Università Politecnica delle Marche
Scuola di Dottorato di Ricerca in Scienze dell'Ingegneria
Curriculum in Ingegneria Civile, Edile e Architettura
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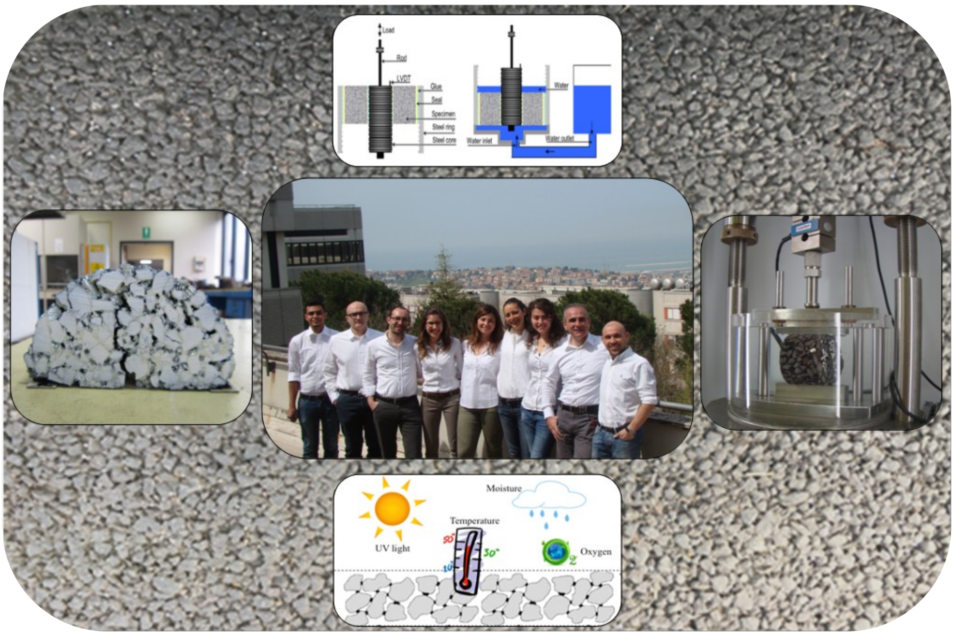
Development of Sustainable (Hot and Warm Recycled) Porous Asphalt Mixtures through Laboratory and Field Investigation

Ph.D. Dissertation of:

Francesca Frigio



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Ancona, January 5th 2017

Abstract

Nowadays, porous asphalt (PA) mixtures are extensively used as motorway surface layers due to their benefits in reducing traffic noise and improving safety during wet conditions. Despite these advantages, PA layers are characterized by limited durability due to the high air void content that makes them more susceptible to ravelling and water damage. As a consequence, maintenance processes in the motorway network are often performed on PA layers leading to a considerable amount of milled material. Moreover, only the use of virgin aggregates is currently allowed in PA mixtures in many countries due to their low durability and delicate volumetric properties. In order to promote the use of Reclaimed Asphalt Pavement (RAP) also in the PA layers, milled material coming from old PA mixtures needs to be stockpiled separately and re-used in PA courses. Given this background, the first objective of this research is to evaluate the feasibility of including milled materials coming from old PA layers as partially substitution of virgin materials without compromise pavement performance.

Furthermore, it must be taken into account that the substitution of virgin aggregates with RAP requires the use of higher production temperatures, due to the presence of aged bitumen. This implies significant energy consumption and, thus, higher production costs as well as environmental issues related to harmful gasses emission. The latest aspect is becoming crucial since most of the production plants are located near urban agglomerations. Urgent solutions are needed in order to solve the energetic and environmental issues without reducing the use of recycled materials. In this sense, the use of Warm Mix Additives (WMA) can represent a valid solution since it allows significant reduction in production temperatures without compromising the workability and compactability properties of asphalt mixtures. In the second part of this experimental study, the feasibility of using different WMA additives available on the market were analyzed involving PA mixtures prepared with 15% of RAP.

Sommario

I conglomerati bituminosi drenanti sono ampliamente utilizzati come strati di usura autostradali grazie ai benefici apportati in termini di riduzione del rumore veicolare e il miglioramento delle condizioni di sicurezza in caso di pioggia. Nonostante i numerosi vantaggi, tali strati sono caratterizzati da una durabilità limitata a causa dell'elevato numero di vuoti che li rende più suscettibili a sgranamento e danni legati alla presenza d'acqua. Di conseguenza, i lavori di manutenzione autostradale sono spesso effettuati su strati drenanti, il che comporta l'accumulo di grandi quantità di materiale fresato (RAP). Allo stesso tempo, occorre considerare che solo gli aggregati vergini sono attualmente utilizzati per la produzione di miscele drenanti a causa delle loro delicate proprietà volumetriche. Al fine di promuovere l'uso di fresato anche in miscele drenanti è necessario che il RAP proveniente da strati di usura aperti sia stoccato separatamente dal resto e ri-utilizzato in tali strati. La prima parte del presente lavoro consiste in un'estesa ricerca sperimentale volta alla valutazione delle prestazioni meccaniche e di durabilità di miscele drenanti in cui i materiali vergini sono stati parzialmente sostituiti da materiale fresato proveniente esclusivamente da vecchie pavimentazioni drenanti.

Occorre altresì considerare che la sostituzione di aggregate vergini con materiale fresato implica l'utilizzo di temperature di produzione maggiori a causa della presenza di bitume invecchiato. Ciò comporta un maggiore consumo di energia e, quindi, maggiori costi di produzione e problemi ambientali legati all'emissione di gas dannosi. Quest'ultimo aspetto sta diventando cruciale dal momento che gli impianti di produzione si trovano sempre più spesso vicino ad agglomerati urbani; soluzioni urgenti sono quindi necessarie per risolvere sia il problema energetico che quello ambientale senza dover ridurre l'uso di materiale fresato nelle miscele. Una possibile soluzione è l'uso di additivi WMA (Warm Mix Additives) al fine di ridurre le temperature di produzione senza comprometterne la lavorabilità né la compattabilità delle miscele. In questo senso, la seconda parte del presente lavoro descrive l'indagine effettuata per valutare la possibilità d'impiego di vari additivi WMA per la produzione a temperature ridotte di miscele drenanti contenenti 15% di RAP.

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Problem statement

Problem statement

The research work described in this thesis focuses on environmental friendly and sustainable solution in designing asphalt mixtures for Porous Asphalt (PA) surface layers.
Porous asphalt mixtures are specially designed mixtures to quickly drain water from the pavements surface thanks to a large number of air voids that are interconnected allowing the water to easily pass through the layer without persisting on the pavement surface during rain fall weather. Hence, the use of PA as pavements surface layers improves safety during wet conditions and enhanced riding quality as well as helps to significantly reduce traffic noise. Due to high number of benefits, PA mixtures are worldwide employed, especially in country characterized by warm wheatear conditions. In particular, the use of PA in the Italian motorway infrastructure had been growing rapidly over the last years achieving the maximum percent possible (83%) considering that the remaining parts are mountain sections or tunnels where the use of PA is not advisable. On the other hand, many disadvantages are often reported for PA mixtures, mainly related to layer durability. PA surface layers are, in fact, characterized by reduced structural durability that can be attributed to raveling distresses and water susceptibility as well as reduced functional durability that is associated to a premature voids clogging that limits their potential to drain water and absorb noise. As a consequence, PA mixtures need frequent maintenance interventions and their durability can be estimated around 10-12 years whereas the typical service life of a dense-graded layer is around 15-20 years. Moreover, the need to use high-quality aggregates and polymer modified bitumen lead to high construction costs compared to dense-graded mixtures.
Frequent maintenance interventions on PA layers lead to a considerable amount of milled material from old PA layers that is growing dramatically. This material consists in valuable and re-usable aggregates since high quality materials are employed for the preparation of PA mixtures. However, Reclaimed Asphalt Pavement (RAP) can be usually re-used as partially substitution of virgin aggregates only in dense-graded mixtures for new base and binder layers. Several national specifications, in fact, currently allow only the use of virgin aggregates in PA mixtures due to their low durability and the difficulties related to the control of the volumetric properties. RAP aggregates, in fact, usually consists in un-fractioned materials from different asphalt layers and the use in delicate mixtures such as PA could compromise their volumetric properties and, thus, their durability as well as their ability to properly drain water and absorb noise. Based on background described, it appears clear that sustainable solutions for the production of useful mixtures such as porous asphalt are needed. At the same time, there is also the need to properly re-employed milled materials without fulfill landfills.
In order to promote the use of RAP also in the PA surface layers, milled material coming from PA mixtures needs to be stockpiled separately and re-used in a PA wearing course. This possibility is confirmed by recent studies (Hagos et al., 2007) that demonstrated that using RAP coming from milled wearing courses do not compromise performance of new high quality wearing courses. As a matter of fact, in Netherland, reclaimed materials are already used in PA layers up to 20% (Hagos et al., 2008). Given this background, the first

objective of this research work is the evaluation to include milled materials coming from old PA layers as partially substitution of virgin materials without compromise pavement performance. It is well known, in fact, that the re-use of milled materials in new asphalt mixtures optimizes the use of natural resources and sustains the asphalt pavement industry since it allows considerable savings in materials, cost and energy. With the increase of demand and limited aggregate and bitumen supply, along with strict environmental regulations, recycling had become a priority goal for both administrations and producers. Thus, a technical support to evaluate any potential deterioration or improvement in performances compared with the mixtures currently used in road construction is necessary.
In case of PA mixtures, the contact between aggregate particles through asphalt mastic occurs in a very small area due to the high void content of the mixtures. Thus, the "affinity" at the bitumen-aggregate interface should be deeply investigated as the new virgin bitumen interacts with the thin film of aged mastic that covers the RAP aggregates. In this sense, the performance of the final mixture will sensibly depend on the adhesion developed between new virgin bitumen and RAP aggregates. Specific mix design attempts, laboratory evaluations regarding adhesion properties, ravelling resistance, indirect tensile strength, fracture propagation and water sensitivity were carried out in order to evaluate if the presence of RAP aggregates could affect PA mixture performance.
In addition, it must be also considered that the substitution of a significant amount of virgin aggregates with RAP requires the use of higher mixing and compaction temperatures, due to the presence of aged bitumen. The bitumen within RAP, in fact, is highly oxidated and, thus, more viscous than a virgin bitumen; as a consequence, higher production temperatures are needed in order to guarantee adequate workability. Higher production temperatures require significant energy consumption and, thus, higher production costs as well as environmental issues related to harmful gasses emission. The latest aspect is becoming more and more crucial since most of the production plants are located near urban agglomerations; thus, urgent solutions are needed in order to solve the energetic and environmental issues without reducing the use of recycled materials.
A possible solution is the use of Warm Mix Additives (WMA) in asphalt mixtures allowing significant reductions of production temperatures without compromise workability and compactability properties during mixing at the asphalt plant as well as the in situ lay-down phase. Hence, both environmental and economic benefits can be guaranteed ensuring also better working conditions due to a reduction of harmful gasses. Such mixtures (usually called warm mixtures) were investigated for the first time in Germany at the late '90s but only during the last decade, the scientific community has been working for their development. As a consequence, limited experimental data are nowadays available concerning warm mixtures performance as well as their effective durability (long term issues). In this sense, WMA technologies and their applicability in asphalt industry need to be further investigated. In particular, possible advantages and disadvantages must be carefully considered with respect to hot mixtures, taking into account both environmental, technical as well as economical parameters. Moreover, it should be taken into account that most of the new produced asphalt mixtures include RAP. The combination of the two above mentioned solutions (RAP and WMA) must be carefully investigated and developed

in order to evaluate the feasibility of warm recycled mixtures. Given this background, the feasibility of using different WMA additives available on the market were analyzed in the second part of this experimental study involving PA mixtures where 15% of virgin aggregates were substituted with milled materials coming from old PA layers. Advanced laboratory tests involving adhesion properties, water sensitivity and the overall durability of the produced mixtures were carried out throughout this PhD program. The positive results obtained using the chemical WMA additives leads to further in-plant studies where the feasibility of in-plant production, workers conditions as well as final mixture performance were properly evaluated.

Finally, on the basis of the abovementioned discussions it can be stated that the present study has a twofold objective: first it evaluates the possibility to include milled materials coming from old PA layers as partially substitution of virgin materials without compromise pavement performance. Moreover, the effectiveness of different Warm Mix Additives (WMA) is taken into account in order to significantly reduce production temperatures of recycled PA mixtures with both environmental and economic benefits. Hence, the present thesis is organized in two separate parts: the first part (*Part 1. Hot recycling of PA mixtures*) describes the attempt to use RAP and increase its content in PA mixtures by means of different mix design studies, laboratory evaluations based on the moisture sensitivity and durability tests as well as in field study throughout experimental sections. The second part (*Part 2. Warm recycling of PA mixtures*) describes laboratory and field evaluations that were carried out to optimize the use of WMA additives in PA mixtures including RAP aggregates.

The overall research activities were carried out in the Department of Civil and Building Engineering and Architecture of the Polytechnic University. Part of the experimental program described in this dissertation was carried out in cooperation with the Modified Asphalt Research Center (MARC) at the University of Wisconsin (Madison, USA) which made available its laboratories to perform the low temperature characterization of the investigated mixtures.

Chapter 1.

Porous Asphalt mixtures

1.1. General Introduction of Porous Asphalt mixtures

Porous asphalt (PA) mixtures consists of Hot Mix Asphalt (HMA) mixtures specially designed to quickly drain water from the pavements surface and are usually employed as surface layers on the top of impermeable bitumen layers. The peculiarity of these mixtures is the large number of air voids (Figure 1.1) that allows the water to easily pass through the layer without persisting on the pavement surface during rain fall weather. The large air void content (around 20%) is created by a substantial modification of the aggregates grading curve from a typical dense graded mixture: a larger percentage of coarse aggregates and a reduced amount of fine particles are typically used for designing an open-graded mixtures in such way that fine particles partially fill the voids in the coarse aggregates but sufficient voids remain in the total aggregates mixture to allow for the bitumen content without clogging up the voids. The aggregates grading curve requirements for porous asphalt layers as well as for bitumen courses, as specified by the Italian technical Specification for motorway are reported in Figure 1.2 as examples.

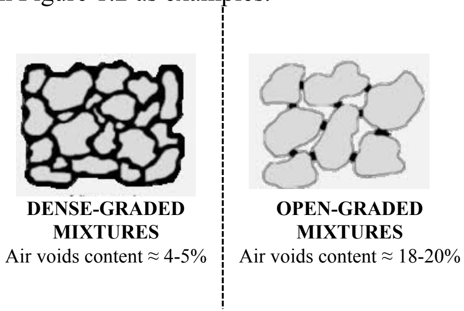
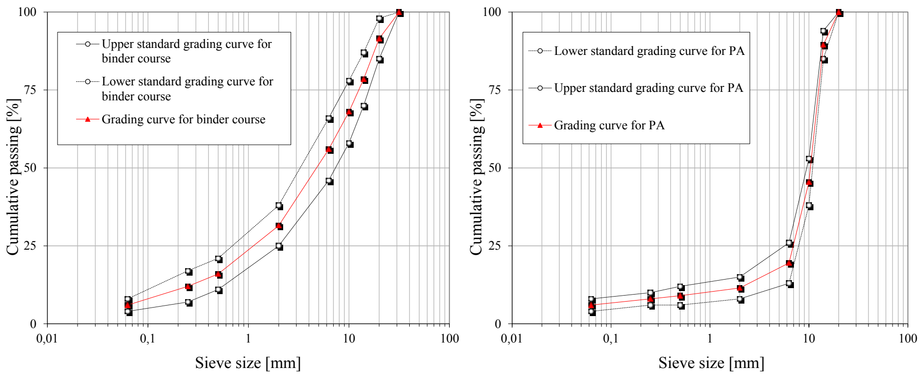


Figure 1.1: Air voids content of dense and open graded mixtures

The presence of a large amount of voids in the mixture implies that the contact between the aggregate particles occurs in very small area and, as a consequence, bitumen phase with enhanced properties is needed in order to hold the aggregates in place during the entire pavement service life. Thus, polymer modified bitumen (PMB) as well as high-quality aggregates are typically used for the production of open-graded mixtures to improve adhesion properties ensuring resistance to disintegration through raveling. Moreover, the use of PMBs increases the mixtures resistance to permanent deformation, durability as well

as resistance to water damage. Most countries also use PMBs in PA mixtures to improve resistance to aging (Nielsen, 2006).
The total bitumen content for PA mixtures is generally slightly higher than that for dense-graded mixtures with the same maximum aggregates size. This implies that PA mixtures are characterized by higher film thickness than dense-graded mixture, since coarse aggregates have low surface area; an high film thickness of bitumen that cover the aggregates particles is needed in PA mixtures in order to improve durability. As a consequence, during construction draindown issues are likely to occur if specific precautions are not taken into account. The use of PMBs helps to minimize draindown but the addition of fibers to the bitumen phase has shown to be the best approach for preventing such a phenomena. Polymer modified bitumens as well as fibers proved the better overall performance without draindown problem (Brown et al., 2009), in particular studies has been proved that the combination of cellulose fibers and styrene-butadiene-styrene (SBS) PMBs led to improved resistance to disintegration (Hassan and Al-Jabri, 2005).



(a) (b)

Figure 1.2: Typical aggregates grading curves for bitumen courses (a) and porous asphalt mixtures (b).

The main advantages of using porous asphalt mixtures as pavements surface layers are related to safety driving conditions during wet weather. In particular, the presence of PA mixtures ensure significant reduction of splash and spray effects (Figure 1.3), risk of hydroplaning, glare and wet skidding as well as enhancements of visibility during rainfalls. In addition, PA mixtures provide improved riding quality and traffic noise reduction effectiveness as compared to dense-graded HMA (Alvarez et al., 2011). As a matter of fact, PA layers allow a reduction of the tire/pavement noise approximately of 3 dB(A) compared to dense-graded HMA. To put a 3 dB(A) reduction in tire/pavement noise into perspective, this reduction also can be achieved by reducing the traffic volume in half (Kandhal, 2002).

Benefits include also improved smoothness that ensures better as well as reduced fuel consumption. Additional environmental benefits can derived from the use of PA mixtures, mainly related to cleaner runoff; Barret (2008), in fact registered smaller concentrations of pollutants related to particulate material and total suspended soils in the runoff obtained from PA surface layers as compared to that of dense-graded HMA.

On the other hand, many disadvantages are often reported for PA mixtures, mainly related to layer durability. PA surface layers are characterized by reduced structural durability that can be attributed to raveling distresses and water susceptibility as well as reduced functional durability that is associated to a premature voids clogging that limits their potential to drain water and absorb noise (Alvarez at al., 2010). As a consequence, PA mixtures need frequent maintenance interventions and their durability can be estimated around 10-12 years whereas the typical service life of a dense-graded layer is around 15-20 years. Moreover, the needs to use high-quality aggregates and PMBs lead to high construction costs compared to dense-graded mixtures. Finally, winter maintenance problems (e.g., black ice formation) are an important disadvantage of PA surface layers; as a consequence, PA mixtures are not usually employed in cold weather regions (Yildrim et al., 2006).

(a) (b)

Figure 1.3: Spray effect during wet weather in case of PA mixtures (a) and conventional dense-graded mixture (b). (Barret, 2008).

1.2. Porous asphalt performance

Several factors play a role in determining PA surface layers performance such as aggregates and bitumen characteristics, mix design, construction variables as well as environment

conditions and traffic loads. In particular, the air void content of the mixture, as a design variable, can be cited as one of the major factor that can affect performance even if all other variables remaining equal. The high air voids content, in fact, allows an easy entrance of water, UV light, oxygen exposing the bitumen of PA mixture to premature aging and extensive moisture damage. Thus, due the inherent nature of PA mixture, environmental conditions are the main cause of the low PA durability associated to premature bitumen oxidation, extensive raveling distress and water damage (Figure 1.4).
Thus, the major disadvantage of porous asphalt is the limited durability with respect to dense graded mixtures due to the high sensitivity of PA mixtures to traffic and climatic loading the damaging effect of water.
In the present paragraph, the main PA mechanical characteristics that determine the overall in service performance are described in details.

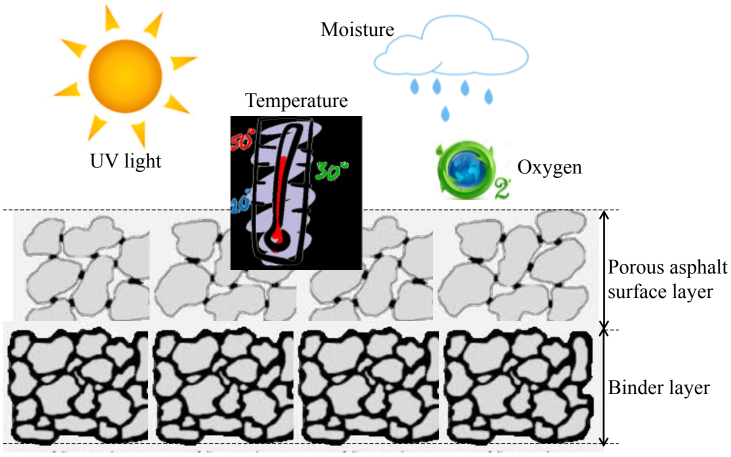


Figure 1.4: Environmental conditions that affects PA layers

1.2.1. Aging characteristics

The large number of air voids that characterized porous asphalt mixtures and the fact that they are interconnected lead to an easy entrance of oxygen, light and water into the pavement structure causing changes in the properties of the bitumen. It is well known that such environmental factors cause oxidation and, thus, aging of the bituminous phase throughout the entire thickness of the PA layer with significant consequences on the long term performance of the mixture.

The aging of bitumen can be defined as a change in the physical properties of the material over time caused by a modification of its chemical composition. It leads to changes in chemical, rheological and mechanical characteristics of bitumen that depend on the bitumen characteristics and bitumen source. The effect of asphalt mixture aging can be classified in two major groups: short and long-term aging (Robert, 1996).
The short-term aging is a very fast process that happens during the production phase of the mixture (mixing, transport and lay down phase) when the thin film of bitumen that coats the aggregates is exposed to temperatures around 140÷160°C. In this phase, an increase in viscosity (between 1.5 and 4 times depending on the bitumen type) can be observed as well as an increase in the asphaltenes content (between 1 and 4% by weight) (Lesueur, 2009).
The long-term aging is a process that the mixture undergoes in the field during the whole in service life mainly caused to climate factors. The effects of this phenomenon depends on the thickness of the bitumen film that coats the aggregates, on the mixtures air voids content and on the pavement layer considered (the surface layers are much more exposed to the aging phenomenon compared to the deeper layers). A key factor that affects the magnitude of the long-term aging is obviously played by the geographic location of the area where the pavement is built since it determines different climate conditions (Lesueur, 2009).
The main mechanisms that contribute to the aging effects development are the oxidation, the volatilization, the steric hardening and the physical hardening. The first two mechanisms cause changes in the chemical composition and are consequently irreversible. On the contrary, the steric and the physical hardening are the results of physical changes in the chemical structure of the material (a re-organization of the molecular chains) and do not alter the molecular composition. Therefore, they can be considered as totally reversible. As a consequence, only the oxidation and the volatilization are usually considered as mechanisms related to the aging process. Thus, aging is usually associated to an initial loose of volatile components during the mixing, transport and lay down phases (short-term aging) of asphalt mixtures and a secondary progressive oxidation in the field (long-term aging). Specifically, the volatilization mechanism manifests as the removal of bitumen substances. In fact, during the production phases of an asphalt mixture (mixing, transport and lay down) the temperature exceeds the boiling point (around 150°C) of some components that consequently leave the bitumen making it stiffer and more viscous. However, the extent of this process depends on the chemical composition of the material and, hence, it is different form bitumen to bitumen. Moreover, the volatilization mechanism is always combined with oxidation reactions that contribute to make the material stiffer. In fact, as the most part of organic materials, the bitumen undergoes a slow oxidation process when it comes into contact with the oxygen. Such a phenomenon is due to the creation of polar groups containing oxygen that tend to associate in molecules with high molecular weight causing an increase in the bitumen viscosity. The oxidation mechanism happens at every temperature and during the whole life of a pavement. It represents the main mechanism associate to the long-term aging.

The speed and the extent of the oxidation mechanism are also strongly related to the chemical composition of the bitumen and the average temperature experienced in the field, besides of the air voids content of the asphalt mixture, the surface roughness, the absorbance grade of the aggregates, the pavement layer thickness, the exposition to the UV rays and the bitumen content that affects the thickness of the bitumen film that coats the aggregates (Robert, 1996).

As far as the chemical composition effects on bitumen are concerned, many full scale experimental sections have been monitored over the years. Each one was built with different types of mixtures, aggregates and bitumen contents (Read and Whiteoak, 2003). Figure 1.5 summarizes the results of these investigations in terms of chemical composition of the bitumen and aging index, defined as the ratio between aged RAP bitumen viscosity and virgin bitumen viscosity at a temperature of 25°C.

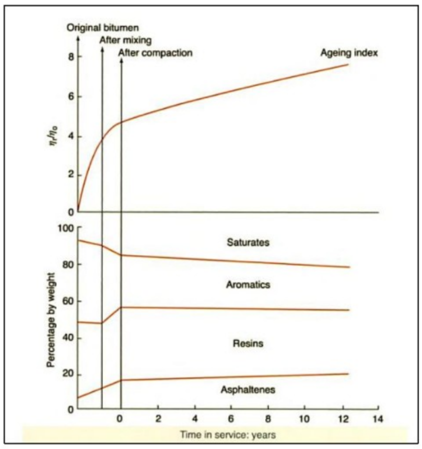


Figure 1.5: Short and long-term aging effect on the chemical composition of bitumen (Read and Whiteoak, 2003)

In terms of chemical composition, it is possible to observe that the asphaltenes content considerably increases during the mixing phase and keep increasing over time, even if at a lower rate. On the contrary, the resins and aromatics percentages decrease over time.

Based on the analysis of the bitumen extracted from RAP, it was also possible to observe that the aging process does not alter the molecules of satures neither those of the asphaltenes, but converts the aromatics into resins and the resins in asphaltenes, with a consequent further increase in bitumen viscosity and stiffness. Another direct effect due to the chemical conversions caused by the aging process is the slight increase in the glass transition temperature.

One of the first study carried out in the Sixties (Corbett and Swarbrick, 1960) showed an increase in the molecular weight of the asphaltenes due to aging, so suggesting the

existence of polymerization reactions and allowing the hypothesis that the asphaltenes produced during the aging process are different than the initial ones. This result was partially confirmed by Kats et al. (1976) who observed for one of the two bitumens studied, the conversion of the asphaltenes into carbenes and carboidi due to a photo-oxidation.
Further studies on the bitumen oxidation (Moschopedis and Speight, 1977) showed that the exposition to the air at 260°C for few hours causes an increment in the asphaltenes content and an increment in their molecular weight, simultaneously. Additionally, other researches (Siddiqui et Ali, 1999) highlighted the linear increment of the asphaltenes over time due to the oxidation caused through the Rolling Thin Film Oven Test (6-7% increment after 340 minutes).
Moreover, the aging process causes significant changes in the rheological properties of bitumen. One of the major parameter used to evaluate the modification caused by aging is the viscosity, which undergoes alterations more or less evident depending on the aging extent. In that sense, already some years ago, the analysis of the bitumen viscosity at ambient temperature as function of the exposition time of thin films of bitumen (3.2 mm) to a temperature of 163°C (Traxler, 1961) showed the linear increment of viscosity over time, up to 15 hours of heating. The increments recorded varied between 5 and 20 times, depending on the bitumen origin. On the contrary, aging time longer than 15 hours induced lower viscosity increments, that allowed to model the viscosity over time by an hyperbolic interpolation line (Bell, 1989).
As far as the bitumens behavior as function of the asphaltenes content is concerned, it was demonstrated (Petersen, 1993) that bitumens with high asphaltenes content show different behaviors depending on the aging temperature applied. On the contrary, bitumens with a lower asphaltenes content seem to behave independently to the oxidation temperature (Figure 1.6 – A).
Such a behavior could be interpreted (Petersen, 1993) considering that at lower temperatures (around 60°C), bitumens with high asphaltenes content can easily aggregate, so limiting the possibility for the oxygen to penetrate the bitumen and, subsequently, the aging process to take place. On the contrary, at high temperatures (around 130°C), the higher dispersion of the solid particles of bitumen allows an easy penetration of the oxygen and, hence, a subsequent higher degree of aging (Figure 1.6 – B). In bitumens with low contents of asphaltenes the dispersion of the particles is always guaranteed. Thus, their behavior is independent to the aging temperature.

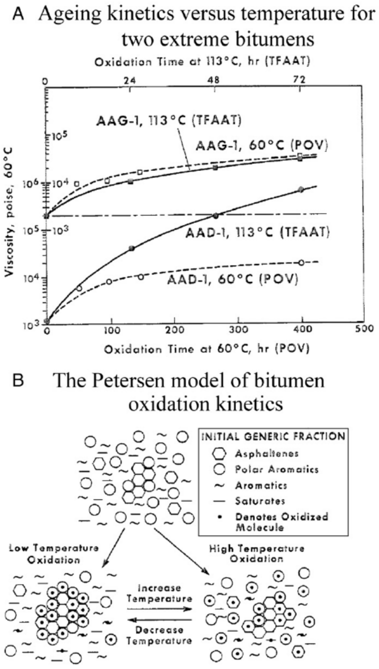


Figure 1.6: Aging model of bitumen (Petersen, 1993)

From a rheological point of view, it was observed (Bahia and Anderson, 1995) that the trends of the complex modulus G* and the phase angle δ of some bitumens subjected to oxidation in laboratory by applying different aging durations and aging temperatures are significantly different than those of unaged bitumens (Figure 1.7). In particular, within the frequency and temperature range typical of road applications, the general effect of aging can be summarized as an increment in the complex modulus and a reduction in the phase angle.

The increment in the complex modulus implies an increase in the stiffness and, hence, a lower aptitude to accumulate permanent deformations under traffic loading. The benefits in terms of rutting are balanced by the negative impact induced in terms of fatigue and thermal cracking. On the contrary, the reduction in the phase angle allows different analysis to be drawn. In fact, the tangent of the phase angle represents the ratio between the dissipated energy and the storage energy at each loading cycle. Therefore, a reduction of such a parameter indicates a lower amount of dissipated energy in comparison to the storage energy. Therefore, due to the oxidation process the changes in the phase angle make the bitumen more resistant in terms of fatigue and rutting, but more sensible to the effects induced by the thermal strains since the tensile tension accumulated after each loading cycle tends to increase due to the decrease in the dissipated strains (Bahia and Anderson, 1995).

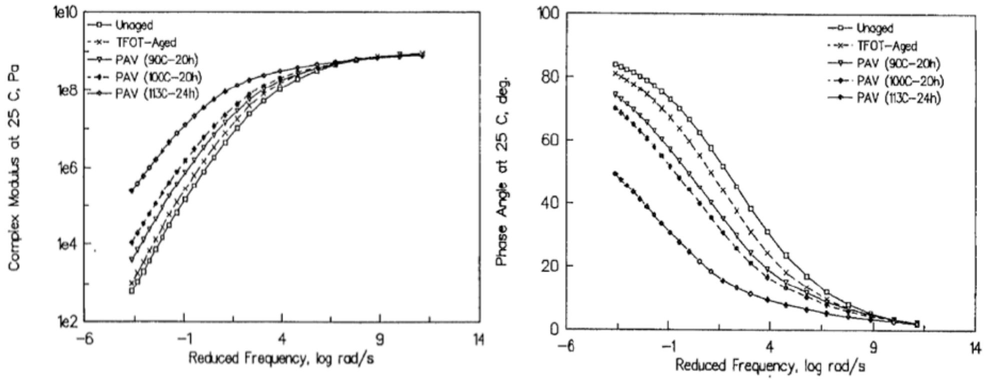


Figure 1.7: Aging effect on the complex modulus and phase angle master curve (Bahia and Anderson, 1995)

As far as PA mixtures are concerned, the premature aging of bitumen component has been demonstrated by many studies (Voskuilen et al., 2004; Hagos et al., 2007). In particular, Voskuilen et al. (2004) showed that the effect of aging in PA mixtures results in a dramatic change of the bitumen properties in the first few years of the pavement life. Moreover, Hagos et al. (2007) compared the aging effects on artificial laboratory aged bitumens and bitumen recovered from field core specimens in a PA surface layer. Results showed that the standard bitumen aging is not the same as the aging of PA layers in the field. The laboratory long term aged bitumen properties were comparable to the 1 year aged material in the field in case of PA whereas it simulates the aging of bitumen in dense asphalt pavements after 10 years. Such an outcome showed that a new aging method is crucial for PA since they undergo an excessive aging damage that affect the overall mixture performance. In this sense, Hagos et al. (2007) evaluated that aging has a positive influence on PA performance at intermediate and high temperatures since it minimizes possible drainage of the bituminous mortar and improves the mixture stability. On the other hand, aging increases the stiffness of the binding material (mastic/mortar) and reduces the relaxation potential of the bitumen that is a fundamental factor to enhance PA performance in terms of raveling at low temperatures as well as. In reality, they found out that the initial increase in PA mixtures stiffness was followed by a decrease in stiffness with time by testing the field cores by means of Indirect Tensile tests. The most likely explanation for such a behavior was attributed to the aging effect and subsequent damage development in the binding material and/or stone-mastic contact points. Aging considerably increases the stiffness of binding material during the initial periods of the pavement life due to high aging rate. In fact, during the initial periods of the pavement life, traffic loadings are sustained because the bituminous mortar has sufficient stiffness and stress relaxation potential (flexibility) until the critical aging of the bitumen is reached. With time, the increase in stiffness of the aged bitumen/mastic allows stress relaxation mainly through development of damage (cracking) in the material. Such damage development is critical at low temperatures, which is also consistent with observation of the occurrence of raveling in the field. Thus, the decrease in stiffness modulus of the asphalt mixtures could be associated with damage propagation, which overcomes the effect of aging on the mixtures stiffness. The damage in the mixture causes the aggregates at the surface to ravel, because the surface aggregates are with insufficient support in the mixture and more susceptible to raveling (Hagos et al., 2007).

1.2.2. Raveling resistance

Raveling is defined as the loss of aggregates from the pavement surface and it is a common distress mainly related to traffic loads and climate conditions. Of course, such a distress involves solely the pavement surface layer and is one of the main causes of the reduce durability of porous asphalt layers (Figure 1.8). Environmental conditions and the action of traffic are responsible for raveling of porous asphalt. Environmental effects cause aging of the bitumen due to reaction with the atmospheric air resulting in hardening of the bitumen and the action of water that weakens the bond between bitumen and aggregate resulting in stripping of the stones. The raveling of the surface of porous asphalt is further aggravated by the action of traffic.

Raveling of a HMA pavement surface is usually caused by a combination of various factors such as low bitumen content, insufficient amount of fine aggregate matrix to hold the coarse aggregate particles together, high air voids content, excessive aged/ brittle bitumen phase and lack of adhesion between bitumen and aggregates particles (Brown et al., 2009). Thus, it appears clear that both the mix design and the quality of the mixtures components as well as factors related to the quality of the construction work can easily induce premature raveling distresses. Those factors includes workmanship such as lack of compaction that results in excessive air voids contents or weather conditions during the construction phase that can lead to insufficient mixing and compaction temperatures with consequent lack of compaction or reduced bond between bitumen and aggregates.

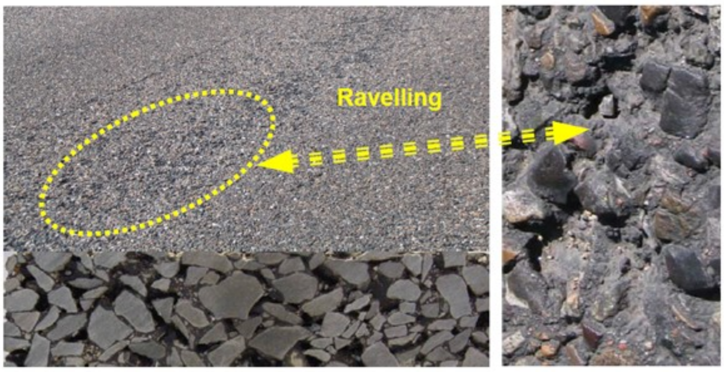


Figure 1.8: Example of raveling distress in a PA surface layer (Hagos, 2008)

As mentioned earlier, the aging of the bitumen cause a progressive increase in brittleness of the bitumen that affect the bond with the aggregates inducing raveling distresses. Moreover, high air avoids content accelerate such a phenomena since it allows an easy entrance of UV light, water and oxygen. Many studies demonstrated a close relationship between air voids content and the extent of raveling distress (Frigio et al., 2013). Such a relationship is not only attributed to the aging effects of bitumen but it is also associated to the fact that the contact between aggregate and bitumen occurs in a small area in mixtures characterized by high air voids content. Moreover, water can easily entry in mixtures pores and weakens the bond between aggregates and bitumen; as a matter of fact, raveling is often considered as a moisture related distress. Thus, it is evident that PA mixtures are extremely prone to such a distress due to the structural nature (high air voids content).

The progressive loss of surface material (raveling distress) can be caused by two different mechanisms that affect the bitumen/aggregate bond: adhesive or cohesive failure. The adhesive failure is associated to the loss of adhesion at the interface between aggregates and the bitumen/mastic phase whereas the cohesive failure occurs within the bitumen/mastic phase (Figure 1.9). The nature of the failure depends on the nature of the mastic and the relative thickness of the mastic around the coarse and the fine aggregate. Usually, asphalt mixtures with thin bitumen films fail in tension by adhesive bond rupture whereas thick bitumen/mastics films fail because of damage within mastic as opposed to interfacial debonding. The thickness that differentiates these two types of failure is dependent on the rheology of the bitumen or the mastic and the amount of damage that it can withstand before failure, the rate of loading and the temperature (Little and Jones, 2003). Kim et al., (2002) demonstrated that the rate of damage and the amount of damage that mastics can accumulate before failure depend on the nature of the mastic. In particular, mastics with proper amount and type of filler can resist to more damaging than unfilled systems as well as polymer-modified mastics can accommodate more damage before failure than

unmodified systems. Thus, a proper mastics mix design is essential to ensure mixture resistance to raveling.
Also, Mo et al., (2010) confirmed that adhesive failure and cohesive failure are the failure mechanisms within the stone contact and the weak link is responsible for raveling. In particular, adhesive failure is predominant at low temperatures whereas cohesive failure is the main cause at high temperatures. Moreover, they pointed out that although raveling is a surface defect, the pavement structure itself has a great influence on raveling resistance since this is reflected by the deflection of the pavement under load. In fact, raveling resistance at high temperatures strongly depends on the confining stresses that follow from the pavement deflection. However, the tensile strains due to the combined effect of pavement deflection and thermal contraction are the main cause for raveling at low temperatures (Mo et al., 2010).

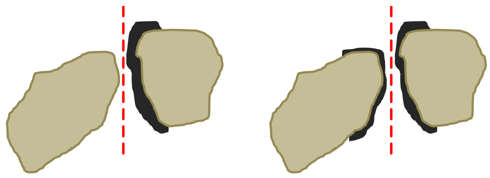


Figure 1.9: Failure mechanisms in case of raveling: adhesive and cohesive.

Given this background, it is essential to design PA mixtures characterized by improved raveling resistance. In this sense, many studies have been carried out with the aim at investigating the PA performance in terms of raveling and the technique to improve their resistance. (Mo et al., 2009; Mo et al., 2010). In fact, the raveling of the PA surface decreases the noise reducing potential of the pavement layer and threatens the technical durability of the surface layer. As a consequence, it requires early maintenance interventions that increase the costs and imply frequent interruption of traffic flow. In other words, there is a high demand for durable PA surface courses. An extensive study was carried out by Voskuilen et al. (2004) on Dutch motorways where the service life of 26 Porous Asphalt test sections was monitored. The projects involved mixtures prepared with different bitumen contents and bitumen type (unmodified and PMBs). The observation of the raveling distresses development showed that the use of 4.5% polymer modified bitumen did not ensure any life extension in comparison with standard PA even if polymer modifications allow the use of a higher bitumen content without segregation problems, which in general increases the service life. On the other hand, the increase in bitumen content was found to ensure better durability of the monitored PA (PA prepared with 5.5% bitumen content, both unmodified and PMB, generally acted better).

1.2.3. Moisture sensitivity

Moisture damage can be defined as the loss of strength and durability in asphalt mixtures due to the effects of water. Such a phenomena can occur because of a loss of bond between bitumen or mastic and the aggregates particles or because the moisture permeates and wakens the mastic phase, making it more susceptible to moisture during cycling loading.
The presence of water in the pavement is mainly due to infiltration through the pavement surfaces and shoulders, melting of ice during freezing/thawing cycles, capillary action, and seasonal changes in the water table (Yilmaz and Sargin, 2012). In the case of PA mixtures, the water can easily infiltrates from the surface pores and pass through the entire layer, making the mixture highly susceptible of water damage. Moreover, as mentioned before, the stone-stone contact occurs in a small area in PA mixtures due to the high air voids content. Thus, the presence of water can easily causes a loss of bond at the bitumen/aggregates interface leading to adhesive or cohesive failure.
Literature reports at least five different mechanism of stripping related to moisture damage (Little and Jones, 2003):
- detachment: is a retraction of bitumen along the surface of the aggregates due to introduction of water at the bitumen/aggregate interface. The theory able to explain displacement relates to the thermodynamic equilibrium of the three-phase bitumen-aggregate-water system. When such a system exists, water reduces the free energy of the system more than bitumen to form a thermodynamically stable condition of minimum surface energy, meaning that the aggregate has a strong preference for water over bitumen. In fact, water have highly polar molecules that can easily replace the bitumen at the aggregate/bitumen interface.
- <u>displacement</u>: involves displacement of the bitumen at the aggregate surface through a break in the bitumen film. The source of the break may be incomplete coating of aggregate surface, film rupture at sharp aggregate edges, pinholes in the bitumen film etc…
- <u>spontaneous emulsification</u>: is an inverted emulsification of water droplets in the bitumen phase. It occurs when bitumen films are immersed in water and its rate depends on bitumen nature and the presence of additives.
- <u>pore pressure</u>: occurs when water is entrapped in the mixture pores. Stresses imparted to the entrapped water from repeated traffic loads (Figure 1.10) worsens the damage as the continued buildup in pore pressure disrupt the bitumen film from the aggregate surface or cause the growth of microcracks in the bituminous mastics.

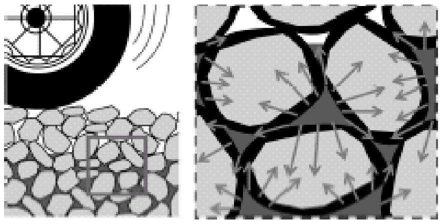


Figure 1.10: Pore pressure

Terrel and Al-Swailmi (1994) described the concept of pessimum air voids content between 8% and 10% (Figure 1.11). Above this level, the air voids become interconnected and moisture can flow out under a stress gradient developed by traffic loading, as in PA mixtures. Below this value, the air voids are disconnected and are relatively impermeable and thus do not become saturated with water. In the pessimum range, water can enter the voids but cannot escape freely and is, thus, subjected to pore pressure buildup upon repeated loading.

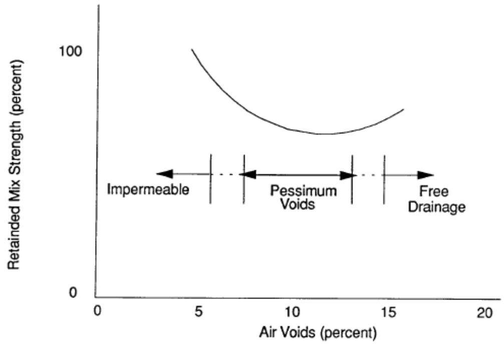


Figure 1.11: Relationship between strength of mixtures and air void content (Terrel and Al-Swailmi; 1994)

Other studies highlighted that also the voids structure in terms of size, distribution, connectivity and tortuosity of the flow path influence the moisture sensitivity. Chen et al. (2004) classified air voids in asphalt mixtures into three categories: effective, semi-effective and impermeable (Figure 1.12). However, the identification of these different types of air voids in laboratory or field samples is difficult because of the complex internal structure of the material and the limited ability to explore its interior composition.

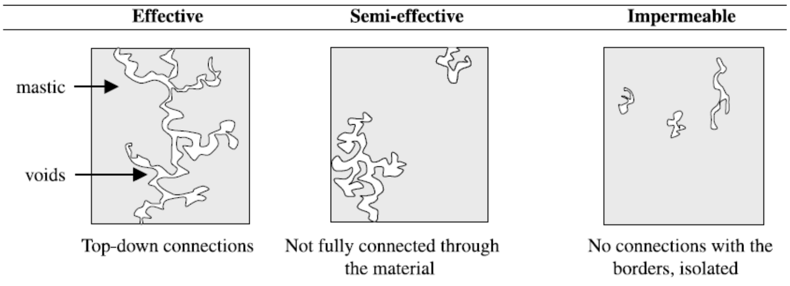


Figure 1.12: Air voids classification in asphalt mixtures (Chen et al., 2004)

- hydraulic scour: occurs at the pavement surface where stripping results from the action of tires on a saturated surface. Water is sucked under the tire into the pavement by the tire action. Osmosis occurs in the presence of salts or salt solutions in aggregate pores and creates an osmotic pressure gradient that actually sucks water through the asphalt film.
- pH instability: the adhesion at the bitumen/aggregate interface is strongly influenced by the pH of the contact water. In particular, the pH of contact water affects the value of the contact angle and the wetting characteristics at the aggregate–asphalt interface region.

Moreover, Terrel and Al-Swailmi (1994) identified other additional factors that have a profound effect on the durability of HMA mixtures such as water from precipitation of groundwater sources, temperature fluctuation that can includes freeze-thaw conditions and aging of the bitumen. Also traffic and construction techniques, that are external to the environment, were found as important factors in terms of durability.
The causes that contribute to moisture sensitivity problems in asphalt pavements are various (Little and Jones, 2003). First of all, it is important to take into account the moisture sensitivity of the aggregates during the mix design process, particularly for mixtures that are significantly affected by water damage such as PA mixtures. In particular, the aggregate surface chemistry and the presence of clay fines are important factors affecting the adhesion between aggregate particles and bitumen. Common methods of combating these factors are through the use of antistrip agents and by the elimination of detrimental clay fines through proper processing or specification. Also the bitumen chemical properties can influence both the adhesion with the aggregate and the cohesion within the mastic. Adhesion is influenced by the chemistry of the bitumen as well as by its stiffness. The cohesive strength of the bituminous matrix in the presence of moisture is also influenced by the chemical nature of the bitumen and processing techniques.
Moisture-related problems do not occur without the combined presence of water and traffic, which provides energy to break the adhesive bonds and cause cohesive failures. Repeated freeze–thaw cycles can also accelerate the distress in the pavement. Moreover, pavement design considerations as well as construction issues must be taken into account since

pavements may have fundamental design flaws that trap water or moisture within the structural layers. There must be good drainage design, both surface and subsurface, since water causes moisture-related distress. Weather conditions during construction are important as they can affect mix compaction or trap mix moisture. Mixture handling techniques can influence segregation and affect the permeability of the mixtures. Joint construction techniques can also affect compaction and permeability. Finally, the amount of compaction achieved (relative density) has a major effect on the air void content, the permeability of the finished pavement, and the mixture sensitivity to moisture damage.

The major pavement distresses that can derived from moisture damage are:

- stripping: debonding of bitumen and aggregate particles;
- rutting: surface depression along wheel path (Figure 1.13a);
- cracking (Figure 1.13b);
- raveling: progressive disintegration of the surface layer (Figure 1.13c);
- localized failure derived by a progressive loss of adhesion between bitumen and aggregate or cohesion within the bitumen phase (Figure 1.13d).

(a)

(b)

(c)

(d)

Figure 1.13: Types of distresses associated to water damage

1.3. Advantages and disadvantages of PA

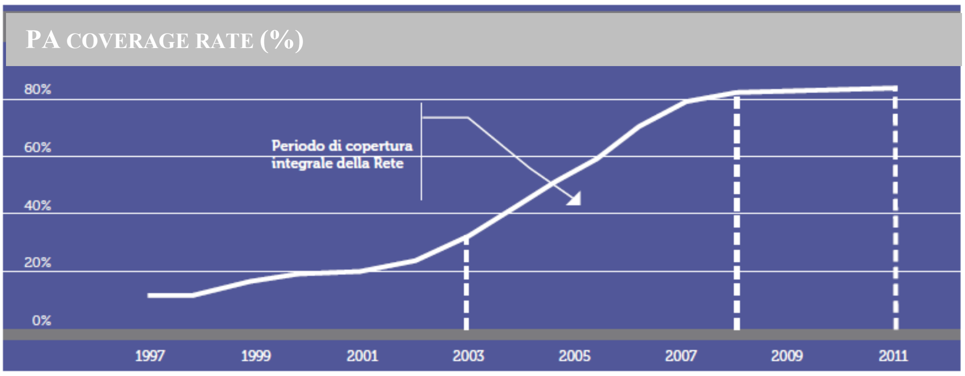
The main advantages as well as disadvantages related to the use of porous asphalt mixtures as surface layers are listed in this paragraph, based on the mixtures characteristics that have been described in the previous part.
The advantages of PA are related to improvements in three basic areas: safety, economy and environment (Alvarez et al., 2006). The use of PA can improve traffic safety, especially under wet conditions since the material is highly permeable allowing direct flow of water from the surface to the bottom of the layer. This is one of the most important advantages of PA and constitutes a substantial difference from dense graded mixtures that require longer times to disperse water from the pavement surface and facilitates accumulation in low areas. It is well known that water accumulations at the surface limit or eliminate the contact between the tire and the pavement leading to hydroplaning and, as a consequence, loss of control for braking and steering. Moreover, drainage of water during rainfall minimizes spray and splash phenomena that contribute to reduced visibility. Spray is related to very fine water particles from pavement surfaces generated by rolling wheels and vehicle bodies that advance on wet pavements whereas splash is related to the coarser water particles created when rolling wheels move over pools in poorly drained areas.
Further benefits in wet conditions is the improved wet frictional resistance compared with dense graded mixtures layers, as reported by Kandhal (2006). In particular, higher wet skid resistance is reached at high speed on porous mixtures compared to that on wet dense graded mixtures, leading to reduction in accidents under wet conditions associated with the use of PA. However, at low speed, differences in response of these two types of mixtures are not noticeable.
An additional advantage in terms of traffic safety is the glare reduction that can constitute an issue, particularly at night. PA layers have the ability to mainly diffuse reflection both during darkness and daylight, improving visibility of road markings during the night and day, which is affected by the presence of reflected light.
Economic benefits are mainly related to reduction in fuel consumption on the order of 2 percent due to enhanced smoothness. In addition, reduction in the rate of tire wear on PA was suggested based on a decrease in tire stresses generated by the improved macrotexture of this type of mixture (Khalid and Pérez, 1996)
As far as environment is concerned, the ability of PA mixtures to reduce noise at the tyre-pavements interface becomes an important advantage to reduce or control highway noise levels. This aspect has widely motivated the use of porous mixtures in Europe. Kandhal (2004) summarized a comprehensive set of studies including information from several European countries and Canada and reports that the use of PA mixtures allows a noise reduction in the range of 3 to 6 dB(A) with respect to standard dense-graded mixtures. Moreover, higher driver comfort levels can be achieved with the use of PA, since noise reduction is perceived not only outside the vehicle, but also inside.

In addition, PA mixtures can be considered an environmentally friendly product because crumb rubber from old tires can be integrated into the bitumen to produce this type of mixture.
Finally, cleaner runoff is produced when PA surface layers are used than that obtained from conventional dense-graded mixtures since lower total suspended solids, total metals, and chemical oxygen demand are reported. The benefits obtained from the use of PA mixtures are comparable with those attained from a vegetated filter (Kearfott et al., 2005).
As far as disadvantages are concerned, the main issues related to the use of PA are reduced performance, high construction costs, winter maintenance and minimal structural contribution. The performance reduction involves both durability and functionality issues. As already mentioned, durability issues are mainly associated with raveling as well as water susceptibility due to the high air voids content. As for functionality, accelerated loss of permeability and noise reduction capacity due to clogging of pores is the main concern for these mixtures.
Construction and maintenance costs are usually considered higher for porous asphalt when compared with dense graded mixtures. The higher construction cost is mainly related to the use of polymer modified bitumen that needs to be incorporated in PA mixtures. Moreover, frequent maintenance and rehabilitation processes need to be taken into account to maintain the pavement functionality and ensure good service performance. In particular, winter maintenance is considered a significant issue for PA since they have a tendency to cool faster than dense graded mixtures leading to earlier exhibition of frost and ice formation. Therefore, larger amounts and more frequent application of deicer agents and higher care in the homogeneity of the application are required. Moreover, the lower temperatures and greater air voids of PA allow water to become trapped more easily and freeze more quickly than other pavement surfaces. This is known as black ice, and it is a serious road hazard for drivers. Sand and salt are not effective on PA surfaces. Sand clogs the air voids eliminating their special benefits. Salt drains away too quickly within the open-graded structure of the pavement, proving ineffective against ice (Yildirim et al., 2006).
As far as structural contribution is concerned, for pavements structural design PA mixtures are typically considered to have no or minimal structural contribution.

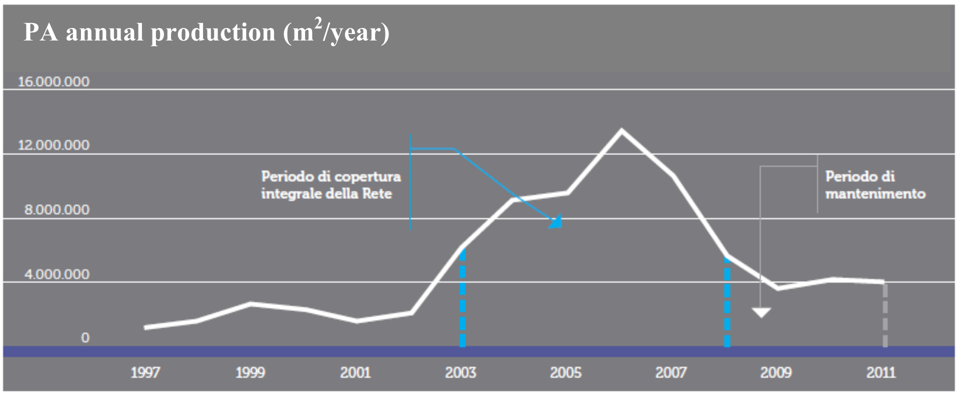
1.4. The use of PA in Italy, Europe, USA

Porous asphalt mixtures are worldwide employed as drainage surface layers as well as noise reducer. In particular, noise reduction capacity of PA surface layers has prevalently motivated the use of porous mixtures in Europe, while in the United States the safety improvements under wet conditions have been the primary motivation (Alvarez et al., 2006). Of course, PA mixtures are mainly employed in warm weather countries due to the winter maintenance issues.
As far as European countries are concerned, a common agreement on PA design procedures was never reached since the first developments at the University of Cantabria in Spain.

Nevertheless, this has not stopped the development of porous asphalt mixes. The European countries characterized by main PA construction rate are Denmark, The Netherlands, Australia, Belgium, Switzerland, Great Britain, Spain and Italy. In particular, the use of PA in the Italian motorway infrastructure had been growing rapidly over the last years: the percentage of PA mixtures as surface layer in the motorway grew from 14% of 2003 to 83% of 2008 (Figure 1.14). Such a percentage can be considered as the maximum possible taking into count sections under construction or rehabilitation as well as section where the use of PA is not advisable (i.e. mountain sections, tunnels).



(a)



(b)

Figure 1.14: Use of PA in Italian motorway: percentage of use over the years (a) and annual production

Part 1. Hot recycling of PA mixtures

Chapter 2.

Literature review: hot recycling

Traffic loads as well as climate agents causes distresses in road pavements during their entire service life. Therefore, pavements require constant rehabilitation and maintenance activities that in turn cause production of large quantity of milled material. In order to avoid the disposal of huge pilots of milled materials in landfills, over the last decades the most industrialized countries have developed production techniques of new asphalt mixtures that allow the re-use of old pavements. As a matter of fact, the design of asphalt mixtures with high Reclaimed Asphalt Pavement (RAP) content as well as the performance evaluation of recycled mixtures has been a challenging topic for researchers all over the world (Al-Qadi et al., 2007; Al -Qadi et al., 2012; Li et al., 2008; McDaniel et al., 2000; Sondag et al., 2002). Although experimental studies were not sufficient to fully characterize such a complex topic as hot recycling, they constitute an important reference to identify the main issues related to the re-use of milled material.

It is important to underline that recycling is not a new concept. It started to become relevant in the Seventies when the oil crisis involved all the Western countries and forced them to renounce to the oil supplied by OPEC. During that period, many productive fields tried to find alternative energy supplies and new technologies to limit the use of oil. In road market, such a necessity arose again in the last two decades, when the companies had to deal with a big increment of the oil price along with a low availability of aggregates of good quality. In that sense, the need to decrease the use of virgin aggregates had as direct consequence the re-use of the material coming from the milling of old pavements, allowing recycling to become a good and convenient alternative. Figure 2.1 shows a summary concerning recycling practice of the main European countries along with the conventional percentage of RAP employed as a function of the pavement layer considered.

In this paragraph, an extensive literature review of the main experiences concerning the different RAP techniques available as well as the effect of RAP on mixture performance is provided. Moreover, the state of the art related to the use of Reclaimed Asphalt Pavement (RAP) in Europe and USA is reported and detailed described in the present chapter.

Country	Hot technique	Max RA content in base / binder layers	Max RA content in surface layers	Cold technique
Belgium (F+W)	Mainly	50% binder homogeneous RA 20% binder heterogeneous RA 20% binder RA in EME (Enrobé à Module Élevé)	prohibited	20% in base layer
Czech Republic		25 to 40% when fresh bitumen pen 30 to 70 60% for softer grade	No	Foamed bitumen Emulsions and Cement
France	Mainly	15% with no testing on national network. Up to 40%	Up till 10% with limited restriction and till 40% with restriction on the binder content	Emulsions or foamed bitumen
Germany	Mainly	20% in binder course Up to 100% in base	20% (Direct-Mat report states 100%)	Emulsions or foams Recommended for tar contaminated RAP
Italy	Mainly	Never above 50%, usually below 30%	No, except for special wearing course – below 20%	Mainly emulsions but foam increase Higher % RAP allowed
Netherlands	Mainly	50% in base layer 50¨% in binder layer 50% in DAC surface layers	50%	
Sweden, Finland				
Norway	Mainly	No Limit1	No limit	Emulsions and foamed bitumen
Denmark	Mainly, but a large percentages goes to unbound application	5 to 10% with no testing (Direct-Mat report states 100% for base course and 30% for binder course)	5 to 10% with no testing (Direct-Mat report states 30%)	Almost none.
Spain		10% with no testing 11% -50%	Not permitted	100% RAP in base layer. Emulsions

Figure 2.1 Use of the recycling techniques in Europe (de la Roche et al., 2012)

2.1 Possible issues related to the use of RAP

The material coming from demolition and milling of old asphalt pavements is commonly referred as Reclaimed Asphalt Pavement (RAP) and can be re-inserted in the production process of new asphalt mixtures (Copeland, 2011). In order to obtain a recycled asphalt mixture that guarantees suitable characteristics needed to consider the material acceptable according to the current technical specifications, several studies highlighted the necessity to address the following issues (Copeland, 2011; Ipavec et al., 2012):

- quality control of the material and the production process adopted;
- characterization of RAP (according to the European standard EN 13108-8):
- amount and type of external materials eventually included in the RAP;
- type and age of the bituminous component;
- average bitumen content;
- type and grading distribution of the original aggregates;
- nominal maximum aggregate size;
- homogeneity;
- type and percentage of the virgin bitumen added.

The recycling technique chosen and the properties of the materials that compose RAP are of fundamental importance to obtain good recycled mixtures that can guarantee suitable

performance in the field. In particular, recycling techniques can be divided in two main categories: hot and cold recycling. In this literature review, only the hot recycling technique is addressed describing the main experiences collected on this subject, in order to provide a comprehensive picture of the current state of the art.
The hot recycling process for the production of new asphalt mixtures requires that aggregates and bitumen are heated at high temperatures prior to be mixed together with milled materials. Usually, milled materials constitute a percentage of the total amount of materials around 10 to 50%. Two different production methods can be adopted:

- in plant recycling: milled material is moved from old pavement location to the plant site where it is processed through crushing and screening to discard foreign matters and clumps too large. Then, it is usually sieved and divided in more fractions prior to be re-used. Thanks to the higher possibility to monitor and control the milled material prior to be re-introduced in the production process, this method avoids high variability of RAP and ensures a better quality and a more uniform homogeneity of the final mixture. Moreover, it allows the advantage of having higher flexibility in the production sequences that can be adapted according to several requirements and different types of mixture. At the same time, this method has few drawbacks mainly related to the higher costs due to the transport and the stocking of the milled material in the plant where it is necessary to ensure a big amount of appropriate spaces;
- in place recycling: all production activities (milling, mixing, compaction) are realized directly in the field by means of specific equipment. Minor quality of milled materials as well as lower control of the production process have to be accounted for. However, these drawbacks are balanced by higher economic benefits and shorter production time. The so produced mixtures are usually employed to build binder or base layers, depending on type and volume of traffic.

The choice to adopt the in plant production process is mainly determined by the following factors:

- type of application: the in plant recycling is usually taken into account when the recycled material is used to build a new layer of the pavement;
- in site material: since high quality and good properties are required for surface layers, when they are produced with milled material, the variability usually related to RAP can justify a pre-treatment that can be realized only in plant.

Another aspect to be mentioned, strongly associated to the production method adopted, is the water content of RAP. As a matter of fact, RAP humidity represents one of the most important characteristics to be checked when RAP is re-used since it can negatively affect the overall quality of final mixtures.
In fact, a pavement is subjected to maintenance and rehabilitation activities when its distresses are already significant and compromise the integrity of the surface, hence allowing the access of the water inside the pavement structure. In particular, in case of in place recycling, RAP is used right after the milling in the exact conditions that it has in situ.

Therefore, it is necessary to control that the water content in the old pavement is able to guarantee the goodness of the re-building activity.
On the other hand, production the initial water content of RAP is less important when in plant production is considered since it can be easily monitored over time and varied if needed. In fact, once RAP is moved to the plant site, it can be managed and preserved as everyone else traditional aggregate fraction. However, it is necessary to remind that milled materials tend to retain humidity without allowing the water to progressively drain as usually happens for virgin aggregates. Therefore, also in plant the water content of RAP must be regularly monitored and the contact between RAP and water should be avoided (for example stoking the material in a covered space).
As initially highlighted, the quality of recycled asphalt mixtures is also affected by the homogeneity of RAP, especially in terms of type and dimension of original aggregates and type of the original bitumen.
The ideal production process should allow the use of RAP coming from only a single source (intended as pavement layer). Such a precaution can be realized by stocking the material coming from different layers separately. This is particularly important since different layers of a pavement are made with different materials in order to obtain specific characteristics. For example, the surface layers are usually composed by bitumen with improved quality and aggregates of smaller size (Al-Qadi et al., 2007; Copeland, 2011; de la Roche et al., 2012). Such a practice requires efforts in terms of logistic organization and costs (time and a lot of spaces for the stockpiles) and it is difficult to realized; hence, milled materials coming from different locations and pavement layers are usually mixed together.
In any case, in the attempt to increase the homogeneity of milled materials, a previous fractioning and sieving of RAP is fundamental. Obtaining several RAP sizes (e.g. 0/4 mm and 4/11 mm or 0/8 mm and 8/16 mm) should be recommended also for the in plant production, since it helps to meet easily the aggregate grading curve requirements (de la Roche et al., 2012). Such a precaution can also encourage the use of higher RAP content in new mixtures since it is believed that the fractioning in more sizes enhances the integrity and the homogeneity of RAP with positive consequences on the interaction with the virgin aggregates (Copeland, 2011).
Among the aspects initially mentioned, other two factors are of fundamental importance in the production of optimum hot recycled mixtures: the aggregate gradation of the recycled mixture and the type and quantity of the virgin bitumen added.
Specific investigations about these two fundamental aspects are needed prior to produce recycled mixtures. In particular, one of the major concerns is the degree of blending between the virgin bitumen and the aged bitumen coming from RAP and the effects of the blending on properties of the final bituminous component of recycled mixtures. The degree of blending can significantly affect performance of asphalt mixtures and hence, it determines type and quantity of virgin bitumen to be used as well as the maximum amount of RAP that can be included in a recycled mixture. Due to the fundamental importance of this aspect, a specific paragraph of the literature review hereby presented is completely dedicated on it (see § 2.4).

2.2 State of the art in Europe

2.2.1 The re-use of RAP

Recycling techniques applied to the pavement industry are a common practice in most of the European countries (de la Roche et al., 2012). In this sense, the statistical data available highlighted that the production of new asphalt mixtures in 2008 allowed the recycling of 57% of all produced RAP. Moreover, the statistical data demonstrated the most used technique was the hot recycling method.

An approximate estimation of the potential re-use of RAP in Europe can be easily guessed taking into account that, during the last decade, the total quantity of Hot Asphalt Mixtures (HMA) and Warm Asphalt Mixtures (WMA) was about 320-350 Mt/year and the total quantity of RAP produced was 50 Mt/year. Therefore, hypothesizing a re-use of RAP equal to 15% in each new mixture produced, it would be theoretically possible a re-use of RAP equal to 100% (Ipavec et al., 2012).

Regarding the percentage of RAP currently employed in new mixtures, it can be noted that usually low amounts of RAP are used in the surface layers (about 10% by total aggregate weight), whereas higher percentages (up to 50%) are allowed for base layers. Generally, the construction companies do not overcome the 30% of RAP even for deeper layers, both for quality issues and many constrains related to plant production equipment that are not solved yet (de la Roche et al., 2012).

In any case, RAP is a complex material and re-using it in high quantity needs a very accurate monitoring of its characteristics and properties of the virgin materials to be combined with, essential element for achieving acceptable performance (Mouillet et al., 2012).

In that regard, the European standard EN 13108 recommends to pay particular attention to choice the virgin bitumen added to the recycled mixture when RAP percentages higher than 10% are used for surface layers and 20% for base layers. In the light of what just said, the most practical way to proceed could appear to use RAP in percentage not higher than 10%, as done by most of the companies, but so renouncing to many potential environmental and economic benefits.

In terms of performance, both the European and the national standards prescribe that the asphalt mixtures made with recycled materials meet all the requirements asked to the virgin traditional mixtures. However, some countries impose further investigations when RAP content is more than 15-20%.

Currently, the increment in the use of recycled aggregates for the production of new asphalt mixtures is a key point of the European policies, above all for the construction and maintenance of the highway system. Many European countries have established the use of 100% of RAP as one of the main long-term objectives to achieve without compromising the performance of the material.

Nowadays, RAP is mostly employed for the deeper layers (unbound layers or base layer), but the tendency is pushing towards the ordinary use of such material also for the production of the upper bituminous layers (such as porous asphalt layers), with the advantage to have the possibility of exploiting also the bituminous component of RAP.

2.2.2 Characterization of the aged bitumen coming from RAP

As previously anticipated, the choice of the best virgin bitumen type is strictly related both to percentage of RAP used and properties of the aged bitumen coming from RAP.
In order to improve mechanical properties of mixtures including RAP (with particular attention to the cracking resistance) and to increase the percentage of RAP without affecting mixture performance, many European countries use polymer modified bitumen as virgin bitumen in combination with RAP.
Regarding the use of additives named as rejuvenators, the latter are not often added to the mixture since not a clear evidence of the benefits related to their use exists, especially if used as alternative to low polymer modified bitumens that are more effective and also more economically convenient.
Another aspect concerning the bitumen coming from RAP that deserves further investigations is the presence of external substances, with particular attention to the contamination of tar (Polycyclic Aromatic Hydrocarbons, PAH), that could be included in very old pavements.
The use of RAP contaminated with tar is forbidden according to the Netherland and Belgium standards, whereas in Switzerland the maximum amount of PAH allowed is 5000 ppm. Since many countries are very attentive to this topic, the research project SAMARIS (Sustainable and Advanced Materials for Roads and Infra-Structures (Piau et Christensen, 2005) activated by the FEHRL (Forum of Europe's National Road Research Centres) from 2002 and 2006, provided some recommendations in terms of procedures useful to detect and quantify the presence of tar in secondary recycled materials.

2.2.3 European Projects

In Europe, several international projects were entirely dedicated to improve the existing knowledge and to develop new technologies concerning the re-use of valuable material such as RAP from milled asphalt pavements.
The research project RE-ROAD is one of the most significant program developed over the last years about hot recycling in the pavement field (Kalman, 2013). The most important research centers in all Europe took part to this project (i.e. FEHRL, the Laboratoire Central des Ponts et Chaussées (LCPC), the University of Nottingham and the Swedish National Road and Transport Research Institute). It ended in November 2012 with an international conference held in Bruxelles. This project aimed at developing innovative knowledge and technologies that could help to create long lasting asphalt pavements as well as improving the energy efficiency and the environmental impact of the pavement production process.
The strategy followed to achieve these goals is perfectly aligned with the support promoting by the European governments towards more attentive environmental policies. In fact, the growing diffusion of recycling techniques allows a significant reduction of the use of raw materials and territories for disposal purposes. Moreover, the use or RAP allows the

minimization of transport from and to the construction site with benefits in terms of energy saving, emissions and pollution (Bocci et al., 2010; de la Roche et al., 2012; Mouillet et al. 2012;).
The program RE-ROAD was developed focusing on some fundamental points that were initially analyzed separately to finally compose a unique summary about the issues and the corresponding possible solutions related to recycling in the pavement field.
In details, the major investigations realized were focused on the following points:

- demolition techniques of old pavements: evaluation of potential negative impact of milling methods on the quality of RAP;
- protocols to characterize RAP: improvements of characterization methods and technical evaluation of RAP, analysis of RAP heterogeneity and study on the specific industrial processes adopted for the production of recycled mixtures;
- management system: optimization of recycling techniques and strategies to improve disposal of secondary materials that cannot be recycled;
- environmental analysis: evaluation of risks and benefits related to the environment;
- cost analysis: short and long-term analysis of performance and estimation of in service life through the study of recycled asphalt mixtures produced with different amounts of RAP by means of different production techniques;
- industrial processes: analysis of potential negative effect of the procedure adopted to introduce RAP in the in plant production process of new asphalt mixtures. Evaluation of advantages and disadvantages related to the use of RAP containing polymer modified bitumens.

