Examining the Effects of Contributory Factors on Curing process of Cold Bitumen Emulsion Mixtures

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ABSTRACT:

Curing of Cold Bitumen Emulsion Mixtures (CBEMs) is influenced by different factors such as curing temperature, curing time, humidity and presence of cement. In this study, the influence of these contributory factors on CBEMs has been evaluated in terms of indirect tensile stiffness modulus. During the curing period, the mix moisture content has been monitored. The results showed that the high curing temperature is responsible for additional stiffness gain by increasing the binder stiffness due to ageing and by increasing the moisture loss by evaporation during the curing process. However, at high curing temperature the moisture loss by evaporation may hinder the hydration of cement. Moreover, the results also indicate that the high relative humidity level influences the stiffness modulus of CBEMs negatively.

Keywords: Cold bitumen emulsion mixtures, curing temperature, cement, relative humidity, indirect tensile stiffness modulus.

1. INTRODUCTION

In recent years, environmental issues regarding reducing energy consumption, reducing CO_2 emissions and managing wastes have been increasingly articulated and have been gaining more attention worldwide. One of the most important trends in road materials and pavement engineering is the use of cold asphalt mixes (CAMs) in roads construction.

The performance of CAM is intimately related to the properties and proportions of materials that are used in the mixture and to the curing condition. One of the most common types of CAM is cold bitumen emulsion mixture (CBEM) treated with cement (Needham, 1996, Thanaya, 2003, Oruc et al., 2007, Niazi and Jalili, 2009, Bocci et al., 2011). In general, the incorporation of cement into CBEMs can increase: stiffness modulus, resistance to permanent deformation, resistance to fatigue cracking and resistance to moisture damage (Needham, 1996). The CBEM requires a certain time which is necessary to cure and build up the ultimate mechanical properties such as strength and stiffness. This process is termed "curing" and is a process whereby the CBEM gradually gains both strength and stiffness over time. This process accompanied by emulsion breaking, moisture loss and/or hydration of cementitious compounds in case of CBEMs treated with cement. It is a well-established fact that the curing process has a significant effect on the mechanical properties and performance of CBEMs (Jenkins, 2000).

Despite the fact that a wide range of studies have been undertaken to investigate the effect of incorporation of cement into CBEMs, considerable issues still need to be addressed. In particular, there is a lack of clarity regarding the influence of the curing process on the performance of CBEMs treated with cement. This is because of the complex combination of three phenomena acting together during the curing process: emulsion breaking, moisture loss and hydration of cementitious compounds (García et al., 2013, Cardone et al., 2014, Serfass et al., 2004). It is important that the bitumen emulsion breaking process is achieved as soon as possible after emulsion application. However, it must not occur until after the completion of the mixing and compaction phases. Accordingly, the presence of water after emulsion breaking can negatively affect early strength gain. Increasing the curing temperature leads to an increase in the rate of water evaporation, resulting in an increase in the strength gain process. Furthermore, the presence of cement accelerate the emulsion breaking process, increase the rate of bitumen coalescence and reduce the amount of evaporable water (Needham, 1996). More strictly, hydration of cementitious compounds is linked to moisture loss; the chemical reactions that take place during the hydration of cementitious fillers require the presence of water and do not require any moisture loss. However, the increasing of curing temperature results in an increase in the amount of water evaporation.

The main objective of the present study therefore, is to investigate the level of impact of climatic parameters such as curing temperature and humidity in addition to the impact of curing time and the presence of active filler (cement) on the curing process in CBEMs. The effect of such factors on the curing process has been evaluated in the laboratory in terms of indirect tensile stiffness modulus, water loss evaluation and binder characterization before and after curing. A series of mixtures having different amounts of cement (0, 1, 3, 5%) and cured at constant temperature (5, 20, 40^{O}C) has been evaluated. Moreover, two relative humidity levels (less 50% and higher 85 %) have been adopted in this study.

2. MATERIALS CHARACTERIZATION:

2.1. *Aggregate*

The aggregate used in this study was crushed limestone. The physical properties of the aggregate were: apparent density 2.70 Mg/m^3; absorption 0.4%; Los Angeles Coefficient 28. The gradation of the aggregate (Figure 1) was within the limits of 0/14 mm size dense graded surface course, according to European Committee for Standarization (2005a).

