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Abstract

In cold regions, the production of Cement-Bitumen Treated Materials (CBTM) represents an issue in terms of annual time available for production. The objective of this research is to study the influence of different combinations of production temperatures for mixing, compacting and curing (developed in two steps) on the mechanical properties of CBTM produced with two sources of bitumen emulsion. Workability, compactability, indirect strength and other additional tests were involved in the analysis. Findings highlighted the critical effect of transportation and compaction temperatures on CBTM workability. Moreover, the emulsion source significantly affects the mixture strength when produced at low temperatures.

Keywords: Cold recycling, Compactability, Bitumen emulsion, Indirect Tensile Stiffness Modulus, Indirect Tensile Strength, Scanning Electron Microscope

1 INTRODUCTION

Production of traditional hot mix asphalts (HMA) in road industry, intended as mixing, transportation and compaction, is normally performed at a range of temperatures between 150 °C and 170 °C [1, 2]. The reasons that lead to the definition of such temperatures is the necessity to reduce the bitumen viscosity in order to well coat aggregates, to provide a workable mixture and to be properly compacted in the field.

The economical and environmental crisis that characterized the last decades brought to the introduction of new techniques to obtain materials addressed to the production of the pavement structure: warm mix asphalts (WMA) [3, 4] and cold asphalt mixtures (CAMs) [5-7]. In the first case, production temperatures can be decreased by around 30 °C thanks to the use of additives able to reduce the bitumen viscosity [8]. In the second case, the entire production process can be performed at atmospheric temperature employing the bitumen in form of foam or emulsion. The use of water in these mixtures ensures workability and compactability, allowing at the same time the use of wet aggregates. For such reasons, this technique brings high environmental and energy-saving benefits if compared to standard HMA or WMA mixtures [9-12].

A further improvement in terms of sustainability is obtained when reclaimed asphalt pavement (RAP) is used as aggregate material [11, 13-16]. The re-use of RAP instead of virgin aggregates in Cold Recycled Mixtures (CRMs) leads to the possibility to have performant mixtures for base or binder layers with a material that is normally available in high quantities [15, 17].

To improve short-term and long-term mechanical properties, a small amount of Ordinary Portland Cement is added to CRMs obtaining cement-bitumen treated materials (CBTM) [18]. The quantity of cement used is usually lower than the bitumen content in order to have materials that are considered having a bituminous behaviour [19]. For this reason, the balance between the two binding agents is an important parameter to control. As mentioned previously, bitumen can be added in the form of foam or emulsion in CRMs. In this paper, we concentrate on emulsion treated materials. Bitumen emulsions are obtained by sheering the bitumen in a colloidal mill, which is then suspended in a watery phase in form of droplets. The suspension of bitumen droplets is ensured by the presence of an emulsifier in the system, that is responsible for the repulsive effect [20]. This phenomenon allows storing the emulsion for a certain period (2–3 months) and to have a good breaking on the RAP material. The nature of bitumen emulsion makes it extremely sensitive to temperatures, from the storage to the long-term performance of the final mixture [21].

At present, no specific standard establishes the minimum temperature required to produce a CBTM material, but many manuals recommend different temperatures based on their experience, without distinguish the three different processes: mixing, transportation and laydown and compaction. For example, in some cases, the minimum temperature for laydown must be above 5 °C, whereas in other cases a temperature of at least 10 °C is required to carry out a cold recycled project [22-24]. An AASHTO report (1998) establishes that for projects using bitumen emulsions, a minimum atmospheric temperature range between 10 and 16 °C should be respected during production. If cement or fly ash are used as additional binders in CBTM, the minimum atmospheric temperature can be 4 °C [25]. The Asphalt Recycling & Reclaiming Association (ARRA) also provided construction guidelines for cold in-place recycling (CIR) using bitumen emulsion, specifying that operating temperatures are extremely variable depending on the emulsion used and/or RAP temperature, requiring in some cases atmospheric temperatures higher than 16 °C [26]. Many other studies report the production temperature in the laboratory equal to room temperature, or able to represent as close as possible the field conditions [27-32]. This aspect of CBTM mixtures is of fundamental importance when construction projects are carried out in cold regions such as

Canada, North-East USA or North-Europe. In fact, average climate conditions throughout the year do not allow a wide time span for CBTM production and laydown.

Not only production's temperatures are important for the CBTM mechanical properties, but also the conditions characterising the curing process. During this time, the water present in the mixture evaporates, accelerating the emulsion breaking process and improving the mechanical properties. When cement is used in addition to bitumen emulsion, a certain amount of water is used for the hydration process. Therefore, the amount of time to allow a complete curing is highly dependent on environmental conditions, such as temperature, relative humidity and wind [19, 33-37]. Because of this high variability, it is impossible to establish a single laboratory procedure to represent field curing. At the same time, the evolution of curing in the field is difficult to follow, because of the distortion brought by performing cores [6].

However, Bocci et al. (2011) [38] showed that changing the curing temperature in the laboratory from 40 °C to 20 °C, it is possible to reach the same level of stiffness, although the curing time required is very different (10 days and 50 days, respectively). On the other hand, a curing temperature of 5 °C for 60 days did not allow to increase the stiffness enough; but, when an additional curing of 14 days at 40 °C was carried out, the tested mixture reached the same stiffness as the ones of the other curing conditions. In that research, the double step curing can be seen as a simulation of a material cured during the cold season first, and with a long-term curing afterwards. It is highlighted that in that case, mixtures were mixed and compacted at room temperature, and only the curing temperature effect was studied [38].

The objective of this research is to understand how the low production temperatures (mixing, transportation and compaction, and curing) are affecting the long-term mechanical properties of CBTM treated with bitumen emulsion, changing the emulsion source. For this purpose, different combinations of temperatures for the three processes were reproduced in the laboratory, focusing the work towards low temperatures.

2 EXPERIMENTAL APPROACH

Cold in-plant recycling (CIPR) projects are characterized by the presence of a production plant (fixed or mobile) located several kilometres from the construction site. In such cases, the entire process is developed in different steps. At first, the existing pavement is milled at a specified depth according to the thickness of the damaged layer or layers. During this operation, the RAP material is obtained and collected, in order to be moved to the production plant. At this point, the CBTM mixture is prepared, adding to the RAP aggregate cement, bitumen emulsion, and water. If required, the RAP aggregate gradation can be corrected to respect local gradation specifications. At the moment of mixing, only the temperature of the emulsion is known, since it is stocked at a precise temperature. On the other hand, all the other raw materials characterizing the CBTM mixture are kept at atmospheric temperature. The obtained mixture is then transported to the construction site, in order to be laid and compacted. During transportation and compaction, atmospheric temperature and time are very important, to avoid a premature breaking of the emulsion (in case of low temperatures) or rapid water evaporation (in case of high temperatures). In both cases, laydown and compaction characteristics of the material could be changed. When the required density is reached, the compaction stops and a certain amount of time is often required before that the upper layer is placed. This time is necessary to allow the water to evaporate, in order to let strength and stiffness of the mixture to increase. Normally, this process is considered finished when around 1% of residual water is present in the mixture [6].