Furthermore, the research project DIRECT-MAT represents another important program in the European background for the promotion and development of recycling techniques (Bocci et al., 2010). It is aimed at shearing and spreading the knowledge and the practices of each European country about recycling. Also in this case, as already mentioned for the RE-ROAD project, the main goal of the program is to establish guidelines useful for the construction of long lasting pavements produced through secondary materials. To this aim, an on-line database was created to make the current state of the art about the research on recycling and the corresponding experimental data always available. Downhill of the comparative evaluation of all documents and several national experiences collected over time, specific guidelines about recycling techniques will be defined. Such a database should constitute a reference point constantly updated according to a continuous and accurate standard methodology.
Thus, the final goal of the project DIRECT-MAT is to improve the cooperation and coordination between several European research groups through the implementation of a sheared scientific vision and research methodology. Within the scope of this project, also the collaboration between theoretical research and practical application has a fundamental importance. Therefore, the research activities are always carried out in accordance with pavement construction companies in order to find a good balance between the theoretical aspects and practical issues.

Based on the first results collected in the DIRECT-MAP database, RAP can be used successfully for the in plant production of new mixtures. Moreover, the percentages of RAP that can be included vary depending on the equipment used and the type of mixture realized (upper or deeper layers).
The main recommendations for the use of high percentages of RAP are mainly focused on the necessity to completely characterize the milled material prior to use it. Moreover, the experimental results showed that it could be possible to employ recycled mixtures also for asphalt roads that require high performance (i.e. wearing layers or highway pavements). Finally, it is worth noting that all the experiences made within the DIRECT-MAT project did not reveal particular or specific issues related to the use of RAP material containing aged polymer modified bitumen (Ipavec et al., 2012). This aspect assumes fundamental importance particularly in Italy, since the most part of the Italian highway system is currently made with polymer modified bitumens. Therefore, the majority of the RAP available now or in the future contains aged polymers, making the analysis of recycled asphalt mixtures further difficult.

2.3 State of the art in USA

Due to the first oil crisis during the Seventies between West and East countries, also in USA the recycling techniques gained more attention as a useful alternative to decrease the dependence of USA from oil.
Therefore, recycling was quite known even before the implementation of the Strategic Highway Research Program (SHRP) and the corresponding Superpave sub-task that nowadays is still a fundamental reference point for the study and the characterization of bitumen and asphalt mixtures. Nevertheless, these programs did not provide guidelines or specific standards for the using of RAP in the production of new asphalt mixtures, since there were not urgent reasons yet that promoted RAP exploitation (Copeland, 2011).
The percentages of RAP initially used were lower than 15% per mixture since for so low amounts the technical standards did not require modifications of the virgin bitumen used, nor further investigations or analysis for the evaluation of recycled mixture performance. However, the continuous growing of material prices, as well as more and more strict laws about environmental issues and growing attentions for the “green technology” encouraged the increase in RAP percentages used as well as the identification of specific technical standards available for the Departments of Transportation (DOT) of each State. Over the last decades, these efforts found a tangible application with a re-use equal to 80% of the 90 mil of RAP tons produced per year.
Milled materials coming from old asphalt pavements are employed as simple virgin aggregates in the deeper layers of a pavement (i.e. base, sub-base, stabilized base and embankments) as well as in the upper layers for the substitution of part of the virgin bitumen by exploiting its bitumen component. As already shown in the European context, also in USA the reasons behind the growing development of recycling are related to a more conscious awareness of the environmental issues as well as the economic benefits mainly achievable thanks to the following factors:

- reduction in the use of virgin aggregates and bitumen;
- reduction in the energy consumption;
- reduction in the transport costs;
- lower amounts of waste materials to disposal in the landfills.

In particular, regarding the costs, the Federal Highway Administration (FHWA) has identified four main cost items related to the production of traditional asphalt mixtures that could be significantly reduced by the adoption of recycling techniques: materials, production plants, transport and lay down phase.
Among these aspects, the one that assumes the highest relevance in the overall costs computation is certainly the materials (70% of the total costs). Moreover, among the different materials, the bitumen is the component with the highest price. Therefore, the most convenient way of exploiting RAP is represented by its use in bound layers (binder or surface layers) where the bitumen coming from RAP can be re-activated and partially substitute the more expensive virgin bitumen.

2.3.1 Current practices in USA

Nowadays in USA, the construction of new pavements needs an amount of asphalt mixtures equal to 500 mil tons per year. Within this quantity, the RAP currently constitutes about 60 mil tons. However, also in USA specific standards that impose the use of a certain quantity of RAP do not exist. The inclusion of RAP in new mixtures is not mandatory and can be proposed by the construction company involved in construction or rehabilitation activity.
In 2007, the average use of RAP was estimated equal to 12% in the all USA (Copeland, 2011). A summary of the current use of RAP by the Federal State Agency and an estimation of the potential use of RAP in each flexible pavement layer (surface, binder and base layers) is reported in Figures 2.2 and 2.3. For the surface layers (Figure 2.2) the RAP percentage usually varies between 10% and 30%, whereas for base and binder layers (Figure 2.2) the RAP content is about 20% and 50%. However, in the cases where RAP exceeds the 30%, it is necessary to demonstrate that the final pavement performance are not penalized by the use of RAP through a series of quality controls. Finally, it is worth noting that almost 20% of the States does not allow the use of RAP in the surface layers, especially for roads with high traffic loads.

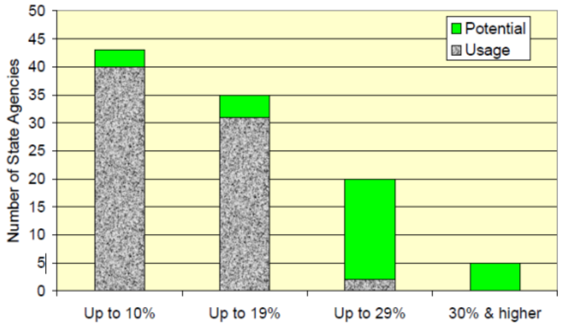


Figure 2.2 Current and potential use of RAP in the surface layer (Copeland, 2011)

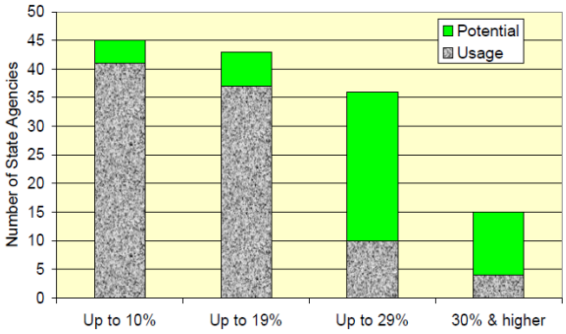


Figure 2.3 Current and potential use of RAP in the base and binder layers (Copeland, 2011)

As shown in Figure 2.4, even if several DOTs already allow the possibility to include more than 25% of RAP, in the 50% of them construction companies still prefer amounts of RAP lower than 20%. Despite the efforts to promote the use of growing amount of RAP in the production of new asphalt mixtures, recycling is not a consolidated production technique in USA, yet.

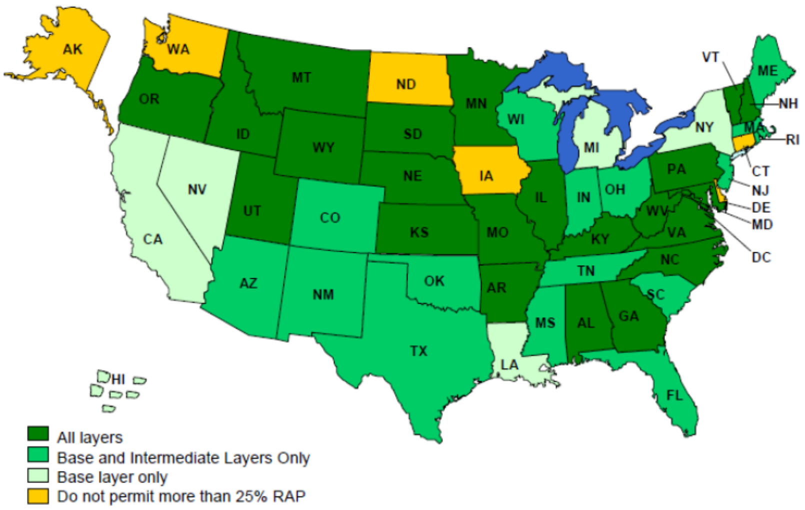


Figure 2.4 Asphalt mixture layers where RAP can be incorporated for each State (Copeland, 2011)

The low exploitation of secondary materials could be partially due to the further controls and performance parameters that the State Agencies require to characterize recycled mixtures (i.e. type of aged bitumen coming from RAP, type and dimensions of the original aggregates). The limits of these parameters established by technical standards can cause limitations in the use of RAP, both in terms of percentages and locations (pavement layers where the RAP can be included).
Besides that, other complementary factors that contribute to limit the amount of RAP must be taken into account:

- specific limitation imposed by DOTs;
- lack of appropriate manufacturing and mix design procedures;
- necessity of high homogeneity and good quality of RAP;
- low availability of RAP near to the construction site;
- high performance standards required for the final recycled mixture;
- low experience.

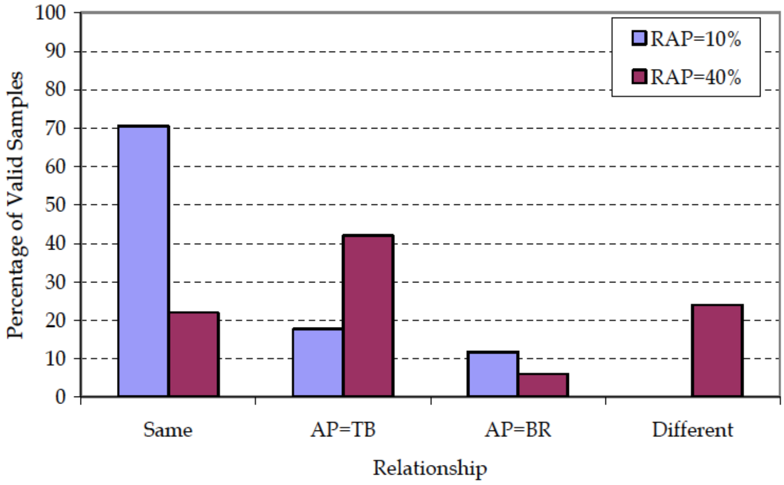
However, based on a series of surveys commissioned by DOTs, it can be noticed that the main concerns about the use of high percentage of RAP are mainly related to the type and quality of the virgin bitumen as well as the properties of the aged bitumen coming from RAP. In particular, the major issues to be addressed are the effect of RAP bitumen on the stiffness of recycled mixtures (with particular focus on the cracking aptitude) and the possible interactions between virgin and aged bitumen with consequent effects on overall pavement performance.

A further aspect not solved yet, concern for several USA Federal State Agencies as in Europe, is the use of RAP containing polymer modified bitumens. In fact, it is commonly believed that the presence of aged modified bitumen in RAP could further reduce quality of the final mixture, principally for the alleged deterioration of polymeric components caused by oxidation phenomena underwent by the recycled material.
Moreover, the issues related to the lack of a direct and reliable method to evaluate the degree of blending between virgin and aged bitumen and the assessment of the corresponding interactions can be mentioned as a further important factor that limits the use of RAP in new mixtures.
It is important to underline that recycled asphalt mixtures are asked to meet the same requirements of traditional mixtures, both in terms of permanent deformation, fatigue and thermal cracking resistance.
About the mix design and the production procedures to be followed for mixtures including RAP, specific and mandatory technical standards are not available yet and the experience collected is still insufficient to provide recommendations. Therefore, even for high amounts of RAP, the mix design method more commonly adopted is still the one provided by the Superpave.
Finally, the analysis of homogeneity of RAP is a further aspect highlighted by the FHWA in order to optimize the recycling process. In that sense, the production plants are asked to regularly monitor the water content, the gradation and the origin of RAP, with particular attention on separately stocking RAP coming from different pavement layers.

2.4 Effect of RAP on bituminous phase: degree of blending

As observed in the previous paragraph, one of the main concerns for the use of RAP in the production of new asphalt mixtures is related to the properties of the aged bitumen included in RAP and how it can affect the quality of the final bitumen phase of the recycled mixture. This aspect deserves particular attention and specific investigations to be fully understood.
A comprehensive picture of the effects caused by aging on the chemical and rheological properties of bitumen is provided in paragraph 1.2.1. In the present chapter, the current state of the art concerning the knowledge acquired on the interactions between virgin and RAP aged bitumen is given.
The production of recycled HMAs implies a heating process that involves RAP aggregates when they are mixed with the pre-heated virgin aggregates and bitumen. In turn, this heating phase implies a partial melting of the aged bitumen that coats RAP aggregates. Such bitumen represents the “re-activated” bitumen that could interacts with the virgin bitumen contributing to the overall performance of the final recycled HMA. Mixture performance change as a function of the re-activated bitumen amount released by RAP during the heating process that depends on several factors mainly related to RAP properties (e.g. bitumen type and content, aggregate gradation, aging degree) and production process parameters (e.g. production temperature, RAP pre-heating time, storage and transportation time) (Shirodkar et al., 2011).

The re-activated bitumen degree varies between two limit conditions: full RAP bitumen re-activation (100% re-activation) and "black rock" condition (0% re-activation). Several studies consistently concluded (Al-Qadi et al. 2007; Al-Qadi et al. 2009; Kandhal and Foo, 1997; Khosla et al. 2012; McDaniel et al. 2000; Navaro et al. 2012; Shirodkar et al. 2011; Shirodkar et al. 2013) that the real re-activated bitumen degree is comprised between the above-mentioned limit conditions. Therefore, wrong assumption of the re-activated bitumen degree can cause inaccurate choice of virgin bitumen quantity and grade with negative effects on pavement performance. In fact, if part of the aged RAP bitumen is actually not contributing, the assumption of 100% re-activation can lead to "under-asphalted" mixtures (Al-Qadi et al. 2007; Shirodkar et al. 2011) leading to early pavement distresses. On the other hand, the adoption of an underestimated re-activation degree could induce to excessive virgin bitumen content that could affect also in this case the final mix performance (Al-Qadi et al. 2007).
In the literature, many studies concerning the blending process between reclaimed and virgin bitumens are available (Huang et al., 2005; McDaniel et al., 2000; Oliver, 2001; Sthephens et al., 2001). In particular, the research project NCHRP 9-12 (McDaniel et al., 2000) used mechanical SuperPave performance related parameters (mainly obtained through frequency sweep tests, Simple and Repeated Shear tests at constant height, indirect tension tests and creep tests) to compare mixtures with different RAP content. Various levels of interaction between virgin and RAP bitumens were experimentally investigated (McDaniel et al., 2000): the "black rock" condition (0% blending), 100% blending, the partial blending and the actual practice situation (blending as it usually occurs in practice). Based on the results, the AASTHO M323 specification was prepared indicating that the use of 15% or less of RAP does not require any change in the virgin bitumen selection since the blending conditions are always very similar (Figure 2.5). On the other hand, for mixtures including more than 30% RAP a specific blending chart should be used in order to adjust the virgin bitumen in terms of content and grade accordingly. For intermediate percentages of RAP, the specification recommends to use one-grade softer PG bitumen. Anyway, a recent experimental research (Mohajeri et al., 2014) demonstrated that a bitumen grade change is not necessary up to 25% RAP. This research also studied the diffusion process between RAP and virgin bitumen by means of a Dynamic Shear Rheometer (DSR) to estimate the degree of blending.



Same: Actual Practice = Total Blending = Black Rock
AP=TB: Actual Practice = Total Blending ≠ Black Rock
AP=BR: Actual Practice ≠ Total Blending = Black Rock
Different: Actual Practice ≠ Total Blending ≠ Black Rock

Figure 2.5 Interaction between virgin and reclaimed bitumen (McDaniel et al., 2000)

Previous researches (Khosla et al. 2012; Navaro et al. 2012; Shirodkar et al. 2011; Shirodkar et al. 2013) were pointed on the development of methods for determining the proper re-activated bitumen degree by comparing the main rheological properties of the virgin bitumen with those estimated or measured for blends between re-activated and virgin bitumens. Other studies addressed the analysis by performing mechanical tests on recycled HMA prepared with various RAP content and different mix design (Al-Qadi et al. 2009; Kandhal et al. 1997; McDaniel et al. 2000).

Shirodkar et al. (2013) studied the impacts of partial RAP bitumen re-activation on the critical grade of the blended bitumen (re-activated + virgin) developing a chart for different degrees of re-activation (blending). Their study was based on the critical grade of blends related to different proportions between re-activated and virgin bitumens. Further studies were carried out at micro and nano-levels. Navaro et al. (2012) used a microscopic observation technique and image analysis to qualitatively investigate how virgin and re-activated bitumens blend together. Mohajeri et al. (2014) studied the blending between re-activated and virgin bitumens by applying testing procedures usually employed to assess the diffusion phenomenon between different substances.

All the above-mentioned research studies provide a qualitative assessment of the degree of re-activation, but are not able to provide a quantitative estimation. Few attempts have been made in this direction.

Among them, Shirodkar et al. (2011) proposed a “blending ratio” that involves the main rheological properties measured for the virgin bitumen and for the bitumen extracted from hot recycled mixtures prepared with different amount of RAP. Although this methodology

allows a numerical determination of the re-activated bitumen, it requires at least three solvent extractions and recoveries of the bitumen. Moreover, the procedure is very time consuming and must be carefully adjusted when different RAP, virgin aggregates and bitumen type are used.
The re-activation degree of the bitumen released by RAP aggregates was quantified also by Huang et al. (2005). They defined the re-activated bitumen as the extent at which the RAP bitumen coats the virgin aggregates when RAP and virgin aggregates are mixed together without any virgin bitumen. Anyway, it must be considered that the degree of re-activation is not only affected by the pure mechanical mixing. Many other factors (e.g. mixing time and temperature, virgin and RAP aggregate gradation, bitumen type) influence this process. Moreover, a portion of the re-activated bitumen could coat also RAP particles, not only the virgin aggregates, making the method proposed by Huang et al. questionable.
In addition, Huang et al. (2005) assumed the viscosity as reference parameter to assess the re-activation degree. In the case of mixes prepared with 100% RAP and proper amount of virgin bitumen, it was found that the viscosity increased from the external bitumen to the internal bitumen closer to the aggregate surface that behaved similarly to pure RAP aged bitumen. However, also this method implies laboratory practices that involve the use of solvents or methodologies that can alter the bitumen properties, making the calculation method not sufficiently reliable.
In summary, although over the last years the re-activation degree of bitumen released by RAP aggregates has been widely debated, currently no direct and reliable method is available to accurately quantify the amount of re-activation that occurs within recycled HMA.
The impossibility to estimate the actual amount of the re-activated RAP bitumen prevents the design of the proper quantity and grade of the virgin bitumen to be added to hot recycled bituminous mixtures and discourage the use of higher amounts of RAP.

2.5 Mix design of recycled asphalt mixtures

Nevertheless the in place recycling processes (both hot and cold) allow quantity of RAP up to 80-100% to be used, the common practices usually do not exceed RAP amounts equal to 20-25% by aggregate weight. One of the main reasons that limits RAP quantity is related to the extreme variability of RAP gradation. This aspect is especially important when RAP is not appropriately processed and stocked, with previous sieving and fractioning actions that avoid excessive variability and segregation.
Additionally, the milling process produces a recycled material with high content of fine aggregates, RAP component rich in aged bitumen that, hence, could cause a significant increment in mixture stiffness, with consequences in terms of production process and lay down phase. For workability reasons, in fact, a stiffer mixture needs the virgin aggregates heated at higher temperature in the plant, with subsequent problems related to mix design optimization as well as adoption of the best production process.
Besides the issues related to the manufacturing phase, it is necessary to analyze the potential impact that the presence of RAP can cause on durability and mechanical performance of pavements in terms of rutting, fatigue and thermal cracking.

Over the last years, an extensive and detailed amount of studies have been carried out in terms of methods and strategies to re-use RAP, but only recently the attention has been focused on the analysis of recycled mixtures performance. Therefore, no enough and reliable data about the long-term behavior of such mixtures are available, yet.
The first experiences showed that trial sections made with recycled materials were characterized by higher quantity of cracks, smaller ruts and a surface roughness similar to that one of a traditional asphalt mixture.
On the basis of the results collected so far, it is possible to state that the pavement layers made with a RAP content around 35% can guarantee the same performance of a traditional virgin mixture for the whole in service life as long as an accurate mix design and lay down process are performed (Copeland, 2011).
The following paragraphs provide a summary of the main issues to address in the mix design of mixtures including RAP. Two main aspects must be analyzed: the major factors to be taken into account during the mix design of a recycled mixture and the performance evaluation of the mixture through appropriate laboratory experimental plans and procedures.

2.5.1 Mix design

The first experiences that tried to apply recycling techniques of milled material for asphalt mixtures considered RAP as a traditional virgin aggregates both for the mix design and for the production process. However, the corresponding applications in the field showed that this assumption led to mixtures not able to achieve the expected performance. It was understood that a specific mix design criterion was needed for recycled mixtures, especially when considerable RAP content are used.
Over the last years, several mix design methods have been developed to define the best "recipe" for a recycled mixture. Based on the main characteristics of the various mixture components, a more rational and reliable quantification of the maximum amount of RAP incorporable and the best type of virgin bitumen to be combined with that specific RAP aggregate can be identified (Bukowski, 1997; Kandhal and Mallick, 1997).
The USA technical standards are currently the reference point at international level for the mix design of recycled mixtures; they do not provide any modification in terms of amount and type of virgin bitumen used when less than 15% of RAP is included in the mixture. On the contrary, when the amount of RAP varies between 16% and 25%, the FHWA suggests the use of a virgin bitumen with a lower Performance Grade (PG) (lower both in the highest and in the lowest limit) compared to that used for the corresponding virgin mixture. Finally, when percentages of RAP higher than 25% are considered, the definition of the most appropriate bitumen in terms of PG and quantity is determined through specific "blending charts" (AASHTO M 323) that are built on the basis of the physical properties assumed by the virgin and the reclaimed bitumen at various temperature ranges. Such guidelines developed by the FHWA were developed on the basis of the results of the NCHRP Project 9-12 (McDaniel et al., 2000), which is one of the most important research programs that were carried out about hot recycling, specifically created to introduce in the SuperPave (initially born only for traditional virgin mixture) specific mix design

procedures for mixtures containing RAP (McDaniel and Anderson, 1997). Hence, the Superpave mix design was updated and modified according to the indications provided by the several practical experiences that were progressively realized; in this sense, two alternative approaches are provided in order to define the best "recipe" to use for recycled mixtures with high RAP content, once all the physical and rheological properties (e.g. the PG) of the reclaimed bitumen coming from RAP are known.
The first approach fixes a priori the amount of RAP to be included in the mixture. Then, specific blending charts are available to determine the PG and the amount of virgin bitumen on the basis of RAP content.
In the second approach, the amount of RAP to be included in the mixture is defined by using additional blending charts where the type of virgin bitumen is initially established.
However, the development of such blending charts is very time consuming and requires many efforts and, often, the use of dangerous solvents. Therefore, over time researchers have tried and are still trying to implement other alternative procedures.
In particular, Figure 2.6 shows a graph (developed based on the bitumen stiffness values of some experimental field sections in USA) which allows the evaluation of the superior temperature limit of the bitumen PG on the basis of the RAP content to be included in the mixture, so allowing the estimation of RAP effect on mixture stiffness (Copeland, 2011).

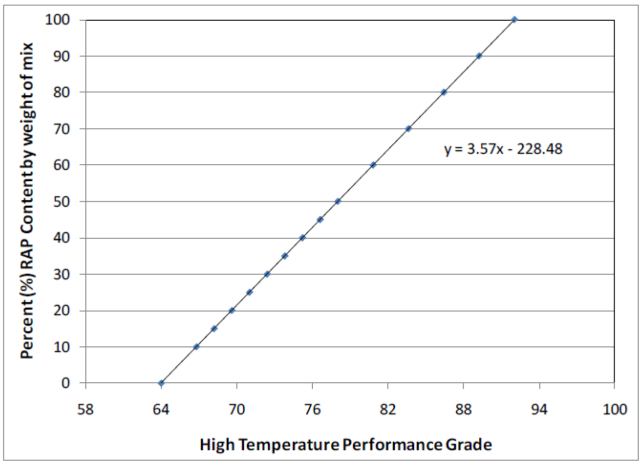


Figure 2.6 RAP amount Vs. upper temperature limit of virgin bitumen (Copeland, 2011)

Additionally, a procedure was developed aimed at identifying the degree of blending between virgin and aged reclaimed bitumen in mixtures containing RAP. This method requires the measure of the dynamic modulus |E*| of recycled asphalt mixtures. Afterwards, the mixture is subjected to a solvent extraction in order to rescue bitumen components of the recycled mixture (composed by virgin and reclaimed bitumen). The complex modulus |G*| of the extracted bitumen is determined as it constitutes the input data of a specific calculation model able to estimate |E*| of the final mixture. Such a calculated value is compared with the |E*| initially measured. In case that the two data are comparable, it is

possible to assume that a proper blending between virgin and reclaimed bitumen occurs. Such a procedure could be taken into account during the mixture mix design in order to choose the most appropriate virgin bitumen to be used in that specific case (Copeland, 2011).
Besides the degree of blending between virgin and aged bitumen, other aspects that contribute to determine the maximum amount of RAP incorporable in a mixture must be taken into account. In fact, factors as characteristics and productive capacity of the production plant or volumetric properties of the final asphalt mixture play a fundamental role to guarantee the minimum performance that a pavement must provide as established by the technical standards.
In that sense, the mix design procedure optimized by the Superpave program for mixture containing RAP seems not so different from the procedure usually adopted for traditional virgin mixtures produced without recycled materials (Al-Qadi et al., 2007). The main difference consists in the calculation of the RAP amount that differs from the classical relationship used to determine the weight of the virgin aggregates:

$$M_{dryRAP} = \frac{M_{RAPagg}}{(100-P_b)} * 100 \quad (2.1)$$

where

- M_{dryRAP} = amount of aggregates contained in the RAP;
- M_{RAPagg} = total amount of RAP;
- P_b = amount of bitumen in the RAP.

Moreover, an important role is played by the volumetric requirements that recycled mixtures have to meet. They concern the Voids in Mineral Aggregate (VMA), the Voids Filled with Bitumen (VFB), the amount of fine aggregates, the compactability properties and the geometric characteristics of the aggregates, especially in terms of shapes.
Also in this mix design procedure, the amount of virgin bitumen, that should be partially reduced to take into account the bitumen coming from RAP, assumes a fundamental importance that significantly affects the volumetric properties. Almost every volumetric characteristic, in fact, is calculated on the basis of the total amount of bitumen in the mixture (virgin bitumen plus re-activated bitumen coming from RAP). Therefore, if the total amount of bitumen is not properly estimated, volumetric properties will not meet the specifications with severe consequences on the pavement durability. For example, if the final quantity of total bitumen in the mixture should be 4.5% and the contribution due to the aged RAP bitumen is estimated equal to 0.3%, the virgin bitumen to be added should be 4.2%. Actually, considering all the bitumen included in the RAP as re-activated is unlikely. Usually only a part of it really contributes to the total bitumen amount. Therefore, this procedure to calculate the virgin bitumen can be considered inaccurate and could lead to rough mistakes. In fact, if only a certain part of the reclaimed bitumen of RAP is re-activated (i.e. 50%), the effective total amount of bitumen in the mixture will be 4.35%

instead of the designed value equal to 4.5%, with evident consequences on volumetric properties as well as mechanical performance of the final mixture.
From the aspect analyzed so far, it can be concluded that several challenges need to be addressed in the mix design of mixtures containing RAP. Factors that mainly affect the final results are the extreme heterogeneity of RAP, the uncertain properties of the bitumen coming from RAP and the unknown amount of re-activated bitumen (Al-Qadi et al., 2007).
Regarding the variability of RAP, many studies (Bukowski, 1997; Huang et al., 2004) showed that mixtures with low RAP content (around 15%) are not significantly affected by the potential heterogeneity of milled materials, whereas when high quantities of RAP are used, the variability of the milled material compromises mixture performance. In that sense, particular attention must be focused on the high amount of fine aggregate usually present in the RAP, also produced as a consequence of the milling process to break up the coarse aggregates in smaller particles. Such a high presence of fine fraction has consequences on the dosage of the other components (virgin aggregates and bitumen) and it is not always taken into proper account, with detrimental effects in terms of performance.
Another important aspect to consider concerns the gradation of RAP aggregates. Since a relevant part of the fine fraction agglomerates with bitumen and tends to combine with the coarse aggregates forming larger clumps, it must be remember that the original gradation of the old pavement does not reflect the gradation of RAP aggregates once they are milled out from the pavement. Therefore, it is necessary to accurately study the RAP aggregate gradation without rely on the original formulation. This aspect confirms again the relevant importance of an initial sieving and fractioning of RAP prior to be included in the mixture in order to achieve a better control of the gradation. In fact, as previously mentioned, also the amount of the fine fraction strongly affects the volumetric properties of the final recycled mixture and, hence, it must be accurately taken into account. In particular, an excessive amount of fine fraction can cause a too thin film thickness of bitumen around the aggregates. Moreover, too high amounts of fine fraction can be the reasons of higher rutting susceptibility and lower fatigue resistance. In order to better monitor the amount of fine fractions coming from RAP with the aim to increase the total quantity of milled material in the recycled mixture, a good precaution is to divide the RAP in at least two fractions that must be separately accounted for in the mix design.
The analysis of the effects of various amounts of RAP on the mechanical and volumetric properties of recycled mixtures (Al-Qadi et al., 2007) showed that the optimum bitumen amount should not significantly change for different RAP contents, whereas the VMA variation as a function of RAP quantity could be much more evident, even if not a clear trend was observed. In fact, some cases showed a decrease in the VMA when the RAP percentage increased, others demonstrated an opposite behavior. Several authors investigated the effects of high RAP contents, but the results are not always in accordance, demonstrating again the complexity of the topic and the need to further investigate the related aspects.
In particular, West et al. (2009) found evidences of a reduction in the total optimum bitumen content due to an increase in the RAP percentage as well as a decrease in the VMA value. Viceversa, Daniel and Lachance (2005) observed an increase in the VMA and VFB values as the percentage of RAP increased (from 25% to 40%). In particular, they

hypothesized that the VMA variation can be attributed to the mixing time between RAP and virgin materials. On the basis of this study, an optimum mixing time was identified in order to obtain the best blending of the mixture. Within the same research activity, the influence of the pre-heating time of RAP on the volumetric properties was also investigated. It was observed that the VMA value decreased of 0.5% when the pre-heating time changed from 2 to 3.5 hours and of 3% when the pre-heating time changed from 2 to 8 hours. Longer pre-heating times seem able to provide an amount of heat to the RAP sufficient to break up potential clumps too large, allowing a better interaction and a easier mixing with the virgin materials. The amount of the pre-heating time is directly related to some fundamental parameters that characterize the recycled mixtures, such as the amount of RAP included in the mixture, the water content of RAP, the quality of the demolition and milling process, the mixing temperature and the efficiency of the equipment in the production plant (Ipavec et al., 2012).
Finally, the results of several various investigations highlighted how particular attention must be focused on the analysis of the volumetric properties variation as function of the RAP content, since they directly affect pavement performance.

2.5.2 Performance

As already highlighted in the previous paragraph, there are several aspects that concurrently affect the characteristics of a recycled asphalt mixture and each one must be accurately analyzed.
Among them, the further oxidation process underwent by the aged material coming from milled pavements that takes place over time in the stockpiles due to the continuous exposition to the air is not always sufficiently considered.
The extent of this further oxidation varies as a function of the stocking time and conditions prior to be used. At equal amount of RAP, a different stocking process can significantly affect the overall performance of the mixture.
Another aspect to be mentioned, is the uncertain degree of blending between virgin and reclaimed bitumen coming from RAP. In fact, when RAP is added to other material components, an unknown percentage of RAP bitumen is re-activated and blends with the virgin bitumen, so producing a bitumen phase in the recycled mixture with unknown properties and, hence, performance difficult to predict.
Many international studies aimed at predicting the performance of recycled mixtures once they are lay down in the field and reducing the uncertainties in the mix design phase, focused the attention on the cracking resistance (through an analysis of the stiffness properties and fatigue behavior) and on the rutting aptitude. The evaluation of such characteristics allowed the judgment of the goodness of the mix design method and the production process adopted.
The first investigations in this direction (Al-Qadi, 1997) evaluated the mixture properties at low temperature, since it was assumed more likely that the stiffening effect due to the adding of a certain amount of RAP could mainly affect the pavement behavior in the most critical conditions in terms of cracking. These investigations were mainly developed by means of the Superpave Indirect Tensile Creep Test, in a temperature range between -20

and 0°C, along with tests for the measuring of the resilient modulus, in a temperature range between 5 and 40°C, with the aim to analyze the thermal susceptibility of the mixture, too. The results showed that the inclusion of RAP in asphalt mixtures causes a reduction in the permanent deformation aptitude as well as in the thermal susceptibility. On the contrary, an increase in the fatigue cracking tendency was detected. Moreover, a stiffness increase of almost 100% was assessed at intermediate temperatures, whereas the stiffness varied much less at low temperatures (Al-Qadi, 1997).

Another experimental investigation (Sondag et al., 2002) aimed at evaluating the resilient modulus of mixtures compacted with different types and quantities of RAP and various types of virgin bitumen, showed that, at a temperature of 25°C, the adding of 40% of RAP caused an increment between 74 and 164% of stiffness as function of the type of virgin bitumen used. Such results highlighted that the properties of recycled mixtures did not vary linearly as function of the RAP percentage.

In terms of fatigue, tests carried out within the NCHRP 9-12 Project confirmed that recycled mixtures including more than 20% of RAP manifested lower fatigue resistance than traditional virgin mixtures (McDaniel et al., 2000). A possible solution to solve such an issue can be the use of a softer bitumen characterized by a lower PG, especially when high amount of RAP are considered.

Further studies (Huang et al., 2004; Pereira et al., 2004) showed that an increase in the bitumen content in mixtures produced with high RAP content did not significantly affect the fatigue resistance and confirmed the improved rutting behavior compared to traditional virgin mixtures.

Other researches (Shu et al., 2008) highlighted the significant effect due to the aging underwent by the RAP component on the fatigue life of a recycled mixture by investigating this aspect through Indirect Tensile Strength (ITS) and Semi-Circular Bending (SCB) test. Additionally, it was found that the adding of RAP caused a significant increment in the indirect tensile resistance, a reduction in the post-failure tenacity, an increase in the material stiffness and a reduction in the viscosity properties. On the contrary, percentages of RAP lower than 20% demonstrated very limited effects on the stiffness and the indirect tensile resistance.

Opposite results were found in other research studies (Widyatmoko, 2008) concerning the production of asphalt mixtures for surface and base layers containing 10, 30 and 50% of RAP. In fact, the results showed that mixtures with higher RAP content had lower resistance to permanent deformation and improved fatigue behavior. However, such findings were attributed to the large amount of rejuvenators included in the mixtures.

In fact, when high RAP contents are employed, higher contents of rejuvenators (or virgin bitumen much softer) are often used as well in order to obtain mixtures less viscous and with good workability.

A further aspect to addressed concerns the durability of the mixtures containing RAP, strongly related to the effect of water and humidity on the material properties. In that sense, with the aim to evaluate the water susceptibility of recycled mixtures, some researchers (Sondag et al., 2002) used the tensile strength ratio (ratio between the tensile strength of the mixture not subjected to wet conditioning and the tensile strength of the same material after conditioning in water). They found that this parameter was not affected by the presence of RAP.

However, it must always be considered that the RAP can come from pavements deteriorated due to the effect of water. In that case, it is likely to assume that the same problems can manifest again. Therefore, it is highly recommended not to use RAP that comes from pavements that had significant problems related to humidity.

At the same time, it must also be considered that the milled material could guarantee an improved water resistance since RAP aggregates are pre-coated and protected by a thin film of bitumen that could ensure a sort of further sealing element (Al-Qadi et al., 2007).

2.6 The use of RAP in surface layers

As already mentioned, the use of RAP in surface layers of asphalt pavements is often limited to a very low amount and, in some case, it is even forbidden from some national specification all over the world, especially when delicate mixtures are selected such as Porous Asphalt (PA). Figure 2.1 shows that European countries tend to limit such an amount to RAP content lower than 20%.

The uncertainty related to RAP gradation as well as RAP storage conditions plays a major role in such a limitation of RAP content for surface layers. Both aggregates gradation and water content, in fact, represents two of the most important parameters than need to be properly taken into account during the mix design of surface layers. In this sense, a separate storage of RAP based on layers and sources as well as a proper RAP gradation control could improve RAP quality and allowing the use of higher amount of recycled materials also in surface layers up to 30%. In particular, RAP coming from milled surface layers should be store separately from RAP coming from binder or base layers in such way that good quality materials are re-employed for surface layers. Moreover, in order to better control RAP gradation, at least two fractioned classes are required.

Such a goal represents a very important step forward in terms of asphalt materials recycling since surface layers needs frequent maintenance and rehabilitations.

2.6.1 The use of RAP in SMA mixtures

Surface layers made with Stone Mastic Asphalt (SMA) are widely spread in many countries such as Denmark. However, in order to use high RAP content in new recycled SMA layers it is necessary the RAP aggregates only came from milled SMA layers.

A recent research study (Jensen, 2012) was carried out in Denmark focusing on the performance evaluation of a recycled SMA layer including RAP coming from old milled SMA mixtures. In particular, two real scale experimental sections were realized including a traditional SMA layer prepared with only virgin aggregates and a recycled SMA layer including 15% RAP. The aggregate gradations of the two SMA mixtures were properly studied during the mix design in such way that the two layers were characterized by similar final aggregates grading curve. Several cores were taken from the experimental sections and properly tested showing that the two SMA mixtures were identical both in terms of

rutting resistance and water susceptibility. In particular, the rutting resistance (Figure 2.7) of the recycled SMA mixture was found to be even better that the virgin SMA one.
Thus, the presence of selected RAP in new SMA layers did not show any negative effect (Jensen, 2012).

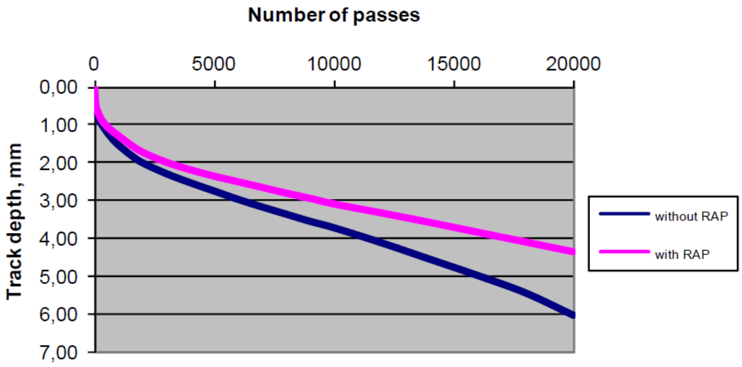


Figure 2.7 Wheel Tracker tests on virgin and recycled SMA mixtures (Jensen, 2012)

2.6.2 The use of RAP in PA mixtures

As already extensively described in paragraph 1.4, the use of Porous Asphalt (PA) layers is widely common all over the world; in Italy more that 80% of motorway surface layers are made with PA mixtures due to their ability to drain water as well as reduce traffic noise. The largely use of PA, both for new construction and rehabilitation interventions, require sustainable solutions that need to be properly studied for such delicate mixtures. Many European countries are moving in that direction developing sustainable solution for PA mixtures. In particular, in the Netherlands, the use of RAP in porous asphalt layers is permitted up to 20% (Molenaar et al., 2011).
The Michigan Technology University (MIT) carried out a very interesting research study in 2012 focused on the use of recycled materials as partial substitution of valuable virgin materials in new PA layers (Shu et al., 2012). In particular, the Compaction Energy Index (CEI), the permeability, the Indirect tensile resistance as well as the dynamic modulus (E*) of different porous asphalt mixtures were compared. Four PA mixtures were prepared and compacted by means of a Gyratory compactor:

- one control PA mixture prepared without any RAP aggregates or WMA additives
- one PA mixture without any RAP aggregates and 0.25% of WMA additive
- one PA mixture with 15% of RAP and without any WMA additives
- one PA mixture with 15% of RAP and 0.25% of WMA additive

The Compaction Energy Index represents the energy require to in situ compact mixtures until reaching the desired voids content; lower CEI values are desirable since it indicates that the mixture can be compacted easily. In the study (Shu et al., 2012), the PA mixtures

including RAP aggregates showed higher CEI values, meaning that the presence of the aged bitumen from RAP tends to stiff the mixture.
Permeability was evaluated by measuring the flow time of water through the surface of the specimens, in accordance to the American specification ASTM PS 129-01 (2001). All the mixtures analyzed exceeded the minimum required in terms of permeability coefficient (10^{-2} cm/s) meaning that also the recycled PA mixtures are capable to guarantee good drainage of the surface during rainfalls. However, it is important to underline that the two recycled PA mixtures demonstrated the lower values in terms if permeability.
Moreover, Indirect Tensile tests were carried out in accordance to AASHTO T283 showing that the recycled PA mixtures are characterized by slightly higher resistance than the virgin ones (Figure 2.8), as already shown by other Authors (Shu et al., 2008). Since Indirect Tensile resistance is considered to be related to the fatigue resistance, such an outcome allows concluding that recycled PA mixtures are more resistance in terms of fatigue than virgin ones.

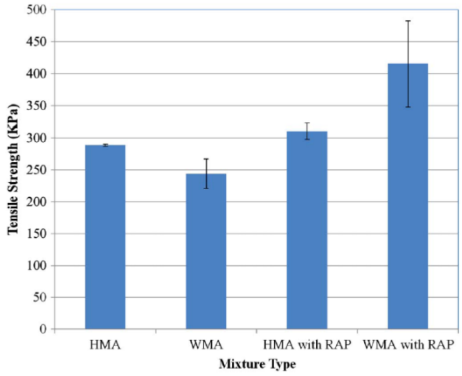


Figure 2.8 Results of Indirect Tensile tests at T= 24°C for PA mixtures (Shu et al., 2012)

Finally, data of the dynamic modulus E* at various temperatures and frequencies allowed the determination of the master curves of each PA mixture at the reference temperature selected at -5°C. Results (Figure 2.9) show that both the recycled PA mixtures are characterized by higher modulus values with respect to the virgin ones due to the presence of the aged bitumen from RAP. Moreover, since dynamic modulus is strongly related to the rutting resistance (43), results also demonstrated that PA mixtures including WMA additives can be affected by premature rutting compared to traditional PA mixtures.

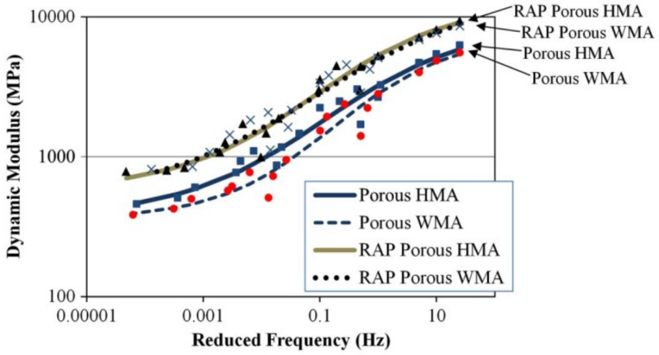


Figure 2.9 Comparison of dynamic modulus among different PA mixtures (Shu et al., 2012)

Chapter 3.

Preliminary study on adhesion properties between bitumen and RAP aggregates

The durability of porous asphalt mixtures is strongly related to the adhesion properties developed at the interface between bitumen and aggregates. The high air voids content that characterized PA mixtures, in fact, leads to small area of contact between particles: as a consequence, the adhesion between aggregates through the bituminous/mastic phase needs to be carefully evaluated. The loss of adhesion implies a rapid deterioration (e.g. stripping, raveling) of pavement layers under traffic loads, especially when the pavement is affected by the presence of moisture. The presence of water, in fact, can interfere with the bitumen-aggregates bond leading to premature de-bonding effects. Adhesion characteristics can be influenced by the presence of Reclaimed Asphalt Pavement (RAP) within the mixtures. RAP consists of aggregates particles cover with aged bitumen; thus, adhesion properties at the interface between virgin bitumen and aged bitumen that coats RAP aggregate surface need to be properly investigated. An innovative procedure to evaluate the compatibility of the system virgin bitumen/RAP aggregate is proposed in this preliminary study. This procedure allows simulating in laboratory the substrate of a Reclaimed Asphalt aggregate and can integrate the Bitumen Bond Strength test currently used to investigate bonding properties and water sensitivity of the system bitumen-virgin aggregates. Tests were conducted using basalt aggregates and a polymer modified bitumen and two conditioning types (dry, wet). The investigation suggests that the presence of a thin film of aged bitumen in the artificial RAP aggregates provides higher adhesion with the virgin bitumen. This result is detectable both in terms of failure type (failure within the bitumen) and bond strength values, regardless of the conditioning type (dry and wet). In particular, the loss in performance due to the effect of water experienced by the virgin aggregates is much more evident than the loss experienced by the coated aggregates. Experimental results obtained in this phase strongly encouraged to proceed with the study of recycled porous mixtures.

3.1. Introduction

Adhesion is a complex phenomenon related to the mineralogical and morphological nature of aggregates, as well as to the chemical bitumen composition and the environmental conditions. Nowadays, its evaluation becomes even more complicated as an increasing percentage of Reclaimed Asphalt is used in the production of new asphalt mixes. Thus, adhesion properties are also related to the mechanisms developed at the interface between virgin bitumen and aged bitumen that coats the RAP aggregate surface.

It is well known that when RAP is mixed with virgin aggregates and virgin bitumen, many chemical and physical interactions (e.g. partial blending of RAP bitumen, aging effects) occur in the hot recycled mix asphalt (Shirodokar et al., 2011). Although these interactions have not been fully identified, they are believed to strongly affect the properties of recycled mixtures (Daniel and Lachance, 2005; Singh et al., 2012) making the hot mix asphalt more prone to premature failure. In this sense, over the last years, several national agencies have limited the amount of RAP to use in the different layers of the pavement structure (Copeland, 2011; Mollenhauer and Gaspar, 2012). In particular, the use of RAP is limited in surface layers since they are directly expose to environmental conditions and traffic loads; in case of porous asphalt mixtures, the limitation is more restricted (or RAP is even forbidden in some countries) due to their inherent low durability and mix design issues. At the same time, the growing availability of milled pavement materials, along with environmental and economic issues related to the limited supply of virgin aggregates and bitumen, are inducing to increase the amount of RAP incorporated in new mixtures.

Taking into account that RAP is constituted by aggregates covered with a thin film of bitumen, the virgin bitumen added for the production of new mixtures has to develop appropriate bond strength with such coated particles. Furthermore, all the oxidation phenomena experienced by a flexible pavement during the mixing and construction phase and the whole service life could alter the RAP bitumen composition and its ability to react with virgin bitumens (Lesueur, 2009; Ruan et al., 2003), especially in case of PA layers due to the high air voids content. The adhesion developed at the interface between RAP aggregates and virgin asphalt bitumen is one of the main mechanisms that influences the behavior and the performance of a recycled mixture. Flexible pavement distresses (e.g. fatigue cracking and rutting) are strongly related to loss of this type of adhesion, particularly in presence of water which induces less compatibility between bitumen and aggregates (moisture damage). The interactions that develop at the bitumen-aggregate interface are strongly affected by the materials characteristics, both from a chemical (e.g. molecular orientation and polarity, aggregate charge distribution) and a physical (e.g. surface texture and porosity) point of view (D'Angelo and Anderson, 2003; Kanitpong and Bahia, 2005; Robert et al., 1996). It has been shown (Kiggundu and Roberts, 1988) that several phenomena (e.g. detachment, displacement, pore pressure, hydraulic scour, pH instability, environmental effects), directly related to the adhesion between aggregate and bitumen, can initiate and amplify moisture damage.

In this context, an effective and practical method, successfully employed to identify the bitumen/aggregate affinity and the related moisture damage, is the Bitumen Bond Strength (BBS) test developed by Moraes et al. (Moraes et al., 2011) starting from the initial attempts of Kanitpong et al. and Canestrari et al. (Canestrari et al., 2010; Kanitpong and Bahia, 2003). The BBS test allows the quantification of the bond strength that develops at the interface between aggregates substrates and bitumens in both dry and wet condition. It is able to guarantee optimum reproducibility and repeatability measuring the effects due to different type of conditioning and materials with various chemical and rheological characteristics (Canestrari et al., 2010; Moraes et al., 2011). Since the adhesion issue becomes even more complex in recycled mixtures considering the presence of RAP aggregates that directly interact with the virgin bitumen, in this study an innovative testing protocol has been employed in order to integrate the current BBS test with a method able to assess bond properties and water sensitivity of hot recycled asphalt mixtures (Canestrari et al., 2014). In particular, the experimental investigation proposes an original procedure for the laboratory preparation of aggregate substrates aimed to properly simulate the RAP surface. The aggregate plates prepared following this method are then used to perform the standard BBS test.

This preliminary study focuses on the evaluation of the bond strength properties (adhesion and cohesion) between virgin bitumens and an artificial RAP substrate simulating the real RAP aggregate. By comparing the results obtained performing BBS test on traditional (virgin aggregate) and on coated aggregate (artificial RAP aggregate) samples, in both dry and wet conditions, the effects of RAP used for the production of new asphalt mixes can be investigated in terms of cohesion/adhesion properties and moisture damage resistance.

Basalt substrates were employed to simulate both virgin and RAP aggregates since PA mixtures for Italian motorway are currently designed with basalt aggregates and the RAP considered in this experimental research program comes only from milled PA surface layers. Moreover, SBS (Styrene-Butadiene- Styrene) polymer modified bitumen was selected since it represents the most common type of bitumen currently employed for the production of PA mixtures.

3.2. Experimental investigation

3.2.1 Materials

As already mentioned, a basalt aggregate was selected as substrate since it represents the aggregate type that is currently employed to prepare PA mixtures in Italian motorway. Perfectly horizontal and flat square aggregate plates were prepared by sawing stone blocks in adequate dimensions ($10 \times 10\ cm^2$) to allow the positioning of five specimens. A SBS polymer modified bitumens was employed for the evaluation of the adhesion properties and it is the bitumen that has been used in the entire experimental program. The main bitumen characteristics are reported in Table 3.1.

Table 3.1 Bitumen main characteristics.

Bitumen characteristics	Standard	Unit	Value
SBS polymer content by weight	–	%	3,8
Penetration [25°C; 100g; 5s]	EN 1426	0.1mm	54
Ring and ball softening point	EN 1427	°C	71
Elastic recovery [25°C; 5cm/min]	EN 13398	%	89
Dynamic viscosity @ 135°C	EN 12595	Pa·s	1,24
Mass loss after RTFOT	EN 12607-1	%	0,05
Penetration after RTFOT	EN 1426	0.1mm	27
Ring and ball softening point after RTFOT	EN 1427	°C	77

3.2.2 Experimental program

The experimental program is summarized in Table 3.2 and involved one polymer modified bitumen and one aggregate source (basalt). Two different surface treatments were considered: a virgin aggregate with untreated surface and an artificial RAP aggregate having a surface coated with a thin film of bitumen. The same SBS polymer modified bitumen was selected because of a great amount of RAP available in Italy is currently constituted by bitumen with high modification levels; in particular, the RAP employed in this experimental program comes only from milled PA surface layers that are prepared with modified bitumens.
For each test configuration, samples were conditioned in dry or wet environment before testing. Dry specimens were left at 25 °C for 24 hours in a climatic chamber whereas wet specimens were conditioned at 40 °C for 24 hours in a water bath and then at 25 °C for 2 hours in air dry conditions. For each test configuration, five replicates were performed in order to obtain reliable results.

Table 3.2 Experimental Program

Aggregate substrate	Surface treatment	Conditioning	Replicates
Basalt	Virgin	Dry	5
		Wet	5
	Coated	Dry	5
		Wet	5

Procedure for the preparation of the artificial RAP substrate

The preparation of the artificial RAP aggregate consists in the following steps:

– heat bitumen as long as a fluid consistency is obtained at a temperature ranged between 135 to 160 °C, depending on bitumen used (neat or modified). The consistency of bitumen is an important aspect that can play a crucial role in the development of good bonding properties between bitumen and aggregate. In this study, the bitumen was heated at 160 °C in order to guarantee a suitable viscosity;

- heat aggregate plates at 135 °C for 1 hour in a forced-draft oven;
- coat the aggregate surface with a uniform and homogeneous thin film of bitumen by means of a brush. The amount of bitumen used to coat the aggregate plate ranged between 0.2 and 0.3 g, depending on the aggregate surface porosity and on its tendency to absorb asphalt bitumen. This quantity was found to ensure a bitumen film thickness of about 9.5 μm which is believed to reproduce the bitumen thickness coating a RAP aggregate adequately. In fact, in a Hot Mix Asphalt with a bitumen content of about 5.5%, an average optimum asphalt film thickness should vary from 9.5 to 10.5 μm (Ruan et al., 2003; Sengoz and Agar, 2007). Considering that during the pavement service life and the storage period in the stockpiles RAP aggregates have experienced many phenomena that could reduce the coating bitumen thickness, it was decided to adopt the lower limit of the above-mentioned range;
- age the coated aggregate plates at short and long-term in a forced-draft oven by heating the plates at 135±3 °C for 4±0.5 hours and subsequently at 85±3 °C for 120±0.5 hours, according to AASHTO Standard R30 specification on the aging procedures for hot mix asphalt samples.

The last step of the procedure aimed to properly simulate the oxidative phenomena experienced by the milled material during the whole service life. This is a key aspect as aging produces chemical changes in the asphalt bitumen composition (Dehouche et al., 2012; Le Guern et al., 2010; Kandhal et al., 1998) that can have significant effects also in terms of bond strength interactions at the interface between virgin and aged bitumen. The coated aggregate plates produced with the above described procedure have provided the artificial RAP substrates needed to prepare the BBS specimen (Figure 3.1) used to perform the pull-off test.

BBS test method

The BBS test was recently accepted as provisional AASTHO test method (TP 91) and it is used to quantify the cohesion/adhesion properties and the moisture sensitivity of bitumen-aggregate systems. It is a very quick, simple and practical test able to guarantee optimum repeatability and reproducibility (Canestrari et al., 2010; Moraes et al., 2011). It allows the measurement of the bond strength between aggregates and bitumens in both dry and wet conditions, on different aggregate substrates and considering bitumens with different chemical and rheological characteristics (Canestrari et al., 2010; Moraes et al., 2011). The test is performed by means of specific equipment, composed by a portable pneumatic adhesion tester according to ASTM D4541. The device (Figure 3.2-a) is also equipped by a pressure hose, a piston, a reaction plate, and a metal pull-stub (diameter = 12.7 mm, surrounding edge = 0.3 mm, Figure 9.3-b). The bond strength is considered as the tensile force required breaking the bond between the bitumen and the aggregate component. A small amount of virgin bitumen (about 0.08 g) is placed onto the pull-stub surface previously heated at 65 °C for a minimum of 30 minutes along with the aggregate plates.

Afterwards, the pull-stub is pressed onto the aggregate surface avoiding twisting stresses that could entrap air between the bitumen and the aggregate (Moraes et al., 2011). After the required conditioning (dry or wet), the piston and the reaction plate are screwed to the stub and the air pressure is steadily transmitted through the pneumatic system with a loading rate of 315 kPa/s (Figure 3.3). In this way, an increasing pulling force is applied to the specimen until the failure is reached. Then, the pressure value displayed in the test equipment is recorded and the failure type is visually identified. Finally, the pressure value is converted into the Pull-Off Tensile Strength (POTS) as a function of the bonding surface area.

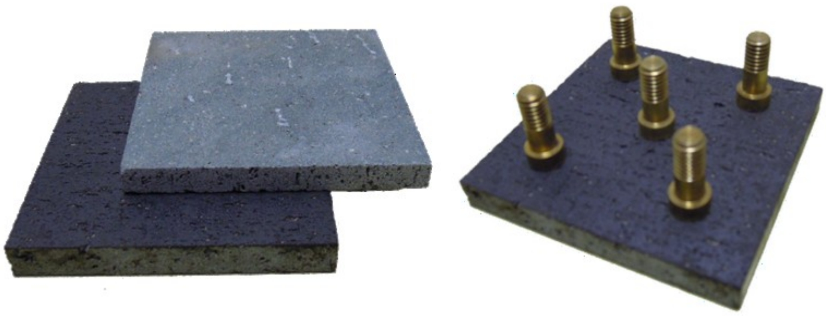
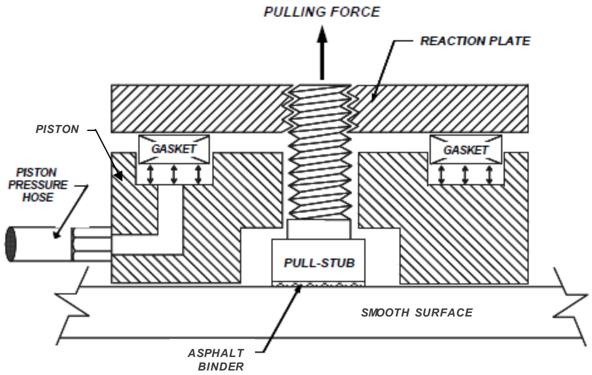


Figure 3.1 BBS test samples (pull-stubs/aggregates substrate system)

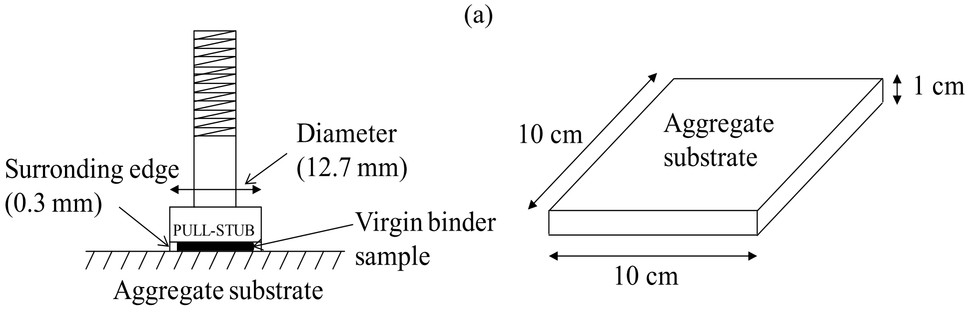
The debonding phenomenon of the bitumen-aggregate system may occur with three different failure mechanisms (Bhasin et al., 2006):

- failure at the interface (coded A), defined as loss of adhesive bond strength between bitumen and aggregate;
- failure within the bitumen (coded C), characterized by loss of cohesive strength within the bituminous component caused by the rupture of bonds in the asphalt film;
- intermediate failure (coded B), recorded when the two failure mechanisms previously described occur simultaneously.

The failure type was visually identified after each repetition.



(a)

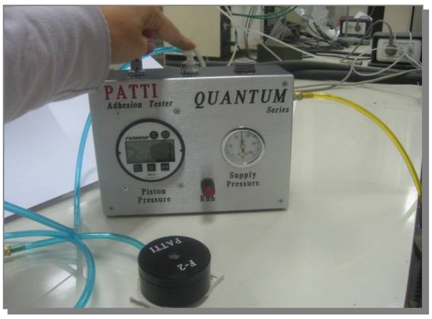


(b)

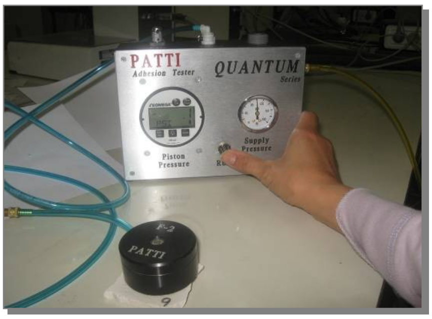
Figure 3.2 Schematic representation of BBS equipment: (a) cross-section of BBS test device (Canestrari et al., 2010), (b) pull-off stub and aggregate plate dimensions

a – Installation of the piston around the pull-stub

b – Positioning and screwing of the reaction plate around the pull-stub



c – Connection of the air to the equipment through the blue pressure hose



d – Transmission of the air to the piston through the pneumatic system

e – Failure of the adhesion between the pull-stub and the aggregate plate

f – Final configuration

Figure 3.3 Operational sequences of the BBS test

3.3. Experimental results

The mean BBS test results in terms of pull-off tensile strength of the studied materials are shown in Figure 3.4, along with error bars reporting the minimum and the maximum value obtained for each testing condition. Moreover, the prevalent failure type that was identified for each configuration is also reported in the Figure 3.4.

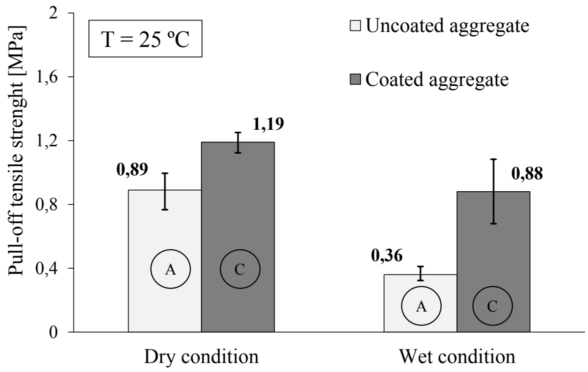


Figure 3.4 BBS mean test results

The coated aggregates, representative of RAP aggregates within recycled mixtures, showed significantly higher bond strength compared to the untreated surface, representative of virgin aggregates. The better performance demonstrated by recycled aggregates with respect to virgin aggregates are evident in both dry and wet conditions: the increase in performance is equal to 25% in dry condition and 59% in wet condition. Such a result suggests that the presence of a thin film of aged bitumen that covers the aggregates enhances the adhesion properties at the interface with the virgin aggregates. This improvement in adhesion performance was even more accentuated in presence of water. It is also interesting to notice that the coated aggregate performance after the water conditioning were almost equal to the virgin aggregate performance in dry condition. Moreover, the loss of bond strength due to the effect of water experienced by the coated aggregates (−26%) is significantly lower than the loss experienced by the uncoated aggregates (−60%), suggesting that the artificial RAP substrate is able to strongly reduce the water sensitivity of the bitumen-aggregate systems, improving the adhesion properties.
As far as failure type is concerned, it is possible to notice that virgin aggregates demonstrated adhesive failure whereas cohesive failure was demonstrated by coated aggregates in both dry and wet conditions. Therefore, RAP aggregate appears able to guarantee a better affinity and an improved bond between the substrate and the virgin bitumen, also improving the material performance in wet condition respect to the virgin aggregate. In fact, the failure within the bitumen (type C) recorded for coated aggregates in dry condition does not change into failure at the interface (type A) after the water conditioning. This result could be partially explained considering that the oxidation process experienced by the artificial RAP substrate reduces the free radicals of the material, making the artificial aggregate more resistant to stripping than the virgin one (Little and Jones, 2003). Moreover, results allow admitting that the thin film of aged bitumen that coats the aggregate surface is able to reactivate and develop chemical interactions with the virgin bitumen, ensuring an optimum collaboration between the two bituminous components and

an improved bond with the aggregate surface (Canestrari et al., 2014). Thus, the bond strength reduction due to water conditioning experienced by the system bitumen-coated aggregates is principally due to the effects of moisture on inner cohesion of the bituminous component. On the other hand, the virgin aggregate, that exhibited a failure at the interface (type A) in both dry and wet condition, showed that water conditioning causes a further reduction in the affinity between bitumen and aggregates.

The overall investigation suggests that the use of RAP in the production of new bituminous mixes does not penalize the bond interactions between virgin bitumen and aggregates. On the contrary, the artificial RAP substrate, used to simulate a real RAP aggregate, appears able to provide an improvement in the adhesion performance of the mixture and to reduce the water sensitivity of the system bitumen-aggregate considerably. The presence of a thin film of aged bitumen in the artificial RAP aggregates, in fact, ensures the development of higher adhesion with the virgin bitumen. This result is detectable both in terms of failure type (failure within the bitumen) and bond strength values, regardless of the conditioning type (dry and wet). In particular, the loss in performance due to the effect of water experienced by the virgin aggregates is much more evident than the loss experienced by the coated aggregates. Such promising results strongly encouraged to proceed with the mix design and performance evaluation of recycled porous mixtures.

Chapter 4.

Use of 15% RAP in PA mixtures

4.1. Introduction

It is well known that the re-use of milled materials in new asphalt mixtures optimizes the use of natural resources and sustains the asphalt pavement industry since it allows considerable savings in materials, cost and energy. With the increase of demand and limited aggregate and bitumen supply, along with strict environmental regulations, recycling had become a priority goal for both administrations and producers (Jensen, 2012; Molenaar et al., 2011). Thus, a technical support to evaluate any potential deterioration or improvement in performances compared with the mixtures currently used in road construction is necessary.
As already mentioned, in many countries porous asphalt mixtures are nowadays extensively used as motorway surface layer due to their benefits in reducing traffic noise and improving safety during wet conditions (enhanced skid resistance and reduced spray and splash). Despite these advantages, it is well known that the major disadvantage of porous asphalt is the limited durability due to its high air void content that makes PA more susceptible to ravelling and water damage (Alvarez et al., 2011; Copeland, 2011; De La Roche et al., 2013). Ravelling, defined as the loss of aggregates from the surface layer, is caused by two main failure mechanisms: cohesive (damage within the mastic) or adhesive (loss of bond between asphalt bitumen or mastic and the aggregates). Both cohesive and adhesive failure are strongly influenced by the effect of water and aging (Partl et al., 2010, Poulikakos et al., 2012). In PA mixtures, the contact between aggregate particles through asphalt mastic occurs in a very small area due to the high void content of the mixtures. Thus, the “affinity” at the bitumen-aggregate interface should be deeply investigated as the new virgin bitumen interacts with the thin film of aged mastic that covers the RAP aggregates. In this sense, the performance of the final mixture will sensibly depend on the adhesion developed between new virgin bitumen and RAP aggregates.
As a consequence of their limited durability, maintenance processes in the motorway network are often performed on porous asphalt layers. Such activities lead to a considerable amount of milled material from old PA layers that is growing dramatically. However, since in the technical specifications of several Countries only virgin aggregates are currently allowed in PA, reclaimed asphalt pavement (RAP) can be usually re-used as aggregate only in hot dense-graded mixtures for new base and/or bitumen layers. In order to promote the use of RAP also in the PA surface layers, milled material coming from PA mixtures needs to be stockpiled separately and re-used in a PA wearing course. This possibility is confirmed by recent studies (Hagos et al., 2007) that demonstrated that using RAP coming

from milled wearing courses do not compromise performance of new high quality wearing courses. As a matter of fact, in Netherland, reclaimed materials are already used in PA layers up to 20% (Hagos, 2008).

4.2. Laboratory mix design and investigation

4.2.1 Objectives

This experimental investigation has a twofold objective. First of all, it has been verified if the performance of porous asphalt mixtures can be still guaranteed when a certain percentage (15%) of course RAP aggregates is added to the mix. Moreover, the influence of different total bitumen contents on the recycled porous asphalt mixtures performance has been evaluated in order to optimize the mix design.
For these purposes five recycled asphalt mixtures prepared with different total bitumen content were investigated in terms of volumetric properties, durability and water resistance. The total bitumen content takes into account the virgin bitumen that is added during the mixing phase and the aged bitumen from RAP. Furthermore, a reference PA mixture was prepared by employing virgin aggregates and a virgin polymer modified bitumen, in accordance to the Italian specifications for motorway pavements (Autostrade SpA, 2013).
The research project focuses on the evaluation of durability issues as they are the main failure causes in PA. In this sense, Indirect tensile strength tests, particle loss (Cantabro) tests, semi circular bending (SCB) tests and repeated indirect tensile tests were carried out in both dry and wet conditions in order to evaluate acceptability, durability, fracture resistance and water sensitivity of recycled mixtures. Further mechanical properties (e.g. complex modulus) were not taken into account as performance indicators because PA surface layers do not play a structural role in the pavement.
A rheological characterization of the bitumen was not essential in this research study because only 15% RAP course aggregates (8/16 mm), with minor contribution of aged bitumen, had been included in the investigated porous asphalt mixtures. In fact, according to McDaniel and Anderson (2001), RAP can be used up to 15% without changing the virgin bitumen performance grade.

4.2.2 Materials

One reference PA mixture and five recycled PA mixtures prepared with different bitumen contents were investigated in this research study. The reference mixture, hereafter named P00_5.00%, was a typical porous mixture including only virgin aggregates and 5.00% of virgin bitumen by aggregate weight. The recycled mixtures, hereafter named R15_bitumen content%, were characterized by 15% of coarse RAP aggregates and five different total bitumen contents from 4.50% to 5.50% with steps of 0.25%. The overall mixtures and their codes are summarized in Table 4.1.

