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Effect of temperature and relative humidity on cement-bitumen treated materials produced in the laboratory using emulsion or foamed bitumen

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ABSTRACT

Full-depth reclamation (FDR) is an asphalt pavement rehabilitation technique that increases the structural capacity of deteriorated pavements while reducing costs, energy consumption, and resource use, compared to a reconstruction. Despite these advantages, FDR application remains limited, primarily due to gaps in knowledge regarding its design and quality control methods. The mechanical properties of FDR mixtures vary significantly with composition, making an appropriate curing period essential for optimal performance. This study investigates the effects of temperature and relative humidity on emulsion and foamed cement-bitumen treated materials (CBTM) used in FDR applications. Three curing regimes were compared by measuring water loss, indirect tensile strength, and indirect tensile stiffness modulus at 1, 3, 7, and 28 days for both emulsion and foam mixtures. Additionally, emulsion samples underwent extended curing up to 90 days to simulate and assess the long-term effects of climatic variations on material stiffness. Results showed that curing at high temperature and low relative humidity (first regime) produced the highest mechanical properties, while an extended transition from low to high humidity (second regime) reduced performance. Simulating nighttime conditions with 12-hour cycles of high humidity (third regime) delayed mechanical development. For emulsion samples, extended curing at high temperature and low humidity after 28 days did not significantly affect stiffness, while humidity and temperature variations influenced material stiffness. These findings demonstrate CBTM's adaptability to varying environmental conditions, offering practical guidelines to predict long-term performance based on climate, enhance durability, and reduce construction timelines for FDR-treated pavements.

1. Introduction

Full-depth reclamation (FDR) is a pavement rehabilitation technique that pulverizes distressed asphalt and underlying materials to create a stabilized base layer [1]. This technique is cost effective, environmentally sustainable and extends pavement lifespan, particularly when combined with stabilization [2]. Among the Cold Recycled Mixtures (CRM) produced through FDR, Cement-Bitumen Treated Materials (CBTM) are widely used due to their ability to balance stiffness and flexibility, making them resistant to traffic loads and environmental stresses [3].

Despite these advantages, significant gaps remain in understanding the curing process of CBTM, which leads to the development of mechanical properties such as tensile strength and stiffness. Laboratory studies have focused primarily on fixed curing conditions, often neglecting the combined effects of temperature and humidity. Moreover, while both bitumen emulsion and foamed bitumen are commonly used as binders in FDR, limited research directly compares their curing behavior under identical conditions. A review of 151 studies on CRM highlighted this gap, showing that 121 studies used bitumen emulsion, 21 used foamed bitumen, and only 9 considered both stabilizers [4].

This research addresses these gaps by analyzing how curing conditions, involving combinations of temperature and relative humidity, influence the evolution of CBTM's physical and mechanical properties. Conditions were chosen based on Canadian industry practices and climate, providing practical guidelines to predict long-term performance and enhance the durability of FDR-treated pavements for diverse field applications.

The paper is organized as follows: The next section provides a detailed literature review on the FDR technique, its benefits, CBTM curing behavior, and laboratory curing practices. It also presents the volumetric composition of CBTM, a key aspect of the mix design in this study. Section 3 describes the experimental methodology, including material characterization, mix design, sample preparation, curing

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regimes, and testing procedures. Section 4 discusses the results, focusing on the impact of curing conditions on material properties. Finally, Section 5 concludes with key findings and their relevance for field applications.

2. Literature review

FDR is an asphalt pavement rehabilitation process that creates a uniform, upgraded base layer by pulverizing the entire thickness of a distressed asphalt layer along with a portion of the underlying materials, up to depths of 300 mm [1]. This process can be performed through simple in-situ pulverization, blending, grading, and compaction with water as the only additive, or by incorporating aggregates (virgin or recycled) and/or binders to enhance the quality and strength of the stabilized base, all without the addition of any heat [1,2]. Common binders include bitumen emulsion or foamed bitumen for bituminous stabilization and cement or lime for chemical stabilization. These stabilization methods can be combined to optimize pavement performance [2,5].

The benefits of FDR include its cost-effectiveness, as it reuses existing asphalt and base materials to create a new base layer, eliminating the need to transport additional aggregates or dispose of old materials, thereby conserving valuable landfill space [6,7]. FDR also enhances the structural capacity and lifespan of the pavement and with the addition of chemical or bituminous stabilizers and an asphalt overlay, the pavement's lifespan can reach 20 years, equivalent to that of full reconstruction [2,8]. Additionally, FDR allows for roadway geometry corrections, such as adjustments to horizontal and vertical profiles [9,10].A comparative environmental assessment of the FDR technique versus the conventional "Mill and Fill" rehabilitation process for a base layer revealed, using life-cycle assessment (LCA), a notable 51 % reduction in greenhouse gas (GHG) emissions and a significant 64 % decrease in energy consumption when employing FDR [11]. Similarly, another recent study compared three pavement rehabilitation methods: FDR with stabilization, FDR without stabilization, and traditional reconstruction to evaluate their environmental performance across multiple impact categories. Results highlighted significant environmental benefits of FDR with stabilization compared to traditional reconstruction, showing FDR as a better approach for advancing circular economy goals and reducing environmental impacts [12].

During the stabilization process, the binder content can produce a wide range of CRM with varying mechanical behaviors. Among these, CBTM are produced using 1–3 % of bituminous binder, resulting in a less brittle, more flexible material, with the addition of 1–3 % of cement to reduce moisture content and resist early raveling under traffic [13–15]. The combination of the bituminous binder and cement results in mechanical properties characterized by both stiffness and thermal sensitivity [16].

CBTM are characterized by evolving behavior over time due to moisture content changes, resulting from evaporation, emulsion breaking, and cement hydration [17]. Initially, the moisture content corresponds to that chosen during compaction, whether in the field or in the laboratory. Over time, the moisture content in the material changes at varying rates depending on the environmental conditions. This process, known as curing, leads to the development of the material's mechanical properties, such as tensile strength and stiffness [18].

Laboratory curing aims to simulate field curing conditions as closely as possible while accelerating the attainment of long-term properties [19]. However, replicating field conditions in a laboratory setting is challenging due to the variability and complexity of factors influencing field curing, which are difficult to standardize [3]. Therefore several researchers have explored various laboratory curing times, temperatures, and humidity levels to approximate effective field curing simulations. For example, the following conditions were used to represent Italy's typical seasons: summer — 28 days of curing at 40°C; spring and fall — 63 days at 20°C; and winter — 56 days at 5°C [20]. The authors concluded that curing at high temperatures accelerates stiffness development and leads to higher stiffness modulus values, whereas curing at lower temperatures slows the process but does not compromise the potential performance of the mixture. Another study used a curing time of 14 days at three temperatures (25, 40, and 60°C) with both ambient and controlled humidity at 50 % [21]. Their findings highlighted the critical importance of the first 3 days of curing in the material's strength development, showing that higher curing temperatures result in greater mechanical properties. They also observed that the presence of humidity reduced the rate of strength development. Other researchers used a curing duration of 100 days with two temperatures of 25 ± 2°C and 40 ± 2°C and a relative humidity of 70 ± 5 %, considering that high humidity offers a reasonable balance for curing both bituminous stabilizers and cement [22]. Their study showed that higher curing temperatures promote faster development of mechanical properties. Additionally, they noted that water loss during curing had a more pronounced effect on tensile strength evolution, whereas stiffness modulus was more closely linked to cement hydration.