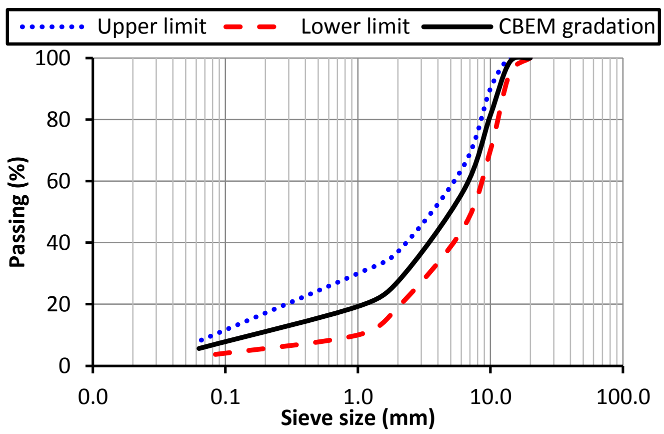

Figure 1. Limestone aggregate gradation of 0/14 mm size according to BS EN 4987-1.

2.2. *Bitumen emulsion*

The bitumen emulsion used was C60B5. This is a cationic slow setting bituminous emulsion, with 60 % of bitumen content and density 1.016 g/cm^3. 40/60 penetration grade bitumen was used in emulsion production. The high stability and high adhesion of cationic emulsion was the reason in which this type of emulsion was selected, as recommended by Thanaya (2003).

2.3. *Fillers*

Two types of filler material were used in the CBEMs; natural limestone filler (LF) and Ordinary Portland cement (OPC). The OPC used in this study was CEM I 52.5R. Scanning electron microscopy (SEM) was used for determining the morphology of these two fillers, as shown in Figure 2. SEM analysis was implemented under a resolution of 3-4 nm and an accelerated voltage of 15 kV.

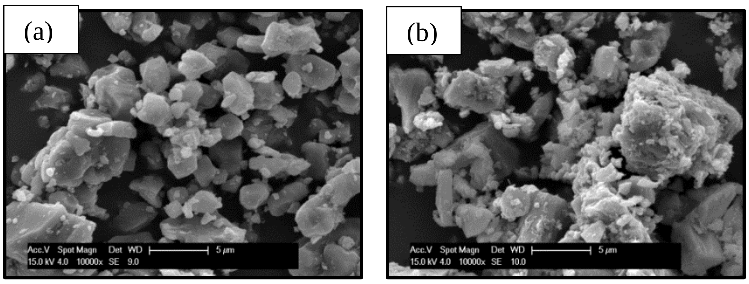

Figure 2. Morphology of (a) natural limestone filler and (b) ordinary Portland cement.

2.4. *Mix proportions, sample manufacture and curing procedures:*

A performance based mix design approach was adopted in order to optimize the mix proportions for CBEM using a statistical technique. The details regarding the mix proportions based on this statistical technique have been described in a previous work (Nassar et al., 2016). According to this procedure, pre-wetting water content and optimum bitumen emulsion content were 2.12% and 6.75% of total weight of aggregate, respectively.

The same mixture proportions were used to prepare CBEMs with cement. The total amount of filler was fixed at 5% by total weight of the aggregate. Specimens of CBEM were prepared using different ratios of OPC, by replacing LF with 0%, 1%, 3% and 5% of OPC. In this paper, C designates OPC; 0, 1, 3 and 5 represent the amount of OPC by mass of dry aggregate. For example, 1C-CBEM-40^{O}C, is a CBEM mix with 1%

OPC by mass of dry aggregate (and therefore 4% LF) cured at 40^{O}C. Marshall specimens were prepared for all CBEMs. Mixing was carried out using a Sun and Planet mixer. Thereafter, impact compaction (Marshall Hammer) was utilized to compact the specimens; following a pilot study 75 blows were applied to each face in order to produce a suitably dense mixture. After compaction, the curing protocol followed was divided into two stages as recommended by Jenkins (2000). In the first stage, the specimens were left in their moulds (in a sealed condition) after compaction for 24hrs. This was due to the fragile nature of the specimens in early life. In the second stage, the specimens were extruded and must were conditioned in a thermostatically controlled air chamber at 5, 20 or 40^{O}C with low relative humidity level, i.e. less than 50%. Specimens were not sealed during the second stage of curing to guarantee free water evaporation. Table 1 summarizes the curing procedure for CBEMs.

Table 1. CBEM curing protocol.

