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1 **Rheological and performance characterization of the bitumen**
2 **recovered from different emulsions for cold mixtures**

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13 **Rheological and performance characterization of the bitumen**
14 **recovered from different emulsions for cold mixtures**

15 In recent years, over-stabilized polymer-modified bitumen emulsions have been
16 widely used to improve the mechanical properties of cold bituminous mixtures for
17 base and subbase layers. However, the effect of the process for the polymer
18 modification of the emulsions on the binder performance have not been
19 investigated. The present paper deals with the analysis of the binders recovered
20 from a reference unmodified bitumen emulsion and two polymer-modified
21 emulsions, respectively including SBR-latex added after bitumen emulsification
22 (post- blending) and SBS added before bitumen emulsification (pre-blending). The
23 investigation included laboratory tests using a dynamic shear rheometer for the
24 linear viscoelastic characterization (frequency sweep) and the evaluation of the
25 resistance to permanent deformations (multiple stress creep and recovery) and
26 damage (linear amplitude sweep). The results showed that the polymer-modified
27 bitumen emulsions have a more pronounced elasticity with respect to the
28 unmodified emulsion, allowing achieving a higher performance against permanent
29 deformations and fatigue. In addition, the SBS modified bitumen emulsion (pre-
30 blending) has better properties than the latex modified emulsion (post-blending),
31 as a consequence of the different chemical structure that the modification process
32 produces.

33 Keywords: Bitumen emulsion; Blending method; Cold bituminous mixtures;
34 Latex; Polymer modification

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41 **Introduction**

42 Nowadays, cold bituminous mixtures (CBM) are taking more and more importance in the
43 context of road construction materials (E. Bocci et al., 2020; Godenzoni et al., 2017; Xiao
44 et al., 2018). This need arises from the fact that, in recent decades, institutions are
45 increasingly looking for eco-sustainability (Al-Merzah, Al-Busaltan, & Nageim, 2019;
46 Dulaimi, Shanbara, & Al-Rifaie, 2020). In particular, these materials allow obtaining
47 several advantages, in economic and environmental terms, with respect to both hot and
48 warm mixtures, due to the reduction of the energy necessary for bitumen and aggregate
49 heating (Godenzoni et al., 2017; Sangiorgi et al., 2017). Moreover, these materials can be
50 produced with a high amount of reclaimed asphalt (RA), reducing the use of the virgin
51 aggregate (M. Bocci et al., 2010; Godenzoni et al., 2017; Stimilli et al., 2013; Xiao et al.,
52 2018). The technical solution to reduce binder viscosity without heating deals with the
53 use of foaming processes or bitumen emulsification (Khan et al., 2016).

54 Foamed bitumens are obtained through the injection of cold water in hot
55 bitumen (160-180 °C). The heat transfer from bitumen causes the almost instantaneous
56 evaporation of the water and the generated steam determines the immediate expansion
57 of the binder in the form of foam.

58 Bitumen emulsions (BE) are constituted by two immiscible liquids: the solution
59 of water and emulsifying agent is the "continuous phase" while the bitumen, dispersed in
60 the form of small droplets, is the "internal phase" (Querol et al., 2019). The BE needs to
61 have a dispersion that is stable under storage, mixing, and pumping, but that breaks down
62 (or "sets", which means that bitumen and water separate from each other) upon contact
63 with aggregates to form a final film of binder (Marasteanu & Clyne, 2006). To improve
64 this storage stability, the emulsifying agents are used, as they decrease the interfacial
65 tension between bitumen droplets and water in order to stabilize the system against

66 coalescence and allow the emulsification (Ronald & Luis, 2016; Tadros, 2013). The
67 production of BE is not a simple task, because for its formulation must take into account
68 a series of different parameters related not only to the manufacturing process, but also to
69 the final properties for which the emulsion has been designed (Querol et al., 2019). There
70 are two different methods of BE production, the conventional method (thought colloid
71 mills) and the high internal phase ratio method, but currently, the most widely used
72 system is the first one (Querol et al., 2019).

73 The main properties that describe a BE, and that are generally named by the
74 various international standards for their classification, are particle polarity, binder
75 content, breaking index and presence/absence of polymers (D. Lesueur, 2011). BEs can
76 be anionic, cationic or nonionic and can include up to 70% of bitumen (Querol et al.,
77 2019). Currently, cationic emulsions with a residual bitumen content about 60% are most
78 used in the asphalt industry (Marasteanu & Clyne, 2006; Ronald & Luis, 2016). The
79 breaking index represents the breaking speed when the emulsion is put in contact with
80 the aggregates. In particular, an emulsion can be classified in rapid setting (RS), medium
81 setting (MS) and slow setting (SS). The choice depends on the BE application. RS
82 emulsions set quickly in contact with clean aggregates of low-surface area, such as the
83 chippings used in chip seals (surface dressings). MS emulsions set sufficiently less
84 quickly that they can be mixed with aggregates of low surface area, such as those used in
85 open-graded mixes. They can also be used in applications such as tack coat or fog seal.
86 SS emulsions are mixed with reactive aggregates of high surface area and would be used
87 for a dense cold mix which has a high filler content and consequently high reactivity,
88 such as micro-surfacing and slurry seal (Salomon, 2006). In reality, there is a further
89 category, namely the over-stabilized (OS) emulsions, which set more slowly than SS and

90 are typically used in the production of CBM, as they can withstand the contact with
91 Portland cement without breaking, for a certain time (Grilli et al., 2018).