To monitor the evolving behavior of CBTM, several studies [23–25], have shown that evaluating both physical properties (water content) and mechanical properties (tensile strength and stiffness modulus) is effective in assessing laboratory curing regimes.

2.1. Volumetric properties of cement-bitumen treated materials (CBTM)

The volumetric characterization of CBTM is essential in both mix design and quality control, as it affects the material's mechanical performance and durability. In mix design, the selection of compaction methods and energy levels is important, as the resulting density influences both the curing process and the resulting mechanical properties [15]. Additionally, the volumetric composition of CBTM evolves between its fresh state (during mixing and compaction) and its cured state (in service) [26,27]. Therefore, a proper volumetric characterization ensures better control over the compaction process and the quality of the resulting samples.

In CBTM, reclaimed asphalt pavement (RAP) aggregates are not heated, so the aged bitumen from RAP does not act as an active binder, unlike the bitumen coming from the added binders. Therefore, RAP is considered a solid component, also known as "Black rock" [18]. During compaction, the residual bitumen in the emulsion act like a liquid, meaning at this stage, the emulsion with its two phases (residual bitumen and water) contributes to the lubrication of the aggregates, which influences the compaction of the material. However, after breaking, there is a separation between the water and the residual bitumen, causing them to act as distinct materials. For foamed bitumen, its contribution to the lubricating effect of water is minimal because, during mixing, foam bubbles burst and form tiny bitumen splinters. These splinters disperse by adhering to the finer particles and aged bitumen on RAP [13]. Only a small number of bitumen splinters attach to larger virgin aggregate particles during compaction, resulting in localized, non-continuous bonds, or "spot welding" [13,18]. However, since both CBTM (produced with emulsion or foam bitumen) are treated as four-phase composites (with solids (aggregates and cement), bitumen, water, and air), using the same volumetric approach by considering a single liquid phase (foamed bitumen and water) is useful. This approach enables direct comparisons of mechanical properties and performance outcomes under similar conditions, thereby isolating the effect of the bituminous binder type on the material's behavior [18].

In laboratory studies, it is more practical to use the saturated surface dried density (ρ_{SSD}) of the aggregate mix for the volumetric analysis of CBTM, given that the pre-dried aggregates are moistened before compaction [26]. Fig. 1 presents the volumetric composition for both CBTM types used in this study where V_t represents the total volume of the material, V_S represents the volume of solids (cement and aggregate mix in SSD state), and VMA represents the volume of voids of the mineral aggregate, that is the porosity of the aggregate skeleton and which is

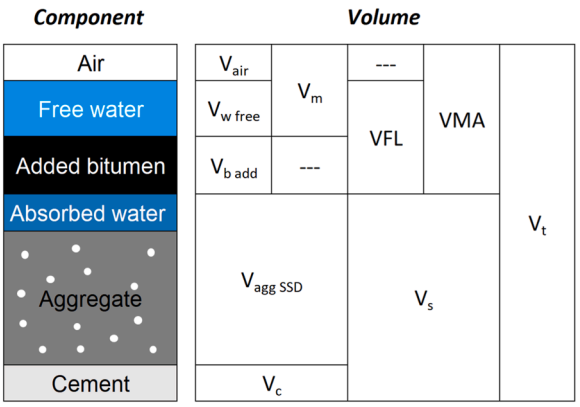

Fig. 1. Volumetric composition of CBTM.

filled by added bitumen, free water and air. It is important to note that the total water content of the material includes the absorbed water and the free water. Free water is provided by the emulsion and the water added to improve workability and compaction for both foamed bitumen and emulsion mixes [15,18].

Based on this information, the following volumetric parameters during compaction can be defined: voids in the material (V_m) and voids filled with liquids (VFL). Voids content (V_m) represents the sum of air voids and free water and it is calculated by the Eq. (1).

$$V_m = \frac{V_{air} + V_{w\ free.}}{V_t} = \frac{V_t - V_{agg\ SSD} - V_c - V_{b\ add}}{V_t} \quad (1)$$

This parameter allows for the optimization of water dosage, bitumen dosage, and compaction energy to achieve a target V_m value [15]. Water and added bitumen occupy a significant volume and act as a liquid during compaction. Therefore, it is essential to consider the voids filled with liquids (VFL), which indicate the level of material saturation and are calculated by the Eq. (2).

$$VFL = \frac{V_{w\ free} + V_{b\ add}}{V_{air} + V_{w\ free} + V_{b\ add}} = \frac{V_{w\ free} + V_{b\ add}}{VMA} = \frac{V_{w\ free} + V_{b\ add}}{V_t - V_{agg\ SSD} - V_c} \quad (2)$$

Previous studies have shown that VFL values higher than 90 % generally imply a visible loss of liquids and fines from the compaction mold, indicating that the actual sample composition may differ from the designed composition [17,22,26]. Therefore, it is important to consider and control both V_m and VFL in CBTM mix design.

3. Experimental program

3.1. Objective and methodology

The objective of this study was to investigate the influence of different curing environments on the physical and mechanical properties of CBTM produced with emulsion or foamed bitumen. Various combinations of temperature and relative humidity were used to simulate three distinct curing conditions in laboratory. A CBTM mix design incorporating 1.5 % cement served as the baseline, using either bitumen emulsion or foamed bitumen to evaluate how each binder type interacts with these conditions.

The three curing regimes, each lasting 28 days, were selected to reflect current industry practices material composition that prevails in Canada, and real field conditions, as detailed in Section 2.2. Additionally, extended curing up to 90 days was applied to emulsion samples to evaluate the effects of prolonged temperature and humidity variations on material stiffness.

Fig. 2 presents an overview of the research methodology followed for the current study. First, preliminary compaction tests were carried out to

