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Using fine aggregate matrix mortars to predict the curing behaviour of cement bitumen treated materials produced with different cements

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ABSTRACT

Cold recycling is a sustainable pavement rehabilitation technology. Among the different techniques, cement-bitumen treated materials (CBTM) take advantage of the presence of the two co-binders to achieve satisfying performance. A multiscale study addresses the effect of different cementitious binders on the mechanical behaviour of CBTM mixtures and fine aggregate matrix mortars produced with bitumen emulsion. The evolution of stiffness and strength during curing is measured and compared. Results showed that the cement type has a critical effect on the mechanical behaviour and, under fixed curing conditions, changing the strength is equivalent to changing the dosage. Finally, fine aggregate matrix mortars offer an excellent prediction of mixture mechanical properties.

Keywords

Cold recycling, bitumen emulsion, mortar, curing, mechanical characterisation, Sulfoaluminous cement, cement, Fine Aggregate Matrix, ITS, ITSM

1 INTRODUCTION

Environmental sustainability and cost-effectiveness are promoting the worldwide diffusion of low-energy and low-emission technologies for pavement construction and rehabilitation [1]. In this context, cold recycling of bituminous pavements is one of the most effective and low environmental impact techniques [2].

Cold recycled mixtures are produced at ambient temperature employing a high amount of reclaimed asphalt (RA), generally between 50% and 100% of the total aggregate blend. The limited use of virgin aggregate and bitumen heating reduces energy consumption and pollutants emissions [3], [4] while the reuse of RA preserves natural resources and limits the disposal costs [5], [6]. Bituminous (i.e. foamed bitumen or bitumen emulsion) and cementitious binders (e.g. ordinary Portland cement, composite cement, fly ash or ground granulated blast) are generally employed together to achieve structural and durability properties of the mixture [2]. Water is added to enhance mixing, laying and compaction operations [6], [7].

The performance of cold recycled mixtures must be evaluated taking into account their curing behaviour [6], [8]–[10]. In fact, the microstructure of the mixtures evolves and their mechanical properties improve because of drying, emulsion breaking and setting, and cement hydration [11], [12]. Curing is influenced by the aggregate blend nature [13], the dosage of binders and their interaction. Cement hydration affects the pH and reduces water content, promoting emulsion setting [14], [15]. Likewise, emulsion affects the hydration rate and the formation of well-structured cementitious bonds [12], [16], [17]. Environmental conditions affect the curing process as well. High temperature and low relative humidity (RH) promote water evaporation and thus increase the curing rate [18], [19]. High temperature also increases the rate of cement hydration but may hinder the long-term strength of the cementitious matrix. Besides, a moist environment [14] enhances cement hydration and hinders emulsion breaking.

2

54   Among cold recycled mixtures, cement-bitumen treated materials (CBTM) mixtures have

55 properties which are half-way between those of asphalt concrete mixtures and cement-treated

56 mixtures. The bituminous binder gives to the CBTM mixture its typical frequency and

57 temperature dependent behaviour and fatigue susceptibility [8], [20]–[24]. The cementitious

58 binder improves strength, stiffness and permanent deformation resistance, but may lead to a

59 prone-cracking behaviour of the material [6], [14], [25]–[27].

60   Rapid hardening cementitious binders, like calcium aluminate and calcium

61 sulfoaluminate binders, can accelerate the strength development improving the mechanical

62 properties of CBTM mixtures in the early stage of curing [17], [28], [29]. They bound a higher

63 amount of water compared to ordinary Portland-based cement, which may affect the emulsion

64 setting. Besides, compared to Portland cement, their production requires less energy and

65 reduces CO_2 emissions [30], [31], leading to sustainability benefits. The use of other

66 supplementary blended cementitious fillers results in the increased early strength, due to an

67 enhanced hydration reaction. Moreover, these kinds of filler can improve the mechanical

68 response and water sensitivity [32]–[34].

69 1.1   **The fine aggregate matrix concept**

70 The mechanical behavior of asphalt concrete mixtures has been studied at different scales of

71 observation [35]. At the mixture scale, asphalt concrete can be considered a particulate

72 composite where coarse aggregate particles (inclusions) are dispersed in the fine aggregate

73 matrix (FAM) phase [36]. Several studies have shown that FAM properties affect the

74 viscoelastic, fatigue and fracture behavior of asphalt concrete mixtures [35], [37]–[44] as well

75 as their moisture damage resistance [45]. It was also shown that healing, oxidation and ageing

76 occur in the FAM phase [39], [46], [47]. Besides, FAM have been used to predict the overall

77 behaviour of the mixture using multiscale computational models [41], [43], [48]–[50].

3

78   The FAM material is a mortar composed of fine aggregate, filler, bitumen and voids [51].

79   Its grading distribution derives from the fine part of the mixture grading [38], [39], with upper

80   sieve size comprised between 1.18 and 2.36 mm [38], [39], [44], [48]. The bitumen content of

81   FAM mortars can be obtained from the total binder of the mixture, subtracting the fraction

82   absorbed by the coarse aggregate [39] or the fraction included in the mastic film coating the

83   coarse aggregate [35]. The air voids content of the FAM was found to be between 2.5 and 3.5%

84   [38], or about 50-70% of the total air voids of the mixture [35].

85   Recent studies applied the FAM concept also to study CBTM mixtures produced with

86   bitumen emulsion and cement [51], [52]. The FAM mortars had a maximum aggregate size of

87   2 mm and were produced using all the bitumen emulsion and cement content of the mixture

88   (the residual bitumen to cement ratio was 0.8 and 1.3 or 2.0). The authors found that FAM

89   mortars including about 75 - 80% of mixture water and 50% or 17% of mixture air voids could

90   predict the stiffness and strength of mixtures, throughout the curing process [51], [52] and their

91   LVE behaviour [52].

92   Cold bituminous mortars, were also proposed as a model system for characterizing

93   bitumen emulsion [53], [54]. This approach was inspired by the common practice for

94   characterising the strength of cementitious binders (EN 196-1, ASTM C109 / C109M). The

95   authors selected the same upper sieve size, 2 mm, and a similar grading distribution [55], [56].

96   Since the focus of those studies was the bitumen emulsion, the mortars had a bitumen-

97   dominated behaviour with bitumen to cement ratio ranging between 3 and 7. Cold bituminous

98   mortars were also employed for evaluating the effect of mineral additions on the failure

99   properties of CBTM using a bitumen to cement ratio of 1 [55], [57].

100   The FAM concept was also used to investigate the interaction mechanism between

101   cement and bitumen, assisting the computational modelling of CBTM mixtures [58].

4

1.2 **Objective and methodology**

In this research we investigated the physical and mechanical properties of CBTM mixtures produced with bitumen emulsion and cement, throughout the curing process. The objectives were to compare the effect of different cement types and to evaluate the predictive potential of FAM mortars. We also evaluated the impact of different curing conditions.

Figure 1 describes the research methodology and the paper structure. In detail, we considered eight CBTM mixture compositions obtained with four cement types and two dosages. First, we studied the effect of water on their volumetric and mechanical properties. Then, for each mixture, we investigated nine potential FAM mortar compositions, focusing on the effect of water and air voids on their early-age strength. These two tasks allowed to define eight pairs of materials (CBTM mixture and corresponding FAM mortar) that we tested to evaluate the evolution of water loss by evaporation, stiffness and strength. We considered one curing temperature (25 °C) and two curing conditions (sealed and unsealed specimens). Finally, we assessed the predictive potential of the FAM mortars comparing their mechanical properties to those of the corresponding CBTM mixtures.

5

MIXTURE COMPOSITION - Sections 3.1, 4.1

Volumetry & Compactability

- 4 cement types, 1 dosage
  - Fresh state

Mechanical properties @25°C

- 4 cement types, 2 dosages
- 3 unsealed curing days @40 °C
- 28 unsealed curing days @ 25 °C + 3 - 7 days water immersion @40 °C

FAM MORTAR COMPOSITION - Sections 3.2, 4.2

Effect of water and air voids content on the strength @25 °C

- 4 cement types, 2 dosages
- 1 unsealed curing day @ 25 °C

CURING BEHAVIOUR - Sections 3.3, 4.3

MIXTURE	FAM MORTAR
Effect of cement type	Effect of cement type
• 4 cement types, 2 dosages	• 4 cement types, 2 dosages
• Water loss, stiffness and strength @ 25 °C	• Water loss, stiffness and strength @ 25 °C
• 1, 3, 7, 28, 90 unsealed curing days @25 °C	• 6 h, 1, 3, 7, 28 unsealed curing days @25 °C
Effect of curing RH	Effect of curing RH
• 2 cement type, 2 dosages	• 2 cement type, 2 dosages
• Stiffness and strength @ 25 °C	• Stiffness and strength @ 25 °C
• 1, 3, 7, 28, 90 unsealed & sealed curing days @25 °C	• 6 h, 1, 3, 7, 28 unsealed & sealed curing days @25 °C

MIXTURE and FAM MORTAR COMPARISON - Section 4.4

Prediction of mixtures properties using FAM mortars

- 4 cemen types, 2 dosages
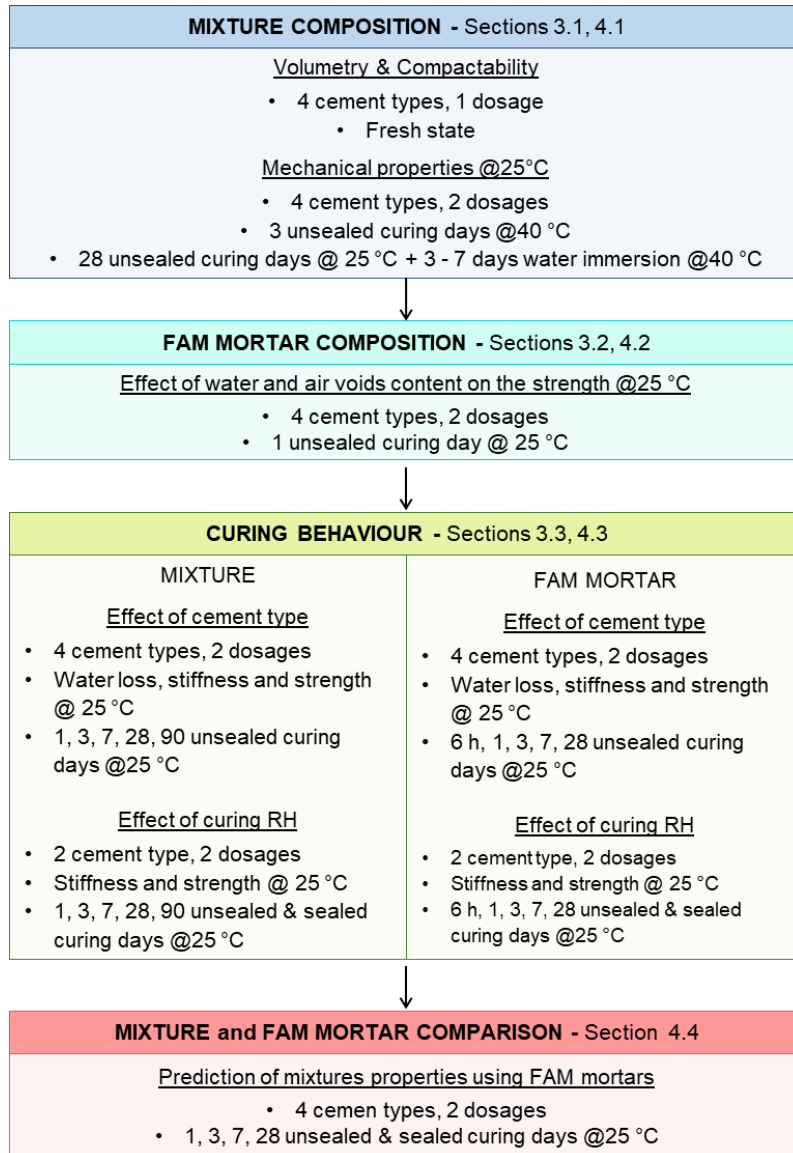- 1, 3, 7, 28 unsealed & sealed curing days @25 °C

Figure 1. Overview of the research methodology and paper structure

2 MATERIALS AND METHODS

2.1   Materials

The granular blend of mixtures and FAM mortars were composed of RA aggregate, virgin fine

aggregate and natural filler. The RA aggregate, supplied by Società Cooperativa Braccianti

Riminese, from the San Leo mixing plant (Rimini – Italy), had a nominal maximum dimension

6

125 of 16 mm (RA 0/16). It was further sieved in the laboratory to produce a separate fraction with

126 an upper sieve size of 2 mm (RA 0/2). The fine aggregate was a crushed limestone sand with a

127 nominal maximum dimension of 2 mm and the filler was a finely ground limestone powder.