92 In order to improve some performance characteristics of the residual bitumen, the
93 BEs can be modified with polymers (Lesueur, 2011). Particularly in the case of CBM, the
94 BEs including polymers allow increasing flexibility and plasticity in low-temperature
95 conditions, strength and thermal stability at high temperatures, anti-ageing ability and
96 fatigue resistance (Zhang & He, 2007), extending the whole pavement service life (Shafii
97 et al., 2013). In the past, the CBMs were used only for low-traffic roads or deep pavement
98 layers (subbase and foundation) because of the use of low-performing materials and the
99 lack of a good knowledge on the correct mix design and material internal interaction
100 mechanism (Xiao et al., 2018). Recently, the high properties of the polymer-modified
101 BEs have encouraged the pavement engineers to use CBM not only for subbase and
102 foundation layers but also in place of the hot-mix asphalt base, even in high-trafficked
103 roads (E. Bocci et al., 2020; Buczyński & Iwański, 2017; Sangiorgi et al., 2017; Stimilli
104 et al., 2013).

105 There are three different techniques to produce a polymer-modified BE and the
106 blending method has an important influence on the polymer network distribution, thus on
107 the performance of the final product (Forbes et al., 2001). The polymers can be added
108 through pre-blending method, simultaneous-blending method or post-blending method
109 (Shafii et al., 2011). In the pre-blending method, the polymer is added directly to the
110 bitumen prior to the emulsification process, obtaining a mono-phase emulsion. This
111 method is used for solid polymers and allows obtaining a finer polymer network within
112 the bitumen emulsion, which improves its performance. However, the high temperature
113 for production (up to 180 °C) implies an alteration of the polymer network within the
114 bitumen (as in the typical polymer-modification process of the bitumen for hot mixtures).

115 In the simultaneous-blending method, the separate streams of bitumen, polymer and
116 emulsifying agent are contemporarily milled together during the BE production whereas
117 in the post-blending method the polymer is added after bitumen emulsification. These
118 latter methods are carried out at lower temperatures (usually below 80-90 °C) and
119 therefore a reduction of energy consumption and of the polymer chain alteration is
120 achieved (Shafii et al., 2013). These techniques are used for liquid polymers and allow
121 producing the so-called “bi-phase” emulsions.

122 The most common polymers that are used as BE modifiers are latexes of different
123 types, Styrene Butadiene Styrene (SBS) and Ethylene Vinyl Acetate (EVA) (Khadivar &
124 Kavussi, 2013). According to the physical state, each polymer type can be included in a
125 BE through a specific method (pre-, simultaneous- or post-blending). SBS and EVA, in
126 a solid state, are added by pre-blending method. Differently, latex consists of Styrene
127 Butadiene Rubber (SBR) or Natural Rubber (NR) polymers dispersed in a water phase,
128 thus it is incorporated in the BE by simultaneous- or post-blending method (Lesueur,
129 2011). The result is an emulsion consisting of latex and bitumen particles that are
130 dispersed into the aqueous phase. When BE breaks, the latex particles are spontaneously
131 transformed into a continuous polymer film covering bitumen particles (Takamura &
132 Lubbers, 2000), allowing increasing the bitumen viscosity at high temperatures
133 (thickening) and reducing the thermal cracking at low temperatures (Ruggles (2004)).

134 In the recent years, many researches have focused on the characterization of
135 polymer-modified BE.

136 Zhang and He (2007) studied the performance of the BEs modified by a
137 compound of water-epoxy resin emulsion and SBR latex (post-blending) and showed that
138 these materials, in terms of low-temperature cracking and permanent deformation
139 resistance, adhesion and durability, exceed ordinary BE.

140 Li et al. (2019) also investigated the performance of waterborne epoxy modified
141 BE (manufactured through a particular process, where epoxy was pre-blended with
142 bitumen and curing agent was post-added to the emulsion) as a potential high-
143 performance cold binder and found that this modification allows a higher complex
144 modulus (G^*) and a lower angle phase (δ) to be obtained with respect to the base bitumen,
145 indicating that the epoxy resin raised the elastic features of the binder. Moreover, the
146 multiple stress creep-recovery (MSCR) test results showed that the percent recovery
147 increased and the non-recoverable creep compliance decreased, as the epoxy polymer
148 network strengthened the elastic properties of bitumen. Finally, the linear amplitude
149 sweep (LAS) test results demonstrated that the fatigue life of bitumen emulsion residue
150 was improved by waterborne epoxy resin.

151 Waterborne epoxy was also used in BE modification by Pan et al. (2019), who
152 compared its effects with those of SBR latex and colloidal silica sol (polymers added by
153 post-blending process). Test results showed that the modifiers allow improving the
154 residual bitumen bonding strength and the rheological properties (increased complex
155 modulus and reduced phase angle at the low frequency region), but the polymer
156 combination and dosage need to be carefully selected.

157 Khadivar and Kavussi (2013) studied the rheological characteristics of SBR and
158 NR modified BEs (post-blending) at average temperatures, and proved that the addition
159 of these polymers allows increasing the stiffness of the binder residue and reducing its
160 temperature susceptibility, with respect to a traditional BE. Moreover, the SBR
161 modification (but not NR modification) allows decreasing the non-recoverable creep
162 compliance and increasing the elastic recovery.