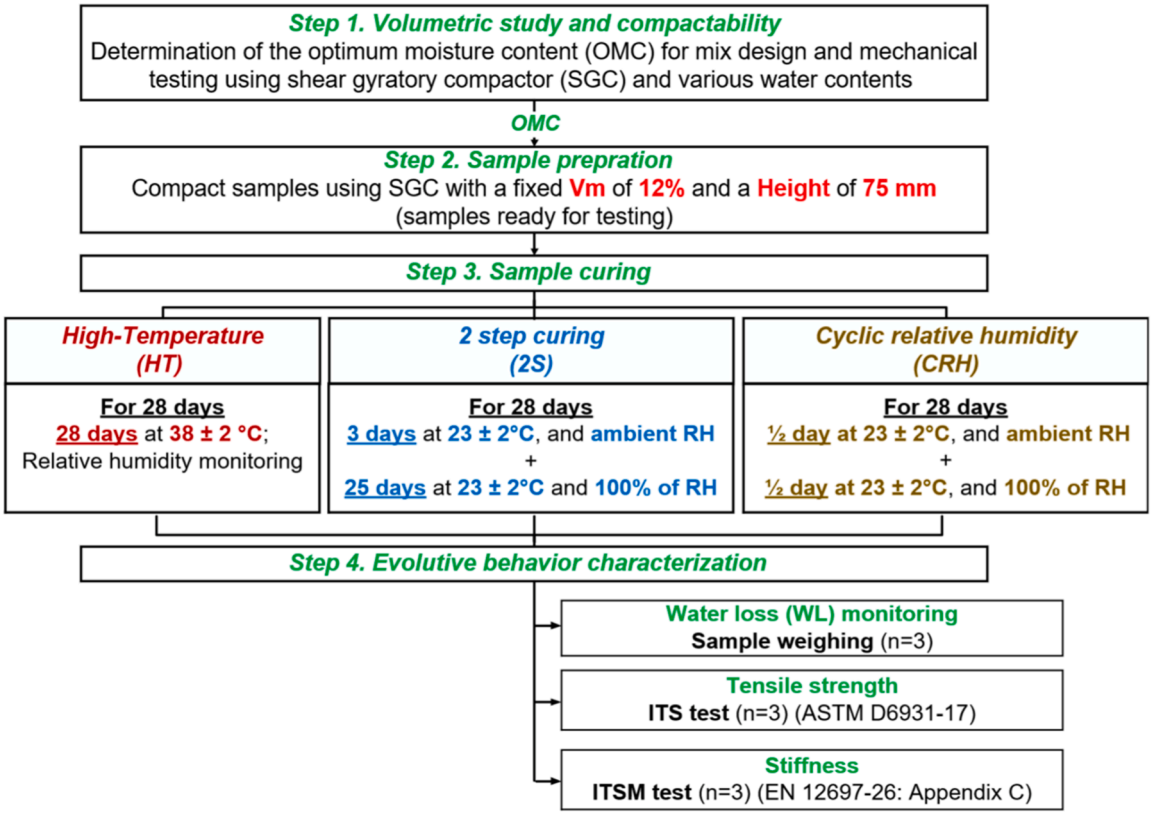

Fig. 2. Overview of the methodology of the study.

verify the compactibility of the mixtures and identify target moisture content for the testing samples based on their volumetric properties. After determining the optimum moisture content (OMC) for the chosen mix design, samples were compacted at a fixed V_m of 12 % and a fixed height of 75 mm. This ensured that the samples were ready for testing after undergoing the chosen curing regime. For each binder type (emulsion or foamed mixtures), three samples were prepared for ITS testing at curing ages of 1 day, 3 days, 7 days, and 28 days, resulting in a total of 36 samples per binder type for the three curing regimes. For ITSM testing, three samples were used per curing regime, resulting in 9 samples per binder type. Combined, a total of 90 samples were prepared and tested. Foamed bitumen samples were demolded immediately after compaction, whereas bitumen emulsion samples were subjected to overnight preliminary curing in the mold at an ambient temperature of 20–23°C. The purpose of this preliminary step was to increase the material's cohesion and prevent breaking during demolding.

3.2. Laboratory curing regimes and conditions

The three laboratory curing regimes used in this study, as shown in Fig. 2, were selected based on current industry practices, material composition, and real field conditions that are observed in Canadian climate. They are described as follows:

1. High temperature curing regime (HT): This regime involved curing at a high temperature of 38°C to accelerate water loss from the material, without any humidity control. In this study, humidity inside the oven was measured/monitored to enable a proper comparison with the second and third curing regimes.
2. Two-step curing regime (2S): The goal of this regime is to provide favorable conditions for each binder (bitumen and cement) by separating the laboratory curing into two distinct steps.

- Step 1 consists of a 3-day cure at low RH (30 %) and a temperature of 23 ± 2°C to facilitate water loss and the cure of the bituminous binder and;
- Step 2 consists of a 25-day cure at high RH (near 100 %) and a temperature of 23 ± 2°C to promote cement hydration.

3. Cyclic relative humidity curing regime (CRH): This regime aims to reproduce real field environmental conditions by simulating day/night cycles through varying RH over 12-hour periods, alternating between low (day: RH of 30 %) and high humidity (night: RH of 100 %). The goal is to evaluate the impact of humidity cycles on the evolution of material properties.

Fig. 3 presents a chart plan of the target temperatures and relative humidity values for each curing regime.

3.3. Materials

CBTM samples for this study were produced in the laboratory using RAP, virgin aggregates (VA), Portland cement (C) and bitumen emulsion or, foamed bitumen. The bitumen emulsion used in this study was a cationic slow-setting emulsion (CSS-1). The foamed bitumen was produced in the laboratory using a PG58S-28 bitumen and a Wirtgen WLB10 foaming device, with target parameters set for an expansion ratio of 10 and a half-life of 6 seconds, considered optimal for foamed bitumen applications. The cement used in the mixtures was a general-use (GU) type (CSA A3000 classification). The main properties of the presented components are summarized in Table 1. Standards and test methods used were from the following organizations: 1) from Quebec province, *Ministère des Transports et de la Mobilité Durable* (MTMD), *Laboratoire des Chaussées* (LC), 2) from Canada, Canadian Standard Association (CSA) and, 3) from United States, American Society for Testing Materials (ASTM).

The added bitumen and cement contents used in the mix design (as a percentage of the dry aggregate mass) were 3.0 % and 1.5 %, respectively. This corresponds to an emulsion dosage of 4.61 % (water + bitumen) and a foamed bitumen content of 3.0 %, resulting in a bitumen-to-cement ratio (B/C) of 2.

The aggregate blend requirements to be met were those for a recycled material of type MR-5 according to Quebec's provincial standard BNQ 2560–600 (*Bureau de Normalisation du Québec*). The recycled material was composed of 35 % RAP, 60 % coarse VA and 5 % of fine VA. Table 2 shows the results of the sieve analysis performed on the aggregates used, as well as the grading of the aggregate blend and the required specifications.

3.4. Mix design and sample preparation

For emulsion samples, the total water content (W_{tot}) was composed by water from the emulsion (W_{em}) and additional water (W_{add}), added in two phases. The first part (W_{abs}) corresponded to the water absorption of the aggregate blend, which was added to the dry aggregate blend. The wet aggregate blend was then sealed in a plastic bag for a minimum of 12 hours at room temperature to ensure homogeneous moisture conditions and allow for absorption by the aggregate. Next, the remaining water and the binders were added in the following order: Portland cement, remaining water to reach the target water content and finally bitumen emulsion. After each addition, the blend was mixed manually until it had a uniform appearance. The entire mixing process required five to eight minutes, starting with the addition of cement.