Curing Temperatures	First stage	Second stage
5^{O}C	24hrs at 20^{O}C (sealed)	3 months at 5^{O}C (unsealed, low RH)
20^{O}C	24hrs at 20^{O}C (sealed)	3 months at 20^{O}C (unsealed, low RH)
40^{O}C	24hrs at 20^{O}C (sealed)	3 months at 40^{O}C (unsealed, low RH)
20^{O}C	24hrs at 20^{O}C (sealed)	3 months at 20^{O}C (unsealed, high RH)

These curing temperatures were selected to represent different conditions that may apply during CBEM curing. The severe condition at 5^{O}C was selected to simulate cold climatic conditions (Bocci et al., 2011) while the curing at 20^{O}C was chosen to simulate a more usual site condition while avoiding any early ageing of the binder(Serfass et al., 2004). Finally, curing at 40^{O}C was used to represent typical summer temperature in some European countries (Bocci et al., 2011) also to represent the accelerated curing suggested by literature (Thanaya, 2003, Jenkins, 2000).

A plastic box with dimensions 605×370×355 mm was used to design a simple humidity chamber to cure the final batch of specimens. A saturated NaCl solution was used to maintain a high level of humidity as recommended by Kubo (2007). The actual recorded humidity varied between 85% and 95% through the period of curing with a temperature of between 19 and 23 ºC. The specimens were stored over a steel mesh to avoid any contact with the saturated NaCl solution and the chamber sealed to keep the moisture inside. A schematic diagram and photograph of the chamber are shown in Figure 3.

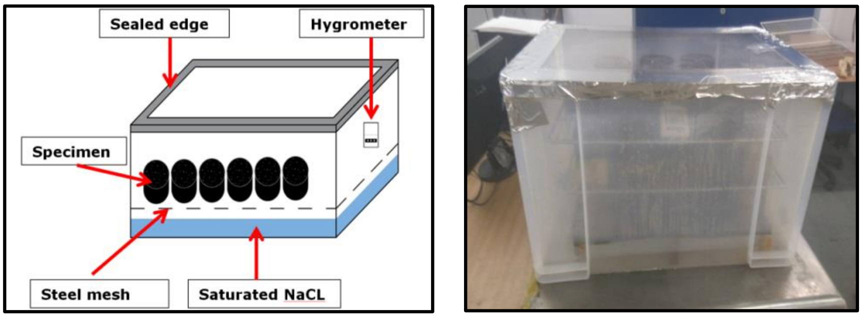

Figure 3. Schematic diagram and photograph of the relative humidity chamber.

3. EXPERIMENTAL PROGRAMME

3.1. *Moisture loss monitoring*

The moisture in the CBEM comprises of the water added during mixing and the water in the bitumen emulsion. Moisture loss by evaporation is defined as the difference between the initial weight of the specimen and the weight at a given time divided by the weight of the specimen. During the curing period, weight measurements were made at 2, 3, 5, 7, 10, 14, 28, 54 and 84 days.

3.2. *Indirect tensile stiffness modulus (ITSM) evaluation*

The stiffness gain (curing trend) of CBEMs was monitored over a period of time. The non-destructive stiffness test, ITSM, was selected for assessing the stiffness modulus over a period of approximately 3 months, as shown in Figure 4. Three specimens per mix were conditioned before the test for 4hrs at 20^{O}C then tested at 20^{O}C. The test was carried out according to BS EN 12697-26 (European Committee for Standarization, 2012), as shown in Table 2. The ITSM was chosen to allow the test to be carried out repeatedly on the same set of specimens to nullify variability in the mixtures and to derive reliable trends for stiffness evolution. Stiffness modulus is considered as an indicator of the structural condition of a mixture because it is directly related to the capacity of the asphalt material to distribute traffic loads.

Table 2. ITSM test configuration based on BS EN 12697-26.

Item	Range
Specimen diameter	100±2 mm
Specimen thickness	40-80 mm
Transient peak horizontal deformation	3 μm
Rise time	124±4 ms
Poisson's ratio (assumed)	0.35
Conditioning prior the test	4hrs at 20^{O}C
Testing temperature	20^{O}C

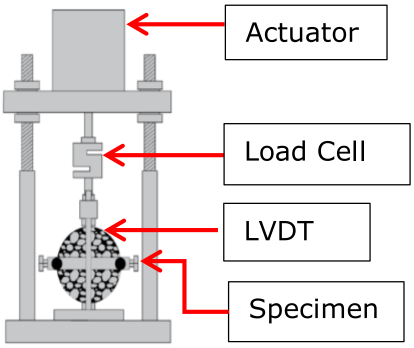

Figure 4. Schematic diagrams and test configurations for ITSM.

3.3. *Bitumen binder characterization before and after the curing*

After 28 days curing at 5, 20 or 40^{O}C, the bitumen was extracted based on European Committee for Standarization (2005b) from the 0C-CBEM specimens. Also, the bitumen was extracted from the bitumen emulsion by distillation method. The extracted bitumen was characterised using frequency sweep and softening point tests. The test programme is shown in Table 3.