128 Figure 2 and Table 1 report the grading distribution and the main physical properties of the

129 aggregates, respectively.

130 The aggregate blend of the mixtures consisted of 80% RA 0/16, 17% of fine aggregate

131 and 3% of filler (by dry mass). The resulting grading curve was close to the maximum density

132 curve with a maximum nominal size of 16 mm (Figure 2). The aggregate blend of the FAM

133 mortars was obtained by removing the volume of coarse aggregate (retained on the 2 mm sieve)

134 and consisted of 61% RA 0/2, 32% fine aggregate and 7% filler.

135 The total water content of mixtures and FAM mortars included emulsion water and

136 additional water. The latter was necessary to improve the workability and compactability of

137 mixtures. A fraction of the additional water was absorbed by the aggregates, while the

138 remaining fraction, along with the emulsion water, was identified as intergranular water. The

139 procedure for selecting the total water content of mixtures and FAM mortars will be described

140 in Sections 3.1 and 3.2, respectively.

141 The bituminous binder was a cationic slow-setting bitumen emulsion, supplied by Valli

142 Zabban S.p.a. (Bologna - Italy), with a residual bitumen content of 60%. The emulsion was

143 specifically designed for cold recycling applications and coded C60B10 (EN 13808). The

144 emulsion base bitumen was 70/100.

145 Four cementitious binders, supplied by Italcementi S.p.a. (Bergamo - Italy), were

146 selected to provide an extensive overview of the effect of cement on the mechanical properties

147 of CBTM mixtures (Table 2):

148 • C1 - Portland limestone cement type II/B-LL with strength class 32.5R (EN 197-1);

- C2 - Sulfoaluminous cement (not standardised within the European standards framework);

- C3 - Portland-slag cement type II/B-S with strength class 52.5N (EN 197-1);

- C4 - Hydraulic binder non-structural applications type HB3.0 (EN 15368).

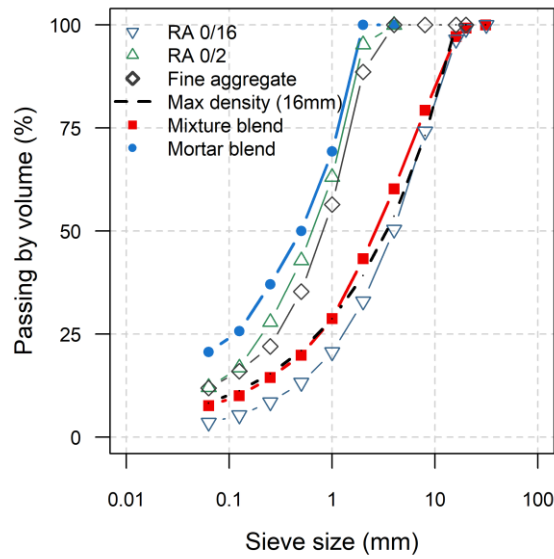C1 was considered the reference cementitious binder, because it is commonly used in cold recycling in Italy [59], [60]. C2 was characterised by rapid setting and hardening, and very high 28-days strength; moreover, it had a lower pH with respect to conventional Portland-based cements. C3 was characterised by an ordinary early strength and a high 28-days strength. C4, is typically used in Italy to produce mortars for non-structural masonry or rendering and plastering; it was characterised by low values of both early and long-term strength, and high water retention in the fresh state. The selected cementitious binders allowed us to investigate a wide range of strength. Also, the non-Portland cement, i.e. C2, was chosen for verifying if its different nature and the lower pH influenced the microstructure of the CBTM, leading to a different mechanical response.

The procedures for selecting the emulsion and cement dosages for the mixtures and FAM mortars will be described in Sections 3.1 and 3.2.

Table 1. Main physical properties of the aggregate

Material	Particle density (EN 1097-6) kg/m^3	Absorption (EN 1097-6) %	Rigden voids (EN 1097-4) %	Bitumen content* (EN 12697-1) %
RA 0/16	2482	1.14	-	4.9
RA 0/2	2424	1.32	-	8.3
Fine aggregate	2732	1.50	-	-
Filler	2650	-	23.8	-

* by dry aggregate mass

Figure 2. Grading distribution of granular materials, CBTM mixtures and FAM mortars (by volume).

Table 2. Main characteristics of cementitious binders employed (provided by the supplier).

Property	Standard	Cementitious binder type			
		C1	C2	C3	C4
Particle density (kg/m^3)	EN 1097-7	3020±10	2900±030	3090±10	2900±10
Blaine surface area (cm^2/g)	EN 196-6	3800±100	5700±200	4090±100	6100±350
Rigden voids (%)	EN 1097-4	33.2	33.5	33.5	32.3
pH value		12-13.5	11-12	12-13.5	12-13.5
Initial setting time (min)	EN 196-3	170	15	180	180
Final setting time (min)	EN 196-3	220	25	245	300
Compressive strength @2 days (MPa)	EN 196-1	19±2	45±2	20±2	3±1
Compressive strength @28 days (MPa)	EN 196-1	39±3	80±3	56±3	10±2

2.2 Specimen preparation and volumetric properties

For both mixtures and FAM mortars, aggregates, water, bitumen emulsion and cement were

mixed at room temperature according to a procedure developed in previous studies [6], [51].

After mixing, specimens were compacted with a gyratory compactor, applying constant

176 pressure of 600 kPa, gyration speed of 30 rpm and angle of inclination of 1.25°. Moulds with

177 diameters of 150 mm and 100 mm were used for mixtures and mortars, respectively. For

178 evaluating mixture composition, specimens were compacted at 180 gyrations. All the other

179 specimens were compacted until reaching a fixed height value, with the aim to control their

180 volumetric properties. Thus obtained specimens were directly tested after the selected curing

181 times.

182 During compaction, the specimen height was recorded at each gyration, which allowed

183 monitoring the voids (V_m) and the voids filled with liquids (*VFL*) [7]:

$$V_m = \frac{v_A + v_W}{v} = \frac{v - (v_S + v_B)}{v} \tag{1}$$

$$VFL = \frac{v_B + v_W}{v_A + v_B + v_W} = \frac{v_B + v_W}{v - v_S} \tag{2}$$

184 where v is the total volume of the specimen, v_A is the volume of the air voids, v_s is the bulk

185 volume of solids (aggregates, filler and unhydrated cementitious binder), v_B is the volume of

186 residual bitumen from the emulsion and v_W is the volume of intergranular water (the volume of

187 absorbed water is comprised in the bulk volume of the aggregate) (Figure 3a). The volumetric

188 analysis refers to CBTM at the fresh state, before emulsion breaking, water evaporation and

189 cement hydration begin. Indeed, during curing part of the water evaporates while a specific

190 amount of water is bonded by the cement, whose hydration products will occupy a greater

191 volume compared to the volume of the unhydrated cement. In addition , a minor amount of

192 water can be trapped in the specimen, constituting not-interconnected voids. [61].

193 According to Equations (1-2), V_m is the volume fraction that is occupied by air and

194 intergranular water, while *VFL* is the fraction of voids occupied by residual bitumen and

195 intergranular water. During compaction, V_m decreases whereas *VFL* increases (Figure 3b), with

196 *VFL* = 100% indicating the theoretical saturation condition of the specimen. Previous studies

have shown that when *VFL* approaches 90%, a loss of material (water, bitumen droplets and fines) occurs from the mould [6], [7], [51]. This indicates that a small amount of entrapped air (voids not interconnected) must always be included in the specimen to avoid altering its composition during the compaction process. Thus, the number of gyrations corresponding to *VFL* = 90% has been assumed as a practical compaction limit, for a given specimen composition (Figure 3b).

To check possible material loss during compaction, in this study we compared the mass of the loose material before compaction with the mass of the compacted specimen. All the specimens compacted with fixed height had a mass loss lower than 0.5%, testifying the goodness of the compaction process.

a)

b)
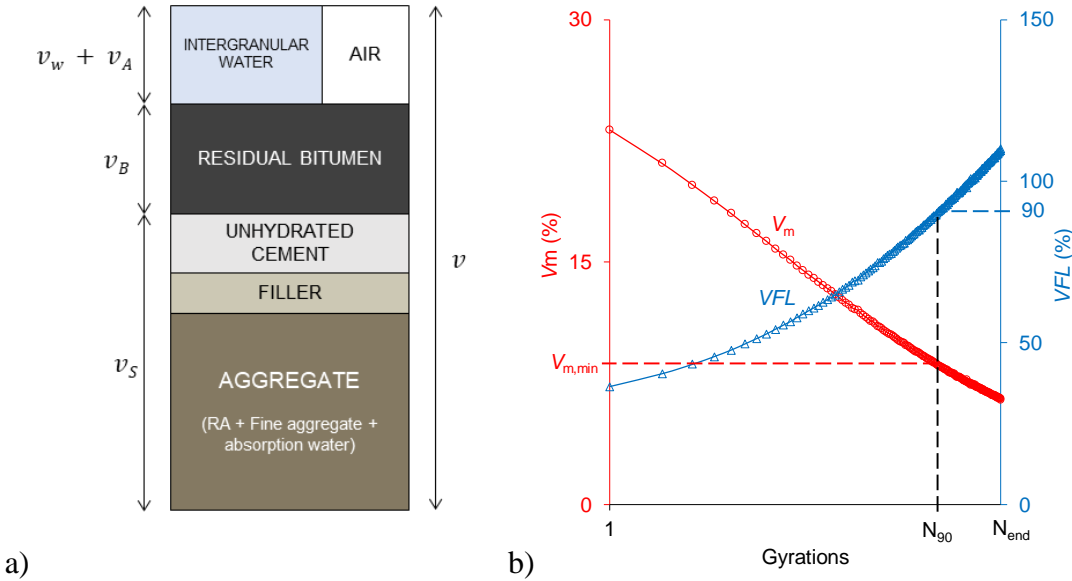
Figure 3. Volumetric study of CBTM: a) specimen volume at the fresh state, b) volumetric properties progress during compaction (Equations 1 and 2)

212   ## 2.3   **Testing methods**

213   Since CBTM are evolutive materials, we selected relatively quick testing methods to measure

214   stiffness and strength. This allowed linking the measured property value to the actual curing

215   time.