Preliminary compaction tests were carried out using a shear gyratory compactor (SGC) and following this protocol: 150 mm diameter mold, constant pressure of 600 kPa, gyration rate of 30 rpm, and an inclination angle of 1.16°. The objective was to identify the water content to be used for the preparation, curing and mechanical testing of samples. The procedure was based on the volumetric properties of CBTM, compacted at a fixed number of gyrations of 180 gyrations. Sample height was

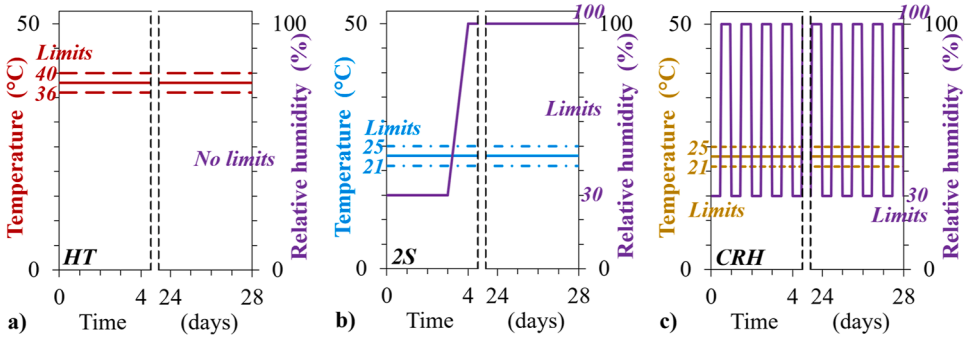

Fig. 3. Chart plan of target temperatures and relative humidity for the: a) HT regime; b) 2S regime; c) CRH regime.

Table 1
Properties of CBTM components.

Component		Property	Unit	Value	Standard or test method
Virgin aggregate (VA)	Coarse	Grading	mm	0–20[a]	MTMD 2101
		Bulk Density	—	2.655	LC 21–065, −066 & −067
		Apparent Density	—	2.744	LC 21–065, −066 & −067
		Absorption	%	1.21	LC 21–065, −066 & −067
	Fine	Grading	mm	0–5[a]	MTMD 2101
		Bulk Density	—	2.628	LC 21–065, −066 & −067
		Apparent Density	—	2.742	LC 21–065, −066 & −067
		Absorption	%	1.57	LC 21–065, −066 & −067
Reclaimed asphalt pavement (RAP)		Grading	mm	0–10[a]	MTMD 2101
		Bitumen content	% mass	4.6	LC 26–006
		Bulk Density	—	2.381	LC 21–065, −066 & −067
		Apparent Density	—	2.492	LC 21–065, −066 & −067
		Absorption	%	1.88	LC 21–065, −066 & −067
Bitumen emulsion		Type	Letters	CSS−1[b]	ASTM D2397
		Bitumen grade	PG Hn-L[c]	58S—28	MTMD 4101
(BE)		Bitumen content	% mass	65.1	ASTM D6997
Foam bitumen (FB)		Bitumen grade	PG Hn-L[c]	58S—28	MTMD 4101
		Temperature	°C	175	—
		Cold water added[d]	% mass	4	—
Cement (C)		Use	—	General	CSA A3000
		f'c[e]	MPa	43.9	ASTM C109

[a] Nominal Maximum Aggregate Size (NMAS).
[b] Cationic Slow Setting with Low viscosity.
[c] Performance Grade defined by High temperature, Traffic level and Low temperature.
[d] Added to hot bitumen during foam production (% by weight of bitumen).
[e] Compressive strength at 28-day.

Table 2
Grading distribution of aggregates and blend, and required grading specifications.

Sieve	Passing at each sieve (%)				
(mm)	RAP 0–10	VA 0–20	VA 0–5	Blend	Specifications*
40	100	100	100	100	100
31.5	100	100	100	100	—
28	100	100	100	100	80–100
14	100	73	100	83.8	50–90
5	65	36	97	49.2	25–55
0.315	6	11	20	9.7	5–20
0.080	0.6	8.8	12	6.1	3–10

* Specifications according to « *Ministère des Transports et de la Mobilité durable (MTMD)* » in Quebec for FDR work [28].

recorded at each gyration, during the compaction allowing for the monitoring of V_m and VFL. As the number of gyrations increases, V_m decrease while VFL increase until it reaches 100 %, which signifies the theoretical saturation condition of the sample. However, visual confirmation showed that when VFL approaches 90 %, there is a loss of material (including water, bitumen droplets, and fines) from the mold. These results led to choosing $W_{tot} = 3.07$ % as the optimum moisture content (OMC) for the mix design, meaning that the free water during the mixing process corresponds to that coming from the emulsion.

Similar to emulsion, foam samples were designed with the same volumetric composition considering that foamed bitumen does not include water and consequently, the free water (water coming from emulsion in the case emulsion mix) is represented only by the water added in the mixture. First, water corresponding to the aggregate absorption was added to the batch, then sealed in a plastic bag for at least 12 hours at room temperature. A twin-shaft pug-mill type mixer was used as it provides a mixture quality similar to site mixes [13] and allows easy connection to the foaming device for direct discharge of the foam into the mixer. The mixing process followed this sequence: wet aggregates and cement were added to the mixer and blended for 60 seconds. While mixing, the remaining water was added to ensure a good distribution throughout the aggregate blend. Finally, the foam was discharged directly into the mixer, and the material was mixed for an additional 30–60 seconds [29]. Both emulsion and foamed samples compacted at a fixed V_m content of 12 % reached a VFL value of 52 %, indicating that they were far from saturation.

3.5. Water loss monitoring and mechanical testing program

The ITS 28-day series was used to monitor water loss (WL), following the demolding of the samples for each curing regime. The goal was to evaluate how different curing conditions influence water content in CBTM materials. For the HT and 2S regimes, samples were weighed on days 1, 3, 7, and 28 to measure their water content. For the CRH regime, samples were weighed every 12 hours, simulating daily cycles, over 28 days. The WL was calculated using Eq. (3).

$$WL(\%) = \frac{W_0 - W_i}{W_0} \quad (3)$$

where WL represents the water loss after i curing days, W_0 is the initial moisture content of the sample after demolding, and W_i is the moisture content of the sample after i curing days, calculated with Eq. (4).

$$W_i(\%) = W_0 - \frac{M_0 - M_i}{M_0} \quad (4)$$

where M_0 is the initial mass of the sample after demolding, and M_i is the mass of the sample after i curing days.

3.5.1. Indirect tensile strength (ITS)

The ITS test was performed according to ASTM D6931, after conditioning the samples at 25°C. For the HT regime, samples were placed in a closed oven for a minimum of 4 hours. For the 2S and CRH regimes, samples were placed in a leak-proof plastic bag and then in a water bath for a minimum of 2 hours. The test involves loading the sample across its vertical diametral plane and recording the peak load at failure, which is then used to calculate the ITS strength of the sample according to Eq. (5).

$$ITS(kPa) = \frac{2000 \times P(N)}{\pi \times t(mm) \times D(mm)} \quad (5)$$

where ITS is the tensile strength of the sample, P is the maximum compressive load, t is the sample thickness (or height) before testing and D is the sample diameter.