In the present study, the entire process is simulated in the laboratory, in order to investigate the effect of temperature on each step of the production process. In fact: a) mixing, b) transportation and compaction and c) curing, are considered separately, with a specific assigned time and temperature.

3 MATERIALS AND METHODOLOGY

3.1 Materials and mixtures

The mixes were produced using a single RAP source sampled from a stockpile in Italy. The main characteristics of the RAP aggregate are listed in Table 1. The gradation of the RAP material was modified to obtain a distribution close to the maximum density curve with exponent 0.45. For this reason, the aggregate blend was composed of 94% of RAP and 6% of crushed limestone filler (Figure 1).

The cement used was a GU type (CSA A3000) with compressive strength at 28 days of 43.9 MPa (ASTM C109). The cement content was fixed at 1.5% by mass of aggregates.

Table 1 RAP aggregate properties

Property	**Standard**	**Unit**	**Value**
Binder content	ASTM D6307	%	5.51
Nominal maximum particle dimension	ASTM D448-03	mm	16
Maximum specific gravity	ASTM C127-128	-	2.482
Average bulk density	LC 21-065-066-067	-	2.323
Water absorption	ASTM C127-128	%	1.10

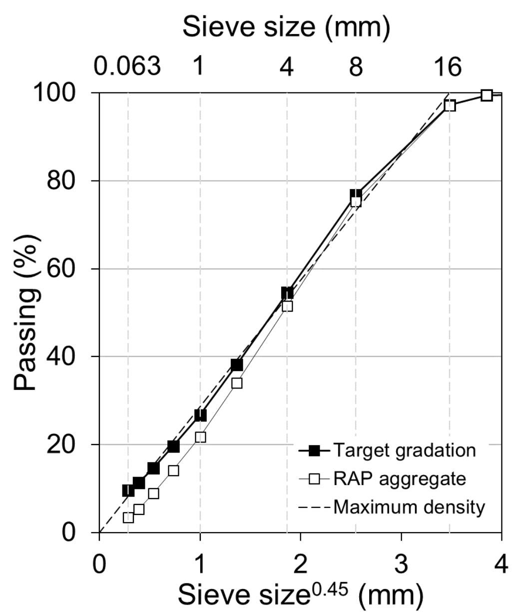

Figure 1 Target aggregate blend

Two bitumen emulsions were used for this study: one is a slow-setting cationic emulsion produced in United Kingdom classified as CSS-1 (ASTM D2397), whereas the other is a slow-setting cationic emulsion produced in Italy and classified as C60B10 (EN 13808). The main properties of both emulsions are listed in Table 2, where for simplicity are named from now on as Emulsion A and Emulsion B, respectively. It is possible to observe that the main difference between the emulsions regards the residual bitumen penetration value. In fact, Emulsion B is characterized by a softer residual bitumen. Moreover, this is confirmed by the lower softening temperature. In both cases, the bitumen emulsion dosage was kept constant at 5% (3% of residual bitumen) by mass of aggregates for the mixes. A mix design protocol was performed to fix the amount of total water, characterized by the water absorbed by the aggregates, the water from bitumen emulsion and the added water to improve compactability. Such amount was fixed at 4.0% by mass of aggregate, in order to reach the target air voids (15%) without employing high compaction energy and avoiding any material loss (water, bitumen and/or fine particles) during compaction.

Table 2 Bitumen emulsions properties

Emulsion A			
Bitumen emulsion properties	**Standard**	**Unit**	**Value**
Density	ASTM D6397-16	g/cm^3	1.0
Residue content (bitumen)	ASTM D6997-12	%	60.3
Storage stability @ 24 hours	ASTM D6930-10	%	0.6
Residual bitumen properties			
Penetration @ 25 °C	ASTM D5-13	mm	4.1
Softening point	ASTM D36-14	°C	48.6
Emulsion B			
Bitumen emulsion properties	**Standard**	**Unit**	**Value**
Residual bitumen	EN 1428	%	60.0
Viscosity @ 40 °C	EN 13302	s	42.5
Breaking Index	EN 13075	%	2
Residual bitumen properties			
Penetration @ 25 °C	EN 1426	mm	10.0
Softening point	EN 1427	°C	43.0

3.2 Mixtures production

In order to investigate the effect of production temperature, loose mixes and specimens were obtained dividing the entire process in four steps: mixing, transportation and compaction, first period of curing and finally the second period of curing. Table 3 summarizes the details regarding the production process.

The mixing protocol required from 5 to 10 minutes and was performed by adding to the humid aggregate blend cement, water for compaction and bitumen emulsion, in this order. The mixing was carried out after conditioning materials and mixing tools (except for bitumen emulsion) at the target temperature for more than 12 hours. At the same time, the two bitumen emulsion sources were stored at room temperature (Emulsion A) and at 40 °C (Emulsion B) [39].

A rest period was planned to simulate the transportation process for in-situ applications. In the laboratory, the mixture was poured in a plastic bag and sealed carefully to avoid any water loss by evaporation. The material was then placed in an environmental chamber at the target temperature for 2 hours. After the simulated transportation time, the compaction process was carried out by means of a Superpave Gyratory Compactor (SGC) in a 100 mm undrained mould, with a constant pressure of 600 kPa, gyrations rate of 30 rpm and internal angle of 1.25 °. Prior to compaction, the mould and all the tools employed were placed in the environmental chamber for conditioning at the target temperature for at least 12 hours. The compaction was performed at fixed height, to obtain the same amount of voids in the mixture (V_m):

$$V_m = \frac{V_{V,A} + V_{W,I}}{V} \cdot 100 = \frac{V - (V_S + V_C + V_{B,R})}{V} \cdot 100 \quad (1)$$

where V is the total volume of the specimen, V_S is the bulk volume of aggregates, V_C is the volume of cement, $V_{B,R}$ is the volume of residual bitumen from emulsion, $V_{W,I}$ is the volume of intergranular water and $V_{V,A}$ is the volume of air. The specimens' volume, V, was fixed to obtain a V_m of 15 ± 1%.

After compaction, the specimens started a first period of curing of 14 days in the environmental chamber, after which a first series of test was performed. In this first part, two temperatures were chosen: 5° C and 25 °C. The first represents the minimum temperature that several manuals recommend to produce cold mixtures, whereas the second represents the typical environmental temperature, often used in literature as reference temperature for the study of such materials [40, 41]. All the specimens not tested were cured for an additional period of 14 days at 40 °C, regardless of the mixture, and another series of test was performed afterwards. This step was necessary to have specimens representing a long-term curing, in order to understand the effect of the initial production and curing temperatures.