163 A research carried out in southern Spain (Carrera et al., 2014; Cuadri et al., 2016)
164 experienced the possibility of using a recycled polyurethane modifier in BE. In terms of

165 applicability, polyurethane-modified bitumens can be emulsified (pre-blending process)
166 at much lower temperatures than in the case of other modifiers. The rheological
167 characterization conducted on the BE residues at 60 °C showed an improved rutting
168 resistance with respect to unmodified recovered bitumen.

169 Abedini et al., (2017) studied the rheological properties of BE modified (post-
170 blending) with different percentages of nitrile-butadiene rubber (NBR). From dynamic
171 shear rheometer (DSR) analysis it was observed that higher NBR dosages increase
172 complex modulus and decrease phase angle, which determine an increase in the elastic
173 properties. Moreover, increasing NBR polymer content (up to 6%) led to an increase of
174 $G^*/\sin\delta$ and consequently an improvement of rutting resistance. The same research team
175 also investigated the effect of two types of SBR latexes, ordinary linear SBR and carboxyl
176 X-structure SBR (included through post-blending process), as BE modifiers. The results
177 demonstrated that X-SBR, more than ordinary SBR, proved to increase the recovered
178 bitumen stiffness (Abedini et al., 2016) and to reduce the non-recoverable creep
179 compliance and the temperature sensitivity (Abedini et al., 2018).

180 Jiang et al. (2018) tested three different types of recovered bitumen from polymer-
181 modified BE, respectively including SBR latex (post-blending), Styrene-ethylene-
182 butylene-styrene SEBS copolymer (pre-blending) and Chloroprene rubber CR latex
183 (post-blending). The study mainly focused on the high-temperature performance,
184 investigated in terms of $|G^*/\sin\delta$ and non-recoverable creep compliance J_{nr} . The results
185 showed that the residues from modified BEs, especially the one including CR latex, have
186 lower high-temperature sensitivity and higher rutting resistance with respect to the other
187 recovered bitumens.

188 The recalled researches, which are very topical, denote that the characterization
189 of polymer-modified BE is a very felt theme by the scientific community. Even if many

190 studies have been carried out, only few of them seem to investigate the effect of the
191 modification process in the recovered bitumen properties. At the light of this, the
192 objective of the present research was the rheological and performance analysis of the
193 bitumen recovered from different BEs specifically designed for CBM. In particular, the
194 binders recovered from traditional, SBS-modified (pre-blending) and SBR latex-modified
195 (post-blending) BEs were investigated.

196 **Objective**

197 The study focused on the comparison between the bitumens recovered from 3 different
198 types of BEs for CMB: an SBR latex-modified BE produced through post-blending
199 process, an SBS-modified BE produced through pre-blending process and a reference
200 unmodified BE. It is highlighted that the tested polymer-modified BEs are characterized
201 by similar values of ring-and-ball softening point of the residue, as required in the
202 specifications for base-layer CBM by Italian national highways authority (ANAS, 2010).

203 The comparison between the 3 recovered bitumens was carried out in terms of:

- 204 • linear viscoelastic (LVE) behaviour, investigated by means of strain sweep and
205 frequency sweep tests with dynamic shear rheometer (DSR) for the construction
206 of the complex modulus and phase angle master curves. In addition, the 2S2P1D
207 model parameters for the 3 bitumens were analysed;
- 208 • resistance to permanent deformations, examined through multiple stress creep and
209 recovery (MSCR) test;
- 210 • fatigue behaviour, studied through linear amplitude sweep test (LAS).

211 In order to clearly distinguish between the manufacturing process of the polymer-
212 modified BEs, the residue from SBR latex-modified BE was coded with LMEB (latex-
213 modified emulsified bitumen), as the emulsification was carried out on the unmodified

214 bitumen before polymer addition, while the residue from SBS-modified BE was coded
215 with EPMB (emulsified polymer-modified bitumen), as the emulsification was carried
216 out on the SBS-modified bitumen. Finally, the residue from the unmodified emulsion was
217 coded with EB (emulsified bitumen). Table 1 summarizes the material nomenclature.

218 **Materials**

219 The BEs selected for the present study were commercial products typically marketed in
220 Italy for CBM applications. Table 2 shows the physical properties of the BEs and of the
221 recovered bitumens as declared by the producer. It can be observed that the 3 emulsions
222 have the same characteristics in terms of water content (60%) and breaking index. The
223 latter, in particular, which is equal to 10, identifies the over-stabilized emulsions (EN
224 13808, 2013), which have a mixing stability with cement lower than 2% according to a
225 specific test procedure (EN 12848, 2009) and thus are suitable to be used in CBM.

226 On the contrary, some differences can be noticed in the recovered bitumens
227 properties (ring-and-ball softening point and elastic recovery). In fact, the BE producer
228 adjusts the modifier dosage in order to match the acceptability interval defined by Italian
229 standards (ANAS, 2010) for the recovered bitumen softening point (55-75 °C). To this
230 aim, LMEB and EPMB respectively include 3.3% of SBR and 4.0% of SBS by total
231 bitumen weight, resulting in a ring-and-ball softening point value of 62 °C for both.
232 However, the different polymer dosage and blending process determine different elastic
233 recovery values, higher for EPMB, but complying with the specifications (elastic
234 recovery > 55%).

