Comparison of accelerated laboratory curing and maturation under uncontrolled conditions of gravel emulsions using a nonlinear weighted least-squares predictive model

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Word count: 7,649

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Abstract

Cold-asphalt mixtures (CAMs) are versatile, environmentally friendly, and less costly than other bituminous options. Moreover, they provide a more favourable work environment for workers. Nevertheless, CAMs are evolved materials whose mechanical strength increases with their field maturation. There is a lack of knowledge regarding the correlation between laboratory-accelerated curing protocols and field maturation for such mixtures. Moreover, there is no consensus regarding the most appropriate laboratory-accelerated curing protocol. This study, therefore, analysed the accelerated curing protocol for a CAM-type gravel emulsion and compared it with its uncontrolled maturation and performance. For this purpose, a gravel emulsion was exposed to either of two different curing processes: natural curing (from 0 days to 6 months at 20 ± 3 °C in the laboratory, and at an unfixed temperature outside of the laboratory) or accelerated curing (from 0 to 7 days in a 50 °C oven). We analysed, as functions of time, the moisture, indirect tensile stiffness modulus (ITSM), and moisture damage resistance through indirect tensile strength (ITS) under immersion. A curing protocol of 2 days at 50 °C yielded ITSM values closer to those obtained under uncontrolled conditions, overestimating them by 22.9%. Nevertheless, total maturation of the mixture was achieved after 3.5 days of curing. The nonlinear weighted least-squares method yielded an expression that better predicted the performance of the gravel emulsion for the first six months of life. The model parameters were easily fitted using ITSM measurements derived from samples cured for 2 days in a 50 °C oven.

Keywords: cold-asphalt mixture; gravel emulsion; evolutive material; uncontrolled maturation; accelerated curing protocol; nonlinear weighted least squares

Introduction

Cold-asphalt mixtures (CAMs) are bituminous mixtures that are manufactured and executed at ambient temperature (Al-Busaltan *et al.* 2012). CAMs are typically composed of mineral aggregates and bitumen emulsion, cutback bitumen (Asphalt Institute 1989), or foamed bitumen (Shanbara *et al.* 2021). Also, pre-wetting water is normally added to improve the coating ability of the binder (Al-Busaltan *et al.* 2012). In addition, 1% (or more) of cement is usually added to improve the mechanical properties of the CAMs (Al-Busaltan *et al.* 2012, Wirtgen GmbH 2012, Cardone *et al.* 2015, Li *et al.* 2020, Yang *et al.* 2021). CAMs have well known benefits and drawbacks, compared with hot-mix asphalt. Their notable outstanding advantages and disadvantages are summarised in Table 1.

CAMs are evolutive materials because their mechanical strength increases with curing time owing to the breaking of the bitumen emulsion during the curing process (Li *et al.* 2020) or to free water evacuation while curing in the case of foamed bitumen (Chen *et al.* 2021). In this regard, in the initial stages of service, they behave similarly to an unbound granular material (Casillas and Braham 2021, Orosa *et al.* 2022) and not like hot-mix asphalt. This means that a considerable amount of time (Tebaldi *et al.* 2014) is required for CAMs to achieve stability, that is, to reach their maximum strength. According to some authors (Leech 1994), 2 to 24 months are required to achieve complete field maturation of CAMs manufactured with bitumen emulsion, depending on weather conditions. Other authors (Serfass *et al.* 2004) indicated a period that includes a complete cycle of seasons for CAM-type gravel emulsion (GE). Different maturation periods have been reported for the type GE CAMs, such as a minimum of 1 to 2 months under favourable weather conditions, that is, with high temperatures and good aeration (Junta de Castilla y León 2004).

Curing (CEN 2019) is the process by which a bituminous mixture manufactured with a bitumen emulsion or foamed bitumen evolves over time until it reaches stability. The curing process is affected by several factors, such as the type of binder, the bituminous layer thickness, the climate, and water drainage (CEN 2019). Laboratory curing of CAMs attempts to simulate an accelerated field maturation process (Jenkins and Moloto 2008). Several investigations have examined the effect of accelerated laboratory curing protocols on the performance of certain CAM types. According to Kim et al. (2007), the laboratory curing temperature affects the stiffness of cold-in-place recycling (CIR) mixtures with foamed asphalt, such that the indirect tensile strength (ITS) increases with the curing temperature. In addition, the curing length affects the properties of CIR mixtures made with bitumen emulsion or foamed bitumen (Kim *et al.* 2011). Some authors (Chen *et al.* 2021) reported a negative influence of excessive water content on the strength acquisition of foamed CIR mixtures. This effect is even more remarkable in the case of cold climates because freeze-thaw cycles can reduce the stiffness modulus (Chen *et al.* 2021). Yang et al. (2021) reported that for cold-recycled mixtures with bitumen emulsion (CRMAE), the use of a single curing condition (2 days at 60 °C) instead of mixed curing conditions (3 days at 20 °C followed by 2 days at 60 °C) produced mixtures with higher strength and lower air voids. There is no consensus on the laboratory-accelerated curing time, humidity, and temperature conditions for CAM samples. As shown in Table 2, researchers have used various curing protocols for emulsified and foamed CAM mixtures. Some even established a correspondence between accelerated curing time and field maturation.

A particular case of CAM is GE. This mixture is a type of dense-graded cold mix asphalt with a bitumen emulsion, first used in France in the 1950s as a reprofiling bituminous mixture (Day *et al.* 2019). Currently, its use has been extended to other countries such as Ireland (Bullen *et al.* 1994) or Spain (Bardesi 1994), not only for reprofiling but as a base course for low to medium traffic and as an overlay to strengthen distressed pavements (Shanbara *et al.* 2021). GE is usually manufactured in a plant by mixing wet aggregates with a continuous grain-size distribution, slow- or medium-setting bitumen emulsion, water, and some additives (Bordes *et al.* 1987, Needham 1996, Shanbara *et al.* 2021). In particular, the additives used for manufacturing gravel emulsions are used for bitumen emulsion breaking control (ATEB n.d.). In general, the residual binder content of a GE varies from 3.8 to 4.8% (Charentais n.d.). Nevertheless, some countries use lower proportions of residual binder contents, such as 2.5% residual binder for low-traffic roads in Spain (ATEB n.d.). After mixing, spraying, and compacting the GE at ambient temperature, two phases can be distinguished: a binder-rich mortar that provides cohesion, and a mineral skeleton partially covered with binder that provides internal friction (Needham 1996). However, the mechanical performance of GE changes significantly during the curing process, particularly in the first two years. This curing process is affected by traffic, climate, and water drainage (Lafon and Puggelli 1997). However, GE can be trafficked immediately after compaction. In this regard, despite its low stiffness in early life, it has demonstrated good field performance (Needham 1996). This type of mixture displays good performance against permanent deformation and fatigue (Charentais n.d.). However, GE is highly water-sensitive, making it suited to warm and dry regions (Needham 1996).