216   The indirect tensile stiffness modulus (*ITSM*) was measured using a servo-pneumatic

217   machine. Repeated load pulses with a rise time of 124 ms and a pulse repetition period of 3.0 s

218   were applied (EN 12697-26 Annex C). The peak load was adjusted using a closed-loop control

219   system to achieve a target peak horizontal deformation of 2 microns. For each specimen, the

220   test was repeated along two diameters and the average *ITSM* value was calculated:

$$ITSM = \frac{F \cdot (v + 0.27)}{z \cdot h} \tag{3}$$

221   where F is the peak load of the applied repeated pulse, z is the amplitude of the horizontal

222   deformation, h is the mean thickness of the specimen and v is the Poisson's ratio (assumed as

223   0.35).

224   The indirect tensile strength (*ITS*) was measured using a servo-hydraulic testing machine.

225   A constant rate of deformation of (50 ± 2) mm/min was applied along the two generatrixes of

226   the cylindrical specimen until failure (EN 12697-23):

$$ITS = \frac{2 \cdot P}{\pi \cdot D \cdot h} \tag{4}$$

227   where P is the maximum load, D is the specimen diameter and h is its mean thickness.

228   Both *ITSM* and *ITS* were measured at 25 °C. The specimens cured at 40 °C, tested for

229   defining the composition of the mixtures, were conditioned 4 hours at the testing temperature.

230   The specimens cured at 25 °C for the curing behaviour characterisations, were not subjected to

231   further temperature conditioning.

The water resistance of mixtures was evaluated using the indirect tensile strength ratio (*ITSR*), quantifying the strength reduction due to the specimen soaking in water. There is no standard for measuring the water resistance of CBTM. The procedure required by the Italian construction specifications [62], [63] for hot mix asphalt (HMA) was followed, considering 3 days of immersion in water at 40 °C of specimens. An additional immersion time of 7 days was considered as well. Before the water resistance testing, specimens were cured 28 days at 25 °C.

Before mechanical testing, the water loss by evaporation (*DW*) of each specimen was measured by weighing the specimens:

$$DW = \frac{M_0 - M_i}{M_W} \cdot 100 \tag{5}$$

where M_0 is the specimen mass right after compaction, M_i is the specimen mass after i curing days and M_W is the total mass of water in the specimen (derived from its gravimetric composition).

The evolution of material properties was modelled using a non-linear asymptotic function, obtained as a modified version of the Michaelis-Menten model [51]:

$$y(t) = y_i + (y_a - y_i)\frac{t - t_i}{(h_y - t_i) + (t - t_i)} \tag{6}$$

where $y(t)$ is the property under investigation (*DW*, *ITSM* or *ITS*), y_i is its value at the time t_i and y_a is its long-term asymptotic value. The time t_i represents the early-stage of curing, and here we assumed $t_i = 1$ day. The parameter h_y represents the time to reach the value $(y_a - y_i)/2$ by $y(t)$. We estimated the model parameters y_i, y_a and h_y using non-linear least-squares minimization and used the residuals standard error to evaluate the goodness of fit.

3 EXPERIMENTAL PROGRAM

3.1   **Mixture composition**

253 The emulsion and cement dosages (by dry aggregate mass) were fixed at the beginning of the

254 research, based on the Italian practice for cold recycled subbase and base courses [62].

255 Specifically, we selected a 3.3% emulsion dosage (corresponding to 2.0% of residual bitumen),

256 and two cement dosages, 1.5% and 2.5%. The eight mixtures were identified using the cement

257 type (C1, C2, C3 and C4) and the residual bitumen to cement mass ratio (B/C), 1.3 and 0.8.

258   The total water content was established by analysing the volumetric properties and the

259 compactability of the mixtures [7], [51], [64]. Trial mixtures were produced at four total water

260 contents: 3.2%, 4.2%, 5.2% and 6.2%. The mixtures produced with binders C1 and C2 at the

261 lowest water content (3.2%) were discarded because the material was too dry and not

262 homogeneous. The mixtures produced with binders C3 and C4 at the highest water content

263 (6.2%) were discarded because the material was too wet, and the water drained out. From each

264 batch, three specimens were compacted at180 gyrations. This allowed to measure V_m and *VFL*

265 as a function of the number of gyrations. By analysing the compaction curves, first we chose a

266 common V_m value for all mixtures and then we selected the optimal water content for each

267 mixture.

268   The final composition of the mixture was tested to verify the mix design requirements set

269 by Italian construction specifications [62], [63]. First, *ITSM* and *ITS* were evaluated on

270 specimens subjected to an accelerated curing regime of 3 days at 40 °C in unsealed condition.

271 Then, the *ITSR* was evaluated on two additional series of specimens that were first cured

272 28 days at 25 °C and then submerged in water at 40°C for 3 and 7 days.

3.2 **FAM mortar composition**

274 We obtained the composition of the FAM mortar by considering the mixture as a three-phase

275 composite (Figure 4), consisting of:

276 • FAM (Phase I):

277 • Coarse aggregate (Phase II);

278 • Voids (Phase III).

279 The FAM was considered as the continuous binding phase of the composite. It had a

280 maximum aggregate dimension of 2 mm and included all the bituminous and cementitious

281 binders of the mixture. Hence, based on the selected mixture composition, the emulsion dosage

282 was 7.8% (corresponding to 4.7% of residual bitumen), and the cement dosages were 3.5% and

283 5.9%. FAM mortars had the same B/C ratios as the corresponding mixtures: 1.3 and 0.8. The

284 voids of the mixture (i.e. air and intergranular water) were dived in two parts: those that were

285 part of the FAM and those that constituted the Phase III, that we call the external voids (Figure

286 4).

287
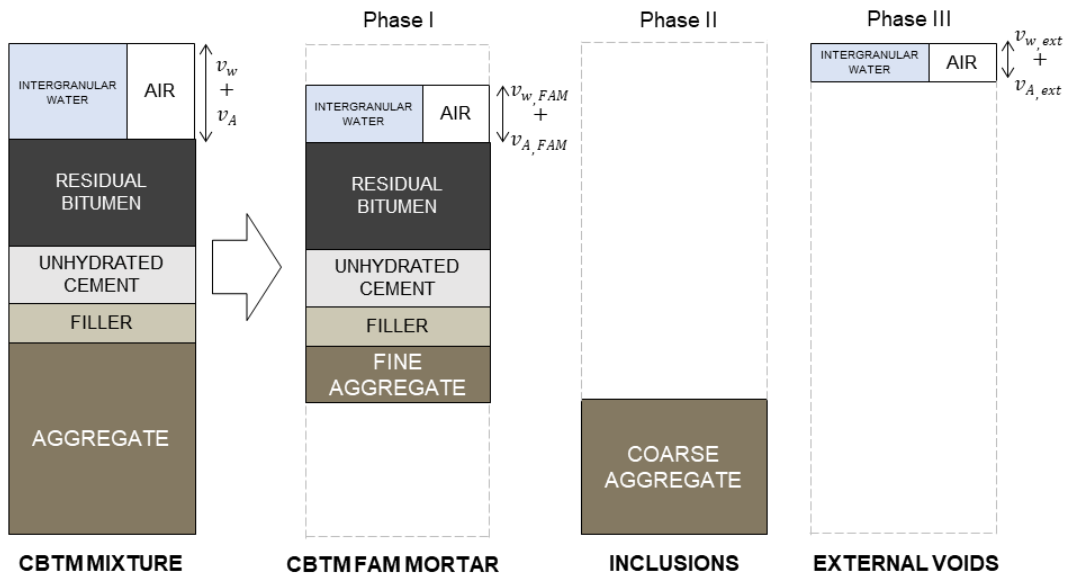
288 **Figure 4. CBTM mixture and FAM mortar volumetric composition model (fresh state)**

In order to investigate the effect of voids content on the FAM properties, for each mixture we produced nine FAM mortars characterised by different water and air voids fractions (Table 3). The mortars characterised by $v_{w,FAM} / v_{w,mixture} = 1.00$ and $v_{A,FAM} / v_{A,mixture} = 1.00$ were prepared with all the water and all the air voids of the mixture. According to the mixture model depicted in Figure 4, in this case Phase III would not be present (i.e. the mixture would be a two-phase composite). On the other hand, mortars characterised by $v_{A,FAM} / v_{A,mixture} = 0$ were compacted trying to remove all the air voids and reach saturation. n this case all the air of the mixture would be included in Phase III. As explained in Section 0, this latter condition was impossible to obtain in practice, and thus a small amount of entrapped air voids was always be included in the FAM mortar. During production, it was found that FAM mortars produced only with the water of the emulsion resulted too dry and difficult to mix. Thus, additional water was always used. Specifically, for mortars produced with binders C1 and C2, the minimum gravimetric water content was 6.9% (corresponding to $v_{w,FAM} / v_{w,mixture} = 0.77$). For mortars produced with C3 and C4, the minimum gravimetric water content was 5.8% (corresponding to $v_{w,FAM} / v_{w,mixture} = 0.84$).

Table 3. Water and air voids fractions adopted for the trial FAM mortar compositions.

C1 – C2			C3 – C4		
w* (%)	$v_{w,FAM} / v_{w,mixture}$	$v_{A,FAM} / v_{A,mixture}$	w* (%)	$v_{w,FAM} / v_{w,mixture}$	$v_{A,FAM} / v_{A,mixture}$
9.4	1.00	1.00 / 0.50 / 0.00	7.1	1.00	1.00 / 0.50 / 0.00
8.2	0.89	1.00 / 0.50 / 0.00	6.5	0.93	1.00 / 0.50 / 0.00
6.9	0.77	1.00 / 0.50 / 0.00	5.8	0.84	1.00 / 0.50 / 0.00

* dosage by dry aggregate mass

307 On all materials we measured the *ITS* after 1 day of curing at 25 °C and, based on the volumetric

308 and mechanical properties we selected one representative FAM mortar composition for each

309 mixture (Section 4.2).

310

3.3 Characterising the curing behaviour of mixtures and FAM mortars

312 The procedures described in Sections 3.1 and 3.2 allowed us to define eight mixture

313 compositions and the corresponding eight FAM mortar compositions. We tested these materials

314 to evaluate and compare the evolution of *DW*, *ITSM* and *ITS* throughout the curing process.

315 The mixture and FAM mortar specimens were cured in a climatic chamber at (25 ± 2) °C and

316 (70 ± 5)% RH, in unsealed condition. Additional series of specimens, produced only with

317 cementitious binders C1 and C2, were cured inside sealed plastic bags (identified with "S"). In

318 sealed condition, the higher RH due to restricted water evaporation enhances cement hydration

319 but hinders emulsion breaking. Therefore, bituminous bonds were expected to be more

320 developed in unsealed specimens, whereas cementitious bonds were expected to be more

321 developed in sealed specimens [65]. We considered five curing periods: 1, 3, 7, 28 and 90 days

322 for the mixtures and 6 hours, 1, 3, 7 and 28 days for the FAM mortars. For each curing condition

323 and period three replicate specimens were tested.

4 RESULTS AND DISCUSSION

4.1 Mixture composition

326 Figure 5a reports the average compaction curves obtained for mixture specimens with a total

327 water content of 4.2%. Naturally, V_m reduced and *VFL* increased, as compaction proceeded.