It is important to remark that all the temperatures reported in Table 3 had a variability of ± 2 °C. As it can be seen, not all the mixtures produced with Emulsion A were repeated with Emulsion B. In fact, Emulsion A was chosen to investigate the different phases of the production process in different temperature conditions, whereas Emulsion B was used only for production at standard temperature and low temperature. It must be highlighted that both emulsions are designed for cold recycling purposes, even though they are employed in two different climates and markets. The letter at the beginning of the mixes names represents the emulsion, the first number is the mixing temperature, and the second number represents transportation, compaction and first cure temperature.

Table 3 Mixtures naming and production process

Processes		**Production**		**Curing**	
Steps		Mixing	Transportation + compaction	1^{st} period	2^{nd} period
Allowable time		5–10min	2 hours + 30 min	14 days	14 days
Emulsion A	**A_25_25**	25 °C	25 °C	25 °C	40 °C (2)
	A_25_5	25 °C	5 °C	5 °C (2)	
	A_5_25	5 °C	25 °C	25 °C	
	A_5_5	5 °C	5 °C	5 °C (2)	
	A_5_5_0C (1)	5 °C	5 °C	5 °C (2)	
Emulsion B	**B_25_25**	25 °C	25 °C	25 °C	40 °C
	B_5_5	5 °C	5 °C	5 °C	

(1) The mixture does not contain cement. The volume of cement was replaced by filler.
(2) The curing was performed with controlled relative humidity at 55 ± 5%

3.3 Testing program

3.3.1 *Workability and compactability*

SGC compaction curves can be described using several parameters. In this specific study the Compaction Energy Index (CEI), voids in the mixture after 10 gyrations $V_m(10)$ and the compaction curve slope k were chosen to analyse the mixtures in terms of compaction behaviour. In case of HMA, the CEI indicates the area under the compaction curve from the 8th gyration to the number of gyrations related to 92% of the mixture maximum density. Eight gyrations are selected to simulate the process of laydown performed by the paver in the field. In this case, the CEI is calculated between gyration number 10 and the number of gyrations required to reach the target V_m of $15 \pm 1\%$. Mixtures with low values of CEI are preferable because of improved constructability [42]. Nevertheless, other authors have elaborated several compaction indexes based on the relationship between maximum density and air voids ratio [43-45].

Starting from 10 gyrations, the SGC compaction curve can be represented in a semi-logarithmic plot as a straight line having slope k. Parameters $V_m(10)$ and k are obtained by experimental data by means of a linear regression:

$$V_m(n) = V_m(10) - k \log n \quad (2)$$

where $V_m(n)$ are the voids in the mixture at the gyration number n.

In order to describe the mixture workability, i.e. the ease to be mixed and the laydown effort, the value of V_m at 10 gyrations, $V_m(10)$, is selected as the workability parameter. Low values of $V_m(10)$ characterise mixtures with improved workability. On the other hand, the slope is selected as a compactability parameter, and it is directly related to the mixture densification [40–41]. High k values represent better compactability.

As mentioned before, all the mixtures studied were compacted at fixed height to reach the same amount of voids in the mixture. Hence, in order to compare the CEI index results, it is not useful to consider the compaction area below the target value of V_m. Consequently, the area of the triangle is considered and named CEI_T^+, as shown in Figure 2.

Normally the CEI_T^+ is calculated as the area under the graph according to the trapezoidal rule. However, an alternative way to calculate the CEI_T^+ is proposed in this research, as the area of the triangle characterized by the parameters $V_m(10)$ and k:

$$CEI_T^+ = \frac{\left(V_m(10) - V_{m,t}\right)^2}{2 \cdot |k|} \quad (3)$$

where $V_{m,t}$ is the target voids in the mixture (15% in this case).

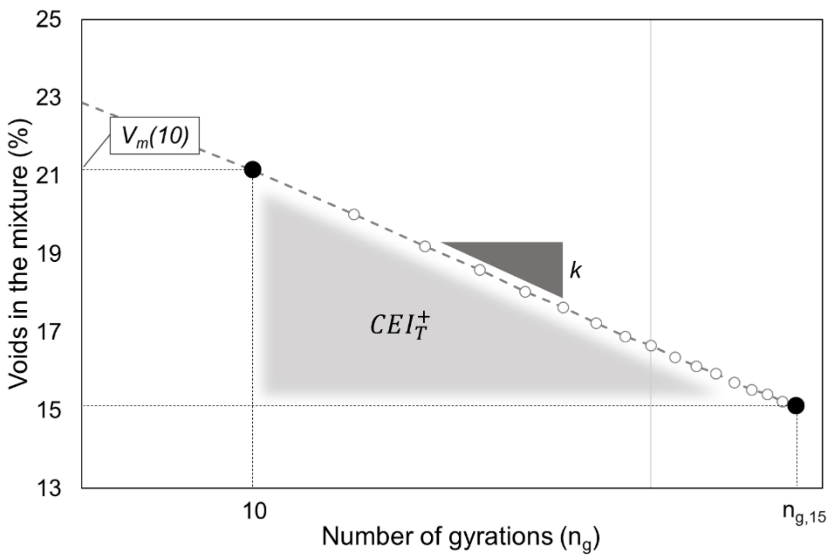

Figure 2 Graphic meaning of CEI_T^+, $V_m(10)$ and k parameters

3.3.2 Water Loss

Water loss was monitored along curing, measuring the specimens' mass after 1, 3, 5, 7, 14, 21 and 28 days for mixes produced with Emulsion A and after 1, 3, 7, 14, 15, 17, 21 and 28 days for mixes produced with Emulsion B. The water loss was calculated as:

$$\Delta W(t) = \frac{W_0 - W(t)}{W_{TOT}} \cdot 100 \tag{4}$$

where $\Delta W(t)$ is the water loss (%) at the curing time t, W_0 is the initial mass of the specimen; $W(t)$ is the mass of the specimen at the curing time t and W_{TOT} is the total amount of water in the specimen, constituted by absorbed water, bitumen emulsion water and added water for compaction.

3.3.3 Indirect Tensile Strength (ITS)

The Indirect Tensile Strength (ITS) test is used to investigate both the effect of the production temperature and of the emulsion source, as the resistance of the binding phase is assessed [46]. The test was performed according to ASTM D6931, at a testing temperature of 25 °C and on three replicates for each mixture produced. The test measures the tensile strength along the vertical diametral plane of the specimen as:

$$ITS(kPa) = \frac{2000 \cdot P(N)}{\pi \cdot D(mm) \cdot l(mm)} \tag{5}$$

where ITS is the tensile strength, P is the maximum compressive load, l is the specimen height and D is the specimen diameter.