Aims and scope

The technical literature (Table 2) related to laboratory-accelerated curing protocols for CAM-type GEs is limited. Moreover, the relationship between laboratory-accelerated curing and field maturation remains poorly understood. Therefore, the objectives of this study were:

- to determine the optimal artificial curing time for a GE and compare it with the curing time recommended by the current standards or typically used protocols for CAM design;
- to compare the artificial (laboratory) curing time with maturation under uncontrolled conditions, similar to those experienced by a mixture in the field;
- to determine how accurately the stiffness of GE subjected to artificial curing time represents the stiffness of the mixture when it acquires resistance under uncontrolled conditions;
- to design a model to predict GE performance under uncontrolled conditions using simple laboratory measurements, e.g., the indirect tensile stiffness modulus (ITSM) of samples subjected to an accelerated curing protocol.

Our achievements are threefold: We compare the artificial conventional curing protocol with uncontrolled maturation, investigate the differences between the "laboratory" ITSM and the "uncontrolled" ITSM, and develop a model that predicts the ITSM of a GE under uncontrolled curing conditions that are representative of those encountered by a mixture during its in-service life.

For this purpose, a CAM-type GE was manufactured with an optimum bitumen emulsion content of 5% by weight of dry natural aggregates. The GE was subjected to two different curing processes: natural curing (for 6 months at 20±3 °C in the laboratory, and at an unfixed temperature outdoors) or accelerated curing (for 7 days in a 50 °C oven). The moisture content over time, stiffness, and moisture damage resistance were analysed. The ISTM as a function of time was fitted to a nonlinear weighted least-squares regression, using a model that accurately predicted the GE stiffness under uncontrolled conditions. A weighted regression, inversely proportional to the variance of each data point, was used. The parameters of the resulting equation were estimated using ITSM measurements after two days of accelerated curing in a 50 °C oven.

Materials and methods

Aggregates

Natural siliceous aggregates were used, in particular hornfel (62.30% SiO_2), which was supplied by a local quarry.

The bulk specific gravity (ρa) and water absorption (W_{24}) of the hornfel was determined following EN 1097-6 (AENOR, 2014). The sand equivalent (SE) was obtained following EN 933-8 (AENOR, 2015). The Los Angeles (LA) abrasion coefficient was determined according to EN 1097-2 (AENOR 2021). The EN 933-3 (AENOR 2012) was used to determine the flakiness index (FI). Finally, the percentages of crushed and broken surfaces were obtained using EN 933-5 (AENOR, 2005). Table 3 compares these properties with the ATEB (n.d.) requirements. The quarry aggregates are visibly suitable for all heavy-traffic categories. To compose the GE, we used (see Table 4) 20% of the fraction 0/2 mm, 40% of the fraction 2/6.3 mm, and 40% of the fraction 10/20 mm by weight.

Commercial bitumen emulsion

We selected a slow-setting cationic bitumen emulsion type C60B5 GE (AENOR 2013). According to the supplier, the bitumen content was no less than 58%.

Mix design and sample preparation

We selected gravel emulsion type GE-1 (ATEB n.d.). The grain-size distribution is specified in Table 4. The GE dosage was determined by performing envelope water tests (MOPT 1995), modified proctor tests (AENOR 1994), and immersion-compression tests (MOPT 2000). Thus, 5.0% by weight of dry aggregate was selected as the optimal bitumen emulsion content, representing 3.0% of the residual binder. The optimum envelope water content was 3.0% by weight of the dry aggregate.

The most suitable mixing procedure was applied by mixing the envelope water with the aggregate for 1 min and then mixing the emulsion. The total mixing time was 2 min. The mix was prepared in an automatic mixer with kneading paddles undergoing planetary movement. A gyratory compactor was used for compaction by applying 80 gyrations. The resulting gravel emulsion is suitable for low-volume heavy-traffic category T3 (the annual average of the daily number of heavy traffic vehicles, AADHT, ranges from 50 to 200) according to the ATEB (n.d.).

Curing procedure

Two curing procedures were selected: natural curing and accelerated curing.

- Natural curing: Eight identical GE-1 cylindrical specimens were manufactured at their optimal residual binder content (3%) and water content (3%) and compacted with 80 gyrations of the gyratory compactor. Four of these specimens were left inside the laboratory at room temperature (22±3 °C), while the other four were left outside. Between May and December 2019, temperature measurements were made at 12:30 h using a digital thermometer. (From experience, the mean temperature of the day is displayed at this hour.) This temperature varies by approximately ±5 °C over a daily cycle. The test samples were then reweighed. Measurements were taken daily during May, weekly in

June, and monthly from July to December. Notably, the relative humidity ranged from 76 ± 3% during the measurement period outside the laboratory to 50 ± 5% inside. There were also 5.6 rainy days (>1 mm rain fall) in July and 14.6 rainy days (>1 mm) in August. The outside samples were subjected to variable temperature, humidity, and rain to determine the actual weather conditions.

- Accelerated curing: The Spanish guidelines (ATEB n.d.) for the design of GE-1 recommend subjecting the GE samples to a curing time of 3 days in an oven at 50 °C before analysing its performance. We, therefore, maintained this temperature because it was not very time-consuming, but the curing time was modified. In this regard, the GE-1 samples with optimal water and residual binder contents, compacted with 80 gyrations of the gyratory compactor, were subjected to an accelerated curing time in an oven at 50 °C for 1, 2, 3, 4, 5, 6, and 7 days before testing.

Moisture as a function of time

Some authors reported that GE cannot be dried in the field, and that after a long time, its moisture content ranges from 0.5 to 1.5% (Serfass *et al.* 2004). In addition, some design guidelines indicate the need for maturation time in the field to avoid the GE residual water content exceeding 1% (Junta de Andalucía 2007). This justified determining the humidity as a function of time for the samples of GE-1 subjected to natural curing, and regarding this parameter as an indicator of maturation. In addition, for the GE-1 mixtures subjected to artificial curing, the time-dependent humidity was determined until the mass saturated. The moisture content (%) was calculated as

$$w = \frac{P_w}{P_s} \times 100 \qquad (1)$$

where Ps (g) is the dry weight of the sample and Pw (g) is the weight of water in the sample.