3.5.2. Indirect tensile stiffness modulus (ITSM)

To measure the stiffness of the samples after curing, an indirect tensile stiffness modulus (ITSM) test was performed using an MTS device based on the standard EN 12697–26 (Annex C). The test was conducted after conditioning the samples at 25°C in a closed oven for a minimum of 4 hours. The test involves measuring the average stiffness modulus after applying 5 pulses with a rise time of 124 ± 4 ms and a pulse repetition period of 3.0 seconds. For each pulse, the stiffness modulus is obtained by Eq. (6).

$$ITSM = \frac{F(N) \times (0.27 + \nu)}{z(mm) \times h(mm)} \quad (6)$$

where F is the peak load of the applied repeated pulse, z is the amplitude of the horizontal deformation, h is the height (or thickness) of the sample tested and ν is the Poisson's ratio (assumed as 0.35). For each sample, the test was repeated along the vertical and horizontal diameters, and the final stiffness modulus value is the average of both measurements.

4. Results and analysis

4.1. Recorded temperature and relative humidity

Temperature and RH were recorded using a USB data logger, with data sampled every hour throughout the curing process. Fig. 4 presents the daily averages of temperature and RH for the HT and 2S curing regimes, as well as the recorded hourly temperature and RH values for the CRH regime. These measurements were taken from the demolding of the samples until day 28 of curing.

As shown at Fig. 4a, for the HT curing regime, temperature was consistently maintained at 38°C ± 2°C, and the RH remained very low, with an average of 17 % over the entire curing period. For the 2S curing regime (Fig. 4b), the samples were cured in a closed environmental chamber at ambient relative humidity and a temperature of 23 ± 1°C for the first 3 days. Following this, the samples were transferred to a humid chamber with a fixed RH of 100 % and a temperature of 23 ± 1°C for the next 25 days. The lowest RH recorded during the first 3 days was 41 %* , and the highest was 50 %* , which is slightly higher than the target values (*measured value per hour, not averaged daily). For the CRH regime the lowest and highest ambient RH recorded were 36 % and 58 %, respectively.

4.2. Water loss monitoring from 1 to 28 days

The evolution of water loss in both materials throughout the studied curing regimes is presented Fig. 5. The values shown represent the average weight of three samples for each regime. For the HT curing regime, we can see that after just one day of curing, water loss values are 48 % for emulsion mixtures and 56 % for foam. The rate of water loss decreases over time as water content starts approaching an equilibrium state, by day 7 for both emulsion and foam. By day 28, water loss reached 74 % for emulsion and 79 % for foam. Overall, it appears that water loss is similar for both emulsion and foam when subjected to high temperature curing.

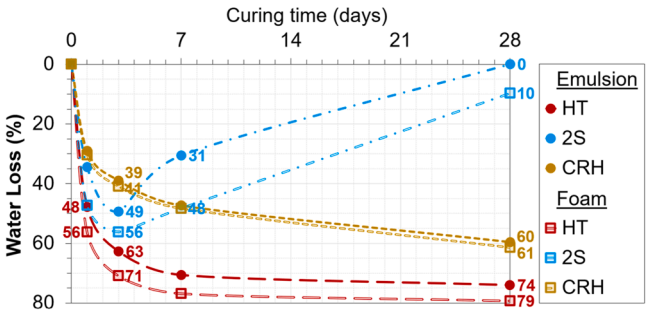

Fig. 5. Evolution of water loss for emulsion and foam mixtures according to the studied curing regimes.

In the first step of the 2S curing regime (3 days at 23°C and ambient RH), water loss values are 49 % for emulsion and 56 % for foam. These values after 3 days of curing are very similar to those observed after 1 day for HT, confirming that a higher temperature in the case of HT has the effect of accelerating water loss. After transferring the samples to the humid chamber at 100 % RH, a reverse trend was observed, with water content increasing instead of continuing to decrease. By day 7, water loss compared to the initial values was reduced to 31 % for emulsion and 48 % for foam. By day 28, emulsion mixtures not only recovered all the water lost but also surpassed its initial water content, indicating that no water loss occurred. Similarly, foam reached 10 % WL by day 28, indicating near-complete water content recovery. Therefore, no equilibrium

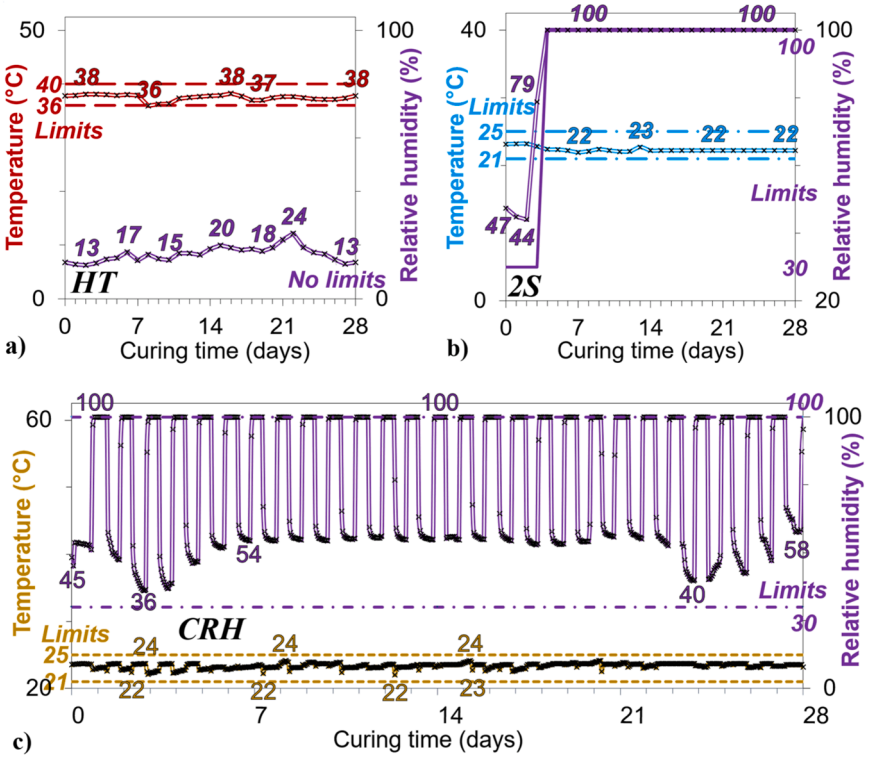

Fig. 4. Recording of temperature and relative humidity for 28 days: a) HT curing regime (daily average values); b) 2S curing regime (daily average values); c) CRH curing regime (recorded values per hour).

state was observed for this curing regime.

For the CRH regime water loss evolution was relatively similar for emulsion and foam. The use of alternating humidity cycles (12 hours at 30 % and 12 hours at 100 %) revealed that CBTM materials tend to lose water faster in dry environments than they regain it in humid environments. The rate of water loss for this curing regime seems similar to HT, but the amplitude of water loss is much smaller. Moreover, this curing regime did not allow an equilibrium state to be reached after 28 days.

4.3. Indirect tensile strength results from 1 to 28 days

Fig. 6 illustrates the evolution of ITS values for emulsion and foam mixtures under the studied curing regimes. Each series represents the average of three tested samples, with error bars indicating the standard deviation. The coefficient of variation for all results remained within 16 %.