235 **Recovery of the bitumen from the emulsion**

236 The main processes to recover the bitumen from the emulsion, modified or unmodified,
237 are based on three different methods: "distillation and high-temperature evaporation

238 method", "low-temperature evaporation method" and "solvent precipitation method"
239 (Waters et al., 2008). Any national specification has a different indication on the method
240 to be followed for the recovery of BE bitumen and this can influence the rheological
241 properties of the recovered binder. In particular, if the procedure is carried out at high
242 temperatures (over 180 °C), the rheological properties of the bitumen can be somehow
243 influenced (Shafii et al., 2011). This issue is highly important in the case of polymer-
244 modified BE, for which the polymer morphology can be significantly affected by the
245 temperature and the duration of the recovery method (Marasteanu & Clyne, 2006). In
246 particular, the high temperatures required by some of the methods significantly alter or
247 damage the microscopic structure of the emulsion and therefore the residues do not
248 represent the field conditions, where the CBM construction is done at ambient
249 temperature (Takamura & Lubbers, 2000). This is especially true with latex modified
250 emulsions, for which the simultaneous- or post-blending manufacturing process does not
251 entail any polymer swelling or digestion (Lesueur, 2011).

252 In the present research, the bitumen was recovered from the bitumen emulsion by
253 means of a low-temperature evaporation method, according to EN 13074-1 (2019). The
254 European standard provided the BE conditioning at room temperature for 24 h
255 (approximately 20 °C) followed by 24 h at 50 °C. The recovered bitumens were stored in
256 a cool room (at a temperature of approximately 15 °C) in opaque and sealed containers
257 for at maximum 7 day before testing.

258 **Experimental programme and test procedure**

259 The experimental programme included the analysis of the rheological properties, the
260 resistance to permanent deformations and fatigue behaviour of the recovered bitumens.

261 The rheological characterization was carried out by means of strain sweep and
262 frequency sweep tests. Strain sweep tests were performed in order to determine the

263 threshold of LVE region γ_{lim} , i.e. the strain value corresponding to a $|G^*|$ deviation equal
264 to 95% of its initial value $|G^*_{in}|$. The test parameters were frequency $f = 1.59$ Hz,
265 temperature $T = 5, 15$ and 30 °C, a range of strains γ between 0.005-5% and 2 repetitions
266 for each material. Frequency sweep tests aimed to measure the complex modulus of the
267 bitumens. In particular, these tests were performed at a constant strain $\gamma = 0.2\% < \gamma_{lim}$,
268 over a range of frequencies (from 0.1 to 10 Hz) and temperatures (0, 4, 10, 16, 22, 28, 34,
269 40, 46, 52, 58 °C). Temperatures > 58 °C were not investigated as CBMs, used for base,
270 subbase or foundation layers, rarely reach this value in their service life.

271 The strain and frequency sweep tests were carried out through a dynamic shear
272 rheometer (DSR) in plate-plate configuration, applying sinusoidal load curves in control-
273 strain mode. For $T \leq 40$ °C, a diameter of 8 mm and gap of 2 mm were used, while for
274 $T \geq 28$ °C a diameter of 25 mm and gap of 1 mm were adopted. According to EN 14770
275 (2012), the test protocol provided two repetitions for each test. In case of result dispersion
276 ($|G^*|$ out of the range of 10%), a third repetition was carried out.

277 The resistance of the recovered bitumens against permanent deformation was
278 investigated through multiple stress creep and recovery (MSCR) test, according to
279 EN 16659 (2016). The test was carried out using a DSR with 25 mm diameter and 1 mm
280 gap. The sample was loaded at constant stress for 1 s, then allowed to recover for 9 s. Ten
281 creep and recovery cycles were run at 0.1 kPa creep stress followed by 10 cycles at
282 3.2 kPa creep stress. The test was repeated at the temperatures of 28, 34 and 40 °C, which
283 were assumed to be the typical intermediate/high temperatures for CBM in service. The
284 protocol provided 2 repetitions for each temperature. Non-recoverable creep compliance
285 (J_{nr}), and percent recovery (R) were determined.

286 The fatigue behaviour was studied by means of linear amplitude sweep (LAS)
287 test, according to AASHTO TP101 (2014). The test was carried out using a DSR with 8

288 mm diameter and 2 mm gap. The protocol provided a frequency sweep test at a constant
289 strain $\gamma = 0.1\%$ over a range of frequencies from 0.2 to 30 Hz. This preliminary LVE
290 analysis allowed calculating the undamaged material parameter α , which represents the
291 inverse of the slope of the storage modulus vs frequency curve in the log-log plane. Then,
292 an amplitude sweep test was carried out at 5 Hz by continuously increasing the strain
293 from 0.1% to 30% with a constant rate of 0.05% per second (corresponding to a test
294 duration of 3100 cycles, i.e. 620 s). The sample failure was assumed in correspondence
295 of the peak value in the shear stress τ vs shear strain γ curves. Through the application of
296 the ViscoElastic Continuum Damage (VECD) model the normalized complex modulus
297 C vs damage D curve were obtained for each bitumen. In addition, the binder fatigue
298 behaviour was estimated by the following Equation 1:

$$299 \quad N_f = A \cdot \gamma_{max}^B \quad (1)$$

300 where γ_{max} was the maximum expected binder strain for a given pavement structure and
301 the coefficients A and B were calculated from the parameters in the C - D curve equation,
302 from the LVE parameter α and from the damage at failure D_f , i.e. the D value in
303 correspondence of the peak in the τ vs γ curve. As suggested by Teymourpour & Bahia
304 (2014), the LAS test (both frequency sweep and amplitude sweep steps) were carried out
305 at a temperature which allowed an initial $|G^*|$ value between 10 and 60 MPa to be
306 achieved, to avoid bulging of adhesive issues in the sample. To comply with this
307 indication, the test temperature was fixed to 15 °C. For each material, 3 test repetitions
308 were carried out.