ITSM

The stiffness modulus is demonstrably an adequate parameter for analysing GE maturation (Serfass *et al.* 2012). The stiffnesses of GE-1 mixtures subjected to natural and artificial curing were determined at their optimal water and residual binder contents. EN 12697-26, Annex C) was used (AENOR 2019a). The ITSM was determined using a Cooper NU 14 tester. The test was conducted in a climatic chamber at a controlled temperature of 20 °C. First, ten conditioning Haversine pulses were applied along the vertical diameter of a cylindrical GE-1 specimen compacted with 80 gyrations in the gyratory compactor. Subsequently, five Haversine test pulses were applied with an impulse repetition period of 3 ± 0.1 s. A maximum load was selected to achieve a maximum horizontal strain of 0.005% of the specimen diameter. The rise time was 124 ± 4 ms. The average stiffness modulus of each specimen was determined using five test pulses. After rotating the specimen by 90º, the test sequence was repeated. The average stiffness from the two tested diameters of four GE-1 samples was recorded as the stiffness modulus. The ITSM (MPa) was determined as

$$ITSM = \frac{F \times (\nu + 0.27)}{z \times h} \quad (2)$$

where F is the maximum applied load (N), z is the horizontal deformation (mm), h is the sample thickness (mm), and $\nu = 0.35$ is Poisson's ratio assumed for the GE-1 mixtures). There are no requirements for GE-1 in terms of ITSM.

Water sensitivity

We analysed the water sensitivity of GE-1 mixtures with optimal water and residual binder contents, cured in an oven at 50 °C for 1, 2, 3, 4, and 7 days. The purpose of this analysis was not to study the maturation but to determine whether the accelerated curing time in the laboratory affects the moisture damage resistance of the mixture. To analyse the water resistance of GE-1, the tensile strength ratio (TSR) of the mixture was calculated according to EN 12697-12 (AENOR 2019b):

$$TSR = \frac{ITS_W}{ITS_D} \times 100 \qquad (3)$$

where ITS_W and ITS_D are the average tensile strengths of the "wet" and "dry" groups, respectively. The four cylindrical (100 mm diameter and 60 mm height) specimens of the "wet" group were subjected to a water bath at 40 °C for 3 days, while the four samples of the "dry" group were at room temperature. Subsequently, both groups were conditioned for a minimum of 4 h at 15 °C. Both groups were tested in indirect tensile stress mode.

Predictive model

After analysing the potential outliers according to Iglewicz and Hoaglin (Iglewicz and Hoaglin 1993), we performed a nonlinear weighted least-squares regression, a method widely used in engineering to develop prediction models (Yang and You 2015, Hussan *et al.* 2020). We minimised the weighted squared sum of the residuals by adjusting the model parameters using the reduced gradient engine built in the Excel ® solver. Heteroscedasticity of the data was assumed. Thus, the data were weighted in inverse proportion to the corresponding variance, forcing the curve representing the model to pass nearer the points with a low variance. The data variance was computed using four replicates. Parameter uncertainties were estimated by calculating the variance-covariance matrix at the optimal parameter values, which in turn were calculated using the Jacobian matrix (Englezos and Kalogerakis 2000).

Results and discussion

Moisture as a function of time

Figure 1 shows the evolution of (a) the ambient temperature, (b) the sample weight, and (c) the moisture content as functions of time. Figure 1d plots the moisture evolution over the first 10 days only, to resolve in greater detail the period when the humidity ranges from 0.5 to 1.5%, i.e., the usual range of field values for a GE, as previously stated. For the mixtures cured inside or outside the laboratory, this range was spanned within approximately 1 day or between 1 and 2 days of curing, respectively. It is also interesting to know when the moisture content is less than 1.0%, the threshold specified by some authors where GE-1 completes its field maturation. This threshold is crossed after approximately 1.5 days or within less than 1 day of curing, for the mixtures cured outside or inside the laboratory, respectively.

This difference probably results from the temperatures being higher in the laboratory than outside. In this regard, Figure 1a shows lower environmental temperatures (19.0 ºC on average) outside the laboratory than inside (22.2 °C), i.e., a 3.2 °C difference. Moreover, the minimum temperature was 12.9 °C outside the laboratory, and 19.3 ºC inside, i.e., 6.40 ºC higher. The maximum temperature was 22.10 ºC outside the laboratory, and 24.80 ºC inside, i.e., 2.70 ºC higher. Outside the laboratory, one must also consider the ±5 ºC temperature variation around the measured values, over the daily cycle.

Figures 1b and 1d show the weight and the moisture decreasing rapidly during the first 3 days, with a slower decrease thereafter. In both situations, their mass saturates from day 9, i.e., the weight and moisture vary negligibly thereafter. Similarly, other authors (Li *et al.* 2020) also obtained three differentiated parts in the CAM moisture loss curve plotted as a function of time. A rapid loss is observed during the first three days, a slow loss until day 14, and a plateau thereafter. However, this performance is notably highly dependent on the weather conditions. The time boundaries between these different regimes can therefore shift accordingly.

Figure 2 shows the GE-1 moisture as a function of the accelerated curing time in the oven. After three days of curing in the 50 °C oven, the sample reached a constant mass, that is, consecutive weight measurements differed by less than 0.1% (AENOR 2020). Therefore, measurements of moisture over time for GE-1 cured at 50 °C in the oven were only performed until day 3. As shown in Figure 2a, the moisture content decreased rapidly during the first day of curing in the oven, but very slowly thereafter. In addition, values below 1.0% were reached on the first day of accelerated curing, particularly after 18 h of curing. Thus, 18 h is the minimum curing time at 50 °C in the oven, which reaches the required moisture content of 1.0%.

ITSM

Figure 3a shows the ITSM results for the samples subjected to natural curing inside and outside the laboratory as functions of the curing time. The times of the first plotted points correspond to 9 days (for the samples left outside of the laboratory) and 10 days (inside) because samples cracked when performing the ITSM tests in the first few days. This is an interesting observation because, despite the moisture content being below 1%, the cohesion of these mixtures and their maturation were insufficient to perform the ITSM test without clear damage. As a result, the moisture analysis is insufficient to determine when GE-1 is completely cured in the case of low traffic.