The ITS test was performed on all mixes after the first period of curing (14 days) and after the second period of curing (14 days), to investigate the evolution of strength due to the additional curing period.

3.3.4 Indirect Tensile Stiffness Modulus (ITSM)

The Indirect Tensile Stiffness Modulus (ITSM) test, together with the water loss monitoring, can be carried out during the curing process to evaluate the mechanical properties evolution [37, 47]. The test was performed according to EN 12697-26 (Annex C), at a testing temperature of 25 °C and on three replicates for each mixture produced with only Emulsion B. The test measures the average stiffness modulus after the application of 5 pulses with a rise time of 124 ± 4 ms. For each pulse, the stiffness modulus is obtained as:

$$ITSM\ (MPa) = \frac{F\ (N) \cdot (R + 0.27)}{l\ (mm) \cdot H(mm)} \tag{6}$$

where F is the peak value of the applied repeated vertical load, H is the amplitude of the horizontal deformation, l is the mean thickness of the specimen and R is the Poisson's ratio (assumed as 0.35).

The test was performed to study the development of stiffness along curing, hence the measurements were taken after 1, 3, 7, 14, 15, 17, 21 and 28 days.

3.3.5 Scanning Electron Microscope

The Scanning Electron Microscope (SEM) was performed on samples obtained from the broken specimens produced with only Emulsion A, after 14 and 28 days to verify if changes in the microstructure are seen with different production temperatures. The equipment employed allowed to have pictures of samples with an average dimension of 20 mm. Although organic elements are recommended to be treated on the surface before processing with SEM, no pretreatment was applied in this case, so as not to modify the nature of the material. In other works, researchers performed SEM analysis to evaluate the microstructure in cold bituminous mortars containing cement or other additives [48-50].

4 RESULTS ANALYSIS

4.1 Workability and compactability

Figure 3 shows the compaction curves for the studied mixes. For simplicity, one reference curve for each mixture was chosen. The experimental points collected start from 1 gyration although the part after 10 gyrations is highlighted. In fact, points at 10 gyrations represent the $V_m(10)$ values, whereas the remaining part of the curves is described with the slope parameter k (Eq. (2)). It can be observed that between 1 and 10 gyrations, mixtures are placed in the same order. This means that the parameter $V_m(10)$ is consistent with the value after 1 gyration. Among the studied mixtures, the difference in workability $V_m(10)$ is more visible than the difference in compactability k. As already explained, it is also possible to use these two parameters to evaluate the area of the triangle CEI_T^+. CEI_T^+ values for all the specimens produced were calculated following the trapezoidal rule and by Eq. (3). It was observed that the values obtained with both calculations are perfectly superposing. Such results confirm that the approximation of the compaction curve in the semi-logarithmic plane as a straight line after 10 gyrations is valid. As a consequence, CEI_T^+ can be described using two parameters, workability $V_m(10)$ and compactability k, and used to evaluate the effect of production temperatures and of the emulsion source.

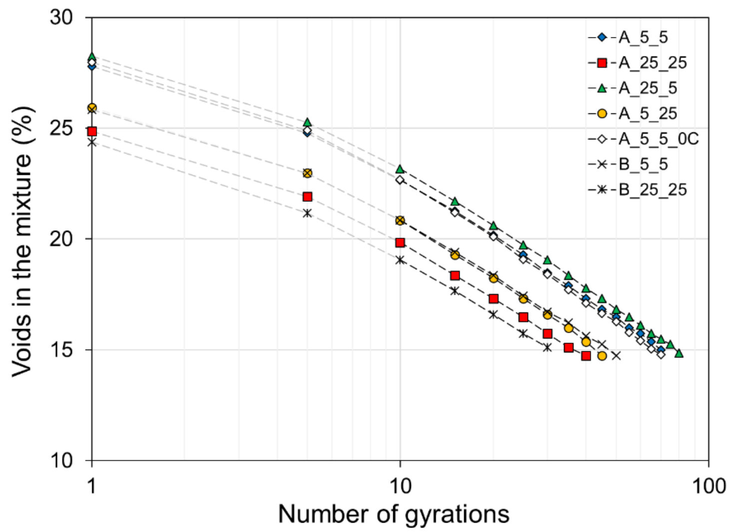

Figure 3 Compaction curves

4.1.1 Correlation between $V_m(10)$, k and CEI_T^+

Regarding the parameters just described, it is interesting to study the respective relationship that could exist among them ($V_m(10)$, k and CEI_T^+). Figure 4 shows the correlation between $V_m(10)$ and k, between CEI_T^+ and k, and between CEI_T^+ and $V_m(10)$. In Figure 4, both mixtures produced with Emulsion A and B are reported. Figure 4 a) globally shows that for both emulsions used, $V_m(10)$ decreases when k decreases. According to this trend, a gain in workability is related to a loss in compactability [51]. However, experimental points related to Emulsion A show that workability significantly improves when transportation and compaction temperature increases from 5 °C to 25 °C (average $V_m(10)$ values of 23% and 19%, respectively). At the same time, average values of compactability decrease from 9.5 to 8.0. An exponential trend line with quite a good R^2 value can describe all the points in the picture (for both emulsions used). Figure 4 b) shows the influence of the compactability parameter k on the CEI_T^+ value. Also in this case, all the experimental points can be represented with an exponential trend line. It can be observed that if the value of k increases, i.e., the slope of the compaction line is higher, the compaction effort increases. In particular this happens for mixes transported and compacted at 5 °C. In fact, such mixes are characterized on one side by higher compactability, but at the same time they showed higher values of $V_m(10)$, which directly affected the compaction effort required to reach the target voids in the mixture. A very good correlation between the compaction effort CEI_T^+ and the workability $V_m(10)$ is shown in Figure 4 c). The experimental points are described by an exponential trend line with R^2 value of 0.971. In particular, points with higher $V_m(10)$ and CEI_T^+ values are related to mixtures with transportation and compaction temperatures of 5 °C. When such temperature is increased to 25 °C, mixtures with increased workability and lower CEI_T^+ are obtained. This trend is observed for both emulsions used, even though the Emulsion B gave globally lower values of $V_m(10)$ and CEI_T^+ than Emulsion A. The softer residual bitumen contained in Emulsion B probably caused a better workability and less compaction effort for the mixtures produced. According to the results, the reduced transportation and compaction temperature (5 °C) lead to an increase of the compaction effort required by the mixture, acting more clearly on the initial workability (laydown process) rather than on the compactability (densification process). Because of the good correlation that exists between the CEI_T^+ and both parameters k and $V_m(10)$

(Figure 4 b) and c), respectively), CEI_T^+ can be considered a reliable parameter that can be used to have an idea of the global compaction effort required by the studied mixes.