Table 3. Testing program summary

Mixture code	Test	Curing Time (days)	Curing Temperature (^{O}C)	Relative Humidity
0C-CBEM, 1C-CBEM, 3C-CBEM, 5C-CBEM	Moisture loss monitoring	2, 3, 5, 7, 10, 14, 28, 54, 84	5, 20, 40	Low level, less than 50%
0C-CBEM, 1C-CBEM, 3C-CBEM, 5C-CBEM	ITSM	3, 7, 10, 14, 28, 54, 84	5, 20, 40	Low level, less than 50%
0C-CBEM, 1C-CBEM, 3C-CBEM, 5C-CBEM	ITSM	7, 28	5, 20, 40	High level, more than 85%
0C-CBEM (extracted Binder)	Frequency sweep, softening point	28	5, 20, 40	Low level, less than 50%

4. RESULTS AND ANALYSIS

4.1. *Moisture loss Monitoring*

The moisture loss by evaporation during the curing process of CBEM is one of the major phenomena occurring. Monitoring the moisture loss by evaporation was carried out over 3 months of curing to explore the rate and sequence of loss of water as shown in Figure 5.

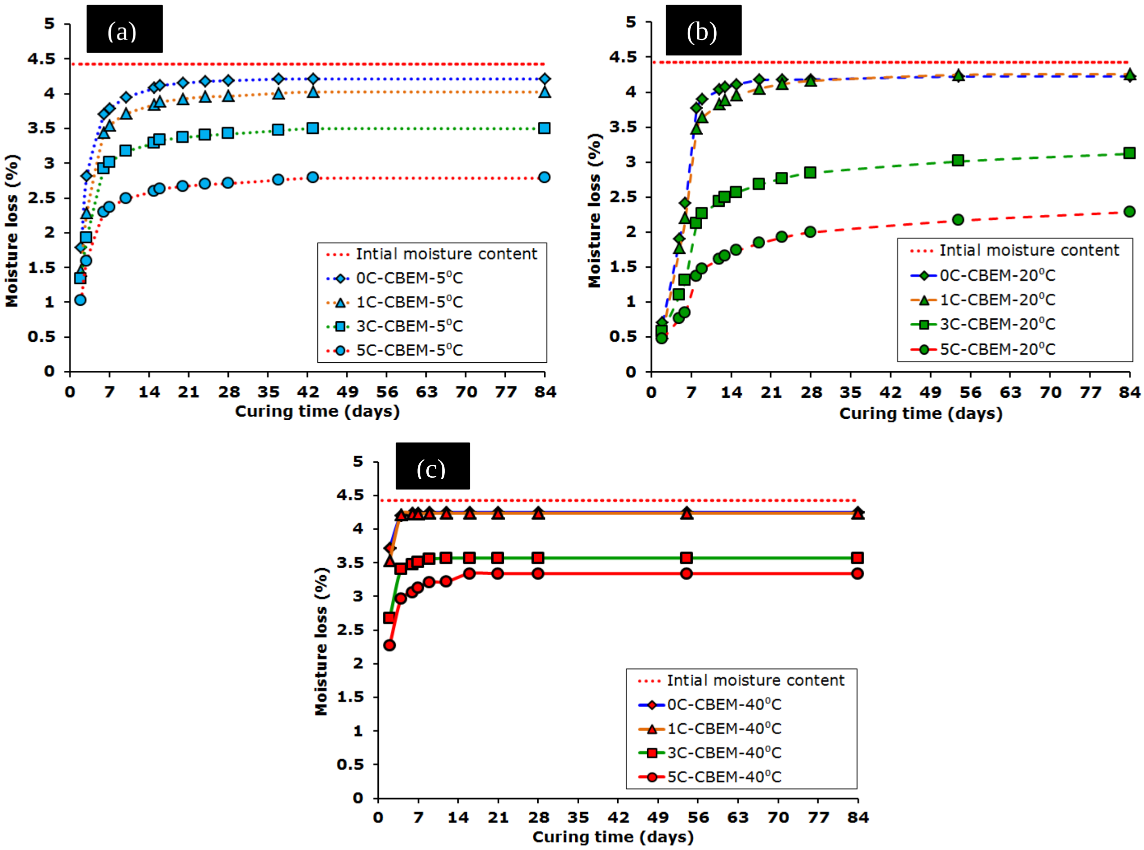

Figure 5. Moisture loss by evaporation for CBEM cured at (a) 5°C (b) 20°C (c) 40°C.