Figure 3a also shows that both groups of samples (naturally cured inside and outside the laboratory) present similar stiffness trends over time. First, the ITSM increases rapidly up to a maximum and decreases slowly thereafter. The stiffness typically increases monotonically with curing time, first swiftly and then slowly, therefore this is not the typical tendency (Needham 1996, Ozsahin and Oruc 2008, Beghin *et al.* 2012, Grilli *et al.* 2019, Ferrotti *et al.* 2020). However, even though the indirect tensile modulus test is nondestructive, when the samples are subjected to numerous measurements over time, as in this instance, some damage may happen due to the cumulative influence of the loads. When most mixture cohesion developed, the cumulative damage produced a slight decrease in the modulus. Needham (1996) found a similar trend for CAMs manufactured with polymer-modified emulsions and 1% Portland cement. Sample damage before curing in the oven arguably explains this trend. This phenomenon may well have arisen in the present study.

Figure 3a also shows that the maximum ITSM occurred after approximately 1 month (30 days) or 2 months (60 days) for the samples cured inside or outside the laboratory, respectively. Moreover, the maximum measured ITSM was 21.34% higher for the samples cured inside the laboratory (436.38 MPa) than outside (359.63 MPa), owing to the higher temperatures recorded inside. Moreover, daily variations in temperature (between day and night) can deteriorate the outside specimens.

Figure 3b plots the ITSM for samples subjected to accelerated curing at 50 °C in an oven as a function of the curing time. It follows a similar trend to that encountered with natural curing. To explain this trend, it must be noted that, according to some authors (Beghin *et al.* 2012), for high curing temperatures (50 °C or higher), samples can develop cracks because of rapid water evaporation (Serfass *et al.* 2004). In this case, longer curing times favour more cracks. When the samples developed their maximum cohesion, the effect of cracks started to increase with the curing time, slightly reducing the ITSM. Other authors have stated that bitumen flow can occur at high curing temperatures (60 °C or higher), resulting in altered properties (Jones *et al.* 2014). This phenomenon might also be responsible for a reduced ITSM.

Figure 3b also shows that the maximum measured ITSM occurred after three days of accelerated curing. This value was 44.37% higher than the maximum in samples subjected to natural curing in the laboratory and 75.18% for samples subjected to natural curing in the laboratory. This result was also expected because higher curing temperatures produce stiffer materials.

Two days of accelerated curing time (442 MPa) made the ITSM 1.3% higher than the maximum value (436.38 MPa) obtained for samples subjected to natural curing in the laboratory, and 22.90% for samples subjected to natural curing out of the laboratory (359.63 MPa).

Water sensitivity

Figure 4 shows the ITSd, ITSw, and TSR for mixtures subjected to different accelerated curing times (from 1 to 7 days). This figure clearly shows the ITSd increasing with the curing time. Similarly, Kim *et al.* (2011) concluded that the ITS increased slightly with the curing time between 0 and 14 d, for both 25 °C and a curing temperature of 45 °C. Other authors (Ojum 2015) also reported the ITS increasing with the curing time. Nevertheless, the ITSw increased for the first 2 or 3 days of curing, then decreased in day 4 before remaining practically constant up to day 7. Figure 4 also shows the TSR increasing from 1 to 3 d of curing. Then, the maximum TSR was produced after 3 d of accelerated curing. Finally, the TSR decreased in day 4 and remained practically constant up to day 7. These two last trends (for ITSw and TSR) could be explained by up to 3 d of curing at 50 °C, and the mixture undergoing accelerated maturation. However, after 3 days of curing at 50 °C, the mixture achieved its maximum ITSM (as shown in Figure 3b) and lost all water (Figure 2). As other authors have demonstrated

(Beghin *et al.* 2012), for curing temperatures of 50 °C or higher, small cracks may appear, allowing water to penetrate and thus reduce ITSd, which is detrimental to the water resistance of the mixture. In addition, the bitumen flow (Jones *et al.* 2014) that occurs at high curing temperatures may affect the water resistance of the mixtures.

Predictive model

In the present investigation, we minimised the weighted sum of squared residuals to optimise the model equation parameters. The resulting model accurately represents the *ITSM* (in MPa) of GE subjected to uncontrolled curing for the first six months of its useful life as a function of the number of days of field life (*t*). The best fit was achieved using the functional form

$$ITSM\ (MPa) = \frac{a \cdot t}{b + t^2} + \frac{c \cdot t}{d + t} \qquad (4)$$

where *a*, *b*, *c*, and *d* are the fitting parameters. This model, expressed as the sum of two rational functions, describes a curve with both a maximum and a positive asymptote at infinite *t*. The first function ensures a maximum value for the resilience modulus, and the second function, similar to the Michaelis-Menten function (Michaelis *et al.* 1913), tends asymptotically to *c* as $t \to \infty$. The latter function, describing the rate of an enzymatic reaction as a function of substrate concentration, was adapted by Graziani et al. (2016, 2017) to describe the evolution of the mechanical properties of the curing process over time. In this case, from the measurements in specimens undergoing natural curing outside the laboratory, we have $a = (26.44 \pm 0.72) \times 10^3$, $b = (26.6 \pm 1.0) \times 10^2$, $c = 117.3 \pm 4.5$ and $d = 1.60 \pm 0.14$. These parameters accurately reproduce the performance of the uncontrolled-cured GE samples, as shown in Figure 5. Based on these estimates, a maximum ITSM of 370.2 MPa was predicted at a curing time of approximately 52 days. The model also predicts a plateau value of 117.3 MPa.

The model has been developed on the basis of the empirical findings. As a result, it is not a general model. Is a model that correctly forecasts the ITSM progression for CAM mixtures type GE under the local climate where the mixture was cured.

Previously, it was shown that the maximum ITSM for a GE subjected to uncontrolled conditions outside of the laboratory occurs at 1 month and 3 weeks of useful life. This maximum value is 22.9% lower than for mixtures cured for 2 days in a 50 °C oven. Then, starting from the ITSM value obtained inside the laboratory for specimens cured for 2 days at 50 °C, and taking all of this into account, the parameters *a*, *b*, c, and *d* can also be estimated using the Excel Solver function. This is possible for CAM mixtures type GE and under the local climatic circumstances.