Table 4.1 Mixtures identification codes

Mixture		Total bitumen content (by aggregate weight)	RAP aggregate content (by aggregate weight)
P00_5.00%	Reference mixture	5.00%	0%
R15_4.50%	Recycled mixture	4.50%	15%
R15_4.75%	Recycled mixture	4.75%	15%
R15_5.00%	Recycled mixture	5.00%	15%
R15_5.25%	Recycled mixture	5.25%	15%
R15_5.50%	Recycled mixture	5.50%	15%

Grading curve optimization

A preliminary study related to virgin and recycled aggregates was carried out in order to build and optimize the final aggregates grading curve for the reference mixture as well as the recycled mixtures.
The same virgin aggregates (Figure 4.1) were employed to prepare all the studied mixtures and consisted in basalt aggregates available in three portions (8/16 mm – 5/11 mm – 0/4 mm) and limestone filler.

Basalt virgin aggregate 8/16mm | Basalt virgin aggregate 5/11mm | Basalt virgin aggregate 0/4mm | Limestone filler

Figure 4.1 Virgin aggregates.

For each fraction of aggregate and for the filler, the grading curve was evaluated as the average of three replicates, in accordance to EN 933-1 (2012). The results are reported in Table 4.2 for all the virgin aggregates.

Table 4.2 Virgin aggregates grading curve

Sieve size [mm]	Cumulative passing Basalt 8/16 mm [%]	Cumulative passing Basalt 5/11 mm [%]	Cumulative passing Basalt 0/4 mm [%]	Cumulative passing Limestone filler [%]
31,5	100	100	100	100
20	100	100	100	100
14	88,52	100	100	100
10	20,63	100	100	100
6,3	0,93	72,47	100	100
2	0,56	1,58	87,74	100
0,5	0,54	1,43	43,08	100
0,25	0,52	1,40	30,37	100
0,063	0,51	1,09	14,59	97,96

Such virgin aggregates were combined in order to obtain the design grading curve reported in Figure 4.2 that was used for preparing the reference mixture; the lower and upper standard grading curve are in accordance to the Italian technical specification for motorway (Autostrade SpA, 2013).

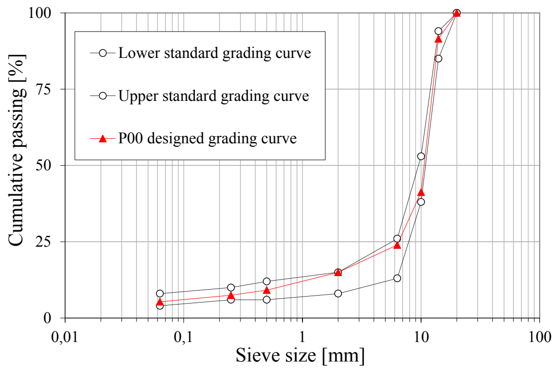


Figure 4.2. Designed grading curve for the reference mixture P00.

In addition, RAP aggregates were preliminary studied in order to optimize the aggregate grading curve for the recycled mixtures. It is important to underline that the recycled aggregates used on this experimental program were purposely selected from old PA asphalt layers. Since porous asphalt mixtures in Italian motorway are prepared only with basalt aggregates and polymer modified bitumen, the selected RAP is constitutes of good quality basalt aggregates and aged modified bitumen. As mentioned before, one of the main concerns related to the use of recycled materials within PA is the volumetric properties of the final mixture due to the high variability of RAP and its unpredictable composition. As a consequence, the use of un-fractioned RAP does not guarantee control on the actual composition of the recycled PA mixture that can results in low air voids content. In order to avoid such issues, milled asphalt mixtures from old PA mixtures were store separately from the others and then sieve at the asphalt plant, in such way that only the coarse aggregates can be employed to produce new recycled PA mixtures. This procedure ensures good quality recycled PA mixtures since the nature and the composition of the employed RAP is known. Figure 4.3 allows a direct comparison of the RAP aggregates and the coarse basalt aggregates employed in this experimental program. It is possible to see that coarse RAP aggregates are very similar to the virgin aggregates cover with a thin film of aged bitumen.

Figure 4.3. Recycled coarse aggregates (left) and virgin coarse aggregates (right).

RAP aggregates were extracted in order to evaluate the bitumen content as well as the extracted aggregates grading curve. The grading curve obtained as the average of three repetitions on the extracted RAP aggregates is reported in Table 4.3. It is possible to notice that the grading curve of the coarse RAP aggregates is similar to the grading curve of the coarse basalt virgin aggregates but it is characterized by higher fine particles content.

Table 4.3 Recycled aggregates grading curve

Sieve size [mm]	Cumulative passing RA 8/16 mm [%]
31,5	100
20	100
14	97,40
10	75,50
6,3	27,70
2	14,88
0,5	10,59
0,25	8,68
0,063	4,49

The same virgin aggregates used for producing the reference mixture were employed to produce all the recycled mixtures along with 15% of RAP aggregates. It is important to underline that the lower and upper standard grading curve selected as limits are those requested from the Italian technical specification for motorway (Autostrade SpA, 2013). The final aggregates grading curve used to produce all the recycled mixtures is reported in Figure 4.4.

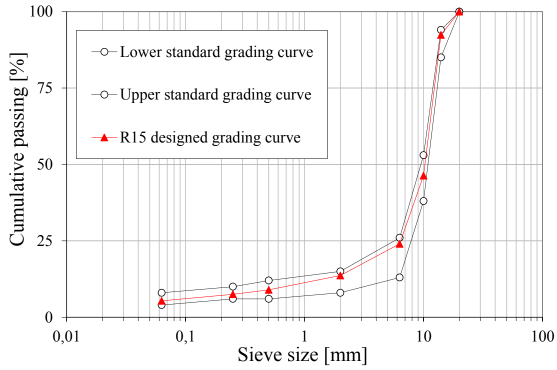


Figure 4.4. Designed grading curve for the recycled mixtures R15.

The aggregates mix design for the reference mixture and all the recycled mixture is reported in Table 4.4 for comparison purposes. It can be observed that coarse RAP mainly substitutes basalt coarse fraction 8/16 and partly substitutes basalt 0/4 whereas basalt 5/11 mm and filler remained unchanged. This fact implies that the recycled mixtures were characterized by almost the same grading curve of the reference mixture without compromising volumetric properties and thus drainability.

Table 4.4 Aggregate mix design

	Percentage combination of aggregate				
	Basalt 8/16 mm	RAP 8/16 mm	Basalt 5/11 mm	Basalt 0/4 mm	Filler
Reference mixture [%]	74	0	13	10	3
Recycled mixtures [%]	63	15	10	9	3

The two grading curves are compared also in Table 4.5 where it appears clear that they are practically equivalent.

Table 4.5 Final aggregate grading curve

Sieve size [mm]	Cumulative passing Reference mixture [%]	Cumulative passing Recycled mixtures [%]	Lower standard limit [%]	Upper standard limit [%]
31,5	100	100	100	100
20	100	100	100	100
14	91,50	92,38	85	94
10	41,26	46,32	38	53
6,3	23,93	23,99	13	26
2	14,98	13,64	8	15
0,5	9,14	8,95	6	12
0,25	7,48	7,51	6	10
0,063	5,32	5,35	4	8

Furthermore, the bulk density of each aggregate fraction was measured by means of a pycnometer, in accordance to EN 1097-6. Since it was not possible to measure the bulk density of the RAP aggregates, it was assumed equal to the one of the coarse basalt virgin aggregate (8/16mm). The results are reported in Table 4.6.

Table 4.6 Aggregate bulk density

	Basalt 8/16 mm	RAP 8/16 mm	Basalt 5/11 mm	Basalt 0/4 mm	Filler
Bulk density [g/cm^3]	2.765	2.765	2.775	2.801	2.690

The bulk density values of each aggregate fraction were combined in order to evaluate the bulk density of the final aggregate mixtures, based on the aggregates percentage combination selected for the reference mixture and the recycled mixtures (Table 4.4). Therefore, the bulk density of the final aggregate mixture was 2.768 g/cm^3 for the reference mixture and 2.767 g/cm^3 for the recycled mixtures.

Bitumen characteristics

The same virgin bitumen was used to produce both the reference PA mixture as well as all the recycled PA mixtures. It was a polymer modified bitumen including 3.8% of Styrene-Butadiene-Styrene and it represents the most common modified bitumen actually used in Italy fir this type of mixtures. The main characteristics of the virgin bitumen are reported in Table 4.7.

It is important to underline that also the aged bitumen included within RAP is a SBS modified bitumen and it is of the same type of the virgin bitumen. In fact, the RAP used in this experimental program came from old PA surface layers that are currently produced by means of polymer modified bitumen.

Table 4.7 Basic characteristics of the virgin bitumen

Binder characteristics	Standard	Unit	Value
SBS polymer content by weight	–	%	3.8
Penetration [25°C; 100g; 5s]	EN 1426	0.1mm	54
Ring and ball softening point	EN 1427	°C	71
Elastic recovery [25°C; 5cm/min]	EN 13398	%	89
Dynamic viscosity @ 135°C	EN 12595	Pa·s	1.24
Mass loss after RTFOT	EN 12607-1	%	0.05
Penetration after RTFOT	EN 1426	0.1mm	27
Ring and ball softening point after RTFOT	EN 1427	°C	77

Mix design

The mix design for the reference mixture was selected according to the Italian technical recommendations for motorway; therefore, the bitumen content was selected equal to 5.00% by the aggregates weight within the recommended range (5.00% and 6.00% by the aggregates weight) and the reference air voids content was fixed at 20% (V≥18% is the recommendation).
As far as the recycled mixtures are concerned, in order to perform a correct mix design, the estimation of the RAP bitumen content is essential. Therefore, three extractions were performed on different RAP sample; the results are reported in Table 4.8.

Table 4.8 RAP bitumen content

	I extraction	**II extraction**	**III extraction**	**Average value**
Bitumen content with respect to the RAP mixture [%]	3.98	4.15	3.98	4.04

It is well known that, during the mixtures production, part of the aged bitumen within RAP acts as a “black rock” whereas part of it becomes “working bitumen” and collaborates with the virgin bitumen. Since it is not easy to estimate how much of the aged bitumen acts as “black rock”, in this experimental program it was considered that all the aged bitumen within RAP becomes “working bitumen” and it is part of the total bitumen of the mixture. Therefore, recycled mixtures were prepared with different total bitumen contents in order to carry out a performance comparison with the reference mixture and evaluate the optimized total bitumen content. Since the recycled mixture must perform as well as the reference virgin mixture, the same standards need to be properly addresses; thus, bitumen content within 5.00% and 6.00% by aggregates weight needs to be guaranteed. It was chosen to produce five recycled mixtures with the same grading curve characterized by five different bitumen contents from 4.50 to 5.50 with a step of 0.25%.

In order to guarantee the various total bitumen contents, it was necessary to considered the RAP bitumen and calculate the virgin bitumen to add during mixtures production. Moreover, it was considered that the grading curve of the recycled mixtures was built by using the RAP aggregates after the bitumen extraction: since RAP consists of aggregates coated by a film of bitumen, a higher quantity of RAP needs to be taken into account. An exemplification of the mix design used for the recycled mixtures is reported below:

- RAP aggregates content: $\%\mathrm{RAP} = 15\%$
- RAP bitumen content: $\%\mathrm{B}_{\mathrm{RAP}} = 4.04\%$ by RAP weight

Therefore, the RAP bitumen content is 0.6% by total aggregate weight, and the following formula can be used to calculate the quantity of materials referred to 10 kg of total aggregates:

- $\mathrm{P}_{\mathrm{b}}^{\mathrm{RAP}} = \mathrm{P}_{\mathrm{T}} * \ \%\mathrm{B}_{\mathrm{RAP}}$
- $\mathrm{P}_{\mathrm{T}} - \mathrm{P}_{\mathrm{b}}^{\mathrm{RAP}} = \%\mathrm{RAP} * \mathrm{P}_{\mathrm{Agg.}} = 1500$
- $\mathrm{P}_{\mathrm{T}} = \frac{1500}{1-4.04\%} = 1562.5\ \mathrm{g}$
- $\mathrm{P}_{\mathrm{b}}^{\mathrm{RAP}} = 4.04\% * 1562.5 = 62.5\ \mathrm{g}$

where:
$\mathrm{P}_{\mathrm{b}}^{\mathrm{RAP}}$ is the RAP bitumen weight;
P_{T} is the RAP weight;
$\mathrm{P}_{\mathrm{Agg.}}$ is the RAP aggregates weight, without taking into account the presence of RAP bitumen;

Thus, $\mathrm{P}_{\mathrm{b}}^{\mathrm{RAP}}$ is the quantity of RAP bitumen that needs to be subtracted from the total bitumen content in order to calculate the virgin bitumen weight.

Finally, 70% cellulose –30% glass fibers were added to aggregates before the mixture preparation for all mixtures (both reference and recycled mixtures) in order to avoid draindown problem. The dosage was selected equal to 0.3% by the aggregates weight, according to the Italian technical recommendations for motorway.

Laboratory protocol: mixtures preparation and compaction

Both reference mixture and recycled mixtures were mixed in the laboratory under the same conditions: the mixing temperature was selected equal to 170°C in order to achieve a proper fluidity of the polymer modified virgin bitumen. The virgin aggregates as well as the virgin

bitumen were kept in a ventilated oven at the mixing temperature for 3 hours in order to ensure a uniform heat of all the components. In case of recycled mixtures, RAP was mixed with the hot virgin aggregates and kept in the oven for 5 minutes before the mixtures preparation. Such a protocol was selected to simulate as close as possible the in-plant procedure.
Afterwards, mixtures were kept in a ventilated oven at the compaction temperature equal to 160°C for an hour in order to reproduce the transportation phase. Then, specimens were compacted by means of a gyratory compactor with 100 mm diameter molds as well as 150 mm diameter molds in order to achieve samples with homogeneous volumetric properties representing real field conditions (22% target air void content). The specimen dimensions and compaction mode (fixed height or fixed gyrations) were selected accordingly to the tests method and it is properly specified in the corresponding sections.

4.2.3 Experimental program

Test program

The mechanical performance related to the most common distresses of porous asphalt surface layers were evaluated in this experimental program in order to optimize the mix design of recycled PA mixtures. Since moisture damage is one of the major issues that limits the PA mixtures durability, all mechanical properties were measured in both dry and wet conditions in order to evaluate also the performance reduction due to water damage. In particular, in order to simulate the water damage, specimens of each mixture were conditioned in air (dry) or in water (wet) at 40°C for a period of 72 h, as suggested by EN 12697-12 (Method A).
Indirect tensile strength tests were carried out to ensure the requirements of the technical specifications as well as evaluate the resistance and the water damage due to the presence of recycled materials and the total bitumen content. Moreover, raveling behavior was evaluated by means of Cantabro tests since it represents one of the major causes of distress for PA surface layers. Raveling resistance is directly related to adhesion properties at the aggregates-bitumen interface that is highly influenced the presence of water: therefore, the comparison of the results before and after water conditioning was evaluated in order to compare the water susceptibility of the different mixtures. Moreover, the resistance of fracture propagation was evaluated by means of Semi-circular bending tests at intermediate temperature. Finally, the fracture initiation behavior of mixtures was established by means of repeated indirect tensile tests that were carried out in three different conditioning configurations: dry (air conditioning at tests temperature), wet condition (specimens kept in a water bath at 40 °C for 72 h and tested in dry condition at tests temperature) and submerged (specimens maintained in a water bath at 40 °C for 72 h and then tested at tests temperature completely submerged in a water bath). The overall experimental program is summarized in Table 4.9.

Table 4.9 Experimental program

Mixtures	*Indirect Tensile tests*		*Cantabro tests*		*Semi-circualr bending tests*		*Repeated indirect tensile tests*		
	T=25°C		*T=25°C*		*T=10°C*		*T=20°C*		
	dry	wet	dry	wet	dry	wet	dry	wet	submerged
P00_5.00%	4	4	4	4	4	4	3	3	3
R15_4.50%	4	4	4	4	4	4	3	3	3
R15_4.75%	4	4	4	4	4	4	3	3	3
R15_5.00%	4	4	4	4	4	4	3	3	3
R15_5.25%	4	4	4	4	4	4	3	3	3
R15_5.50%	4	4	4	4	4	4	3	3	3
TOTAL	**48**		**48**		**48**		**54**		

4.2.4 Equipment, test methods and data analysis

In this paragraph, tests procedure and parameters are given in details and the data analysis is specified.

Indirect Tensile test

The indirect tensile test was carried out at 25°C on gyratory compacted specimens; in particular, the specimens were characterized by 100 mm diameter and were compacted by means of 130 gyrations, in accordance to Italian technical specification for motorway. The testing apparatus applies a diametrical line load (Figure 4.5) by imposing a deformation of 25 mm/min until the maximum failure strength is reached, in accordance to EN 120697-23.

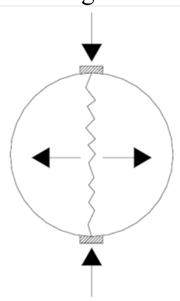


Figure 4.5. Indirect Tensile test configuration.

The Indirect Tensile Strength (ITS) and the Indirect Tensile Coefficient can be calculated using Equation (4.1) and (4.2), respectively:

$$ITS = \frac{2P}{\pi * t * d} \tag{4.1}$$

where P is the maximum load (kN), t is the specimen height (mm) and d is the specimen diameter (mm).

$$ITC = \frac{\pi * d * ITS}{2D_T} \tag{4.2}$$

where d is the specimen diameter (mm), ITS is the Indirect tensile strength (GPa) and D_T is the vertical deformation at failure (mm).

The water susceptibility was evaluated by means of the Indirect Tensile Strength Ratio (ITSR) as specified in Equation (4.3), in accordance to EN 12697-12:

$$ITSR = \frac{ITS_{wet}}{ITS_{dry}} \times 100 \tag{4.3}$$

Cantabro test

Cantabro tests were performed at 25°C on Marshall compacted samples in accordance with EN 12697-17. This test evaluates the resistance to abrasion of porous asphalt mixtures, by estimating the internal cohesion between particles. Particularly, the Cantabro test consists in subjecting an asphalt concrete sample to 300 revolutions (30 revolutions/minute) inside the Los Angeles machine drum without any metal balls. The mass loss at the end of the test of such specimen gives a measure of the resistance to raveling of the corresponding mixture. In particular, the Particle Loss (PL) values were calculated as specified in Equation (4.4):

$$PL = \frac{Wi - W_f}{Wi} \times 100 \tag{4.4}$$

where W_i is the specimen weight before Cantabro test (g) and W_f is the specimen weight after Cantabro test (g).

Semi-circular bending test

The capability of mixtures to resist cracking propagation represents an important property to ensure durability of the pavement layer. Particularly, traffic loads on pavement surfaces produces tensile stress at the bottom of the layer that leads to fatigue cracks. Once cracks have been formed, they start to spread and became visible; the crack growth proceeds until there is the possibility of crack propagation through the stressed body. Semi-circular bending (SCB) tests had been performed on the studied mixtures to analyze crack propagation potential in relation to the presence of recycled material in the PA since SCB

test had been found to be a valid method to characterize the fracture properties of asphalt mixtures (Arabani et al., 2009).
The test was carried out on half cylindrical specimen having a thickness of (50±3) mm and a diameter of (150±1) mm, in accordance to EN 12697-44. In particular, each SCB specimen was cut from gyratory compacted samples with a diameter of 150 mm and a height of 140 mm: four SCB specimens were obtained from one compacted samples. Afterward, an artificial notch was created in the middle of the base (Figure 4.6).

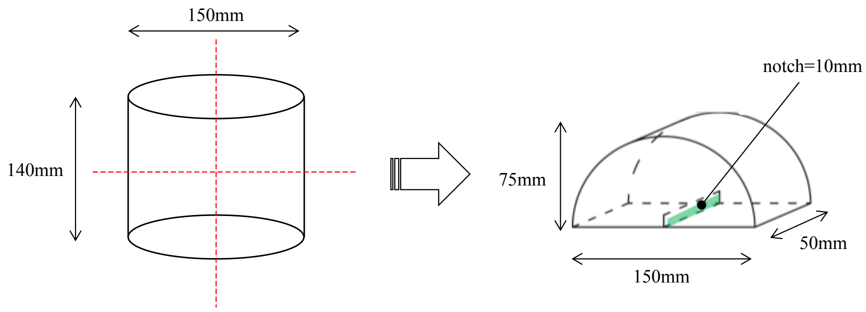


Figure 4.6. SCB specimen preparation.

SCB test consists in applying a three-point bending load (EN 12697-44) to a half cylindrical specimen having a central artificial notch in such way that the middle base of the specimen is subjected to a tensile stress. During the test, the crack starts to propagate from the tip of the artificial notch where the concentration of the stresses is highest; then the crack tends to propagate in the direction of the applied load. Figure 4.7 shows the SCB test configuration.

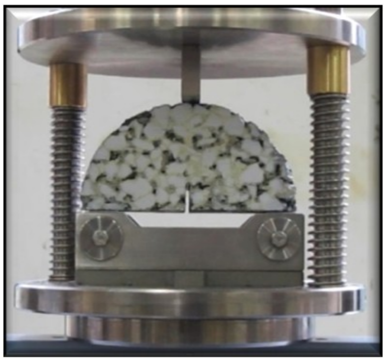


Figure 4.7. SCB test configuration.

In order to consider the test acceptable, the fracture must be comprised within a pre-defined area, not larger than ±15 mm from the central vertical axis of the half cylindrical specimen (Figure 4.8). Such limit is defined in order to have lower dispersion of the test data.

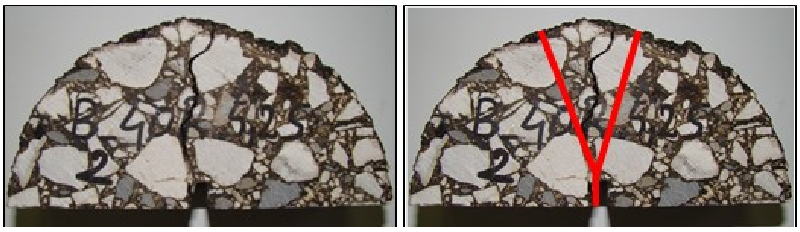


Figure 4.8 Fracture propagation

The test was conducted at 10°C at a constant vertical deformation rate equal to 5 mm/min and the load and the vertical deformation are recorded in continuous.

According to EN 12697-44, results are expressed in terms of fracture toughness and strain at the maximum force.
The results of SCB test allow the calculation of the maximum stress at failure σ_{max} (N/mm^2), as defined in Equation (4.5):

$$\sigma_{max} = \frac{4.263 \times F_{max}}{D \times t} \tag{4.5}$$

where F_{max} is the maximum force (N), D is the diameter (mm) and t is the thickness (mm) of the specimen.

The fracture toughness K ($N/mm^{1.5}$), that represents the capability of mixtures to resist to fracture propagation, can be then calculated using Equation (4.6):

$$K = \sigma_{max} \times f\left(\frac{a}{W}\right) \tag{4.6}$$

where a is the notch depth (mm), W is the specimen height (mm) and $f\left(\frac{a}{W}\right)$ is a geometric factor height (mm) and f(a/W) is a geometric factor equal to 5.956 for a = 9 ÷ 11 mm and W = 70÷75 mm, or otherwise calculated as specified in Equation (4.7) (EN 1697-44):

$$f\left(\frac{a}{W}\right) = -4.9965 - 155.58\left(\frac{a}{W}\right) - 799.94\left(\frac{a}{W}\right)^2 - 2141.9\left(\frac{a}{W}\right)^3 - 2709.4\left(\frac{a}{W}\right)^4 + 1398.5\left(\frac{a}{W}\right)^5 \tag{4.7}$$

The strain at the maximum force ε_{max}, that represents the deformation that the material can accommodate before failure, is determined as defined in Equation (4.8):

$$\varepsilon_{\max} = \frac{\Delta W}{W} \times 100 \tag{4.8}$$

where ΔW is the vertical displacement at the maximum force (mm).

In addition, the total fracture energy G was also determined as a measure of the work required to increase the fractured surface until complete failure (Mobasher et al., 1997; Li and Marasteanu, 2004; Li and Marasteanu, 2010; Biligiri et al., 2012). The fracture energy is calculated as the whole area under the load-displacement curve normalized with respect to the area of ligament that is the surface area where the fracture can propagate before complete failure (Figure 4.9). G (kJ/m^2) is determined as specified in Equation (4.9):

$$G = \frac{\int F ds}{t \times (W - a)} \tag{4.9}$$

where $\int F ds$ is the area under the load-displacement curve (kJ) and $t \times (W - a)$ is the ligament area (m^2).

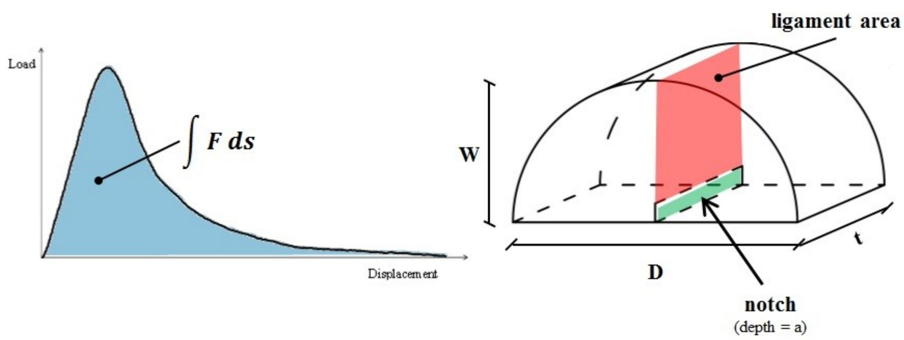


Figure 4.9. Fracture Energy.

As already mentioned, SCB tests were performed in both dry and wet conditions; the water susceptibility was taken into account by means of two parameters, K_{ratio} and G_{ratio}, related to the fracture toughness and the fracture energy and calculated by means of Equations (4.10) and (4.11):

$$K_{ratio} = \frac{K_{wet}}{K_{dry}} \times 100 \tag{4.10}$$

$$G_{ratio} = \frac{G_{wet}}{G_{dry}} \times 100 \tag{4.11}$$

Repeated indirect tensile test

The estimation of the fracture initiation resistance was evaluated by means of repeated indirect tensile tests, performed in accordance to the British standard BS DD ABF. The tests were carried out by means of a Nottingham Asphalt Tester (NAT – Figure 4.10) in indirect tensile configuration (Figure 4.11). The Nottingham Asphalt Tester (NAT) is one of the most efficient and advanced equipment that allows the measuring and assessment of the mechanical properties of asphalt mixtures. The device, developed at the University of Nottingham, is used to performed several different tests in a number of different configurations (e.g. elastic stiffness modulus using repeated load, indirect tension or cyclic compression, instantaneous and total resilient modulus, resistance to fatigue cracking, resistance to permanent deformation with the uniaxial creep test or the repeated load axial test, dynamic creep with both axial and radial strain measurement).
The apparatus consists of a stainless steel load frame fitted with a pneumatic actuator capable of applying vertical loads in the range 0 to 4.3 kN and various load transducers. The device is associated to a temperature controlled chamber capable of controlling the temperature in a range of -10°C to 60°C.

Figure 4.10. Nottingham Asphalt Tester device.

Figure 4.11. NAT indirect tensile test frame.

Preliminarily to the repeated indirect tensile tests, the stiffness modulus was measured for all specimens. The Indirect Tensile Stiffness Modulus (ITSM) was determined in accordance with EN 12697-26 on cylindrical specimens by means of the NAT equipment. Controlled strain tests were carried out for all the investigated mixtures at 20°C. The specimen is placed between two stainless steel loading strips. The face in contact with the specimen is concave and shall extend over the full width of the specimen. The edges of the loading strips shall be rounded to avoid cutting the specimen during testing. A means of centralizing the lower platen with the loading axis of the steel load frame is also provided. The upper platen makes contact with the loading system via a spherical seating. The load actuator applies the stress along the vertical diameter of the specimen via the loading platens. The load has a haversine waveform (Figure 4.12).
The rise-time, measured from when the load pulse commences and which is the time taken for the applied load to increase from zero to maximum value is set up equal to (124 ± 4) ms. The peak load value shall be adjusted to achieve a target peak transient horizontal deformation of 0,005 % of the specimen diameter.

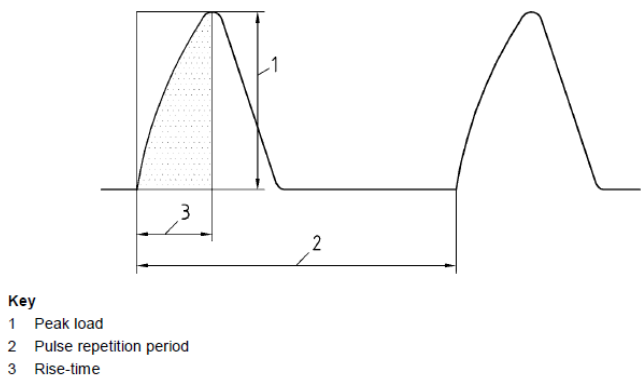


Figure 4.12 Form of load pulse, showing the rise-time and the peak (EN 12697-26)

Two Linear Variable Differential Transformers (LVDTS) are mounted opposite one another in a rigid frame clamped to the specimen. During the test, the frame is only supported by the clamps and it is not in contact with any other part of the equipment They are capable of monitoring the transient horizontal diametral deformation of the specimen during the application of the load pulse. Ten conditioning pulses are applied in order to enable the equipment to adjust the load magnitude and duration to give the specified horizontal diametral deformation and time. After the measurement along a first diameter, the specimen is removed from the test equipment, rotated through (90 ± 10)° about its horizontal axis and replaced in the specimen subframe. The test and calculation are repeated. If the mean value of the stiffness modulus from this test is within +10 % or –20 % of the mean value recorded for the first test, the mean for the two tests is calculated and recorded as the stiffness modulus of the specimen. If the difference between the two values is greater than that specified above, the results are rejected and a third measurement is performed. The measured stiffness modulus is determined for each load pulse using Equation (4.12):

$$S_m = \frac{F \times (\nu + 0.27)}{z \times h} \tag{4.12}$$

where S_m is the measured stiffness modulus, expressed in megapascals (MPa), F is the peak value of the applied vertical load, expressed in Newtons (N), z is the amplitude of the horizontal deformation obtained during the load cycle, expressed in millimeters (mm), h is the mean thickness of the specimen, expressed in millimeters (mm); υ is the Poisson's ratio. In this study, the Poisson’s ratio was assumed equal to 0,35 for all mixtures. The measured stiffness modulus is then adjusted to a load area factor of 0,60.

As far as the resistance to crack initiation of asphalt mixtures is concerned, tests were performed according to the British technical standard BS DD ABF with a rise time of 124 ms at a temperature of 20°C by means of the NAT equipment. An indirect tensile tension of 300 kPa was applied and failure was established when complete fracture of specimens was obtained.
The maximum tensile stress at the center of the specimen (equal to 300 kPa) is evaluated using Equation (4.13):

$$\sigma = \frac{2 \times P}{\pi \times d \times t} \tag{4.13}$$

where P is the vertically applied line loading (kN), d is the specimen diameter (m) and t is the specimen thickness (m).
The same specimens tested to assess the ITSM were used to perform this test. The results were useful to evaluate the crack aptitude of the mixtures and in combination with the results of the SCB test and to investigate the fatigue characteristics of the materials.
The specimens are conditioned at the test temperature for a minimum of 8 hours prior testing. After the conditioning, the specimen is centered in the test subframe.
The flat faces of the specimen must be perpendicular to the lower loading strip. The upper loading strip is then placed on the specimen to fix it and to apply the load through the test apparatus.
In this study, repeated indirect tensile tests were carried out under three different conditions: dry, wet condition (specimens kept in a water bath at 40°C for 72 h and tested in dry condition at 20°C) and submerged (specimens maintained in a water bath at 40°C for 72 h and tested at 20°C completely submerged in a water bath-Figure 4.13). In fact, asphalt pavements in the field experience water damage only under repeated traffic loading and when they are saturated. Hence, in order to simulate closely real field condition, the specimens were immersed in water throughout test: in this way the saturated sample is repeatedly loaded and water can inhaled into and exhaled from the sample at each load application (Kim and Coree, 2005; Poulikakos and Partl, 2009).

Figure 4.13 Repeated indirect tensile test: submerged conditions.

For a reliable evaluation of the performance reduction due to water damage, the specimens of each mixture had been accurately divided into three classes characterized by homogeneous stiffness to be tested in the three different conditions.
For each condition, the number of pulses at failure was taken into account. Moreover, in order to quantify the loss in performance due to the moisture damage experienced by the different mixtures, the percentage pulse decrease (PPD) was calculated for the wet and for the submerged condition compare to the dry condition as defined respectively in Equations (4.14) and (4.15):

$$PPD_{wet} = \frac{N_{dry} - N_{wet}}{N_{dry}} \times 100 \quad (4.14)$$

$$PPD_{submergedt} = \frac{N_{dry} - N_{submerged}}{N_{dry}} \times 100 \quad (4.15)$$

where N_{dry} is the pulse number at failure in dry conditioning and dry test, N_{wet} is the pulse number at failure in wet conditioning and dry test and $N_{submerged}$ is the pulse number at failure in wet conditioning and submerged test.

4.2.5 Results and analysis

Volumetric characteristics

Volumetric properties play a major role in case of PA mixtures in terms of drainability as well as durability. Figure 4.14 shows the mean air voids content of the studied mixtures and demonstrates that the presence of RAP aggregates do not affect the volumetric properties of PA. In fact, all mixtures satisfied acceptance requirements prescribed by Italian technical specification for PA mixtures (Autostrade, 2013) in motorway pavements ($V \geq 18\%$) in case of specimens compacted by means of gyratory compactor with 130 revolutions.

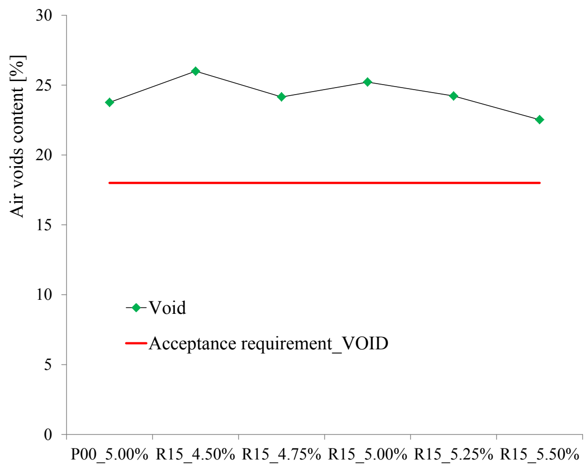
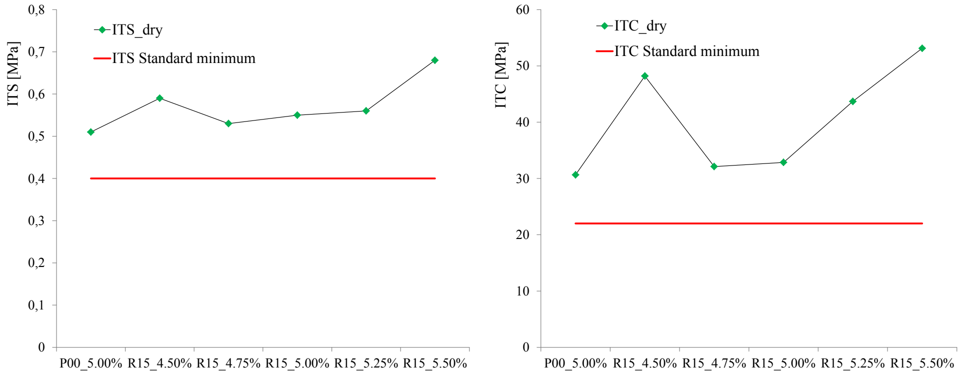


Figure 4.14 Air voids content of recycled and virgin PA mixtures.

Indirect Tensile characteristics

The Indirect Tensile tests were carried out with twofold goals; results in dry conditions allow the assessment of standard recommendations for porous asphalt mixtures whereas the comparison between dry and wet results allows the evaluation of water susceptibility and the possible effects of RAP.
As far as results in dry conditions are concerned, ITS and ITC mean values are shown in Figure 4.15 for all studied mixtures along with the minimum value recommended, according to Italian technical specifications for motorways (Autostrade, 2013). Results show that all PA mixtures analyzed satisfied acceptance requirements prescribed in dry conditions by Italian technical specification for PA mixtures in motorway pavements ($ITS \geq 0.40$MPa and $ITC \geq 22$ MPa); therefore, the presence of 15% RAP does not

compromise the acceptability of PA mixture for motorway application despite the bitumen content.



(a) (b)

Figure 4.15 Indirect Tensile test results in dry conditions in terms of ITS (a) and ITC (b).

Moreover, overall indirect tensile test results in both dry and wet conditions are summarized in Figure 4.16 in terms of mean Indirect Tensile Strength (ITS) and Indirect Tensile Strength ratio (ITSR) that evaluates the water susceptibility of mixtures.

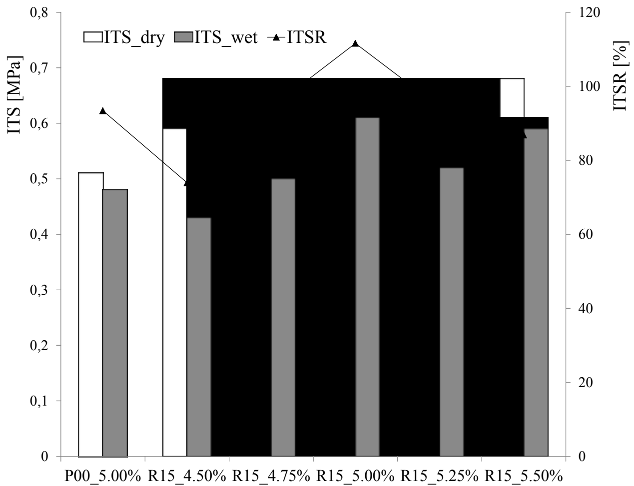


Figure 4.16 Indirect Tensile Strength mean results in dry and wet conditions.

Results in dry conditions showed that all recycled porous asphalt mixtures provide similar or even improved performance than the reference PA. Such an outcome confirms previous research studies on both porous (Goh and You, 2012), dense-graded (Celauro et al., 2010) and rubberized mixtures (Xiao et al., 2007; Xiao and Amirkhanian, 2009) containing RAP. ITS results obtained after the wet conditioning demonstrates that recycled mixtures with bitumen content higher than 4.50% can perform better than the reference PA mixture also in wet conditions. In this sense, ITSR results showed that the water effect is more influenced by the bitumen content than by the presence of RAP. In fact, recycled PA showed a less water sensitivity than the virgin one if a total bitumen content between 4.75% and 5.25% is achieved.

It is worth noting that at the same bitumen content (5.00%), the recycled mixture demonstrated less water sensitivity and higher ITS both in dry and wet conditions than the reference one. These findings could be attributed to the presence of RAP aggregates that can easier adhere to the added virgin bitumen confirming the positive results obtained with the BBS test.

Finally, indirect tensile test results in both dry and wet conditions are summarized in Figure 4.17 in terms of mean Indirect Tensile Coefficient (ITC) and Indirect Tensile Strength ratio (ITCR). Results showed that all recycled porous asphalt mixtures provide improved performance than the reference PA in both dry and wet conditions. As far as moisture susceptibility is concerned, also ITCR results showed that the water effect is more influenced by the bitumen content than by the presence of RAP. In this sense, recycled PA with total bitumen content equal or higher than 5.25% are characterized by similar water sensitivity than the virgin mixture.

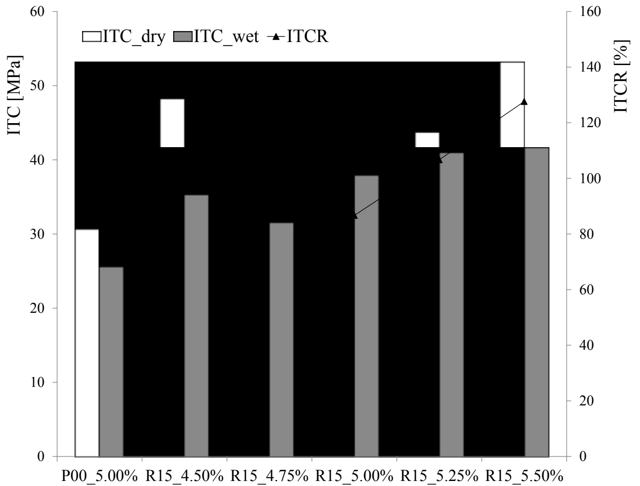


Figure 4.17 Indirect Tensile Coefficient mean results in dry and wet conditions.

Ravelling resistance

As already mentioned, raveling is one of the major distresses that can affect PA mixtures durability; therefore, the influence of RAP on raveling resistance as well as water susceptibility is taken into account by means of Cantabro tests that were carried out in dry and wet conditions.
Cantabro mean results are shown in Figure 4.18 in terms of particle loss in both dry and wet conditions, along with the corresponding mean air void content and the error bars reporting the minimum and the maximum value obtained for each testing condition. In dry conditions all the recycled mixtures show a lower particles loss than the reference PA mixture, meaning that the internal cohesion is even improved when coated RAP aggregates are used. In wet conditions, all the recycled mixtures show similar performance (or even better for high bitumen content) compared to the reference PA mixture, with the exception of R15_5.00%, that exhibits very high particle loss (>70%). However, this behavior can be attributed to the high void content (26.5%) indicating that high porosity can easily lead to poor internal cohesion and thus to high particles loss.

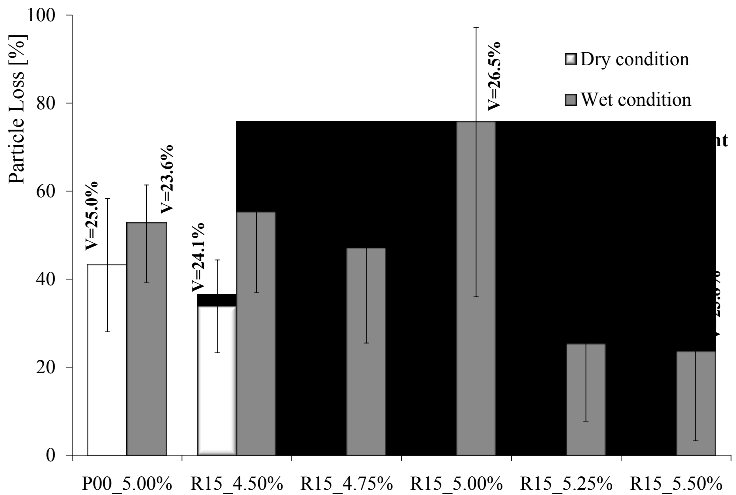


Figure 4.18 Cantabro mean test results in dry and wet conditions.

In order to assess the relationship between particle loss values and the volumetric property of the corresponding specimens, Figure 4.19 relates the Cantabro test result of each specimen with its corresponding air void content for wet conditioned specimens. The graph demonstrates a strong dependency between particle loss and air void content. Furthermore, it is possible to observe that, even in wet conditions, recycled mixtures can provide higher internal cohesion (low particle loss values) than the reference PA mixture, at a given high void content.

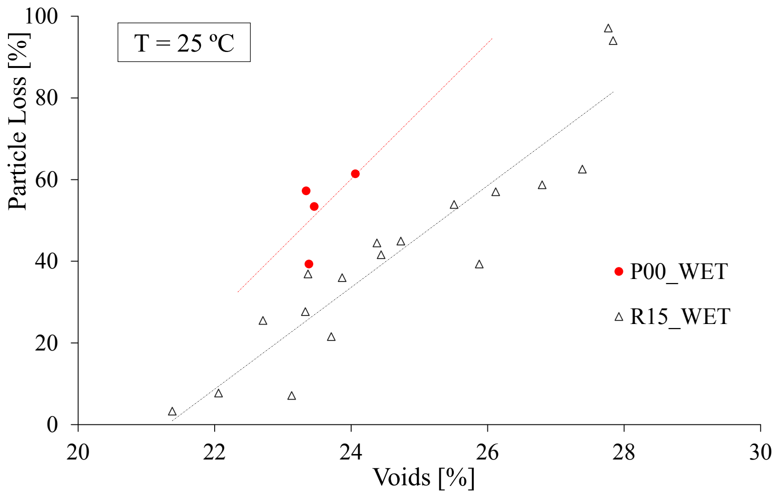


Figure 4.19 Cantabro particle loss vs. air void content.

From the above mentioned considerations, it appears clear that in order to better analyze the ravelling resistance of the studied mixtures, it is necessary to compare specimens at the same air void content. Thus, Cantabro tests were performed again on specimens having the same weight of the Marshall samples but compacted with the gyratory compactor at a target void content (22%); only the reference PA mixture and the R15_5.25% mixture were subjected to this further testing. Mean test results (Table 4.10) showed that the recycled mixture demonstrates improved behavior in both dry and wet conditions, with a lower particle loss increase due to wet conditioning (10.9%) with respect to the virgin mixture (20.2%).

Table 4.10 Cantabro particles loss for mixtures with similar void content

Mixture	Condition	Air void content	Particle Loss	Particle Loss Increase
		[%]	[%]	[%]
P00_5.00%	dry	22,0	17,5	**20,2**
	wet	21,4	21,0	
R15_5.25%	dry	21,6	14,7	**10,9**
	wet	21,7	16,3	

Fracture properties

Traffic load on pavement surfaces produces tensile stress at the bottom of the layer that leads to fatigue cracks. Once cracks have been formed, they start to spread and became

visible; the crack growth proceeds until there is the possibility of crack propagation through the stressed body. Thus, the capability of mixtures to resist cracking propagation represents an important property to ensure durability of the pavement layer. Semi circular bending (SCB) tests had been performed at 10°C on the studied mixtures to analyze crack propagation potential in relation to the presence of recycled material in the PA. As already described in detail in paragraph 4.2.4, fracture propagation characteristics are taken into account by means of three parameters from SCB tests: fracture toughness (K), strain at the maximum force (ε_{max}) and fracture energy (G).
Mean values of the fracture toughness K for all the mixtures in both dry and wet conditions are shown in Figure 4.20 along with error bars reporting the minimum and the maximum value obtained for each testing condition. The values of the parameter K_{ratio} are also reported in the graph as an indication of water susceptibility of mixtures.

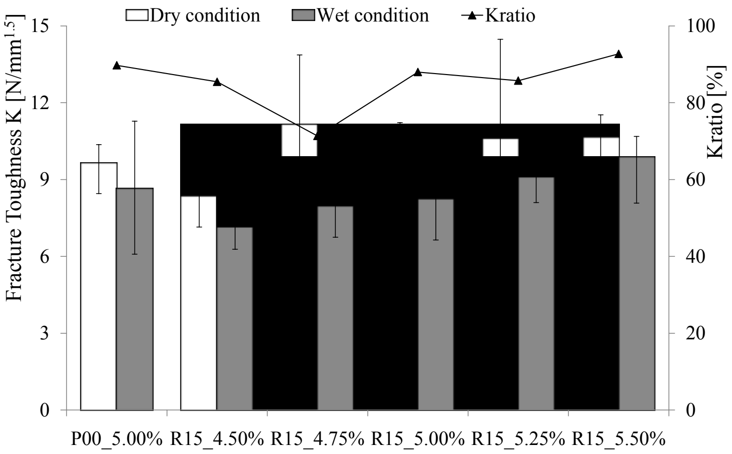


Figure 4.20 SCB mean test results in terms of fracture toughness.

Results showed that the capability of PA mixtures to resist crack propagation is not negatively influenced by the presence of recycled aggregates. In fact, the comparison between P00_5.00% and R15_5.00% (having the same total bitumen content but a different amount of virgin bitumen added) shows that the fracture toughness of the recycled mixture is slightly smaller than the reference one (3% in dry condition and 5% in wet condition). At higher bitumen content (R15_5.25% and R15_5.50%), recycled mixtures perform better than the reference one even if, also in these cases, the amount of the added virgin bitumen is smaller than the amount needed in P00_5.00% due to the presence of the aged bitumen in the RAP aggregates. Such an outcome seems to demonstrate that at least a part of the aged bitumen coming from RAP is still working similarly to virgin bitumen and not as "black aggregate". Results in terms of water susceptibility (K_{ratio}) shows that the capability of

recycled PA mixture to resist to the water effect is comparable to the reference mixture if a total bitumen content equal or higher than 5.00% is selected.
Results in terms of strain at the maximum force are reported in Figure 4.21 in both dry and wet conditions. Low ε_{max} values indicate a material with a brittle behavior, since it cannot accommodate deformations before failure. Results prove that the strain tends to increase with bitumen content, meaning that higher bitumen content provides higher ductility, according to a recent study (Biligiri et al., 2012). Comparing the reference PA mixture with the recycled ones, it is possible to affirm that, except for mix R15_4.50%, recycled PA mixtures show higher ductility than the reference mixture.

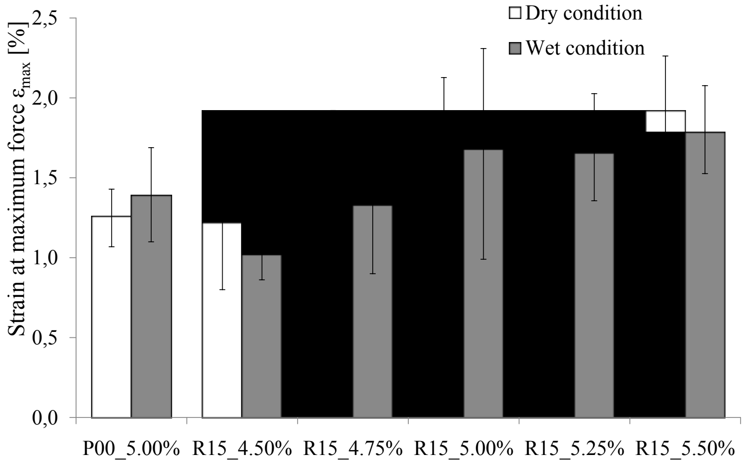


Figure 4.21 SCB mean test results in terms of strain at the maximum force.

Finally, the fracture energy parameter is showed in Figure 4.22 for all the studied mixtures in both dry and wet conditions. It can be observed that the highest fracture energy in dry conditions is provided by the mixture with maximum bitumen content, as already observed by Biligiri et al. (2012). In particular, for the recycled PA the fracture energy tends to increase with the bitumen content, with the exception of R15_5.00%. Moreover, it is possible to affirm that recycled mixtures with 5.25% and 5.50% of total bitumen content show a better behavior in terms of dissipated energy comparing to the reference PA mixture, both in dry and wet conditions. As far as water susceptibility is concerned, results in terms of G_{ratio} confirms previous considerations that only PA mixtures with low bitumen content (R15_4.50% and R15_4.75%) showed lower water resistance that the reference PA mixture.

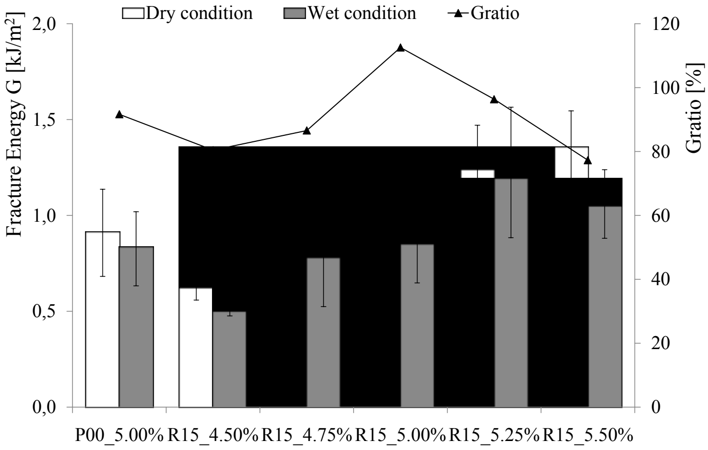


Figure 4.22 SCB mean test results in terms of fracture energy.

<u>Repeated indirect tensile test</u>

Repeated indirect tensile tests were carried out in dry, wet as well as submerged conditions in order to evaluate the water resistance of PA mixtures. Mean results in terms of number of pulse at failure are shown in Figure 4.23 for all the mixtures and testing conditions (i.e. dry conditioning and dry test, wet conditioning and dry test, wet conditioning and submerged test).

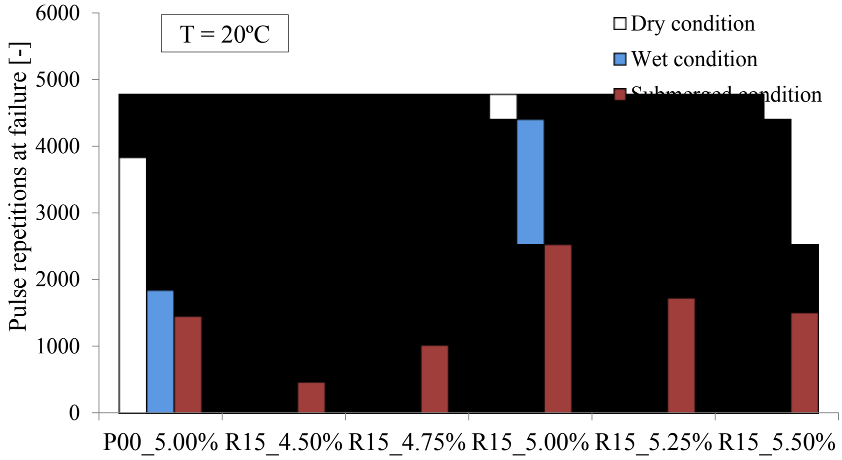


Figure 4.23 Repeated indirect tensile test results.

Results show that the presence of water is detrimental for all mixtures behavior as the pre-conditioning in water leads to a decrease in pulse number at failure compared to the samples that were not subjected to any moisture pre-conditioning. Moreover, the samples tested submerged showed an additional decrease in fracture resistance compared to the other test conditions. Moreover, the water damage parameters (PPD_{wet} and $PPD_{submerged}$) are reported in Figure 4.24 and demonstrate that all the recycled mixtures can perform better than the reference PA mixture (lower decrease of performance) in wet conditions. In submerged condition, the recycled mixtures with low bitumen contents (R15_4.50% and R15_4.75%) show water sensitivity values slightly higher than the reference PA mixture whereas for bitumen content equal or higher than 5.00%, the recycled mixtures perform better than the reference PA mixture as far as water damage is concerned.

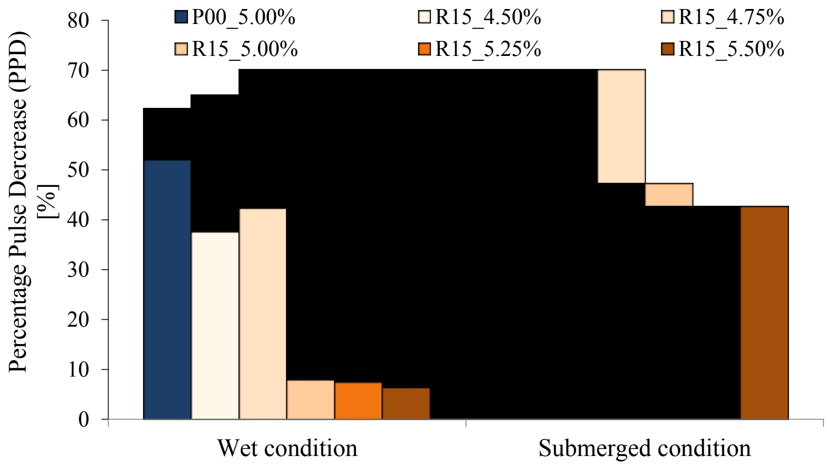


Figure 4.24 Repeated indirect tensile test: water sensitivity.

4.2.6 Conclusions

The present laboratory study evaluates the effect of using coarse reclaimed asphalt (RAP) obtained by milling old porous surface layers, on the performance of porous asphalt (PA) mixtures. A preliminary study through Bitumen Bond Strength (BBS) tests showed that the adhesion strength at the aggregate-bitumen interface improves in both dry and wet conditions using coated aggregates simulating RAP aggregates. Moreover, the loss in performance due to the effect of water experienced by the virgin aggregate is more than double than the loss experienced by the coated aggregate.

On the basis of such encouraging results, during this research study one reference mixture and five recycled mixtures prepared with different bitumen contents were investigated in both dry and wet conditions.

Results from the indirect tensile test showed that in terms of water sensitivity, all the recycled mixtures can perform as well as (or even better than) the reference one, except for the mixture containing low (inadequate) bitumen content (4.50%).

Cantabro tests in dry conditions showed that all the recycled mixtures provided enhanced behavior than the reference one, meaning that the internal cohesion is even improved with the presence of coated RAP aggregates. Moreover, the results of Cantabro test on iso-voids specimens revealed higher performance for the recycled mixtures also in terms of water sensitivity.

The recycled mixtures proved equal or even higher performance than the reference PA mixture in both dry and wet conditions, also when subjected to semi circular bending tests. The strain at the maximum force, that gives an idea of the ductile properties of the mixture, increases with the bitumen content of the recycled mixtures whereas, at the same bitumen content (5.00%), the recycled mixture demonstrates higher strain value than the reference one. In terms of dissipated energy, recycled mixtures with high bitumen content (5.25% and 5.50%) showed improved behavior compared to the virgin mixture. In terms of water sensitivity, only the mixtures with low bitumen content (4.50% and 4.75%) showed lower water resistance with respect to the reference PA mixture.

As far as repeated indirect tensile tests in wet conditions concern, all the recycled mixtures can perform better than the reference PA mixture, whereas in submerged conditions, only recycled mixtures with bitumen contents equal or higher than 5.00% showed higher performance than the reference PA mixture.

In conclusion, the overall experimental results suggest that including 15% of course recycled aggregates in porous asphalt mixtures do not compromise the durability of the PA layers. In particular, it was demonstrated that with total bitumen content from 5.00% to 5.50%, it is even possible to improve the properties of the mixture. These findings could be attributed to the presence of RAP aggregates that can easier adhere to the virgin bitumen as verified through the BBS test during the preliminary phase of the research study.

It is worth noting that, even at 5.50% of bitumen content, the recycled mixture requires less virgin bitumen supply to perform as well as (or even better than) the reference PA mixture. This outcome seems to prove that at least a part of the aged bitumen coming from RAP is still working similarly to virgin bitumen and not as "black aggregate".

Based on the promising results emerged from the present research, advanced laboratory researches as well as field applications of the studied materials are being carried out in order to further evaluate the influence of RAP in PA mixtures performance and durability. In particular, recycled mixtures with 5.00% and 5.25% of total bitumen content are selected for further studies.

4.3. Advanced laboratory characterization: CAST test

4.3.1 Objectives

This study arose from a previous laboratory study (described in detail in paragraph 4.2) concerning the durability and water resistance of recycled PA mixtures (Frigio et al. 2013) that suggested that 15% selected coarse recycled aggregates in PA mixtures would not compromise the durability of PA layers if an adequate total bitumen content (≥ 5.00% by weight) is used. This finding was attributed to the presence of still coated RAP aggregates that can easier adhere to the virgin bitumen. On the basis of such results, laboratory experiments were further extended by cyclic tests on laboratory prepared and compacted mixtures using the CoAxial Shear Test (CAST) apparatus (Partl et al. 2010). CAST tests were performed under both dry and submerged condition for evaluating the behavior under the simultaneous action of traffic and water exposure.
This study was carried out in collaboration to EMPA (Swiss Federal Laboratories for Materials Science and Technology) under the supervisor of Professor Manfred Partl. In particular, the specimens had been prepared at the laboratory of Università Politecnica delle Marche following the same protocol optimized during the first part of the experimentations whereas CAST tests had been performed at EMPA.

4.3.2 Materials

One reference mixture and two recycled mixtures prepared with the same amount of RAP but different bitumen contents were investigated in this research study. The reference mixture, hereafter named P00_5.00%, was a typical PA mixture whereas the recycled mixtures, hereafter named RPA_bitumen content%, were prepared with 15% of RAP aggregates from milled PA dosed. Only the 8/16 mm fraction of RAP aggregates was selected for the recycled PA mixtures. Based on the previous research study (Frigio et al. 2013), the recycled PA mixtures were prepared with two total bitumen contents (5.00% and 5.25% by weight of the aggregates). The material characteristics are the same already described in the paragraph 4.2.

4.3.3 Experimental program

The reference mixture and the recycled mixtures were mixed and compacted with a gyratory compactor under the same conditions (target air void content = 20%, mixing temperature = 170°C and compaction temperature = 160°C). From each mixture, four specimens of 150 mm diameter and 140 mm height were prepared. Volumetric and compactability properties have been taken into account in order to evaluate the possible influence of RAP aggregates on the mixture capability to compact correctly. Mixtures were further investigated using the CoAxial Shear Test (CAST) apparatus in both dry and submerged conditions with the aim at analyzing the effect of RAP aggregates in the fatigue resistance as well as moisture susceptibility of PA mixtures. The overall experimental program is summarized in Table 4.11.

Table 4.11 CAST experimental program

Mixture	***CAST test repetitions***	
	Dry	Submerged
P00_5.00%	2	3
R15_5.00%	2	3
R15_5.25%	2	3

4.3.4 Equipment, test methods and data analysis

Compactability properties

The compactability properties have been taken into account by means of the Compaction Energy Index (CEI). CEI was defined by Mahmoud and Bahia (2004) as the area under the gyratory compaction curve representing the work applied by the roller for compacting the mixture to the required density just before traffic opening (Figure 4.25). In this sense, eight gyrations are generally selected to simulate the effort applied by the paver whereas, in the case of porous asphalt mixtures, according to Goh and You (2012), CEI can be calculated from the whole compaction curve until a density of about 80% of the maximum specific gravity (G_{mm}) is reached. Asphalt mixtures with lower values of CEI are desired since they have better compaction properties (Mahmoud and Bahia 2004; Goh and You 2012). In this study, CEI values were calculated as the average of four repetitions for both standard and recycled PA mixtures.

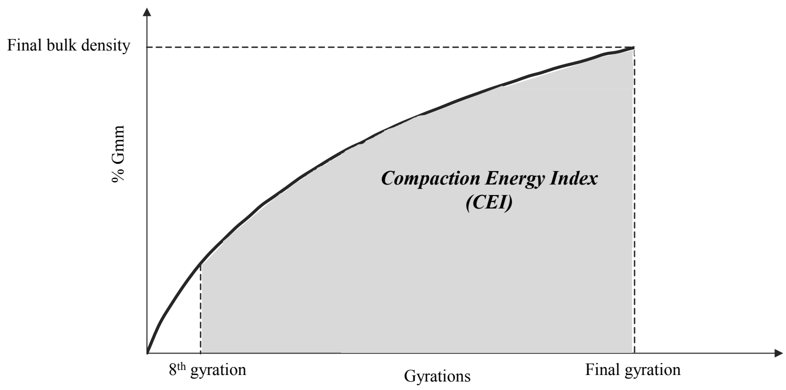


Figure 4.25 Compaction Energy Index.

CAST test

The CoAxial Shear Test (CAST) apparatus has been designed at EMPA (Swiss Federal Laboratories for Materials Science and Technology) and continuously developed and improved since 1987 (Gubler et al. 2005; Sokolov et al. 2005; Poulikakos et al. 2007; Partl et al. 2008; Virgili et al. 2008; Poulikakos and Partl 2009; Kim et al. 2011). It allows testing of donut shaped specimens under simultaneous action of cyclic mechanical loading, temperature changes and water exposure. Tests are performed with lateral deformation constraint in a conventional, temperature controlled, servo-hydraulic tension-compression machine.
A sinusoidal load (stress or strain) in axial direction perpendicular to the specimen's circular surface is applied and the displacement is measured on the upper surface of the steel core by means of a Linear Variable Differential Transducer (LVDT). From the amplitude values of the sinusoidal load and displacement, the complex Young's modulus E* of the material is calculated based on a nonlinear finite element model (Sokolov et al. 2005) as defined in Eq. (4.16):

$$E^{*} = \frac{F_a}{\delta_a} * A\left(E^{*}\right) = \frac{F_a}{\delta_a} * \left(A_1 + A_2 E^{*A_3}\right) \quad (4.16)$$

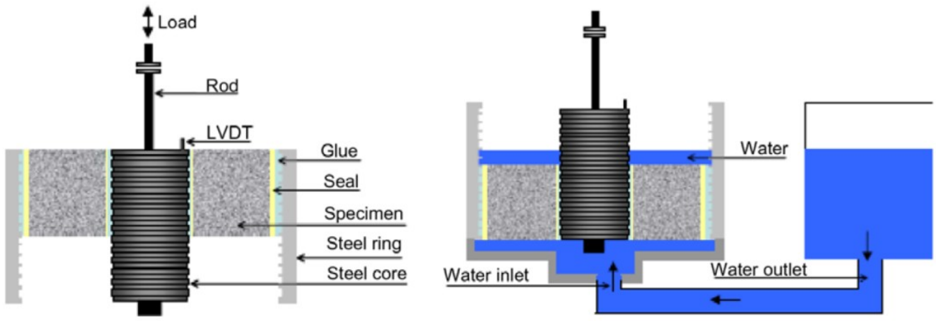
where F_a is the amplitude of the force, δ_a is the amplitude of the displacement of the central core and $A(E^*)$ is a function depending on the complex modulus itself with the geometry coefficients A_1, A_2 and A_3 (Sokolov et al. 2005).
In this study, 50 mm thick specimens were centrally cored to obtain a donut shape (outer diameter of 150 mm and inner diameter of 55 mm). Dry and water submerged tests were

carried out in controlled strain mode at a constant temperature of 27°C; all test parameters are listed in Table 4.12.

Table 4.12 CAST experimental parameters

Test parameter	*Value*
Tempering period	14400 s
Frequency	10 Hz
Deformation amplitude	0.01 mm
Test temperature	27°C
Test duration	144000 s

The CAST setup for testing specimens both in water and in air is shown schematically in Figure. 4.26. For each mixture, five CAST repetitions were carried out (two in dry and three in submerged conditions) as listed in Table 3. Prior to testing, all specimens were pre-conditioned for four hours at 27 °C to ensure initial thermal equilibrium.
Further details about CAST apparatus and testing procedure are given elsewhere (Sokolov et al. 2005; Partl et al. 2008; Poulikakos and Partl 2009; Partl et al. 2010; Kim et al. 2011).



(a) (b)

Figure 4.26 CAST schematic setup for dry (a) and water submerged (b) test.

4.3.5 Results and analysis

Compactability properties

Compactability of standard and recycled laboratory PA mixtures was investigated by means of CEI values. CEI mean results are reported in Figure 4.27 for all the studied mixtures along with the error bars showing the standard deviation values obtained for each set of data. CEI values are in accordance with those of typical dense graded mixes as found by

Mahmoud and Bahia (2004) for selected dense graded mixtures (CEI values between 50 and 1000). Such a significant variation in densification behaviour was achieved by varying aggregate source and mix design.

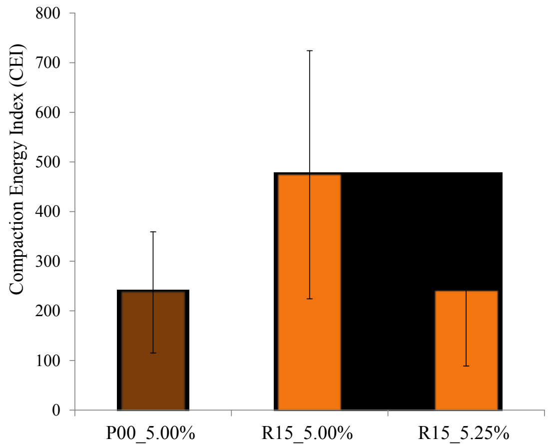


Figure 4.27 CEI mean results.

Experimental data show a significant increase in the CEI value for the recycled PA mixture with 5.00% of bitumen content compared to the standard PA mixture. This finding could be due to the fact that 12% of the total bitumen (0.60% out of 5.00%) consists of aged (stiffer) bitumen from RAP aggregates and a prominent part acts as “black aggregate”. This seems to be confirmed by the fact that the recycled mixture with a higher amount of virgin bitumen (R15_5.25%) showed a CEI value closely comparable to the reference mixture.
As far as repeatability of the test results is concerned, recycled PA mixes with both bitumen contents showed a lower repeatability than the reference mixture. This finding can be explained by probably different quantities of RAP aggregates in the recycled specimens. In fact, for practical reasons and in order to simulate full scale production conditions, materials were mixed in the laboratory in such a way that several specimens were compacted from the same mixed material. This means that, adding the right percentage of RAP aggregates into the mixer, did not guarantee that all specimens contained the same amount of RAP aggregates. In the case of recycled mixtures, this variability is added to the inevitable variability related to the bitumen content and granulometric distribution.
Moreover, it is worth nothing that the recycled mixture with higher bitumen content (R15_5.25%) showed better repeatability than the recycled mixture with lower bitumen content (R15_5.00%). This finding can be explained by the higher supply of virgin bitumen within R15_5.25% mixtures which is able to mitigate the effect of different amount of RAP aggregates among specimens with the same nominal composition.

CAST tests

The influence of water on repeated loading resistance of recycled PA mixtures was investigated performing CAST under dry and submerged condition. The complex modulus E* was calculated according to Equation (4.16) and its decrease with the increasing number of load cycles was used for evaluating the material damage accumulation. Experimental mean results for each mixture and testing condition are given in Figure 4.28.