It can be observed that the rate of evaporation during the first month is significantly influenced by the curing temperature. The moisture loss for mixes cured at 40°C did not increase after 14 days, having reached an equilibrium condition, whereas the equilibrium condition was achieved after 28-35 days for mixes cured at 5°C and 20°C. After CBEMs reached their equilibrium condition, there was very little moisture loss over the rest of curing period. Regardless of curing temperature, the amount of moisture loss decreased with increasing cement content. This is because the water had been absorbed by the cement in the hydration process. The results are very similar to the findings of Needham (1996) and Oruc et al. (2007).

4.2. *Indirect tensile stiffness modulus evaluation*

4.2.1. *Influence of curing time and temperature*

The stiffness modulus measured at 20°C at different curing times and temperatures, is reported in Figure 6. It is clear from Figure 6 (a) that the stiffness modulus of 0C-CBEM increased with increasing curing temperature and time. This gain was rapid for specimens cured at higher temperatures (40°C). This could be attributed to rapid water loss at higher temperature. Also, in cement treated mixes (1C-CBEM, 3C-CBEM, 5C-CBEM), the stiffness modulus increased with increasing curing time. However, this gain was significantly influenced by interaction with both moisture loss and cement hydration. This leads to the interesting observation that improvement in the stiffness modulus did not continue after 28 days for 5°C and 40°C cured mixes while the 20°C cured specimens showed some further development.

4.2.2. *Influence of cement content and curing temperatures*

Figure 6 shows that the stiffness modulus of CBEM increased with increasing cement content at a given curing temperature. This increase might be partly due to the removal of water from the system and partly due to the formation of cementitious bonds.

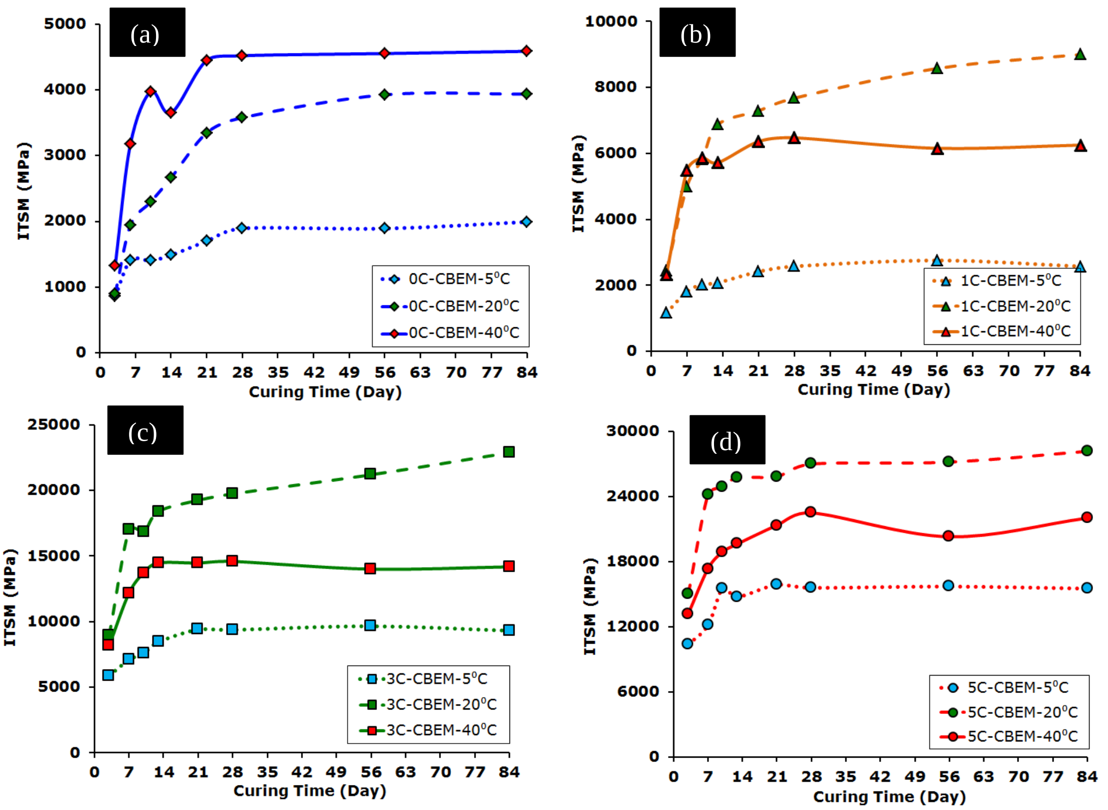

Figure 6. Evolution of ITSM at 20°C with curing time for (a) 0C-CBEM (b) 1C-CBEM (c) 3C-CBEM and (d) 5C-CBEM.