Curing under high temperature (HT) with uncontrolled RH produced the highest ITS values for both emulsion and foam mixtures (Fig. 6). During the early stages (1 and 3 days), emulsion mixtures showed lower ITS values than foam, with 213 and 311 kPa for emulsion compared to 260 and 344 kPa for foam, respectively. By day 7, both materials achieved comparable results, with emulsion at 428 kPa and foam at 432 kPa. However, after 28 days, emulsion mixtures surpassed foam, recording ITS values of 554 and 489 kPa, respectively. This suggests that, when cured at high temperatures, mixes made with foamed bitumen have a higher short-term tensile strength than mixes with emulsion, while in the long term, mixes with emulsion have the highest tensile strength.

In the 2S curing regime, ITS values showed progress in the first step (3 days), with emulsion increasing from 182 kPa at day 1–248 kPa at day 3 and foam from 210 kPa at day 1–278 kPa at day 3. However, transferring the samples to a 100 % RH (second step) notably affected both materials. For emulsion mixtures, this transition led to a slight decrease in ITS values to 228 kPa at day 7, while foam tensile strength continued to increase at a slower rate, reaching 317 kPa. After 28 days, foam mixtures showed a drop to 259 kPa, whereas emulsion showed minimal change reaching a value of 239 kPa. This indicates that curing in a high RH environment tends to slow or even reduce the evolution of tensile strength over time.

The CRH regime showed the slowest development of tensile strength in comparison with HT and 2S. On day 1, emulsion mixtures had a value of 149 kPa, and foam was higher at 184 kPa. By day 3, the ITS values of emulsion (209 kPa) and foam (256 kPa) mixtures approached those of the HT regime on day 1, indicating a delayed tensile strength progression. By day 28, emulsion reached 362 kPa, while foam reached 436 kPa, confirming that although cyclic humidity delays strength development in comparison with high temperature and low relative humidity (HT regime), it does not prevent long-term tensile strength growth. Moreover, the ITS results show that the impact of cyclic exposure to humidity variations is less pronounced for foam mixes than for emulsion mixes. Indeed, at day 28, the ITS values for the emulsion mix were 554 and 362 kPa, with a difference of 192 kPa under the HT and CRH cures, respectively. For foam, the ITS values were 489 and 436 kPa, under HT and CRH cures, respectively, with a difference of 53 kPa. This suggests that foamed bitumen mixes are less sensitive to moisture variations than emulsion mixes in terms of tensile strength development.

4.4. Indirect tensile stiffness modulus results

4.4.1. Curing time: 1–28 days

Fig. 7 illustrates the evolution of ITSM values for both emulsion and foam mixtures from 1–28 days under the studied curing regimes. Each series represents the average of the three tested samples, with error bars indicating the standard deviation. The coefficient of variation generally remaining within 25 %. Exceptions include emulsion mixtures cured for 28 days under the 2S regime (40 %), where a sample's plastic wrap was slightly torn, resulting in increased humidity exposure, and foam mixtures cured for 1 day under the HT regime, which had a coefficient of variation of 27 %.

In Fig. 7, results show that similar to tensile strength values, the HT-curing regime resulted in the highest ITSM values. However, stiffness modulus values for foam mixtures were higher than those for emulsion throughout the 28 days of curing, reaching 3707 MPa, while emulsion reached 3643 MPa by day 28. For the 2S curing regime, stiffness modulus increased during the first step (3 days), with foam displaying higher values than emulsion. After transitioning to high RH conditions, stiffness development slowed down, emulsion's stiffness modulus increased from 2039 MPa on day 3–2171 MPa on day 7, while foam's stiffness modulus increased from 2067 MPa to 2262 MPa. By day 28, stiffness modulus values had decreased to 1874 MPa for emulsion and 2096 MPa for foam, indicating that prolonged curing in high RH conditions (2nd step of 2S regime) reduces material stiffness. These results once again, show that foam mixtures seem to be less sensible to moisture exposition compared to emulsion mixtures. Under the CRH regime, stiffness development was slower compared to both the HT and early stages of the 2S regime, as the use of cyclic humidity delayed the progression of both indirect tensile strength (Fig. 6) and stiffness modulus (Fig. 7). However, stiffness modulus continued to increase over time, with emulsion mixtures reaching 2289 MPa and foam reaching 2638 MPa by day 28.

4.4.2. Curing time: 28–90 days for emulsion mixtures

To further investigate the effect of temperature and relative humidity on long-term stiffness, emulsion samples underwent extended curing up to 90 days under different conditions presented in Table 3.

For HT curing set of samples, the same curing parameters (38°C and low relative humidity) were maintained for up to 90 days. The aim was to verify the effect of exposure to elevated temperature on the development of stiffness modulus over a long period of time. Stiffness modulus was assessed after 56 and 90 days. For 2S and CRH curing set of samples, after exposure to 100 % RH, the samples were subjected to exposure to ambient humidity and temperature of 23°C for a period of

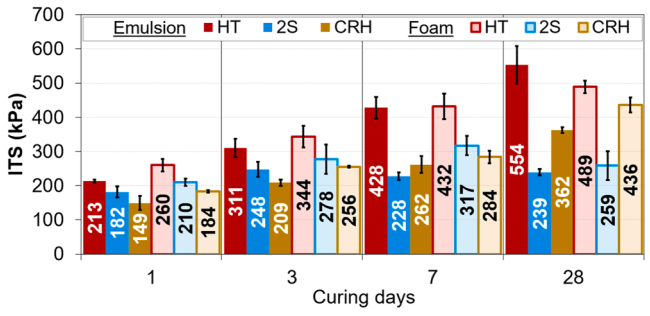

Fig. 6. Evolution of ITS for emulsion and foam mixtures according to the 3 curing regimes from 1 to 28 days.

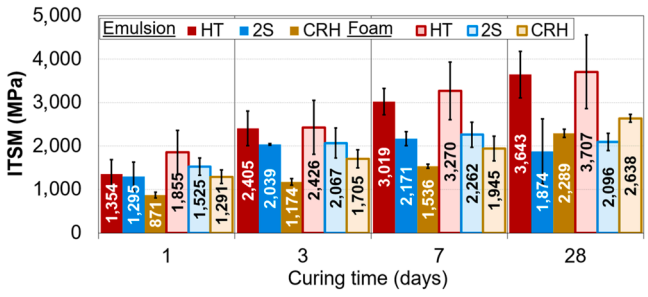

Fig. 7. Evolution of ITSM for emulsion and foam mixtures according to the 3 curing regimes from 1 day to 28 days.

Table 3
Extended curing conditions for emulsion mixtures.