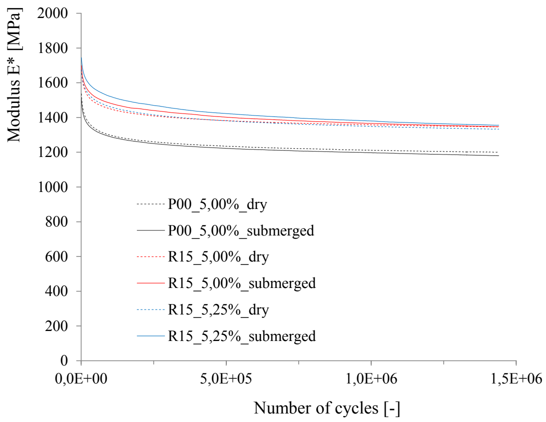
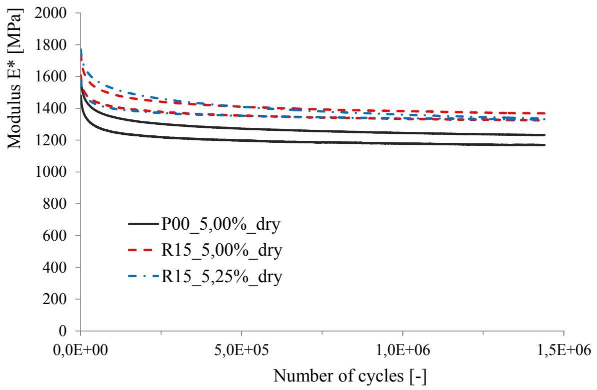
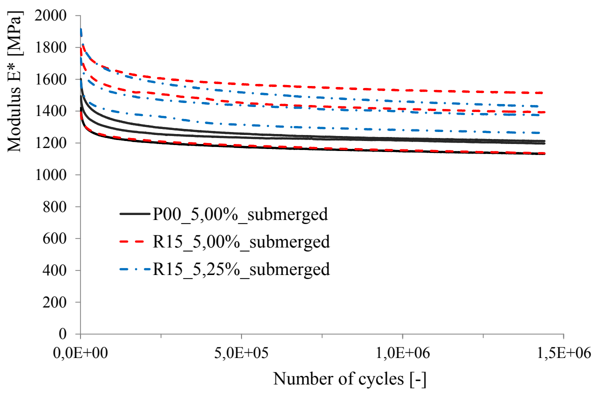


Figure 4.28 CAST mean test results.

It can be noted that the loss in performance due to water exposure in terms of complex modulus decrease is negligible for all mixtures, irrespective of the presence of RAP aggregates. Moreover, recycled PA mixtures at both bitumen contents showed higher complex modulus values than the reference PA mixture under both dry and submerged condition. Thus, it can be stated that CAST results confirmed the findings emerged from the previous laboratory study (Frigio et al. 2013) which demonstrated that repeated loading and water resistance of recycled PA mixtures with 15% coarse RAP aggregates can be equal to (or even better than) the reference PA material.
As far as scattering of the modulus results concerns, Figure 4.29 shows all experimental data obtained in both dry and water submerged conditions.



(a)



(b)

Figure 4.29 CAST experimental test results in dry (a) and submerged (b) conditions

Higher modulus scattering was achieved in case of recycled mixtures with respect to the reference material if submerged testing conditions were taken into account. As mentioned for the CEI values, this fact can be explained considering that recycled specimens were likely characterized by quite different amount of RAP aggregates and, as a consequence, by high variability in bitumen content and granulometric distribution. In this sense, the improvement in adhesion properties due to the presence of coated RAP aggregates principally affect results in submerged conditions. In fact, the presence of coated RAP aggregates is able to improve adhesion properties of the mixtures in the presence of water since they can easier adhere to the virgin bitumen that is the same in both recycled and virgin mixtures (Frigio et al. 2013). Thus, the observed higher scattering of CAST test results in wet conditions due to variability of RAP content seems to be in agreement with the expected behavior of recycled mixtures.

4.3.6 Conclusions

The main conclusions regarding the present study are listed below:
- recycled mixtures with adequate bitumen content (5.25%) demonstrated almost the same compactability than the reference mixture in terms of Compaction Energy Index;
- the effect of water exposure during CAST tests appeared negligible, irrespective of the presence of RAP aggregates;
- recycled mixtures showed higher complex modulus values than the reference mixture in both dry and submerged conditions.

4.4. Field investigations

4.4.1 Objectives

The present experimental study on recycled PA was further investigated by means of in plant production involving recycled PA mixtures and the construction of full scale trial section along an in-service motorway. The same PA mixtures that were investigated in the laboratory experimentation were
Such experimentation involved the same PA mixtures that were already evaluated during the laboratory investigation and consist in a laboratory evaluation of the PA mixtures produced at the asphalt plant during the construction of a real scale trial (hereafter named “field mixtures”) in order to perform a comparison between laboratory produced and in plant produced mixtures and to verify the feasibility of large scale production of recycled PA. Moreover, the construction of the trial section also allowed the field evaluation of the drainage properties of the studied materials. It is worth noting that field mixtures were prepared in the asphalt plant according to the mix design optimized during the previous laboratory study (Frigio et al. 2013) and adopted for preparing the laboratory mixtures (described in detail in paragraph 4.2). In order to evaluate the durability and water susceptibility of the field mixtures, a comprehensive experimental program similar to the previous study (Frigio et al. 2013) was carried out.

4.4.2 Materials

A reference PA mixture (P00field _5.00%) and two recycled PA mixtures with 15% of recycled aggregates with different bitumen contents (R15field _5.00% and R15field _5.25%) were investigated. As already done for the advanced laboratory investigations

(paragraph 4.3), only two total bitumen contents were selected for further in-plant evaluations, based on the previous research study (see paragraph 4.2).
The materials were taken from the field during the construction of a full scale trial section realized as a part of an in-service motorway rehabilitation project (Italian motorway A1). The three mixtures were prepared at the asphalt plant according to the mix design developed in the laboratory (see paragraph 4.2).
Asphalt mixtures from the field were analyzed in the laboratory for determining the actual total bitumen content and aggregate grading curves (Table 4.13). As it can be seen, the materials prepared in the asphalt plant strictly adhered to the composition requirements developed in the laboratory.

Table 4.13 Field mixtures characteristics: bitumen content and aggregate grading curve

Sieve size [mm]	P00field_5.00%		R15field_5.00%		R15field_5.25%	
	Passing [%]		Passing [%]		Passing [%]	
	Design	**Actual**	Design	**Actual**	Design	**Actual**
20	100,0	**100,0**	100,0	**100,0**	100,0	**100,0**
14	91,5	**87,6**	92,4	**90,9**	92,4	**90,1**
10	41,3	**44,9**	46,3	**45,4**	46,3	**50,8**
6,3	23,9	**19,1**	24,0	**20,0**	24,0	**23,9**
2	15,0	**12,7**	13,6	**12,2**	13,6	**14,1**
0,5	9,1	**8,2**	8,9	**8,0**	8,9	**9,1**
0,25	7,5	**7,1**	7,5	**7,0**	7,5	**7,6**
0,063	5,3	**4,3**	5,4	**4,2**	5,4	**4,5**
Bitumen [%]	5,00	**4,93**	5,00	**4,91**	5,25	**5,16**

4.4.3 Experimental program

Similarly to laboratory mixtures, materials taken from the asphalt plant were compacted in the laboratory using the gyratory compactor. In order to simulate water damage, specimens of each mixture were conditioned in air (dry) or in water (wet) at 40°C for a period of 72 h, as suggested by EN 12697-12 (Method A). Indirect tensile strength (ITS) tests, particle loss (Cantabro) tests and semi-circular bending (SCB) tests were carried out in both dry and wet conditions, according to the experimental program of the previous phase of the research (paragraph 4.2). Repeated indirect tensile tests were also performed but only on specimens fully immersed in water that is the most severe condition, as demonstrated in the previous phase (paragraph 4.2).
Finally, pavement drainability was measured in the field in order to evaluate if recycled PA surfaces are comparable to the reference PA surface.
The overall experimental program is summarized in Table 4.14.

Table 4.14 Field mixtures experimental program

Mixture	ITS test		Cantabro test		SCB test		Repeated indirect tensile test	In situ drainability test
	Dry	Wet	Dry	Wet	Dry	Wet	Submerged	
P00field_5.00%	4	4	4	4	4	4	3	6
R15field_5.00%	4	4	4	4	4	4	3	6
R15field_5.25%	4	4	4	4	4	4	3	6

4.4.4 Equipment, test methods and data analysis

Indirect tensile strength (ITS) tests, particle loss (Cantabro) tests, semi-circular bending (SCB) tests as well as repeated indirect tensile tests are already described in detail in paragraph 4.2.4.
On the other hand, the measurement of PA surface drainability is described in the present paragraph. Surface drainability was measured in the field using the outflow time test method according to the Italian technical specifications (Autostrade SpA 2013). The test consists in placing a cylindrical container of known volume, called permeameter, above the pavement before sealing the outer part of the pavement (out of the tested area) and the bottom of the permeameter in order to avoid any lateral leaking of water. Then, the permeameter is filled with water and the time of a fixed decrease of water volume (3.85 litres) is recorded. This time is governed by the water seepage through an annular area of the pavement layer under standardized hydraulic head conditions. In this study, a permeameter with an internal diameter of 140 mm and a maximum capacity of 6 liters water has been used (Figure 4.30). The drainage capability of the tested surface was expressed as the ratio between the known volume of water and the corresponding outflow time (l/min).

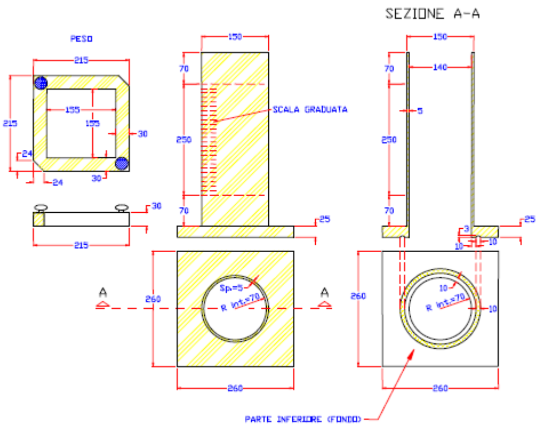


Figure 4.30 Model of the permeameter used for field surface drainability measurements.

4.4.5 Results and analysis

Experimental results coming from the study of the PA mixtures taken from the asphalt plant during the construction of a full scale trial section are presented in this section. Moreover, such results were also compared with those obtained in the previous phase of the research on the corresponding laboratory mixtures.

Indirect tensile tests

Indirect tensile test mean results are shown in Figure 4.31 in terms of Indirect Tensile Strength in both dry and wet conditions for all the field mixtures. The error bars representing the minimum and maximum values are also reported in the figure for each testing condition.
Results showed that all recycled field mixtures satisfied acceptance requirements prescribed in dry conditions by Italian technical specification (Autostrade SpA 2013) for PA mixtures in motorway pavements (ITS ≥ 0.40MPa). Moreover, recycled porous asphalt mixtures provided higher performances than the reference mixture confirming previous studies (Goh and You 2012) as well as previous observation based on the laboratory experimentation (paragraph 4.2). As far as moisture susceptibility is concerned, results showed that recycled PA mixtures can perform as well as the reference material even after a wet conditioning demonstrating similar water sensitivity in terms of ITSR.

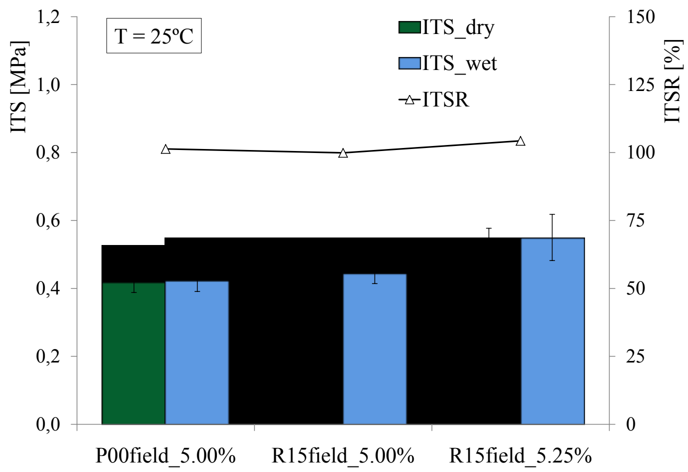


Figure 4.31 ITS mean results: field mixtures

In order to compare performance of laboratory and in-plan produced (field) mixtures, Figure 4.32 shows all results obtained from the reference mixtures as well as the recycled mixtures with total bitumen content equal to 5.00% and 5.25%. The error bars representing the minimum and maximum values are also reported in the figure for each testing condition. The comparison shows a slightly decrease in ITS in case of field mixtures with respect to laboratory mixtures, in both dry and wet conditions. Such a difference can be attributed to the different procedures used in laboratory and during the in-plant production that can lead to different aging degree in mixtures. Such a decrease in PA mixtures performance does not compromise the acceptability of the mixture and involves both the reference and the recycled mixtures; hence it cannot be attributed to the presence of RAP aggregates. As far as water susceptibility is concerned, results are fully comparable between laboratory and field mixtures.

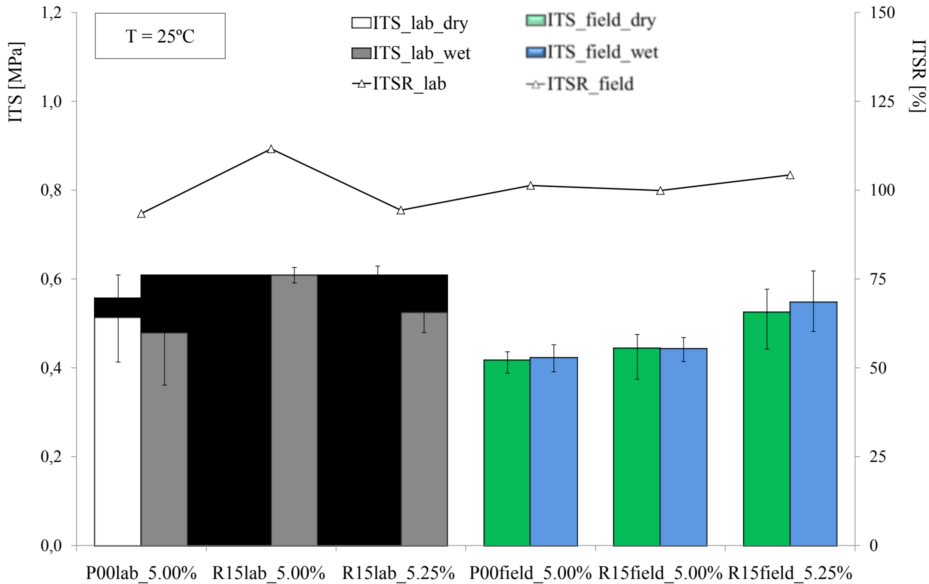


Figure 4.32 ITS mean results: comparison between laboratory and field mixtures

Cantabro tests

Mean Cantabro test results for both dry and wet conditions are shown in Figure 4.33 in terms of particle loss percentage along with the corresponding mean air void content (the error bars indicate the minimum and the maximum value for each testing condition).

Results in dry conditions show that the recycled PA mixture with 5.00% of total bitumen content is characterized by higher particle loss value than the reference PA mixture whereas the recycled mixture with higher bitumen content (R15_5.25%) demonstrates improved raveling performance. Such an outcome can be related to the higher bitumen content that improve the adhesion properties at the aggregate/bitumen interface but it can also be related to the lower air voids content with respect to the other mixtures.

In any case, particle loss exhibited by reference and recycled field mixtures was very low (< 22%) and consistent with international requirements for high traffic highways corresponds to a maximum of 20% in dry condition (Alvarez et al., 2010; Kline and Putman, 2011). Hence, it can be asserted that the particle bonding of PA mixtures is not compromised due to the presence of RAP aggregates.

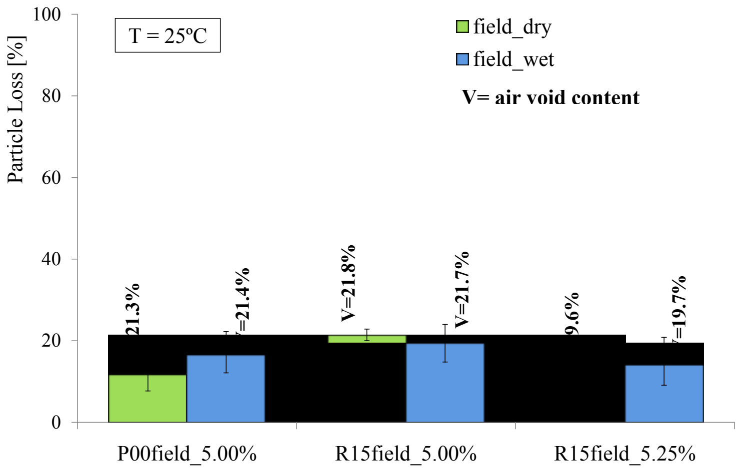


Figure 4.33 Cantabro mean results: field mixtures.

Moreover, as far as water sensitivity concerns, the increase in particle loss due to wet conditioning is not affected by the presence of RAP aggregates in the mixture confirming the positive results obtained in the case of laboratory mixtures (paragraph 4.2.5). Also the results in wet conditions are consistent with international requirements that correspond to a maximum of 35% in wet condition (Alvarez et al., 2010).

In order to compare performance of laboratory and in-plan produced (field) mixtures, Figure 4.34 shows all results obtained from the reference mixtures as well as the recycled mixtures with total bitumen content equal to 5.00% and 5.25%. The error bars representing the minimum and maximum values are also reported in the figure for each testing condition. The comparison demonstrates that the field mixtures can perform significantly better that the corresponding laboratory mixtures, especially in wet conditions, meaning that the in-plant production of recycled PA mixtures is possible without compromise the ravelling performance. Such difference in response between laboratory and field mixtures can be attributed to the lower air voids content of the mixtures produced at the asphalt plant; as a matter of fact, it is known a clear dependency of the particle loss values to the volumetric properties of the specimen, particularly in wet conditions (see paragraph 4.2.5).

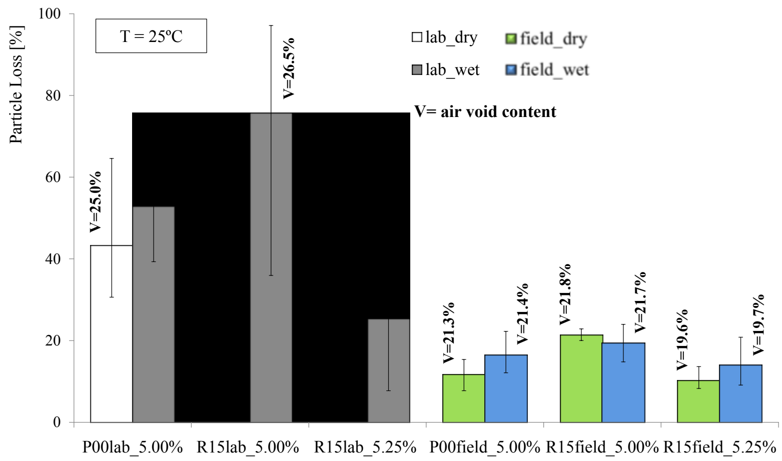


Figure 4.34 Cantabro mean results: comparison between laboratory and field mixtures

Fracture properties

Semi-circular bending (SCB) tests were carried out in both dry and wet conditions in order to evaluate the crack propagation aptitude in relation to the presence of recycled material in the PA.
Results of field mixtures in terms of fracture toughness are given in Figure 4.35 as the average of four repetitions for both dry and wet conditions.
Recycled mixtures can perform similar to the reference PA in dry conditions and even better in wet conditions, meaning that the presence of coated aggregates (RAP) can improve the water susceptibility of PA mixtures. Such an outcome is also confirmed by the parameter Kratio that show a batter resistance of recycled mixtures when compared to the virgin one.
The comparison between laboratory and field mixtures in terms of K values is shown in Figure 4.36. Fracture propagation resistance is fully comparable between the studied mixtures, demonstrating the feasibility of in-plant production of recycled PA. As a matter of fact, better performance in wet conditions are shown by the field recycled mixtures when compared to the corresponding laboratory mixtures.

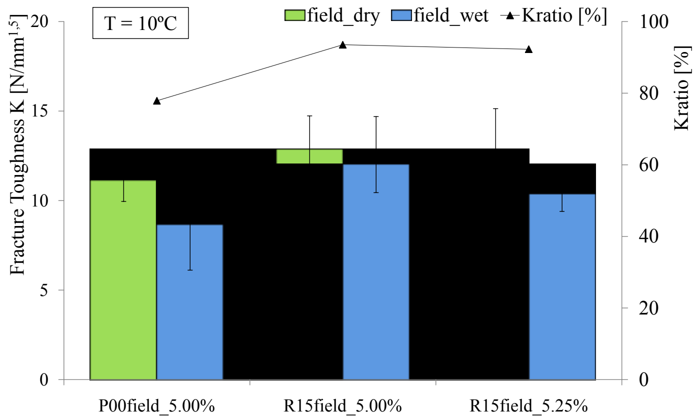


Figure 4.35 SCB mean results in terms of fracture toughness: field mixtures.

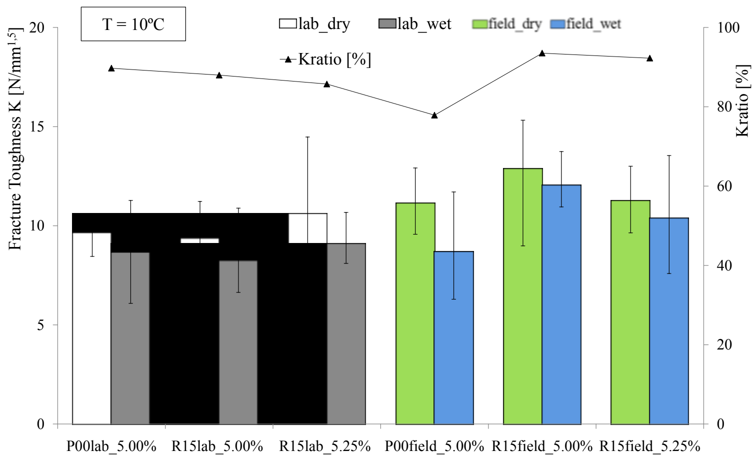


Figure 4.36 SCB mean results in terms of fracture toughness: comparison between laboratory and field mixtures

Results of field mixtures in terms of strain at the maximum force are given in Figure 4.37 as the average of four repetitions for both dry and wet conditions. Recycled mixtures demonstrated to perform similarly to the reference mixture in terms of ability to deform before failure (ductility). In particular, R15_5.25% may exhibit a slight improvement even though such differences do not assume statistical significance due to scattering of the results.

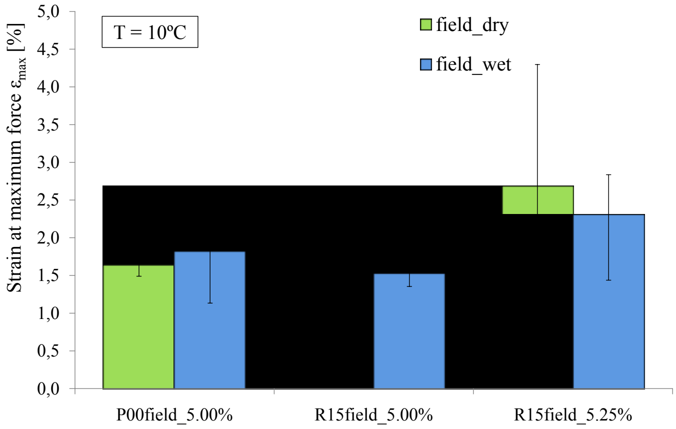


Figure 4.37 SCB mean results in terms of strain at the maximum force: field mixtures.

The comparison between laboratory and field mixtures, showed in Figure 4.38, confirm previous observation that in-plant produced mixtures can perform similarly or even better that the corresponding laboratory one.

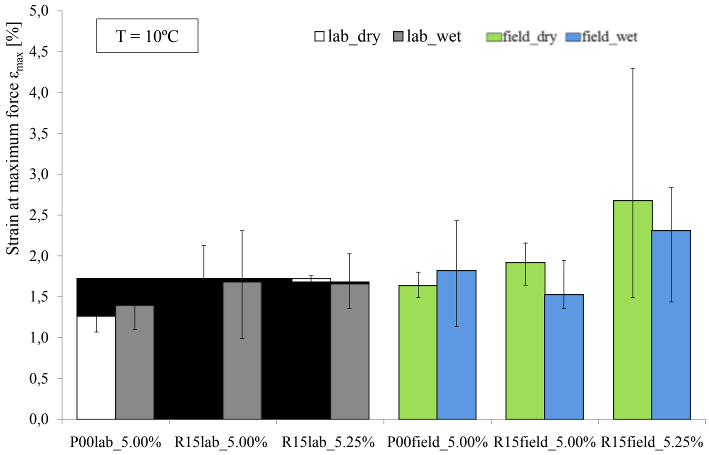


Figure 4.38 SCB mean results in terms of strain at the maximum force: comparison between laboratory and field mixtures

As far as fracture energy is concerned, mean results of field mixtures are given in Figure 4.39 for both dry and wet conditions. Recycled mixtures demonstrated to perform similarly or even better than the reference mixture in terms of ability to dissipate energy before failure. In particular, the fracture energy for the recycled mixtures tends to increase with the bitumen content according to Biligiri et al. (2012).
Also the water susceptibility does not seem to be affected by the presence of RAP aggregates.

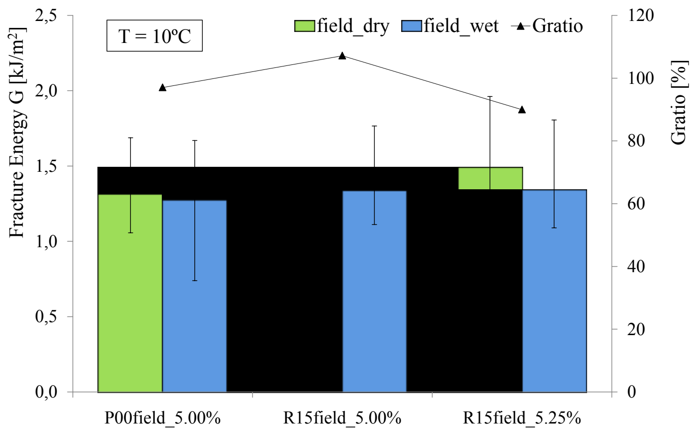


Figure 4.39 SCB mean results in terms of fracture energy: field mixtures.

The comparison between laboratory and field mixtures, showed in Figure 4.40 in terms of fracture energy, confirm previous statements that in-plant produced mixtures can perform even better that the corresponding laboratory one.

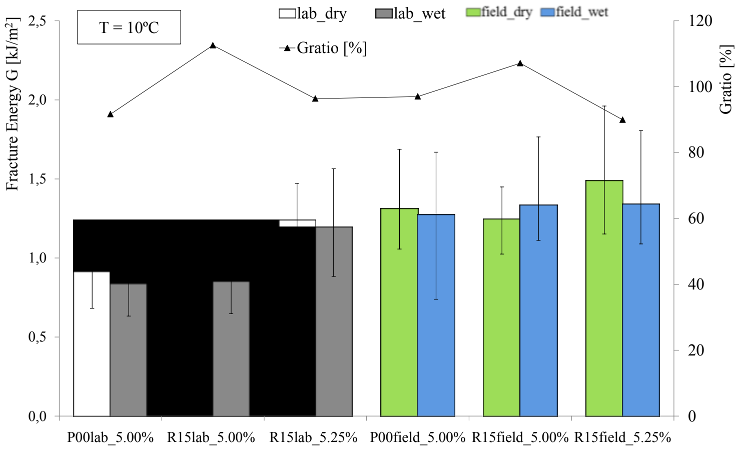


Figure 4.40 SCB mean results in terms of fracture energy: comparison between laboratory and field mixtures

Repeated indirect tensile test

According to Kim and Coree (2005), repeated indirect tensile tests were performed in water submerged conditions to investigate moisture susceptibility of the studied field mixtures. Tests were carried out only in submerged condition that was recognized as the most sever in a previous laboratory study (paragraph 4.2.5).

Experimental results in terms of number of pulse at failure are given in Table 4.15. Results clearly showed that the presence of RAP aggregates does not affect the performances of the mixtures under such testing conditions. Contrarily, recycled mixtures showed a sensibly better behavior than the reference mixture confirming results of the previous laboratory study.

Table 4.15 Mean results of repeated indirect tensile tests for field mixtures

Mixture	Number of pulse at failure [-]
P00field_5.00%	1011
R15field_5.00%	2118
R15field_5.25%	1571

Field drainability test

Drainage properties of the studied PA mixtures were evaluated in the field after the construction of the full-scale trial section. Mean results in terms of drainage capability are given in Table 4.16; each value is the average of six measurements spaced 50 meters apart. It is worth noting that the drainage capabilities of the three pavement sections are fully comparable. Thus, field tests confirmed that the use of selected coarse RAP as partial substitution of basalt coarse aggregates (fraction 8/16 mm) does not compromise pavement drainability.

Table 4.16 Field drainability results

Mixture	Drainage capability	Coefficient of variation
	[l/min]	[%]
P00field_5.00%	29	15,3
R15field_5.00%	31	20,4
R15field_5.25%	30	20,6

4.4.5 Conclusions

Mixtures prepared at the asphalt plant during the construction of a full-scale trial section along an in-service motorway were evaluated in this study in order to verify the performances of “real” mixtures. Such mixtures were prepared according to the laboratory mix design defined in the previous phase of the research and with the same materials.
The following main conclusions can be drawn:
- recycled mixtures are able to satisfy acceptance requirements typically prescribed by technical specification for PA mixtures in terms of indirect tensile strength (ITS);
- recycled porous asphalt mixtures can perform as well as corresponding mixtures without recycling in terms of ITS and water sensitivity (Indirect Tensile Strength Ratio – ITSR);
- Cantabro test results showed that the presence of recycled aggregates does not sensibly affect the resistance to ravelling (bitumen-aggregates bonding properties) and water susceptibility of the PA mixtures;
- semi-circular bending (SCB) tests showed similar crack resistance potential and fracture energy of the recycled and the reference PA mixtures. Moreover recycled mixtures demonstrated higher water resistance than the reference mixture;
- recycled field mixtures showed enhanced resistance to repeated indirect tensile loading in submerged conditions;
- field mixtures exhibited similar or even enhanced performances with respect to the corresponding laboratory materials meaning that the large scale production of recycled PA is possible without compromise mixtures performance and durability;
- recycled mixtures are able to assure the same drainage properties of the reference porous surface.

In conclusion, the overall experimental results confirmed that including 15% of coarse recycled aggregates in porous asphalt mixtures does not compromise performances and durability of the PA layers if adequate bitumen content is selected. As a matter of fact, it was demonstrated that recycled mixture with 5.25% of total bitumen content and prepared following an accurate mix design can perform as well as (or even better than) standard PA.

Chapter 5.

Attempt to increase RAP content in PA mixtures

5.1. Objectives

Based on the promising results of the first experimental research involving the use of 15% of selected RAP in porous asphalt mixtures, the experimental activity was extended to involve higher amount of RAP. As a matter of fact, previous laboratory and field investigations (chapter 4) were able to optimize the mix design (aggregates grading curve as well as total bitumen content) and to evaluate the mechanical performance and the drainability of recycled PA mixtures including 15% of RAP aggregates. It is important to underline that RAP aggregates used for such experimentation come from old PA surface layers and it was sieve so that only the coarse (8/16mm) can be re-used in PA mixtures. In this way, the RAP aggregate grading curve as well as the RAP bitumen content can be considered almost constant so that the presence of recycled materials do not compromised the volumetric and in-situ performance of the PA mixtures.
The present chapter describes the attempt to extend such experimentation involving higher amount of selected RAP. It is well known, in fact, road pavement maintenance and rehabilitation are more frequently performed on porous asphalt surface layers due to their inherent low durability. Such activities lead to the production of considerable amount of reclaimed asphalt mainly from PA layers and to a large use of virgin non- renewable natural resources due to the fact that the use of RAP is not usually allowed into PA. In this sense, the use of higher amount of milled materials from old PA wearing courses in new PA layers promotes an important cycle of re-using that should be encouraged.
This research aims to investigate the performance of recycled PA mixtures with different amount of RAP (20% and 25% by aggregates weight) from old PA surface layers. First of all, it was verified if the performance of PA mixtures can be still guaranteed when higher percentages (20% and 25%) of coarse RAP are added to the mixture. Moreover, different total bitumen contents (5.25%, 5.50% and 5.75% by aggregate weight) were adopted for each mixture in order to optimize the mix design.
For these purposes, six recycled PA asphalt mixtures prepared with two different amount of RAP and three total bitumen contents (new bitumen plus aged bitumen from RAP) were investigated in terms of compactability, durability and water resistance. Furthermore, a reference PA mixture made only with virgin aggregates and prepared with a polymer modified bitumen was also investigated for comparison purposes.
The research project focuses on the evaluation of durability issues as they are the main failure causes in PA. In this sense, an experimental program similar to that of the first

experimentation was carried out, involving Indirect tensile strength tests, particle loss (Cantabro) tests, semi circular bending (SCB) tests in both dry and wet condition in order to evaluate acceptability, durability and water sensitivity of recycled mixtures. Repeated indirect tensile tests were also performed on specimens fully immersed in water for evaluating fracture resistance properties under the most severe condition. Moreover, compactability properties of the reference and the recycled PA mixtures were compared.

5.2. Laboratory mix design and investigation

5.2.1 Materials

One reference PA mixture and six recycled PA mixtures prepared with different amount of RAP and various bitumen contents were investigated in this research study.
The reference mixture, hereafter named P00_5.00%, was a typical porous asphalt mixture prepared with virgin basaltic aggregates and a polymer (SBS) modified bitumen (5% by weight of the aggregates). Such a mixture has the same characteristic of the reference mixture that was prepared in the first experimental activity (paragraph 4.2) and the same components were used.
The recycled mixtures were prepared with two different percentages of RAP, equal to 20% and 25% by total weight of the aggregates (hereafter named R20_bitumen content% and R25_bitumen content%, respectively), and three total bitumen contents (5.25%, 5.50% and 5.75% by the aggregates weight). Only RAP from milled PA surface layers was used and sieved so that only the 8/16 mm fraction was employed in the preparation of recycled PA mixtures. The total bitumen content includes the virgin bitumen and the bitumen within the RAP which was equal to 4.0% by weight of RAP. The three total bitumen contents investigated for the recycled PA mixtures were chosen taking into account the optimum total bitumen content (5.25%) of the previous studied PA mixtures with 15% RAP (paragraph 4.2 and 4.3). In fact, as the amount of RAP in the mixtures increases, a larger amount of total bitumen is composed of aged bitumen within the RAP whose a prominent part acts as “black aggregate”.
Basalt aggregates (8/16 mm – 5/11 mm – 0/4 mm), calcareous filler and RAP aggregates (after bitumen extraction) were sieved in order to find the optimum aggregate mixture grading curve for both the reference mixture and the two recycled mixtures, according to the Italian specifications for motorway pavements (Autostrade, 2013).

Table 5.1 Aggregates mix design.

	Basalt 8/16 mm	RAP 8/16 mm	Basalt 5/11 mm	Basalt 0/4 mm	Filler
	[%]	[%]	[%]	[%]	[%]
Reference mixture	**74**	**0**	**10**	**13**	**3**
Recycled mixtures with 20% of RAP	**58**	**20**	**10**	**9**	**3**
Recycled mixtures with 25% of RAP	**55**	**25**	**10**	**7**	**3**

The aggregate mix design is shown in Table 5.1. As far as the reference mixture is concerned, the aggregates mix design is the same of the fist experimental program and it is the mix design currently used for the production of virgin PA mixtures for surface layers in Italian motorways (Autostrade, 2013). For both the recycled mixtures, it can be observed that coarse RAP mainly substitutes basalt coarse fraction 8/16 mm and partly substitutes basalt sand 0/4 mm whereas basalt 5/11 mm and filler remained unchanged. This fact implies that the recycled mixtures were characterized by almost the same grading curve of the reference mixture (Figure 5.1) without compromising volumetric properties and thus the balance between mixture durability and mixture functionality (Alvarez et al., 2009b).
Moreover, 70% cellulose-30% glass fibres dosed at 0.3% by the aggregates weight were also added to the mixtures, as required by Italian technical specification (Autostrade, 2013).

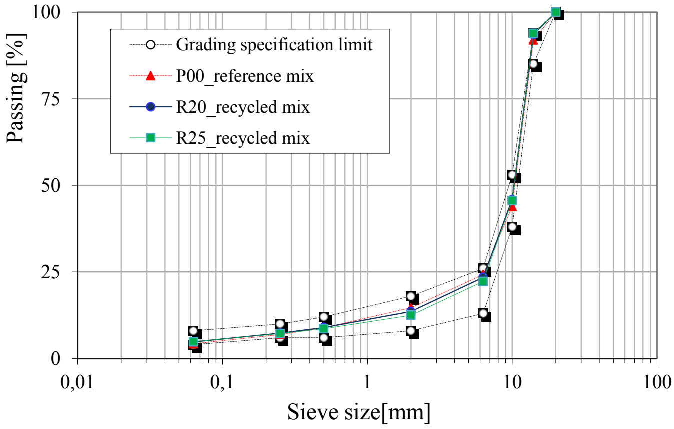


Figure 5.1 Aggregates grading curves.

5.2.2 Experimental investigation

The reference and the recycled PA mixtures were mixed and compacted in the laboratory using the gyratory compactor under the same conditions (target air void content = 22%, mixing temperature = 170 °C and compaction temperature = 160 °C).
In order to evaluate possible effects of RAP aggregates on the compactability properties of PA mixtures, the Compaction Energy Index (CEI) was taken into account. CEI was already described in detail in paragraph 4.3.5. It is important to underline that asphalt mixtures with lower value of CEI are desired since they have better compaction properties. In this study, CEI values were calculated on specimens of 100 mm diameter as the average of eight repetitions and on specimens of 150 mm diameter as the average of four repetitions for both reference and recycled PA mixtures.
The mixtures were also evaluated in terms of mechanical properties and water sensitivity. To this aim, indirect tensile strength (ITS) tests, particle loss (Cantabro) tests and semi

circular bending (SCB) tests were carried out in both dry and wet conditions. In fact, in order to investigate the water susceptibility, specimens of each mixture were conditioned in air (dry) at the test temperature or in water (wet) at 40 °C for a period of 72 h, as suggested by EN 12697-12 [28]. Moreover, repeated indirect tensile tests were also carried out only in submerged conditions after wet conditioning. The overall experimental program is summarized in Table 5.2.

Table 5.2 Experimental program.

Mixture	ITS test		Cantabro test		SCB test		Repeated indirect tensile test
	dry	wet	dry	wet	dry	wet	submerged
P00_5.00%	**4**	**4**	**4**	**4**	**4**	**4**	**3**
R20_5.25%	4	4	4	4	4	4	3
R20_5.50%	4	4	4	4	4	4	3
R20_5.75%	4	4	4	4	4	4	3
R25_5.25%	4	4	4	4	4	4	3
R25_5.50%	4	4	4	4	4	4	3
R25_5.75%	4	4	4	4	4	4	3
TOTAL	**56**		**56**		**56**		**21**

All the tests were already described in detail in paragraph 4.2.4 and the same test parameters were used.

5.2.3 Results and analysis

Compactability properties

Compactability of reference and recycled PA mixtures was investigated by means of CEI values. Mean results obtained on 100 mm and 150 mm diameter specimens are given in Figures 5.2 and 5.3, respectively (along with the corresponding error bars reporting the minimum and the maximum value for each test condition).
Results show an increase in CEI value for the recycled PA mixtures with lower bitumen content compared to the reference PA mixture. This finding can be attributed to the fact that a significant part of the total bitumen is aged bitumen within the RAP whose a prominent part acts as “black aggregate”. This seems to be confirmed by the fact that, for the recycled mixtures, CEI value tends to decrease as the amount of total bitumen content increases. In particular, recycled mixtures with 20% and 25% of RAP demonstrate compactability properties similar to the reference PA mixture if adequate total bitumen contents are adopted.

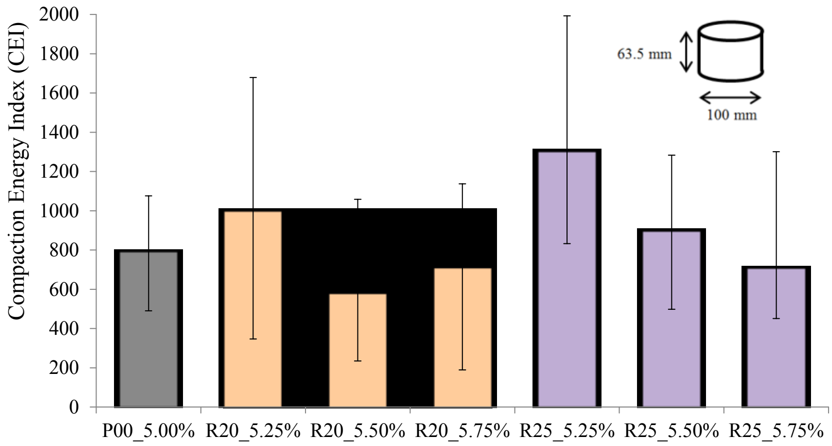


Figure 5.2 CEI mean results for 100mm diameter specimens.

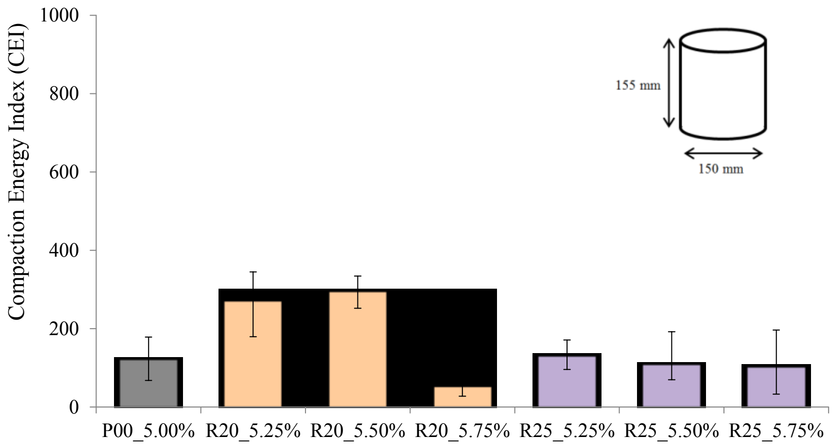


Figure 5.3 CEI mean results for 150mm diameter specimens.

A one-way ANOVA at 95% confidence level has been used to verify the statistical significance of calculated CEI values for the recycled mixtures with respect to the reference mixture. Experimental data (Table 5.3) show that, in general, the presence of RAP is not statistically significant for the compactability of PA mixtures, except for some cases at low total bitumen contents.

Table 5.3 ANOVA: the influence of RAP and total bitumen content on compactability properties

CEI values (D=100mm)			CEI values (D=150mm)		
Mixtures	Significant?	*p* -value	Mixtures	Significant?	*p* -value
P00_5.00% vs R20_5.25%	NO	0,3295	P00_5.00% vs R20_5.25%	YES	0,0232
P00_5.00% vs R20_5.50%	NO	0,1891	P00_5.00% vs R20_5.50%	YES	0,0076
P00_5.00% vs R20_5.75%	NO	0,6319	P00_5.00% vs R20_5.75%	NO	0,1175
P00_5.00% vs R25_5.25%	YES	0,0240	P00_5.00% vs R25_5.25%	NO	0,7931
P00_5.00% vs R25_5.50%	NO	0,5036	P00_5.00% vs R25_5.50%	NO	0,7649
P00_5.00% vs R25_5.75%	NO	0,6168	P00_5.00% vs R25_5.75%	NO	0,7414

Indirect Tensile characteristics

The Indirect Tensile tests were carried out with twofold goals; results in dry conditions allow the assessment of standard recommendations for porous asphalt mixtures whereas the comparison between dry and wet results allows the evaluation of water susceptibility and the possible effects of RAP.
As far as results in dry conditions are concerned, ITS and ITC mean values are shown in Figure 5.4 and 5.5 for all studied mixtures along with the minimum value recommended (ITS ≥ 0.40MPa and ITC ≥ 22 MPa), according to Italian technical specifications for motorways (Autostrade, 2013). Results show that all PA mixtures analyzed satisfied acceptance requirements prescribed in dry conditions by Italian technical specification for PA mixtures in motorway pavements; therefore, the presence of 20 and 25% RAP does not compromise the acceptability of PA mixture for motorway application despite the bitumen content.

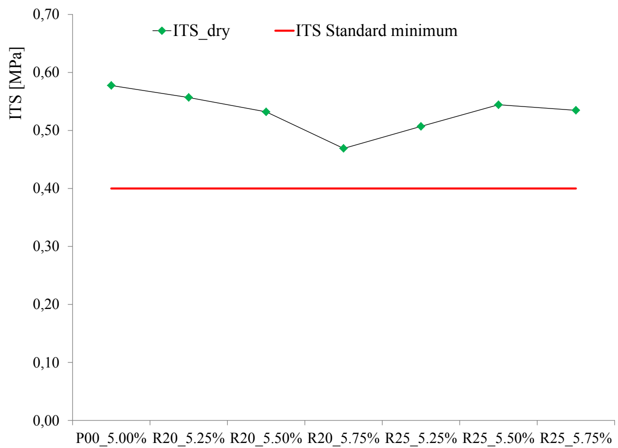


Figure 5.4 Mean ITS results in dry conditions.

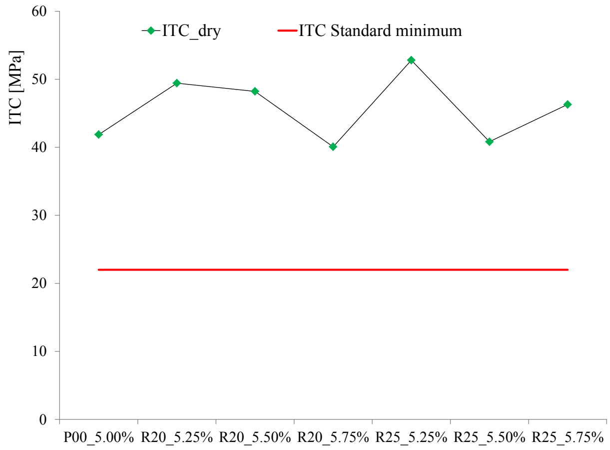


Figure 5.5 Mean ITC results in dry conditions.

Moreover, overall indirect tensile test results in both dry and wet conditions are given in Figure 5.6 in terms of mean Indirect Tensile Strength (ITS) and Indirect Tensile Ratio (ITSR); the error bars represents the minimum and the maximum values obtained for each testing condition.

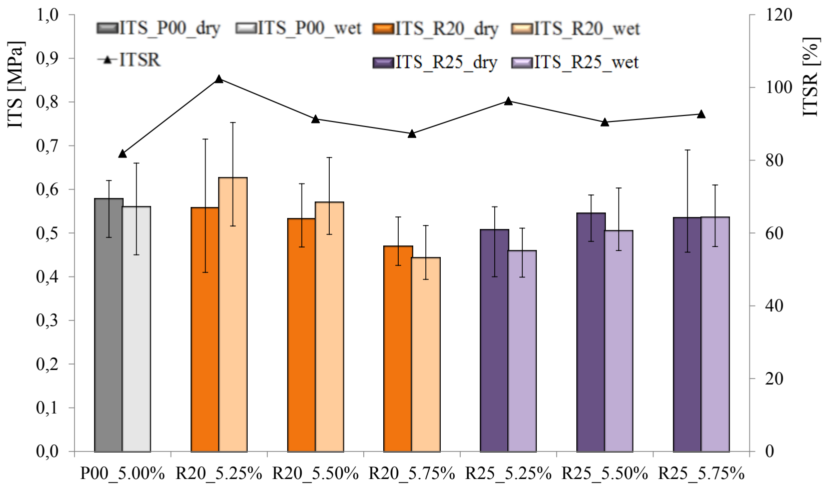


Figure 5.6 ITS results in dry and wet conditions.

ITS results in dry conditions show a slight decrease for recycled PA mixtures with respect to the reference one. ITS tests were also performed on wet conditioned samples, in order to

estimate if the moisture susceptibility is affected by the presence of RAP. In this sense, all recycled mixtures showed lower water sensitivity in terms of ITSR than the reference mixture irrespective of the amount of RAP and the bitumen content.
A one-way ANOVA at 95% confidence level has been used to verify the statistical significance of the results, in terms of ITS values for the recycled mixtures with respect of the reference mixture. The results of ANOVA are summarized in Table 5.4 in terms of test outcome along with the relevant p-value. Experimental data suggested that the presence of RAP in PA mixtures does not provide statistically significant improvement or worsening in terms of ITS in both dry and wet conditions, except for the case of mixture R20_5.75% that showed a slightly decrease in performance with respect to the reference mixture.

Table 5.4 ANOVA: the influence of RAP and total bitumen content on Indirect Tensile test results

	ITS values_dry			ITS values_wet	
Mixtures	Significant?	*p*-value	Mixtures	Significant?	*p*-value
P00_5.00% vs R20_5.25%	NO	0,8101	P00_5.00% vs R20_5.25%	NO	0,4466
P00_5.00% vs R20_5.50%	NO	0,3243	P00_5.00% vs R20_5.50%	NO	0,8855
P00_5.00% vs R20_5.75%	YES	0,0354	P00_5.00% vs R20_5.75%	NO	0,1040
P00_5.00% vs R25_5.25%	NO	0,1922	P00_5.00% vs R25_5.25%	NO	0,1481
P00_5.00% vs R25_5.50%	NO	0,4304	P00_5.00% vs R25_5.50%	NO	0,4234
P00_5.00% vs R25_5.75%	NO	0,5069	P00_5.00% vs R25_5.75%	NO	0,7144

Ravelling resistance

Mean Cantabro test results for both dry and wet conditions are shown in Figure 5.7 in terms of particle loss percentage along with the corresponding mean air void content (the error bars indicate the minimum and the maximum value for each testing condition).

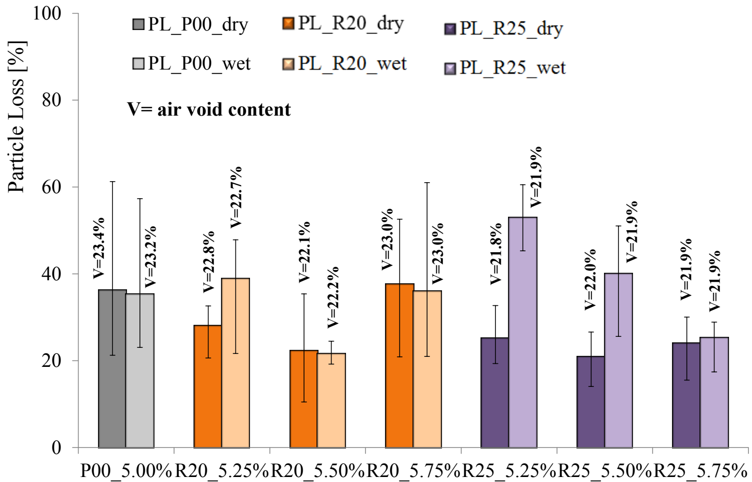


Figure 5.7 Cantabro test results in dry and wet conditions.

All recycled mixtures show lower particles loss than the reference PA mixture in dry conditions, meaning that the aggregate-bitumen bonding is even improved when coated RAP aggregates are used. The mixture R20_5.75% is the only case that shows a slightly worst behavior than the other recycled mixtures even though its performance are still comparable to the reference mixture. A one-way ANOVA at 95% confidence level has been used to verify the statistical significance of the results, in terms of Cantabro values for the recycled mixtures with respect of the reference mixture (Table 5.5). The results of ANOVA in dry conditions show that the influence of 20% and 25% RAP in PA mixtures is not statistically significant in terms of Cantabro test.
On the other hand, only the mixture R25_5.25% demonstrates a statistically significant worsening with respect to the reference mixture in wet conditions.
In particular, at low total bitumen contents the recycled mixtures exhibit higher water damage (particle loss increase) that tends to decrease with the increase of bitumen content. PA mixtures with 20% RAP were able to perform as well as the virgin PA mixture in term of water sensitivity if an adequate total bitumen content ≥ 5.50% is adopted whereas PA mixtures with 25% RAP required a total bitumen content of 5.75%. It is worth noting that, as the RAP content increases, the amount of added virgin bitumen required to achieve comparable performance to the reference mixture needs to be increased as well. That confirms the fact that a prominent part of the aged bitumen within the RAP acts as "black aggregate".

Table 5.5 ANOVA: the influence of RAP and total bitumen content on Cantabro test results

	Cantabro values_dry			Cantabro values_wet	
Mixtures	Significant?	*p*-value	Mixtures	Significant?	*p*-value
P00_5.00% vs R20_5.25%	NO	0,5512	P00_5.00% vs R20_5.25%	NO	0,7194
P00_5.00% vs R20_5.50%	NO	0,3455	P00_5.00% vs R20_5.50%	NO	0,1256
P00_5.00% vs R20_5.75%	NO	0,9365	P00_5.00% vs R20_5.75%	NO	0,9654
P00_5.00% vs R25_5.25%	NO	0,4306	P00_5.00% vs R25_5.25%	YES	0,0353
P00_5.00% vs R25_5.50%	NO	0,2833	P00_5.00% vs R25_5.50%	NO	0,6442
P00_5.00% vs R25_5.75%	NO	0,3867	P00_5.00% vs R25_5.75%	NO	0,2621

Fracture properties

Semi circular bending (SCB) tests were carried out in both dry and wet conditions in order to evaluate the capability of the PA mixtures to resist cracking propagation in relation to the presence of recycled material.
Mean values of the fracture toughness K for all mixtures in both dry and wet conditions are shown in Figure 5.8 along with error bars reporting the minimum and the maximum value

for each testing condition. Results show that the capability of PA mixtures to resist crack propagation is not negatively influenced by the presence of recycled aggregates. In fact, in both dry and wet conditions all the recycled mixtures show fracture toughness values similar or even higher than the reference mixture.

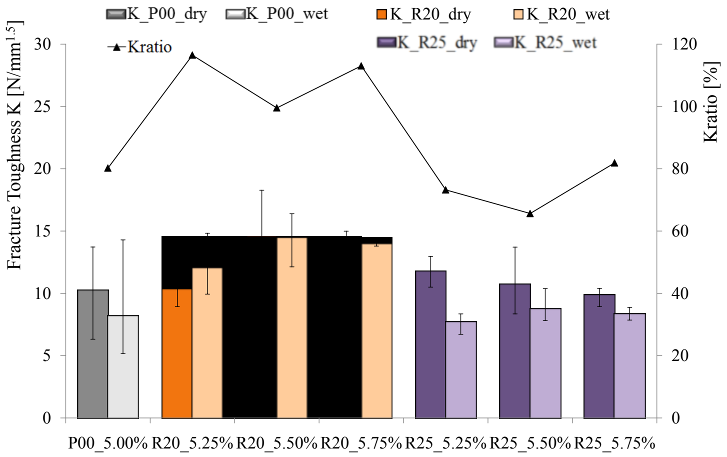


Figure 5.8 SCB test results in dry and wet conditions in terms of fracture toughness.

A one-way ANOVA at 95% confidence level has been used to verify the statistical significance of the results, in terms of fracture toughness values for the recycled mixtures with respect to the reference mixture (Table 5.6). The results of ANOVA confirm that the presence of 20% and 25% RAP does not significantly influence the performance of PA mixtures in dry conditions. Moreover, ANOVA results in wet conditions show a statistically significant improvement in performance for the mixtures with 20% RAP and high bitumen contents (5.50% and 5.75%) with respect to the reference mixture.

Table 5.6 ANOVA: the influence of RAP and total bitumen content on SCB test results in terms of fracture toughness.

	Fracture toughness_dry	
Mixtures	Significant?	*p*-value
P00_5.00% vs R20_5.25%	NO	0,6177
P00_5.00% vs R20_5.50%	NO	0,1110
P00_5.00% vs R20_5.75%	NO	0,3673
P00_5.00% vs R25_5.25%	NO	0,4133
P00_5.00% vs R25_5.50%	NO	0,8151
P00_5.00% vs R25_5.75%	NO	0,8472

	Fracture toughness_wet	
Mixtures	Significant?	*p*-value
P00_5.00% vs R20_5.25%	NO	0,1552
P00_5.00% vs R20_5.50%	YES	0,0342
P00_5.00% vs R20_5.75%	YES	0,0332
P00_5.00% vs R25_5.25%	NO	0,8341
P00_5.00% vs R25_5.50%	NO	0,7994
P00_5.00% vs R25_5.75%	NO	0,9483

Furthermore, mean values of the fracture energy G for all mixtures in both dry and wet conditions are shown in Figure 5.9 along with error bars reporting the minimum and the maximum value for each testing condition. In dry condition, it can be observed a significant decrease of the fracture energy for all recycled mixtures with respect to the reference one. This was confirmed by the ANOVA at 95% confidence level (Table 5.7).

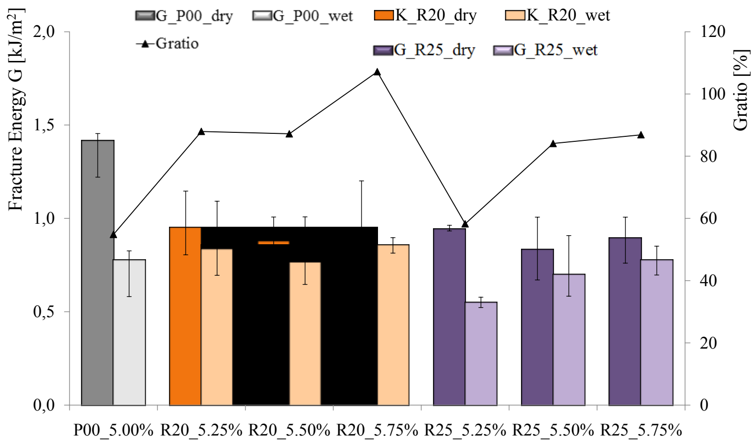


Figure 5.9 SCB test results in dry and wet conditions in terms of fracture energy.

As far as wet conditions concern, all mixtures with 20% RAP were able to perform as well as the reference mixture. On the other hand, in the case of the mixtures with 25% RAP only the mixture with the highest total bitumen content (R25_5.75%) was able to perform similarly to the reference mixture. The results of ANOVA at 95% confidence level (Table 5.7) showed that the mixture R20_5.75% was able to provide a statistical significant improvement in terms of G whereas only the mixture R25_5.25% showed a statistically significant performance decrease with respect to the reference mixture.

Table 5.7 ANOVA: the influence of RAP and total bitumen content on SCB test results in terms of fracture energy.

	Fracture energy_dry			Fracture energy_wet	
Mixtures	Significant?	*p*-value	Mixtures	Significant?	*p*-value
P00_5.00% vs R20_5.25%	YES	0,0019	P00_5.00% vs R20_5.25%	NO	0,5388
P00_5.00% vs R20_5.50%	YES	0,0001	P00_5.00% vs R20_5.50%	NO	0,9005
P00_5.00% vs R20_5.75%	YES	0,0136	P00_5.00% vs R20_5.75%	YES	0,0183
P00_5.00% vs R25_5.25%	YES	1,1E-06	P00_5.00% vs R25_5.25%	YES	2,9E-05
P00_5.00% vs R25_5.50%	YES	0,0003	P00_5.00% vs R25_5.50%	NO	0,3345
P00_5.00% vs R25_5.75%	YES	0,0001	P00_5.00% vs R25_5.75%	NO	0,9863

As far as water susceptibility is concerned, all the recycled mixtures with 20% of RAP are able to perform better that the virgin mixture, both in terms of Kratio and Gratio. On the other hand, the mixtures with 25% of RAP demonstrate to perform as well as the reference mixture in terms of moisture susceptibility if adequate total bitumen contents are used (≥ 5.50%).

Repeated indirect tensile test

The ability of an asphalt pavement containing RAP to resist fracture from repeated loads is essential for the design of hot mix asphalt (HMA) mixtures (Xiao et al., 2011). In this study, repeated indirect tensile tests were performed in submerged conditions in order to investigate moisture susceptibility of the mixtures similarly to what proposed by Kim and Coree (2005). As already mentioned, the specimens were immersed in water throughout the test in such way that the saturated sample is repeatedly loaded and water can inhale into and exhale from the sample at each load application. The submerged condition is capable to simulate closely the real field condition and it was found to be the most severe condition for repeated indirect tensile tests in a previous study.
Tests results are given in Table 5.8 in terms of number of pulse at failure; the error bars representative of the minimum and the maximum values are also reported. Results show that recycled PA mixtures with 20% RAP are able to perform as well as the reference mixture irrespective of the total bitumen content. On the other hand, PA mixtures with 25% of RAP demonstrate a significant decrease in performance with respect to the reference PA mixture at higher total bitumen contents.

Table 5.8 Mean results in terms of pulse at failure of repeated indirect tensile tests.

	Submerged conditions	
Mixture	Pulse No.	Voids
	[-]	[%]
P00_5.00%	2500	20,02
R20_5.25%	2584	17,55
R20_5.50%	2636	18,36
R20_5.75%	2276	15,60
R25_5.25%	2638	20,24
R25_5.50%	964	19,27
R25_5.75%	879	19,20

A one-way ANOVA at 95% confidence level has been used to verify the statistical significance of the results, in terms of number of pulse at failure for the recycled mixtures with respect of the reference mixture. The results of ANOVA (Table 5.9) showed that the presence of RAP is not statistically significant due to the high scattering of the data.

Table 5.9 ANOVA: the influence of RAP and total bitumen content on fracture resistance in terms of pulse at failure.

	Number of pulse at failure	
Mixtures	Significant?	*p*-value
P00_5.00% vs R20_5.25%	NO	0,9589
P00_5.00% vs R20_5.50%	NO	0,9393
P00_5.00% vs R20_5.75%	NO	0,9185
P00_5.00% vs R25_5.25%	NO	0,9112
P00_5.00% vs R25_5.50%	NO	0,2977
P00_5.00% vs R25_5.75%	NO	0,1674

5.2.4 Conclusions

The present experimental program evaluates the effect of using different amount of coarse reclaimed asphalt (RAP) from old porous surface layers on the performance of new porous asphalt (PA) mixtures. For this purpose, a reference PA mixture and six recycled PA mixtures with two different amount of RAP (20% and 25%) and three total bitumen contents (5.25%, 5.50% and 5.75%) were investigated in terms of compactability, durability and water resistance.
The following main conclusions can be drawn:
- recycled PA mixtures at low total bitumen contents show worst compactability properties than the reference PA mixture due to the fact that a significant part of the total bitumen content is aged bitumen within the RAP and a prominent part acts as “black aggregate”. In fact, at higher total bitumen content the recycled mixtures demonstrate compactability properties similar to the reference PA mixture;
- recycled mixtures are able to satisfy acceptance requirements typically prescribed by technical specification for PA mixtures in terms of indirect tensile strength (ITS);
- recycled PA mixtures show performance similar to the reference mixture in terms of Indirect Tensile Strength in both dry and wet condition and less water sensitivity;
- Cantabro test results show that recycled PA mixtures can perform as well as (or even better than) the reference PA mixture in dry condition whereas the presence of RAP affect bitumen-aggregates bonding properties after the wet conditioning;
- Semi circular bending (SCB) tests show that the capability of PA mixtures to resist crack propagation is not negatively influenced by the presence of RAP. On the other hand, it can be observed a decrease of the fracture energy for the recycled PA mixtures that can be attributed to the larger presence of aged bitumen within the RAP that leads to less ductile response;
- recycled PA mixtures with 20% of RAP show resistance to repeated indirect tensile loading in submerged conditions similar to the reference PA mixture whereas PA mixtures with 25% of RAP demonstrated a significant decrease in performance at high total bitumen content.
In conclusion, the overall experimental suggests that 5.50% of total bitumen content can be considered as the optimum value for PA mixtures with 20% of coarse RAP whereas both

5.50% and 5.75% of total bitumen content can be adopted as optimum value for PA mixtures with 25% of coarse RAP. Based on the promising results emerged from the present research and previous studies, it is possible to conclude that the hot recycling is a suitable solution also for PA mixtures, as already largely accepted for dense-graded mixtures.

5.3. Field investigation

Based on the results from the previous laboratory experimentation, recycled PA mixtures including 20% of RAP were selected for further studies. In particular, the promising results obtained on both durability and water resistance of laboratory recycled PA mixtures leads to further study on similar PA mixtures prepared in the asphalt plant during the construction of a real scale trial section along an in-service motorway (hereafter named field mixtures). In order to evaluate the durability and water susceptibility of the field mixtures, a comprehensive experimental program was carried out. The study focuses on the evaluation of durability issues as they are the main failure causes in PA (Hagos et al., 2007). In this sense, indirect tensile strength tests, particle loss (Cantabro) tests, semicircular bending (SCB) tests and repeated indirect tensile tests were carried out in both dry and wet condition to evaluate acceptability, durability, and water sensitivity of recycled mixtures. Moreover, compactability properties of the reference and the recycled PA mixtures were compared in order to evaluate the effect of recycled aggregates on the ability of PA mixtures to be properly compacted.
Furthermore, the construction of the trial section also allowed the field evaluation of drainage properties of the studied materials.

5.3.1 Experimental investigation

<u>*Materials*</u>

The aim of this study is to verify if standard PA mixtures performance (containing only virgin aggregates) can still be guaranteed if 20% of virgin aggregates are substituted with selected coarse RAP aggregates from old PA mixtures. In this sense, one reference PA mixture and two recycled PA mixtures prepared with 20% of RAP and different bitumen contents were investigated.
The reference mixture, hereafter named P00_field, was a typical porous asphalt mixture prepared with virgin basaltic aggregates and a polymer (SBS) modified bitumen (5% by weight of the aggregates).
The recycled mixtures (hereafter named R20_5.25_field and R20_5.50_field) were prepared with 20% of RAP and two total bitumen contents (5.25% and 5.50% by the aggregates weight). Recycled materials from milled PA surface layers were sieved so that only the 8/16 mm fraction is used in the recycled PA mixtures. The total bitumen content includes the virgin bitumen and the bitumen within the RAP which was equal to 4.0% by

weight of RAP. Higher total bitumen contents were selected for the recycled PA mixtures to take into account that a part of the aged bitumen within the RAP acts as "black aggregate".
The materials were taken from the field during the construction of a full-scale trial section as a part of an in-service motorway rehabilitation project. The mixtures were prepared at the asphalt plant according to the mix design previously developed in the laboratory (paragraph 5.2). Asphalt mixtures from the field were analyzed in the laboratory for determining the actual total bitumen content and aggregate grading curves. The materials prepared at the asphalt plant comply with acceptance requirements prescribed by Italian technical specification (Autostrade, 2013). Moreover, as shown in Table 5.10, all the aggregate grading curves strictly adhered to the composition requirements developed in the laboratory. It is also possible to notice that the recycled mixtures were characterized by almost the same grading curve (obtained after bitumen extraction from the produced PA mixtures) of the reference mixture without compromising volumetric properties and thus drainability. Also the actual bitumen contents were very similar to the design bitumen contents, meaning that the production in large scale of recycled PA mixtures is possible without compromising the mixtures composition.

Table 5.10 Field mixtures composition.

Sieve size (mm)	**P00_field**		**R20_5.25_field**		**R20_5.50_field**	
	Passing (%)		Passing (%)		Passing (%)	
	Design	**Actual**	Design	**Actual**	Design	**Actual**
20	100	**100**	100	**100**	100	**100**
14	91.5	**88.3**	93.5	**91.1**	93.5	**91.8**
10	41.3	**46.9**	45.9	**45.9**	45.9	**52.8**
6.3	23.9	**23.8**	23.4	**25.0**	23.4	**25.1**
2	15	**14.3**	13.6	**15.0**	13.6	**15.3**
0.5	9.1	**10.0**	8.9	**10.7**	8.9	**8.9**
0.25	7.5	**8.0**	7.3	**8.4**	7.3	**7.2**
0.063	5.3	**5.7**	4.8	**5.3**	4.8	**5.1**
Bitumen	5.00	**4.91**	5.25	**5.22**	5.50	**5.39**

Test program and methods

Materials taken from the asphalt plant were compacted in the laboratory using a gyratory compactor. In order to study the compactability properties of the mixtures, the Compaction Energy Index (CEI) was evaluated. The mixtures were also evaluated in terms of mechanical properties and water sensitivity. In order to simulate water damage, specimens of each mixture were conditioned in air (dry) or in water (wet) at 40°C for a period of 72 h, as suggested by EN 12697-12 (Method A). Indirect tensile strength (ITS) tests, particle loss (Cantabro) tests and semi-circular bending (SCB) tests were carried out in both dry and wet conditions. Repeated indirect tensile tests were also performed in submerged condition with specimens fully immersed in water throughout tests in order to simulate real field conditions.

Finally, pavement drainability was measured in the field in order to evaluate if recycled PA surfaces are comparable to the reference PA surface and complies the technical specifications.
The overall experimental program is summarized in Table 5.11.

Table 5.11 Experimental program: laboratory and in-situ tests.

Type of test	Test condition	Test repetitions		
		P00_field	R20_5.25_field	R20_5.50_field
ITS	dry	4	4	4
	wet	4	4	4
Cantabro	dry	4	4	4
	wet	4	4	4
SCB	dry	4	4	4
	wet	4	4	4
Repeated indirect tensile	dry	3	3	3
	wet	3	3	3
	submerged	3	3	3
In situ drainability		6	6	6

5.3.2 Results and analysis

Compactability properties

Compactability of reference and recycled PA mixtures was investigated by means of CEI values. Results are obtained as the average of eight repetitions for 100 mm diameter specimens and as the average of four repetitions for 150 mm diameter specimens. Figure 5.10 shows the mean results for both reference and recycled PA mixtures along with the corresponding error bars reporting the minimum and the maximum value for each test condition.