However, it is noticeable that 20^{O}C cured mixes showed the highest stiffness modulus over the curing period in comparison to 5^{O}C and 40^{O}C cured mixes. For example, the 5C-CBEM specimens achieved stiffness modulus of 15600, 27000 and 22500 MPa after 28 days cured at 5, 20 and 40^{O}C, respectively. It is believed that the low curing temperature (5^{O}C) limited the stiffness modulus gain since a low hydration reaction temperature tends to prevent hydration development (Puertas et al., 2000), although this effect has been little studied in cement treated CBEM under different curing temperatures. Moreover, the low rate of moisture evaporation also contributes in this case. More surprisingly, Figure 6 illustrates that the stiffness modulus for 1C-CBEM-20^{O}C, 3C-CBEM-20^{O}C and 5C-CBEM-20^{O}C specimens was higher than for those cured at 40^{O}C.

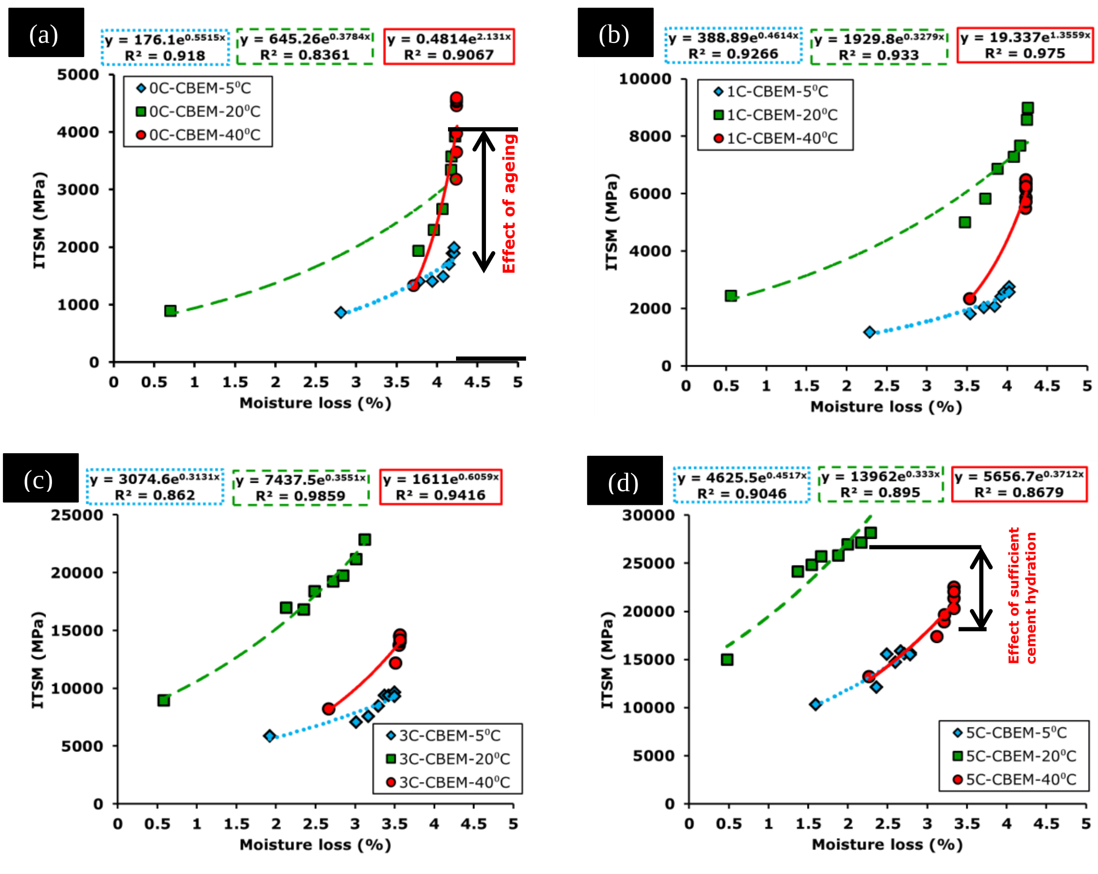

Figure 7. The relationship between moisture loss and stiffness modulus under different curing temperatures (a) 0C-CBEM (b) 1C-CBEM (c) 3C-CBEM (d) 5C-CBEM

A similar observation was made by Cardone et al. (2014). This could be because the rate of moisture loss is greater at higher curing temperature (40^{O}C), particularly at early age. This may lead to insufficient water which may hinder the hydration process, see Table 4. Furthermore, it is interesting to mention that the density of hardened cement tends to be less uniform at higher curing temperature, which can lead to weaker spots in the microstructure (Rovnaník, 2010). Thus, further research is needed to study the effect of curing temperature on the microstructure of cement treated CBEM. As a general trend, with increase in moisture loss the stiffness increased. Therefore further analysis of the relationship between the moisture loss by evaporation and stiffness modulus was carried out, see Figure 7.