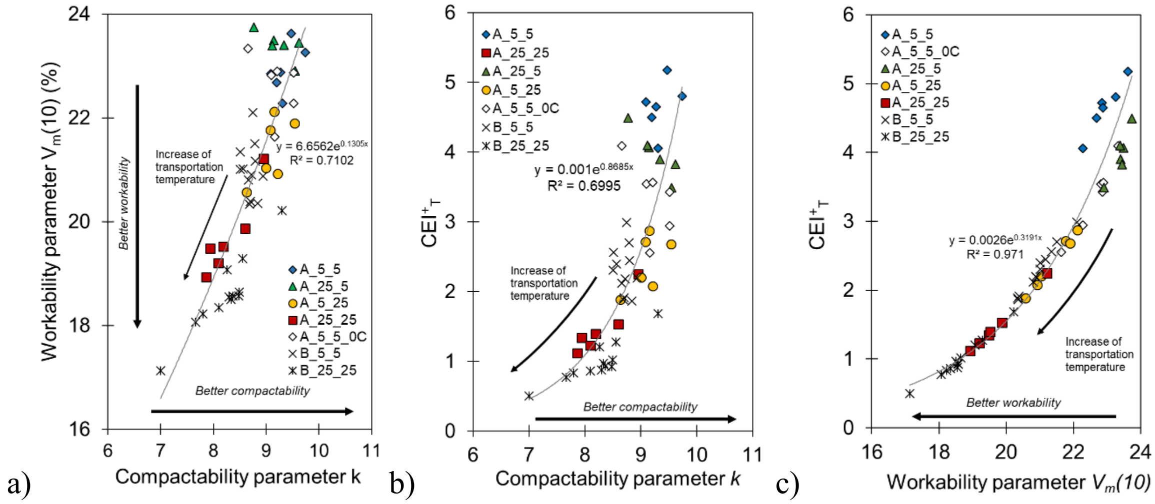

Figure 4 a) Relationship between $V_m(10)$ and k; b) Relationship between CEI_T^+ and k; c) Relationship between CEI_T^+ and $V_m(10)$

4.1.2 Effect of mixing and transportation temperatures on CEI_T^+

Figure 5 shows the effect of production temperatures (mixing, transportation and compaction) on the CEI_T^+ values of the mixes produced with Emulsion A. Mixtures produced with Emulsion B are not reported because no distinction was made between mixing and transportation temperatures. In the graphs, each point represents a compacted specimen, which were 6 for each mixture. A low mixing temperature (5 °C) did not result critical to the compaction effort required by the mixture to reach the target voids. In fact, values of CEI_T^+ are ranging between 1.1 and 5.2 regardless the mixing temperature.

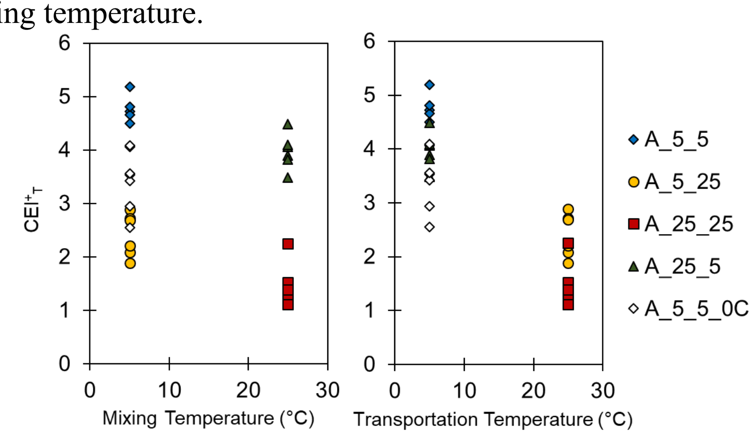

Figure 5 Relationship between CEI_T^+, mixing temperature and transportation temperature

On the other hand, the influence of transportation (and compaction) temperature is more visible. When the mixture is transported and compacted at 5 °C, the lowest CEI_T^+ obtained is around 2.6. Increasing the transportation temperature from 5 °C to 25 °C, a CEI_T^+ of 1.1 can be reached. Such results show that, during the production process of CBTM mixtures, the mixing temperature is not critically affecting the effort required for the mixtures compaction, which is instead more influenced by the transportation and compaction temperature. This also highlights that the emulsion did not prematurely break in case of low mixing temperatures (5 °C), because it is reasonable to assume that this would lead to an increase of the CEI_T^+.

In order to prove the above-mentioned statements, an analysis of variance (ANOVA) was also performed considering only the mixtures with added cement and with a level of significance $\alpha = 0.05$ (Table 4). It can be observed that both mixing and compaction temperatures statistically affect CEI_T^+, since the F statistic is higher than the critical value. Among the two temperatures studied, the transportation and compaction temperature influences more the CEI_T^+ value than the mixing temperature. Furthermore, there is no connection between the two variables.

Table 4 Results of two-way ANOVA for CEI_T^+

Source of variance	*SS*	*df*	*MS*	*F*	*p-value*	*F crit*
Transportation and compaction temperature	33.798	1	33.7978	235.8242	1.56E-12	4.3512
Mixing temperature	3.842	1	3.8419	26.8068	4.58E-05	4.3512
Interaction	0.099	1	0.0986	0.6881	0.4166	4.3512
Error	2.866	20	0.1433			
Total	40.605	23				

4.2 Water Loss

Figure 6 shows water loss evolution along curing time for all the mixes studied. It is possible to observe, for both emulsions, the increasing trend of the experimental points, which are characterized by a step in proximity of the additional curing after the first 14 days. Experimental data for each mix were modelled thanks to a modified version of the Michaelis-Menten model [30, 52, 53], which is a non-linear hyperbolic function characterized by three parameters and valid starting from 1 day:

$$y(t) = y_1 + \frac{(y_A - y_1) \cdot (t-1)}{(t-1) + (H-1)} \tag{7}$$

where $y(t)$ is the material property under investigation (in this case, water loss), t is the curing time (days), y_A is the asymptotic value, y_1 is the value related to 1 day and H is the time (days) for $y(t)$ to reach half of the gap between y_A and y_1.

It is important to highlight that terms $(t-1)$ and $(H-1)$ are used to describe the function after the first day, since what happens in the first hours of curing is dominated by a different and faster mechanism. In order to employ the model also in the second curing, the terms in Eq. (7) become $(t-14)$ and $(H-14)$, respectively.

In Figure 6 the model related to each mix is superposed to the average experimental points and standard deviation, whereas the model parameters are reported in Table 5. Figure 6 a) shows results of mixtures produced with Emulsion A. In the second part of the curing at 40 °C, water loss was measured only at 21 and 28 days, so the model is not reported in the period between 14 and 21 days, as well as the parameter H is not listed in Table 5 for the second curing.