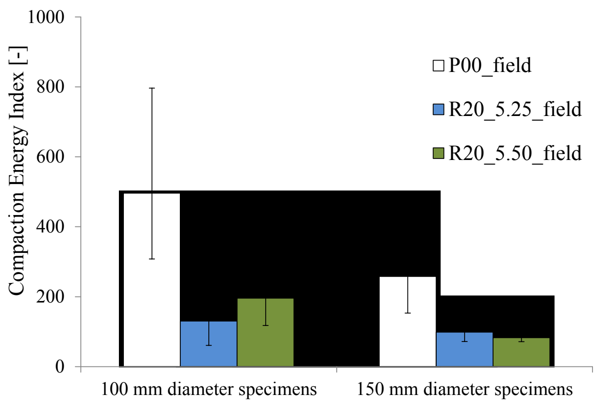


Figure 5.10 Compaction properties in terms of CEI values for the field mixtures.

Results show a significant decrease in CEI value for recycled PA mixtures compared to the reference one, in case of both 100 mm and 150 mm diameter. Such outcome suggests that recycled PA mixtures can be compacted easily than the reference one and this can be attributed to the higher total bitumen content of recycled mixtures. Moreover, the presence of pre-coated RAP aggregates tends to reduce the intergranular friction helping the compaction of the recycled PA mixtures.

Indirect tensile tests

Indirect tensile test results in both dry and wet conditions are given in Figure 5.11 and Figure 5.12 in terms of mean Indirect Tensile Strength (ITS) and Indirect Tensile Coefficient (ITC), respectively. Moreover, water susceptibility is shown in both graphs by means of Indirect Tensile Strength Ratio (ITSR) and Indirect Tensile Coefficient Ratio (ITCR); the error bars represents the minimum and maximum values obtained for each testing condition.

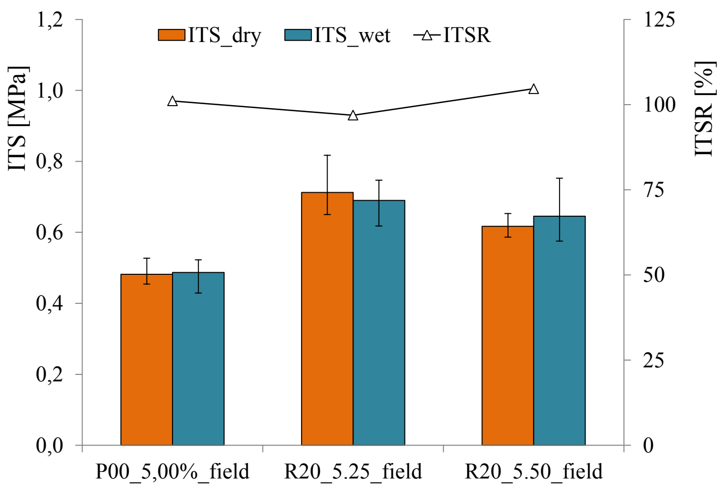


Figure 5.11 Indirect tensile tests results in terms of ITS for the field mixtures R20.

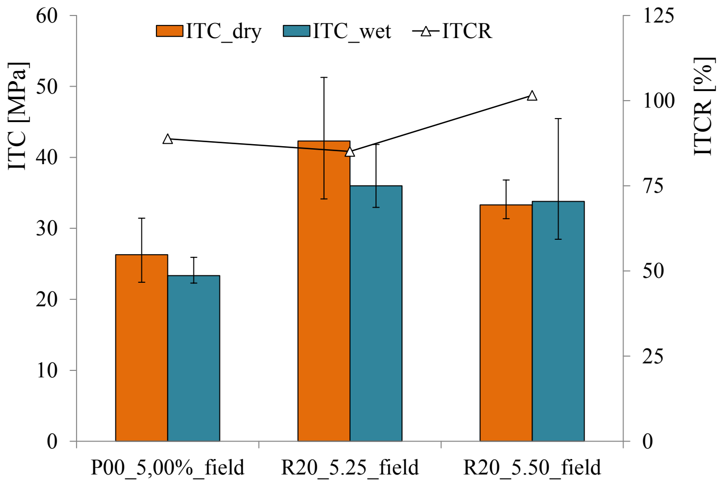


Figure 5.12 Indirect tensile tests results in terms of ITC for the field mixtures R20.

Results show that all mixtures satisfied acceptance requirements prescribed in dry conditions by Italian technical specification (ASPI 2013) for PA mixtures in motorway pavements in terms of ITS and ITC (ITS ≥ 0.40 MPa; ITC ≥ 22 MPa). Moreover, recycled porous asphalt mixtures provide higher performance than the reference mixture in both dry and wet conditions, confirming previous studies (Goh & You 2012, Frigio et al., 2013, Frigio et al., 2014).

In fact, the presence of still coated RAP aggregates allows better adhesion properties within the mixture improving performance under indirect tensile configuration tests.

As far as moisture susceptibility is concerned, results show that recycled PA mixtures can perform as well as the reference one, demonstrating comparable or even improved water sensitivity characterized by high values of ITSR (≈100%).

As already described in details in paragraph 5.2, similar recycled PA mixtures including 20% of RAP aggregates and the same total bitumen contents were prepared in laboratory for a preliminary evaluation and mix-design optimization. Laboratory and field mixtures were prepared using the same basalt aggregates, filler, recycled materials as well as SBS-modified bitumen. Moreover, the same production temperatures were selected in the laboratory for preparing the previous mentioned mixtures in order to simulate as closely as possible the in-plant production. Hence, a performance-based comparison between laboratory and field mixtures can be carried out in order to evaluate the effect of large scale production of recycled PA mixtures. Such an evaluation is shown in Figure 5.13 in both dry and wet conditions. Both recycled PA mixtures are characterized by higher indirect tensile resistance in both dry and wet conditions whereas the reference mixture shows a decrease in performance. Results confirm that a large scale in-plant production of recycled PA mixture is possible and lead to even improved ITS with respect to laboratory produced mixtures.

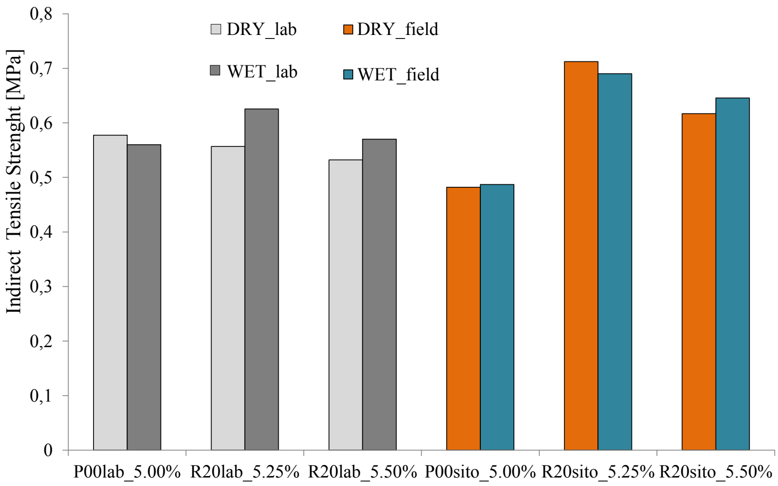


Figure 5.13 Performance-based comparison in terms of ITS for laboratory and field mixtures.

Furthermore, an additional comparison can be carried out by considering all the in-plant produced recycled PA mixtures. As a matter of fact, previous field mixtures were produced for a trial section using recycled PA mixtures including 15% of RAP aggregates. Thus, it is interesting to compare performance of the two types of field mixtures including different amount of RAP (15% and 20%) and total bitumen contents. In particular, during the previous study, three PA mixtures were prepared: one reference mixture including only virgin aggregates and 5.00% of bitumen content by aggregate weight (here-after named P00_field1) and two PA mixtures including 15% of selected RAP aggregates and two total bitumen contents equal to 5.00% and 5.25% by aggregate weight (hereafter named R15_5.00_field and R15_5.25_field, respectively).
It is interesting to notice that, as the RAP content in the mixture increases, the optimum total bitumen content was found to increase as well since a larger amount of total bitumen is composed of aged bitumen within RAP whose prominent part acts as “black aggregate”.
Results of all field mixtures are reported in in Figure 5.14. The two types of field mixtures demonstrate comparable performance both in case of reference and recycled mixtures. In particular, field mixtures including 20% of RAP show improved performance in terms of indirect tensile strength in both dry and wet conditions. Such outcome can be attributed to the higher presence of coated RAP aggregates that tends to improve adhesion properties within mixtures, as shown by previous results (Frigio et al. 2013).

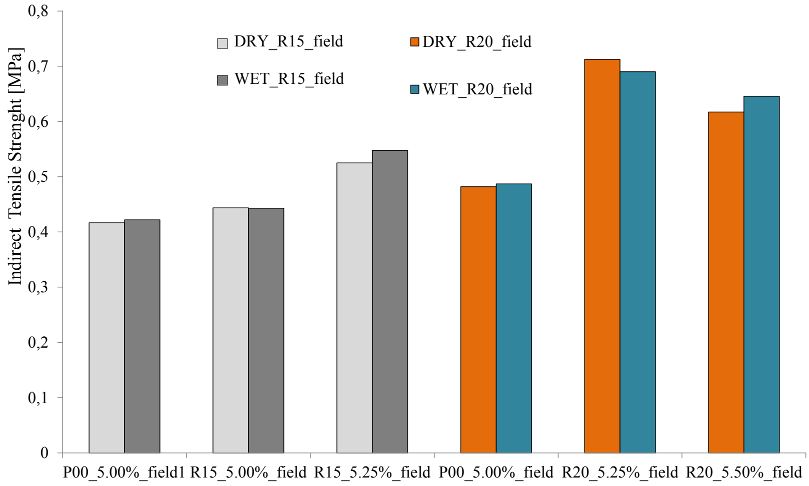


Figure 5.14 Performance-based comparison in terms of ITS for field mixtures R15 and R20.

Cantabro tests

Mean Cantabro test results for both dry and wet conditions are shown in Figure 5.15 in terms of particle loss percentage (the error bars indicate the minimum and the maximum value for each testing condition) along with the corresponding mean air void content.

All studied mixtures demonstrate good performance in terms of ravelling in both dry and wet conditions as the results are consistent with international requirements for high traffic highways corresponds to a maximum of 20% and 35% loss in dry and wet conditions, respectively (Alvarez et al. 2010).

Moreover, results measured on reference and recycled mixtures can be considered fully comparable and, thus, it can be asserted that the particle bonding of PA mixtures is not compromised due to the presence of RAP aggregates.

However, it is worth noting that the recycled PA mixture with higher total bitumen content (R20_5.50_field) exhibit slightly higher particle loss than the reference mixture in both dry and wet conditions, even if specimens were characterized by similar air void contents.

On the other hand, reference mixture experienced water damage (even if minor) whereas both recycled mixtures demonstrate the same performance even after the wet conditioning. According to previous experimental study, the presence of coated RAP aggregates tends to improve the aggregate-bitumen bonding, ensuring enhanced water resistance properties. Thus, as far as water sensitivity is concerned, the ability to preserve ravelling resistance after a wet conditioning is not affected by the presence of RAP aggregates in the mixture confirming the positive results obtained in the case of laboratory mixtures (Frigio et al. 2015).

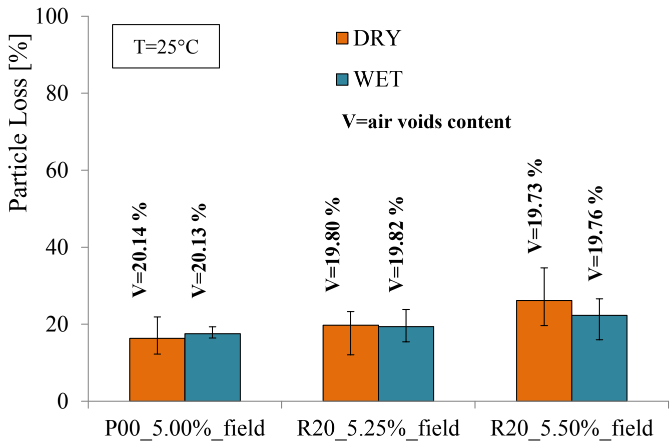


Figure 5.15 Cantabro tests results for the field mixtures R20.

As already described for the Indirect tensile tests, a performance-based comparison between laboratory and field mixtures is carried out in order to evaluate the effect of large scale production of recycled PA mixtures also in terms of Cantabro test results. Such an evaluation is shown in Figure 5.16 in both dry and wet conditions. All in-plant produced PA mixtures demonstrate significant lower particle loss values that the corresponding laboratory mixtures in both dry and wet conditions, indicating higher raveling resistance. It was already shown that Cantabro particle loss values are strictly related to the air voids content of the specimen: in this case, it is possible to notice a significant difference in volumetric properties between laboratory specimens and the in-plant produced specimens. Thus, the improvement in raveling resistance shown by field mixtures can be mainly attributed to the lower voids content.

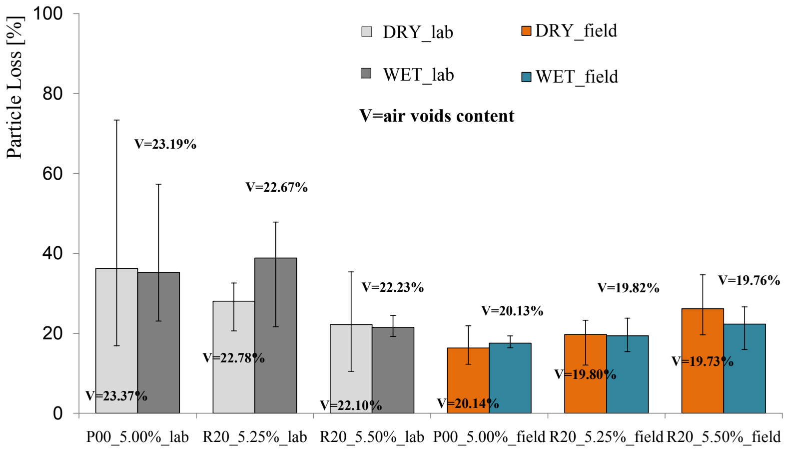


Figure 5.16 Performance-based comparison in terms of Cantabro particle loss for laboratory and field mixtures.

Furthermore, an additional comparison was carried out by considering all the in-plant produced recycled PA mixtures including different amount of RAP (15% and 20%) and total bitumen contents. Results (Figure 5.17) show that all field PA mixtures are characterized by good raveling resistance in both dry and wet conditions and comparable performance. Only the recycled PA mixtures including 20% of RAP aggregates and 5.50% of total bitumen content demonstrate a slightly increase in particle loss values.

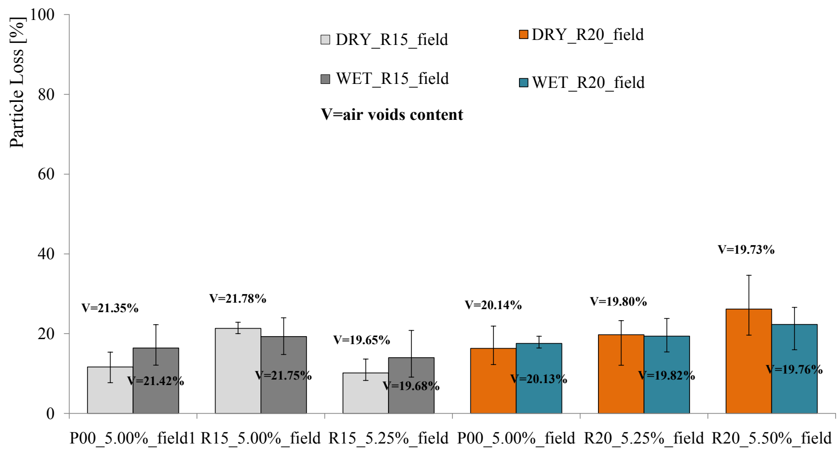


Figure 5.17 Performance-based comparison in terms of Cantabro particle loss for field mixtures R15 and R20.

Fracture properties

The evaluation of the crack propagation aptitude in relation to the presence of recycled material in the PA was carried out by means of Semi-circular bending (SCB) tests. Both fracture toughness and fracture energy were evaluated in dry and wet conditions in order to take into account the water effect on PA mixtures durability.
Mean values of fracture toughness K of all mixtures in both dry and wet conditions are shown in Figure 5.18 along with the water sensitivity parameter (Kratio) calculated as the ratio between K_{wet} and K_{dry}. Recycled mixtures show slightly higher resistance to crack propagation than the reference mixture in both dry and wet condition, meaning that the presence of RAP aggregates tends to stiff the material. Moreover, the water sensitivity is found to be fully comparable between mixtures meaning that the presence of RAP does not affect the ability of PA mixtures to resist to water damage.
Moreover, mean values of fracture energy G for all mixtures in both dry and wet conditions are shown in Figure 5.19. Recycled mixtures exhibited a decrease in fracture energy with respect to the reference one in both dry and wet condition, meaning that the presence of RAP leads to more brittle materials. The fracture energy for recycled mixtures tends to increase with the bitumen content according to Biligiri et al. (2012). As far as water sensitivity is concerned, both recycled PA mixtures show similar or even better resistance to water effects with respect of the reference mixture.

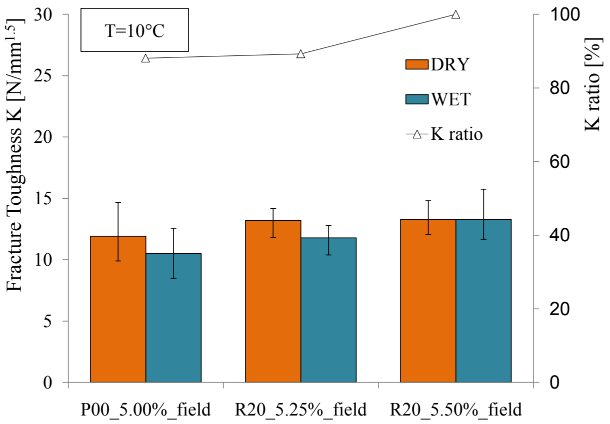


Figure 5.18 SCB tests results in terms of fracture toughness for the field mixtures R20.

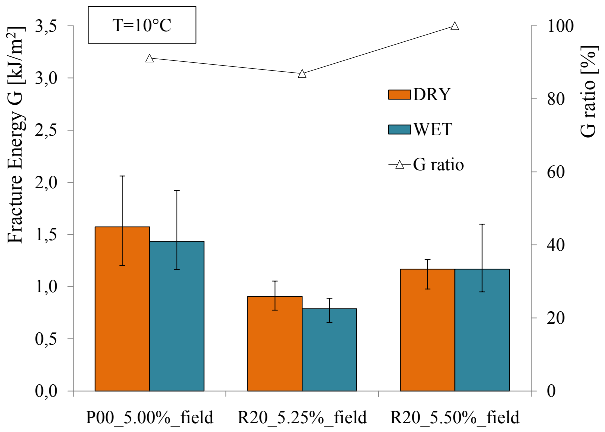
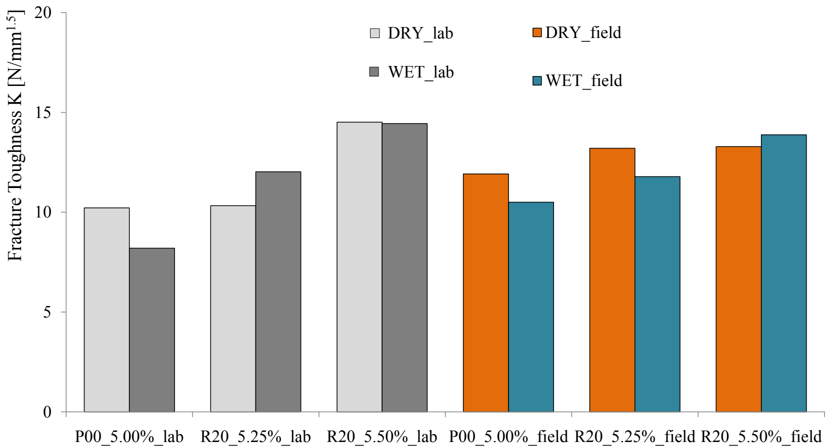
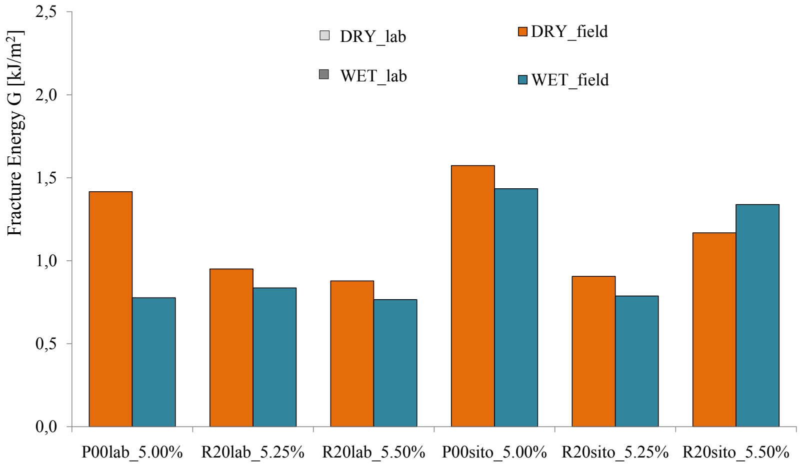


Figure 5.19 SCB tests results in terms of fracture energy for the field mixtures R20.

A performance-based comparison between laboratory and field mixtures is carried out in order to evaluate the effect of large scale production of recycled PA mixtures also in terms of fracture propagation resistance. Such an evaluation is shown in Figure 5.20 in both dry and wet conditions for the K and G parameters. It is possible to notice that results in terms of fracture toughness as well as fracture energy are very similar between the corresponding laboratory and field mixtures. In particular, recycled PA mixtures show similar or even improved fracture toughness values than the reference PA both in case of laboratory and in-plant produced mixtures. Analogously, the decrease in fracture energy due to the presence of RAP is evident regardless the fact that the mixtures were produced in the laboratory or taken during the in-plant production of an experimental trial section.



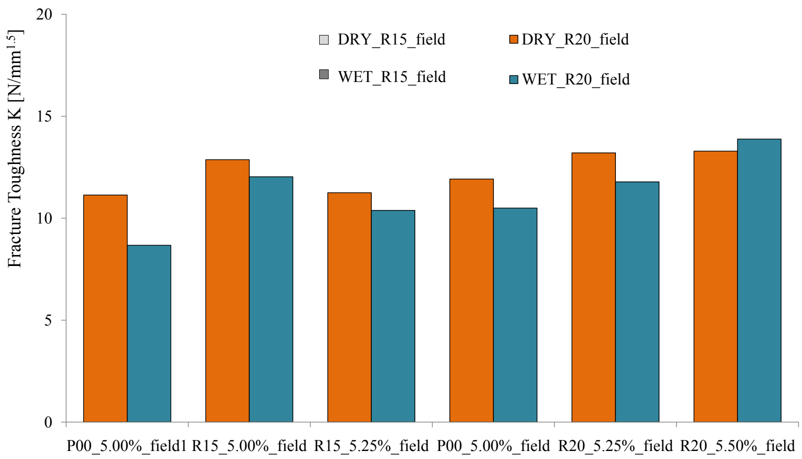
(a)



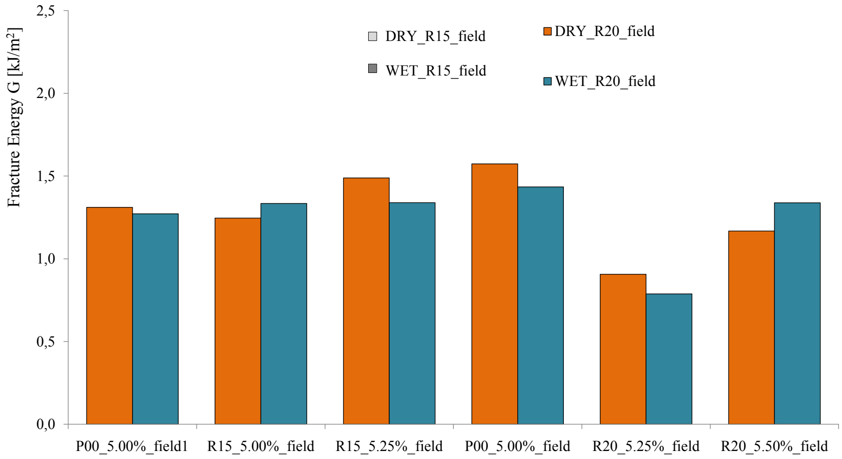
(b)

Figure 5.20 Performance-based comparison for laboratory and field mixtures in terms of fracture toughness (a) and fracture energy (b).

Furthermore, an additional comparison was carried out by considering all the in-plant produced recycled PA mixtures including different amount of RAP (15% and 20%) and total bitumen contents. Results (Figure 5.21) show that all field PA mixtures are characterized by similar fracture toughness regardless the amount of RAP included or the total bitumen content.



(a)



(b)

Figure 5.21 Performance-based comparison for field mixtures R15 and R20 in terms of fracture toughness (a) and fracture energy (b).

As far as the fracture energy is concerned, it is possible to notice a significant decrease in G values for the recycled PA including 20% of RAP, regardless the bitumen content. Such a result indicate that as the amount of recycled material increases, the mixture tends to became more brittle at intermediate-low temperatures due to the presence of aged bitumen.

Repeated indirect tensile test

Repeated indirect tensile tests were performed in order to investigate the water resistance of the mixtures similarly to what proposed by Kim and Coree (2005). It is well known that, in the field, asphalt pavements experience water damage only under repeated traffic loading and when they are saturated. Thus, in order to simulate closely real field condition, the specimens tested in submerged conditions were immersed in water throughout test: in this way the saturated sample is repeatedly loaded and water can inhaled into and exhaled from the sample at each load application (Poulikakos & Partl 2009).

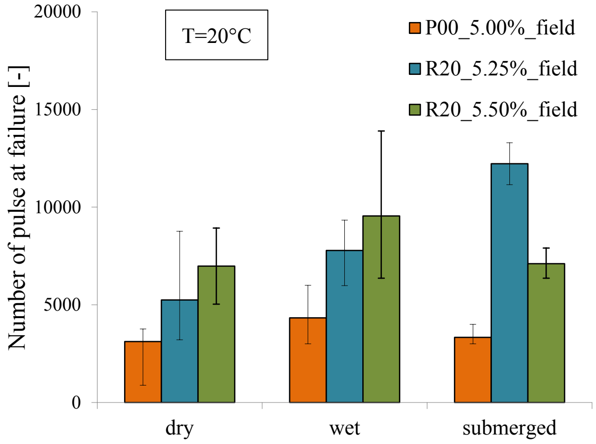


Figure 5.22 Repeated indirect tensile test results for the field mixtures R20.

The number of pulses at failure was taken into account for each specimen; the average data, along with error bars reporting the minimum and the maximum value obtained, are reported in Figure 5.22 for all the mixtures and testing conditions. First of all, it is interesting to notice that the water conditioning (both wet and submerged conditions) does not seem to significantly affect the PA mixtures performance.

Both recycled PA mixtures demonstrate to outperform the reference mixture for all tested conditions, meaning that the presence of RAP aggregates is able to improve the PA mixtures performance under repeated loading cycles. Moreover, the performance of recycled PA mixtures tends to improve as the total bitumen content increases, with the exception of the submerged condition case: the mixture R20_5.50_field show a decrease in performance with respect of R20_5.25_field in submerged condition, still ensuring better results than the reference mixture.

A performance-based comparison between laboratory and field mixtures is carried out in order to evaluate the effect of large scale production of recycled PA mixtures also in terms of repeated load resistance. Such an evaluation is shown in Figure 5.23 in both dry and wet conditions. It is possible to notice that the reference PA mixture behavior is very similar between laboratory and in-plant produced mixtures whereas both recycled mixtures perform significantly better in case of in-plant produced specimens. Hence, the large production of PA mixtures leads to improved performance in terms of repeated load resistance if compared to laboratory produced mixtures.

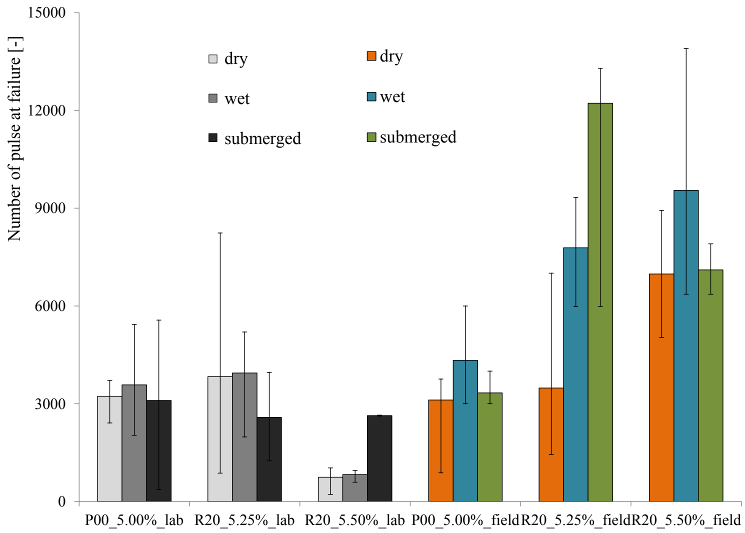


Figure 5.23 Performance-based comparison in terms of pulse number at failure for laboratory and field mixtures.

Furthermore, an additional comparison was carried out by considering all the in-plant produced recycled PA mixtures including different amount of RAP (15% and 20%) and total bitumen contents. Results (Figure 5.24) show that in-plant produced PA mixtures including 20% of RAP aggregates can perform significantly better that in-plant produced recycled PA with 15% of RAP. Thus, it is possible to conclude that the presence of larger amount of recycled aggregates is able to enhance repeated load resistance probably due to increased adhesion at the aggregate-bitumen interface.

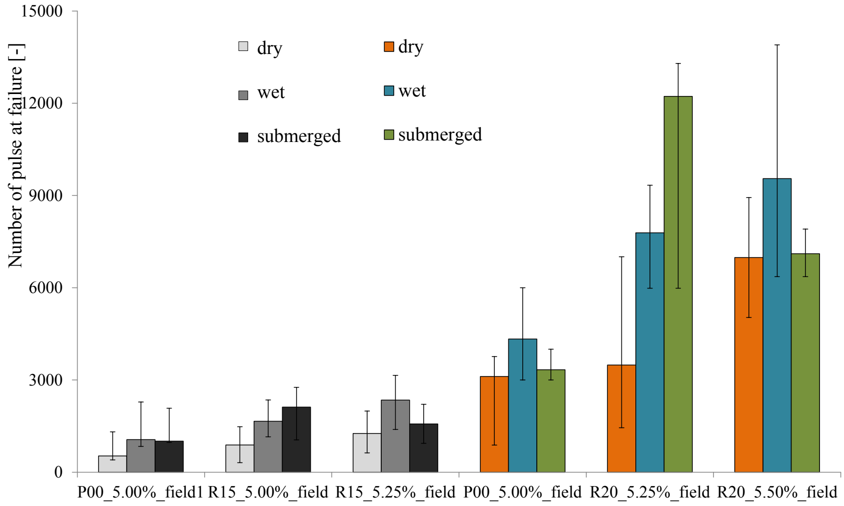


Figure 5.24 Performance-based comparison in terms of Cantabro particle loss for field mixtures R15 and R20.

Field drainability test

Drainage properties of the studied PA mixtures were evaluated in the field. The first measurements were taken 1 day after the construction of the full-scale trial section (before traffic opening) whereas the second measurements were taken 90 days after the construction in order to investigate the evolution in drainage performance.
Mean results in terms of drainage capability are given in Figure 5.25; each value is the average of six measurements spaced 50 meters apart. The percent-age loss in drainage capability (Δ) due to post compaction and traffic loads is also shown in the figure.
Results show that all mixtures satisfied acceptance requirements prescribed by Italian technical specification (ASPI 2013) for PA mixtures in motorway pavements in terms of drainage capability measured within 14 days after the construction (drainage capability = 10 ÷ 25 l/min). Moreover, only the recycled mixture with high total bitumen content (R20_5.50%_field) show a slight decrease in drainage capability with respect to the reference mixture both in terms of the measurements taken 1 day and 90 days after the construction.
As far as percentage loss is concerned, results are fully comparable for all mixtures demonstrating that the drainage evolution is not affect by the presence of RAP aggregates within PA mixtures and the results are within the limits even after 90 days.

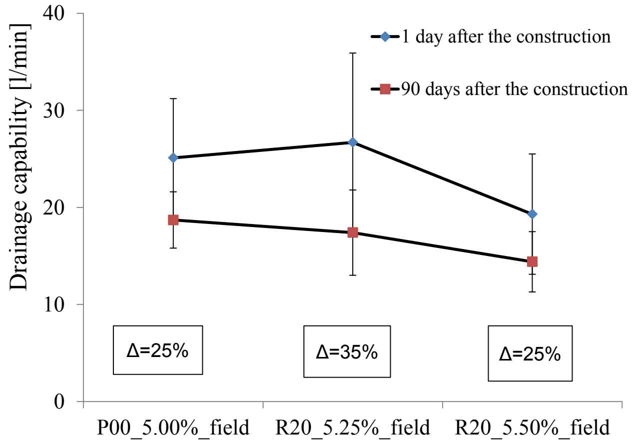


Figure 5.25 In field drainage capability for the mixtures R20.

Moreover, drainage capability measurements were taken 1 day after the construction also after the previous trial section that involved recycled PA mixtures with 15% of RAP aggregates (R15). Hence, it is interesting to compare drainability properties in case of virgin and recycled PA mixtures including different amount of recycled materials as well as different bitumen contents. Measurements of both trial sections are given in Table 5.12.

Table 5.12 Comparison of drainage capability for the filed mixtures R15 and R20.

Measurements date	Mixture	Drainage capability [l/min]	Coefficient of variation [%]
06.09.2013	P00_5.00%_field1	29	15,3
	R15_5.00%_field	31	20,4
	R15_5.25%_field	30	20,6
19.06.2014	P00_5.00%_field	25	6,1
	R20_5.25%_field	27	9,2
	R20_5.50%_field	19	6,2

The results are fully comparable and, in both cases, drainage properties exceed the acceptance requirement pre-scribed by Italian technical specification (ASPI 2013) for PA mixtures in motorway pavements. All experimental results demonstrate that the partial substitution of virgin aggregates with different amount of selected RAP aggregates in PA mixtures is possible and guarantee improved performance with respect to the virgin PA mixture, if an accurate mix design is performed.

5.3.3 Conclusions

The present experimental study focuses on the usability of selected reclaimed asphalt pavement (RAP) into porous asphalt (PA) mixtures. Only coarse RAP aggregates (8/16

mm) obtained by milling old PA surface layers were used at a rate of 20% of the total aggregate weight.
A comprehensive laboratory experimental program for investigating acceptability, compactability, durability, fracture resistance and water sensitivity was carried out along with the in situ monitoring of drainage properties.
The following main conclusions can be drawn:
- recycled PA mixtures demonstrate better compactability properties than the reference mixture in terms of Compaction Energy Index;
- recycled mixtures were able to satisfy acceptance requirements typically prescribed by technical specification for PA mixtures in terms of indirect tensile strength (ITS);
- recycled PA mixtures provided higher performance in terms of ITS with respect to the reference mixture in both dry and wet conditions and similar water sensitivity (Indirect Tensile Strength Ratio – ITSR);
- Cantabro test results showed that the presence of recycled aggregates does not sensibly affect the resistance to ravelling (bitumen-aggregates bonding properties) and water susceptibility of the PA mixtures;
- recycled PA mixtures were characterized by similar or even enhanced crack resistance potential but lower fracture energy properties. Such effect on fracture energy tends to reduce as the bitumen content within recycled mixtures increases. Moreover the water sensitivity is found to be fully comparable between mixtures meaning that the presence of RAP does not affect the ability of PA mixtures to resist to water damage;
- results in terms of repeated indirect tensile tests showed that both recycled PA mixtures demonstrate to perform better than the reference mixture for all tested conditions;
- all mixtures satisfy acceptance requirements for PA mixtures in motorway pavements in terms of drainage capability. Moreover, the drainage evolution due to post compaction and traffic loads is not affect by the presence of RAP aggregates within PA mixtures.
In conclusion, the overall experimental results confirmed that including 20% of coarse recycled aggregates in porous asphalt mixtures does not compromise performance and durability of the PA layers if an adequate bitumen content is selected.
Moreover, the results were compared with those of previous research studies regarding laboratory PA mixtures including the same amount of RAP aggregates and total bitumen contents, demonstrating that the laboratory research phase was representative of the real plant conditions and the selected mix design was accurate. Generally, the field mixtures outperform the corresponding laboratory ones, suggesting that the production of such delicate mixtures is possible even in large scale if an accurate mix deign is performed.
Finally, the comparison with previous field PA mixtures including 15% of coarse RAP was carried out. The overall performance are found to be comparable; enhanced results obtained for field mixtures including 20% of RAP in terms of indirect tensile configuration tests suggest that the higher presence of coated RAP aggregates improves adhesion properties within mixtures, confirming previous studies.

Chapter 6.

Reactivated bitumen investigation

6.1. Introduction

As already detailed explained in Chapter 2.4, one of the main concerns for the use of RAP in the production of new asphalt mixtures is related to the properties of the aged bitumen included in RAP and how it can affect the quality of the final bitumen phase of the recycled mixture. In general, asphalt bitumen demonstrates two stages of aging: one during construction (short-term aging) and one during the service life (long-term aging). The level of aging that asphalt bitumen experiences during production and service depends on the void content of the HMA. In addition, properties of aged bitumen depend on the level of damage to the recycled pavement (Smiljanic et al., 1993). The greater the damage to the pavement prior to recycling, the greater the changes are in the properties of the bitumen. This is illustrated by the reduced oxidation susceptibility in pavements that are better preserved. Stockpiling also accelerates bitumen aging as the material is more prone to air exposure and oxidation (McMillan and Palsat, 1985).
As asphalt bitumen reacts and loses some of its components during the aging process, its rheological behavior will naturally differ from virgin materials. This suggests the importance of controlling the blending process between recycled and virgin bitumens. If the old bitumen is too stiff, the blend of old and virgin bitumens may not perform as expected. At small percentages (up to 20%), an aged bitumen does not significantly affect the properties of the blend of virgin and RAP bitumen (Kennedy et al. 1998). However, when used at intermediate to higher percentages, aged bitumen can significantly influence the properties of the blend and may affect the resultant bitumen grade. Recent modifications have been introduced to conventional asphalt plants in order to reduce aging of the old bitumen during mix production.
In order to predict and estimate the amount of blending between virgin and aged bitumen and, thus, the properties of the final bitumen, it is essential to evaluate what percentage of the bitumen from RAP will be re-activated during the mixing process. The production of recycled HMAs, in fact, implies a heating process that involves RAP aggregates when they are mixed with the pre-heated virgin aggregates and bitumen. In turn, this heating phase could lead to a partial melting of the aged bitumen that coats RAP aggregates. Such bitumen represents the “re-activated” bitumen that could interact with the virgin bitumen contributing to the overall performance of the final recycled HMA. Mixture performance change as a function of the re-activated bitumen amount released by RAP during the heating process that depends on several factors mainly related to RAP properties (e.g.

bitumen type and content, aggregate gradation, aging degree) and production process parameters (e.g. production temperature, RAP pre-heating time, storage and transportation time) (Shirodkar et al., 2011).
As already explained, the re-activated bitumen degree varies between two limit conditions: full RAP bitumen re-activation (100% re-activation) and "black rock" condition (0% re-activation). Several studies consistently concluded that the real re-activated bitumen degree is comprised between the above-mentioned limit conditions.
Given this background, an attempt to estimate the degree of re-activation of the bitumen from RAP is reported in the present chapter regarding the PA mixtures. Further studies will be needed in order to extend such an hypothesis to different mixtures, including dense-graded mixtures.

6.2. Experimentation

On the basis of a performance-based equivalence principle, a reliable approach for a practical method able to predict the amount of "re-activated" bitumen within the RAP is proposed. The method is based on the assumption that the "re-activated" bitumen content is proportional to the average "re-activated" bitumen film thickness (R_{TF}) which is considered constant for a given heating process (mixing temperature and time) and aged bitumen type. The calculation of R_{TF} is based on the average asphalt film thickness (A_{FT}) concept that relate the calculation of A_{FT} to aggregate surface area and effective volume of bitumen. Thus, it is possible to evaluate the corresponding "re-activated" bitumen content from such R_{TF} as long as the RAP aggregates gradation is known.

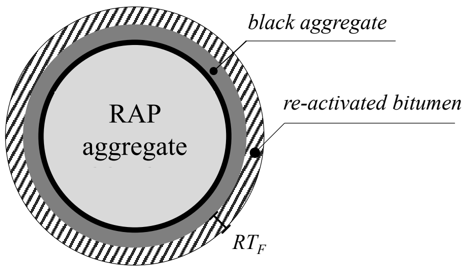


Figure 6.1 Re-activated film thickness.

The estimated bitumen content values seem congruent with the experimental results presented in detail in this paper.
As described above, three total bitumen contents (5.25%, 5.50% and 5.75%) were considered for each mixture in order to select the suitable mix design for each recycled PA. Overall, based on the experimental data, taking into account all the selected performance-related parameters as well as the statistical significance of the test results, it is possible to conclude that 5.50% of total bitumen content can be assumed as the optimum value for PA

mixtures with 20% of coarse RAP. On the other hand, tests results seems to suggest that both 5.50% and 5.75% of total bitumen content could suit for PA mixtures with 25% of coarse RAP.

It is interesting to notice that, as the amount of RAP in the mixture increases, the total bitumen content needs to increase as well, since the total bitumen includes the aged bitumen within the RAP whose a prominent part acts as "black aggregate".

Based on this remark, a closer examination has been performed about the quantification of the "re-activated" bitumen of the RAP.

Both laboratory and field studies demonstrated that PA mixtures including 15% of coarse RAP and prepared with a total bitumen content of 5.25% can perform as well as virgin PA mixtures prepared with a bitumen content of 5.00%. Since RAP included 4.0% of aged bitumen (by weight of RAP), a recycled mixture with 15% of RAP and 5.25% of total bitumen is characterized by 0.6% of aged bitumen and, consequently, 4.65% of virgin bitumen.

Thus, assuming a performance-based equivalence principle, it is likely to assert that about half of the bitumen within the RAP acts as "black aggregate" while the other half (namely the "re-activated" bitumen of the RAP equal to 0.3% of the total bitumen) blends with the virgin bitumen so that the mixture can count on 4.95% (≈5.00%) of "working" bitumen. At the same time, it is correct to state that the "re-activated" bitumen content is proportional to the bitumen film thickness which covers the aggregate particles.

Thus, it is truthful that the deepest film of aged bitumen acts as "black aggregate" while the heat propagation during mixing allows a separation of the external film from the RAP ("re-activated" bitumen), as shown in Fig. 11. The basic principle assumed here is that the average "re-activated" bitumen film thickness (R_{TF}) is constant for a given heating process (mixing temperature and time) and aged bitumen type. Consequently, it is possible to evaluate the corresponding "re-activated" bitumen content from such R_{TF} as long as the RAP aggregates gradation is known.

The method proposed in this paper is based on the average asphalt film thickness concept that has been adopted for the Hot Mix Asphalt mix design (Elseifi et al., 2008; Attia et al., 2009; Kandhal et al., 1998; Brown et al., 2009). The calculation of the average film thickness (A_{FT}) for a given mixture is based on the assumption that its aggregate particles are spherical and covered with the same film thickness (average value). Thus, A_{FT} can be related to aggregate surface area and effective volume of bitumen (Brown et al., 2009). The same concept can be applied to the "re-activated" bitumen whose content within a RAP can be calculated as a function of its film thickness and RAP aggregates surface area (SA) as shown in Equation (6.1):

$$RB = \frac{SA \times P_a \times G_b \times RT_F}{1000} \tag{6.1}$$

where RB is the percentage (by weight) of "re-activated" bitumen, SA is the RAP aggregates surface area (m^2/kg), P_a is the percentage (by weight) of aggregate, G_b is the

specific gravity of asphalt bitumen (g/cm^3) and R_{TF} is the average "re-activated" bitumen film thickness (μm).
The RAP aggregates surface area (SA) is given by the aggregates gradation on the basis of the percentage passing a specific set of sieves, according to the Equation (6.2):

$$SA = \frac{1}{100} \times \sum_{i}^{N} P_i \times CP_i \tag{6.2}$$

where N is the number of sieves considered in the surface area calculation, P_i is the percentage of aggregate passing sieve i and CP_i is the surface area factor for a given sieve size.
Therefore, knowing the RAP aggregates gradation it is possible to calculate the RAP aggregates surface area and then the "re-activated" bitumen content.
In the case of the RAP studied in this experimental research, SA is equal to 4.67 m^2/kg (Table 6.1) whereas RB is assumed equal to 50% of RAP bitumen which corresponds to 2.00% (0.5×4.00%). Thus, considering the specific gravity of bitumen (G_b) equal to 1.02 g/cm^3, it is possible to calculate an average "re-activated" bitumen film thickness R_{TF} equal to 4.28 μm.

Table 6.1 Calculation of the aggregates surface area.

Sieve size	Passing P_i	Surface Area Factor CP_i	Surface Area P_i×CP_i
mm	%	m^2/kg	m^2/kg
20	100	0,41	41,00
4,75	32,23	0,41	13,21
2,36	19,8	0,82	16,24
1,18	15,80	1,64	25,91
0,6	13,0	2,87	37,31
0,3	10,4	6,14	63,86
0,15	7,50	12,29	92,18
0,075	5,40	32,77	177,06
			SA= 4,67

Since the RAP aggregates surface area remains unchanged for all the studied PA mixtures, the percentage of "re-activated" bitumen can be considered the same (50% of RAP bitumen). In this sense, Table 6.2 shows the appropriate virgin bitumen content (E) predicted in the case of PA mixtures with 20% and 25% of RAP in order to achieve the same performance (i.e. the same working bitumen content) of the PA mixtures with 15% of RAP. The corresponding values of the total bitumen content reported in Table 6.2 (5.35% and 5.45%, respectively) are congruent with the experimental results presented in detail in this thesis. In fact, as illustrated above, 5.50% of total bitumen content was found to be the optimum value for PA mixtures with 20% of coarse RAP whereas both 5.50% and 5.75% of total bitumen content could be adopted for PA mixtures with 25% of RAP.

Therefore, using Equation (6.1), the proposed method should be able to predict the percentage of “re-activated” bitumen by assuming a constant “re-activated” bitumen film thickness as long as RAP aggregates gradation is known. Such an assumption requires to be confirmed with further studies on different RAP but it prefigures a reliable approach to predict the amount of “re-activated” bitumen from RAP, which needs to be taken into account during the mix design of hot recycled mixtures.

Table 6.2 Application of the “re-activated” bitumen method.

RAP content (A)	Aged Bitumen (B)	Re-activated bitumen (C)	"Working" bitumen (D)	Required Virgin Bitumen (E=D-C)	Total bitumen (E+B)
%	%	%	%	%	%
20	4.0% of A= 0.8%	50% of B= 0.4%	4.95%	4.55	5.35
25	4.0% of A=1.0%	50% of B= 0.5%	4.95%	4.45	5.45

Part 2. Warm recycling of PA mixtures

Chapter 7.

Literature review: warm recycling

The use of Warm Mix Additive (WMA) in asphalt mixtures allows a significant reduction of production temperatures without compromise workability and compactability properties during mixing at the asphalt plant as well as the in situ lay-down phase. Hence, both environmental and economic benefits can be guaranteed ensuring also better working conditions due to a reduction of harmful gasses.

Such mixtures (usually called warm mixtures) were investigated for the first time in Germany at the late ‘90s but only during the last decade, the scientific community has been working for their development. As a consequence, limited experimental data are nowadays available concerning warm mixtures performance as well as their effective durability (long term issues). In this sense, WMA technologies and their applicability in asphalt industry need to be further investigated. In particular, possible advantages and disadvantages must be carefully considered with respect to hot mixtures, taking into account both environmental, technical as well as economical parameters.

Moreover, it should be taken into account that most of the new produced asphalt mixtures include RAP. In fact, nowadays the use of significant amount of recycled materials for the production of new asphalt pavements is a reality all over the world, also when hot techniques are selected since adequate in-service performance and economic advantages are guaranteed. From an environment point of view, the use of milled materials limits the use of virgin materials such as aggregates and bitumen, helping the preservation of natural resources. On the other hand, it must be considered that the substitution of a significant amount of virgin aggregates with RAP requires the use of higher mixing and compaction temperatures, due to the presence of aged bitumen. The bitumen within RAP, in fact, is highly oxidated and, thus, more viscous than a virgin bitumen; as a consequence, higher production temperatures are needed in order to guarantee adequate workability. Higher production temperatures require significant energy consumption and, thus, higher production costs as well as environmental issues related to harmful gasses emission. The latest aspect is becoming more and more crucial since most of the production plants are located near urban agglomerations; thus, urgent solutions are needed in order to solve the energetic and environmental issues without reducing the use of recycled materials.

A possible solution is the use of warm mix additives to reduce production temperatures especially when recycled materials are involved. The combination of the two above mentioned solutions (RAP and WMA) must be carefully investigated and developed in order to evaluate the feasibility of warm recycled mixtures.

7.1. Advantages and disadvantages of WMA

As already mentioned, the reduced production temperatures that are used in WMA technology leads to a number of both environmental and economic benefits.
First of all, a significant reduction on pollutant and harmful emission has been reported by many studies. Specific researches demonstrated that the production of asphalt mixtures at temperatures up to 80°C do not theoretically causes any emission whereas the standard production at 170-180°C causes a release in the atmosphere of a considerable amount of pollutant (D'Angelo et al., 2008). Generally, depending on the WMA technology used, emissions declared in the literature have some variation. Nevertheless, irrespective of the WMA production process, a significant reduction of emissions is always observed. Evaluations carried out in a number of European countries (EAPA, 2010) showed a significant decrease of various emissions during the production process in plant, as following specified: 30–40% for CO_2 (carbon dioxide) and SO_2 (sulfur dioxide), 50% for VOC (volatile organic compounds), 10–30% for CO (carbon monoxide), 60–70% for NO_x (nitrous oxides), and 25–55% for dust (Figure 7.1).

Emission	Norway	Italy	Netherlands	France
CO_2	31.5	30–40	15–30	23
SO_2	NA	35	NA	18
VOC	NA	50	NA	19
CO	28.5	10–30	NA	NA
NO_x	61.5	60–70	NA	18*
Dust	54.0	25–55	NA	NA

Figure 7.1 Percent reductions in plant emissions with WMA (D'Angelo et al., 2008)

Moreover, a reductions from 30% to 50% for asphalt fumes and polycyclic aromatic hydrocarbons (PAHs) have also been reported, which have a substantial influence on the exposure of the workers and the surrounding area of construction sites to those products.
Furthermore, since operating temperature and emissions are lower, it is easier for plants to be allowed in the proximity of urban areas, in accordance with current environmental policy. Figure 7.2 shows two pictures taken in the same asphalt plant during the production of standard hot temperature (≈160°C) and the production at reduced temperature (≈110°C) by means of warm technologies; clear differences in air quality can be seen due to the significant reduction of production temperatures.

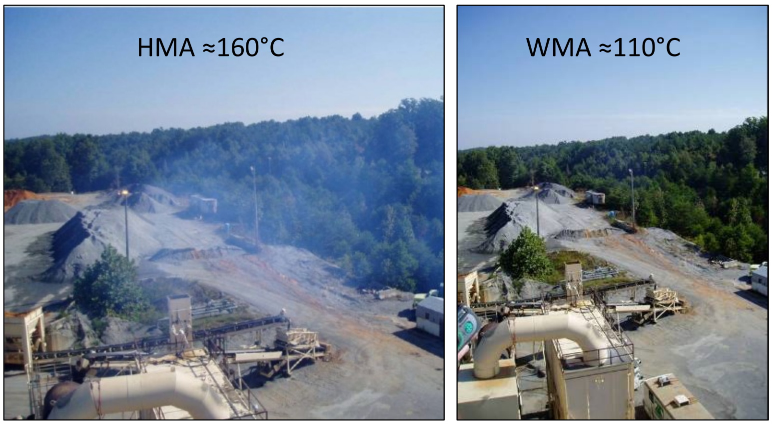


Figure 7.2 Asphalt plant during hot (left) and warm (right) productions

In addition, the use of WMA techniques allows higher haulage distances and reduces the risk of compaction troubles, especially in cold weather; moreover, lower compaction temperatures in turn implies that less time is required to cool the laid material before opening it to traffic or to place the next layer (Button et al., 2007; Capitão et al., 2012).
Among various environmental benefits, it also must be considered the possibility to increase RAP dosage into new asphalt mixtures thanks to low production temperatures as well as enhanced mixtures workability (Brown, 2008; Bonaquist, 2011). The use of reduced production temperatures, in fact, leads to a low aging rate of the bitumen (lower short term aging) that help to compensate the stiffening effect related to the use of high RAP dosage. On the other hand, it must also be taken into account that the use of RAP at low temperatures could lead to a limited reactivation of the aged bitumen within RAP that can affect the mix design and the final mixture performance. The issues related to the combined use of WMA and RAP will be further analyzed in this thesis.
As far as economic savings are concerned, it must be considered that lowering the production temperature allows reducing the energy consumption up to 35%, or more, depending on the WMA process applied (D'Angelo et al., 2008) and on how much the temperature is reduced. Of course, savings in energy consumption must be also considered as an environmental friendly aspect. However, a number of WMA processes require initial investment to partially modify the asphalt plant, whereas some of them require permanent purchase of additives. Therefore, the costs issues must be carefully exanimated, taking into account all the variables (Capitão et al., 2012).
It also needs to be taken into account that the WMA technology is also beneficial compared to cold technology since it does not need curing time before opening up to traffic. In addition, the laying and compaction operations, and the coating of aggregates by the bitumen are better than for cold asphalt mixtures, leading to a better in-service pavement (Button et al., 2007).

As a result of the abovementioned economic and environment advantages related to the use of WMA additives to reduce mixtures production temperatures, nowadays, WMA technologies are gained more acceptances among asphalt industry and a huge number of WMA additives are available on the market.
At the same, some drawbacks must be taking into account when selecting WMA technology for the production at reduced temperatures of asphalt mixtures. First af all it must be mentioned that there are some concerns that the carbon emissions related to the production of WMA additives could actually offset some of the environmental advantages recognized to WMA technology.
Moreover, since there is not enough experience yet on long-term performance of WMA, administrators as well as contractors are still reluctant to a widespread use of these technologies. Some authors (Button et al., 2007) refer to concerns related to in-service moisture susceptibility of WMA mixtures associated to the reduced optimum bitumen content derived from the reduced production temperatures. In fact, low in-plant temperatures can cause an incomplete drying of the aggregates as well as a not uniform coating of the aggregates by the bitumen. Such an issue could be more sever when RAP aggregates are used since they tend to retain more water. As a consequence, poor adhesion at the bitumen/aggregates interface can arise leading to significant water susceptibility of the warm mixtures as well as premature raveling failure, especially in case of open-graded mixtures.
It is also important to highlight that current research are studying the effect of low production temperatures on the interface shear strength between two asphalt layers (Pasquini et al., 2015). First results show that low production temperatures can cause a significant drop on interlayer resistance with consequent de-bonding in case of upper layers where shear stresses related to the traffic are considerable.

7.2. WMA technologies

Due to both economic and environment benefits related to the reduction of production temperatures, a significant number of WMA technologies are reported in the literature and nowadays available in the market (Figure 7.3). Despite that, WMA additives can be, basically, classified in three main groups, based on their chemical compositions and operating processes (EAPA, 2010; Prowell et al., 2011): organic additives, chemical additives and foaming technologies.
Each WMA technology is based on different operating system such as viscosity reduction, introduction of water into the process and reduction of friction at that interface with the aim at lowering mixture production temperatures, as well as achieving the same in-service performance compared to HMA. For a proper selection of the most suitable WMA technology, a careful evaluation of the aspects involved are requires and it must be taken into account that some of the WMA technologies involve a temporary or permanent adjustment of various bitumen properties, such as viscosity.

In the following paragraphs, the three main categories of WMA additives are detailed described.

WMA processes	Product	Company	Description	Dosage of additive	Country where technology is used	Production temperature °C (or reduction range)
Foaming processes						
Water-containing	Aspha-Min®	Eurovia and MHI	Water-containing technology using zeolites	0.3% by total weight of the mix	USA, Germany, France, worldwide	(20–30 °C)
Water-containing	Advera®	PQ Corporation	Water-containing technology using zeolites	0.25% by total weight of the mix	USA	(10–30 °C)
Water-based	Double Barrel Green	Astec	Water-based foaming process	2% water by mass of bitumen; anti-stripping agent	USA	116–135 °C
Water based	Ultrafoam GX	Gencor industries	Water-based foaming process	1–2% water by mass of bitumen	USA	Not specified
Water-based	LT Asphalt	Nynas	Foam bitumen with hydrophilic additive	0.5–1 % by mass of bitumen	Netherlands and Italy	90 °C
Water-based	WAM-Foam	Shell and Kolo-Veidekke	Soft binder coating followed by foamed hard binder	2–5 % water by mass of hard binder	Worldwide	100–120 °C
Water based	Low Energy Asphalt	LEACO	Hot coarse aggregate mixed with wet sand	3% water with fine sand	USA, France, Spain, Italy	<100 °C
Water based	Low Emission Asphalt	McConnaugh–ay Technologies	Hot coarse aggregate mixed with wet sand, combined with chemicals	3% water with fine sand; 0.4% bitumen weight	USA	90 °C
Water based	LEAB	Royal Bam Group	Direct foam with binder additive. Mixing of aggregates below water boiling point	0.1% of bitumen weight of coating and adhesion additive	Netherlands	90 °C
Organic						
FT Wax	Sasobit	Sasol	Fischer-Tropsch wax	Approx. 2.5% by weight of binder in Germany; , 1.0–1.5%, in the U.S.A	Germany as well as 20 other countries	(20–30 °C)
Montan Wax	Asphaltan B	Romonta GmbH	Refined Montan wax with fatty acid amide for rolled asphalt	2.0–4.0% by mass of bitumen	Germany	(20–30 °C)
Fatty Acid Amide wax	Licomont BS	Clariant	Fatty acid amide	3.0% by mass of bitumen	Germany	(20–30 °C)
	3E LT or Ecoflex	Colas	Proprietary	Yes, but not specified	France	(30–40 °C)
Chemical						
Chemical	Evoterm Technologies	Mead Westvaco	Chemical packages, with or without water	0.5% of mass of bitumen emulsion. Emulsion contains 70% of bitumen	USA, France, Worldwide	85–115 °C
Chemical	Cecabase RT	CECA	Chemical package	0.2–0.4% by mixture weight	USA, France	(30 °C)
Chemical	Rediset	Akzo Nobel	Cationic surfactants and organic additive	1.5–2% of bitumen weight	USA, Norway	(30 °C)
Chemical	Revix	Mathy-Ergon	Surface-active agents, waxes, processing aids, polymers	Not specified	USA	(15–25 °C)
Chemical	Iterlow T	IterChimica		0.3–0.5% by mass of bitumen	Italy	120 °C

Figure 7.3 Different WMA products available on the market (Rubio et al., 2012)

7.2.1 Chemical additives

Many and different WMA additives can be considered as chemical additives: usually they are formed by surfactants, emulsification agents, aggregate coating enhancers and anti-stripping additives. Such additives are able to improve ability of bitumen to coat aggregate particles by regulating and reducing the slip forces at interface rather than reducing bitumen viscosity (Button et al., 2007).

Chemical additives usually need to be mixed with the bitumen before batching the bitumen into the asphalt mixer, although there are also techniques in which the package of products is used by means of a bituminous emulsion.

Rediset™ WMX and Cecabase® RT are both chemical additives formed by surfactant and adhesion agents, among other components. Those types of products chemically enhance active adhesion and improve the wetting of aggregates by bitumen without changing considerably the bitumen performance, such as viscosity (Capitão et al., 2012).

An additional chemical technology commercially called Evotherm™ is a typical case wherein a package of additives is used in a form of emulsion (Hurley and Prowell, 2006; Bonaquist, 2011). Since the aggregates are heated before mixing, the water within the emulsion vaporizes during the production process and the bitumen covers the aggregate particles. Meanwhile, the initial technology has evolved: firstly, to a process in which chemical additives are introduced into the plant's bitumen line (Evotherm Dispersed Additive Technology), incorporating much less water than with emulsion; recently, to a third generation process (Evotherm 3G), which is a water-free WMA technique, where the additive is incorporated into bitumen before its deliver to asphalt plants (Bonaquist, 2011).
As stated in the bibliography, the use of chemical additives allows a reduction of the mixing and compaction temperatures up to 30°C (EAPA, 2010; Silva et al., 2010).

7.2.2 Organic additives

WMA technologies based on organic additives consist on adding organic waxes to the asphalt mixture. Such a wax is a high molecular hydrocarbon chains with a melting point of 80 - 120 °C which is able to significantly modify the properties of the original bitumen. At temperatures above the melting point of the waxes, in fact, there is a decrease in bitumen viscosity (Zaumanis, 2010), allowing the mixing and the lay-down phases to be completed at reduced temperatures. As the mixture cools, these organic additives solidify into microscopically small and uniformly distributed particles, which increase the stiffness of the bitumen similarly to fiber-reinforced materials. Hence, waxes within bitumen should be resistant and solid at service temperatures leading to asphalt pavement more resistant to permanent deformation.
Obviously, the type of wax must be carefully selected in order to avoid possible temperature problems (Silva et al., 2010): if the melting point of the wax is lower than in-service temperatures, this can lead to complications. Moreover, a proper selection of the wax minimizes the embrittlement of the asphalt at low temperatures (Shang et al., 2011).
The use of organic additives usually allows a temperature reduction up to 20-30 °C.
Among organic waxes, three different categories which differ in the type of wax used to reduce viscosity can be identified: Fischer-Tropsch wax, fatty acid amide, and Montan wax.

Fischer-Tropsch wax

Fischer-Tropsch (FT) wax is a pure hydrocarbon without functional groups, and is characterized by high chemical stability and ageing resistance. Although it melts at approximately 100 °C in its pure state, when it is blended with bitumen, its melting point is lower (80-85 °C), which allows asphalt compaction at less than 100 °C. As it cools, crystallization begins at 105 °C and is completed at 65 °C, thus forming regularly distributed, microscopic, stick-shaped particles. Research has shown that these waxes have good oxidation and ageing stability, and can be stored indefinitely (Hurley and Prowell, 2005).

Fatty acid amide

Amide waxes are products synthetically manufactured in order to cause amines to react with fatty acids. They melt at temperatures of 140-145 °C, whereas solidification takes place at 135-145 °C. As the fatty acid amides cool, they form crystallites in the bitumen, thus increasing asphalt stability and deformation resistance (D'Angelo et al., 2008).
Synthetic fatty acid amides are available on the market under different trade names.

Montan wax

Montan wax is extracted from special waxy lignite. In chemical terms, Montan wax consists mainly of fossil fatty acid esters. Since the melting point of this wax in its pure state is approximately 75 °C, it is often blended with materials with a higher melting temperature such as amide waxes. Montan waxes can be fed directly into the mixer, which requires additional mixing time or into the mobile stirrer for mastic asphalt.

7.2.3 Foaming processes

Foaming process includes several methods that entail the addition of small amounts of water, either injected into the hot bitumen or directly into the mixing chamber (Larsen, 2001). When the water is mixed with the hot bitumen, high temperatures cause the water to evaporate and the steam remains entrapped within the bitumen. This generates a large volume of foam, which temporarily increases the volume of the bitumen reducing its viscosity which leads to a significant reduction of mixture viscosity. This effect remarkably improves the coating and workability of the mixture, but its duration is limited: this means that the material must be produced and compacted soon after production.
Special attention must be taken on the dosage of water: the quantity of water should be just enough to produce the foaming effect, but not so much as to cause a stripping problem (Smith, 2007).
Although the basic process is the same for most of the products and technologies, the way in which water is added to the bitumen can vary. Thus, the foaming processes can be divided in water-based (direct method technologies) or water-containing (indirect method technologies) (Zaumanis, 2010).

Water-based technologies

Water-based technologies use directly the water to produce the foaming effect: the water is injected into the hot bitumen flow with special nozzles (Figure 7.4). As the water rapidly evaporates, this produces a large volume of foam that slowly collapses.

This category can be subdivided into the types of product used to make the mix (Zaumanis, 2010):

- Double Barrel Green, Ultrafoam GX, LT Asphalt: Although the equipment for injecting water into the hot bitumen differs (since each company makes its own equipment), the basic principle is the same. Several nozzles are used to inject the cold water to microscopically foam the bitumen

- WAM Foam: it is a two-component bitumen system that feeds a soft bitumen and a hard foamed bitumen at different times into the mixing cycle during production. The soft bitumen is first mixed with the aggregates to allow pre-coating. Then the hard bitumen is added to the mixture, which has been foamed by the previous injection of cold water in a quantity ranging from 2% to 5% of the mass of the hard bitumen. This combination of soft bitumen and foaming of the hard bitumen reduces mixture viscosity to provide the necessary workability.

There are other technologies that use water differently, and thus do not fall into either category. One example is Low Energy Asphalt, which uses wet fine aggregate: the foaming effect is produced at contact with the hot bitumen when aggregate of a certain particle size is partially dried.

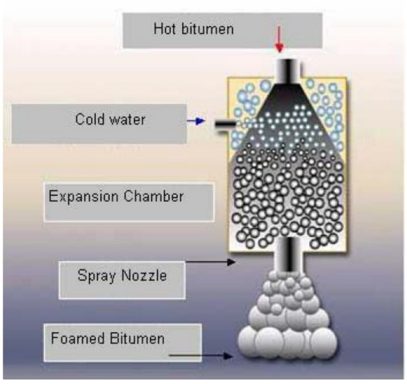


Figure 7.4 Foaming process due to water-based technologies (EAPA, 2010)

Water-containing technologies

Water-containing technologies use synthetic zeolite to produce the foaming process. The product is composed of alumino-silicates of alkali metals that has been hydro-thermally crystallized. The structure of the zeolites has large air voids where water can be hosted (Figure 7.5). Their ability to lose and absorb water without damaging the crystalline structure is the main characteristic of this silicate framework (Chowdhury and Button, 2008).

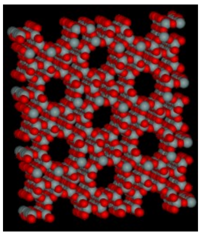


Figure 7.5 Structure of zeolite additives

In the specific, zeolite usually contains approximately 21% water by mass; during the production process, water is released and vaporizes in contact with hot bitumen. The steam remains encapsulated within bitumen leading to a temporary expansion of bitumen volume with a reduction of its viscosity. Such a micro-foaming effect in the asphalt mixture lasts about 6-7 h (Chowdhury and Button, 2008; D'Angelo et al., 2008).

7.3. Warm mixtures performance

Warm mixtures have to guarantee the same performance as hot produced mixtures in order to be applied for the production at reduce temperatures of asphalt mixtures. Thus, the same standard and quality control procedures usually applied for HMA mixtures, both in-plant and laboratory produced mixtures, have to be taken into account also for WMA mixtures: volumetric properties, stiffness, rutting resistance, fatigue resistance and water susceptibility must be checked before application.

Special attention must be taken for laboratory produced WMA mixtures since it has been demonstrated that mixture performance can by highly influenced by the production procedure. In particular, the water-based technology is the most difficult to closely reproduce in laboratory since it requires specific equipment. In order to avoid such a issue, it is recommended to prefer studies involving WMA mixtures that were actually produced in the asphalt plant for a reliable characterization of short and long term performance.

Moreover, since the use of WMA additives is relatively recent, concerns still exist related to in-service and long term performance.

In addition, adhesive failures at the bitumen-aggregates interface and water susceptibility represent two of the major concerns since the aggregates drying and their coating with bitumen can results insufficient due to the reduced mixing temperatures. Moreover, the rutting resistance of WMA mixtures can be affected since they undergo a less oxidative hardening during the production process (Capitão et al., 2012; Kheradmand et al., 2014; Rubio et al., 2012).

In this sense, WMA mixtures need to be investigated in order to evaluate the most appropriate WMA additive and their feasibility, in particular when recycled mixtures are

involved due to the presence of aged bitumen within recycled aggregates. In case of porous asphalt mixtures, one of the major concerns is the low durability due to high air void content (≈20%) that makes PA more susceptible to ravelling and water damage (Alvarez, Martin and Estakhri, 2011; Partl, Pasquini, Canestrari and Virgili, 2010; Poulikakos & Partl, 2012). In particular, ravelling is strongly related to the adhesive properties between bitumen/mastic and aggregates that are in turn strongly influenced by the effect of water and aging (Hagos, Molenaar, Van de Ven and Voskuilen, 2007) and can be easily be affected by the reduced production temperatures. Thus, specific research studies are needed in order to assess the feasibility of WMA technologies for PA mixtures.
In the next paragraph, the major issues related to the use of reduced production temperatures due to the use of WMA additives are detailed described.