Curing time (days)	Curing regime		
	HT	2S	CRH
28–56	38 ± 2°C in the oven	T = 23 ± 1°C + ambient RH	T = 23 ± 1°C + ambient RH
56–70	38 ± 2°C in the oven	T = 23 ± 1°C + 100 % RH	T = 23 ± 1°C + 100 % RH
70–90	38 ± 2°C in the oven	38 ± 2°C in the oven	38 ± 2°C in the oven

28 days, the stiffness modulus was then evaluated at 56 days. The samples were then returned to an environment with high relative humidity (100 %) for a period of 14 days (total of 70 days). The stiffness modulus was once again evaluated. Finally, the samples were placed in an oven at 38°C and low relative humidity for a period of 20 days, after which the final stiffness modulus was evaluated (total 90 days). The aim was to verify the effect of prolonged exposure to various temperature and humidity conditions on stiffness modulus development. Emulsion-treated mixes were chosen for this portion of the study as they showed higher sensitivity to humidity. Extended curing results of emulsion mixtures are presented in Fig. 8.

Initially, for HT curing, prolonged exposure to high temperature and low relative humidity for a further 62 days (28–90 days) did not appear to have any effect on stiffness modulus evolution (Fig. 8). Indeed, the modulus values measured after 56 and 90 days are very similar to the values measured at 28 days. These results suggest that exposure to high temperature and low relative humidity for 28 days enables the ultimate stiffness modulus of the material to be measured. For the 2S curing regime, where samples were transferred from 100 % RH (28 days) to ambient RH (56 days), there was a significant increase in stiffness caused by changes in humidity exposure. ITSM values reached 3625 MPa by day 56, which is similar to the stiffness observed under HT conditions. This suggests that material stiffness temporarily decreases when subjected to high RH environments, but regains and even surpasses its initial values once transferred to drier conditions. However, continued curing in high RH for another 14 days caused a notable reduction in ITSM, with values dropping to 2398 MPa by day 70, confirming the material's sensitivity to relative humidity. Further curing at 38°C for an additional 20 days led to a stiffness increase, as ITSM values reached 3479 MPa by day 90, showing that stiffness loss in high RH is reversible when reintroduced to higher temperatures or drier environments. Samples under the CRH regime were subjected to the same alternating temperature and RH curing conditions presented in Table 3. ITSM increased from 2289 MPa on day 28–2868 MPa after 28 days of ambient RH (Fig. 8). However, like in the 2S regime, a 14-day curing period at 100 % RH resulted in a decrease in stiffness, with ITSM values dropping to 2280 MPa by day 70. Once cured at 38°C for another 20 days, stiffness showed a significant increase, reaching 3335 MPa by day 90, confirming that the long-term stiffness of CBTM varies significantly depending on climatic conditions the materials are exposed to.

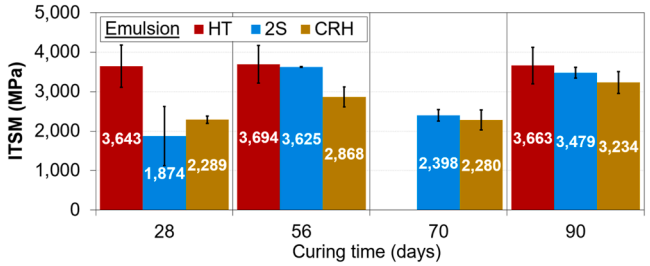

Fig. 8. Extended curing results of ITSM for emulsion mixtures.

4.5. Correlation between ITS and ITSM results from 1 to 28 days

To better understand the effect of curing conditions on the development of indirect tensile strength and indirect stiffness modulus, examining their correlation provides a more comprehensive understanding of CBTM's mechanical performance. Fig. 9 shows a strong linear correlation between ITSM and ITS for both materials under the HT and CRH regimes. This linear relationship suggests that, for these curing regimes, there is a steady progression in both tensile strength and stiffness modulus over time, but according to a different rate of growth. However, under the 2S regime, where extended exposure to 100 % RH occurs, the development of ITS and ITSM values diverges. This results in a slower growth or even a decrease in mechanical properties, causing deviations from the regression line and leading to lower coefficients of determination (R^2) for both foam and emulsion. These observations indicate that while the HT and CRH regimes maintain a unique linear relationship between indirect tensile strength and indirect stiffness evolution, this correlation does not hold for the 2S regime. Mechanical properties in the 2S regime are highly influenced by the amount of moisture absorbed (Fig. 5) and the duration of exposure to high RH, leading to the observed variations in both ITS and ITSM values.

4.6. Influence of curing time on ITS and ITSM values

A statistical study was conducted to evaluate the impact of curing time on ITS and ITSM values from 1 to 28 days, following the same approach. A one-way ANOVA was performed for each curing regime and type of binder separately. Table 4 presents the results from the one-way ANOVA results for 1–28 days comparison. The results revealed significant differences for both ITS and ITSM values across curing times (F-value > F-crit and p-value < 0.05). This means that for all the curing combinations evaluated, the results obtained after 28 days are significantly different from those evaluated after 1 day. A 28-day laboratory cure is therefore appropriate for assessing the effect of temperature and/or humidity on indirect tensile strength and indirect stiffness modulus development regardless of the type of curing involved.

Following this, pairwise comparisons using t-tests with Bonferroni correction were conducted to identify which specific curing times differed. This post hoc test is applied only after an initial analysis indicates statistically significant differences, to determine where those differences lie. The Bonferroni correction is a statistical method used to adjust the significance level when performing multiple comparisons in a dataset. Without this adjustment, conducting multiple hypothesis tests increases the probability of detecting false positives meaning that results that appear significant purely by chance (a Type I error). To address this, the Bonferroni correction reduces the threshold for statistical significance by dividing the original significance level ($\alpha = 0.05$) by the number of comparisons [30]. In this study, the significance level was adjusted to $\alpha = 0.05/3$ where 3 is the number of comparisons conducted per curing regime. The detailed results of the statistical tests are shown in Table 5.

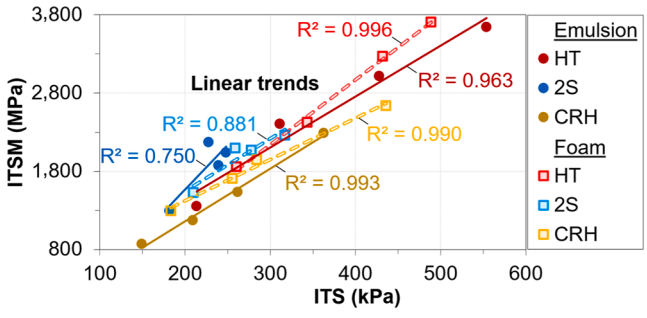

Fig. 9. Correlation between ITSM and ITS according to the studied curing regimes for emulsion and foam mixtures.

Table 4
One-way ANOVA results for ITS and ITSM values from 1–28 days for each mixture (significance level = 0.05).

Curing regime	ITS values					
	Emulsion			Foam		
	F-Value	p-Value	F-Crit	F-Value	p-Value	F-Crit
HT	55.543	0.000	4.066	40.055	0.000	4.066
2S	11.122	0.003	4.066	5.261	0.026	4.066
CRH	83.972	0.000	4.066	160.549	0.000	4.066
Curing regime	ITSM values					
	Emulsion			Foam		
	F-Value	p-Value	F-Crit	F-Value	p-Value	F-Crit
HT	34.574	0.000	3.098	10.997	0.000	3.098
2S	9.445	0.000	3.098	8.730	0.001	3.098
CRH	132.723	0.000	3.098	34.445	0.000	3.098

Table 5
Pairwise Comparisons for ITS and ITSM values from 1 day to 28 days (adjusted significance level = 0.017).