It can be observed that in mixtures produced with Emulsion A, lowest values for $y_{A,14}$ are related to a first curing temperature of 5 °C, which are also characterized by a slower evaporation rate H_{14}. In those two mixes (A_5_5 and A_25_5) only the mixing temperature is different, and it seems to have affected the water loss after 14 days. This can be due to the cement that immediately trapped some water when mixed at 25 °C, leading to a lower water loss (72.9% instead of 77.9%). However, after a long-term curing of 28 days, all the four mixes with cement tend to similar values (comprised between 88.8% and 91.2%), whereas the mixture without cement reached 97.4%, since no water was used for the cement hydration.

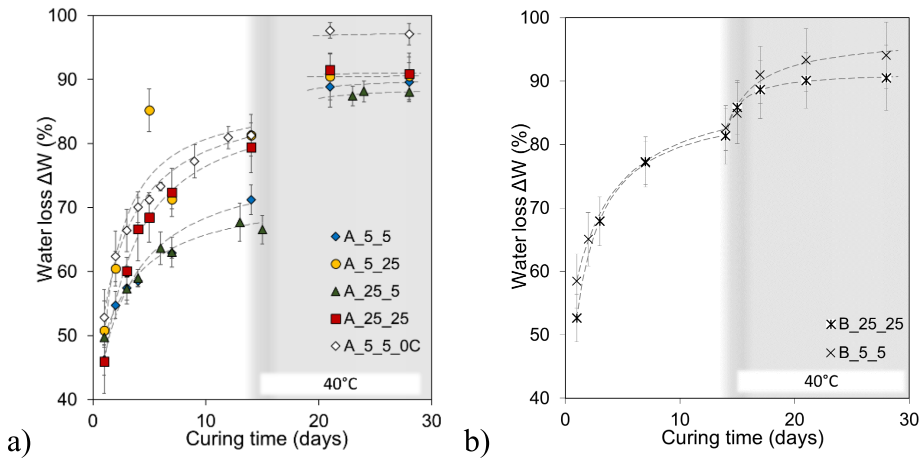

Figure 6 Water loss experimental data and superposed model for mixtures with: a) Emulsion A and b) Emulsion B

Table 5 Water loss model fitting parameters

	1st curing			2nd curing	
Mixtures	y_1 (%)	$y_{A,14}$ (%)	H_{14} (days)	y_{14} (%)	$y_{A,28}$ (%)
A_5_5	46.7	77.9	5.0	70.6	90.4
A_5_25	50.2	87.8	3.1	82.6	90.6
A_25_5	49.7	72.9	5.0	67.7	88.8
A_25_25	45.9	88.2	4.5	79.3	91.2
A_5_5_0C	53.6	88.3	4.4	81.1	97.4
B_25_25	52.6	86.9	3.4	81.5	91.5
B_5_5	58.6	89.7	4.9	82.5	97.1

Regarding mixtures produced with Emulsion B, mixture B_5_5 lost more water than mixture B_25_25 after 14 days, which is comparable to the same mixes produced with Emulsion A. This basically shows that the water evaporation mechanism does not strictly depend on temperature, but mostly on relative humidity. At the end of curing, at 28 days, the mixture B_5_5 lost 97.1% of the total water, which is very close to the value obtained for the mix A_5_5_0C, with no cement. In this case, it could mean that the cement hydration was eventually prevented in mixture B_5_5. At the same time, comparing mixes A_25_25 and B_25_25, it is highlighted that the water loss after 28 days was 91.2% and 91.5%, respectively; hence, at standard production temperatures, the emulsion did not really have an effect on the water evaporation of the mixture.

4.3 Indirect Tensile Strength (ITS)

Figure 7 shows the influence of the mixing and compaction temperatures on the ITS results at 28 days. In the picture, all the mixes produced in this study are reported. It can be seen that for mixtures produced with Emulsion A at different temperatures, both mixing and transportation temperatures do not affect the ITS. In fact, at the end of curing, all the mixes show similar strength if compared to the mixture produced at room temperature (A_25_25). As expected, a drop in the ITS values is observed in the mix without cement (A_5_5_0C).

On the other hand, the Emulsion B used to produce mixes B_5_5 and B_25_25 gives different results. On one side, the mixture produced at room temperature (B_25_25) shows a remarkable lower strength compared to the same mixture produced with Emulsion A (A_25_25). This can be caused by the softer bitumen contained in Emulsion B, which caused a lower ITS resistance [54]. Moreover, Emulsion B results to be more sensitive to low production temperatures. In fact, at 28 days, the mixture B_25_25 is characterized by an ITS value 24% higher than the mixture B_5_5.

Also in this case, a two-way ANOVA analysis was performed with the level of significance $\alpha = 0.05$ (Table 6). For mixtures with emulsion A, the F statistic is lower than the critical value, hence it is possible to conclude that both factors (mixing and transportation and compaction temperatures) do not affect the ITS results and there is no interaction between them. On the other hand, low production temperatures affect the strength of the samples produced with Emulsion B. In fact, the t-test confirms that the ITS of mixture B_5_5 is significantly lower than the ITS of mixture B_25_25 (p-value = 0.0217).

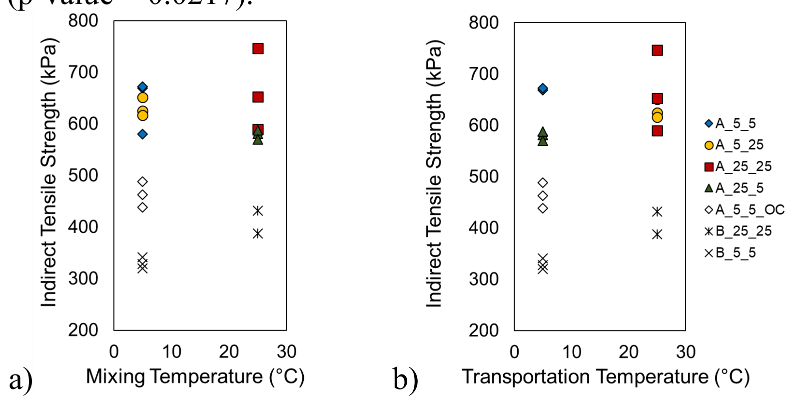

Figure 7 Effect of mixing and transportation temperatures on Indirect Tensile Strength (28 days)

Table 6 Results of two-way ANOVA for ITS results (Emulsion A)

Source of variance	SS	df	MS	F	p-value	F crit
Transportation and compaction temperature	4073.2	1	4073.2	1.7391	0.2237	5.3177
Mixing temperature	621.7	1	621.7	0.2655	0.6203	5.3177
Interaction	6490.5	1	6490.5	2.7712	0.1345	5.3177
Error	18736.8	8	2342.1			
Total	29922.3	11				

Concluding, the emulsion source resulted to be critical for the final strength level at 28 days, as well as in terms of production temperature sensitivity.