7.3.1 Mix design of WMA mixtures

Mix design of asphalt mixtures involved several aspects such as materials selection, bitumen content and volumetric properties. The use of a correct mix design is essential for ensuring adequate performance of the final mixture. Based on the scientific literature, in most cases the optimization of warm mixtures uses the same procedure commonly employed for HMA mixture such as Marshall method in Europe and Superpave method in the USA.
One parameter that needs to be carefully considered in case of warm mixture is the additive dosage to be incorporated in the mixtures as well as the operating system (prior addition to the bitumen or directly addition with the mixtures). Moreover, an additional factor that should be considered during the mix design is the selection of the proper temperature for mixing and compaction that should guarantee proper workability of the mixture. Since some WMA additives tend to vary the bitumen viscosity to allow the production at low temperature, several studies demonstrated that the equi-viscous principle for the determination of the mixing and compaction temperatures is not fully applicable for WMA mixtures (Bonaquist, 2011; Silva et al., 2010). On the other hand, it is preferable to estimate the progressive variation of density and volumetric properties as a function of temperature. In this sense, preliminary studies are required to evaluate such a trend by preparing the mixture at different temperatures.

7.3.2 Water sensitivity

As already mentioned, one the major issue of asphalt pavement is the performance reduction over time due to the presence of water, especially when surface layer (such as porous mixtures) are involved. Such a reduction is due to the lack of adhesion at the bitumen/aggregates interface. This aspect can be amplified by the production at reduced temperatures due to an incomplete drying of aggregates during the mixing phase: some

water can remain entrapped within the mixtures leading to a quick deterioration of the mixture. Hence, it appears clear how important is the stocking process of the aggregates, especially the fine gradation in order to reduce the water content allowing a complete dying even at warm production temperatures.
Hence, in case of warm mixtures, the water sensitivity is a parameter that needs special consideration, especially in case of PA mixtures.
Several research studies showed that the loss of Indirect Tensile strength due to water conditioning tends to reduce as the compaction temperature increase; generally, the performance of warm mixtures in terms of water susceptibility decreases in 79% of the case with respect of similar HMA mixtures (Bonaquist , 2011; Sanchez-Alonso et al., 2011).
Other researchers suggested to enhance the WMA mixture resistance to moisture by adding anti-stripping agents that are characterized by a specific polarity which help the coating of the aggregates by the bitumen limiting the negative effect of water (Bonaquist 2011; Kavussi and Hashemian, 2011). The use of anti-striping agents is particularly useful in case of PA mixtures where the water sensitivity is more severe than dense-graded mixtures. In this matter, Hamzah et al. (2011) have conducted a study on PA mixtures prepared with an organic WMA additive (Sasobit®) and two different types of anti-stripping agents (hydrated lime and PMD- Pavement modifier used as filler) with the objective to evaluate the raveling resistance and water sensitivity. Results showed that the raveling resistance, measured by means of Cantabro tests, tends to decrease as the production temperatures decrease; such an effect seemed to be limited by the presence of PMD. Moreover, mixtures prepared with PMD showed a higher water resistance with respect to those prepared with hydrated lime.

7.3.3 Mixtures stiffness

In terms of mixture stiffness, it is generally possible to affirm that reduction of production temperatures leads to a decrement of stiffness properties of warm mixtures with respect to hot mixtures (Bennert et al., 2011; Cardone et al., 2009; Sanchez-Alonso et al., 2011). Such a reduction in terms of stiffness is dependent on WMA additive types as well as the compaction mode and production temperatures adopted (Zaumanis, 2010).
The reduction in stiffness characteristics results more significant at high service temperatures (>45°C) and less important at low temperatures (4°C) due to the worst aggregate coating by the bitumen that occurs in WMA mixtures for the reduced production temperatures (Bennert et al., 2011). This leads to a less adhesion between coarse aggregates that, in turn, causes less resistant mixtures (Cardone et al., 2009).
However, several studies (Akisetty, 2008; Bennert et al., 2011; Sanchez-Alonso et al., 2011) demonstrated that the use of organic WMA additives helps to partially compensate the stiffness reduction, especially at low service temperatures (4-20°C) when mixture production temperatures between 100°C and 140°C are selected.
An additional aspect to be considered for a proper evaluation of warm mixtures stiffness properties is the selection of virgin bitumen. Jenkins (2011) demonstrated that WMA mixtures prepared with unmodified bitumen suffer a stiffness reduction, at standard

condition (25°C and 10Hz) between 40% and 60% with respect to a reference hot mixture. On the contrary, similar performance between WMA and HMA in terms of mixtures stiffness had been measured for mixtures prepared with polymer modified bitumen, if the same modification type, granulometric composition as well as test condition have been adopted. Such a result was demonstrated using both elastomer and plastomer and allows to conclude that the use of polymer modified bitumen in WMA mixtures helps to compensate possible performance reduction due to the use of low production temperatures.

7.3.4 Fatigue resistance

It is well know that different methods as well as several data interpretation systems exist to evaluate mixture performance in terms of fatigue. The scientific literature (Diefenderfer and Hearon, 2008; Jenkins, 2000) agrees that, despite the test method adopted, WMA mixtures are characterized by low fatigue resistance than traditional HMA mixture, especially when small deformation level are selected. Nevertheless, it seems that WMA mixtures are less sensitive to increments of the deformation rate with respect to HMA mixture and thus, they are preferable in heavy traffic roads (Diefenderfer and Hearon, 2008; Jenkins, 2000; Petit et al., 1993). Moreover, even if a performance reduction of 20-25% with respect of HMA mixtures is demonstrated, the fatigue resistance of WMA mixtures can be considered satisfying (i.e. deformation after 10^6 load cycles > 200 μm/m) in case of both laboratory prepared specimens and cores from in-service pavement (the variation between the two cases is less than 10%). In this sense, it is important to underline that the low aging rate that warm mixtures undergo during the production phase can help to improve flexibility of the pavement allowing an overall improvement in fatigue performance (Perkins, 2009).
Additional evaluations had been carried out on WMA mixtures including RAP and modified bitumen (Jenkins, 2011; David, 2011). Results demonstrated that the combined use of foaming techniques and RAP do not affect the indirect tensile deformation of materials, especially at high service temperatures. However, some results outside the trend suggest that a special attention is needed on the evaluation of repeated loads resistance, especially when recycled warm mixtures are considered. Finally, some research studies focused in the evaluation of organic WMA additives and zeolite demonstrated that the use of Sasobit® can slightly improve fatigue performance (Petit, 2012) whereas the use of zeolite seems to penalize the resistance to repeated traffic loads (Xiao et al., 2009b).

7.3.5 Resistance to thermal cracking

Resistance to thermal cracking represents a significant issue especially form asphalt pavements in cold climate regions. Such a phenomenon is more severe when low adhesion between bitumen and aggregate occurs; thus, it must be considered in case of warm mixtures where weak adhesion at the bitumen/aggregate interface could potentially occurs

due to the reduced production temperatures. However, several research studies (Min-Yong, 2011; Das, 2012) suggested that warm mixtures are able to guarantee performance similar to HMA mixtures at low service temperatures (i.e. 0, -10 e -20 °C).
The worst results in terms of thermal cracking resistance were obtained in case of warm mixtures prepared with organic WMA additives that tend to increase the mixture stiffness; at service temperatures, in fact, the crystallization of the organic additives could lead to excessive embrittlement of WMA mixtures.

7.3.6 Rutting performance

Mixtures evaluation in terms of rutting resistance allows understanding pavement behavior at high service temperatures. Based on past researches (Bennert, 2011) it is possible to affirm that rutting resistance tends to decrease as the production temperatures decrease, even if such variation depends on the WMA technology adopted as well as the amount of WMA additives used. Therefore, warm mixtures are characterized by low resistance to permanent deformation with respect to similar HMA mixtures, especially when foaming techniques are used. However, rutting performance of WMA mixtures can be improve using organic additives, such as Sasobit®; below its melting point, in fact, organic additives form a crystalline network structure in the bitumen that increases bitumen stiffness as well as mixture resistance to permanent deformation (Kristjansdottir, 2006; Silva et al., 2010b).
Additional studies demonstrated that rutting resistance of WMA mixtures tends to decrease when water conditioning of specimens is taken into account (Bonaquist 2011).

7.4. Use of RAP in warm mixtures

The use of milled materials as partial substitution of virgin aggregates in new produced in Hot Mix Asphalt mixtures is a widely recognized solutions for economic as well as environment benefits (Al-Qadi et al., 2007). However, it is noteworthy that the use of RAP requires higher production temperatures due to the presence of aged bitumen within recycled mixtures. The aged bitumen from RAP, in fact, results stiffer than similar virgin bitumen since it underwent short and long term aging processes and that could lead to difficulties during the laying down phase. Thus, recycled mixtures requires high production temperatures (180°C or higher) for achieving the proper compactability characteristics and such temperatures tend to increase as the percentage of RAP increases. The use of high mixing and compaction temperatures implies both environmental and health problems due to hazardous emissions as well as harmful gases and, at the same time, requires the consumption of higher amount of fuel that leads to an increment of the production costs. Such an issue is becoming more and more relevant since most of the production sites are located near urban centers leading to strict environmental regulations.
In order to avoid this problem, the use of WMA additives to reduce mixture production temperatures can provide a valid alternative able to ensure both environmental and economic benefits as well as to guarantee pavement in-service performance (Zhao et al.,

2013). Given this background, experimental research focused on the use of reduced production temperatures by means of WMA additives for recycled mixtures are needed in order to provide proper answers to different aspects. In the present paragraph, the main international scientific studies on the matter are analyzed with the aim at highlighting possible issues and the relative solutions to guarantee proper performance of the final mixtures.

Since the use of RAP requires high production temperatures, the combined use of WMA additives and RAP helps to guarantee good compactability standards without the use of higher production temperatures. In particular (D'Angelo et al., 2008), the effect of using WMA technology in recycled mixtures including high percentage of RAP is comparable to the effect of using virgin bitumen characterized by a lower Performance grade (one or two points lower). Moreover, it is noteworthy that the use of reduced produced temperatures leads to a lower short-term aging rate of the bitumen with consequent benefits for the final mixtures, especially in case of recycled mixtures since it could help to compensate the effect of the aged bitumen from RAP. Several studies demonstrated the possibility to use very high content of RAP (up to 90-100%) and still assuring proper workability as well as reduced compactability problems (D'Angelo et al., 2008; Mallick et al., 2008; Shu et al., 2012b; Tao and Mallick, 2009). At the same it needs to be taken into account that the use of low production temperatures could alter the degree of reactivation of the aged bitumen within RAP. The use of hot recycling techniques, in fact, ensures the reactivation of a certain amount of bitumen from RAP due to the high temperatures used during the mixing phase; the reactivated bitumen from RAP blends with the virgin bitumen and it is taken into account for the evaluation of the optimum bitumen content during the mix design. Thus, in case of warm mixtures, a reduced reactivation degree of the aged bitumen from RAP must be considered in the mix design as well as in the evaluation of the final mixture performance. An overvaluation of such a parameter could lead to a final mixture with insufficient bitumen content that is not able to provide proper volumetric characteristics and adhesion properties. Based on recent studies (Bonaquist, 2011), it seems that the blending between aged and virgin bitumen could occur also in case of WMA mixtures if the mixing temperatures are maintained for a sufficient amount of time. In particular, laboratory studies showed that two hours of conditioning at the compaction temperature should be sufficient to guarantee a reactivation degree of the bitumen from RAP comparable to the one of an equivalent HMA mixture. However, it is recommended to use slightly higher production temperatures in case of recycled mixtures compared to virgin mixtures in order to ensure a more efficient blending process between virgin and aged bitumen. Such a solution also helps to dry the aggregates and control the humidity within WMA mixtures.

Furthermore, rutting performance needs also to be considered in case of warm recycled mixtures. Warm mixtures, in fact, tend to rut easily than HMA mixtures due to the lower short term aging of the bitumen. In this sense, the use of RAP could help to compensate the tendency to rut due to the presence of aged bitumen that stiff the final mixtures.

Mogawer et al (2009) focused on recycled mixtures produced using WMA additives Advera® e Sasobit® and evaluated their performance in terms of bitumen properties, workability and durability of mixtures. Results showed that adding Sasobit® lead to variations on the bitumen Performance Grade and to a viscosity reduction of final bitumen at high temperatures whereas Advera® did not implicate any modification of bitumen components. All warm mixtures demonstrated better workability than the HMA mixture and higher water sensitivity. In this sense, Authors suggest the use of anti-stripping agents along with WMA additives in order to avoid or delay the stripping phenomenon due to the action of water.

Other research studies (Copeland et al., 2010) evaluated performance of recycled mixtures with high content of RAP (up to 75%) by comparing the results obtained from hot mixtures and similar warm mixtures produced using foaming techniques or adding Sasobit®. Tests for the bitumen PG determination as well as for evaluation of the resistance to permanent deformation showed that the use of foaming techniques leads to soft mixtures that tends to rut easily, even if no significant differences in terms of stiffness was detected.

Moreover, it was demonstrated (Mallick et al., 2008) that the use of Sasobit® dosed at 1.5% (by virgin bitumen weight) at a temperature of 135°C significantly helps to realize a uniform mixture with performance similar to a reference hot mixture. In addition, the use of Sasobit® leads to an increase in Indirect tensile strength due to the presence of wax that tends to stiff the mixture at intermediate and low service temperatures.

Nevertheless, it is important to underline that many studies (Lee et al., 2009; Kim et al., 2011a; Kim et al., 2011b) suggests the use of a virgin bitumen with lower PG grade in case of the combined use of RAP and Sasobit® to compensate the stiffening effect of the organic additive and the aged bitumen from RAP.

A research experimental study conducted by Zhao et al. (2013) showed that mixtures produced with WMA additives and RAP can perform better than standard hot mixtures in terms of rutting despite of the amount of RAP used. An increase in RAP content leads to an improvement in performance both in case of HMA and WMA mixtures but such an increment is more significant in case of HMA mixtures; such a difference in performance is not influenced by the type of WMA technology.

Encouraged results related to the combined use of WMA additives and RAP were obtained also in case of open-graded mixtures. In particular, a study conducted by Goh and You (2012) focused on the performance comparison between HMA and WMA recycled porous mixtures produced with Advera®. Results showed a decrease in mixtures stiffness due to the use of reduced production temperatures in case of virgin WMA mixtures whereas such an effect was not detected for recycled WMA ones. Thus, the presence of RAP helps to compensate the reduction in terms of mixture stiffness that could affect warm produced mixtures.

One of the first field experimentation able to combined recycling and WMA techniques was realized by the Washington Department of Transportation (2009). An USA motorway pavement (18 km) was build using a warm recycled mixture including 20% RAP and 2.0% (by virgin bitumen weight) of Sasobit®; the selected virgin bitumen was characterized by a PG 76-28. Test results did not shoe any particular issues for warm mixtures that were able to perform similarly or even better that the reference hot mixture.

Water sensitivity of warm recycled mixtures was investigated by Shu et al. (2012b) and by Zhao et al. (2013) on mixtures including several RAP percentages up to 50%. Results did not show significant differences in terms of water resistance between virgin and recycled mixtures; moreover, better rutting as well as fatigue performance were demonstrated by the recycled warm mixtures.

7.5. WMA technologies and modified bitumen

The use of polymer modified bitumen for the production of asphalt mixtures is common all over the world. In general, polymer modified mixtures (both elastomer and plastomer) are characterized by higher resistance to rutting, fatigue and thermal cracking as well as a reduced tendency to aggregates stripping (Jamshidi et al., 2013; Yildirim, 2007). At the same time it must be taken into account that the use of polymer modified bitumen requires higher production temperatures to achieve a proper workability and that, in turn, leads to higher energy consumption and production costs as well as higher release of pollutant emissions. Hence, the possibility to reduced production temperatures of polymer modified asphalt mixtures by means of WMA additives is very interesting and needs to be properly analyzed.

A recent study (Kim et al., 2010) investigated the effects of adding two different WMA additives (Sasobit® and Asphamin®) on the rheology characteristics of polymer modified bitumen. Results showed that the use of both types of WMA additives allows the use of bitumen characterized by higher PG superior temperature that implies a higher rutting resistance. On the contrary, it tends to decrease the fatigue resistance at intermediate in-service temperatures. Moreover, the presence of WMA additives leads to bitumen stiffening at low in-service temperatures that could implicate more cracking.

Furthermore, during the same research project (Kim et al., 2010), additional modified asphalt mixtures by means of SBS polymers were produced using the same WMA additives at a temperature 10°C lower than HMA. Results showed that performance in terms of Indirect Tensile strength and water sensitivity of warm mixtures fully satisfy acceptance requirements of HMAs and no significant difference in performance between WMA and HMA was detected. As far as rutting resistance is concerned, all the modified mixtures (both HMA and WMA) showed performance higher than the standards minimum and the mixtures produced with Sasobit® were characterized by the highest rutting resistance.

It is interesting to underline that all the performance analyzed such as resilient modulus, water damage and rutting resistance were highly dependent from the type of aggregates. Moreover, the study was also conducted on artificial long-term aged specimens and results did not show significant differences between WMA and HMA mixture performance. Thus, the study confirmed that the use of WMA additives in asphalt mixtures including polymer modified bitumen does not lead to negative effect in terms of mixture performance.

An additional aspect that needs to be taken into account in case of modified bitumen is the affinity between polymer and bitumen that could significantly affect the proper dispersion of the polymer phase within the bitumen and the storage stability. In this sense, the combined use of WMA technologies and polymer modified bitumen could results beneficial for the final mixture; in particular, several studies showed that the use of

Sasobit® improves the storage stability of polymer modified bitumen (Ho et al., 2008; Edwards et al., 2012).

Chapter 8.

Preliminary study on bituminous phase and adhesive properties

8.1. Introduction

It has been well established that the rheological properties of the bitumen substantially affect the asphalt pavement performance (Brown et al., 2009). Since bitumens for road paving applications experience a variety of thermo-mechanical states during their service life, it results extremely important to investigate their rheological properties under different temperature and loading conditions. Most pavement distresses, such as rutting at high temperatures and crack initiation and propagation at low temperatures, can be attributed not only to traffic loads but also to the thermal susceptibility of asphalt bitumens (Pérez-Lepe et al., 2003). In order to improve asphalt mixture performance, the bitumen properties are often enhanced by means of polymer modification. Polymers are traditionally used to decrease the temperature susceptibility of the bitumen by increasing its stiffness at high service temperatures as well as reducing its stiffness at low service temperatures (Airey, 2002; Collins et al., 2008). This leads to enhanced pavements having higher resistance to rutting and thermal cracking and lower fatigue damage, stripping and thermal susceptibility.

Given this background, it is essential to properly analyze the effect of WMA additives on the properties of bitumens, especially when polymer modified bitumens are involved. Hence, mechanical behavior in terms of stiffness, rutting and fatigue resistance needs to be evaluated in case of warm produced bitumens including difference WMA additives. In particular, the mixing and compaction temperatures of mixtures are closely related to the viscosity properties of the bituminous and mastic component that should be properly investigated in order to select the warm production of mixtures.

Furthermore, it needs to be taken into account that one of the major promising “green” techniques employed for the production of bituminous mixtures is given by the combination of warm mix technologies and recycling. Warm Mix Asphalt (WMA) mixtures are produced at temperatures approximately 25-30°C less than traditional Hot Mix Asphalt (HMA) mixtures thank to physico-chemical bitumen composition changes during the mixing process (D’Angelo et al. 2008). Reclaimed Asphalt Pavement (RAP) is the primary product coming from the milling of old asphalt pavements. Its adding generally causes stiffer and more brittle mixtures due to the presence of aged bitumen and, for this reason, the total amount of RAP has to be kept lower than a maximum limit. The combination of the two techniques (use of WMA additives for producing mixtures containing RAP at reduced temperatures) guarantees a lower aging rate of the virgin bitumen, allowing the incorporation of higher amounts of RAP in warm mixtures without any need to select softer virgin bitumen grade (Prowell & Hurley 2007). As a consequence, it should be taken into account that most of the new produced warm asphalt mixtures include RAP.

Therefore, further investigations are needed to efficiently exploit possible advantages related to the combination of the abovementioned techniques as well as to optimize the mix design of recycled WMA mixtures. In particular, due to the lack of long-term performance data from the field, laboratory performance tests continue to fulfill a fundamental role in the design and deployment of existing and innovative WMA technologies.

Warm recycled mixture performance are strictly related to a number of factors (e.g. type of warm technology, bitumen, production temperatures, environmental conditions, RAP amount).

Among various parameters, the adhesion at the bitumen-aggregates interface represents one of the most critical aspects associated to the failure of warm mixtures. In fact, reduced production temperatures could cause poor bond strength due to incomplete and not homogeneous coating of aggregates by bitumen as well as a not proper drying process of wet aggregate particles. Low adhesion properties mean higher water susceptibility which in turn implies stripping of bitumen from the aggregate surface or cohesive failure within the bitumen leading to a rapid deterioration of overall pavement performance and durability.

Among other possible advantages (decrease in material costs, energy savings, higher rutting resistance) (Canestrari et al. 2014), the use of reclaimed material could be helpful to partially reduce the detrimental effect of water since RAP aggregates can be seen as a water-resistant material due to the presence of the thin film of aged bitumen. Nonetheless, two aspects related to RAP could counteract and compromise the potential benefits. First, milled material stockpiles tend to retain high moisture (often higher than virgin aggregates). Since usually RAP aggregates are not pre-heated (or pre-heated at very low temperature) prior to being mixed with the other material components, great amount of undesired water remains entrapped during mixing, negatively affecting bitumen-aggregate interactions. Second, the reduced production temperatures can alter the degree of reactivation of RAP bitumen with consequences on the total bitumen amount. The effective "working" bitumen is lower than what expected for traditional HMAs prepared at standard high temperatures with same mix design and equal amount of RAP. Without taking into account this aspect, the resulting overestimation of the effective "working" bitumen leads to lower aggregate coating and so higher risk of water damage mostly resulting in raveling and stripping. This explains the reason that often leads to the adding of more anti-stripping agents in WMA mixtures.

Several studies have addressed the evaluation of water susceptibility of recycled WMA mixtures (Mallick et al. 2008, Zhao et al. 2012), but analyses were usually based on mechanical test results or analytical methods, rather than conducting direct measurements of the interactions between bitumen and aggregates. This latter aspect is directly linked to

the adhesion properties that represent a key point to properly identify the best materials combination and to consciously design mixtures with enhanced moisture resistance.
Given this background, this chapter is divided in two major sections. The first one (Section 8.2) analyzes the bitumen and mastic mechanical characteristics in order to evaluate the possible consequences related to the use of WMA bitumen. In order to take into account the warm production, both virgin and short-term aged bitumens were investigated.
The second part (Section 8.3) involves the evaluation of the adhesion properties between aggregates and bitumen when WMA additives as well as RAP materials are involved. The experimental research faces the problem with a double analysis aimed at evaluating the interaction between the component materials (bitumen and aggregates), measured in terms of fundamental adhesion properties and the water susceptibility of the corresponding asphalt mixtures.

8.2. Rheological analysis of WMA bitumens

This section describes the rheological analysis that was carried out on WMA bitumens. Viscosity characteristics as well as mechanical properties were evaluated by means of a Rotational Viscometer (RV) and a Dynamic Shear Rheometer (DSR).

8.2.1 Materials

Materials involved in this experimental section were produced and aged in the laboratory with the aim at simulating as closely as possible the real in-field conditions. The same polymer modified (SBS) bitumen that was used throughout this entire research project (already described in section 4) was selected as reference bitumen (hereafter named "HMA"). Moreover, it was employed for the production of three WMA bitumens by adding three different WMA additives, selected as representative of the three main categories nowadays available on the market:
- WMA-O: it was produced by including 3.0% of a WMA organic additive to the hot bitumen and mixing for 15 minutes;
- WMA-C: it was produced by including 0.5% of a WMA chemical additive to the hot bitumen and mixing for 15 minutes;
- WMA-Z: it was produced by including 6.0% of a WMA zeolite to the hot bitumen allowing the temporarily foaming process to occur.
Unaged as well as short term aged bitumens were tested in order to simulate all the different stages that pavements undergo during the construction; short term aging conditions, in fact, simulate the bitumen mixed with the aggregates and compacted and could lead to different results in case of HMA and WMA due to the different production temperature used. It is well known, in fact, that mixing and compaction temperatures play a fundamental role for the aging process during the production phase. On the other hand, as

far as long term conditions are concerned, no significant differences in bitumen behavior are expected due to the reduced temperatures used for WMA production. Thus, such an aspect was not analyzed in terms of bitumen rheology but it will be further taken into consideration in terms of long term mixtures performance and detailed described in Chapter 9 – Section 3.
As already mentioned, HMA and WMA bitumens undergo different short term aging conditions; thus, the HMA bitumen was short term aged at 163°C whereas the WMAs bitumen were short term aged at 120°C, in order to simulate as closely as possible the real mixing and laydown phases that bitumens undergo during production.
As far as viscosity evaluation is concerned, also mastics were produced in order to analyze and compare viscosity properties when mineral filler is involved. Thus, each bitumen was used to produce mastic using a standard bitumen/filler ratio equal to 1 and selecting the same limestone filler for all the mastics.
The overall experimental program is summarized in Table 8.1.

Table 8.1 Rheological analysis: experimental program

Bitumens	**WMA additives**	**Viscosity**	**Master curve**	**MSCR test**	
				Virgin	Short-term aged
HMA	-	X	X	X	
WMA-C	Chemical	X	X	X	
WMA-O	Organic	X	X	X	
WMA-Z	Zeolite	X	X	X	
Mastics	**WMA additives**				
HMA	-	X			
WMA-C	Chemical	X			
WMA-O	Organic	X			
WMA-Z	Zeolite	X			

8.2.3 Experimental investigation

Short-term aging procedure

The test method based on the Rolling Thin Film Oven Test (RTFOT) was used in this study (Figure 8.1) to reproduce the short-term aging undergone by the bitumen during the mixing, transport and compaction phases. The test procedure was applied according to the European standard EN 12607-1. The RTFOT was originally developed by the California Highway Department to simulate aging that occurs in asphalt plants during the manufacture of hot asphalt mixtures. This test can be used for two main goals: providing aged bitumen to be used for further testing of physical properties and determining the mass quantity of volatiles

lost from the bitumen during the test. The amount of volatile loss is an indication of the amount of aging that may occur in the bitumen during mixing and construction phases.
The RTFOT procedure requires an electrical heated convection oven that contains a vertical circular carriage to accommodate eight sample bottles. The oven is also equipped with an air jet that blows air in each bottle while the carriage circulates.

Figure 8.1 RTFOT equipment

The basic RTFO procedure takes unaged asphalt bitumen samples in cylindrical glass bottles and places these bottles (Figure 8.2) in a rotating carriage within an oven. The carriage rotates within the oven while the 325°F (163°C) temperature ages the samples for 85 minutes.
The RTFO aging procedure is used to simulate aging during mixing and placement, while the PAV aging procedure is used to simulate aging during in-service life. Therefore, asphalt bitumen tests concerned with mix and placement properties are conducted on RTFO aged samples, while asphalt bitumen tests concerned with in-service are performed on samples first aged in the RTFO and then in the PAV.

Figure 8.2 RTFOT glass bottles

The basic procedure is described below:
- heat a sample of asphalt bitumen until it is fluid to pour. Stir sample to ensure homogeneity and remove air bubbles.
- if a determination of mass change is desired, label two RTFO bottles and weigh them empty. These are designated as the “mass change” bottles. Record the weights.
- pour 35 g of asphalt bitumen into each bottle (Figure 8.2). Immediately after pouring each bottle, turn the bottles on their side without rotating or twisting and place them on a cooling rack.
- allow all bottles to cool 60 to 180 minutes.
- after cooling, weigh the two mass change bottles again. Record the weights.
- place the bottles in the RTFO oven carousel, close the door, and rotate carousel at 15 RPM for 85 minutes. During this time, maintain the oven temperature at 163°C and the airflow into the bottles at 4000 ml/min
- remove the bottles one at a time from the carousel, setting the mass change bottles aside. Residue from the remaining bottles should be transferred to a single container.
- remove residue from each bottle by first pouring as much material as possible, then scraping the sides of the bottle to remove any remaining residue. There is no standard scraping utensil but at least 90 percent of the asphalt binder should be removed from the bottle.
- RTFO residue should be tested within 72 hours of aging.

The test temperature is fixed at 163°C by the standard in order to simulate the mixing procedure of HMA mixtures; hence, in the present study, the temperature of 163°C was used for the HMA bitumen in accordance with the recommendation whereas a reduced temperature equal to 120°C was selected for the WMAs bitumen is order to simulate the warm production procedure.

Viscosity evaluation: Rotational viscometer

As previously mentioned, the viscosity can be evaluated as the resistance opposed by a fluid to an object that is rotated at a constant speed within the fluid. Based on this definition, one of the most common system to measure the viscosity is based on the rotational coaxial cylinder viscometer like that specified by the European standard EN 13302, used in this study to carry out viscosity measurements. In this research, the Brookfield rotational viscometer was employed (Figure 8.3).

Figure 8.3 Rotational viscometer Brookfield (left); spindle SC21 (right)

The rotational viscosity is determined by measuring the torque required to maintain a constant angular velocity of a cylindrical spindle while submerged in a sample at a constant temperature (Figure 8.4). The torque required to rotate the spindle at a constant speed is directly related to the viscosity of the bitumen, automatically calculated by the viscometer through a digital controller. Many spindles are available with different dimensions. The proper spindle dimension is selected based on the viscosity of the bitumen being tested.

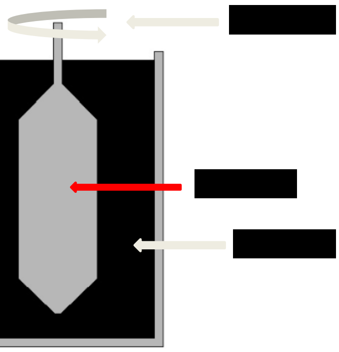


Figure 8.4 Rotational viscometer configuration

The equipment allows the calculation of viscosity over a wide range of temperature and angular velocity. Once the spindle dimension and the velocity are known, the instrument directly calculates the shear rate $\dot{\gamma}$ (s^{-1}), the shear stress τ (Pa) and the viscosity η (Poise) as follows:

$$\dot{\gamma} = \frac{2\omega R_c^2 R_b^2}{x^2(R_c^2 - R_b^2)} \qquad (8.1)$$

$$\tau = \frac{M}{2\pi L R_b^2} \quad (8.2)$$

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (8.3)$$

where ω is the angular velocity of spindle (rad/sec); R_c is the radius of container (cm); R_b is the radius of spindle (cm); x is the radius at which shear rate is being calculated (cm); M is the torque input by instrument (%) and L is the effective length of spindle (cm).
Rotational viscosity is used to evaluate high temperature workability of bitumens. High temperature viscosity is measured to ensure that bitumen is sufficiently fluid during mixing and construction.
In order to determine the limit values of the mixing and compaction temperature, the Superpave mix design (West et al., 2010) provides a specific procedure based on the iso-viscosity principle, which assumes that asphalt mixtures should be mixed and compacted at temperatures corresponding to a given range of viscosity as follows:

mixing: $\eta\_M = 0,17 \pm 0,02$ Pa·s
compaction: $\eta\_C = 0,28 \pm 0,03$ Pa·s

Figure 8.5 shows the procedure currently used to identify the mixing and compaction temperatures. The abovementioned viscosity ranges are reported in a semi-logarithmic plot and their intersection with the viscosity curve as function of temperature allows the estimation of mixing and compaction temperatures. The procedure is described in the American standard ASTM D2493.
Based on the viscosity values recorded for every materials studied, the above described procedure was used in this study to calculate mixing and compaction temperatures.

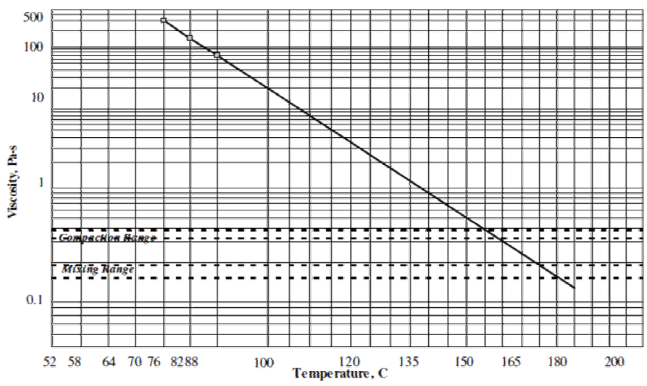


Figure 8.5 Determination of mixing and compaction temperatures (ASTM D2493)

Viscoelastic properties: Dynamic Shear Rheometer (DSR)

The Dynamic Shear Rheometer is an instrument that allows the concurrent evaluation of time and temperature effects on bitumen samples. In this research study, the Anton Paar Modular Compactor Rheometer (MCR 302, Figure 8.6) was used to perform many rheological investigations.

Figure 8.6 Anton Paar Modular Compactor Rheometer MCR 302

When used for testing bituminous materials, the DSR measures the rheological properties (e.g. complex shear modulus, phase angle) at low, intermediate and high temperatures. The test can be performed in control strain or control stress mode.
Two main geometric configurations are usually employed: the plate-plate configuration or the cone-plate configuration depending on the type of measurements required. For the purposes of this study, only the plate-plate configuration was used to carry out oscillatory tests in control strain mode.
The principle of operation is simple: the bitumen sample is sandwiched between a fixed plate and a moving plate that oscillates back and forth (Figure 8.7). The distance between the plates is maintained constant and is referred as gap. The dimension of the plates varies depending on the bitumen stiffness. In this study plates with a diameter of 8 and 25 mm were employed. The gap was set equal to 2 and 1 mm respectively.

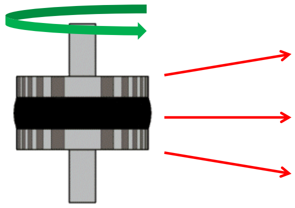


Figure 8.7 DSR configuration: plate-plate geometry

- Specimens preparation

The bitumen specimens are little disks of bitumen with a diameter equal to the oscillating plates and a height equal to the gap. They are prepared by heating the bitumen until fluid enough to pour into a rubber mold of appropriate dimensions (slightly larger than the space between the parallel plates). The heating temperature may vary depending on the bitumen stiffness. Afterwards, the disk is left at ambient temperature for at least 15 minutes to cool down (Figure 8.8). Then, after removal from the mold, the specimen is placed between the plates of the DSR to be tested. Prior to start the test, the trimming of the edge specimen through a spatula is needed in order to remove the bitumen in excess and obtain a specimen with the required dimensions.

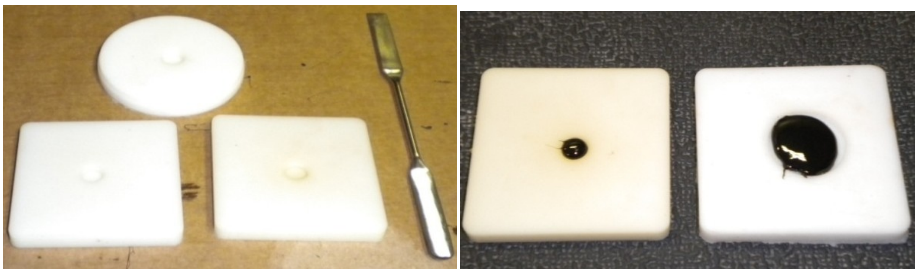


Figure 8.8 Rubber molds and trimming spatulas

The test configuration, the loading steps and the setting of the equipment can be controlled through the software associated to the instrument. In particular, all the tests carried out with the DSR provided two fundamental steps:

- a conditioning phase of 15 minutes at the test temperature to allow the specimen to reach the desired temperature homogeneously. In this step, no loads are applied;
- a loading phase where the different loading conditions are applied, depending on the type of test performed.

- Data analysis

Frequency sweep test: master curve construction

The frequency sweep test is one of the most common test performed by means of the DSR and is used for determining the viscoelastic properties of undamaged bituminous samples. It is an oscillation test performed at a given temperature and load rate. The test procedure provides the application of a constant load over a range of loading frequency. In the case of this study the test was carried out in control strain mode, by applying a low level of strain (i.e. 1%) to ensure the respect of the LVE and a frequency range from 1 to 30 Hz in a temperature range from 5 to 35°C.
The test is carried out at various temperature values to ensure the application of the Time-Temperature Superposition Principle (TTSP). In fact, in order to completely characterize the material behavior, the bitumen should be tested at very high and low frequencies, too. However, due to laboratory and technical restrictions, some test conditions cannot be performed. For example, tests at very low frequency are too long and not compatible with the laboratory practice, as well as tests at too high frequency cannot be performed due to equipment limitations. These drawbacks can be overcame by applying the TTSP that allows the construction of the master curve and the determination of the viscoelastic behavior of the bitumen across a wider range of reduced frequencies than originally tested. For this reason, tests at different temperatures must be performed. Based on the TTSP, the data measured at each temperature are horizontally shifted and aligned to the data measured at a temperature T_0 assumed as reference (Figure 8.9).

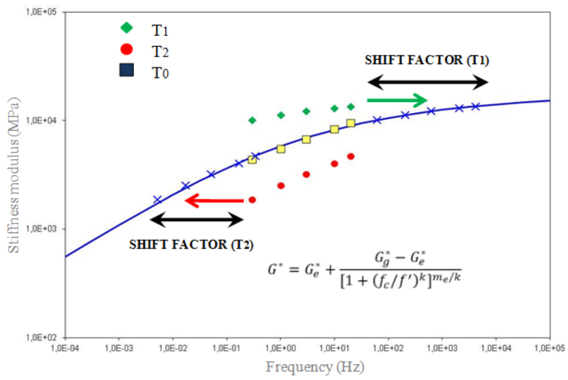


Figure 8.9 Time-Temperature Superposition principle: schematic representation

Thus, the master curve describes the rheological behavior of a material at a specific temperature and represents the trend of a rheological property (for bitumens. usually the complex shear modulus or the phase angle) as a function of loading frequency. Therefore,

the master curve can be considered as an iso-thermal representation of the mechanical behavior of viscoelastic materials over a wide range of frequency.
The master curve construction is realized by exploiting the solver functionality in Microsoft Excel that allows the difference between the measured data and the theoretical model adopted to represent the master curve to be minimized. The least-squares regression is performed in order to fit the recorded data (for bitumens, usually the complex shear modulus) to a rheological model that relates that specific rheological property to the loading frequency. The solver functionality allows the simultaneous determination of the model parameters and the horizontal shift factors a_T (at each temperature). The shift factors are function of the reference temperature T_0 and the generic test temperature T considered.
A number of different rheological model and shift factors variation laws can be used to construct the master curve. In the following section, the methods used in this research study are described. The Williams-Landel-Ferry (WLF) equation (Williams et al., 1955) was assumed as variation law for the shift factors as follows:

$$log\,\frac{a_T(T)}{a_T(T_0)} = -\frac{c_1(T-T_0)}{c_2+(T-T_0)} \tag{8.4}$$

where:

- a_T = shift factor;
- T_0 = reference temperature;
- T = test temperature;
- C_1, C_2 = material constants.

The following model (Figure 8.10) was used to represent the complex shear modulus as function of reduced frequency (Bahia et al., 2001):

$$G^* = G_e^* + \frac{G_g^*-G_e^*}{\left[1+(f_c/f')^k\right]^{m_e/k}} \tag{8.5}$$

where:

- G_e^*= equilibrium complex modulus, measured for frequencies close to zero. It is equal to zero for bitumens;
- G_g^*= glassy complex modulus, measured for very high frequencies, it is assumed equal to 1 GPa for bitumens;
- f'= reduced frequency, function of temperature and strain [Hz];
- f_c= location parameter with dimensions of frequency;
- k, m_e= shape parameters, dimensionless.

Also the relationship between phase angle and reduced frequency can be modeled through the formulation proposed by Bahia et al (2001):

$$\delta = 90I - (90I - \delta_m)\left\{1 + \left[\frac{\log(f_d/f')}{R_d}\right]^2\right\}^{-m_d/2} \qquad (8.6)$$

where:

- δ_m= phase angle at f_d, the value at inflection;
- f'= reduced frequency;
- f_d = location parameter with dimensions of frequency;
- R_d, m_d = shape parameters;
- I = 0 if $f > f_d$ I=1 if $f \leq f_d$

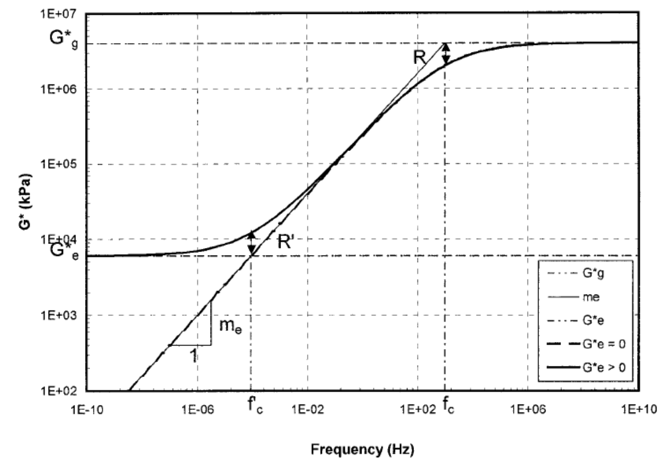


Figure 8.10 Complex modulus representation as a function of frequency (Bahia et al, 2001)

Besides the master curve, also the Black diagram (|G*| Vs. phase angle) and the Cole-Cole plane (|G''| Vs. |G'|) are useful tools to characterize the viscoelastic material behavior. Combined with the master curve trend, they provide relevant information about the overall rheological behavior of bitumen.
Finally, it is important to bear in mind that the applicability of the TTSP provides the determination of identical shift factors for each rheological property (i.e. |G*| and δ) and can be verified by the determination of a unique continuous curve in the Black diagram or in the Cole-Cole plane. Such a behavior is usually respected for unmodified bitumens, but it is generally not applied to polymer modified bitumens. For the latter, it is more suitable the application of the Partial Time-Temperature Superposition Principle that allows the

hypothesis to have different shift factors for the complex modulus and the phase angle (Di Benedetto et al. 2011).

Multiple Stress Creep and Recovery (MSCR) test: rutting behavior

It is well known that the use of polymer modified bitumens has been growing rapidly over the last decade. However, the current SuperPave method (AASHTO M 320) to determine the bitumen resistance of permanent deformation was based on study of neat (unmodified) bitumens and may not properly characterized the rutting behavior of polymer modified bitumens. Thus, a recent test protocol has been developed with the aim at better predict the rutting susceptibility of both neat and modified bitumens.
In this sense, the rutting behavior was evaluated by performing multiple stress creep recovery (MSCR) tests, according to ASTM D7405 (2010). This test provides a means to investigate the reversible and non-reversible strain behavior as well as the stress dependence of bituminous materials. The MSCR tests are carried out adopting a parallel plate geometry (25 mm plate diameter) and a gap equal to 1 mm. Tests consists in 10 creep/recovery cycles in which a creep loading time of 1 second and a recovery time of 9 seconds were selected. In accordance with the specifications, the MSCR tests are conducted on both virgin and short-term aged bitumens. In order to evaluate the temperature as well as the stress dependency of HMA and WMA bitumens, four testing temperatures (58 °C, 64 °C, 70 °C and 76 °C) and two stress levels (0.1kPa, and 3.2kPa) were selected.
Two replicates were performed for each testing condition and the average value was thereafter considered in the results analysis.
For each of the twenty creep and recovery cycles, the specimen is loaded at a constant stress of τ_0 for l second and follow with a zero stress recovery of 9.00 second duration. As shown in Figure 8.11, for each cycle, the initial strain value at the beginning of the creep portion of each cycle is denoted as γ_0 whereas the strain value at the end of the creep portion (that is, after 1.0 s) of each cycle is denoted γ_c. The adjusted strain value at the end of creep portion of each cycle is γ (t=1s) is calculated as the difference between γ_c and γ_0.
The strain value at the end of the recovery portion (that is, after 10.0 s) of each cycle is denoted as γ_r. The adjusted strain value at the end of recovery portion of each cycle is γ (t=10s) and is calculated as the difference between γ_r and γ_0.

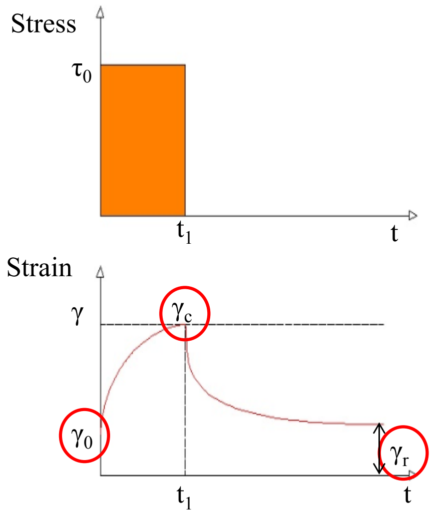


Figure 8.11 Stress and strain trend for each cycle of a MSCR test

For each of the ten cycles at a creep stress of 0.1 kPa as well as for each of the ten cycles at a creep stress of 3.2 kPa, the percent recovery R (100, N) and R(3200, N) is calculated as follows for N = 1 to 10:

$$\varepsilon_r(100, N) = \frac{\varepsilon_1 - \varepsilon_{10}}{\varepsilon_1} * 100 \quad (8.7)$$

$$\varepsilon_r(3200, N) = \frac{\varepsilon_1 - \varepsilon_{10}}{\varepsilon_1} * 100 \quad (8.8)$$

Then, the average percent recovery is calculated at each load value:

$$R(100) = \sum \varepsilon_r(100, N)\,/10 \quad (8.9)$$

$$R(3200) = \sum \varepsilon_r(3200, N)\,/10 \quad (8.10)$$

Afterwards, for each of the ten cycles at a creep stress of 0.1 kPa and 3.2 kPa it is possible to calculate the non-recoverable creep compliance, $J_{nr}(100, N)$ and $J_{nr}(3200, N)$, for N = 1 to 10 as follows:

$$J_{nr}(100, N) = \frac{\varepsilon_{10}}{100} \quad (8.11)$$

$$J_{nr}(3200, N) = \frac{\varepsilon_{10}}{3200} \quad (8.12)$$

Then, the average non-recoverable creep compliance is calculated at each load value:

$$J_{nr}(100) = \sum J_{nr}(100, N)\,/10 \quad (8.13)$$

$$J_{nr}(3200) = \sum J_{nr}(3200, N)\,/10 \quad (8.14)$$

8.2.4 Results and analysis

Viscosity analysis

Results in terms of viscosity are reported in Table 8.2 for all the bitumens selected; no significant differences can be noticed between HMA and WMA bitumens. A slightly decrease in viscosity was detected for the WMA-C that includes chemical additive with respect to the HMA bitumen at the lower tested temperatures.

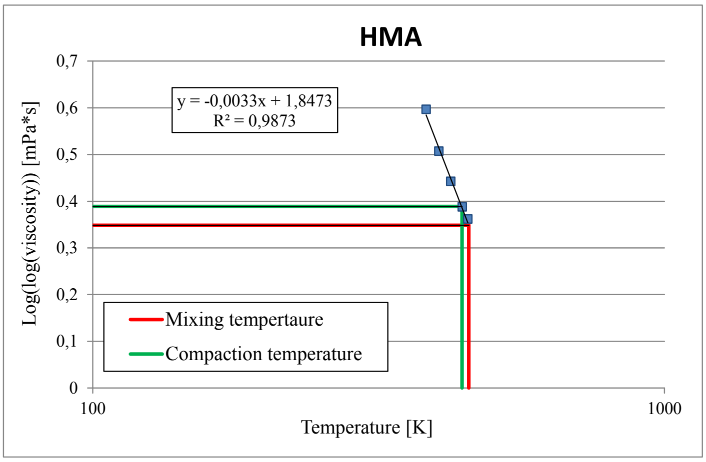
Table 8.2 Bitumens viscosity results

	Brookfield viscosity [Pa*s]				
T [°C]	HMA	WMA-O_3,0%	WMA-O_4,0%	WMA-C_0,5%	WMA-Z_6,0%
110	8,96	7,28	6,56	5,59	7,60
130	1,63	1,51	1,38	1,40	1,65
150	0,59	0,54	0,49	0,54	0,60
170	0,28	0,25	0,22	0,26	0,27
180	0,20	0,18	0,16	0,19	0,19

Even if no significant decreasing in viscosity was measured, such values were used to evaluate the mixing and compaction temperatures using the procedure previously explained (Figure 8.5). In this sense, the iso-viscosity principle was taken into account assuming that asphalt mixtures should be mixed and compacted at temperatures corresponding to 0,17 ± 0,02 Pa·s for mixing and 0,28 ± 0,03 Pa·s for compacting. Results in terms of mixing and compaction temperatures are graphically and numerically reported in Figure 8.9 for all the bitumens.

The mixing and compaction temperatures that were evaluated (Figure 8.12) did not show any significant improvement due to the use of WMA additives, suggesting that the iso-viscosity principle cannot be used in case of warm mixtures. The use of WMA additives, in fact, is known to be able to guarantee a significant reduction of the production temperatures; hence, it is the evaluation method that is not able to predict properly the

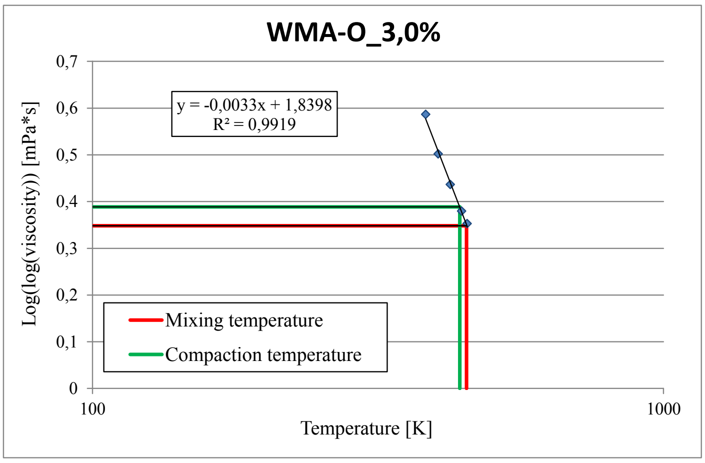
WMA bitumens behavior. Such an outcome can be explained by the fact that the WMA additives does not merely act on the viscosity of the bitumen phase but also influence the adhesion properties between bitumen and aggregates. Hence, the evaluation of the bitumen viscosity does not allow a complete investigation of the WMA additives behavior.



T_{mix}=181,6°C

T_{com}=169,4°C

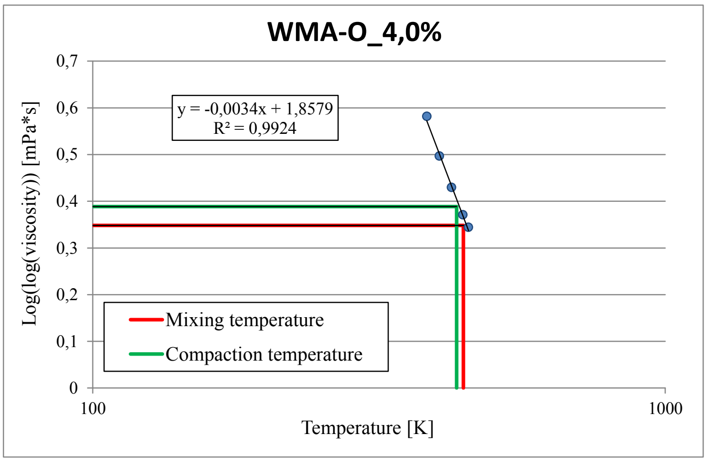
(a)



T_{mix}=178,7°C

T_{com}=166,5°C

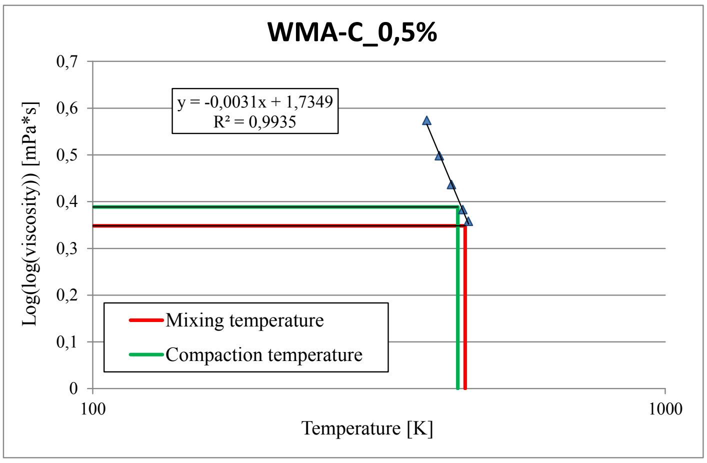
(b)



T_{mix}=170,8°C

T_{com}=158,9°C

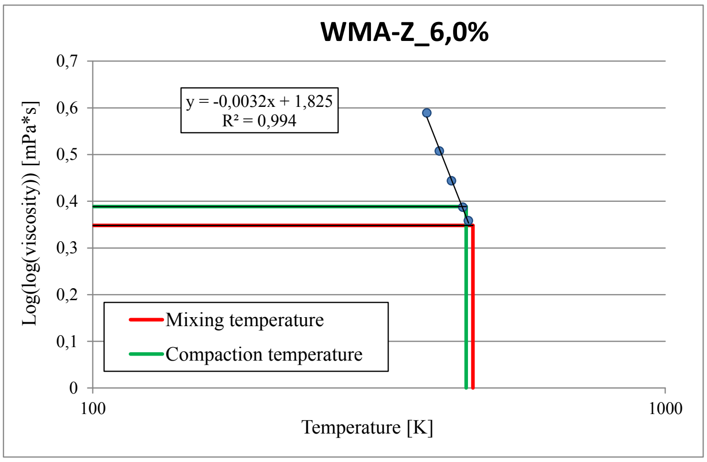
(c)



T_{mix}=174,1°C

T_{com}=161,1°C

(d)



T_{mix}=188,2°C

T_{com}=175,7°C

(e)

Figure 8.12 The mixing and compaction temperatures evaluated for the HMA bitumen (a) and the WMA bitumens including organic additive at low (b) and high content, chemical additive (d) and zeolite (e).

To further investigate such aspect, mastics were prepared using the same bitumens and one filler/bitumen ratio equal to 1 and selecting the same limestone filler for all the mastics. The idea to compare viscosity of mastics instead of bitumens arise from the fact that the action of WMA additives takes place at the bitumen/aggregates interface by reducing and regulating the forces rather than only reducing the bitumen viscosity. Thus, in order to evaluate such effects, also the viscosity of mastics (bitumen+filler) should be analyzed.
The Brookfield viscosity of mastics was measured using the same temperatures that were selected for the bitumen and the results are given in Table 8.3. Only the highest content (4.0%) was selected for the organic additive.

Table 8.3 Mastics viscosity results

	Brookfield viscosity [Pa*s]			
T [°C]	HMA	WMA-O_4,0%	WMA-C_0,5%	WMA-Z_6,0%
110	27,25	9,69	16,47	30,13
130	6,06	2,10	4,44	6,19
150	2,12	0,73	1,66	2,20
170	0,91	0,45	0,70	0,96
180	0,59	0,30	0,50	0,60

Mastics viscosity results highlighted the differences between HMA and WMA: in case of the organic and chemical additives, in fact, a clear reduction in mastics viscosity with

respect to the HMA mastics can be noticed. On the contrary, the effect of the zeolite was not measured by these tests: this could be explained by the fact that the zeolite is the only WMA additive that should be added directly into the mixture rather than to the bitumen. Thus, the procedure used for this experimental section in case of the zeolite was not correct and should not be taken into account.
In order to evaluate the mixing and compaction temperatures of the mixtures, the iso-viscosity principle should be applied but there is not a recommended range of viscosity for mastics. Thus, the HMA mastic viscosity at the mixing and compaction temperatures that were evaluated for the HMA bitumen (181,6 for mixing and 169,4 for compacting) were used to evaluate a reference viscosity range for mastics using the HMA mastic curve, as shown in Figure 8.10. A reference viscosity value of 0.43 mPa*s was found for mixing and 0.46 mPa*s was found for compaction (Figure 8.13) and applied to the WMA mastics, as shown in Figure 8.14.

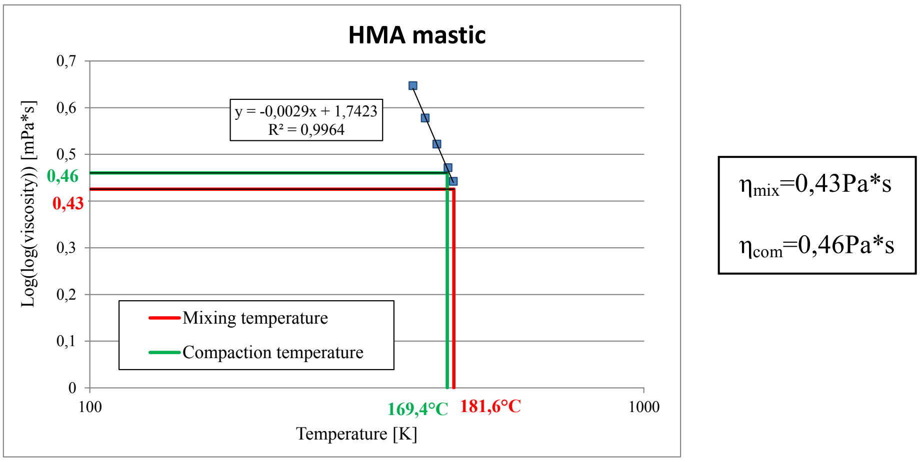
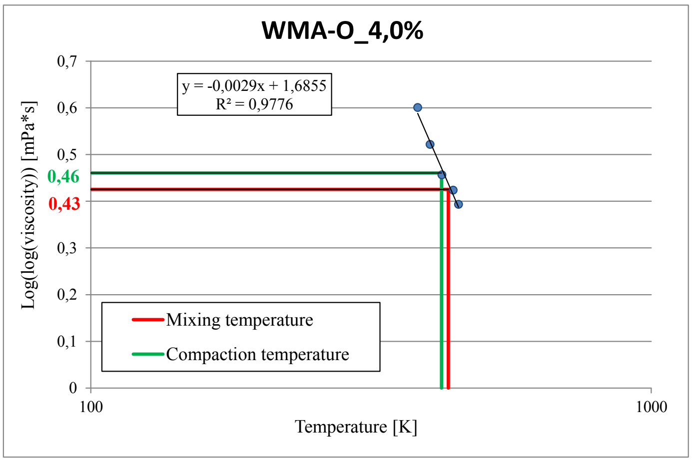


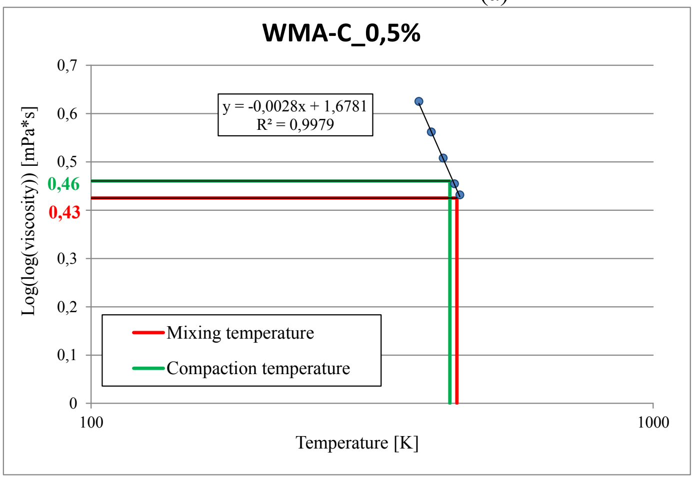
Figure 8.13 Reference viscosity range for mastics



T_{mix}=174,3°C

T_{com}=161,7°C

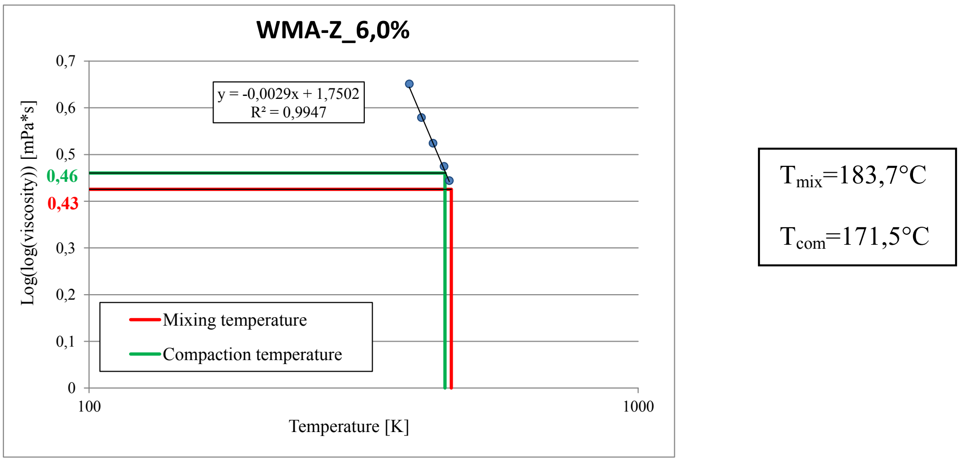
(a)



T_{mix}=161,4°C

T_{com}=149,2°C

(b)



(c)

Figure 8.14 Mixing and compaction temperatures of the WMA mastics including organic additive (a), chemical additive (b) and zeolite (c).

As already explained, results in terms of zeolite should not be taken into account due to the laboratory procedure selected that is not able to simulate the real in-plant conditions. On the other hand, as far as organic and chemical additives are concerned, results showed a clear reduction in terms of mastics production temperatures with respect to the HMA mastic equal to about 7°C for the WMA-O and 20°C for WMA-C. Such a reduction is higher than the one measured in case of the bitumen analysis, meaning that the mastic evaluation allows a better understanding of the WMA additive behavior in a mixture. However, results are not able to properly predict the production temperatures of WMA mixtures since significant reduction (about 40°C) are expected when WMA additives are used.

Master curves

The stiffness properties of the materials were investigated through frequency sweep tests allowing the measurement of complex shear modulus (G*) and phase angle (δ) under several frequency and temperature conditions.
The linear-viscoelastic (LVE) threshold of each bitumen was first analyzed by means of strain-sweep tests with the DSR; such value was found as the strain at which correspond 95% of the initial complex modulus value (G*). LVE values equal to 1.0% were found for all the bitumen studied regardless the presence of WMA additives. Thus, frequency-sweep tests were run at a strain level of 0.5% for the determination of master curves.
Afterwards, the master curves of the bitumen studied were built at a reference temperature of 34°C using the Time-Temperature Superposition Principle (TTSP) and the Bahia model

(Bahia et al., 2001) was selected to represent the complex shear modulus as function of reduced frequency, as detailed described in section 8.2.3. The model parameters are reported in Table 8.4; they were input in the model to obtain some of the values of the master curves, given in Table 8.5.

Table 8.4 Master curve parameters for HMA and WMA bitumens

$E^* = E^*_e+(E^*_g-E^*_e)[1+(f_c/f)^k]^{-me/k}$						
Tipo di bitume	G^*_e [kPa]	G^*_g [kPa]	f_c	k	m_e	R^2
HMA	0	1,00E+06	8,77E+01	0,117	0,900	0,984
WMA-C	0	6,55E+06	2,81E+02	0,094	0,940	0,984
WMA-O	0	1,20E+06	1,60E+01	0,111	0,950	0,984
WMA-Z	0	6,55E+06	2,81E+02	0,091	0,910	0,984

Table 8.5 Master curve values for HMA and WMA bitumens

MASTER CURVES				
Frequency [Hz]	Complex modulud G* [kPa]			
	HMA	WMA-C	WMA-O	WMA-Z
0,0000001	0,005	0,007	0,003	0,005
0,000001	0,030	0,050	0,017	0,028
0,00001	0,188	0,317	0,106	0,162
0,0001	1,10	1,87	0,62	0,89
0,001	5,91	10,08	3,39	4,51
0,01	29,03	48,80	16,97	21,18
0,1	128	210	77	91
1	502	792	319	355
10	1727	2610	1179	1249
100	5177	7474	3887	3951
1000	13501	18637	11404	11201
10000	30713	40691	29776	28471
100000	61393	78519	69392	65041
1000000	109032	135519	145094	134158
10000000	174341	212024	274160	251452

Those values are used to graphically build the master curves as a trend of the complex shear modulus as a function of loading frequency; all the master curves are shown in Figure 8.15 for comparing the rheological behavior of the HMA and WMA bitumens.
The comparison of G^* trend as a function of the frequency shows that for high frequency values (that correspond to low temperatures behavior), all the bitumens have an asymptotic behavior around 1.0 GPa that identify the glass component of the bituminous phase. In particular, at high frequency levels the three WMA bitumens achieved higher stiffness values with respect to the HMA bitumen. Such an outcome could lead to a more brittle behavior of the WMA mixtures at low in-service temperatures.
On the other hand, at low frequency levels (that correspond to high in service temperatures) the behavior of the bitumens studied is significantly different. The bitumen WMA-O is

characterized by higher stiffness values meaning that the presence of the organic WMA additives helps to enhance the rutting resistance. On the other hand, the bitumen including the chemical additive showed lower stiffness values at high in-service temperatures whereas no significant difference in behavior was detected between the bitumen including the zeolite and the reference bitumen HMA.

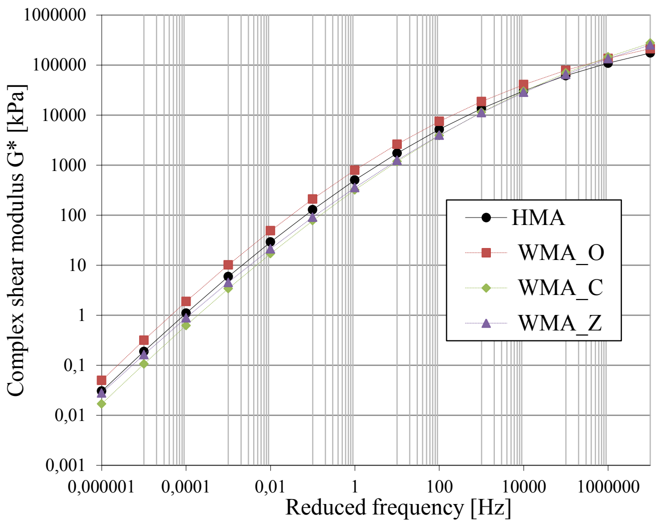
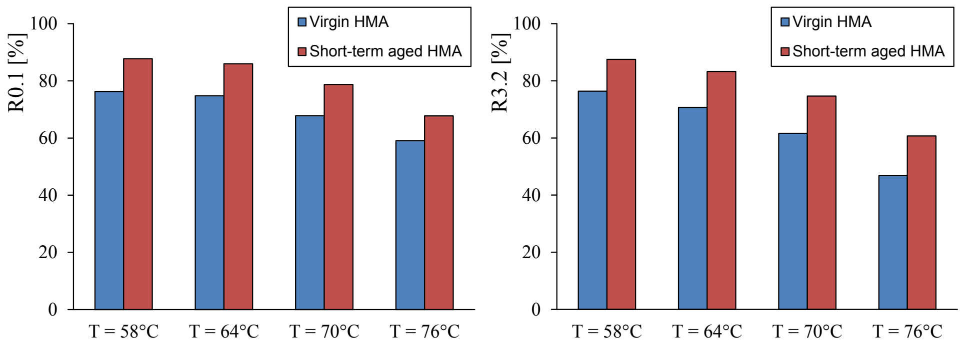


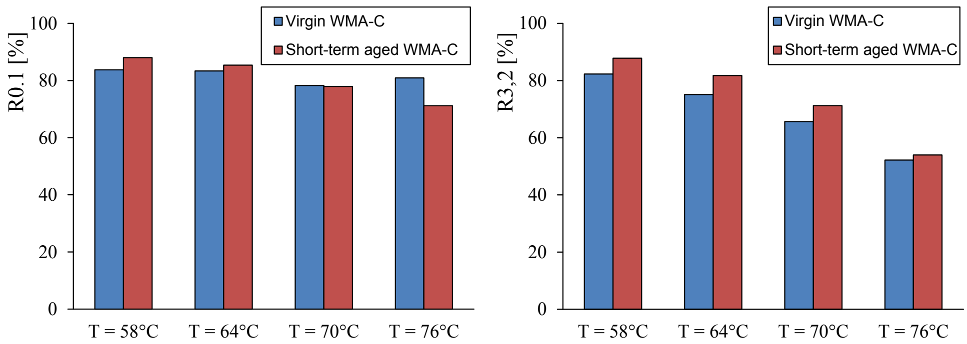
Figure 8.15 Master curves of the HMA and WMA bitumens

Rutting behavior (MSCR test)

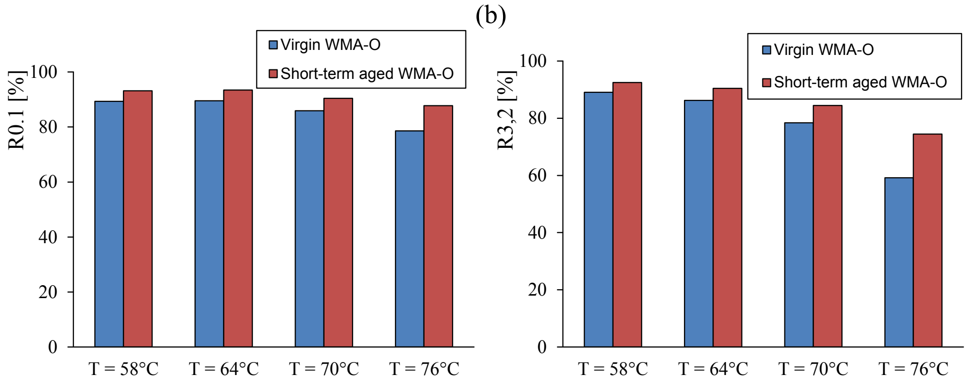
MSCR tests were carried out on both virgin and short-term aged bitumens in order to evaluate the rutting performance of HMA and WMA bitumens prepared using different additives. As already detailed explained, MSCR tests allow the investigation of the reversible and non-reversible strain behavior as well as the stress dependence of bituminous materials. In order to evaluate the temperature as well as the stress dependency of HMA and WMA bitumens, four testing temperatures (58 °C, 64 °C, 70 °C and 76 °C) and two stress levels (0.1kPa, and 3.2kPa) were investigated.
Results in terms of average percent recovery R0.1 and R3.2 at different tested temperatures are represented in Figure 8.16 for each material investigated (both virgin and short-term aged); R0.1 represent the strain recovery for a load of 100 Pa whereas R0.1 represent the strain recovery for a load of 3200 Pa.