Curing regime and number of days	ITS values			
	Emulsion		Foam	
HT	p-Value	Significance	p-Value	Significance
1D vs 3D	0.003	True	0.016	True
3D vs 7D	0.008	True	**0.034**	**False**
7D vs 28D	**0.025**	**False**	**0.076**	**False**
2S	p-Value	Significance	p-Value	Significance
1D vs 3D	0.013	True	**0.057**	**False**
3D vs 7D	**0.221**	**False**	**0.253**	**False**
7D vs 28D	**0.244**	**False**	**0.117**	**False**
CRH	p-Value	Significance	p-Value	Significance
1D vs 3D	0.010	True	0.000	True
3D vs 7D	**0.025**	**False**	**0.056**	**False**
7D vs 28D	0.003	True	0.001	True
Curing regime and number of days	ITSM values			
	Emulsion		Foam	
HT	p-Value	Significance	p-Value	Significance
1D vs 3D	0.000	True	**0.085**	**False**
3D vs 7D	0.013	True	**0.032**	**False**
7D vs 28D	**0.037**	**False**	**0.304**	**False**
2S	p-Value	Significance	p-Value	Significance
1D vs 3D	0.001	True	0.010	True
3D vs 7D	**0.185**	**False**	**0.278**	**False**
7D vs 28D	**0.167**	**False**	**0.237**	**False**
CRH	p-Value	Significance	p-Value	Significance
1D vs 3D	0.000	True	0.005	True
3D vs 7D	0.000	True	0.173	**False**
7D vs 28D	0.000	True	0.001	True

First, the results show that for the HT regime, significant differences in ITS and ITSM were observed between 1 and 3 days, as well as between 3 and 7 days for emulsion, with no significant differences between 7 and 28 days (Table 5). This indicates that emulsion mixtures achieved most of its indirect tensile strength and indirect tensile stiffness modulus gain after 7 days. For foam, a significant difference in ITS was observed only between 1 and 3 days, with no significant differences between other curing times for both ITS and ITSM, suggesting a rapid gain in strength and stiffness in early curing stages, with slower progression afterward. Overall, the results of the statistical analysis show that the effect of exposure to high temperature and low humidity leads to rapid development of the material's properties, and that this effect is even more marked for mixtures with foam.

Under the 2S regime, significant differences were observed between 1 day and 3 days for ITS and ITSM values of emulsion and for ITSM values of foam, while no significant differences were seen for other curing times (Table 5). This confirms the negative effect of prolonged exposure to high relative humidity on the development of the material properties.

For the CRH regime, no significant differences were observed between 3 and 7 days for ITS values of emulsion and for ITS and ITSM values of foam, but significant differences were observed between 7 and 28 days, suggesting a slower gain in indirect tensile strength and indirect tensile stiffness modulus during this intermediate period for both emulsion and foam.

For the extended curing results, the evaluation of the influence of extended curing time on ITSM values was conducted only for the HT regime, as the conditions (temperature and RH) vary significantly across the different curing durations in the extended 2S and CRH regimes. This variation means that changes in ITSM could result from a combined effect of time and changing environmental conditions, rather than curing time alone. The statistical study, performed using a one-way ANOVA (Table 6), confirmed that there is no significant impact of extended curing time on ITSM values within the HT regime. This suggest that evaluation of the stiffness modulus at 28 days following exposure to high temperature and low relative humidity reveals what appear to be the ultimate properties of the material.

5. Conclusion

The present study aimed to evaluate the effect of temperature, relative humidity and curing time on cement-bitumen treated materials (CBTM) produced with bitumen emulsion and with foamed bitumen, characterized by the same volumetric composition. The evolution of physical and mechanical properties of CBTM during curing were monitored in order to evaluate the effect of curing environments and the impact of changing the bituminous binder in the mix design. Additionally, emulsion samples underwent extended curing up to 90 days under different conditions to evaluate the impact of long-term temperature and relative humidity variations on material stiffness. The main findings are presented in the following paragraphs.

Elevated curing temperatures and low relative humidity (curing regime HT: 38°C and an average of 17 % of RH) accelerates water evaporation in the material, while high relative humidity (100 %: curing regimes 2S and CRH) slows evaporation process and can increase water content. Foamed bitumen mixtures lose water faster than emulsion mixtures, reaching equilibrium more quickly under high-temperature and/or dry environment.

The indirect tensile strength (ITS) and indirect tensile stiffness modulus (ITSM) results show that foam bitumen mixtures achieve higher short-term tensile strength and stiffness modulus under high-temperature and low-humidity curing compared to emulsion mixtures. However, long term properties (ITS and ITSM) converge for both mixtures. High relative humidity slows or reduce the evolution of those properties but does not hinder long term evolution. Cyclic humidity delays development compared to high-temperature and low relative humidity curing, but still allows for long term evolution. Foamed bitumen mixtures showed lower sensitivity to moisture variations than emulsion mixtures, as reflected in their indirect tensile strength and indirect tensile stiffness modulus at 28 days.

Prolonged curing results (until 90 days) show that 28 days of high-temperature and low-humidity curing (HT curing) is sufficient to determine the material's ultimate stiffness modulus, as no significant differences were observed between 28- and 90-days results. Also, a 28-day cure is an appropriate timeframe for assessing the effect of temperature and/or humidity on ITS and ITSM development, regardless of the type of curing involved.

Table 6
One-way ANOVA results for extended HT-ITSM values (significance level = 0.05).

Curing regime	Emulsion		
	F-Value	p-Value	F-Crit
HT	**0.017**	**0.984**	3.682

Moreover, the outcomes of prolonged curing under varying relative humidity (ambient vs. 100 %) demonstrated that the loss of stiffness modulus experienced by the material when subjected to high relative humidity is reversible. The stiffness modulus increases when the material is subsequently exposed to an environment with low relative humidity. Consequently, long-term stiffness modulus is significantly influenced by the climatic conditions to which the material is exposed.

These findings provide practical guidelines for predicting the long-term physical and mechanical behavior of CBTM in FDR-treated pavements, enhancing durability and reducing construction time under diverse climatic conditions. Future research should explore the effects of varying binder contents in mix design, focusing on resistance to rutting or fatigue. Additionally, an environmental assessment of greenhouse gas (GHG) emissions from foamed and emulsion mixtures is recommended to identify the more sustainable stabilization method.

CRediT authorship contribution statement

Lachance-Tremblay Eric: Writing – review & editing, Visualization, Validation, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization. **Ben Yahya Maher:** Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis. **Lamothe Sébastien:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Methodology, Conceptualization.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Eric Lachance-Tremblay reports financial support was provided by Natural Sciences and Engineering Research Council of Canada. Eric Lachance-Tremblay reports financial support was provided by Mitacs Canada. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data Availability

Data will be made available on request.

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