Figure 8 shows the relationship between the ITS results and the residual water in the mixtures, measured at 14 and 28 days of curing.

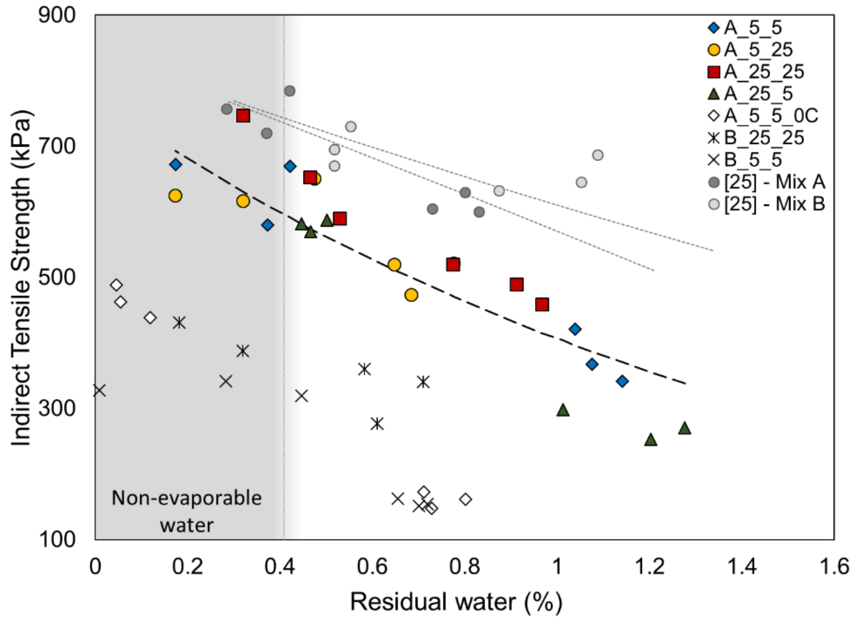

Figure 8 Correlation between residual water and Indirect Tensile Strength (at 14 and 28 days of curing)

The residual water is simply calculated as the difference between the total water and the water loss at the moment of testing [30]. The non-evaporable water, i.e. the amount of water required by the cement hydration, is estimated and reported in the graph (around 0.4% of the mixture mass). The points related to the mixtures produced with Emulsion A (only mixtures with cement) and Emulsion B are modelled separately with the original version of the Michaelis-Menten model:

$$ITS = \frac{a \cdot \Delta W}{b + \Delta W} \tag{8}$$

where a and b are regression parameters obtained through a least square minimisation.

After defining the parameters a and b, the same model is valid for the representation of the ITS results in terms of residual water. The points related to the mixtures produced with Emulsion A at different temperatures show a typical trend which links the increase of ITS with the decrease of residual water in the mixture, regardless of the mixing and transportation temperatures. This confirms that for mixes with Emulsion A, the ITS strength is strictly related to the curing conditions (i.e. residual water), rather than the production temperatures, as also shown in Figure 7. The mixture without cement, A_5_5_0C, is in fact characterized by a residual water content close to 0% at the end of curing. Hence, for mixtures with Emulsion A, the production temperatures did not permanently affect the mechanical properties, and the effect of low curing temperatures is recoverable. Mixtures produced with Emulsion B show more scattered results than Emulsion A mixes, meaning that the ITS strength is sensitive to both residual water and production temperatures.

In order to have a broader view on the results obtained, experimental points from [25] are added to the same graph, and modelled in the same way by Eq. (8). Such results are related to two different CBTM mixtures produced in different laboratories and with different properties (cement, bitumen and water contents, as well as volumetric properties). Nevertheless, even though everything related to mixture's production is different, the two mixtures reach the same level of strength after 28 days of curing, close to 800 kPa. In the present research, mixtures produced at different temperatures with Emulsion A showed a similar trend, as well as close values of ITS after 28 days.

4.4 Indirect Tensile Stiffness Modulus

Figure 9 shows results from ITSM development along curing for mixes produced with Emulsion B. The mixture produced at room temperature, B_25_25, shows a typical increasing trend of the modulus (also shown by the Michaelis-Menten model), due to the contemporary presence of cement hydration, emulsion breaking and water evaporation. After 28 days of curing, the stiffness modulus does not seem to have reached an asymptotic condition, meaning that the curing is still occurring and requires more time, even though the water evaporation is completed (residual water close to 0%).

Regarding mixtures produced at 5 °C, three specimens were tested in the initial 14 days (same three specimens tested at 3, 7 and 14 days), whereas three additional specimens were tested in the second curing period, for a total of six measurements. This was done because in the initial 14 days, the testing temperature (25 °C) could have affected the curing process at 5 °C, leading to unreliable results. The stiffness of the specimens were very low after one day of curing. Because of this, two specimens were slightly damaged during testing, which did affect the results for those specimens in the first 14 days. Between 14 and 28 days, the three not-tested specimens gave reliable results reaching a maximum final value of 2322 MPa. Results between 14 and 28 days of the two damaged specimens were not reported in Figure 9. ITSM results confirmed the temperature sensitivity of Emulsion B also in terms of stiffness. After one day of curing, it was also impossible to run a test in the small deformation field.