R0.1 [%]
R3.2 [%]
Virgin HMA
Short-term aged HMA
100
80
60
40
20
0
T = 58°C
T = 64°C
T = 70°C
T = 76°C

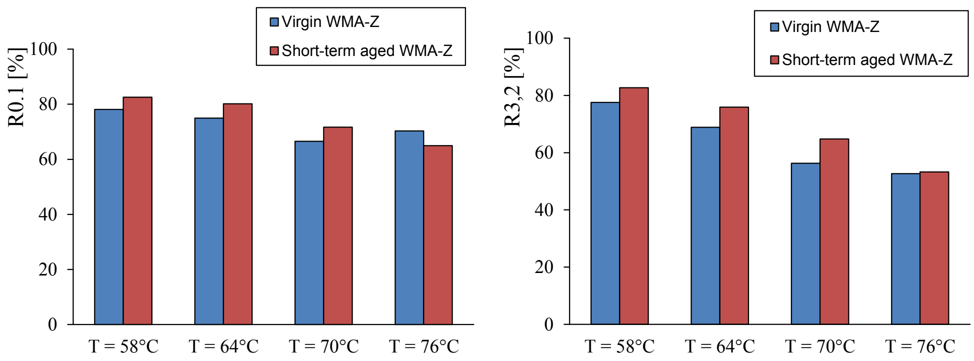
(a)


R0.1 [%]
R3,2 [%]
Virgin WMA-C
Short-term aged WMA-C
100
80
60
40
20
0
T = 58°C
T = 64°C
T = 70°C
T = 76°C

(b)


R0.1 [%]
R3,2 [%]
Virgin WMA-O
Short-term aged WMA-O
100
80
60
40
20
0
T = 58°C
T = 64°C
T = 70°C
T = 76°C

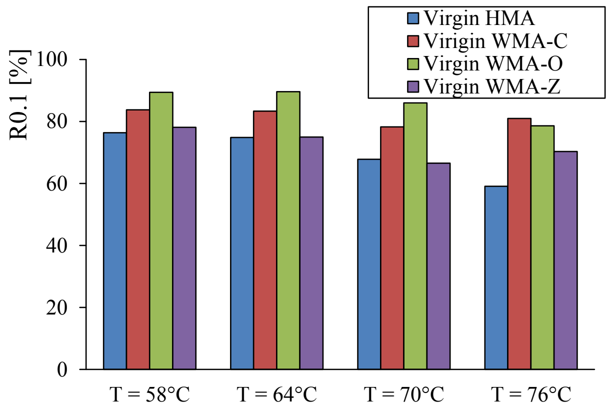
(c)



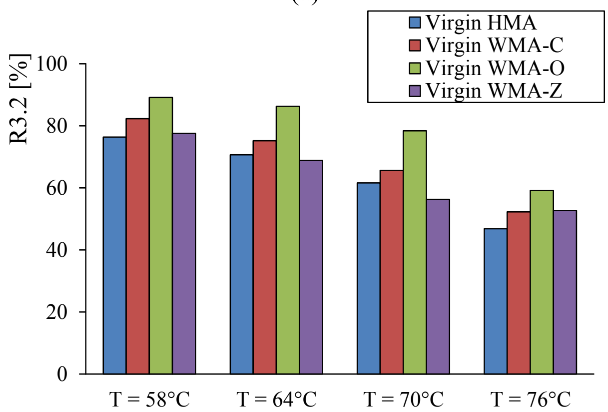
(d)

Figure 8.16 Percent recovery R0.1 (left) and R3.2 (right) for HMA bitumen (a) and WMA bitumens including chemical additive (b), organic additive (c), and zeolite (d).

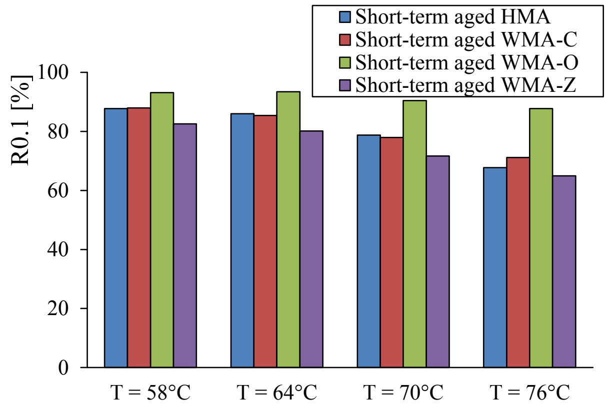
Based on the results, it is possible to notice that the percent recovery is always higher at 100 Pa load when compared to 3200 Pa. Moreover, as the test temperature increases, the recovery strain decreases due to an increment of the viscous component and a decrement of the elastic one. As far as the aging process is concerned, it is possible to notice that the percent recovery is higher for the short-term aged bitumens. Thus, the aging process seems to lead to an increment of stiffness as well as an enhanced elasticity of the bituminous phase. Such an outcome is more evident for the HMA bitumen whereas the difference is less significant for all the WMA bitumens due to the reduced production temperatures used for the aging process.
In order to compare the percent recovery of the different bitumens selected, R0.1 and R3.2 values are given in Figure 8.17 at the different test temperatures for both virgin and aged materials. Results for virgin materials showed that the recovery strain of the WMA bitumens is higher than the one of the virgin bitumen (Figure 8.17 a and b). The presence of the WMA additives does not seem to negatively affect the rutting resistance of the bitumen. On the other hand, in case of short-term aged materials, the difference between HMA and WMA bitumens is less significant and the results are almost comparable. Such an outcome can be attributed to the fact that WMA bitumens were subjected to an aging process at reduced temperatures and, thus, to a less hardening process. In conclusions, the performance gap between WMA and HMA bitumens is less significant in case of aged materials with respect to the virgin ones due to the low aging process that characterized warm mixtures. Such an effect does not significant affect the rutting performance of WMA bitumens including chemical and organic additives whereas the presence of the zeolite leads to a decrement in rutting behavior with respect to the aged HMA bitumen.


Virgin HMA
Virigin WMA-C
Virgin WMA-O
Virgin WMA-Z
R0.1 [%]
100
80
60
40
20
0
T = 58°C
T = 64°C
T = 70°C
T = 76°C

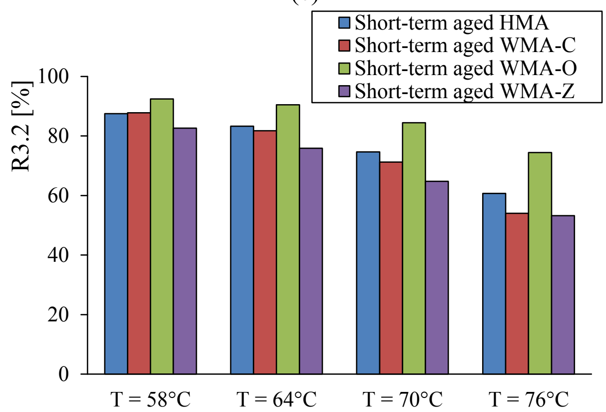
(a)


Virgin HMA
Virigin WMA-C
Virgin WMA-O
Virigin WMA-Z
R3.2 [%]
100
80
60
40
20
0
T = 58°C
T = 64°C
T = 70°C
T = 76°C

(b)



(c)



(d)

Figure 8.17 Comparison between virgin HMA and WMA bitumens in terms of R0.1 (a) and R3.2 (b) and short-term aged HMA and WMA bitumens in terms of R0.1 (c) and R3.2 (d).

Finally, the stress dependence of bituminous materials is investigated by means of the R_{diff} parameter, calculated as expressed in Eq. (8.15):

$$R_{diff} = \frac{R_{0.1} - R_{3.2}}{R_{0.1}} \quad 8.15$$

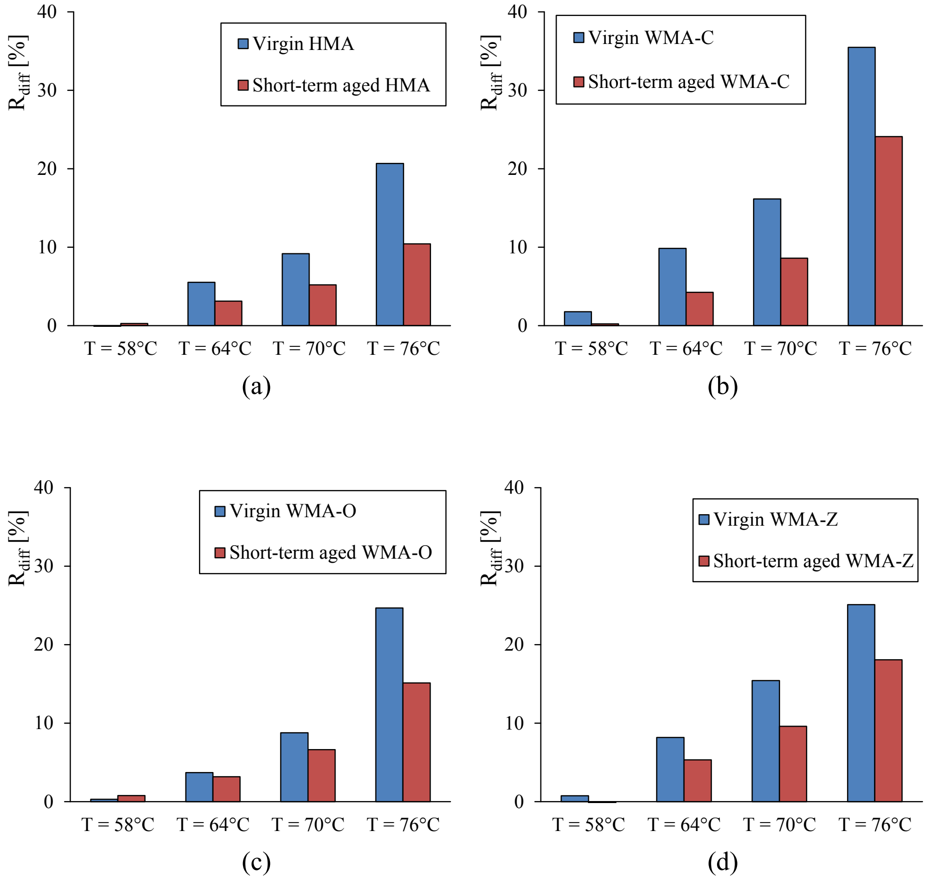


Figure 8.18 Stress dependence of bituminous in terms of Rdiff for virgin and short-term aging HMA (a), WMA-C (b), WMA-O (c) and WMA-Z (d).

Results (Figure 8.18) showed that the difference in terms of the material response at 100 Pa and 3200 Pa (R_{diff}) tends to increase as the temperature increases. Moreover, such difference is always lower for the aged bitumen with respect to the virgin bitumen for all the materials analyzed. In fact, the aged bitumen results more elastic than the virgin one, regardless the hot and warm production. Among the bitumens analyzed, the presence of the chemical additive seems to induce a more significant stress dependency (higher R_{diff}) than the HMA bitumen and the other WMA bitumens.

8.2.5 Conclusions

The rheological analysis concerning WMA bitumens in comparison with a similar HMA bitumen is presented in this chapter. In particular, viscosity characteristics were investigated using a Rotational Viscometer (RV) in order to compare the mixing and compaction temperatures of both WMA and HMA bitumens and mastics. Moreover, mechanical properties such as master curves as well as rutting behavior were evaluated by means of a Dynamic Shear Rheometer (DSR) on WMA and HMA bitumens. Unaged as well as short term aged bitumens were tested in terms of rutting performance in order to simulate all the different stages that pavements undergo during the construction.

Based on the experimental results on the viscosity analysis of bitumens and mastics is possible to conclude that the standard procedures used for the evaluation of mixing and compaction temperatures of HMA mixtures are not able to properly predict the behavior of WMA additives in terms of production temperatures. Such procedure, in fact, is based on the iso-viscosity principle which assumes that asphalt mixtures should be mixed and compacted at temperatures corresponding to a given range of bitumen viscosity that ensures that the bitumen phase is sufficiently fluid during mixing and construction. This procedure is valid in case of HMA mixtures but it cannot be used when warm mixtures are involved since WMA additives do not impact only the bitumen viscosity; their action takes place also at the bitumen/aggregates interface by reducing and regulating the forces rather than only reducing the bitumen viscosity. Further studies are needed in order to define a proper procedure able to evaluate the production temperatures when WMA additives are involved.

As far as the comparison of complex modulus trend as a function of the frequency (master curves) is concerned, it is possible to conclude that all the bitumens have an asymptotic behavior around 1.0 GPa (that identify the glass component of the bituminous phase) at high frequency values (that correspond to low temperatures behavior). In particular, at high frequency levels the three WMA bitumens achieved higher stiffness values with respect to the HMA bitumen. Such an outcome could lead to a more brittle behavior of the WMA mixtures at low in-service temperatures. On the other hand, at low frequency levels (that correspond to high in service temperatures) the behavior of the bitumens studied is significantly different. The bitumen WMA-O is characterized by higher stiffness values meaning that the presence of the organic WMA additives helps to enhance the rutting resistance. On the other hand, the bitumen including the chemical additive showed lower stiffness values at high in-service temperatures whereas no significant difference in behavior was detected between the bitumen including the zeolite and the reference bitumen HMA.

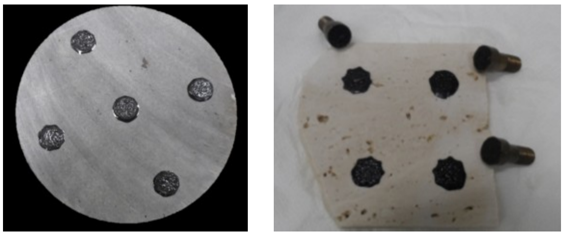
Results in terms of rutting performance (MSCR tests) demonstrated that the aging process leads to an increment of stiffness as well as an enhanced elasticity of the bituminous phase. Such an outcome is more evident for the HMA bitumen whereas the difference is less significant for all the WMA bitumens due to the reduced production temperatures used for the aging process that properly reproduced the in-plant warm production. Results for virgin materials showed that the recovery strain of the WMA bitumens is higher than the one of the virgin bitumen whereas in case of short-term aged materials, the difference between HMA and WMA bitumens is less significant and the results are almost comparable. Such an outcome can be attributed to the fact that WMA bitumens were subjected to an aging process at reduced temperatures and, thus, to a less hardening process. Only the presence of the zeolite leads to a decrement in rutting behavior with respect to the aged HMA bitumen.

8.3. Adhesion properties and raveling resistance

8.3.1 Materials

BBS tests

Bitumen-aggregate adhesion properties were evaluated by means of Bond Bitumen Strength (BBS) tests. Two aggregate types, basalt and limestone (Figure 8.19a, 8.19b, respectively), were selected as they represent the typical materials used to produce porous asphalt mixtures and dense graded mixtures. Aggregate plates were prepared with adequate dimensions (10 × 10 cm^2) in order to allow the positioning of five specimens. For each aggregate type, two different surface treatments were considered: untreated surface (i.e. uncoated) were used to simulate virgin aggregates, whereas a specific protocol (Canestrari et al. 2014) was adopted to reproduce the surface of RAP aggregates (i.e. coated). The protocol adopted for the surface treatments (uncoated and coated) is the same already described in detail in section 3.2.2.



(a) (b)

Figure 8.19 Aggregate types: basalt (a) and limestone (b).

A Styrene-Butadiene-Styrene (SBS) polymer modified bitumen (PG 82-16) was used to coat the substrates since the majority of RAP for motorway pavements currently available in Italy includes SBS polymer modified bitumens; the bitumen is the same that has been used during the entire research project and the main characteristics are given in Table 4.7.
The aggregate-bitumen system was prepared by placing a small amount of virgin bitumen (0.08 g) onto the surface of a pull-stub pre-heated at 170°C for a minimum of 30 minutes to

simulate the in plant production temperature for bitumen. Afterwards, the pull-stub was pressed onto the pre-heated aggregate surface avoiding twisting stresses that could entrap air between the bitumen and the aggregate. In order to simulate the aggregate-bitumen adhesion properties that characterized HMA and WMA mixtures, aggregates surfaces were pre-heated at different temperatures, equal to 170°C to simulate the HMA condition and 130°C to simulate the WMA condition.
All the HMA and WMA conditions were simulated by using the same SBS polymer modified bitumen already mentioned (Table 4.7). For the WMA condition three different technologies were adopted as representative of the three main categories nowadays available in the market: a chemical additive (dosed at 0.5% by bitumen weight), an organic additive (dosed at 3.0% by bitumen weight) and a zeolite (dosed at 6.0% by bitumen weight). Moreover, an additional WMA condition was taken into account by using the same reduced production temperature (130°C) without adding any WMA additive to the bitumen, in order to differentiate the effects due only to the temperature reduction from the ones related to the adding of WMA additives. The identification codes are summarized in Table 8.6.

Table 8.6 Identification codes for BBS specimens.

Aggregate source	**Surface treatment**	**Production temperature**				
		170°C	130°C	130°C	130°C	130°C
		No additive	No additive	Chemical additive	Organic additive	Zeolite
Basalt	Uncoated	B_H_U	B_W_U	B_WC_U	B_WO_U	B_WZ_U
	Coated	B_H_C	B_W_C	B_WC_C	B_WO_C	B_WZ_C
Limestone	Uncoated	L_H_U	L_W_U	L_WC_U	L_WO_U	L_WZ_U
	Coated	L_H_C	L_W_C	L_WC_C	L_WO_C	L_WZ_C

Cantabro test

HMA and WMA mixtures were prepared in laboratory and tested by means of Cantabro tests in order to evaluate the influence of reduced production temperatures on raveling resistance. The experimental program included both porous asphalt (hereafter named PA) and dense graded (hereafter named DG) mixtures; both mixtures type included recycled aggregates (dosed at different amount as prescribed by the Italian technical specification for motorways).
Porous asphalt mixtures were prepared including 15% of selected RAP (coarse fraction 8/16 mm) from milled porous asphalt surface layers using the mix-design that was optimized in the first part of this experimental study (section 4). Basalt virgin aggregates, RAP (8/16 mm) and filler were combined obtaining the final grading curve reported in Figure 8.20a and a total bitumen content was selected equal to 5.25% (by aggregate weight) given by the virgin bitumen and the bitumen within RAP (4.0% by RAP weight).

Dense graded mixtures were prepared including 25% of un-fractioned RAP (0/16 mm). Limestone virgin aggregates and filler were combined obtaining the final grading curve showed in Figure 8.20b. The total bitumen content was equal to 4.80% (by aggregate weight) including the virgin bitumen and the bitumen within RAP (4.68% by RAP weight). The virgin bitumen used to produce both PA and DG mixtures was a SBS modified bitumen, the same used to prepare the BBS specimens.

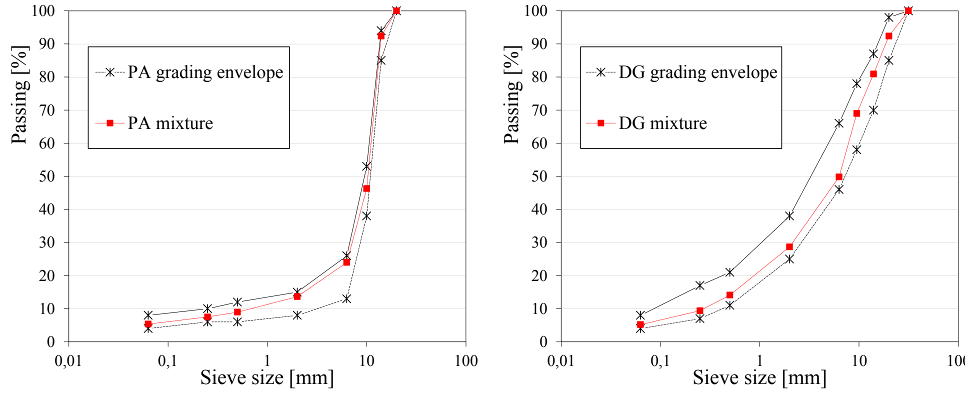


Figure 8.20 Aggregates grading curves: (a) open-graded mixtures; (b) dense-graded mixtures.

For each type of mixture, one HMA reference mixture and three WMA mixtures (prepared with different WMA additives) were investigated; moreover, an additional WMA mixture was prepared at reduced production temperatures without adding any WMA additives for comparison purposes.
The reference mixtures, hereafter named PA_H and DG_H, were mixed and compacted at 170°C and 160°C respectively whereas all the other WMA mixtures were mixed at 130°C and compacted at 120°C. In particular, virgin aggregates were heated at 130°C for about 3 hours whereas the bitumen was heated at 170°C in order to achieve proper fluidity, following the in plant procedure.
The same type and dosage of WMA additives used to produce the BBS specimens were used to prepare the three WMA mixtures. The chemical and the organic additive were added to the hot virgin bitumen and mixed for 15 minutes right before the mixture preparation whereas the zeolite was added directly to the asphalt mixture in the mixing chamber, according to the producer recommendations. The identification codes are summarized in Table 8.7.

Table 8.7 Identification codes for Cantabro specimens.

Mixture type	**Production temperature**				
	170° - 160°C	130° - 120°C	130° - 120°C	130° - 120°C	130° - 120°C
	No additive	No additive	Chemical additive	Organic additive	Zeolite
Porous asphalt	PA_H	PA_W	PA_WC	PA_WO	PA_WZ
Dense-graded	DG_H	DG_W	DG_WC	DG_WO	DG_WZ

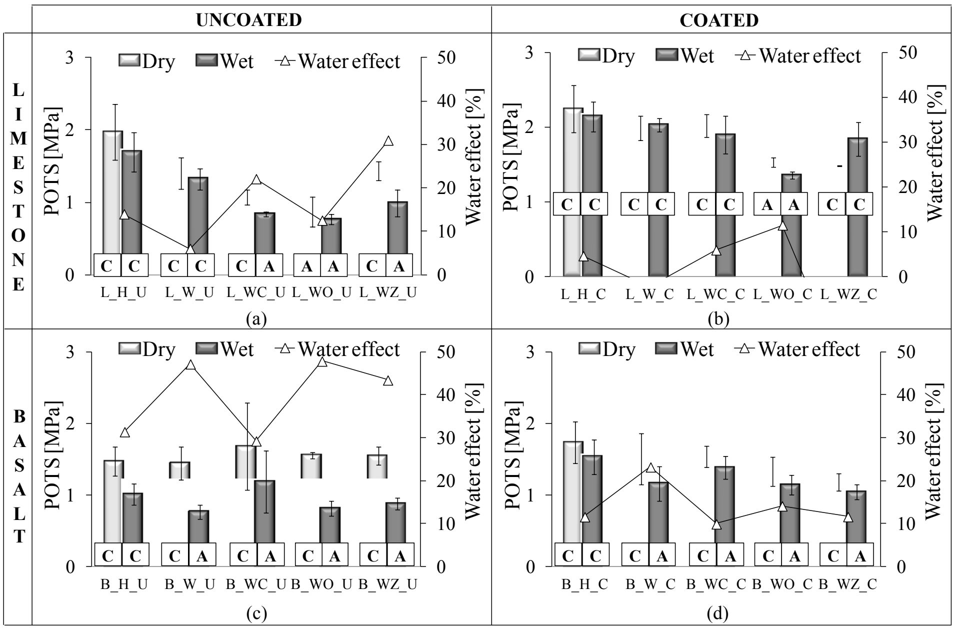
8.3.2 Test program and protocols

The experimental program includes BBS tests and Cantabro tests with the aim at evaluating the influence of reduced production temperature and warm technologies on adhesion properties and moisture susceptibility. To this purpose, BBS and Cantabro tests were performed at 25°C on specimens after both dry conditioning (in air for 24 h at the test temperature) or wet conditioning (in water at 40°C for 72 h, according to EN 12697-12 Method A).
BBS test (AASTHO TP 91) quantifies the cohesion/adhesion properties between aggregate and bitumen within asphalt mixtures; test method as well as data analysis were described in details in Section 3.2.2. It is useful to underline the two main outputs of BBS tests: the Pull-Off Tensile Strength (POTS) is the pressure value necessary to reach the failure of the aggregate/bitumen bond as a function of the bonding surface area. Moreover, the failure type is visually identified as two main mechanisms may occur: failure at the interface (adhesive failure code A), defined as loss of adhesive bond strength between bitumen and aggregate; failure within the bitumen (cohesive failure code C), characterized by loss of cohesive strength within the bituminous component caused by the rupture of bonds in the asphalt film. In the case of this study, five specimens were prepared for each test configuration.
Cantabro tests (EN 12697-17) allow the estimation of the raveling resistance of asphalt mixtures. The test consists in subjecting an asphalt specimen to 300 revolutions inside the Los Angeles machine drum without any metal balls; the particle loss (PL) at the end of the tests is taken into account as a measure of the internal cohesion between particles. For each mixture type (porous asphalt and dense graded mixtures) and for each test condition (dry and wet), four specimens were prepared and tested by means of Cantabro tests. All specimens were compacted using the gyratory compactor at a fixed height (63.5 mm) in order to achieve similar air voids contents for each mixture type (selected equal to 20% for OG and 4% for DG) since it is recognized that particle loss values are strongly dependent on the air void content of the specimen (Frigio et al. 2013).

8.3.3 Results and analysis

BBS test results

Figure 8.21 summarizes all the data collected through BBS tests in terms of POTS (average of five replicates along with the corresponding error bars expressed as standard deviation) and failure type. The graphs show the results for coated and uncoated limestone and basalt aggregates.



C = cohesive failure within bitumen; A = adhesive failure at the bitumen-aggregate interface

Figure 8.21 BBS results. Comparison between dry and wet condition for different substrates: (a) limestone uncoated; (b) limestone coated; (c) basalt uncoated; (d) basalt coated.

Results show that both the uncoated surface (virgin aggregate) and the coated surface (RAP aggregate) were able to develop higher bond strength in dry condition with respect to wet condition, for almost every configuration analyzed. The percentage reduction of POTS due to the water effect is evaluated as the difference between POTS values measured in dry and wet condition with respect to the POTS value after dry conditioning and it is depicted as a continuous line in the diagrams of Figure 8.21.

- Bond strength analysis

It is worth mentioning that the specimens simulating reclaimed aggregates (coated surface) underwent significantly lower water effect than virgin aggregates, regardless WMA additive and aggregate type, as shown by the average POTS values measured after wet conditioning.
Moreover, the comparison between different aggregate types shows that limestone (both coated and uncoated) were less affected by water than basalt aggregates. The mineralogical-chemical nature of the selected aggregates explains this finding. In fact, limestone, characterized by a more basic composition than basalt, is classified as a hydrophobic stone, which means reduced compatibility with water and subsequent more pronounced affinity with bitumen. Anyway, the difference was less pronounced in the case of coated aggregates thank to the thin film of aged bitumen on the surface that reduces the direct interaction between aggregate and water.
As far as "warm technologies" (i.e. specimens prepared at 130°C using bitumen with WMA additives) are concerned, two different behaviors were detected. In the case of basalt plates (coated and uncoated), the mineralogical nature of the surface seemed to prevail over WMA additives and production temperature. On the contrary, the effect of WMA additive and specimen preparation temperature is significantly detectable for limestone uncoated aggregates. In fact, WMA specimens were characterized by lower adhesion performance than HMA specimens and significant differences in results were observed changing WMA additive types. However, when limestone coated aggregates are considered, the differences in terms of bond strength are much less pronounced, so demonstrating that coated aggregates are helpful to reduce the impact of warm technologies. Necessarily, the thin film of aged bitumen that coats RAP aggregates is able to provide an adhesive substrate which guarantees better interaction with the virgin bitumen, although reduced production temperatures. This finding suggests that RAP aggregates are helpful for the production of warm mixtures since they reduce the water sensitivity of the material as well as the negative effect related to the use of warm technologies.
Regarding the comparison between the various warm additives, the overall results suggests that, for both limestone and basalt aggregates (coated or uncoated), the organic was the one characterized by the lowest performance, whereas the chemical additive provided acceptable performance in both dry and wet condition, comparable with the conventional HMA.

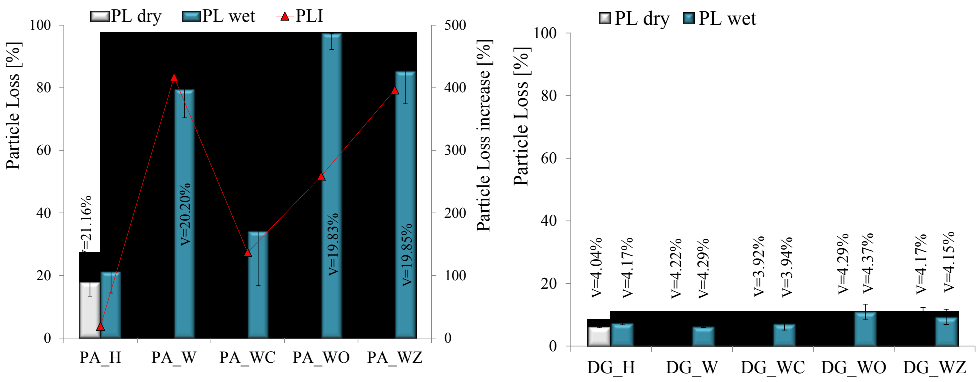
- Failure type analysis

Additives and production temperatures effects are more visible when the failure type is analyzed. First, it is worth noting that regardless of the aggregate type, in dry condition the

failure is always within the bitumen (i.e. cohesive type "C") except in the case of organic additive combined with limestone.
On the contrary, some differences in the material behavior can be detected in wet condition. For basalt aggregates in wet conditions (Figure 8.3c, 3d), the use of reduced temperatures (for both coated and uncoated surface) caused a change in the failure type, from cohesive failure within the bitumen (B_H_U and B_H_C) to failure at the interface bitumen-aggregate (B_W_U and B_W_C). This behavior indicates that the cohesion threshold within the bitumen was higher than the adhesion bond strength in wet conditions, making the failure at the interface bitumen-aggregate system more likely. The adding of WMA additives did not help to restore better interactions between the two component materials and the detected failure remained at the interface.
For limestone aggregates in wet conditions (Figure 8.3a, 8.3b), the reduction of the production temperatures did not cause any change in the failure type (within bitumen for both L_H_U, L_H_C and L_W_U, / L_W_C), whereas the use of WMA technologies appeared detrimental. In fact, the failure detected in wet conditions using uncoated limestone and WMA additives was at the interface. However, the use of coated limestone aggregates was helpful to limit the de-bonding phenomenon of the bitumen-aggregate system when combined with chemical and zeolite additives which showed failure within the bitumen also in wet condition (Figure 8.3b). Thus, the bond strength reduction due to water conditioning experienced by the system bitumen-coated limestone aggregates is principally due to the effects of moisture on inner cohesion of the bituminous component and depends only on the virgin bitumen used.
The latter finding could be partially explained considering that the oxidation process experienced by the artificial RAP surface reduces the free radicals of the material, making the coated aggregate more resistant to stripping than the uncoated one (Little & Jones 2003). Moreover, results allow to state that the thin film of aged bitumen coating the aggregate surface is likely able to partially reactivate (even at reduced temperature) and develop chemical interactions with the applied bitumen (with and without WMA additive). These interactions were significant in case of chemical additive and zeolite whereas they were less pronounced in case of organic additive.

Cantabro test results

Cantabro tests results in terms of particle loss average values are shown in Figure 8.22 for porous asphalt mixtures and for dense-graded mixtures in both dry and wet conditions. The corresponding mean air voids content of each mixture are shown in the figure along with the error bars reporting standard deviation values for each testing condition. It is important to underline that, for both mixture types, the air voids contents of mixtures were similar, meaning that the Cantabro tests results can be considered fully comparable as previously highlighted.



(a) (b)

Figure 8.22 Cantabro test results: (a) porous asphalt mixtures; (b) dense-graded mixtures.

In case of porous asphalt mixtures, similar particle loss values were obtained in dry conditions for all materials, demonstrating that the raveling resistance is not significantly influenced by reduced production temperatures when the effect of water is not considered. Moreover, results were entirely consistent with international requirements for high traffic highways corresponds to a maximum of 20% loss (Alvarez et al. 2010, Kline & Putman 2011), with the exception of WMA mixtures including organic additive. The effect of water is quantified for PA mixtures by means of the particle loss increase (PLI) parameter, evaluated as the difference between PL values measured in dry and wet condition with respect to the PL value after dry conditioning (Figure 8.22a).

Results in wet conditions as well as PLI values demonstrated that all WMA mixtures (with and without additive) were significantly susceptible to the detrimental effect of water in terms of raveling resistance and their behavior was considerably worse than the one shown by the reference HMA. In particular, the presence of the organic additive and the zeolite did not allow any improvement in terms of water susceptibility as their performance in wet conditions were even worst that the WMA mixture prepared without any WMA additive. Only the porous asphalt mixture including the chemical additive (PA_WC) showed limited particle loss values also after the wet conditioning ensuring adequate water resistance, although it does not guaranteed comparable performance than the HMA. Such outcomes confirm previous results (Hamzah et al. 2011, Frigio et al. 2015) suggesting that raveling resistance and water susceptibility are major concerns that affect WMA mixtures. Anti-stripping agents could be added in order to improve performance, especially when organic additives are used.

In case of dense-graded mixtures, the reduced production temperatures did not significantly affect the performance since the particle loss values were found to be comparable between HMA and WMA mixtures in both dry and wet conditions. The use of the organic additive and the zeolite led to a slight increase in particle loss values, whereas the chemical additive

ensured even better raveling resistance than the HMA mixture in both dry and wet conditions.
Overall Cantabro test results showed that the effect of the different WMA additives on the raveling resistance performance was similar in case of porous and dense graded mixtures. In particular, the chemical additive was the only one that ensured good performance in both dry and wet conditions.

Water susceptibility and WMA effects

The water susceptibility expected for porous and dense graded mixtures prepared with WMA technologies can be analyzed by correlating adhesion properties and raveling resistance. It is well recognized that adhesion properties between aggregates and bitumen strongly influence the raveling resistance of asphalt mixtures. However, many aspects related to the mixture type can contribute to determine the overall material response. In this sense, basalt and limestone aggregate selected for the BBS tests were also used for producing porous asphalt mixtures and dense-graded mixtures, respectively. Moreover, the SBS polymer modified bitumen as well as the WMA additives and the production temperature adopted to produce BBS and Cantabro specimens were the same. Thus, the materials of the two set of specimens (BBS and Cantabro) can be considered fully consistent. Furthermore, all tests were carried out at the same temperature equal to 25°C and the same protocol was adopted for conditioning both dry and wet specimens. On these bases, it was possible to relate BBS test results of basalt aggregate to Cantabro test results of PA mixtures, whereas BBS test results of limestone aggregate were compared to Cantabro test results of DG mixtures.
To properly compare the results of the two tests, the presence of different dosages of recycled materials within both PA and DG mixtures (15% for PA mixtures and 25% for DG mixtures) was taken into account. In this sense, BBS test results in terms of POTS obtained for the uncoated substrate (virgin aggregate) and the corresponding coated configuration (RAP aggregate) were proportionally combined in order to consider the presence of a certain amount of RAP within a given asphalt mixture as following specified:

$$POTS_{PA} = 0.85 \times POTS_{BU} + 0.15 \times POTS_{BC} \tag{8.1}$$

$$POTS_{DG} = 0.75 \times POTS_{LU} + 0.25 \times POTS_{LC} \tag{8.2}$$

where $POTS_{PA}$ and $POTS_{DG}$ are the BBS test results which simulate the recycled PA and DG mixtures analyzed in this study, respectively; $POTS_{BU}$ and $POTS_{LU}$ are the BBS test results for uncoated substrates in case of basalt and limestone aggregates, respectively; $POTS_{BC}$ and $POTS_{LC}$ are the BBS test results for coated substrates in case of basalt and limestone aggregates, respectively.

The correlation between adhesion and raveling test results is reported in Figure 8.23 for both PA and DG mixtures and for all test conditions.

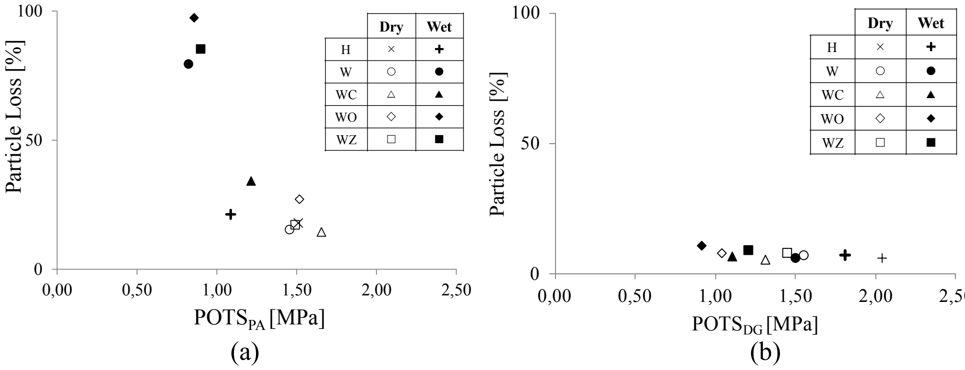


Figure 8.23 BBS vs Cantabro tests results: (a) basalt/PA mixtures; (b) limestone/DG mixtures.

A clear distinction between dry and wet data both in terms of adhesion and mechanical performance (Figure 8.23a) was detected in case of PA mixtures, which showed a significant decrease in raveling performance and lower adhesion properties in wet conditions compared to dry conditions, meaning that the bonding between material components plays a fundamental role when water effects are considered. In fact, PA mixtures are characterized by few and small contact regions between aggregate particles through asphalt mastic due to the high void content of the mixtures. As a consequence, the water can enter in the mixture pores leading to an easy deterioration of the particles bonding. Moreover, it is possible to notice that in the case of PA mixtures the WMA additive type considered strongly affected the mechanical response in wet condition (equal value of adhesion bond corresponded to significant differences in particle loss), whereas in dry condition all PA mixtures were characterized by similar adhesion and raveling results. In particular, among the studied WMA additives, the chemical was the only able to provide good performance in both dry and wet configuration.

On the contrary, in case of DG mixtures the significant difference in the adhesion properties detected through the BBS tests had limited effects on the raveling response of the mixtures, regardless of production temperatures or WMA additives. The water susceptibility was moderated and not affected by the WMA additive type (Figure 8.23b). Again, this finding can be explained in the light of aggregate structure of dense graded mixtures that limited the water access through the pores so preventing potential detrimental water effects.

These findings suggest that adhesion properties play a fundamental role on the development of the raveling resistance of bituminous mixtures, particularly when water effects are considered. Such effects were evident in case of porous asphalt mixtures where the aggregate structure allows continuous and pronounced exposure of the component materials to the water which does not occur in case of dense graded mixtures.

8.3.4 Conclusions

The present experimental study showed that the production of WMA mixtures at reduced temperature may affect adhesion properties between aggregates and bitumen with consequences on raveling resistance as well as water susceptibility. However, other factors (i.e. mineralogical nature of aggregates, volumetric properties, internal aggregate structure) can play a fundamental role on the overall mechanical response. In particular, RAP material is able to partially compensate this loss in performance thank to the presence of pre-coated aggregates which adhere more easily to the virgin bitumen even at reduced production temperatures. Moreover, RAP aggregates provide a water-resistant surface less prone to undergo water damage. Such an effect was found to be significant especially when the chemical WMA additive was used with major benefits on water susceptibility.
Good correlation between adhesion properties and raveling resistance was found mainly in case of porous asphalt mixtures: a very severe decrease in raveling performance with the loss in adhesion properties was detected when these mixtures were subjected to wet condition. Thus, such kinds of WMA technologies are not suggested for porous asphalt WMA mixtures when good water resistance is needed. Contrarily, the aggregate structure of dense graded mixtures prevents from significant water susceptibility and reduces the impact of different WMA technologies on the overall results.

Chapter 9.

Laboratory investigation of PA mixtures using different WMA additives

9.1. Objectives

The objective of this experimental study is to evaluate the feasibility to reduce the production temperatures of good quality recycled porous asphalt mixtures.
In this sense, the production temperatures were reduced of 40°C and different WMA additives were added to the PA mixtures. The performance of the different WMA mixtures produced at reduced temperatures (130°C-120°C) were compared to those of a reference HMA mixture produced at standard temperatures (170°C-160°C) in order to evaluate the effectiveness of WMA additives for ensuring adequate properties. Three WMA additives were selected as representative of the three main WMA techniques available on the market: one organic additive, one chemical additive and one zeolite as a water-containing foaming technique.
The research project consisted in two main phases. The first part was entirely carried out in Polytechnic University of Marche and focused on the evaluation of durability and water susceptibility as they are the main failure causes in PA (section 9.2). Acceptability, durability, ravelling and fracture resistance of the mixtures are evaluated through Indirect tensile strength tests, particle loss (Cantabro) tests, semi circular bending (SCB) tests and repeated indirect tensile tests that were carried out in both dry and wet conditions. Moreover, compactability properties of HMA and WMA recycled PA mixtures were compared.
The second part of this project involved an international collaboration between Polytechnic University of Marche and Vienna University of Technology and focused on long-term performance of WMA mixtures (section 9.3). It was already mentioned, in fact, that WMA additives have been developed over the last decades and long term analysis are still not available. As a consequence, aging effects on WMA mixtures were evaluated by aging WMA porous asphalt mixtures by means of an innovative test procedure (Viennese Aging Procedure - VAPro). Short and long term performance of both WMA mixtures and bitumens were compared with performance of an equivalent HMA.

9.2. Water susceptibility evaluation

9.2.1 Materials

Five porous asphalt mixtures including 15% of coarse RAP aggregates were produced using the same mix design that was optimized in the previous part of the present research project (Frigio et al., 2013; Frigio et al., 2015). Based on previous results, a total bitumen content equal to 5.25% (by aggregates weight) was selected; even if the mix-design optimized is detailed described in section 4, a brief summary is also reported in this paragraph.

The RAP aggregates come from milled PA surface layers that were properly stored separately and sieved so that only the coarse aggregates (8/16 mm) can be used. Basalt virgin aggregates (8/16 mm – 5/11 mm – 0/4 mm), RAP (8/16 mm) and filler were combined obtaining a final grading curve in accordance to Italian technical specification for motorways. Moreover, 70% cellolose-30% glass fibres dosed at 0.3% by the aggregates weight were added to the mixtures. The virgin bitumen was a polymer (SBS) modified bitumen.

One HMA reference mixture and three WMA mixtures with different WMA additives were investigated; moreover, another mixture was prepared at reduced production temperatures without adding any WMA additives for comparison purposes. In this way, it was possible to isolate the effect of WMA additives from the effect of the temperatures reduction.

The reference mixture, hereafter named PA_H, was mixed and compacted at 170°C and 160°C respectively whereas all the other WMA mixtures were mixed at 130°C and compacted at 120°C. The WMA production temperatures were selected 40°C lower than the standard temperatures in order to ensure significant emission reductions and cost savings as well as a comfortable working environment. In particular, the aggregates were heated at 130°C for about 3 hours whereas the bitumen was heated at 170°C in order to achieve a proper fluidity.

The four WMA mixtures were prepared as specified below:

- one mixture, hereafter named PA_W, was prepared without including any WMA additive;
- one mixture, hereafter named PA_WO, was prepared including an organic wax additive dosed at 3.0% by bitumen weight. In the laboratory operations, the additive was previously added to the hot virgin bitumen and mixed for about 15 minutes right before the mixture preparation;
- one mixture, hereafter named PA_WC, was prepared with a chemical additive dosed at 0.5% by bitumen weight. The chemical additive selected includes surfactants, emulsification agents, aggregates coating enhancers and anti-stripping agents; the presence of such chemical additive does not change considerably the bitumen viscosity but improves the ability of bitumen to coat aggregate particles by reducing the slip forces at the interface. It is produced from ammine substances and it is a viscous liquid at 25°C with a density of

about 1.0 g/cm3. It is characterized by a viscosity of 450 cP at 15°C, a pour point of about -8°C and a flash point higher that 140°C.
In the laboratory operations, the additive was previously added to the hot virgin bitumen and mixed for about 15 minutes right before the mixture preparation, according to the producer recommendation;
- one mixture, hereafter named PA_WZ, was prepared with a zeolite additive dosed at 0.3% by mixture weight. In the laboratory operations, the zeolite was added directly to the asphalt mixture in the mixing chamber, according to the producer recommendation.

9.2.2 Experimental investigation

It has been established from various studies (Hurley and Prowell, 2005a; Hurley and Prowell, 2005b; Hurley and Prowell, 2006) that lower mixing and compaction temperatures used when producing warm asphalt may increase the potential for moisture damage (Rubio et al., 2012; Capitão et al., 2012). Reduced production temperatures, in fact, could cause poor bond strength due to incomplete and not homogeneous coating of aggregates by bitumen as well as a not proper drying process of wet aggregate particles. Low adhesion properties mean higher water susceptibility which in turn implies stripping of bitumen from the aggregate surface or cohesive failure within the bitumen leading to a rapid deterioration of overall pavement performance and durability. Given this background, and considering the fact that moisture susceptibility of PA is one of the major issues to be closely investigated, the main focus of this first phase of the experimental study was the water resistance capability of warm produced PA mixtures. Thus, in order to evaluate the performance reduction due to water damage, mechanical properties of the studied mixtures were measured at both dry and wet conditions.
All the specimens were compacted using the gyratory compactor at fixed height or fixed gyrations (varying as a function of the test method).
The compactability properties of the WMA mixtures were evaluated by comparing their capability to compact at reduced temperature to that of a similar HMA mixture at standard temperature. In particular, the Compaction Energy Index (CEI) was calculated for each specimen as the area under the gyratory compaction curve that is considered to represent the work applied by the roller for compacting the mixture to the required density just before traffic opening, according to Mahmoud and Bahia (2004). In this sense, in case of porous asphalt mixtures CEI can be calculated from the 8th gyration (considered to simulate the effort applied by the paver) until a density of 80% of the maximum specific gravity (Gmm) is reached (Fig. 2), according to Goh and You (2012). Asphalt mixtures with lower value of CEI are desired since they have better compaction properties (Mahmoud & Bahia, 2004). In this study, CEI values were calculated for both HMA and WMA mixtures as the average of eight repetitions in case of small specimens (100 mm diameter) and as the average of four repetitions in case of large specimens (150 mm diameter).

After compaction, acceptability, mechanical performance and durability were evaluated. Indirect tensile strength tests, particle loss (Cantabro) tests and semi circular bending (SCB) tests were carried out in both dry and wet conditions. In particular, specimens of each mixture were conditioned in air (dry conditioning) or in water at 40°C (wet conditioning) for a period of 72 h, according to EN 12697-12 (Method A). In addition, repeated indirect tensile tests were performed and three different specimens conditionings were considered: dry condition (specimens were conditioned in air and tested in dry conditions), wet condition (specimens were kept in a water bath at 40°C for 72 h and tested in dry conditions at 20°C) and submerged condition (specimens were kept in a water bath at 40°C for 72 h and tested while completely submerged in water at 20°C). The stiffness moduli were previously measured in order properly divided the specimens of each mixture into three classes characterized by homogeneous stiffness to be tested in the three different conditions. In this way, a reliable comparison between results can be made in order to evaluate the water susceptibility of the mixtures.
All the tests were already described in detail in paragraph 4.2.4 and the same test parameters were used. Table 9.1 summarized the overall experimental program.

Table 9.1 Experimental program.

Mixture	Indirect tensile test		Cantabro test		SCB test		Repeated indirect tensile test		
	T=25°C		T=25°C		T=10°C		T=20°C		
	dry	wet	dry	wet	dry	wet	dry	wet	submerged
PA_H	4	4	4	4	4	4	3	3	3
PA_W	4	4	4	4	4	4	3	3	3
PA_WO	4	4	4	4	4	4	3	3	3
PA_WC	4	4	4	4	4	4	3	3	3
PA_WZ	4	4	4	4	4	4	3	3	3
TOTAL	**40**		**40**		**40**		**45**		

9.2.3 Results and analysis

Compactability properties

Compactability of the reference HMA and the WMA porous asphalt mixtures are investigated by means of CEI values. Mean results obtained on 100 mm and 150 mm diameter specimens are given in Figure 9.1 along with the corresponding error bars reporting the standard deviation value for each test condition.

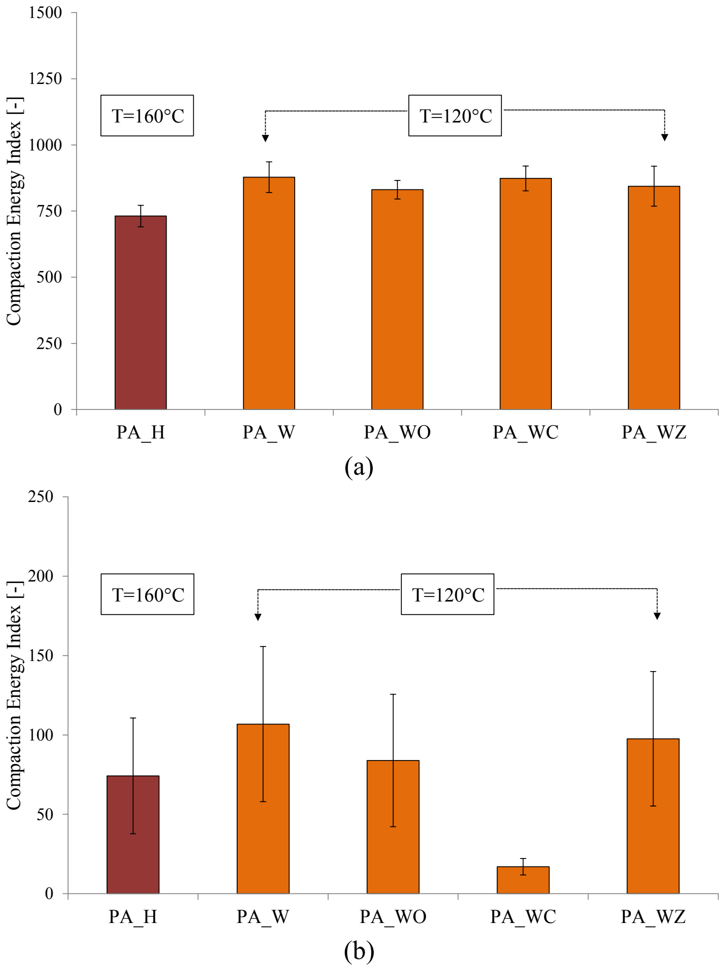


Figure 9.1 Compactability properties in terms of CEI for 100mm diameter specimens (a) and 150mm diameter specimens (b).

First of all, it is possible to notice that CEI values are significantly higher in case of 100 mm diameter specimens with respect to 150 mm diameter specimens, meaning that the mixture tends to be compacted easily when large molds are used.
The CEI results of the 100 mm diameter specimens (Figure 9.1a) are very similar between the different mixtures suggesting that the WMA additives do not seem to affect the compactability properties of PA mixtures.

On the other hand, as far as the 150 mm diameter specimens (Figure 9.1b) are concerned, differences in compactability properties of the mixtures can be highlighted: in particular, the presence of chemical additive significantly improves compactability properties of PA mixtures at reduced production temperature as CEI value is lower than all the other mixtures. Such a result confirms previous findings (Mo, Li, Fang, Huurman and Wu, 2012) and can be attributed to the presence of emulsification agents, surfactants, polymer and additives within chemical additive that helps improving coating, mixture workability and compactability.
The CEI results also highlight that only 150 mm diameter specimens should be used for evaluating mixtures compactability properties: in this case, in fact, actually mixtures properties can be evaluated since the influence of the mixture/mold interaction results less significant. Moreover, scale effect is more pronounced in case of 100 mm diameter specimens, especially in case of PA.

Indirect Tensile characteristics

Indirect tensile test results are summarized in Figure 9.2 in terms of mean Indirect Tensile Strength (ITS) in both dry and wet conditions along with the corresponding error bars reporting the standard deviation value for each test condition. Moreover, Indirect Tensile Strength ratio (ITSR) parameter is also shown as it evaluates water sensitivity of mixtures.
Reduced production temperatures of WMA mixtures can lead to an incomplete aggregate coating and a reduced short-term aging effect; thus lower ITS values can be expected for WMA mixtures.

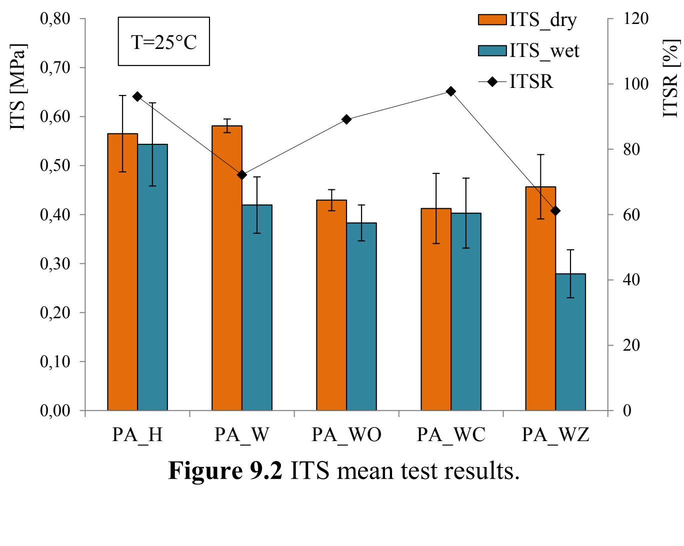


Figure 9.2 ITS mean test results.

Results in dry conditions show that only the mixture PA_W reaches similar performance to the reference mixture whereas all the other WMA mixtures demonstrate a significant reduction in ITS values. Such a decrease in performance for WMA mixtures including WMA additives with respect to PA_H and PA_W may be attributed to the presence of the additives that leads to adjustment of various bitumen properties that can affect mixtures performance. However, all mixtures satisfy acceptance requirements prescribed in dry conditions by Italian technical specification for PA mixtures in motorway pavements (ITS $\geq$ 0.40 MPa).
Results in wet conditions show a significant reduction in ITS values for all WMA mixtures with respect to the HMA one; such a result suggests that reduced production temperatures do not allowed the development of good adhesion properties between bitumen and aggregates since the presence of water affects substantially mixtures performance, when compared to the reference mixture.
Finally, moisture susceptibility can be easily evaluated throughout the ITSR parameter. As expected, the PA_W suffers severe water damage since it does not include any WMA additives that can help proper bitumen coating of aggregate particles and improve mixtures workability, as well as adhesion properties. It is interesting to notice that PA_WZ demonstrated the worst reduction due to the effect of water ($\approx$ 60%): such a result confirms previous studies that demonstrated that the use of zeolite can cause stripping and adhesion problems leading to significant moisture susceptibility if anti-stripping additives are not added (Austerman et al., 2009; Capitão et al., 2012; Kheradmand et al., 2014). It can be hypothesized, in fact, that some of the water trapped in the zeolite structure did not generate foam during the production process and remained within the mixture.
The use of organic additive lead to a limited reduction of water resistance whereas the use of chemical additives ensures results comparable to the HMA mixture in terms of moisture susceptibility. This outcome can be attributed to the fact that the chemical additive contains adhesion promoters and surfactants that improve the water sensitivity of the final WMA mixture.

Cantabro test

Cantabro test results in terms of particle loss mean values are shown in Figure 9.3, for both dry and wet conditions. The corresponding mean air voids content of each mixture are reported along with the corresponding error bars reporting the standard deviation value for each test condition.

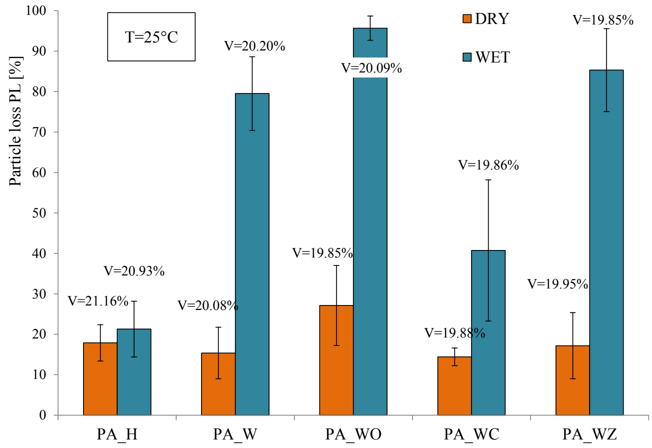


Figure 9.3 Cantabro mean test results.

The air voids content of mixtures were very similar, meaning that the Cantabro tests results can be considered fully comparable.
In dry conditions, mixtures demonstrate similar particle loss results that are entirely consistent with international requirements for high traffic highways corresponds to a maximum of 20% (Alvarez et al., 2010; Kline & Putman, 2011). Such a result suggests that the reduced production temperatures do not affect the resistance to ravelling of PA mixtures if dry conditions are considered. Only the presence of organic additive (PA_WO) does not guarantee adequate raveling resistance in dry conditions.
On the contrary, Cantabro test in wet conditions show that the presence of water causes a significant reduction of WMA mixtures resistance to ravelling compared to the reference mixture. Only the reference mixture that was produced at standard temperatures, in fact, is able to guarantee international requirements for high traffic highways in wet conditions corresponds to a maximum of 35% (Alvarez et al., 2010; Kline & Putman, 2011) whereas the WMA mixtures significantly suffer the effect of water. Only the PA_WC shows adequate particle loss values also in wet conditions: the presence of the chemical additive ensured good particle bonding that is not compromised by reduced production temperatures, confirming previous ITS test results. The other WMA mixtures experience an extensive damage due to the effect of water and the presence of both organic additive and zeolite does not lead to any performance improvement. In particular, almost all the PA_WO specimens subjected to the wet conditioning were completed destroyed during Cantabro tests.
Such a result suggests that anti-stripping agents are needed in order to improve resistance to ravelling and moisture susceptibility of WMA mixtures, especially when organic additives are used. In fact, a previous study (Hamzah et al., 2011) demonstrated that the reduction of

the water damage in terms of abrasion loss is highly related to the anti-stripping type as well as the compaction temperature.

Fracture resistance – SCB test

Semi circular bending (SCB) tests are carried out in order to analyze crack propagation potential in relation to the presence of different WMA additives in porous asphalt mixtures and the effect of reduced production temperatures. Fracture failure represents, in fact, one of the major distresses that can affect asphalt pavements and needs to be closely investigated in order to ensure layers durability.
Mean values of fracture toughness K for all mixtures in both dry and wet conditions are shown in Figure 9.4 along with the corresponding error bars reporting the standard deviation value for each test condition; the water sensitivity parameter calculated as the percent ratio between Kwet and Kdry is also shown.

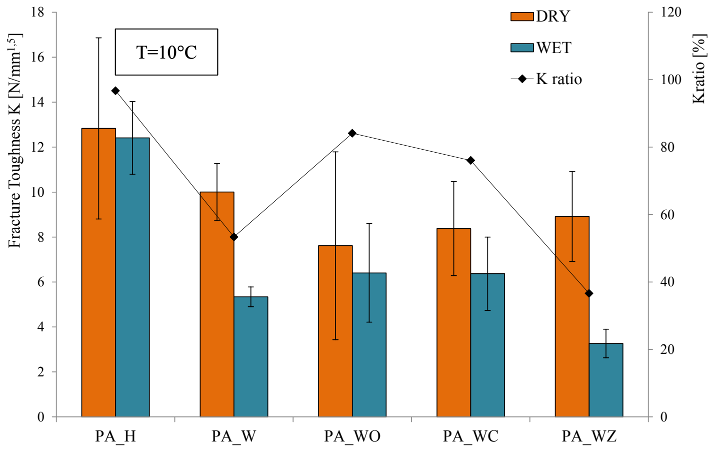


Figure 9.4 SCB mean test results in terms of fracture toughness.

Resistance to crack propagation significantly decreases in the case of WMA mixtures, in both dry and wet conditions. Such a result can be attributed to the reduced production temperatures of WMA mixtures that do not induce a full short-term aging process during mixing and compaction phases, leading to lower bitumen stiffness. In particular, the presence of WMA additives leads to a slight decrease in dry conditions performance when compared to PA_W, confirming previous ITS results. On the other hand, the presence of organic and chemical additives allows a limited reduction in crack propagation resistance due to the effect of water. Water sensitivity of all WMA mixtures, in fact, is lower than the reference mixture, confirming Cantabro and ITS results; however, the PA_WO and the

PA_WC demonstrate a good resistance to the water effect improving significantly the Kratio with respect to the PA_W.
Furthermore, the energy dissipation is also analyzed by means of the total fracture energy parameter (G) that represents the work required to increase the fractured surface until complete failure. Mean values of fracture energy G for all the mixtures in both dry and wet conditions are shown in Figure 9.5 along with error bars reporting the minimum and the maximum value obtained for each testing condition; the water sensitivity parameter calculated as the ratio between Gwet and Gdry is also reported.

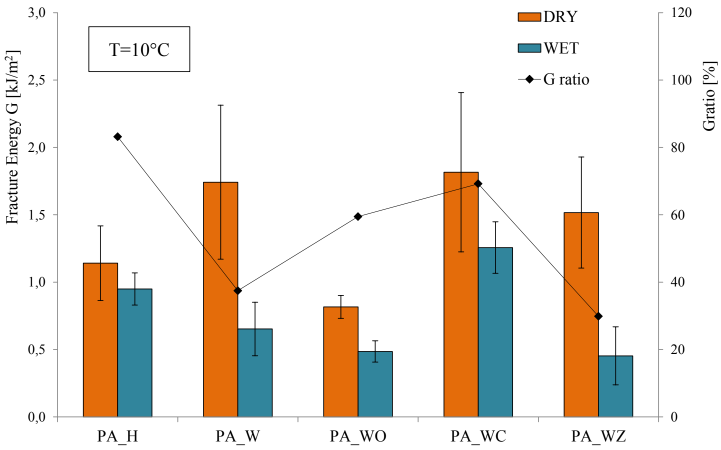


Figure 9.5 SCB mean test results in terms of fracture energy.

Figure 9.5 shows that WMA mixtures outperform the reference mixture in dry conditions since they dissipate more energy before failure, with the exception of the PA_WO. On the other hand, the water effect is more severe for WMA mixtures, particularly for the mixture including zeolite and the mixture that does not include any WMA additive, as expected. The chemical additive is the only WMA additive that guarantees adequate performance in both dry and wet conditions, with acceptable water damage resistance.

<u>Repeated indirect tensile test</u>

Repeated indirect tensile tests are performed under dry, wet and submerged conditions in order to investigate water damage resistance of the PA mixtures under repeated traffic loads. As proposed by previous studies (Kim & Coree, 2005; Poulikakos & Partl, 2009), in fact, the specimens need to be immersed in water throughout tests in order to closely simulate real field conditions.
The number of pulses at failure is taken into account for each specimen. Figure 9.6 reports the mean results along with the corresponding error bars reporting the standard deviation

value for each test condition (i.e. dry conditioning and dry test, wet conditioning and dry test, wet conditioning and submerged test).
The number of pulses at failure obtained in dry conditions for the WMA mixtures including organic, chemical as well as zeolite additives are comparable or even higher that results of the reference mixture PA_H. The WMA mixture PA_W produced at reduced temperature without adding any WMA additive demonstrates very low resistance, meaning that the presence of WMA additives helps improving the mixtures performance in dry conditions. On the other hand, the results in wet and submerged conditions highlight once again the weakness of the WMA mixtures as their performance is significantly affected by the presence of water.

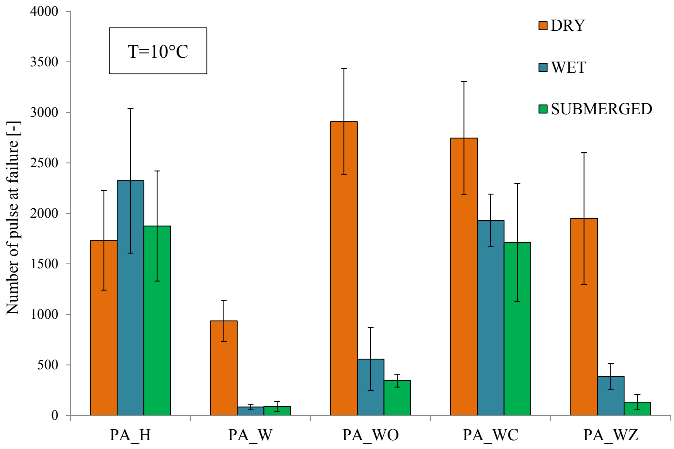


Figure 9.6 Repeated indirect tensile test results.

In order to quantify the loss in performance due to moisture damage, the percentage pulse decrease (PPD) is calculated for the wet and for the submerged condition compared to the dry condition as defined in Equation (9.1) and Equation (9.2) respectively:

$$PPD_{wet} = \frac{N_{wet} - N_{dry}}{N_{dry}} \tag{9.1}$$

$$PPD_{submerged} = \frac{N_{submerged} - N_{dry}}{N_{dry}} \tag{9.2}$$

where N_{dry} is the pulse number at failure in dry condition, N_{wet} is the pulse number at failure in wet condition and $N_{submerged}$ is the pulse number at failure in submerged condition.

Mean results (Table 9.2) demonstrate that in both wet and submerged conditions, all WMA mixtures suffer significant loss in performance due to the presence of water whereas the reference mixture is not affected. In particular, the PA_W demonstrates the worst water damage resistance whereas the presence of the organic additive and the zeolite slightly improves the mixtures performance. Only the PA_WC mixture including the chemical additive demonstrates low water susceptibility, preserving good resistance performance even in the submerged conditions.

Table 9.2. Percentage pulse decrease in wet and submerged conditions.

Mixture	PPD_{wet} [%]	$PPD_{submerged}$ [%]
PA_H	34.0	8.2
PA_W	-91.0	-90.5
PA_WO	-80.8	-88.2
PA_WC	-29.7	-37.7
PA_WZ	-80.2	-93.3

9.2.4 Conclusions

This experimental study evaluates the feasibility to reduce the production temperature of good quality recycled porous asphalt mixtures (including 15% of coarse RAP aggregates). In this sense, performance of the different WMA mixtures produced at reduced temperatures (130°C-120°C) were compared to those of a reference HMA mixture produced at standard temperatures (170°C-160°C) focusing on the evaluation of durability issues as they are the main failure causes in PA. Three WMA additives were selected: one organic additive, one chemical additive and one zeolite. Based on the results obtained during the experimental program, the following conclusions can be drawn:
- WMA mixtures guarantee compactability properties similar to the HMA mixture; only the presence of chemical additive ensures an easily compaction of the PA mixture;
- all mixtures satisfy acceptance requirements prescribed in dry conditions in terms of ITS values; however, WMA mixtures show a significant decrease in performance with respect to the HMA mixture in both dry and wet conditions;
- WMA mixtures guarantee ravelling resistance values comparable to those of the HMA mixture only in dry conditions whereas the loss in performance due to the effect of water is consistent; only the presence of chemical additives shows the capability to avoid such drop in performance ensuring acceptable results also after the wet conditioning;
- resistance to crack propagation significantly decreases in case of WMA mixtures in both dry and wet conditions due to reduced production temperatures that do not induce a full short-term aging process during the mixing and compaction phases, leading to lower

bitumen stiffness. The presence of organic and chemical additives allows a limited reduction in crack propagation resistance due to the effect of water;
- the energy approach in terms of SCB tests shows that WMA mixtures, with the exception of the PA_WO, outperform the reference mixture in dry conditions since they dissipated more energy before failure. The water effect is very severe for WMA mixtures and only the chemical additive guarantees adequate performance in terms of water damage resistance;
- repeated indirect tensile tests show that the presence of all the tested WMA additives helps improving PA mixtures performance in dry conditions whereas the results in wet and submerged conditions highlight once again the weakness of WMA mixtures as their performance are significantly affected by the presence of water.

The overall experimental program highlights the issues related to the reduced temperatures production of PA mixtures, particularly in terms of water sensitivity.

Only the presence of chemical additive in the mixture seems to ensure adequate water resistance, although it does not guaranteed comparable performance than the HMA mixture.

9.3. Aging effects on recycled WMA porous asphalt mixtures

9.3.1 Introduction and objectives

Short-term and long-term aging effects are two aspects that need careful investigation in case of warm mixtures (Xiao et al., 2015; Roja et al., 2016; Sadeq et al., 2016). During mixing and compaction, in fact, WMA mixtures are exposed to lower temperatures compared to conventional HMAs so leading to limited short term aging and premature rutting failure. In addition, concerns still exist related to in-service and long term performance since WMA technologies have been developed over the last decade and long term performance data are not available yet. Further investigations are needed in order to fully understand long term behaviour of WMA mixtures, especially in case of PA that are more sensitive to the detrimental effects of climate and traffic that lead to brittleness as well as ravelling (Molenaar et al., 2012).

Given this background, the objective of this experimental study is to evaluate aging effects on recycled porous asphalt mixtures produced at reduced temperatures using different WMA additives and including 15% of RAP. In this sense, long term aging was simulated in laboratory by means of an innovative test procedure (Viennese Aging Procedure - VAPro) (Steiner et al., 2015; Steiner et al., 2016). Mechanical laboratory tests were carried out on mixtures as well as bituminous components in order to evaluate possible links between bitumen and mixtures performance.