309 **Result analysis**

310 *Identification of LVE region*

311 Table 3 and Figure 1 show the results of the strain sweep tests for the 3 recovered
312 bitumens and allow the threshold of LVE region, γ_{lim} , to be identified. The lowest width
313 of the LVE region was found for LMEB at the temperature of 5 °C, with a $\gamma_{lim} = 0.78\%$.
314 Therefore, the strain value $\gamma = 0.2\%$ (about one third of the minimum γ_{lim} value) was
315 selected for frequency sweep test implementation.

316 *Procedure for LVE analysis and modelling*

317 The measured rheological data were plotted in the Black and Cole-Cole diagrams to
318 evaluate the reliability of the experimental results, assess the time-temperature
319 equivalency and thermo-rheologically simplicity for the tested bitumens, estimate the
320 magnitude of the glassy asymptote and the preponderance of the viscous or elastic
321 behaviour at the different temperatures investigated (Lesueur et al., 1996).

322 As shown in the following sections, all the recovered bitumens were considered
323 as thermo-rheologically simple and the time-temperature superposition principle (TTSP)
324 was applied to determine complex modulus master curves and shift factor relationships.
325 The rheological data were shifted with respect to time until the isothermal curves merge
326 into a single smooth function at the reference temperature of 34 °C. The estimation of the
327 temperature shift factors was carried out according to the closed form shifting algorithm
328 (Gergesova et al., 2011), based on the minimization of the area between two successive
329 isothermal curves. The 2S2P1D model (Olard & Di Benedetto, 2003) was applied to
330 simulate the rheological behaviour of the binders, while the Williams-Landel-Ferry
331 (WLF) law (Ferry, 1980) was used to model the shift factor trend with temperature. The

332 2S2P1D model includes a linear dashpot in series with two parabolic elements and with
 333 the spring of stiffness $G_\infty - G_0$ assembled in parallel with a second spring (G_0) (Figure 2).

334 In detail, according to the 2S2P1D model, the equation for complex modulus is
 335 given by Equation (2)

$$336 \quad G^*(i\omega\tau) = G_0 + \frac{G_\infty - G_0}{1 + \delta(i\omega\tau)^{-k} + (i\omega\tau)^{-h} + (i\omega\tau\beta)^{-1}} \quad (2)$$

337 where G_0 is the static shear modulus when $\omega \rightarrow 0$ ($G_0 = 0$ for binders); G_∞ is the glassy
 338 shear modulus when $\omega \rightarrow \infty$; $\omega = 2\pi f$ is the angular frequency; i is the imaginary unit
 339 defined by $i^2 = -1$; τ is the characteristic time; k , h and δ are dimensionless parameters
 340 such that $0 < k < h < 1$; β is a function of the dashpot viscosity η such that
 341 $\eta = (G_\infty - G_0)\beta\tau$. Based on the TTSP, τ can be determined as in Equation 3:

$$342 \quad \tau(T) = a_T \cdot \tau_0 \quad (3)$$

343 where a_T is the shift factor at the temperature T and $\tau_0 = \tau(T_0)$ is determined at the reference
 344 temperature T_0 . The temperature shift factors can be obtained using the Williams-Landel-
 345 Ferry (WLF) equation for bituminous materials (Equation 4):

$$346 \quad \log \frac{a_T(T)}{a_T(T_0)} = - \frac{C_1(T - T_0)}{C_2 + (T - T_0)} \quad (4)$$

347 where a_T is the shift factor at temperature T , T_0 is the reference temperature, C_1 and C_2
 348 are empirical constants.

349 ***Rheological behaviour***

350 Figures 3 and 4 depict the rheological data measured through frequency sweep tests in
 351 the Black ($|G^*|$ vs ϕ) and Cole-Cole (G_2 vs G_1) diagrams. From the Black diagram, the
 352 variability of $|G^*|$ and ϕ can be observed. In particular, $|G^*|$ ranged between 483 Pa and
 353 110.5 MPa for EB, between 955.5 Pa and 80.3 MPa for LMEB, between 3115 Pa and
 354 91.3 MPa for EPMB. The phase angle ϕ ranged between 27.6° and 87.6° for EB, between
 355 28.9° and 80.0° for LMEB, between 24.5° and 64.4° for EPMB.

356 To assess the validity of the TTSP and thus the thermorheologically simple
357 behaviour, the G^* values measured at different frequencies and temperatures should align
358 on a single smooth curve when plotted in the Black and Cole-Cole diagrams. The results
359 in Figures 3 and 4 confirmed the validity of the TTSP for all the tested bitumens. This is
360 an important finding, especially for LMEB and EPMB, because they are characterized by
361 the presence of polymers.