328 The specimens produced with binders C3 and C4 showed lower voids with respect to the

329 specimens produced with binders C1 and C2. Figure 5b shows that, also for the other total water

17

330 content values, the voids obtained with binders C3 and C4 were about 2% lower with respect

331 to those obtained with binders C1 and C2 at the same water content. This may be related to two

332 kind of phenomena. First, the cement type could alter the viscosity of the pastes obtained by

333 mixing water, emulsion, filler and cement. That, in turn, may have an influence on the

334 lubrication ability [66] of the mixture. Second, cement may affect the demulsifying behaviour

335 of over-stabilised emulsion [67]. Therefore, the effect of cement on the workability and

336 compactability of the mixtures is extremely important, especially for field compaction, and

337 should be considered in the mix design phase.

338 The increase in water content generally led to a decrease in V_m but, in some cases, also

339 caused loss of material from the mould and thus changed the specimen composition. Specimens

340 whose indicator is below the $VFL = 90\%$ line (Figure 5b), thus characterised by VFL values

341 higher than 90%, showed a mass loss greater than 0.5% at the end of the compaction, confirmed

342 by an evident water ejection. This resulted in a remarkable change in their composition (Section

343 2.2).

344 For assessing the curing behaviour, we selected a target voids value $V_m = 11\%$ for all the

345 mixtures. Such a V_m value is considered possible to reach in the in situ compaction of CBTM

346 mixtures [59], [68]. Figure 5c and Figure 5d show the average values of the compaction energy

347 (number of gyrations) needed to reach $V_m = 11\%$ and the corresponding values of VFL. Based

348 on these results we selected the total water content, $w_{tot} = 4.7\%$ for the mixtures produced with

349 binders C1 and C2 and $w_{tot} = 3.7\%$ for the mixtures produced with binders C3 and C4. With

350 these compositions it was possible to obtain the target voids value with a similar compaction

351 effort (between 100 and 80 gyrations) and with VFL values well below the practical saturation

352 limit.

Figure 6a displays the average values of *ITSM* and *ITS* at 25 °C for the selected mixture compositions measured after 3 days of curing at 40 °C. The dashed line represents the minimum value of strength (*ITS* = 0.4 MPa) and stiffness (*ITSM* = 3000 MPa) required by the Italian technical specifications [62], [63]. The increase in the cement dosage always led to an improvement of the mechanical properties. The mixtures produced with high strength cements (C2 and C3) showed higher values of *ITS* and *ITSM* compared to the reference cement C1, whereas the mixtures produced with C4 showed lower mechanical properties (below the *ITS* acceptance limit).

Figure 6b shows the average values of *ITS* and *ITSR* after 28 days of curing at 25 °C. The dashed line is the minimum value (*ITSR* = 70%) required by the Italian technical guidelines [62], [63]. All the mixtures highlighted an *ITSR* greater than 75% and thus a not-pronounced water sensitivity. Increasing the soaking time from 3 days to 7 days, generally led to an increase of *ITSR*. This suggests that the additional curing time at the high temperature of soaking (i.e. 40 °C) improved the mechanical properties of the mixtures, counterbalancing the effect of the immersion in water.

The comparison between Figure 6a and Figure 6b shows that the *ITS* values obtained after 3 days of curing at 40 °C were always lower of those obtained after 28 days of curing at 25 °C. The reduction of strength in the case of the accelerated curing was a function of the type and dosage of cement, and ranged between 0.3% (with C2, B/C = 1.3) and 44% (with C4, B/C = 1.3).
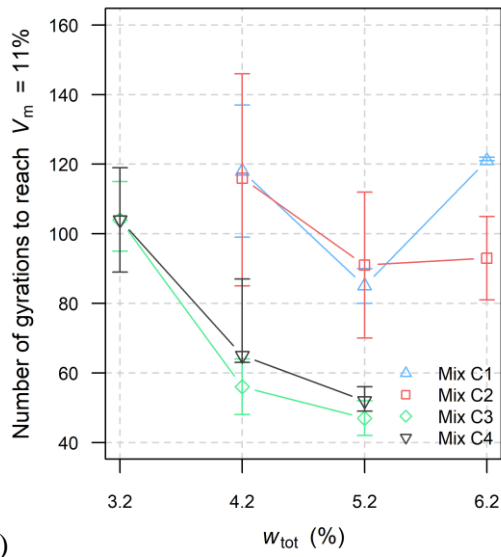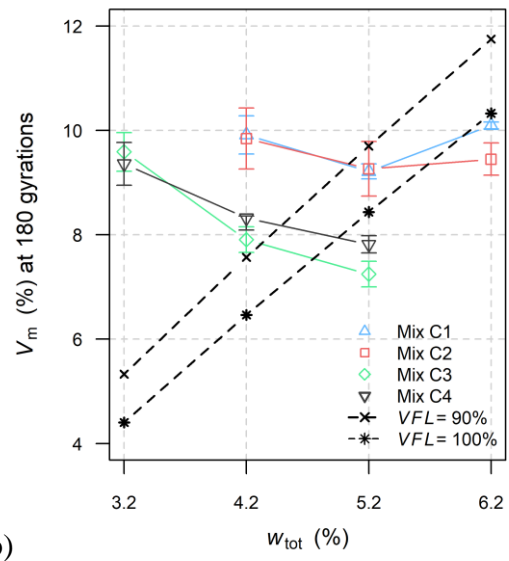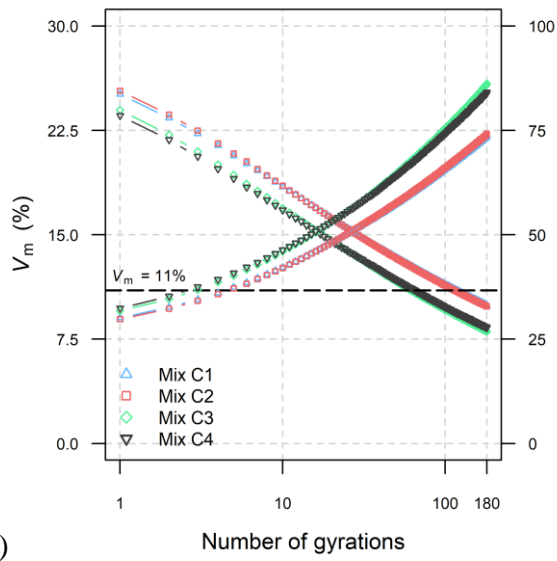
a)

c)

b)

d)
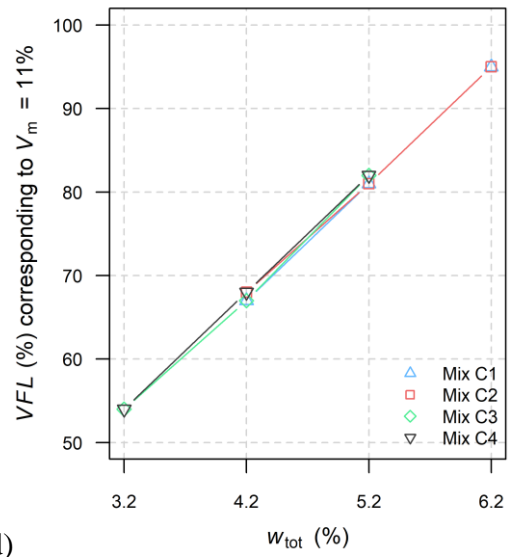
Figure 5 Effect of total water content (w_{tot}) on the volumetric properties of CBTM mixtures: a) compaction curves obtained with w_{tot} = 4.2%, b) V_m at the end of the compaction c) number of gyrations to reach V_m = 11%, d) VFL corresponding to V_m = 11%
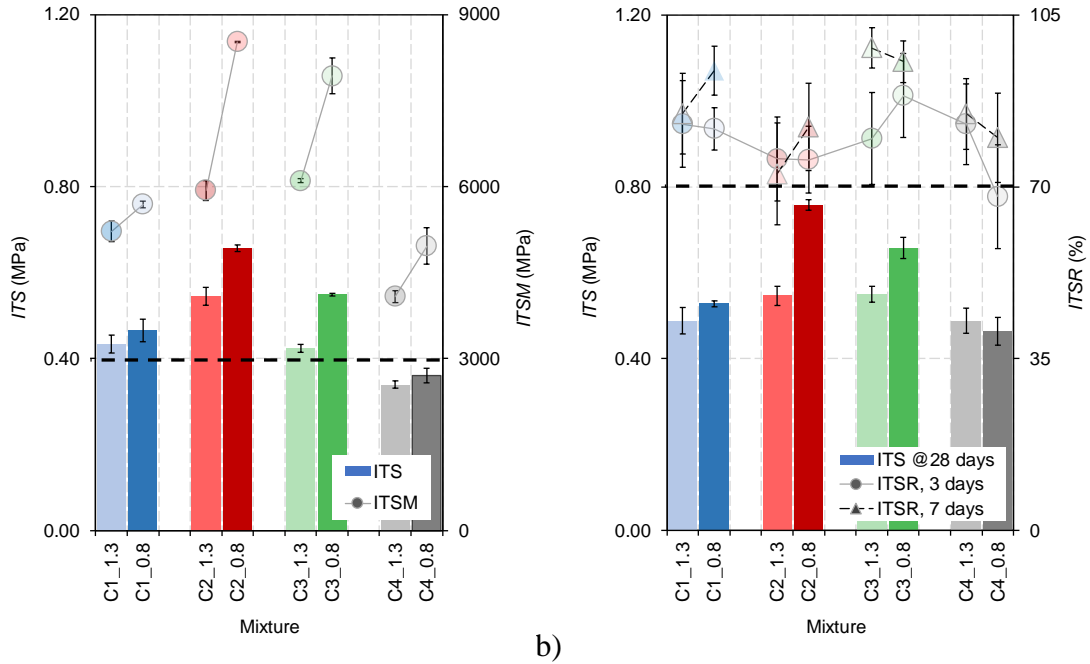
Figure 6 Mechanical properties of CBTM mixtures (25 °C): a) after 3 days of curing at 40 °C, b) water sensitivity

4.2   FAM mortar composition

Figure 7 and Figure 8 show the *ITS* values obtained after 1 day of curing on the nine trial mortars (Table 3) that were produced starting from each mixture. The ratio between the *ITS* of FAM mortar and mixture ($ITS_{FAM} / ITS_{mixture}$) is reported as a function of the air voids fraction contained in the FAM mortar ($v_{A,FAM} / v_{A,mixture}$).

In Figure 7 we plotted the results for the mortars produced using all the water contained in the mixtures ($v_{w,FAM} / v_{w,mixture} = 1.00$). We observe that when the mortars also included all the air voids of the mixture ($v_{A,FAM} / v_{A,mixture} = 1.00$), the *ITS* ratios ranged between 0.47 and 0.71. With this FAM mortar composition, the mixture would be a two-phase composite and thus *ITS* ratios highlight the reinforcing effect of the coarse aggregate. Reducing the air voids fraction contained in the mortar, the ratio $ITS_{FAM} / ITS_{mixture}$ increased. In fact, decreasing $v_{A,FAM} / v_{A,mixture}$ implies that a fraction of air is subtracted from Phase I (FAM) and added to

Phase III (external voids). This leads to an increase in ITS_{FAM}, whereas $ITS_{mixture}$ remains constant because the mixture composition does not change. In Figure 7 we also observe that the FAM mortars produced with binders C3 and C4 had higher ITS ratios with respect to the FAM mortars produced with binders C1 and C2 when the air void volume was reduced. To explain this result we recall that the mixtures produced with the binders C1 and C2 had higher water content and, since V_m of the mixtures was fixed, the volume of air voids was higher. Thus the corresponding FAM also had higher water content with respect to the FAM mortars produced with binders C3 and C4. Since the water volume is not a structural component, the latter FAM mortars will be more resistant in relation to the corresponding mixture. Besides, the reduction of air voids in FAM mortars with C3 and C4 was relatively higher, leading to a lower Vm value and consequently enhanced mechanical response. Finally, we observe that the same considerations are valid for both B/C ratios.