9.3.2 Materials

Materials preparation was entirely carried out in Polytechnic University of Marche (Ancona, Italy) using the same components already selected and described for the first part of this experimental program as well as the same laboratory protocols.
Four porous asphalt mixtures were produced in laboratory using the same mix design that was optimized in that was optimized in the previous part of the present research project (Frigio et al., 2013; Frigio et al., 2015) including 15% of selected RAP aggregates from milled porous asphalt surface layers. The total bitumen content was set equal to 5.25% by aggregate weight and it includes the aged bitumen within RAP (4.0% by RAP weight). The virgin bitumen (hereafter named "B_H_virgin") used to produce all PA mixtures was a polymer (SBS) modified bitumen (PG 82-16) whose main characteristics are already listed in Table 4.7.
One porous asphalt mixture (hereafter named "PA_H") was produced at standard mixing (170°C) and compaction (160°C) temperatures as reference HMA for comparison purposes.
Three different WMA porous asphalt mixtures were produced at reduced temperatures equal to 130°C for mixing and 120°C for compaction. WMA production temperatures were selected 40°C lower than conventional temperatures in order to ensure significant emission reductions and cost savings as well as comfortable and healthy working environment for operators (Rubio et al., 2012).
WMA mixtures were prepared using three different WMA additives, representative of the main categories nowadays available in the market, as specified below:
- one mixture (hereafter named PA_WO) was prepared including an organic additive dosed at 3.0% by bitumen weight;
- one mixture (hereafter named PA_WC) was prepared with a chemical additive dosed at 0.5% by bitumen weight.
- one mixture (hereafter named PA_WZ) was prepared with a zeolite additive dosed at 0.3% by mixture weight.

The three WMA additives selected are the same already used and described in Section 9.2.2. In laboratory, both organic and chemical additives were previously added to hot virgin bitumen (170°C) and mixed for 15 minutes right before mixture preparation at 130°C whereas the zeolite was added directly to asphalt mixture in the mixing chamber at 130°C, according to producer recommendations.
For both HMA and WMA mixtures, after the mixing phase at 170°C for HMA and 130°C for WMAs, short term aging was simulated in laboratory by keeping loose mixtures in a ventilated oven for one hour at the selected compaction temperature (Stimilli et al., 2016) (160°C for HMA and 130°C for WMAs). Afterwards, compacted and cored specimens were subjected to long term aging process by means of VAPro aiming at simulating the in service age hardening process caused by environmental conditions and traffic loads that continues during the pavement entire life. The VAPro test is described in detail in section 9.3.3.

The bituminous phase was then extracted and recovered from all studied PA mixtures, both before and after VAPro specimens aging in order to analyse short and long term aging effects on the rheological behavior. In this sense, the bitumen that was recovered before VAPro simulates short term aging since the specimen underwent only the oxidation due to mixing and compaction whereas the bitumen that was recovered after VAPro simulates long term aging. The bitumen was extracted according to EN 12697-3 with tetrachloroethylene (C_2Cl_4) as a solvent. The solvent-bitumen solution was distilled according to EN 12697-3 to recover bitumen samples. It is important to underline that extraction and recovery processes may affect the bitumen response due to the possible minimal presence of solvent residual. Nevertheless, since the same processes have been applied to all materials, results can be assumed as a relative index for comparison among the different materials.
Materials identification codes used for mixtures and corresponding bitumens are shown in Figure 9.7.

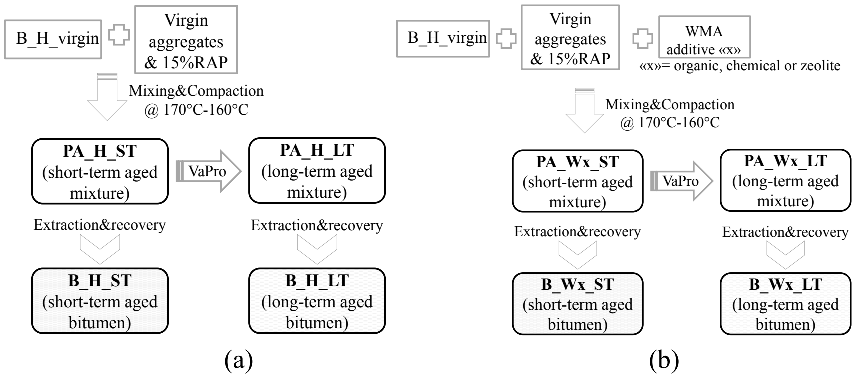


Figure 9.7 Investigated materials: HMA (a) and WMAs (b).

Mixtures aging, mechanical tests as well as bitumen extraction and rheological analysis were entirely carried out in Vienna University of Technology.

9.3.3 Aging process - Viennese Aging Procedure (VAPro)

Figure 9.8 shows the setup and equipment which was used for VAPro. Compressed air at ambient temperature is supplied from the local laboratory system and passed a pressure regulator, which ensures a constant flow rate (1 l/min) and gas pressure.

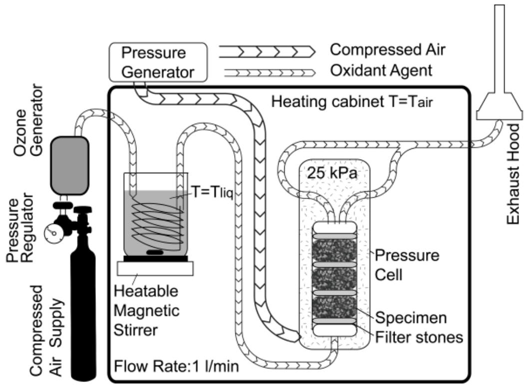


Figure 9.8 VAPro Setup (Steiner et al., 2015)

The subsequent ozone generator, using a dielectric barrier discharge tube (Kogelschatz, 2003), enriches the compressed air with ozone and nitrogen oxides. Using compressed air instead of pure oxygen helps to reduce operating costs and to enhance safety of VAPro. Furthermore, the presence nitrogen and nitrogen oxides enables reaction paths for the formation of ozone molecules, and this corresponds better to what happens in the field, respectively (Stanley, 1999). This gas mixture flows through a coiled Cu-Ni tubing, where it is heated up to Tliq= +70°C. Therefore, the coil is placed in a beaker glass, filled with vegetable oil and positioned on a heatable magnetic stirrer. Three HMA specimens are assembled within a triaxial cell be-tween four filter stones and are covered by an elastic membrane. A slight overpressure of 25 kPa was applied in the triaxial cell to force the gas mixture to flow through the specimen instead of passing on the outside. Since an open graded mixture design was used for this paper, the overpressure in triaxial cell was reduced compared to first applications (Steiner et al., 2015). Concurrently, due to high gas permeability, less flow gas pressure was needed, so that three specimens in series could be aged simultaneous. The triaxial cell and setup for heating up the gas are located in a heating cabinet at temperature Tair=60°C (Figure 9.9).

Figure 9.9 VAPro test

9.3.4 Test program and methods

The experimental program was focused on the evaluation of aging effect and long term performance of WMA bitumens and mixtures compared to HMA.
Rheological properties were measured on extracted bitumen from unaged and aged specimens. A Dynamic Shear Rheometer (DSR) with a plate–plate configuration was used to perform frequency sweep tests over a frequency range from 1 to 10 Hz at 10 tests temperatures from -10°C to 80°C. A 25 mm diameter plate and 1 mm gap were selected for high temperatures (from 50°C to 80°C) whereas an 8 mm diameter plate and 2 mm gap were selected for low and intermediate temperatures (from -10°C to 40°C). Frequency sweep tests were conducted in control strain within the linear viscoelastic range of materials.
As far as PA mixtures are concerned, all specimens were compacted using 150 mm diameter moulds in the gyratory compactor at a fixed height of 155 mm; samples were then cored and cut in such way that three specimens (100 mm diameter and 40 mm height) were obtained from each sample (Figure 9.10). Target air void content was selected equal to 20% for all mixtures.

Figure 9.10 Specimens preparation

Cyclic indirect tensile tests were carried out on specimens before and after long term aging at a temperature of 10°C by applying a sinusoidal load at three frequencies equal to 1, 5 and 10 Hz, according to EN 12697-26. From test data, dynamic modulus |E*| and phase angle φ were determined to examine the viscoelastic behavior of specimen (Di Benedetto et al., 2001). A series of pre-tests were carried out in order to determine the upper stress level of the sinusoidal load so that the elastic horizontal strain amplitude of the specimen during testing is between 5*10-5 m/m and 6*10-5 m/m. It was shown that repeated tests on the same specimen are possible with these loading conditions (Steiner et al., 2016). This is a necessary precondition since all specimens were tested twice, before and after laboratory aging (Steiner et al., 2016). Moreover, long term performance in terms of fatigue resistance was assessed by testing aged specimens. Fatigue tests were carried out at +10°C in stress controlled mode using an indirect tensile configuration. According to EN 12697-24, three diametral vertical loads were applied for each mixture in order to ensure horizontal deformation levels between 50 μm/m and 300 μm/m and a number of loading cycles in a range of 103 and 105. For each stress level, three repetitions were carried out.

9.3.5 Results and analysis

Aging effects on extracted bitumens

Complex modulus values G* were measured for bitumens extracted and recovered from specimens before (i.e. Short Term aging - ST) and after VAPro aging (i.e. Long Term aging - LT) by means of frequency sweep tests through DSR equipment. Results were used to determine master curves at a reference temperature of 10 °C, considering valid the Time-Temperature Superposition Principle. The modified CAM (Christensen Anderson and Marasteanu) Model (Bahia et al., 2001) was adopted to analyze test data and to represent the relationship between complex modulus norm and reduced frequency (Equation 9.3), following a shift factor variation based on the Williams-Landel-Ferry equation (Williams et al., 1955).

$$G^* = G_e^* + \frac{G_g^* - G_e^*}{\left[1 + \left(f_c/f'\right)^k\right]^{m_e/k}} \tag{9.3}$$

where G_e^* is the equilibrium complex modulus (f→0), equal to zero for bitumen; G_g^* is the glass complex (f→∞); f_c is the location parameter with the dimension of frequency; f' is the reduced frequency, function of temperature and strain; k and m_e are shape parameters, dimensionless.

Shifted experimental data as well as master curves are reported in Figure 9.11 for all materials. Moreover, the virgin bitumen used to produce all mixtures was tested and analysed likewise and its results are reported in the graphs (B_virgin) for comparison purposes since it represents the unaged condition (before short and long term aging).

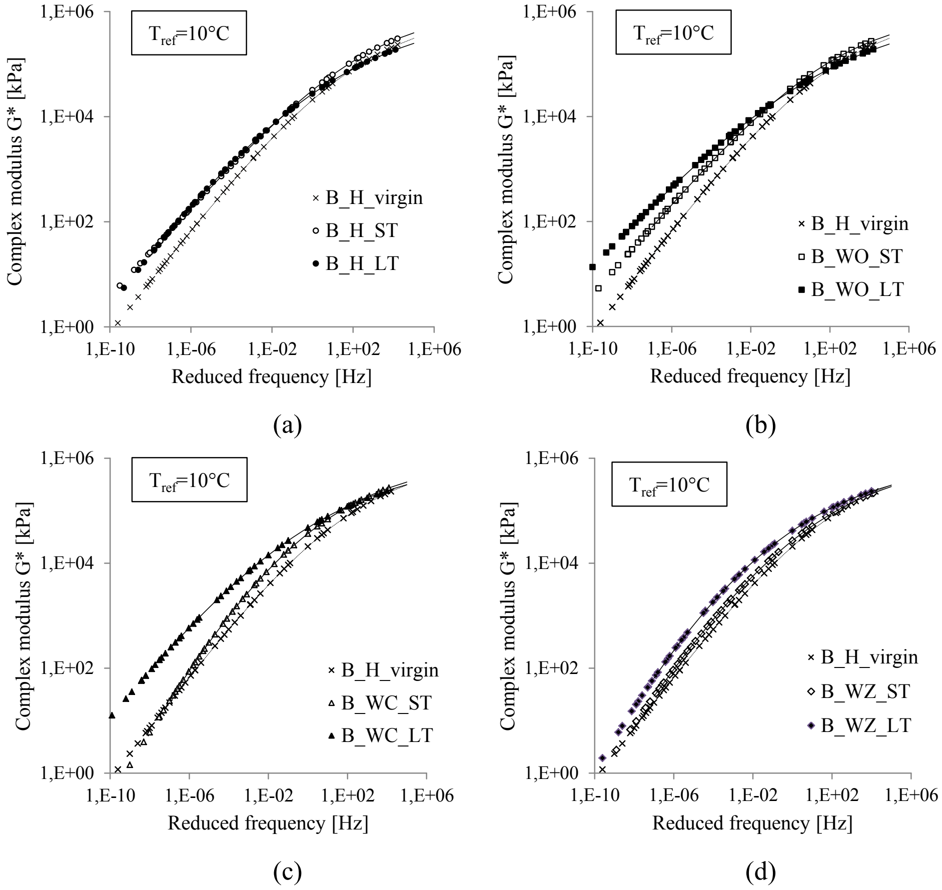


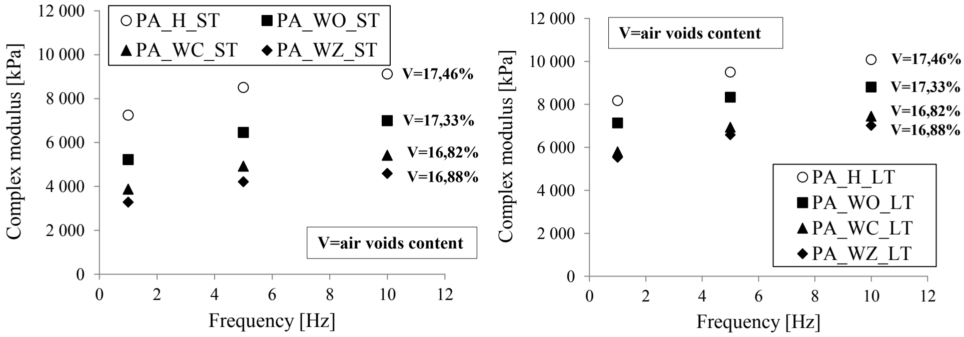
Figure 9.11 Master curves at reference temperature of 10°C of extracted bitumens before and after aging: B_H (a), B_WO (b), B_WC (c) and B_WZ (d).

As regards to short term aging process, aging effects can be deducted by comparing master curves of the virgin bitumen with the bitumens extracted from the specimens before VAPro aging (ST). The bitumen extracted from the HMA mixture is highly influenced by short term aging process as stiffness of B_H results significantly higher than the B_virgin (Figure 9.11a). Such a result can be attributed to the high standard production temperatures that were used to mix and compact HMA mixture specimens. In fact, during mixing time, the bitumen is in very thin films and it is exposed to air at 170 °C; such a process tends to highly stiffen the bitumen due to both air oxidation and loss of more volatile components. Afterwards, the age hardening of bituminous component continues during the transportation and laydown phase, although at a much slower rate (Brown et al., 2009); as

mentioned before, such a stage was properly simulated during laboratory operations by keeping the loose material in a ventilated oven at the selected compaction temperature before being compacted. As far as WMA bitumens are concerned, short term aging process leads to less pronounced increase in complex modulus when compared to HMA bitumen due to the reduced production temperatures used for WMA mixtures (Figure 9.11b, 9.11c and 9.11d). The short term aging stiffening effect results more remarkable in case of organic additive whereas lower complex moduli characterize bitumens including chemical additive and zeolite. The higher complex modulus measured for the WMA bitumen including organic additive can be attributed to the crystalline network structure that organic wax forms in the bitumen below its melting point so increasing bitumen stiffness (Rubio et al., 2012).
As far as long term aging process is concerned, it is possible to notice a clear difference in behaviour between HMA and WMA bitumens. HMA bitumen rheological properties are not affected by VAPro process since comparable G* values were measured for B_H_ST and B_H_LT whereas all WMA bitumens become significantly stiffer due to long term aging process. Such a result can be related to the different mixture production technologies (HMA and WMA) and, in particular, to the lower initial stiffness of WMA bitumens that make them more susceptible to aging effects.

Mixtures stiffness

Indirect tensile modulus results obtained at 10°C are reported in Figure 9.12 as average of 9 repetitions; tests were carried out at three different load frequencies equal to 1Hz, 5Hz and 10Hz. Stiffness values were measured before and after VAPro aging in order to evaluate the effect of short (ST) and long term (LT) aging on WMA mixtures. Stiffness values before VAPro aging (Figure 9.12a) are indicative of the behaviour of short term aged mixtures since the materials were subjected only to mixing and compaction procedures. At the same time, the oxidation that affects upper pavements layers during in-service life is simulated in the laboratory by means of VAPro procedure; thus, stiffness results for aged materials (Figure 9.12b) are representative of long term mixture properties. Moreover, the average air voids content of each mixture is reported in Figure 9.12.



(a) (b)

Figure 9.12 Mean stiffness results at 10°C of HMA and WMA mixtures before (a) and after (b) aging by means of VAPro procedure.

Results show that the stiffness of asphalt mixtures is highly dependent on load frequency as expected; in particular, the stiffness increases as the frequency increases for all mixtures studied. Such a trend is not affected by production temperatures or aging status.

Short-term aged results show that all WMA mixtures are characterized by lower stiffness values than the HMA mixture suggesting that the reduced production temperature could affect stiffness properties of warm mixtures due to the lower temperature-induced oxidation during the production phase. The presence of organic WMA additive partially compensates this stiffness gap; the higher stiffness of the WMA mixture prepared with organic additive with respect to the other WMA mixtures can be attributed to the higher bitumen stiffness, consistently with previous outcomes (§3.1). As a matter of fact, other researches (Zelelewa et al., 2013) found that asphalt mixtures prepared with organic WMA technology measured higher stiffness and improved fatigue cracking resistance than those prepared with other WMA technologies.

Long-term aged results show that, even after VAPro procedure, WMA mixtures do not achieve the same stiffness properties of HMA mixture. The effect of long-term aging is evident for all WMA mixtures but it is not sufficient to compensate the initial stiffness gap respect to HMA due to the reduced production temperatures. The only exception is represented by the WMA mixture including organic additive that show higher stiffness values with respect to the other WMAs, resulting in long term stiffness similar to the HMA mixture. As previously mentioned, the presence of organic additive demonstrate a higher initial stiffness both for bitumen and mixture due to the additive nature that tends to form a crystalline network within bituminous phase. However, it is important to note that such a behavior similar to HMA is not desirable in terms of durability since excessive long term stiffness can lead to premature fatigue and thermal cracking failure.

Moreover, a one-way ANOVA analysis at 95% confidence level is performed to verify statistical significance of results: the p-values are reported in Table 9.3 for short and long term aged mixtures. ANOVA tests confirm a significant difference in stiffness moduli in case of short and long term aged mixtures. In particular, all WMA mixtures are characterized by a significant lower stiffness than HMA and PA_WO demonstrates significant higher stiffness values than all the other WMA mixtures. On the other hand, no statistical significance can be detected between results of WMA mixtures including chemical and zeolite, regardless the aging processes.

Table 9.3. ANOVA test: influence of production temperature and additive type on mixtures moduli after short (left) and long (right) term aging.

SHORT TERM AGING			LONG TERM AGING		
	p-value	significant?		p-value	significant?
PA_H / PA_WO	1,1E-04	NO	PA_H / PA_WO	9,0E-03	NO
PA_H / PA_WC	3,6E-06	NO	PA_H / PA_WC	1,4E-04	NO
PA_H / PA_WZ	8,4E-09	NO	PA_H / PA_WZ	8,4E-07	NO
PA_WO / PA_WC	0,009	NO	PA_WO / PA_WC	0,019	NO
PA_WO / PA_WZ	1,3E-05	NO	PA_WO / PA_WZ	5,8E-04	NO
PA_WC / PA_WZ	0,2	YES	PA_WC / PA_WZ	0,6	YES

In order to evaluate and quantify long-term aging effect on HMA and WMA mixtures, the stiffness increase (ΔE*) due to VAPro aging procedure was calculated as follows:

$$\Delta E^* = \frac{E^*_{LT} - E^*_{ST}}{E^*_{ST}} \times 100 \tag{9.4}$$

where E^*_{LT} is the complex modulus of long term aged specimens after VAPro aging and E^*_{ST} is the complex modulus of short term aged specimens before VAPro aging.

The parameter ΔE* is represented in Figure 9.13 for all frequencies and mixtures. Results show that all WMA mixtures are more affected by long term aging than the HMA mixture. It is well known that age hardening that takes place during the entire pavement service life is highly influenced by the bitumen film thickness around aggregate particles and by the air void content which provides different entry of air, water and light (Brown et al., 2009). In the present study, both parameters can be considered constant since mix design and target air void content were the same for all studied mixtures. Thus, the different long term aging level can be only attributed to mixtures production technologies (HMA and WMA) and, in particular, to the lower initial stiffness of WMA mixtures that makes them more susceptible to aging effects. As a matter of fact, the effect of aging is found to be more pronounced for WMA mixtures including zeolite and chemical additive since they are characterized by lower stiffness before aging.

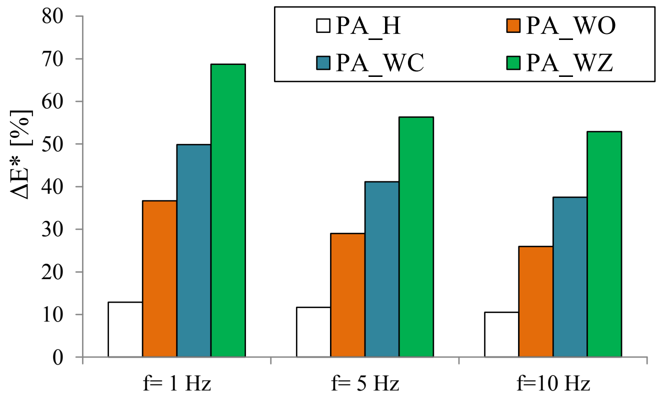


Figure 9.13 Aging effect on stiffness properties for HMA and WMA mixtures.

Such outcomes are consistent with the results obtained for bitumens as all WMA bitumens demonstrate to stiffen significantly more than HMA bitumen due to long term aging process. Thus, the extensive stiffening effect that characterize all aged WMA mixtures can be mainly attributed to the corresponding stiffening of bituminous components. In fact, long term aging effect of WMA bitumens including chemical additive and zeolite result more pronounced with respect to the bitumen including organic additive consistently with the recorded WMA mixtures stiffness increase (Figure 9.13).

Fatigue resistance

Fatigue results for long-term aged mixtures are shown in Figure 9.14 in terms of initial maximum horizontal strain as a function of number of cycles until failure. The fatigue failure of each specimen is determined as number of cycles to reach 50% of initial stiffness. Moreover, fatigue curves are expressed as a generalized relationship incorporating horizontal strain (ε), number of cycles at failure (N) and initial mixture stiffness (E). Many researchers, in fact, had shown that critical horizontal strain is highly influenced by the material stiffness that, as consequence, needs to be incorporated in the fatigue life prediction curve (Kingham, 1973; Mallick and El-Korchi, 2013).

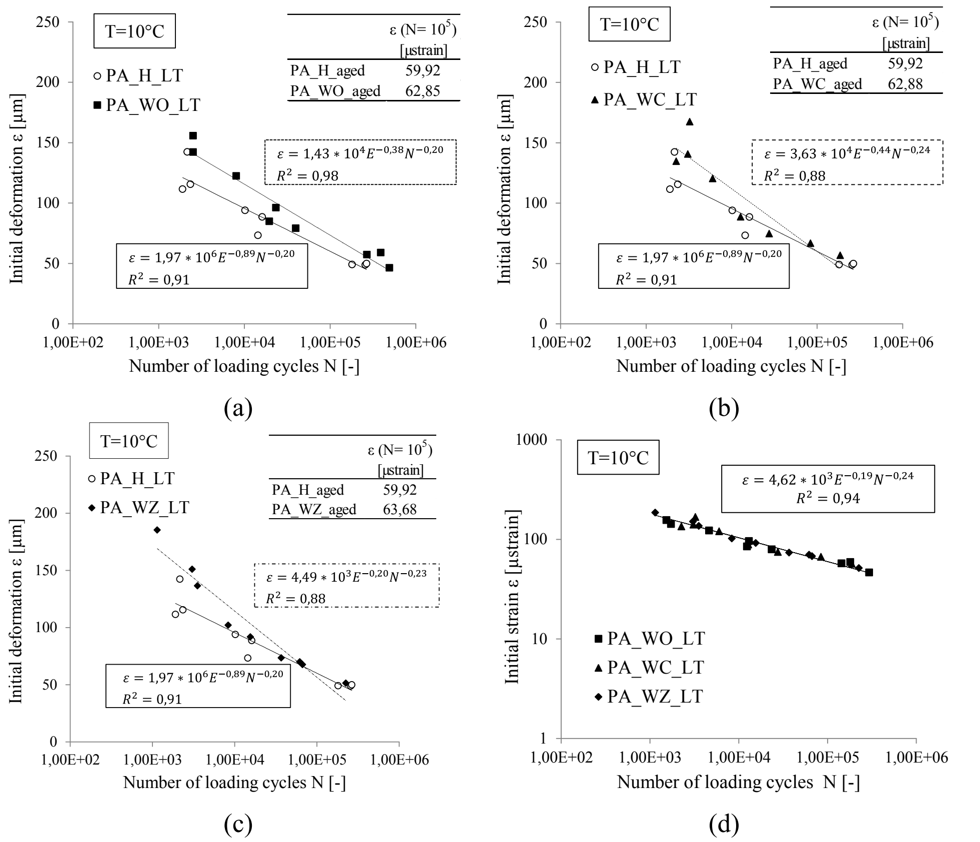


Figure 9.14 Fatigue curves of the HMA and WMA mixtures including organic additive (a), chemical additive (b) and zeolite (c). Fatigue results of all WMA studied mixtures (d).

The fatigue curve obtained for each WMA mixture, directly compared to reference HMA mixture results, is depicted in Figure 9.14a, 9.14b and 9.14c along with the corresponding regression coefficient (R^2). Results show that the WMA mixture including organic additive performs similarly to the HMA mixture in terms of fatigue resistance since they are characterized by the same slope parameter (Figure 9.14a). On the other hand, fatigue behaviour of WMA mixtures including chemical additive (Figure 9.14b) and zeolite (Figure 9.14c) is slightly different than the HMA mixture as the fatigue curves are characterized by higher slope parameters.

In order to directly compare mixtures fatigue behaviour, the horizontal deformation value related to a target number of cycles of 10^5 (selected as target fatigue life) is calculated as considered representative of the predicted deformation after an extensive traffic level. Such deformation parameters, reported in Figure 9.14a, 9.14b and 9.14c, show a full comparability of the studied mixtures as results obtained for all WMA mixtures and the reference HMA mixture were very similar. Thus, the lower stiffness of long term aged WMA mixtures compared to the mixture produced at standard temperature do not provide any benefits in terms of overall fatigue resistance. At the same time, lower production temperatures do not imply any detrimental effects in terms of long term fatigue performance.

Finally, all experimental data obtained for WMA mixtures are reported in the same graph in Figure 9.14d for an overall comparison. It is possible to notice that there is not clear distinction between data which can actually be fitted by a unique regression line. As a matter of fact, considering all test data as representative of a unique material a good regression coefficient is obtained, demonstrating that all mixtures are characterized by very similar fatigue behaviour. Such a result suggests that the fatigue behaviour is more influenced by production temperatures rather than WMA additive type.

9.3.6 Conclusions

Based on the results obtained during the experimental program, the following conclusions can be drawn:

- WMA bitumen and WMA mixtures are characterized by lower stiffness values than the HMA suggesting that reduced production temperature could affect stiffness properties of warm mixtures due to the lower temperature-induced oxidation during the production phase;
- extensive long term aging effects are measured in case of WMA bitumens and mixtures contrarily to HMA materials that are not significantly affected by VAPro process;
- stiffness values of WMA mixtures results lower than HMA mixture even after long term aging due to the initial stiffness gap; the presence of organic additive tends to increase bitumen stiffness and, as a consequence, mixtures stiffness;
- WMA mixture including organic additive performs similarly to HMA in terms of fatigue slope whereas the presence of chemical additive and zeolite leads to a slightly higher fatigue slope parameters.
- overall fatigue results show that long term fatigue performance of WMA mixtures are not significantly affected by low production temperatures or WMA additive type; similar results are found despite the different stiffness values of WMA and HMA mixtures.

Overall experimental results underline the different aging behaviour of WMA and HMA mixtures and the direct correlation with bituminous phase; despite differences in aging effects, long term performance in terms of fatigue resistance are not affected by production temperatures or WMA additive type.

The understanding of temperatures productions and aging effects on PA mixtures performance is a fundamental starting point in order to better interpret and analyse the overall properties of open graded WMA mixtures.

Chapter 10.

In plant production of warm PA mixtures using chemical WMA additives

10.1. Objectives

A previous laboratory investigation of PA mixtures produced at reduced temperatures using different WMA additives was carried out and detailed described in Chapter 9. In this sense, the production temperatures were reduced of 40°C and three WMA additives were selected as representative of the three main WMA techniques available on the market: one organic additive, one chemical additive and one zeolite as a water-containing foaming technique. The overall experimental program highlights the issues related to the reduced temperatures production of PA mixtures, particularly in terms of water sensitivity and only the presence of chemical additive in the mixture proved to ensure adequate water resistance, although it does not guaranteed comparable performance than the HMA mixture.

Based on this finding, a further research project was carried out with the aim at investigate the in-plant production at reduced temperatures of PA mixtures by means of WMA chemical additives. Thus, two chemical WMA additives available on the market were selected with different chemical compositions as well as operating mechanisms aiming at investigating their effectiveness on warm productions. PA mixtures were in-plant produced in order to check the feasibility of large-scale productions when significant reduced temperatures and RAP are concurrently employed. In summary, the main objective of the present experimental study is the evaluation of mechanical performance and durability of warm recycled OG mixtures produced by means of different chemical additives.

Moreover, WMA additives were recently introduced and, as a consequence, the use of WMA technologies to reduce mixtures production temperatures has been mainly investigated in the last years by means of laboratory studies (Mallick et al., 2008; Oliveira et al., 2012; Frigio and Canestrari, 2016). Only few experiences provide results of in-plant productions of WMA mixtures and in situ measurements, both essential to validate the potential of warm technologies. In fact, it is well known that large-scale productions and in-situ compaction present different challenges with respect to laboratory simulations (Sargand et al., 2012; Raghavendra et al., 2016). Therefore, researches focused on in-plant produced warm recycled mixtures are needed to bridge this lack.

10.2. Experimental investigation

10.2.1 Materials

Three open-graded mixtures were in-plant produced using the same mix-design optimized in previous laboratory and field studies to allow the substitution of 15% of virgin aggregates with RAP. Only coarse RAP (8/16 mm) coming from milled PA layers was used in order to better control volumetric properties as well as material characteristics of final PA mixtures. The optimum total bitumen content of all PA mixtures was found equal to 5.25% (by aggregates weight) and includes 4.65% of virgin bitumen and 0.6% of RAP bitumen; the virgin bitumen is a modified bitumen containing 3.8% of Styrene-Butadiene-Styrene (SBS) polymer. Moreover, 70% cellulose-30% glass fibres dosed at 0.3% by aggregates weight were added to each mixture to prevent draindown issues. The optimized mix-design as well as the materials selected for this experimental study are the same already described in Chapter 4.

One PA mixture, hereafter named "PA_H", was in-plant produced at standard temperatures equal to 170°C for mixing and 160°C for compaction and used as reference for comparison purposes. Moreover, two PA mixtures were in-plant produced at reduced temperatures equal to 130°C for mixing and 120°C for compaction by using two chemical additives identified as C1 and C2, (corresponding mixtures coded as "PA_WC1" and "PA_WC2 respectively). The C1 additive was dosed at 0.42% by virgin bitumen weight, whereas C2 was dosed at 0.70% by virgin bitumen weight, accordingly to the recommended range provided by the producers. In both cases, the additive was added to the virgin bitumen through a volumetric pump right before mixtures production without significant modification of the asphalt plant.

The two WMA additives were selected among various chemical additives available on the market so as to analyze products characterized by completely different operating mechanisms. The chemical additive C1 is based on ammine substances and acts as anti-stripping and surfactant agent as well as aggregates coating enhancer. It is a viscous liquid at 25°C with a density of about 1.0 g/cm^3, a pour point of about -8°C and a flash point higher that 140°C. On the other hand, the chemical additive C2 acts as viscous regulator and contains alkylates and fatty acids. It is an amber-colored, inodorous liquid at ambient temperature, insoluble in water and is characterized by a density of 0.86-0.90 g/cm^3 at 20°C, a flash point higher than 220°C and a boiling point between 300°C and 408°C. In order to better understand additives behavior, a preliminary chemical analysis on the two WMA additives was carried out. The Fourier Transform Infrared Spectroscopy (FT-IR) was performed on C1 and C2 additives (Figure 10.1) with the aim of obtaining basic but useful chemical information to better interpret overall performance of the final WMA mixtures. Using electromagnetic waves in a wide and continuous range of frequencies, FT-IR spectroscopy studies the interactions between the material and electromagnetic radiations. Fundamental vibrations (i.e. stretching and bending of chemical bonds, as well as rotational motions in molecules) are analyzed within the Middle Infrared Region (MIR), which includes the interval of wavelengths from 3 µm to 50 µm (extending from wavenumbers 4000 to 400 cm^{-1}). Signal interferences are generated by the interferometer at each wavelength within a selected interval of IR frequencies (or wavenumbers). A detector measures the intensity of the signal passing through the probing sample at each specific wavelength (or frequency, or wavenumber). These data are immediately processed by the Fourier transform mathematical function resulting in an IR spectrum. Therefore, in the absence of absorbing molecules, almost 100% IR light is transmitted to the detector (e.g. background sample); on the contrary, the intensity of IR light transmitted is lower when molecules enter a resonant state with specific frequencies of IR light (Chalmers and Griffiths, 2001).

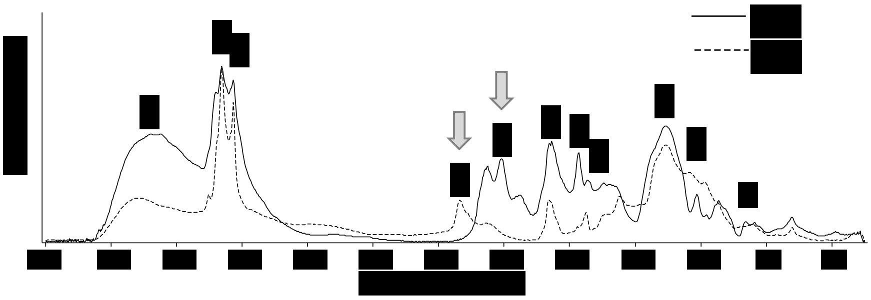


Figure 10.1 FT-IR analysis for the WMA chemical additives.

The FT-IR analysis (Figure 10.1) shows the presence of a considerable amount of water for both WMA additives (high absorbance at 3440 cm^{-1}). In case of additive C1, FT-IR results confirm the presence of ammine substances, as indicated by the absorbance peaks between 1650 cm^{-1} and 1550 cm^{-1}. Moreover, the high absorbance of C1 at 1610 cm^{-1} suggests the presence of aromatics which are substances highly compatible with the bitumen that can improve adhesion properties at the bitumen-aggregate interface. In case of C2, it is possible to notice the presence of chemical bonds C=O, as indicated by the high peak at 1730 cm^{-1}. The additive C2 could likely be the combination of different constituents such as antifoaming agents and organic substances.

10.2.2 Test program and methods

Mixtures produced at the asphalt plant were employed for building motorway trial sections whose in situ performance and durability will be monitored over the years. Furthermore, a certain amount of each mixture was taken during in-plant productions and laboratory compacted by means of a gyratory compactor. Compactability properties were evaluated through gyratory compaction data by calculating the Compaction Energy Index (CEI) as it

is linked to the work applied by the roller for reaching the required density of the mixture just before traffic opening (Mahmoud and Bahia, 2004). In case of open-graded mixtures, CEI index is calculated as the area under the compaction curve from the 8th gyration (simulating the effort applied by the paver) until the final density is reached (80% of the maximum specific gravity), according to Goh & You (2012). Lower values of CEI are desired since they indicate mixtures with improved compaction properties. After specimens compaction, mechanical performance and durability were investigated; in order to evaluate water susceptibility, some specimens of each mixture were conditioned in air (dry) at the specific test temperature and others in water (wet) at 40°C for a period of 72 h prior the dry conditioning, as suggested by EN 12697-12 (Method A). The detrimental effect of water, in fact, is one of the major concerns in case of WMA mixtures (Hasan et al., 2015; Buss et al., 2016), especially when porous asphalt is involved.
Acceptability, mechanical performance and durability were evaluated by means of Indirect tensile strength tests, particle loss (Cantabro) tests and semi circular bending (SCB) test, carried out in both dry and wet conditions. In addition, repeated indirect tensile tests were performed and three different specimens conditionings were considered: dry condition (specimens were conditioned in air and tested in dry conditions), wet condition (specimens were kept in a water bath at 40°C for 72 h and tested in dry conditions at 20°C) and submerged condition (specimens were kept in a water bath at 40°C for 72 h and tested while completely submerged in water at 20°C). The stiffness moduli were previously measured in order properly divided the specimens of each mixture into three classes characterized by homogeneous stiffness to be tested in the three different conditions. In this way, a reliable comparison between results can be made in order to evaluate the water susceptibility of the mixtures. All the tests were already described in detail in paragraph 4.2.4 and the same test parameters were used. For each test, a one-way ANOVA analysis at 95% confidence level was performed to verify the statistical significance of results. The overall experimental program is summarized in Table 10.1.

Table 10.1 Experimental program.

Test replicates										
Mixtures	ITS		Cantabro		SCB		ITSM	Repeated indirect tensile		
	T=25°C		T=25°C		T=10°C		T=20°C	T=20°C		
	dry	wet	dry	wet	dry	wet	-	dry	wet	submerged
OG_H	4	4	4	4	4	4	9	3	3	3
OG_WC1	4	4	4	4	4	4	9	3	3	3
OG_WC2	4	4	4	4	4	4	9	3	3	3

10.3. Results and analysis

10.3.1 Compactability properties

Compactability properties in terms of CEI values based on data acquired during the compaction of 150 mm diameter specimens are reported in Figure 10.2 as the average of four replicates; the standard deviation is also indicated through the error bars and ANOVA test results are shown in terms of p-value.

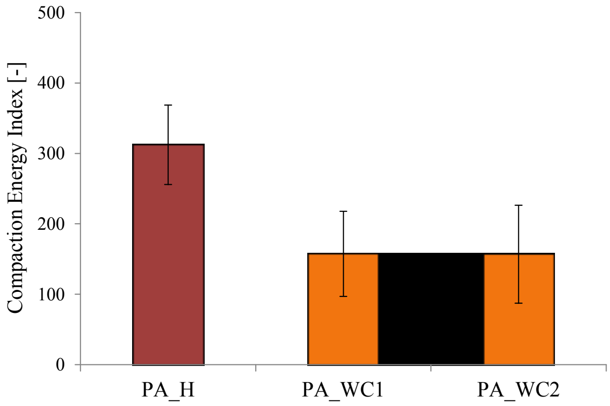


Figure 10.2 Mean results in terms of CEI.

Results clearly show a difference in compaction properties between the reference HMA mixture (PA_H) compacted at 160°C and the two WMA mixtures compacted at 120°C. The latter are characterized by significant lower CEI values than the HMA. Thus, both chemical WMA additives ensured better compactability with respect to the HMA mixture, despite the reduced temperature. The statistical analysis confirms the significant difference in CEI values between WMA and HMA mixtures, whereas no difference in compactability properties between the two WMA mixtures is detected. Therefore, it is possible to conclude that the two chemical additives were effective in terms of compactability, guaranteeing better properties than the reference HMA mixture.

10.3.2 Indirect Tensile characteristics

Indirect tensile strength test results in both dry and wet conditions are reported in Figure 10.3 as the average of four replicates; the standard deviation is also indicated through the error bars and ANOVA test results are shown in terms of p-value. The water susceptibility was taken into

account by means of ITS_Ratio, defined as the percent ratio between the indirect tensile strength of wet and dry specimens, in accordance to EN 12697-12.
Results in dry conditions are very similar between the three mixtures, as also indicated by the statistical analysis. Moreover, all mixtures satisfy typical acceptance requirements (0.4 MPa) meaning that the presence of both types of chemical WMA additives ensure adequate results in dry conditions in terms of ITS. Such an outcome suggests that in-plant production of recycled PA mixtures at reduced temperatures with WMA chemical additives is possible without compromising mixture performance when the effect of water is not considered. As far as results in wet conditions are concerned, it is evident a detrimental effect due to the presence of water for WMA mixtures. In fact, both WMA mixtures show lower ITS values in wet conditions than the HMA mixture, which did not suffered any moisture damage. Consequently, the ITS_Ratio of both WMA mixtures was found lower that the one of the reference mixture. Such a result can be attributed to the lower production temperatures adopted for WMA mixtures that do not allow the development of adequate adhesion properties at the aggregates/bitumen interface, which manifests its weakness mainly under the action of water. The presence of the chemical additives partially compensated such an effect (ITS_Ratio are still acceptable), but it did not ensure the same performance of the HMA mixture in terms of water susceptibility. The effect of the two chemical additives was very similar also in wet conditions and no statistical significance between them was found.

dry conditions		
	p-value	significant?
PA_H / PA_WC1	0,263	NO
PA_H / PA_WC2	0,131	NO
PA_WC1 / PA_WC2	0,970	NO

wet conditions		
	p-value	significant?
PA_H / PA_WC1	0,001	YES
PA_H / PA_WC2	0,013	YES
PA_WC1 / PA_WC2	0,244	NO

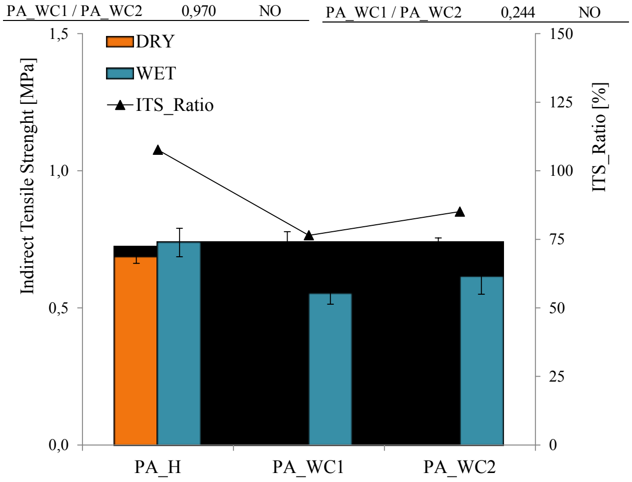


Figure 10.3 Mean results in terms of Indirect Tensile Strength tests.

10.3.3 Ravelling resistance

Raveling represents one of the major failure causes of porous asphalt mixtures and it is strongly affected by the effects of water. In this study raveling resistance was evaluated by means of Cantabro tests in both dry and wet conditions; results in terms of particle loss are represented in Figure 10.4 as the average of four replicates; the standard deviation is also indicated through the error bars and ANOVA test results are shown in terms of p-value. The mean air voids content of each mixture is also shown in the graph since it represents a parameter that highly influences Cantabro test results (Frigio et al., 2013).

Given the fact that all specimens are characterized by similar air voids content, raveling results can be considered fully comparable. Similar raveling performance are observed in dry conditions by WMA and HMA mixtures, demonstrating that low production temperatures does not affect adhesion properties and, consequently, raveling resistance of porous asphalt mixture when chemical additives are employed and the water is not involved. In particular, the WMA mixture including C2 showed dry performance significantly enhanced (lower particle loss) with respect to the HMA mixture, as highlighted by the statistical analysis. On the other hand, both WMA mixtures are strongly affected by the presence of water since higher particle loss values were measured after wet conditioning for WMA mixtures with respect to HMA. Such a reduction in performance is found statistically significant for both WMA mixtures, meaning that both types of chemical additive does not compensate the effect of reduced production temperatures. The WMA mixture including the chemical additive C2 was characterized by the highest water susceptibility as demonstrated by the worst raveling performance in wet conditions; statistically significant differences in performance were measured also with respect to the other WMA mixture including the chemical additive C1. Such an outcome can be attributed to the presence of anti-stripping and surfactant agents within C1 that helps preserving adequate adhesion properties at the bitumen/aggregates interface also in presence of water, confirming other researches (Cucalon et al., 2016).

However, all mixtures demonstrated Cantabro results consistent with international requirements for high traffic motorways corresponding to a maximum of 20% and 35% particle loss in dry and wet conditions, respectively (Alvarez et al., 2010). Such a finding confirms the feasibility of in-plant production of warm PA mixtures since the large-scale production at reduced temperatures leads to acceptable mixtures when chemical WMA additives are employed.

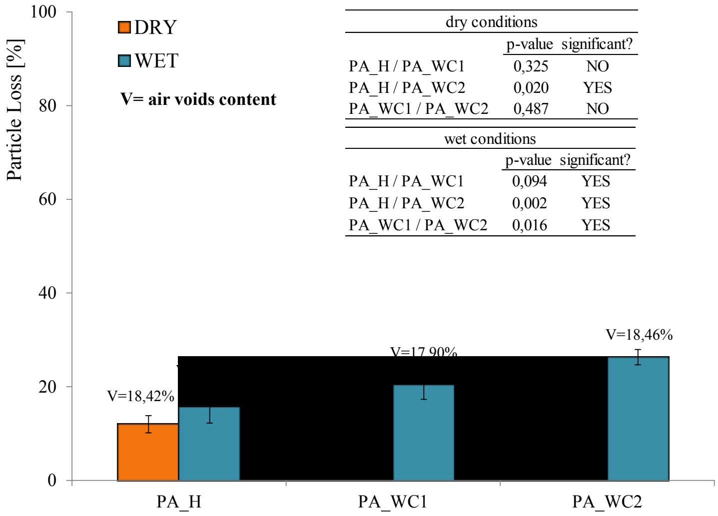
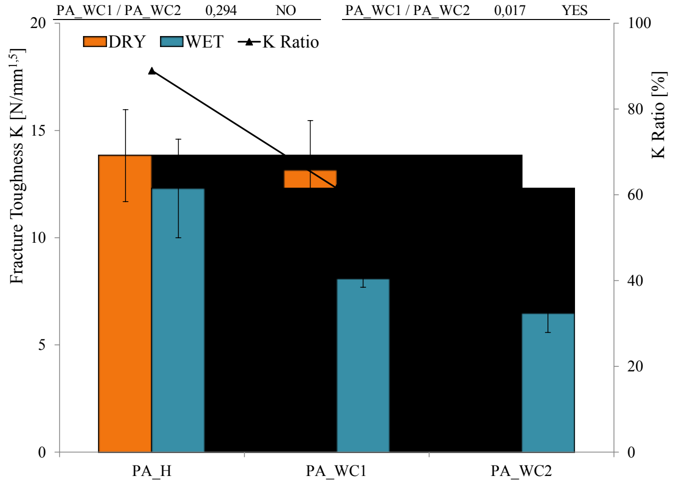


Figure 10.4 Mean results in terms of Cantabro tests.

10.3.4 Fracture properties

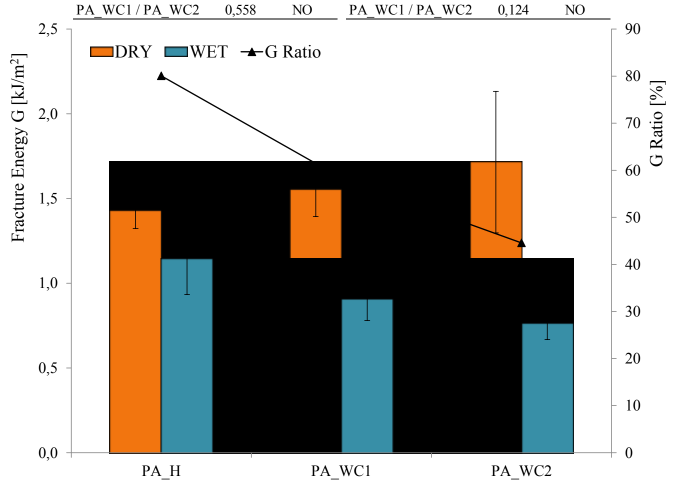
Semi-circular bending test results in both dry and wet conditions are reported in Figure 10.5 in terms of fracture toughness and fracture energy along with the standard deviation and ANOVA test results in terms of p-value. The water susceptibility was taken into account by means of K Ratio and G Ratio, defined as the percent ratio of wet and dry results.

	dry conditions		wet conditions	
	p-value	significant?	p-value	significant?
PA_H / PA_WC1	0,702	NO	0,011	YES
PA_H / PA_WC2	0,121	NO	0,003	YES
PA_WC1 / PA_WC2	0,294	NO	0,017	YES



(a)

	dry conditions		wet conditions	
	p-value	significant?	p-value	significant?
PA_H / PA_WC1	0,260	NO	0,100	NO
PA_H / PA_WC2	0,231	NO	0,017	YES
PA_WC1 / PA_WC2	0,558	NO	0,124	NO



(b)

Figure 10.5 SCB mean results in terms of fracture toughness (a) and fracture energy (b).

Fracture toughness K represents the mixture resistance to the fracture initiation and propagation. Results in dry conditions showed a slight decrease in performance for both WMA mixtures when compared to the HMA, even if no statistical significance can be identified. As far as wet results are considered, it is possible to notice a clear reduction in fracture toughness due to lower production temperatures (WMA mixtures), as also highlighted by the statistical analysis. Among the two warm additives studied, C2 demonstrated worst effectiveness with respect to C1 since significant lower resistance in wet conditions was measured for PA_WC2. This remark confirms previous observations in terms of raveling resistance in presence of water and can be attributed to the anti-stripping and surfactant agents included within C1 that effectively enhance adhesion properties. On the contrary, the additive C2 is a viscous regulator and, even though it ensures good compactability properties at low production temperatures, it does not provide adequate adhesion resistance at the bitumen-aggregates interface leading, in turn, to high water susceptibility.

Fracture energy was also calculated as a measure of the material ductility as it represents the work required to achieve a complete failure of specimens. Results showed similar performance for all mixtures studied in dry conditions (Figure 10.5b), indicating that the presence of chemical additives ensures good performance even if reduced production temperatures are used. However, fracture energy results in wet conditions still demonstrate a higher water susceptibility of both WMA mixtures when compared to the HMA one, consistently with previous findings.

10.3.5 Fracture initiation resistance

Repeated indirect tensile test results in both dry, wet and submerged conditions are reported in Figure 10.6 in terms of number of pulse at failure; ANOVA test results are also shown in terms of p-value whereas the error bars expresses the standard deviation. Stiffness modulus values are also represented in the graph for both hot and warm mixtures as the average of 9 replicates.

Both WMA mixtures are characterized by lower stiffness values than the HMA; such behavior can be attributed to the reduced short term aging process that warm mixtures underwent during production and laydown phases due to the low production temperatures (Capitão et al., 2012). In addition, it needs to be taken into account that recycled aggregates could release less aged stiff bitumen during warm production. Hence, a larger amount of RAP bitumen behaves as “black aggregate” leading to softer mixtures.

As far as the fracture initiation resistance is concerned, both WMA mixtures demonstrated worst performance than the HMA in all test conditions. A remarkable reduction in pulse at failure was shown in dry, wet and submerged conditions, as also highlighted by the statistical analysis. The significant loss in performance, especially in wet and submerged conditions, can be attributed to the reduced adhesion properties at the bitumen-aggregate interface that can developed in case of WMA mixtures due to low production temperatures.

Among the two WMA mixtures analyzed, no significant difference in fracture resistance was found, meaning that the two types of WMA chemical additives behave similarly.

dry conditions			wet conditions			submerged conditions		
	p-value	significant?		p-value	significant?		p-value	significant?
PA_H / PA_WC1	0,046	YES	PA_H / PA_WC1	0,008	YES	PA_H / PA_WC1	0,025	YES
PA_H / PA_WC2	0,032	YES	PA_H / PA_WC2	0,015	YES	PA_H / PA_WC2	0,049	YES
PA_WC1 / PA_WC2	0,936	NO	PA_WC1 / PA_WC2	0,773	NO	PA_WC1 / PA_WC2	0,551	NO

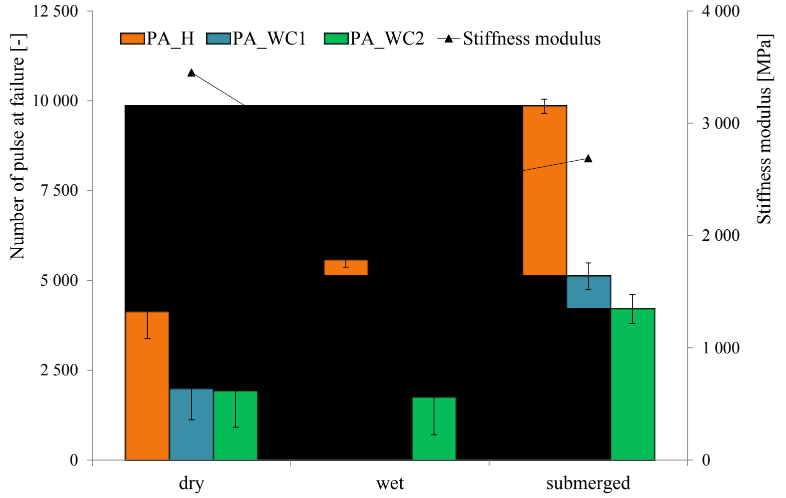


Figure 10.6 Repeated indirect tensile tests results and stiffness modulus values.

10.4. Conclusions

The present research aims at evaluating mechanical performance and durability of recycled porous asphalt (PA) mixtures produced at warm temperatures by means of two WMA chemical additives (C1 and C2) characterized by different composition and operating mechanism. Mixtures were in-plant produced in order to investigate the feasibility of large-scale productions of warm PA mixtures.

Based on the experimental results, the following conclusions can be drawn:

- both WMA mixtures satisfy acceptance requirements of motorway technical specifications and international recommendations for raveling resistance demonstrating the feasibility of in-plant production at reduced temperature of recycled OG mixtures;
- the effect of the two chemical additives is similar in terms of volumetrics and compactability and both guaranteed improved properties than the reference HMA mixture even at reduced production temperatures;
- both types of chemical WMA additives ensure performance comparable to the HMA mixture in terms of Indirect Tensile Strength, raveling resistance as well as fracture propagation resistance when the effect of water is not considered (dry conditions);

- WMA mixtures are more prone to moisture susceptibility with respect to the HMA. However, the chemical composition of the WMA product employed was found essential to improve mixture behavior under the action of water. WMA additives including antistripping agents (C1) seem more effective than viscous regulators (C2), even if equivalent performance to HMA are not guaranteed;
- reduced production temperatures affect fracture initiation resistance, especially in wet and submerged conditions due to the reduced adhesion properties at the bitumen/aggregates interface of WMA mixtures that can easily been deteriorated by the presence of water.

Finally, overall results suggest that the in-plant production of PA recycled mixtures can be successful, but particular attention must be paid on the selection of the most appropriate WMA additive in order to provide adequate performance also in presence of water.

Summary of the overall experimental findings

Summary of the overall experimental findings

Development of Sustainable (Hot and Warm Recycled) Porous Asphalt Mixtures through Laboratory and Field Investigation

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The research work described in this thesis focuses on environmental friendly and sustainable solution in designing asphalt mixtures for Porous Asphalt (PA) surface layers. The study has a twofold objective: it evaluates the possibility to include milled materials coming from old PA layers as partially substitution of virgin materials without compromise pavement performance. Furthermore, the effectiveness of different Warm Mix Additives (WMA) is taken into account in order to significantly reduce production temperatures of recycled PA mixtures with both environmental and economic benefits. Hence, the present thesis is organized in two separate parts: the first part (*Part 1. Hot recycling of PA mixtures*) describes the attempt to use RAP and increase its content in PA mixtures by means of different mix design studies, laboratory evaluations based on the moisture sensitivity and durability tests as well as field study throughout experimental sections. The second part (*Part 2. Warm recycling of PA mixtures*) describes laboratory and field evaluations that were carried out to optimize the use of WMA additives in PA mixtures including RAP aggregates.

The main experimental findings are summarized in this chapter to provide an overall conclusion of this wide research study.

As long as hot recycling in PA mixtures is concerned, optimum results were obtained in terms of adhesion properties which represent a primary aspect for PA mixtures (detailed described in *Chapter 3. Preliminary study on adhesion properties between bitumen and RA aggregates*). In such mixtures, in fact, the contact between aggregate particles through asphalt mastic occurs in a very small area due to the high void content of the mixtures. Thus, the “affinity” at the bitumen-aggregate interface needs deep investigation as the new virgin bitumen interacts with the thin film of aged mastic that covers the RAP aggregates. Hence, a preliminary study focuses on the evaluation of the bond strength properties (adhesion and cohesion) between virgin bitumens and an artificial RAP substrate simulating the real RAP aggregate. By comparing the results obtained performing BBS test on traditional (virgin aggregate) and on coated aggregate (artificial RAP aggregate) samples, in both dry and wet conditions, the effects of RAP used for the production of new asphalt mixes was investigated in terms of cohesion/adhesion properties and moisture damage resistance. The overall investigation suggests that the use of RAP in the production of new bituminous mixes does not penalize the bond interactions between virgin bitumen and aggregates. On the contrary, **the artificial RAP substrate, used to simulate a real RAP aggregate, appears able to provide an improvement in the adhesion performance of the mixture and to reduce the water sensitivity of the system bitumen-aggregate considerably**. The presence of a thin film of aged bitumen in the artificial RAP aggregates, in fact, ensures the development of higher adhesion with the virgin bitumen. This result is detectable both in terms of failure type (failure within the bitumen) and bond strength values, regardless of the conditioning type (dry and wet). In particular, the loss in performance due to the effect of water experienced by the virgin aggregates is much more evident than the loss experienced by the coated aggregates.

Such promising results strongly encouraged to proceed with the mix design and performance evaluation of recycled porous mixtures. In this sense, the effect of using coarse reclaimed asphalt (RAP) obtained by milling old porous surface layers, on the performance of porous asphalt (PA) mixtures was evaluated and detailed described in

Chapter 4. Use of 15% RAP in PA mixtures. This study consisted in a mix design optimization and laboratory evaluations of recycled PA mixtures including 15% of coarse RAP as well as a field study of the optimized mixtures. As long as laboratory experimentation is concerned, it was preliminarily verified if the performance of porous asphalt mixtures can be still guaranteed when a certain percentage (15%) of course RAP aggregates is added to the mix. Moreover, the influence of different total bitumen contents on the recycled porous asphalt mixtures performance had been evaluated in order to optimize the mix design. The research project focuses on the evaluation of durability issues as they are the main failure causes in PA. In this sense, Indirect tensile strength tests, particle loss (Cantabro) tests, semi-circular bending (SCB) tests and repeated indirect tensile tests were carried out in both dry and wet conditions in order to evaluate acceptability, durability, fracture resistance and water sensitivity of recycled mixtures.

The overall experimental results suggest that including 15% of course recycled aggregates in porous asphalt mixtures do not compromise the durability of the PA layers and it was demonstrated that with total bitumen content from 5.00% to 5.50%, it is even possible to improve the properties of PA mixtures. These findings could be attributed to the presence of RAP aggregates that can easier adhere to the virgin bitumen as verified through the BBS test during the preliminary phase of the research study.

Based on the promising results emerged from the present research, advanced laboratory researches were carried in collaboration to EMPA (Swiss Federal Laboratories for Materials Science and Technology) under the supervisor of Professor Manfred Partl. In particular, the specimens had been prepared at the laboratory of Università Politecnica delle Marche following the same protocol optimized during the first part of the experimentations: based on the previous results, the recycled PA mixtures were prepared with two total bitumen contents (5.00% and 5.25% by weight of the aggregates). Afterwards, CoAxial Shear Test (CAST) were performed at EMPA in order to evaluate the influence of water on repeated loading resistance of recycled PA mixtures. Results showed that the effect of water exposure during CAST tests appeared negligible, irrespective of the presence of RAP aggregates. Moreover, recycled mixtures showed higher complex modulus values than the reference mixture in both dry and submerged conditions.

As far as field applications were concerned, recycled mixtures with 5.00% and 5.25% of total bitumen content were selected. Mixtures were prepared at the asphalt plant during the construction of a full-scale trial section along an in-service motorway were evaluated in this study in order to verify the performances of “real” mixtures. Such mixtures were prepared according to the laboratory mix design defined in the previous phase of the research and with the same materials. The overall experimental results confirmed that including 15% of coarse recycled aggregates in porous asphalt mixtures does not compromise performance and durability of PA layers if adequate bitumen content is selected. As a matter of fact, it was demonstrated that recycled mixture with 5.25% of total bitumen content and prepared following an accurate mix design can perform as well as (or even better than) standard PA.

Moreover, it was found that field mixtures exhibited similar or even enhanced performances with respect to the corresponding laboratory materials meaning that the large scale production of recycled PA is possible without compromise mixtures performance and

durability. Recycled mixtures were also able to ensure the same drainage properties of the reference porous surface.
Based on the promising results of the first experimental research involving the use of 15% of selected RAP in porous asphalt mixtures, the experimental activity was extended to involve higher amount of RAP. Performance of recycled PA mixtures with 20% and 25% (by aggregates weight) of RAP from old PA surface layers were evaluated by means of both laboratory and field investigations (the experimental study was detailed described in *Chapter 5. Attempt to increase RAP content in PA mixtures*).
As far as laboratory experimentation is concerned, results showed that recycled PA mixtures at low total bitumen contents are characterized by worst compactability properties than the reference PA mixture due to the fact that a significant part of the total bitumen content is aged bitumen within the RAP and a prominent part acts as "black aggregate". At higher total bitumen content, in fact, the recycled mixtures demonstrate compactability properties similar to the reference PA mixture. However, recycled mixtures were able to satisfy acceptance requirements typically prescribed by technical specification for PA mixtures. The overall experimental suggested that 5.50% of total bitumen content can be considered as the optimum value for PA mixtures with 20% of coarse RAP whereas both 5.50% and 5.75% of total bitumen content can be adopted as optimum value for PA mixtures with 25% of coarse RAP. Based on the promising results emerged from the present research and previous studies, it is possible to conclude that **the hot recycling is a suitable solution also for PA mixtures, as already largely accepted for dense-graded mixtures.**
As far as field experimentation is concerned, similar PA mixtures were prepared in the asphalt plant during the construction of a real scale trial section along an in-service motorway (hereafter named field mixtures). Results showed that recycled PA mixtures have better compactability properties than the reference mixture and they were able to satisfy acceptance requirements typically prescribed by technical specification for PA mixtures. The overall experimental results confirmed that including 20% of coarse recycled aggregates in porous asphalt mixtures does not compromise performance and durability of the PA layers if adequate bitumen content is selected. Moreover, all mixtures satisfy acceptance requirements for PA mixtures in motorway pavements in terms of drainage capability and the drainage evolution due to post compaction and traffic loads was not affect by the presence of RAP aggregates within PA mixtures. The results were also compared with those of previous research studies regarding laboratory PA mixtures including the same amount of RAP aggregates and total bitumen contents, demonstrating that the laboratory research phase was representative of the real plant conditions and the selected mix design was accurate.
In Chapter 6 (*Reactivated bitumen investigation*) a reliable approach for **a practical method able to predict the amount of "re-activated" bitumen within the RAP, on the basis of a performance-based equivalence principle is proposed**. The method is based on the assumption that the "re-activated" bitumen content is proportional to the average "re-activated" bitumen film thickness (R_{TF}) which is considered constant for a given heating process (mixing temperature and time) and aged bitumen type. The calculation of R_{TF} is based on the average asphalt film thickness (A_{FT}) concept that relate the calculation of A_{FT}

to aggregate surface area and effective volume of bitumen. Thus, it is possible to evaluate the corresponding "re-activated" bitumen content from such R_{TF} as long as the RAP aggregates gradation is known. Since the RAP aggregates surface area remains unchanged for all the studied PA mixtures, the percentage of "re-activated" bitumen can be considered the same (50% of RAP bitumen). In this sense, the proposed method should be able to predict the percentage of "re-activated" bitumen by assuming a constant "re-activated" bitumen film thickness as long as RAP aggregates gradation is known. Such an assumption requires to be confirmed with further studies on different RAP but it prefigures a reliable approach to predict the amount of "re-activated" bitumen from RAP, which needs to be taken into account during the mix design of hot recycled mixtures.

As long as warm recycling in PA mixtures is concerned, the second part of this thesis describes laboratory and field investigation to efficiently exploit possible advantages related to the combination of recycling and WMA additives as well as to optimize the mix design of recycled WMA mixtures. A preliminary analysis was carried out and described in *Chapter 8 (Preliminary study on bituminous phase and adhesive properties)* involving a rheological analysis of the bitumen phase as well as an investigation of the adhesion properties at the bitumen/aggregates interface when WMA technologies are included.

The viscosity analysis of bitumens and mastics allowed to conclude that the standard procedures used for the evaluation of mixing and compaction temperatures of HMA mixtures are not able to properly predict the behavior of WMA additives in terms of production temperatures. Such procedure, in fact, is based on the iso-viscosity principle which assumes that asphalt mixtures should be mixed and compacted at temperatures corresponding to a given range of bitumen viscosity that ensures that the bitumen phase is sufficiently fluid during mixing and construction. This procedure is valid in case of HMA mixtures but it cannot be used when warm mixtures are involved since WMA additives do not impact only the bitumen viscosity; their action takes place also at the bitumen/aggregates interface by reducing and regulating the forces rather than only reducing the bitumen viscosity.

Results in terms of rutting performance (MSCR tests) demonstrated that the aging process leads to an increment of stiffness as well as an enhanced elasticity of the bituminous phase. Such an outcome is more evident for the HMA bitumen whereas the difference is less significant for all the WMA bitumens due to the reduced production temperatures used for the aging process that properly reproduced the in-plant warm production. Only the presence of the zeolite leads to a decrement in rutting behavior with respect to the aged HMA bitumen.

The adhesion investigation showed that **the production of WMA mixtures at reduced temperature may affect adhesion properties between aggregates and bitumen with consequences on raveling resistance as well as water susceptibility**. However, other factors (i.e. mineralogical nature of aggregates, volumetric properties, internal aggregate structure) can play a fundamental role on the overall mechanical response. In particular, RAP material is able to partially compensate this loss in performance thank to the presence of pre-coated aggregates which adhere more easily to the virgin bitumen even at reduced production temperatures. Moreover, RAP aggregates provide a water-resistant surface less

prone to undergo water damage. Such an effect was found to be significant especially when the chemical WMA additive was used with major benefits on water susceptibility.
The possibility to significantly reduce the production temperatures of good quality recycled porous asphalt mixtures by means of WMA additives was evaluated by means of laboratory investigations where the production temperatures were reduced of 40°C and different WMA additives were added to the PA mixtures (detailed described in *Chapter 9. Laboratory investigation of PA mixtures using different WMA additives*). The research project consisted in two main phases. The first part was entirely carried out in Polytechnic University of Marche and focused on the evaluation of durability and water susceptibility as they are the main failure causes in PA. Such experimentation highlighted the **issues related to the reduced temperatures production of PA mixtures, particularly in terms of water sensitivity. Only the presence of chemical additive in the mixture seems to ensure adequate water resistance**, although it does not guaranteed comparable performance than the HMA mixture.
The second part of this project involved an international collaboration between Polytechnic University of Marche and Vienna University of Technology and focused on long-term performance of WMA mixtures. In particular, aging effects on WMA mixtures were evaluated by aging WMA porous asphalt mixtures by means of an innovative test procedure (Viennese Aging Procedure - VAPro). Short and long term performance of both WMA mixtures and bitumens were compared with performance of an equivalent HMA. Such an investigation underlined the different aging behavior of WMA and HMA mixtures and the direct correlation with bituminous phase; **despite differences in aging effects, long term performance in terms of fatigue resistance are not affected by production temperatures or WMA additive type**.
Based on this finding, a further research project was carried out with the aim at investigate the in-plant production at reduced temperatures of PA mixtures by means of WMA chemical additives (detailed described in *Chapter 10. In plant production of warm PA mixtures using chemical WMA additives*). In particular, two WMA chemical additives (C1 and C2) characterized by different composition and operating mechanism were selected. Both the **WMA mixtures studied satisfy acceptance requirements** of motorway technical specifications and international recommendations for raveling resistance demonstrating the feasibility of in-plant production at reduced temperature of recycled PA mixtures. Moreover, the effect of the two chemical additives is similar in terms of compactability properties, Indirect Tensile Strength, raveling resistance as well as fracture propagation resistance when the effect of water is not considered. On the other hand, **WMA mixtures are more prone to moisture susceptibility with respect to the HMA**. However, the chemical composition of the WMA product employed was found essential to improve mixture behavior under the action of water. WMA additives including antistripping agents (C1) seem more effective than viscous regulators (C2).

Lists of 3-years Ph.D. publications

1) F. Frigio, E. Pasquini, G. Ferrotti, F. Canestrari. "Improved durability of recycled porous asphalt", *Construction and Building Materials*, vol. 48, pp. 755-763, **2013**. DOI: 10.1016/J.CONBUILDMAT.2013.07.044

2) F. Cardone, F. Frigio, G. Ferrotti, F. Canestrari. " Influence of polymer modification on asphalt binder dynamic and steady flow viscosities", *Construction and Building Materials*, vol. 71, pp. 435-443, **2014**. DOI: 10.1016/J.Conbuildmat.2014.08.043

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6) F. Frigio, G. Ferrotti, F. Cardone. "Fatigue rheological characterization of polymer-modified bitumens and mastics", In: *8th International RILEM Symposium Proceeding*, 7-9 October **2015**, Ancona, Italy. RILEM Bookseries, vol. 11: 655-666, 26 September 2016. DOI: 10.1007/978-94-017-7342-3_53

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