362 The Black and Cole-Cole diagrams already highlighted the different rheological
363 behaviour of the tested bitumens. In particular, EB showed the typical trend of a neat
364 bitumen, with G^* uniformly moving from a purely viscous ($|G^*| = 0$, $\phi = 90^\circ$) to a purely
365 elastic ($|G^*| \approx 1$ GPa, $\phi = 0^\circ$) behaviour when decreasing temperature or increasing
366 frequency. Differently, EPMB curves were positioned on the left and below EB curves in
367 the Black and Cole-Cole diagrams, respectively, denoting a higher relevance of elastic
368 component to detriment of the viscous component. The LMEB bitumen showed an
369 intermediate behaviour between EB and EPMB.

370 Figures 5 and 6 show the master curves of $|G^*|$ and ϕ at the reference temperature
371 of 34 °C. From Figure 5 it can be noted that at low frequencies/high temperatures EPMB
372 gave the highest $|G^*|$, approximately 6 times that of EB and 3 times that of LMEB.
373 Differently, at high frequencies/intermediate-low temperatures the stiffness values were
374 comparable between the different bitumens. In detail, a slightly higher $|G^*|$ was observed
375 for EB with respect for LMEB and EPMB, which provided almost identical values.

376 From Figure 6 it can be observed that EPMB had the lowest phase angle at all
377 frequencies/temperatures, indicating that this bitumen is characterized by the most
378 emphasized elastic features. At low frequencies/high temperatures EPMB and LMEB
379 showed a plateau in the phase angle master curves, indicating that the polymer
380 modification over a certain T (or below a certain f) inhibited the viscous behaviour

381 yielding some elastic features to the binder. By continuing increasing T /reducing f , ϕ
382 values seemed to tend again towards 90° for both LMEB and EPMB (more slowly for
383 this latter). On the left side of ϕ master curves, LMEB provided an intermediate behaviour
384 between EB (markedly viscous) and EPMB (more elastic), while on the right side (high
385 frequencies/intermediate-low temperatures) LMEB showed a higher viscosity, slightly
386 greater than that of EB. This was maybe related to the physical dispersion of the SBR-
387 latex within the binder matrix. The presence of a continuous film of polymer covering
388 bitumen droplets (Forbes et al., 2001) probably allowed a higher molecule mobility, at
389 low temperatures, than in the case of EB or EPMB, where the continuous phase was
390 constituted by bitumen (or polymer-modified bitumen).

391 In general, it is interesting to observe that the rheological behaviour of LMEB was
392 closer to that of EB than to that of EPMB, in spite of its polymer content which is slightly
393 lower (3.3% vs 4.0%). This indicates that the complex modulus of the polymer-modified
394 BE depends not only on the content but also on the type of modifier and in particular on
395 the modification process, which entails a different morphology of the bitumen chemical
396 structure (Forbes et al., 2001).

397 In Figures 5 and 6 the experimental data are superimposed to the curves of
398 2S2P1D model. According to Di Benedetto et al., (2004), the model was calibrated by
399 minimizing the sum of the squared difference between measured and simulated complex
400 modulus, declined in its storage and loss components (Equation 5).

$$401 \quad \sum_{i=1}^N \left((G_{1,meas}(\omega_i) - G_{1,model}(\omega_i))^2 + (G_{2,meas}(\omega_i) - G_{2,model}(\omega_i))^2 \right) \quad (5)$$

402 Both the $|G^*|$ and ϕ plots prove that the 2S2P1D model well simulated the
403 experimental data, even the plateau in the phase angle master curves of LMEB and
404 EPMB.

405 Table 4 shows the parameters of 2S2P1D and WLF models. The graphical
406 comparison between the model parameters is provided in Figure 7. The analysis also
407 included Glower-Rowe parameter, which characterizes the stiffness and relaxation
408 properties of a bitumen (King et al., 2012) and assumes higher values when increasing
409 brittleness. According to Rowe et al., (2014), this parameter can be calculated from DSR
410 data using the following Equation 6:

$$411 \quad GR = \frac{|G^*|(\cos \phi)^2}{\sin \phi} \quad (6)$$

412 where $|G^*|$ and ϕ are the stiffness modulus and phase angle at the temperature of 15 °C
413 and angular frequency of 0.005 rad/s deduced from the 2S2P1D model.

414 The results showed that the parameters k and h , which represent the order of
415 derivation of the two parabolic elements included in the 2S2P1D model, were comparable
416 between the different recovered bitumens. Likewise, also the characteristic time τ_0 was
417 similar for EB, LMEB and EPMB. Differently, the parameters δ and β increased when
418 moving from EB, to LMEB, to EPMB. The parameter δ is a positive non dimensional
419 coefficient balancing the contribution of the first parabolic element in the global
420 behaviour. The higher values of δ measured for the recovered bitumens from polymer-
421 modified BE reflected the lower height of the pinnacle point of the curves in the Cole-
422 Cole diagram, so denoted higher elastic features, in particular for EPMB. The parameter
423 β is a function of the dashpot viscosity ($\eta = G_\infty \beta \tau$) and was found to be associated with
424 asphaltene (polar molecules) content (Yusoff et al., 2013). The higher values of β
425 obtained for LMEB and EPMB indicated the tendency of $|G^*|$ and ϕ master curves to
426 reach more slowly the equilibrium values of 0 Pa and 90°, i.e. to preserve a stiff and
427 elastic behaviour at high temperatures. The GR parameter resulted higher for the
428 recovered bitumens from polymer-modified BE than for EB. In particular, it was the

429 highest for EPMB. This result was due to the higher $|G^*|$ and lower ϕ values and did not
430 indicate a higher brittleness but the predominance of the elasticity over viscosity. In
431 addition, all the GR values were far from the limit of 180 kPa, recognized by Rowe et al.,
432 (2014) as the threshold over which there can be damage onset. Finally, the WLF
433 parameters C_1 and C_2 slightly increased when passing from EB to LMEB, to EPMB, but
434 the comparable values of the ratio C_2/C_1 denoted a similar temperature sensitivity for the
435 recovered bitumens.