In Figure 8 we plotted the results obtained on the FAM mortars produced with binders C2 and C3, and B/C=1.3. Here we observe that reducing the water fraction contained in the FAM mortars ($v_{w,FAM}$ / $v_{w,mixture}$) always led to an increase in the ITS ratio. As explained above, decreasing $v_{w,FAM}$ / $v_{w,mixture}$ implies that a fraction of water is subtracted from Phase I (FAM) and added to Phase III (external voids). Therefore, ITS_{FAM} increases whereas $ITS_{mixture}$ remains constant, because the mixture composition does not change.

Figure 7 and Figure 8 outline the effect of water and air voids content on the FAM mortars properties after 1 day of curing. Based on these results, we selected a univocal FAM mortar composition to investigate the curing process and predict the mixture behaviour: the grading distribution was obtained considering all the aggregate passing the 2 mm sieve; all the bitumen emulsion and cement was contained in the FAM mortar. As regards the air voids content, we had two limiting options: $v_{A,FAM}$ / $v_{A,mixture}$ = 0.00 and $v_{A,FAM}$ / $v_{A,mixture}$ = 1.00. The first would

22

419 imply that the FAM is a saturated mortar and thus all the mixture air voids, even the smallest

420 ones, are part of the external voids (Phase III). The second would imply that all the mixture air

421 voids, even the largest ones, are part of the FAM. Since both these conditions did not appear

422 realistic, we selected the intermediate configuration, $v_{A,FAM} / v_{A,mixture} = 0.50$. As regards the

423 water content, the option $v_{W,FAM} / v_{W,mixture} = 1.00$ (all the water in the mixture is part of the

424 FAM), was excluded for the same reason given above for air voids. In order to avoid the effect

425 of different water content highlighted in Figure 7, we decided to produce all the FAM mortars

426 with the same gravimetric water dosage of 6.90%. This corresponds to values of

427 $v_{W,FAM} / v_{W,mixture} = 0.77$ for C1 or C2 and $v_{W,FAM} / v_{W,mixture} = 0.97$ for C3 or C4. Such a choice

428 allowed the production of mortars with good workability.

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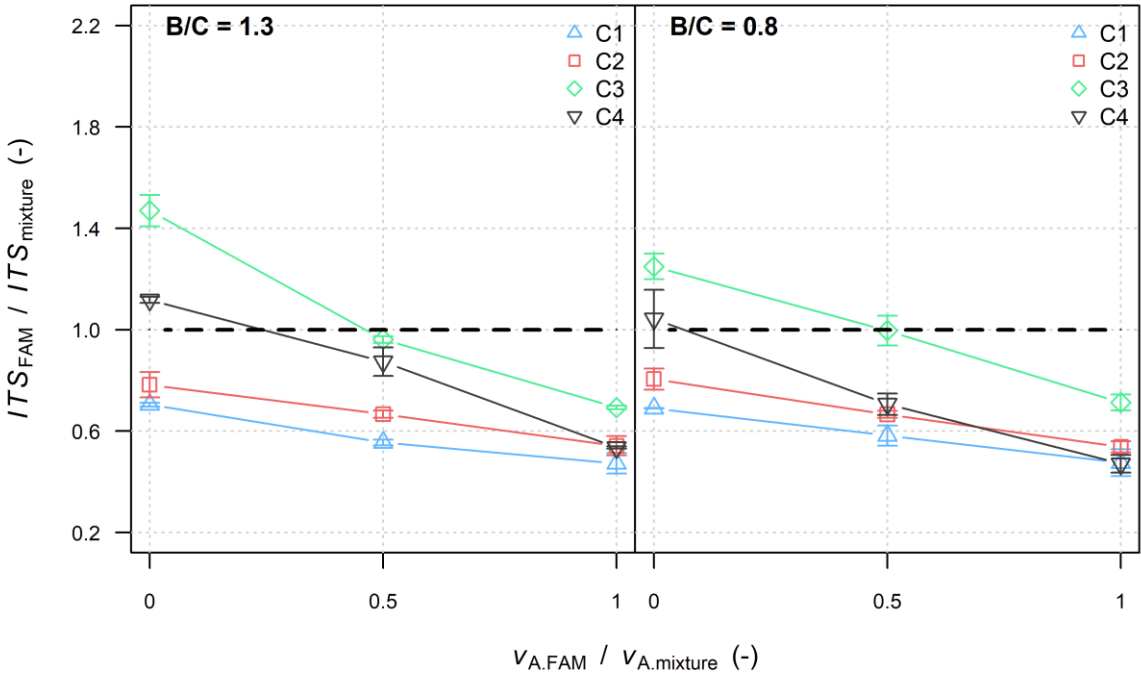
430 **Figure 7. FAM mortar-to-mixture *ITS* ratio as a function of FAM mortar-to-mixture air voids content ratio**
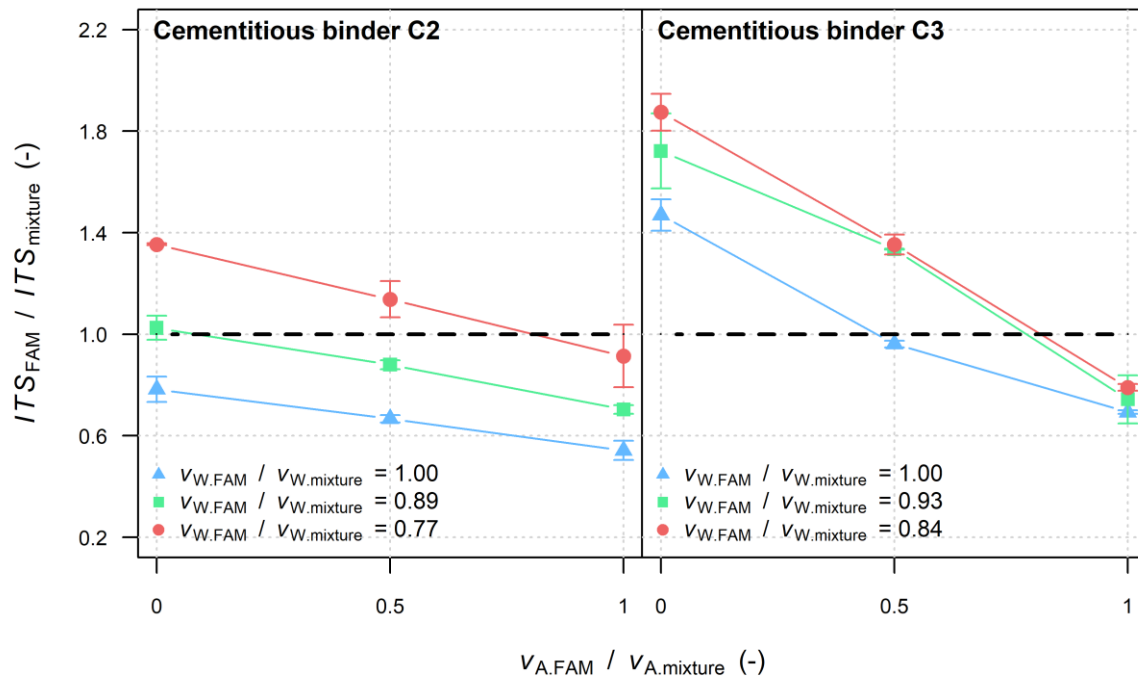431 **obtained with $v_{W,FAM} / v_{W,mixture} = 1.00$**

432

Figure 8. FAM mortar-to-mixture *ITS* ratio as a function of FAM mortar-to-mixture air voids content ratio obtained for CBTM produced with cement C2 and C3, B/C=1.3

In summary, for the study of curing, FAM mortars with binders C1 and C2 were produced with $V_m = 14.1\%$, whereas FAM mortars with binders C3 and C4 were produced with $V_m = 15.8\%$. These values correspond to 67% and 72% of the mixture V_m, and are in agreement with the voids content of FAM of conventional HMA mixtures [35].

4.3 Curing behaviour of CBTM

In this section, we analyse the curing behaviour of the eight mixtures and the corresponding eight FAM mortars. The measured values of *DW*, *ITSM* and *ITS* are plotted as a function of time (logarithmic scale) and the curves representing the fitted curing model (Equation 6) are depicted as well.

Water loss by evaporation

Figure 9 shows the evolution of *DW* for mixtures and FAM mortars produced with binders C1 and C4 in unsealed conditions. The *DW* of specimens cured in sealed conditions was negligible and thus, it was not considered. For all materials, *DW* after the first three curing days was higher than 50%. Afterwards, the evaporation rate decreased and *DW* showed an asymptotic trend. In the long-term, the higher cement dosage (B/C = 0.8) always led to lower *DW*, due the lower availability of evaporable water. In fact, the water physically and chemically bonded by the cement could not evaporate. The amount of evaporated water was related to the cement type [69]. Considering only the mixtures with B/C = 0.8, the *DW* after 90 days was 82.1%, 69.1%, 77.5% and 88.5%, for mixtures produced with binders C1, C2, C3 and C4, respectively. Assuming that all the cementitious binder had the same degree of hydration (very close to 100%), the results highlight the different amount of water bounded by the four types of cement. As expected, the highest amount of water was bounded by C2, the lowest by binder C4.