Figure 5 compares the real measurements made on the samples outside the laboratory, their average, and two model predictions. One model was obtained via the nonlinear weighted least-squares method, based on measured samples outside the laboratory. The other model was estimated from the ITSM of samples cured for 2 days at 50 °C in the laboratory, and using the type of equation that achieves the better fit, according to the weighted least-squares model. The estimated model from the ITSM of the GE samples cured for two days in an oven at 50 °C accurately reproduces their uncontrolled curing performance for the first six months of life.

Conclusions

We subjected CAM-type gravel emulsion GE-1 to two different curing processes. On the one hand, a natural curing was performed inside and outside the laboratory under uncontrolled conditions to simulate field weather conditions. On the other hand, accelerated curing for 1 to 7 days in an oven at 50 °C was also conducted. The moisture, stiffness, and moisture damage resistance were analysed as functions of the curing time.

- To develop an accelerated curing protocol that is practical and not excessively time-consuming, a temperature of 50 °C was adopted, as recommended by national specifications.
- Given the temporal evolution of moisture, the curing time in the oven at 50 °C should not be less than 18 h because the required minimum moisture content (1.0%) cannot be achieved with shorter curing times.
- The ITSM, ITSw, and TSR recommend against curing the GE-1 samples in the oven at 50 °C for more than 3 days, to avoid serious damage to the samples.
- An accelerated curing laboratory protocol involving 3 d of curing in an oven at 50 °C results in an overestimated field stiffness. Thus, the ITSM obtained for laboratory-cured samples is approximately 75% greater than that obtained under uncontrolled conditions. After 2 days of accelerated curing, the uncontrolled ITSM was overestimated by only 22.9%.
- The ITSM obtained after 2 days of curing was closer to the uncontrolled ITSM than that obtained after 3 days of curing, and therefore better represents the GE field performance.
- The nonlinear weighted least-squares method yielded an equation fitted to the uncontrolled GE performance and predicted the final ITSM value.
- Based on this expression, and considering that the maximum ITSM occurred after 1 month and 3 weeks of the sample's life, with a value 22.9% lower than that of the laboratory, a predictive model of the ITSM is easily estimated using only the ITSM measured in the laboratory on specimens cured in an oven at 50 °C for 2 days. This model accurately reproduced the ITSM of the mixture for the first six months of uncontrolled curing.

Since the results of this study were empirically acquired under certain weather conditions, extrapolating its implications would involve running studies in several locations, with various temperatures and humidity levels, as well as using various kinds of CAM.

Acknowledgements

The EMULCELL project was co-financed by the Centre for Industrial Technological Development (CDTI), the Ministry of Economy and Competitiveness-Government of Spain, and the European Regional Development Fund (FEDER) (Ref. IDI-20171097).

The authors would also like to thank “Repsol” and “Áridos de Astariz” for kindly providing the necessary materials to carry out the research.

Declaration of interest statement

There are no relevant financial or non-financial competing interests to report.

Data availability statement

The data that support the findings of this study are available from the corresponding author [A.R.P] upon reasonable request.

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Tables

Table 1. Main benefits and drawbacks of CAMs, according to the literature.

Benefits	**Drawbacks**
Environmentally friendly (Al-Busaltan *et al.* 2012).	Reduced mechanical properties in the first stages of its in-service life (Al-Busaltan *et al.* 2012). Particularly weak strength (Shanbara *et al.* 2021).
Energy/heat savings (Al-Busaltan *et al.* 2012, Li *et al.* 2020). Particularly, 13% of energy savings is achieved when comparing a cold-mix asphalt with a hot-mix asphalt manufactured at 160 ºC (Le Bouteiller 2010).	Extended maturation period (Al-Busaltan *et al.* 2012, Shanbara *et al.* 2021). Some authors indicate the need for up to 24 months for some CAMs to achieve adequate curing (Nikolaides 1983, Dulaimi *et al.* 2016).
Versatile. Can be used for different situations (Asphalt Institute 1989).	Very sensitive to rainfall in the first stages of its service life (Al-Busaltan *et al.* 2012).
Better working environment (Shanbara *et al.* 2021).	High air voids content (Al-Busaltan *et al.* 2012, Shanbara *et al.* 2021).
	Problems in mixing, compacting, and curing with excessive moisture (Asphalt Institute 1989).

Table 2. Accelerated curing laboratory protocols for different CAMs according to the literature and its field maturation correspondence.

Research	Type of CAM (*)	Water, bitumen, and/or cement	Curing condition	Field maturation correspondence
(Konrad and Walter 2001)	Dense graded	Water: 5% Residual binder: 2.5%	Up to 90 days at room temperature with different moisture contents	-
(Serfass *et al.* 2004)	Gravel emulsion and dense-graded asphalt concrete	Water: - Residual binder: 4.0 and 5.0% respectively	Some protocols were tested, but they propose: ✓ 14 days at 35 ºC (20% of humidity) for the mature state ✓ 14 days at 18 ºC (50% of humidity) for the fresh state	1 to 3 years in a field with a temperate climate, or a few weeks after laying the mixture in a field in a temperate climate, respectively
(Kim *et al.* 2007)	Foamed CIR (Cold in-place recycling)	Water: 4% Foamed asphalt: 1 to 3%	Two protocols: ✓ 2 days at 60 ºC ✓ 3 days at 40 ºC	-
(Kim *et al.* 2011)	CIR (foamed and emulsified)	Water: 4 and 3% respectively Residual binder: 2 and 3% respectively	Some protocols: 0, 7, and 14 days in an oven at 25 and 45 °C after the specimens had been cured in the air for 0, 1, 3, and 5 h.	-
(Serfass *et al.* 2012)	Gravel emulsion	Water: 6.6% Residual binder: 4.06%	Some protocols: ✓ 7, 14, and 28 days at 35 ºC. Relative humidity 20% ✓ 60 days at 18 ºC. Relative humidity 50% ✓ 14 days at 35 ºC + 60 days at 18 ºC. Relative humidity 20%	14 days at 35 ºC (20% humidity) for small samples: a period including two summers in a temperate climate
(Tebaldi *et al.* 2014)	CIR	-	Different curing protocols according to the literature: ✓ In United Kingdom 3 days at 60 ºC ✓ In Portugal 1 day at room temperature + 3 days at 50 ºC ✓ In Spain 3 days at 50 ºC ✓ In Ireland 14 days at 35 ºC and 20% of relative humidity	-
(Cardone *et al.* 2015)	Cement-bitumen treated materials	Water: 5% Bitumen emulsion: 3% Cement: 1% and 2%	Different protocols: ✓ 1, 3, 7, 14, 21, and 28 days at 20 ºC. Relative humidity 70±5% ✓ 1, 3, 7, 14, 21, and 28 days at 40 ºC. Relative humidity 70±5%	-
(Ojum 2015)	Recycled CAM	Water: 1% Bitumen emulsion: 1.75% to 7% Cement: 0%, 1% and 3%	Different protocols: ✓ 3 days and 28 days at 40 ºC ✓ 21 days and 28 days at 20 ºC ✓ 28 days at 5 ºC	-
(Li *et al.* 2020)	CAM	Water: 1.5% Bitumen emulsion: 6% to 10% Cement: 0%, 2%, 4% and 6%	1 day at room temperature + 3 days at 60 ºC	-
(Chen *et al.* 2021)	Foamed CAM	Water: 4.2% Foamed asphalt: 1% to 3% Cement: 1.5%	Up to 60 h at 60 ºC	-
(Pérez *et al.* 2021)	CIR	Water: 5.75% Residual binder: 1.5% to 2.5%	0, 1, 3, 7, 21 days at 20±2 ºC + induction heating 3 days at 50 ºC	-
(Yang *et al.* 2021)	Cold recycled mixture.	Water: 4.4% Residual binder: 4% Cement: 0%, 1% and 2%	Two protocols: ✓ 2 days at 60 ºC ✓ 3 days at 20 ºC followed by 2 days at 60 ºC	