436 ***Results of the MSCR tests***

437 Figures 8 and 9 show the results from MSCR tests in terms of average non-recoverable
438 creep compliance (J_{nr}) and average percent recovery (R) at three test temperatures (28, 34
439 and 40 °C) and two different creep stresses (0.1 and 3.2 kPa).

440 From the graphs it can be observed that, in the investigated intervals, EB and
441 LMEB were poorly dependant on the creep stress but had different J_{nr} and R as a function
442 of test temperature. On the contrary, EPMB exhibits the same behaviour when varying
443 temperature and creep stress, especially in relation to the J_{nr} parameter.

444 The EB binder manifested the worst behaviour against permanent deformations,
445 since J_{nr} was higher (i.e. it accumulated a greater deformation with the loading cycles)
446 and R was lower (i.e. the material had a very low recovery during the rest period) with
447 respect to the polymer-modified BE. Instead, the EPMB had very good resistance against
448 permanent deformations and showed a high elasticity, thanks to the low J_{nr} and high R .
449 This material was able to accumulate very little deformation during the loading cycles
450 and also had a very high recovery during unloading. Finally, the rutting resistance of
451 LMEB was intermediate between the other bitumens, since it had lower J_{nr} / higher R
452 compared to EB, but higher J_{nr} / lower R with respect to EPMB.

453 To summarize, it was possible to draw up a ranking with respect to the behaviour
454 against permanent deformations, where the material that performed best was certainly
455 EPMB, followed by LMEB and finally EB. As in the case of the rheological behaviour,
456 this result was probably due to the higher polymer content in EPMB compared to LMEB,
457 but also to the different blending process. In fact, the slight difference in the modifier
458 dosage (4.0% for EPMB, 3.3% for LMEB) did not reflected on a similarly slight
459 difference in the mechanical behaviour observed during MSCR test, as the measured J_{nr}
460 and R for LMEB were very close to the average of the values measured for EB and EPMB,
461 at all the investigated temperatures and creep stresses.

462 ***Damage behaviour of the recovered bitumens***

463 The damage behaviour of the bitumens recovered from BE was investigated by LAS tests.
464 The frequency sweep tests preliminarily carried out on the undamaged bitumen samples
465 showed complex moduli comparable to those measured in the LVE analysis. In particular,
466 $|G^*|$ values of about 15 MPa were determined for all the bitumens at $T = 15\text{ }^\circ\text{C}$ and
467 $f = 5\text{ Hz}$ (confirming the good choice of the test temperature).

468 Figure 10 shows the shear stress τ measured during LAS test for the different
469 recovered bitumens as a function of the shear strain γ . From the graph it can be noticed
470 that EB had the highest peak of τ but, after sample failure (peak of the τ vs γ curves), the
471 stress necessary to achieve the target strain quickly decreased and tended to 0 Pa. The
472 behaviour of the recovered bitumens from polymer-modified BE was very different.
473 EPMB had slightly lower peaks of τ with respect to EB, but the reduction of the stress
474 after failure was very slow. Even at the end of the test, for $\gamma = 30\%$, rather high shear
475 stresses (approximately 50% of the peak values) were necessary to apply the imposed
476 strain. This indicated that, even after failure, the material had a high reserve of energy to
477 withstand the test stresses. LMEB showed a higher sample-to-sample variability in the

478 output of the test, but the general trend was easily identified as peculiarly different from
479 the others. In fact, the peak value of τ for LMEB was the lowest (less than a half of the
480 average peak value of EB) but, as EPMB, it showed a slow decrease of stress after the
481 peak, denoting a good ability to tolerate further cycles after the sample failure.

482 Figure 11 shows the damage curves, which depict the reduction of the normalized
483 complex modulus C (ratio of $|G^*|$ at the i -th cycle and $|G^*|$ at the 1st cycle) when
484 increasing the damage intensity D . The C vs D curves determined through VECD model
485 confirmed the indications obtained from τ vs γ curves. For low D values, EB slowly
486 decreased its stiffness when increasing damage, as a consequence of the higher strength
487 (higher peak of τ). However, the curve of EB did not tend to flatten thus, when continuing
488 the sample stressing and damage increasing, C rapidly went to zero. Differently, LMEB
489 and EPMB curves quickly decreased for low D values, but the slope gradually decreased
490 and for high damages the bitumens from polymer-modified BE demonstrated to still have
491 a certain stiffness. Between the two, EPMB seemed to have the best resistance to damage,
492 as the C values were always higher than for LMEB under the same damage and also the
493 slope of the curve was lower, indicating that the higher performance was also enhanced
494 for high damage values.