It may be observed in Figure 7 that when a certain moisture loss is considered the curing temperatures have different impacts on the stiffness modulus for the same mixtures. On the one hand, mix 0C-CBEM-40^{O}C showed far better stiffness than 0C-CBEM-20^{O}C or 0C-CBEM-5^{O}C with the same moisture loss at the end of the curing period. This can be explained by the fact that specimens cured at 40^{O}C are subject to ageing over the curing period which is consistent with the results from binder characterisation before and after the curing, see section 3.3.

On the other hand, the stiffness modulus of mixes 1C-CBEM, 3C-CBEM and 5C-CBEM showed a marked influence of moisture loss under different curing temperatures. At the same cement content, the stiffness modulus of specimens cured at 20^{O}C was higher than those cured at 5^{O}C or 40^{O}C.

In order to understand more deeply the effect of moisture loss on the moisture content of CBEM, the moisture content is divided into two types. The first type is the evaporated moisture ($M_{Evaporation}$) which is calculated based on the difference between the initial weight and the weight after the end of the curing period. The second type is the trapped moisture ($M_{Trapped}$) inside the mix due to (1) the water physically adsorbed on the surface of aggregate/filler, (2) the water trapped in the closed pores within the bitumen and (3) the water used during cement hydration. The $M_{Trapped}$ was calculated by subtracting the evaporated water from the initial water content present in the CBEM specimens. The amount of moisture required for the hydration of cement ($M_{Hydration}$) was calculated based on equation (1) recommended by Mehta and Monteiro (1995)

$$M_{Hydration} = (0.23 + 0.19) \times \alpha \times k \times C \qquad (1)$$

Where α is the degree of hydration of the cement paste, C is the cement content within the mixture and k is the fraction of Portland cement clinker in the cement. In table 4 it is assumed that k = 0.87 (average of the clinker content range) and α= 0.95 at 100 days of curing.

Table 4. Moisture calculations.

Mixture code	$M_{Evaporation}$			$M_{Trapped}$			$M_{Hydration}$
	40°C	**20°C**	**5°C**	**40°C**	**20°C**	**5°C**	
0C-CBEM	4.25%	4.18%	4.19%	0.18%	0.25%	0.24%	0.00%
1C-CBEM	4.24%	4.08%	3.96%	0.19%	0.34%	0.46%	0.32%
3C-CBEM	3.57%	2.84%	3.42%	0.86%	1.58%	1.01%	0.96%
5C-CBEM	3.34%	1.99%	2.71%	1.09%	2.43%	1.72%	1.59%

The moisture types are summarized in Table 4. It was found that the amount of $M_{Trapped}$ inside the CBEM cured at 5°C and 20°C is more than $M_{Hydration}$ whereas the $M_{Trapped}$ inside the CBEM cured at 40°C is less than $M_{Hydration}$. It is therefore evident that the moisture loss by evaporation affects the hydration of cement, particularly at high curing temperature (40°C). This will hinder the hydration process. This result is in agreement with the finding of Cardone et al. (2014). Moreover, the calculation in Table 4 indicates that most CBEMs still maintained some residual water ($M_{Trapped}$ - $M_{Hydration}$). This moisture may possibly be trapped inside the surface pores of the aggregate or inside the bituminous mortar (García et al., 2013).

A three-way analysis of variance (ANOVA) was conducted to determine the effect of cement content (0, 1, 3, 5%), curing temperatures (5, 20, 40°C) and curing times (3, 7, 10, 14, 28, 54, 84 days) on the stiffness modulus of CBEMs. The result showed that these factors are statistically significant at the 99% confidence level. The p-values obtained for these factors are less than 0.001. Also, the result of ANOVA analysis shows that cement content presented the largest F-values of 314.078, followed by curing temperatures of 72.234 and curing times of 9.357. This means that the cement content and curing temperatures had a higher significant effect on stiffness modulus of CBEM, compared with curing times.

4.2.3. *Influence of relative humidity*

Figure 8 presents stiffness modulus results of specimens cured at 20°C and conditioned at two different levels of relative humidity (RH) (low=less 50%, high= more 85%). The results in Figure 8 show that higher humidity decreases the stiffness development rate of CBEM.