Figure 10 compares the *DW* of mixtures and FAM mortars, from 1 day to 28 days. Except for the short term *DW* of the materials produced with binder C3, the FAM mortars provided an excellent prediction of the mixture *DW*. This confirmed that the selected mortar composition was an effective model of the FAM.
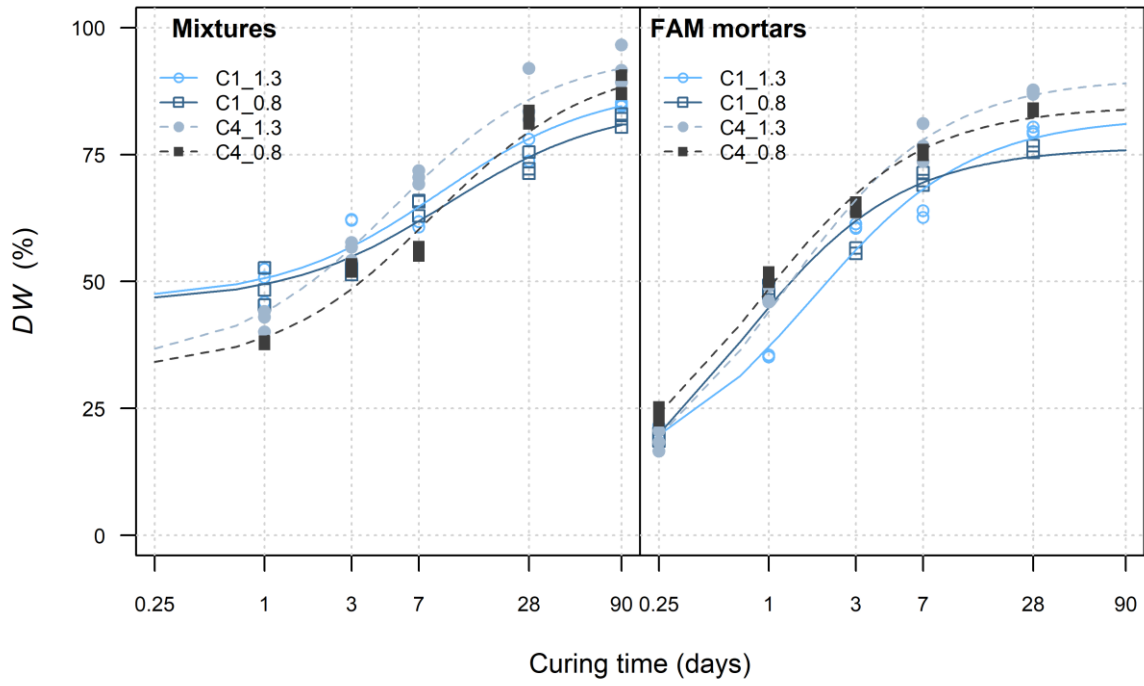
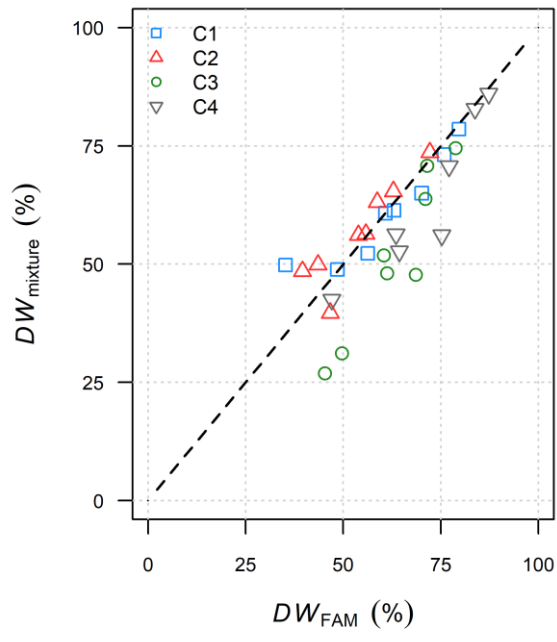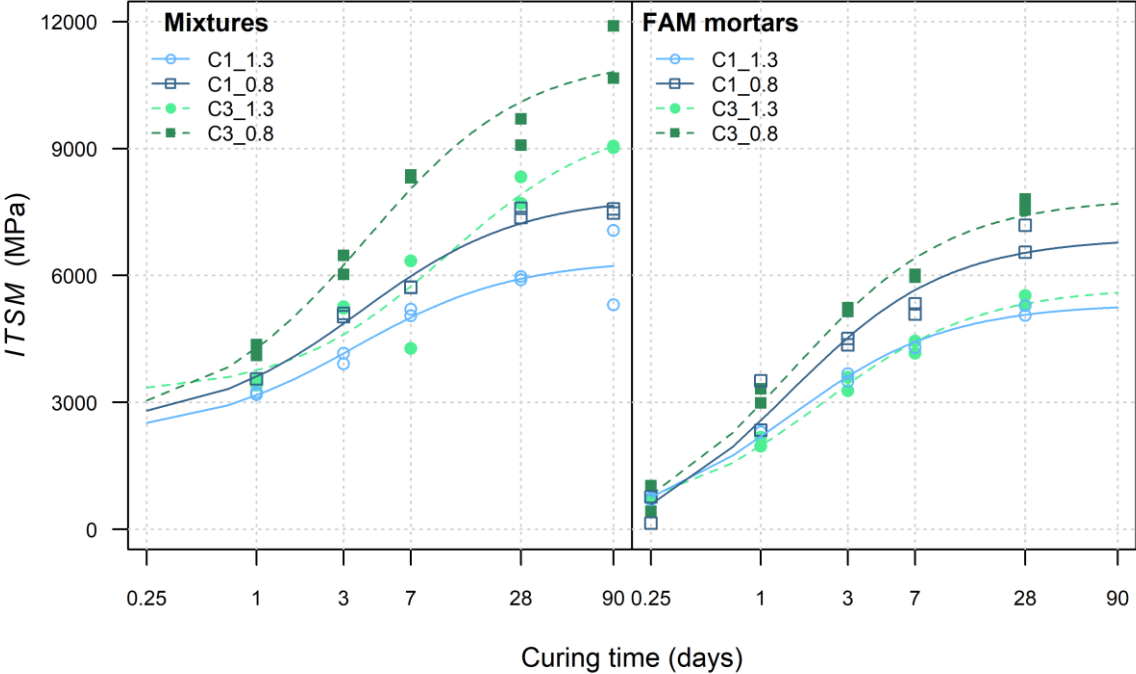**Figure 9. Evolution of *DW* for mixtures and FAM mortars produced with binders C1 and C4**

Figure 10. Comparison of the water loss of mixtures and FAM mortars (from 1 day to 28 days)

Stiffness and strength

469

470 Figure 11 shows the *ITSM* evolution for mixtures and FAM mortars produced with binders C1

471 and C3. Figure 12 summarises the model parameters $ITSM_1$ (*ITSM* after 1 day) and $ITSM_a$

472 (asymptotic value of *ITSM*) for all mixtures. The *ITSM* increased rapidly in the first curing days

473 and then tended towards an asymptotic value. The binder C2 led to the highest short-term

474 stiffness; $ITSM_1$ was 4741 MPa and 7529 MPa, for mixtures with B/C ratios of 1.3 and 0.8,

475 respectively. These values were 50% and 109% higher than those obtained using the reference

476 binder C1. $ITSM_1$ of mixtures produced with binders C1, C3 and C4 was comparable. Using C3

477 and C4, the stiffness was respectively 18% higher and 5% lower, on average, with respect to

478 *ITSM* of mixtures with C1. In the long-term ($ITSM_a$), the highest stiffness was obtained with

479 binders C2 and C3: the increase with respect to binder C1 was between 42% and 56%. Cement

480 C4 did not lead to a significant change in the stiffness: compared to mixtures with C1, $ITSM_a$

481 increased on average of about 8%. For all the cementitious binders, increasing the dosage from

482 1.5% to 2.5% caused an increase in mixtures stiffness at all curing times. The highest increase

483 in $ITSM_a$ was obtained with binder C2 (35.3%), the lowest with binder C3 (14.3%). Finally, we

484 observe that at the same curing, mixtures were generally stiffer than the corresponding FAM

485 mortars (Figure 11).

486   Figure 13 shows the *ITS* evolution for mixtures and FAM mortars produced with binders

487 C1 and C3. Figure 14 summarises the model parameters ITS_1 (*ITS* after 1 day) and ITS_a

488 (asymptotic value of *ITS)* for all mixtures. The binder C2 led to the highest short-term strength;

489 ITS_1 was 0.35 MPa and 0.48 MPa, for mixtures with B/C ratios of 1.3 and 0.8, respectively.

490 These values were 30% and 71% higher than those obtained using binder C1. Differently, using

491 binders C3 and C4 the short term strength was slightly lower (11% and 23%, respectively),

492 compared to C1. However, for all materials, the *ITS* reached 50% of the long-term strength

within the first three days. For all mixtures, the *ITS* increase continued after 28 days, highlighting the long-term contribution of cement hydration and probably also aging of residual bitumen from emulsion. C2 also led to the highest long-term strength; ITS_a was 0.68 MPa and 0.83 MPa for the mixtures with B/C ratios of 1.3 and 0.8, respectively, with an average increase of 35% with respect to the strength of the mixtures produced with the binder C1. Compared to mixtures with C1, the *ITS* increase using C3 was about 22%, whereas with C4, ITS_a was 4% lower, an average. Increasing cement content from 1.5% to 2.5% resulted in higher *ITS* at all curing times and for all mixtures. The gain in strength was limited (about 2% in the long-term) for binder C1, whereas using the other binders, the average increase of *ITS* was 21%. Finally, we observe that, at the same curing, the strength of mixtures was generally lower than the strength of FAM mortars (Figure 13).

Figure 11. Evolution of *ITSM* for mixtures and FAM mortars produced with cementitious binders C1 and C3
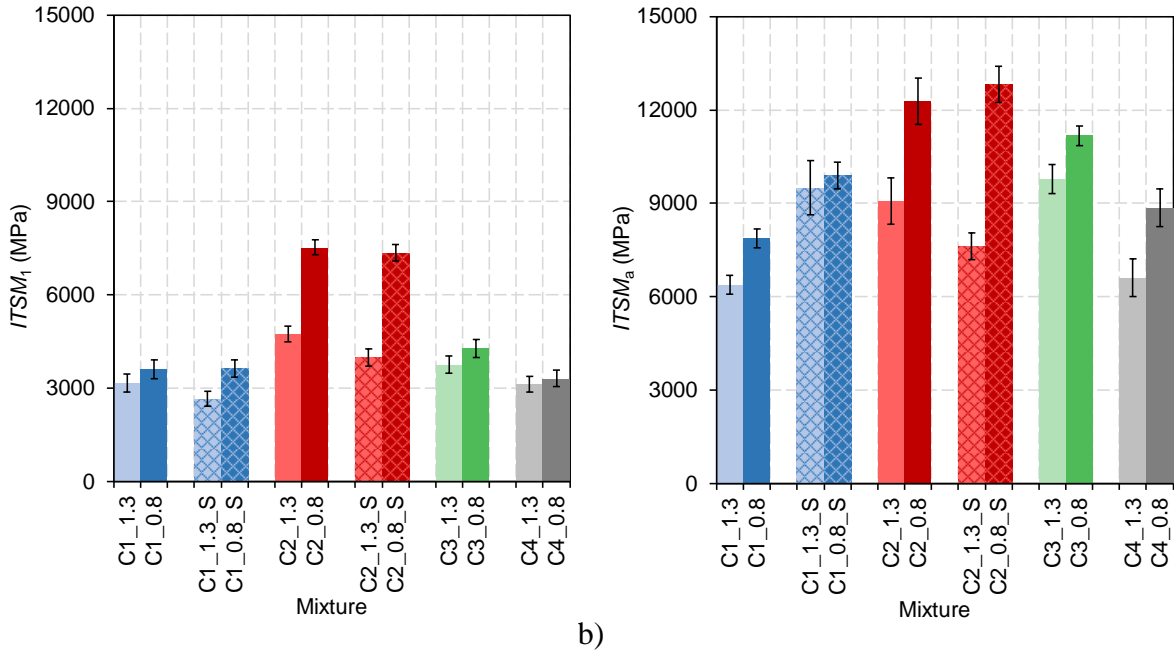
508

509   a)   b)