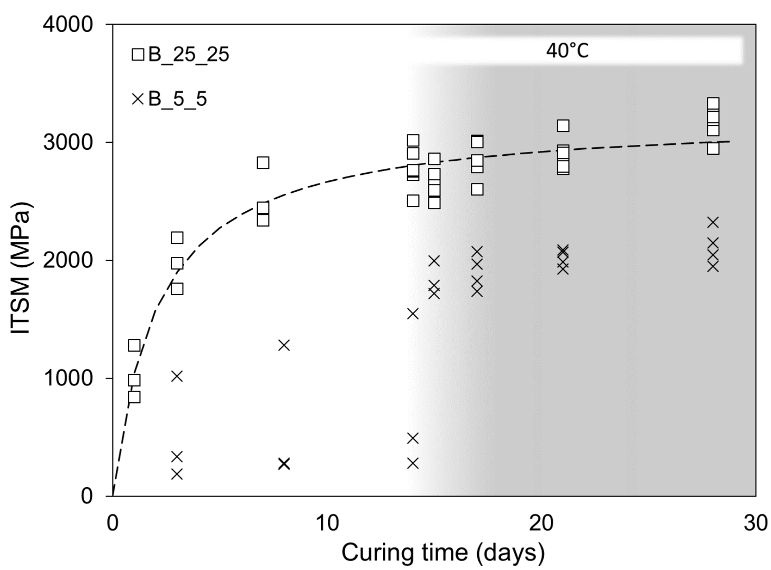

Figure 9 ITSM results

4.5 Scanning Electron Microscope (SEM)

Figure 10 shows images from SEM taken on samples of mixes produced with Emulsion A. Pictures reported are only relative to samples cured for 28 days. It is possible to observe that in mixes A_5_5 and A_5_25, the mastic is visible and the bitumen film looks uniformly spread on the aggregate faces. On the other hand, it is difficult to recognize the hydration products from cement reaction, if not for some little spots. Mixtures mixed at 25 °C (A_25_25 and A_25_5) show similar microstructure, characterized by a bitumen film less dispersed. At the same time, particles of cement seem to have reacted sufficiently to observe points in which the hydration products are visible. These images suggest that both bitumen and cement are sensitive to mixing temperature. The slow-setting nature of the emulsion particularly suitable for low temperatures allowed a more uniform dispersion at 5 °C without affecting the mechanical properties. When the mixture is mixed at 25 °C, the cement hydration is probably favored, whereas the bitumen assumes a spotted dispersion. However, as ITS results confirm, the final mechanical properties were not influenced.

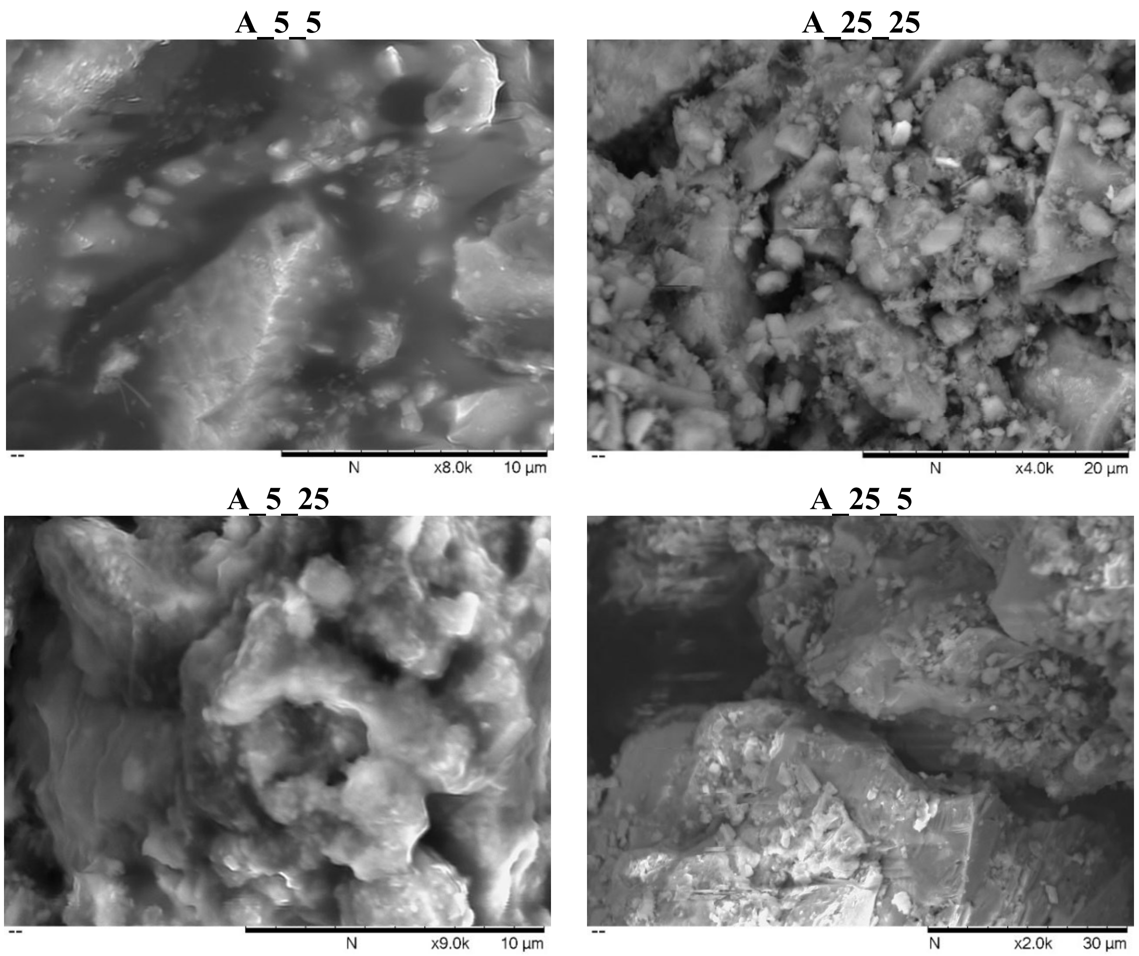

Figure 10 SEM images captured for mixes produced with Emulsion A after 28 days of curing

5 CONCLUSIONS

This study focused on the effect of production temperatures on the mechanical properties of CBTM mixtures produced with two different bitumen emulsions.

In terms of workability and compactability, the compaction energy index CEI_T^+ was selected to link both material characteristics. CEI_T^+ is affected by the low transportation temperature rather than low mixing temperature. In fact, results showed that mixes transported and compacted at 5 °C required more energy to reach the target volumetric properties. Analysing the relationship between CEI_T^+ and the workability parameter $V_m(10)$, this energy increase can be related to the workability of the mixture, i.e. the amount of voids after 10 gyrations. This evaluation was valid for both emulsion sources used, even if the emulsion produced with a softer bitumen was characterized by a better workability and required less compaction energy.

After both 1 day and 14 days of curing, water loss was lower when curing temperature was 5 °C if compared to 25 °C. However, after the long-term curing (28 days), all mixes lost almost the same amount of water, which means that it was not negatively affected by the production temperatures or the emulsion source.

In terms of ITS and ITSM results, the production at 5 °C did not affect long-term strength and stiffness of mixtures with Emulsion A. This suggests that no premature breaking of the emulsion has occurred during the production process at 5 °C, even though the compaction energy required was higher. On the other hand, mixtures produced with Emulsion B showed globally low values for ITS and ITSM, as well as a higher production temperature sensitivity. In general, the CBTM materials studied resulted highly affected by the emulsion used, both at standard (25 °C) and low (5 °C) production temperatures. This means that particular attention should be paid to the emulsion used for the production of CBTM. Results highlighted that two similar emulsion sources (both cationic slow-setting emulsions) significantly affected the final product mechanical properties.

Improved analysis and researches are recommended and strongly encouraged to clarify the effect of production temperatures on cold mixes, aspect still not standardized. More temperature and temperature combinations should be analyzed. Furthermore, attention should be paid to the bitumen emulsion composition and characteristics at the moment of production, such as storage and application temperatures.

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