* Unless otherwise indicated, they are mixtures with bituminous emulsions

Table 3. Characterisation of the quarry aggregates.

Property	Fraction			Specifications (ATEB n.d.)		
	0/2 mm	2/6.3 mm	10/20 mm	T2*	T3*	T4*
Bulk specific gravity (Mg/m^3)	2.77	2.78	2.78	-	-	-
WA_{24} (%)	0.29	0.91	0.66	-	-	-
SE (%)	61	-	-	≥ 45	≥ 40	≥ 35
LA (%)		14.2		≤ 30	≤ 35	
FI (%)		24		≤ 30	≤ 35	
Crushed and broken faces (%)		100		≥ 90	≥ 75	≥ 50

(*) Traffic category T2 or higher refers to a heavy annually averaged daily traffic (AADHT) ≥ 200.
Traffic category T3 refers to 200>AADHT ≥50
Traffic category T4 refers to AADHT<50

Table 4. Selected grain-size distribution for GE-1, according to the lower and upper limits of the granulometric spindle of the ATEB (n.d.).

Sieve size (mm)	Cumulative passing		
	Of the selected GE-1 (%)	**Lower limit of the granulometric spindle (%)**	**Upper limit of the granulometric spindle (%)**
31.5	100.00	100.00	100.00
20	99.74	80.00	100.00
12.5	70.33	66.00	82.00
8	61.47	54.00	69.00
4	53.24	38.00	54.00
2	26.66	26.00	40.00
0.5	13.65	13.00	22.00
0.25	9.02	8.00	16.00
0.125	6.47	5.00	10.00
0.063	4.53	2.00	5.00

Figures

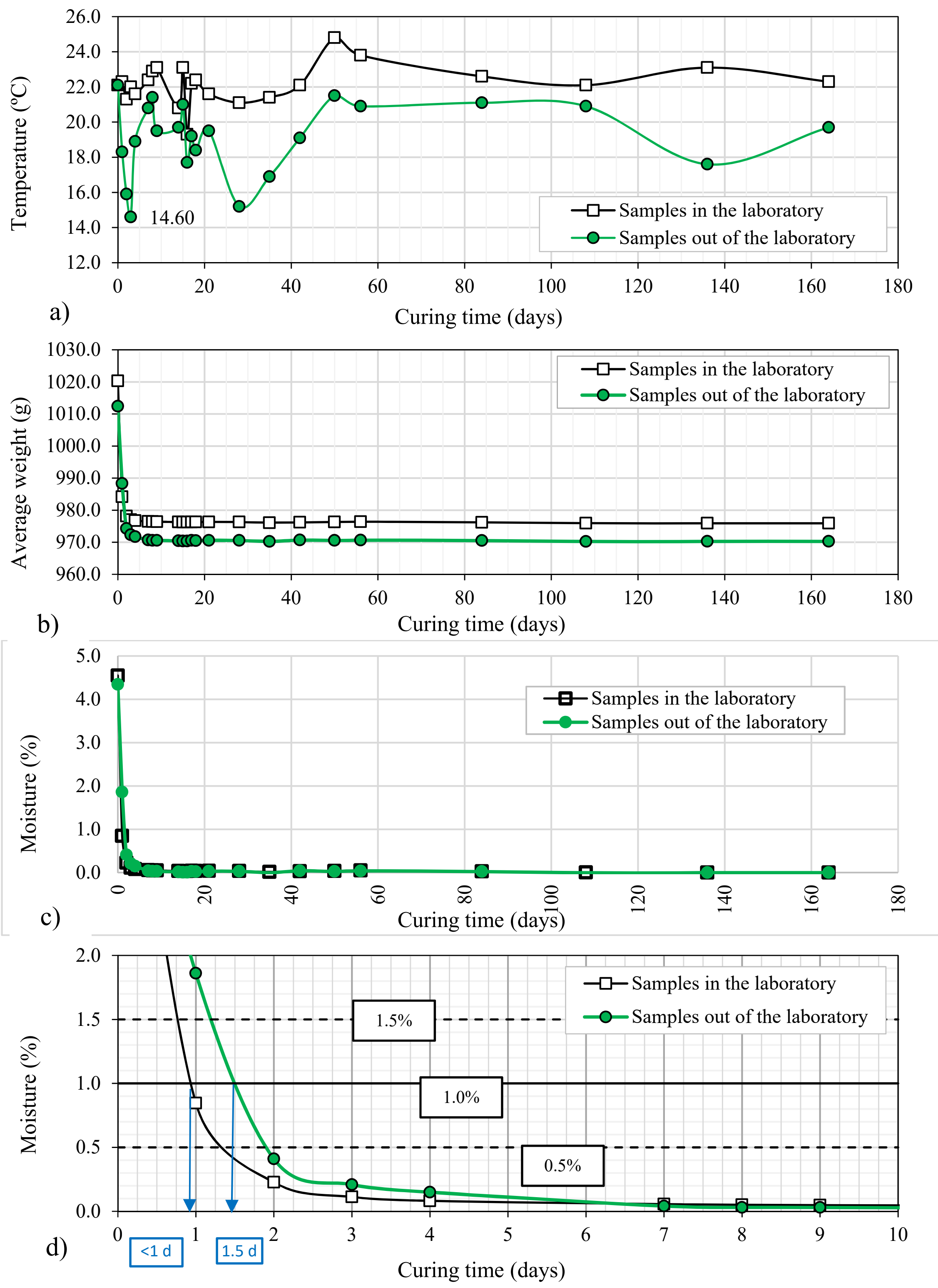
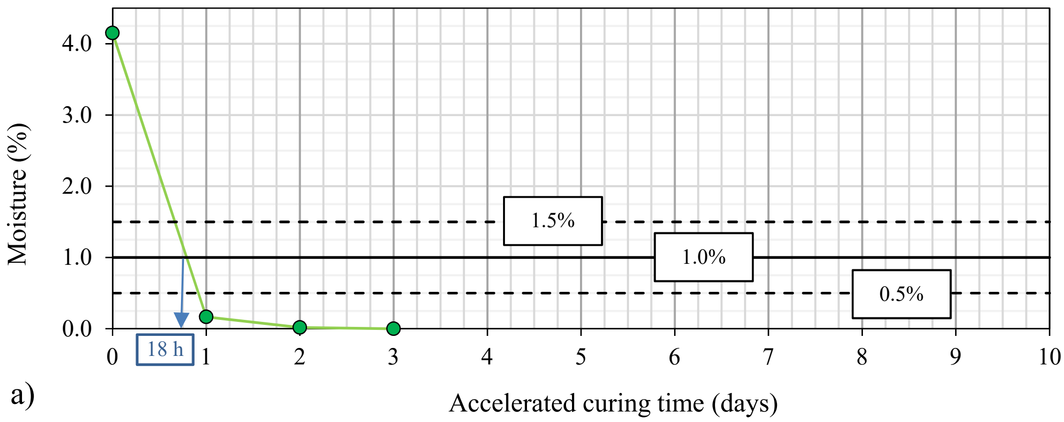