495 Figure 12 shows the fatigue parameters calculated from LAS test. In particular,
496 the parameters A and α are considered good indicators of the behaviour of a binder under
497 repeated stresses: according to Hintz et al., (2011), the bitumen with a good resistance to
498 fatigue is characterized by a high value of A (which indicates the fatigue life of the binder
499 at 1% strain amplitude) and a low value of α (which is a measure of strain susceptibility
500 of the binders, i.e., the rate of reduction in fatigue life with increase in strain). From the
501 graph on the left of Figure 12 it can be observed that α was higher for LMEB and EPMB
502 than for EB. However, like for the β value in 2S2P1D model, this result was probably

503 related to the higher elasticity of the material and did not represent a negative issue for
504 the fatigue behaviour. Indeed, a higher value of α in the case of polymer-modified
505 bitumens was noted by Saboo et al., (2018) in similar temperature conditions. Differently,
506 the value of A noticeably increased when moving from EB to LMEB, to EPMB, indicating
507 a significantly higher performance against fatigue. This was confirmed by the graph on
508 the right of Figure 12, which shows the values of N_f calculated through Equation 1 for
509 strains γ_{max} of 2.5% and 5.0%. The simulation suggested that the binders from polymer-
510 modified BE could withstand a noticeably higher number of cycles before failure for both
511 the strain levels. Respectively, N_f for LMEB and EPMB were approximately 2 and 7 times
512 that of EB at $\gamma_{max} = 2.5\%$, on average. At $\gamma_{max} = 5\%$, the average N_f for LMEB and EPMB
513 were approximately 1.5 and 4 times that of EB. The fatigue life at $\gamma_{max} = 2.5\%$ has to be
514 considered more significant for these binders, since CMB are used in base or subbase
515 layers and are typically subjected to low strain amplitudes. Since the bitumen stiffness
516 $|G^*|$ at the beginning of the test was comparable, this result was not related to how the
517 sample was tested and, thus, to how the binder will operate in its service life (control-
518 stress or control-strain), but highlighted how the presence of elastic polymers in the binder
519 allowed achieving a greater resistance to fatigue. The better performance of EMPB with
520 respect to LMEB indicated that this effect was enhanced in the case of higher polymer
521 content and pre-blending processing.

522 **Conclusions**

523 The present study aimed at comparing the binders recovered from different bitumen
524 emulsions for cold mixtures: a reference over-stabilized BE and two polymer-modified
525 emulsions, including SBR-latex (post-blending) and SBS (pre-blending), analogous
526 according to Italian national standards. The investigation included the analysis of the
527 rheological behaviour, the LVE modelling and the evaluation of the resistance to

528 permanent deformation and damage.

529 On the basis of the results shown in the main body of the paper, the following
530 conclusions can be drawn:

- 531 • All the recovered bitumens showed a thermorheologically simple behaviour,
532 which could be well simulated through 2S2P1D model.
- 533 • At low frequencies/high temperatures, EPMB had the highest $|G^*|$ and lowest ϕ
534 values, denoting a higher elasticity, while LMEB had intermediate properties
535 between EPMB and EB. At high frequencies/intermediate-low temperatures, the
536 $|G^*|$ values were comparable between the different bitumens, while ϕ was lower
537 for EPMB than for EB and LMEB, which presented similar viscous features.
- 538 • In general, the complex modulus of LMEB was closer to that of EB than to that
539 of EPMB, in spite of its polymer content which was slightly lower (3.3% vs 4.0%).
540 This was probably a consequence of the different modification process, which
541 entails a different morphology of the bitumen chemical structure.
- 542 • The parameters k , h and τ_0 of the 2S2P1D model were comparable between the
543 different recovered bitumens, while δ and β increased when moving from EB, to
544 LMEB, to EPMB, reflecting an increase in the elastic features over the viscous
545 ones. Likewise, also the GR parameter resulted higher for the recovered bitumens
546 from polymer-modified emulsions than for EB, due to the predominance of the
547 elasticity over viscosity.
- 548 • The MSCR tests showed that EPMB had the best performance against permanent
549 deformation, followed by LMEB and finally EB. As in the case of the rheological
550 behaviour, this result was probably due to the higher polymer content in EPMB
551 compared to LMEB, but also to the different blending process, since the measured

552 J_{nr} and R for LMEB were very close to the average of the values measured for EB
553 and EPMB, at all the investigated temperatures and creep stresses.

554 • The LAS test results showed different properties of the recovered bitumens
555 against damage. From the τ vs γ curves it was observed that EPMB and LMEB
556 had a reserve of energy against the imposed strain after the sample failure, while
557 the curves of EB quickly decreased and tended to 0 Pa after overtaking the peak.
558 The C vs D curves showed that EB had a higher performance for low damage
559 levels whereas, when increasing damage, EPMB and LMEB had a better
560 behaviour. The analysis of the fatigue parameters indicated that LMEB and
561 especially EPMB, thanks to their elasticity, had a higher resistance against
562 repeated cycles than EB.

563 The previously described findings confirm that the polymer-modified bitumen
564 emulsions for CMB allow achieving a better performance than unmodified emulsions in
565 terms of rheological behaviour and resistance to permanent deformation and damage. In
566 addition, the results show that, even if the two commercial products are equivalent
567 according to Italian national standards, the bitumen emulsion modified with SBS through
568 pre-blending process has better properties against rutting and fatigue. Future
569 developments of the research will focus on evaluating the characteristics of these binders
570 after ageing and validating the performance at mastic and mixture level.

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