Figure 12. Regression parameters of the curing model for the *ITSM* of mixtures a) *ITSM₁*, b) *ITSM*ₐ
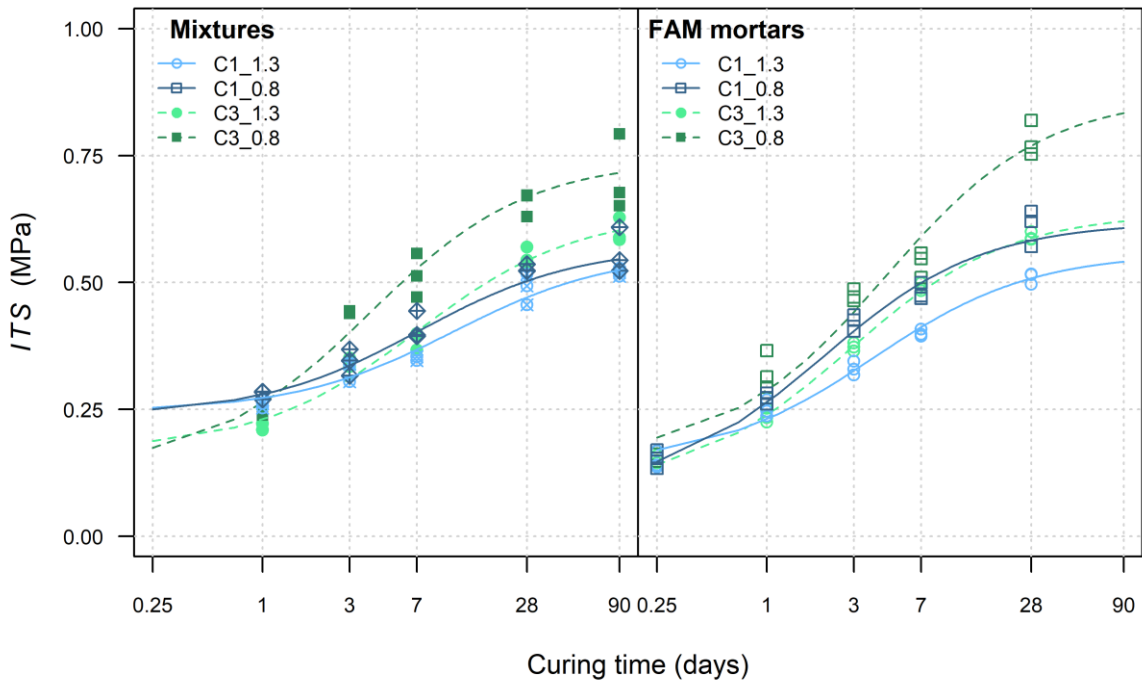
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Figure 13. Evolution of *ITS* as a function of curing time of mixtures produced with cementitious binders C2 and C3
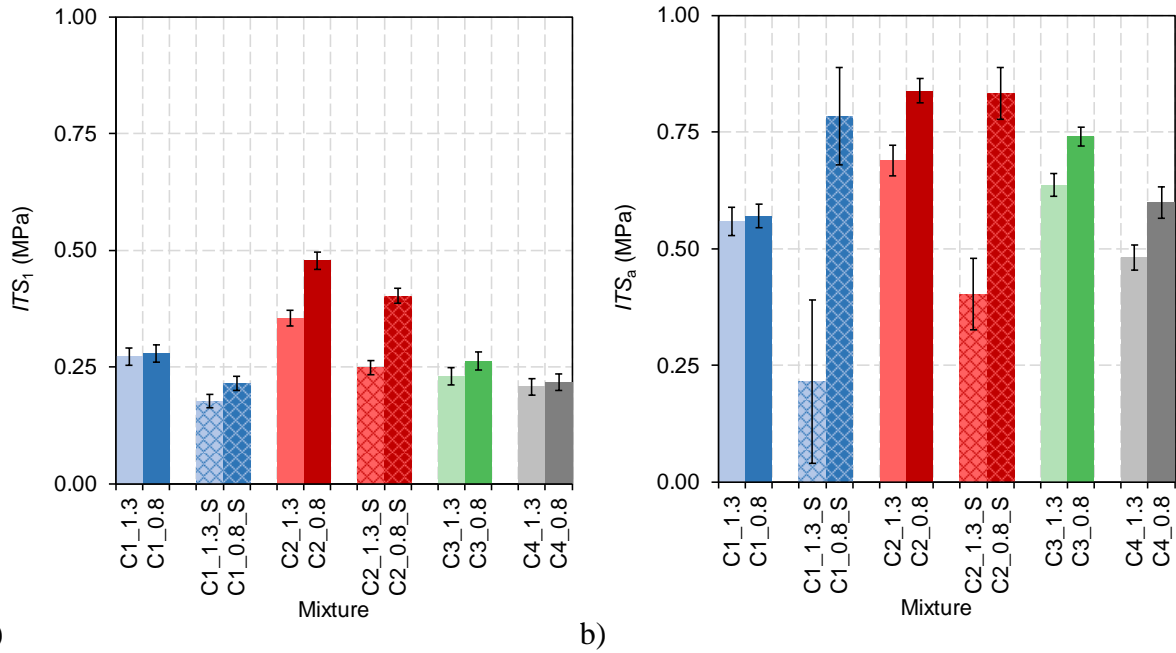
513
514

Figure 14. Regression parameters of the curing model for the *ITS* of mixtures a) *ITS*$_1$, b) *ITS*$_a$

*Effect of sealed curing conditions*

The effect of sealed curing conditions was evaluated on CBTM produced with cementitious

binders C1 and C2. Figure 15 and Figure 16 show, respectively, the evolution of *ITSM* and *ITS*

for mixtures and FAM mortars produced with C2. The regression parameters for all mixtures

are summarised in Figure 12 and Figure 14.

We observe that with sealed curing, although *DW* was negligible, *ITSM* and *ITS* increased

following similar trends to those observed in unsealed curing. Figure 15 shows that sealed

curing led to an increase in *ITSM* at the higher cement content (B/C = 0.8). Considering all the

mixtures, Figure 12 clearly shows that the effect cement content prevailed on the effect of sealed

curing. On the other hand, Figure 16 shows that sealed curing always led to a decrease of *ITS*.

This is confirmed by the model parameters shown in Figure 14. The adverse effect of sealed

curing is particularly evident on the short-term strength (*ITS*$_1$).

530　　　In summary, curing conditions with restricted water evaporation had a limited effect on

531　stiffness but penalised the strength. Since restricted water evaporation delays emulsion

532　breaking, we may conclude that stiffness, i.e. the small-strain behaviour, was mainly affected

533　by the cementitious bonds. On the other hand, strength, i.e. the large-strain behaviour, depended

534　on the development of both bituminous and cementitious bonds.

535　　　The critical role of restricted water evaporation on the development of mechanical

536　properties is confirmed by Figure 17 which shows the correlation between *ITS* and *ITSM* for all

537　the tested mixtures and FAM mortars. As can be observed, unique linear relationships relate

538　the measured stiffness and strength properties, provided that the same curing condition is

539　considered. For fixed values of stiffness, specimens cured in sealed condition were

540　characterised by lower strength. On the other hand, for fixed values of strength, specimens

541　cured in sealed condition were characterised by higher stiffness. Thus, unsealed curing led to a

542　better overall material performance: higher resistance with lower stiffness resulting in a less

543　prone-cracking material.

544
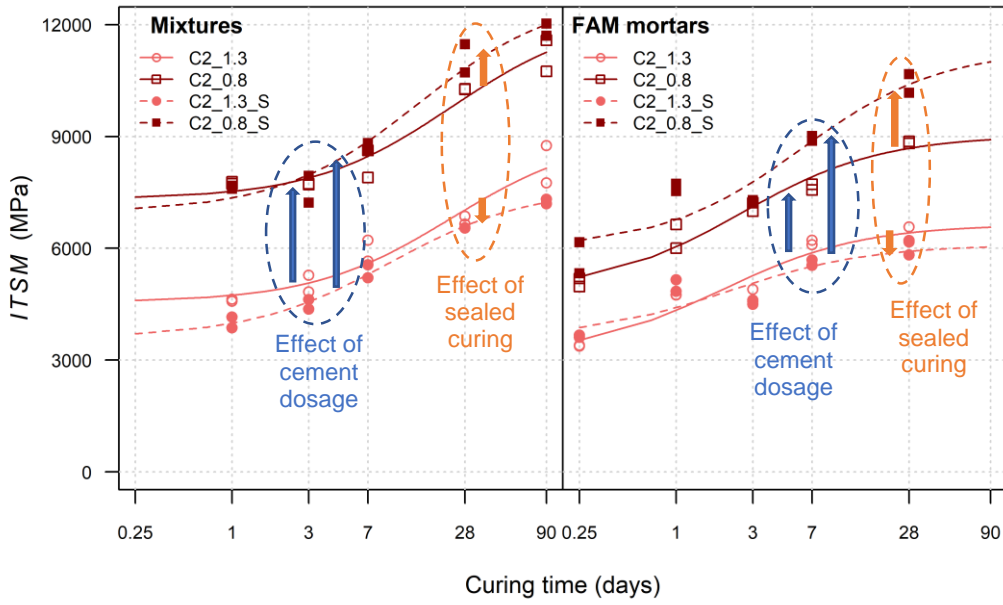
Figure 15. Evolution of *ITSM* as a function of curing time of CBTM mixtures and FAM mortars produced with cement C2 (U and S curing conditions)
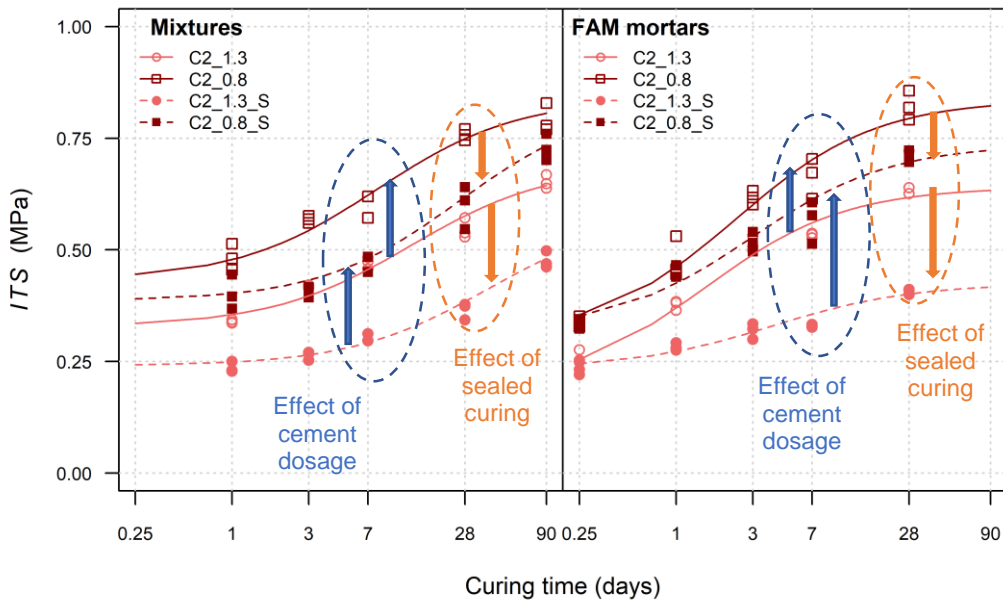
Figure 16. Evolution of *ITS* as a function of curing time of CBTM mixtures and FAM mortars produced with cement C2 (U and S curing conditions)
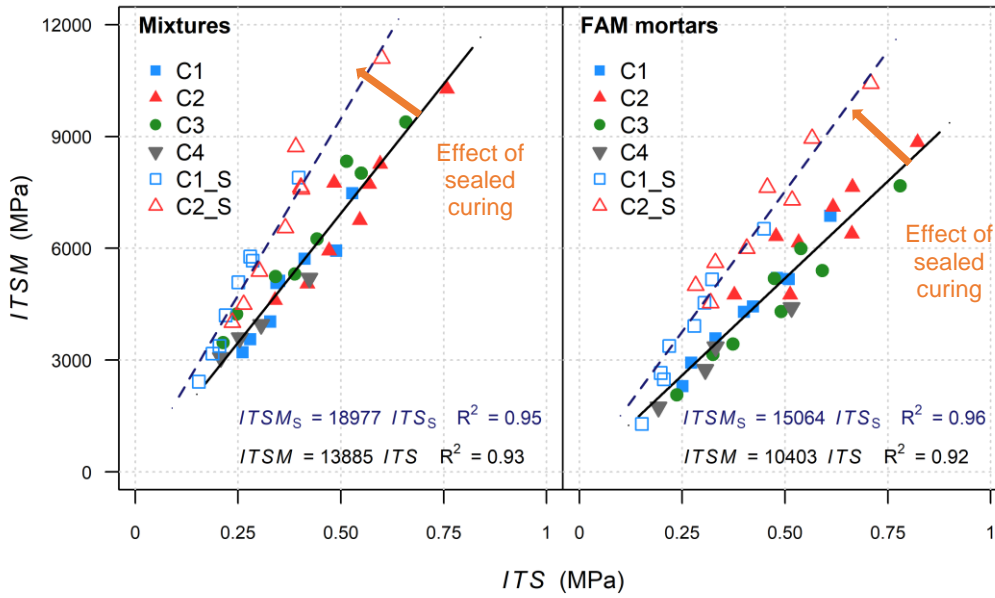
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Figure 17. Correlation between *ITSM* and *ITS* in unsealed (U) and sealed (S) curing conditions