Figure 1. Time evolution of (a) ambient temperature, (b) sample weight, and (c) moisture, for GE-1 samples at their optimal water and bitumen contents, cured inside and outside the laboratory, and (d) detail of the moisture evolution for the first 10 days.



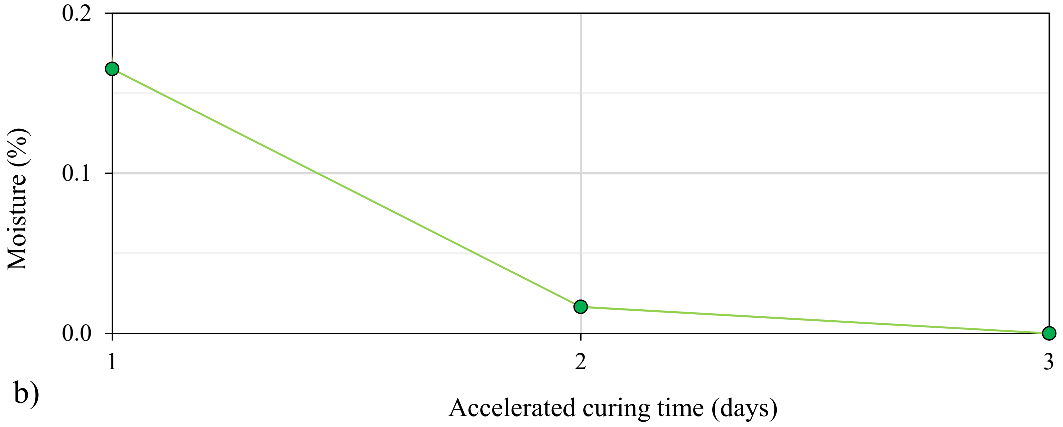
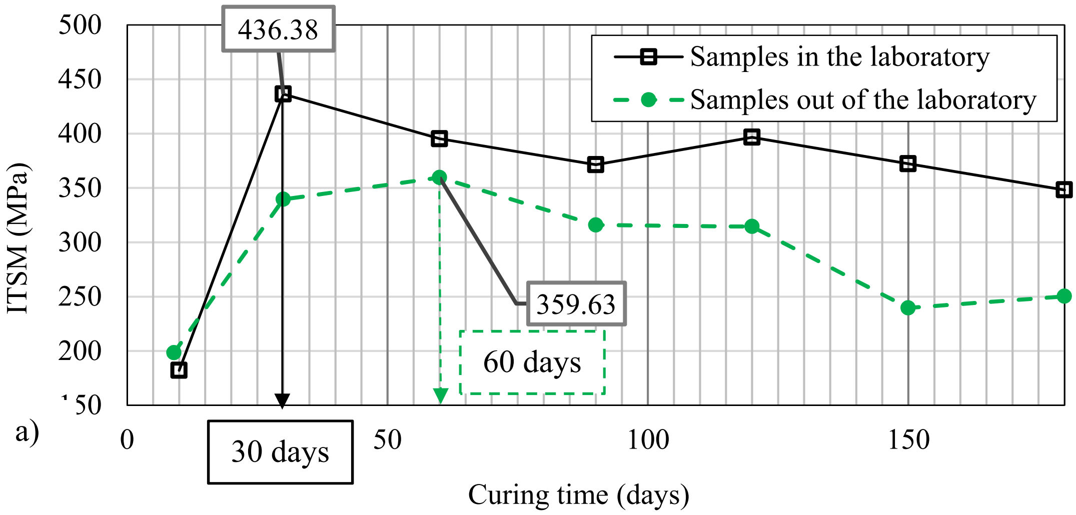
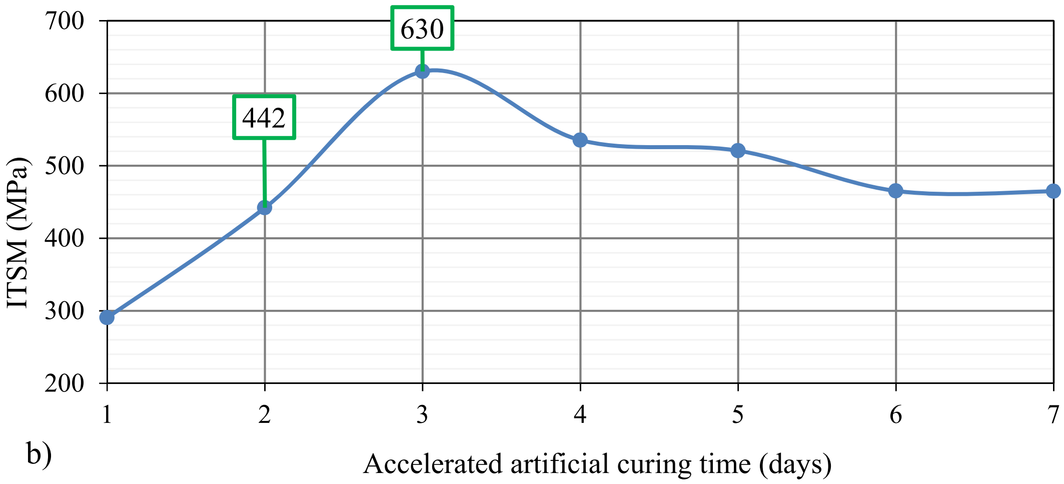