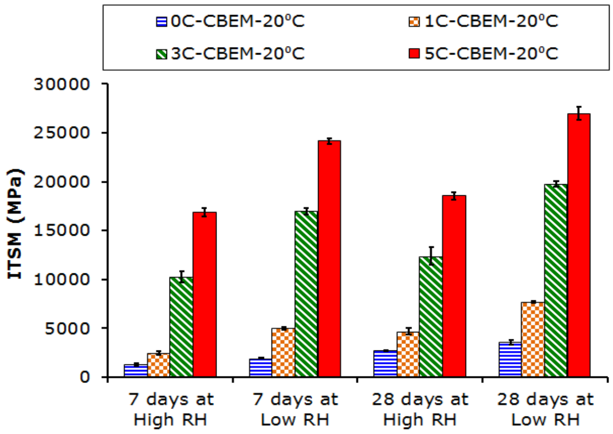

Figure 8. The effect of relative humidity on the stiffness modulus of CBEM.

Thus, longer curing time would be needed to achieve full strength. This behaviour implies that a high level of humidity decreases the rate of water evaporation from the mixture which influences the stiffness negatively. However, high humidity is often desirable for curing mixes with cement, as the hydration process of cement takes place in the presence of water. But, if the moisture content inside the mixture is enough for the hydration process, then exposure to a humid environment would be unnecessary or even detrimental. This result is consistent with the findings of Fu et al. (2009) and Lin et al. (2015). A two-way ANOVA analysis was conducted to determine the impact of humidity level and cement content on CBEMs modulus. The analysis confirms that both the humidity level and cement content are statistically significant at the 95% confidence level ($P_{Humidity\ level} = 0.005 < 0.05$, $P_{Cement\ content} < 0.001$).

4.3. *Bitumen characterization before and after curing*

To study the effect of curing temperature on bitumen properties, bitumen was extracted from 0C-CBEM samples cured for 28 days at 5, 20 and 40^{O}C. Softening point and Frequency sweep tests were carried out and Figure 9 illustrates the complex modulus (G*) master curves at a reference temperature of 20°C.

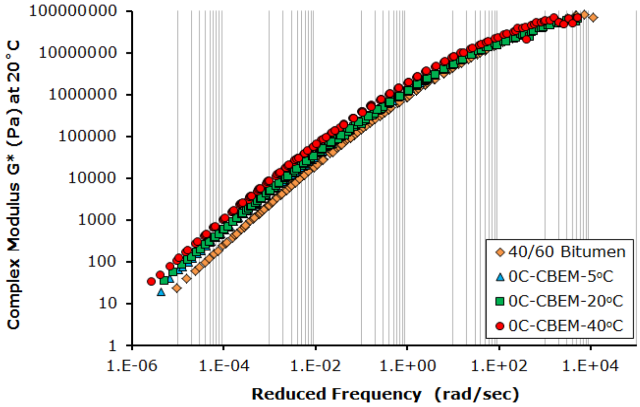

Figure 9. The effect of curing temperatures on bitumen properties.

It can be noticed from Figure 9 that the differences in complex modulus of the recovered bitumen are significant, particularly at low frequency, which indicates that the bitumen ageing effect differs considerably between different curing temperatures. Moreover, the effect of curing temperatures on the softening point of the binder was also evaluated. Before mixing and applying the curing protocols, the softening point of the original base binder was 52.8^{O}C. After applying the curing protocols the softening points of extracted binder were 55.6, 56.2, and 59.2^{O}C for 0C-CBEM-5^{O}C, 0C-CBEM-20^{O}C and 0C-CBEM-40^{O}C, respectively. Over all, based on softening point and frequency sweep test results it may be concluded that the binder properties changed appreciably during the curing process. Hence, it is evident that water loss and the cement hydration are not the only mechanisms involved during curing of CBEMs. High curing temperature is also responsible for additional stiffness gain due to ageing.

5. CONCLUSION:

The effects of several contributory factors on the curing process of CBEM were investigated. These factors were the curing time, curing temperature, cement content and relative humidity. On the basis of the laboratory test results, the following conclusions were derived:

1. An Increase in curing temperature facilitated the evaporation of moisture in the CBEMs leading to improved mechanical and performance properties
2. The results also showed the role of cement and its usefulness in positively improving the stiffness modulus especially during the early life of CBEMs.

3. High curing temperature was responsible for additional stiffness gain by increasing the binder stiffness due to ageing and by increasing the moisture loss by evaporation during the curing process. However, at high curing temperature the moisture loss by evaporation may hinder the hydration of cement.
4. Further investigation is needed to study the effect of curing temperature on the microstructure of cement treated CBEM.

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