4.4 Relationship between CBTM mixtures and mortars properties

Figure 18 shows the correlation between the mechanical properties of mixtures and FAM mortars measured at the same curing time (i.e. 1, 3, 7 and 28 days) and under the same curing conditions.

In general the mixture is stiffer than the mortar (Figure 18a), with a few exceptions represented by specimens produced with cement C2. This indicates that coarse aggregate acts as a reinforcement. The Hirsch model was used to simulate the relation between the stiffness of the mixture and the mortar [70], [71]. The model, originally developed for modelling cement concrete two-phase systems and later applied also to HMA mixtures, combines series and parallel elements (Figure 19a):

$$E_{mixture} = x(E_{CA}V_{CA} + E_{FAM}V_{FAM}) + (1 - x)\left(\frac{V_{CA}}{E_{CA}} + \frac{V_{FAM}}{E_{FAM}}\right)^{-1} \tag{7}$$

33

564 where V_{CA} and V_{FAM} are the volume fractions of coarse aggregate and FAM mortar (here 0.483

565 and 0.473, respectively). $E_{mixture}$ and E_{FAM} are the *ITSM* measured respectively for mixtures and

566 mortars, whereas E_{CA} and x are fitting parameters. E_{CA} represents the stiffness of the coarse

567 aggregate volume. The parameters x and its complementary, $(1-x)$, express proportions of

568 material structure in parallel and in series arrangement, respectively. In composites materials

569 the series arrangement (i.e. equal stress) indicates uniform stress and poor bonding. The parallel

570 arrangement (i.e. equal strain) stands for perfect bonding between the matrix and the inclusions

571 [71]. For cement concrete x value was found around 0.5 [70], [71]. The original model was

572 adjusted (Figure 19b) to take into account the external voids phase (Phase III) assumed in the

573 CBTM mixture conceptual model (Figure 4b) [52].

574 Least squares error minimization provided a satisfactory fitting of the whole experimental data

575 without regard to cementitious binder type, B/C ratio and curing conditions. The estimated

576 model parameters were $E_{CA} = 29511$ MPa and $x = 0.339$. The x value indicates a medium-low

577 bonding between the FAM mortar and the coarse aggregate.

578   Figure 18b displays that the mortar strength is equal or greater than the mixture one

579 throughout all the curing. This outcome can be linked to a scale effect due to the geometry of

580 the specimens adopted and to the dimension of pores that could accelerate the FAM curing

581 process. Additional studies to better understand these finding must be carried out.

582   The power law theory was adopted to modelling the relation between the *ITS* of the

583 mixture and the mortar [71]:

$$ITS_{mixture} = ITS_{FAM}(1 - aV_{CA}^{\,n}) \tag{8}$$

584 where a and n are model parameters function of shape and organization of the inclusions. The

585 model was proposed for modelling the strength of particulate composite characterised by poor

586 bonding between the inclusion and the matrix, and consequently considering no stress

34

587 concentration at their interface. Using the least square minimization, an excellent fitting was

588 obtained, with $a = 0.368$ and $n = 1.567$.

589 The existence of unique relationships between the mechanical properties of the two

590 CBTM composites, regardless of cement type, B/C ratio and curing conditions confirms that

591 mixtures and mortars showed similar sensitivity in terms of strength and stiffness evolution.

592 Thus, correctly designed FAM mortars can be potentially used to predict mixtures mechanical

593 behaviour over all the curing time regardless of their constituents. Further studies are though

594 needed to assess the prediction ability of FAM mortars considering a wider range of CBTM

595 compositions and types of components.

596 In CBTM, the RA aggregate is generally considered as a "black rock". This means that,

597 during the mixing at ambient temperature, no significant blending occurs between the bitumen

598 coming from the emulsion and aged bitumen coating RA. This allows considering the coarse

599 RA aggregate particles as perfect inclusions submerged in the FAM. Mortar composition was

600 obtained removing from the mixture the coarse RA aggregate and part of its voids (air and

601 water) (Sections 3.2 and 4.2). Considering that assumptions, it can be likely stated that the

602 evolutive behaviour of the mixture may be attributed to the FAM mortar properties evolution

603 and that the contribution of the coarse aggregate to the mixture mechanical properties is nearly

604 constant. For each CBTM investigated the ratios $ITSM_{FAM} / ITSM_{mixture}$ and $ITS_{FAM} / ITS_{mixture}$

605 were almost constant after three day of curing. This suggests that the structure of CBTM is

606 already developed after the first curing days. Emulsion breaking and cement hydration

607 processes are nearly completed and bituminous and cementitious bonds are mostly formed. The

608 increase in the mechanical properties overtime, then, can be attributed to the consolidation and

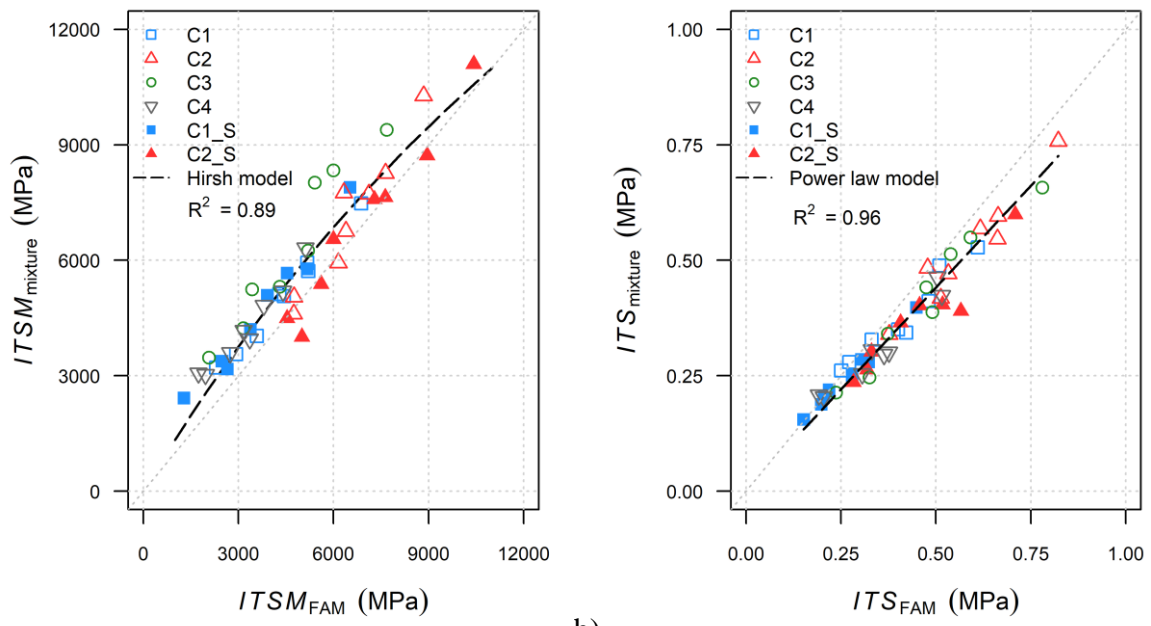609 strengthening of the existing matrix bonds and eventual aging effects.

610

a)   b)

Figure 18. Mechanical properties of CBTM mixtures as a function of mechanical properties of FAM mortars: a) *ITSM*, b) *ITS*

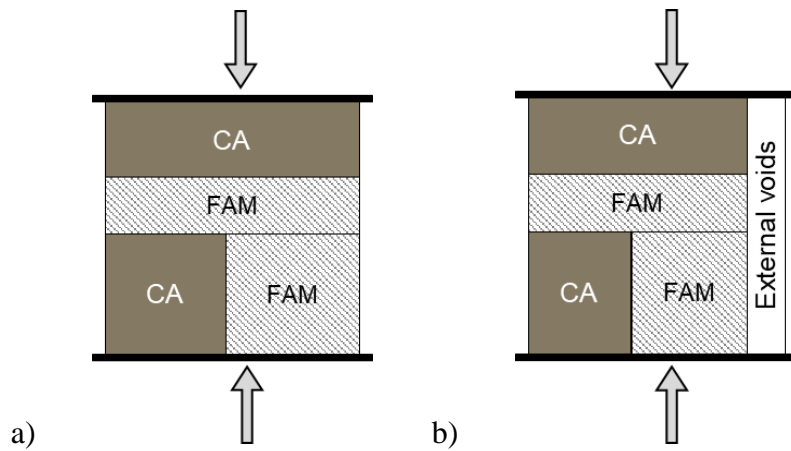
a)   b)

Figure 19. Schematic 1D representation of modulus composite models: a) original Hirsch model b) modified Hirsch model

5 CONCLUSIONS

The present study deals with a multiscale study of CBTM mixtures and FAM mortars. The evolution during curing of stiffness and strength is assessed to evaluate the effect of four types and two dosages of cementitious binders (two B/C ratios) and two curing conditions (i.e. unsealed and sealed curing). Finally, the predictive ability of properly designed FAM mortars is evaluated relating their mechanical behaviour to those of the mixtures. The main findings of the paper are the following:

- Use of rapid-hardening Sulfoaluminous cement C2 can improve the mechanical properties of CBTM in the early-stage of curing;

- Sulfoaluminous (C2) and high strength cement (C3) allow enhancing the mechanical properties of CBTM in the long-term;

- FAM mortars show a similar sensitivity in terms of stiffness and strength evolution compared to mixtures;

- Unsealed curing improved material performances resulting in an higher strength and lower stiffness;

- Properly designed mortars showed a satisfactory potential ability to predict mixtures behaviour regardless of curing conditions, cementitious binder type and dosages. It is worth highlighting that such a result could be related to the composition of mixtures examined.

Use of CBTM FAM mortar as a tool for predicting mixture behaviour and support mechanical modelling is a new tool. Despite that, it has turned out to be very promising. Further studies are needed to evaluate the influence of scale effects on the predicting ability.

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