Figure 2. Evolution of the GE-1 moisture as a function of the curing time in the 50 ºC oven: (a) complete and (b) magnified time ranges.



a)



b)

Figure 3. ITSM of the samples subjected to (a) natural curing inside and outside the laboratory, and (b) accelerated curing in the 50 ºC oven.

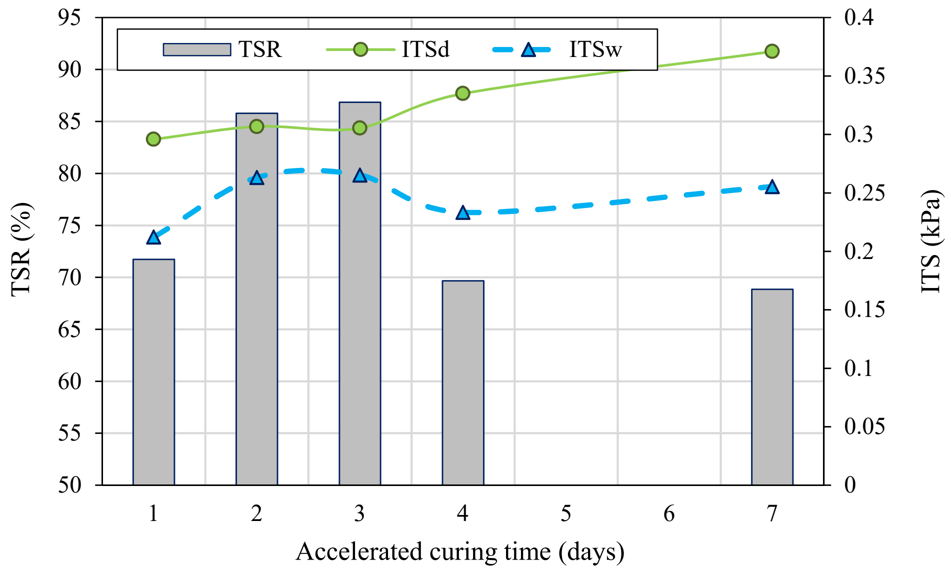


Figure 4. Moisture damage to the samples subjected to accelerated curing in the 50 ºC oven.

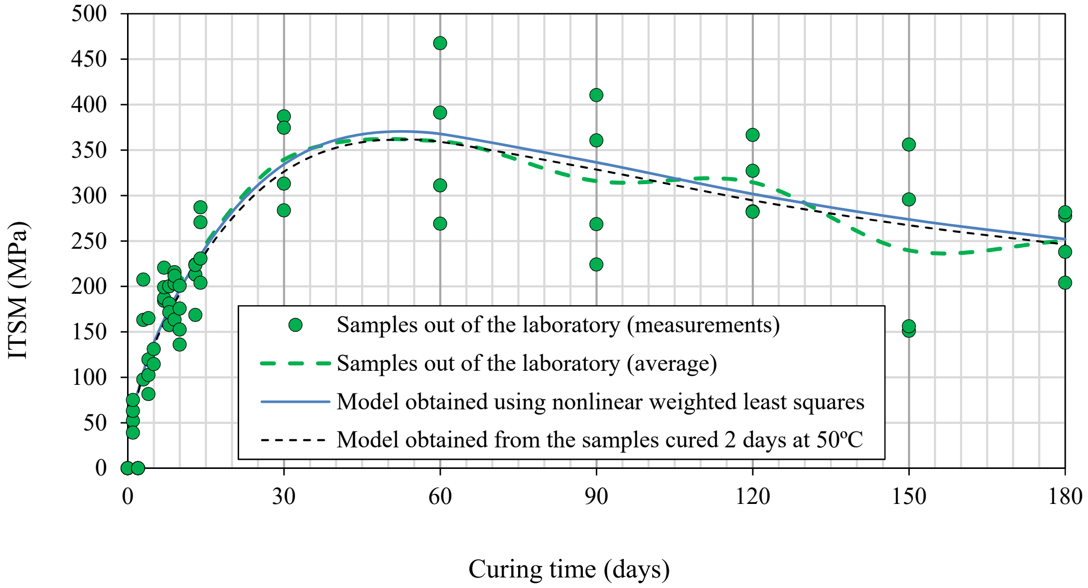


Figure 5. Comparing model predictions and uncontrolled curing results for the ITSM of the GE.

Figure captions

Figure 1. Time evolution of (a) ambient temperature, (b) sample weight, and (c) moisture, for GE-1 samples at their optimal water and bitumen contents, cured inside and outside the laboratory, and (d) detail of the moisture evolution for the first 10 days.

Figure 2. Evolution of the GE-1 moisture as a function of the curing time in the 50 ºC oven: (a) complete and (b) magnified time ranges.

Figure 3. ITSM of the samples: a) subjected to a natural curing in and out of the laboratory and b) subjected to accelerated curing in the oven at 50ºC.

Figure 4. Moisture damage to the samples subjected to accelerated curing in the 50 ºC oven.

Figure 5. Comparing model predictions and uncontrolled curing results for the ITSM of the GE.