Mechanical, Durability and Microstructure Properties of Cold Asphalt Emulsion Mixtures with Different Types of Filler

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Abstract

The primary aim of this study is to investigate the enhancement of Cold Asphalt Emulsion Mixtures (CAEMs) using binary and ternary blended fillers (BBF and TBF), including an in-depth assessment of the microstructure. Ordinary Portland cement (OPC), fly ash (FA) and ground granulated blast furnace slag (GGBS) were used for the BBF while silica fume (SF) was added to the BBF to obtain TBF. The mechanical and durability results indicated that the TBF was more suitable than the BBF for the production of CAEMs. The microstructural assessment indicated that the effect of BBF on the internal microstructure of CAEMs was slightly negative and more noticeable in CAEMs containing FA. It is proposed that the addition of SF to BBF mixtures can eliminate the delay in formation of hydration products caused by the bitumen emulsion. Overall, the research suggests that the use of BBF-CAEMs might be appropriate for pavements in cold climate whereas TBF-CAEMs would be effective in road pavements exposed to severe conditions both in hot and cold climates.

Keywords: Cold Asphalt Emulsion Mixtures; Blended fillers; Stiffness modulus; Repeated load axial test; Durability; Microstructure; Delay hydration process

1. Introduction

Nowadays, environmental issues about reducing energy consumption, reducing CO_2 emissions and managing wastes are being increasingly articulated and have been gaining attention worldwide. One of the most significant trends towards more eco-friendly asphalt mixes is the use of material such as recycled asphalt pavement, municipal solid waste incineration ash, construction and demolition waste material, cement kiln dust and coal ash [1-8].

Due to the many significant environmental and economic benefits that can be derived from using cold asphalt mixtures (CAMs), several research projects have been performed to study and develop the properties of these mixtures. The development of CAMs has commonly been by utilizing waste material/ by-products while achieving satisfactory hot mix asphalt (HMA) properties [6, 7, 9, 10]. However, CAMs for sustainable and resilient pavements still has to meet the requirements of carrying heavy traffic loads from a mechanical perspective. Additionally, the properties of CAMs need to resist the impact of the environment from a durability perspective. In the current research, these two features will be termed as engineering properties. The enhancement of the engineering properties of CAMs mainly depends on the type, quantity and quality of the raw materials used. This might be considered as one of the most important factors to extend the use of CAMs for the use as a surface course.

Fillers can play a major role controlling the engineering properties of asphalt mixtures. It has been demonstrated that filler can significantly influence permanent deformation resistance, stiffness, fracture resistance, and moisture susceptibility of asphalt concrete [11, 12]. A recent development aimed at achieving excellent engineering properties of CAMs is the use of manufactured fillers. This kind of filler is produced as a blend of reactive, semi-reactive and non-reactive natural fillers [6, 7, 13, 14]. In this study, binary blended fillers (BBFs) contain a combination of fly ash (FA) or granulated blast furnace slag (GGBS) with ordinary Portland cement (OPC), while the ternary blended fillers (TBFs) are a combination of BBFs with silica fume (SF). These fillers are used for many reasons. FA, GGBS, SF are considered to be some of the most used supplementary cementitious material alternatives. Furthermore, their availability worldwide in substantial quantities can provide social, economic, and environment benefits.

An experimental study by Oruc, Celik and Akpinar [13] assessed the mechanical properties of emulsified asphalt mixtures including 0-6% OPC, which was substituted for mineral filler. Their findings showed a remarkable improvement with a high percentage of OPC treatment, recommending these mixtures to be used as a structural layer. Al-Busaltan, Al Nageim, Atherton and Sharples [6] confirmed the enhancement of close graded CAEM to a stage where its mechanical properties were comparable to those of traditional asphalt concrete mixtures. The improvement was due to the replacement of the conventional mineral filler with a domestic fly ash. Al-Hdabi, Al Nageim, Ruddock and Seton [15] showed a significant improvement in mechanical properties and water damage resistance of cold rolled asphalt by incorporating a biomass fly ash with cement. Recently, the influence of chemical additives including OPC, hydrated lime (HL), and a combination of HL and GGBS on recycled mixture performance was investigated by Du [16]. The results showed that hydration products can increase the stiffness and cohesion of the asphalt mastic of the recycled mixture [16].

A comparison study between using coal ash and OPC in cold recycled asphalt mixtures was carried out by Modarres and Ayar [1]. The results revealed that the application of coal waste powder improved the mechanical properties of cold recycled asphalt material, but it could not achieve a positive impact on moisture damage resistance. Based on these comparisons, coal ash was found to have comparable effects to OPC. While previous studies have highlighted the importance of using OPC, HL and some specific kinds of fly ash in developing CAMs, there is a need to develop more sustainable CAMs using artificial by-products such as FA, GGBS and SF in combination with OPC. The purpose is to reduce the potential increase in production costs and environmental issues particularly in the manufacturing process of both cement and lime. However, the availability of alternative cement materials should be considered when choosing the type of fillers in CAMs.

Pouliot, Marchand and Pigeon [17] showed that the presence of a small quantity of bitumen emulsion in cement mortar had a delaying effect on the cement hydration. Furthermore, Du [18] agreed with this study and proposed a mechanism for the hydration process delay. He suggested that some of the bitumen droplets and cement particles come into contact due to intensive chemical adsorption, and as a result, some of the cement particles are encapsulated by an asphalt film, causing a delay or interruption in the hydration reaction. A study carried out by Wang, Shu, Rutherford, Huang and Clarke [19] showed that the total hydration heat decreased with an increase in bitumen to cement ratio in CAEM. Also, the study revealed that the addition of bitumen emulsion significantly influenced cement hydration behaviour in CEAM. In contrast, a recent experimental study carried out by Fang, Garcia, Winnefeld, Partl and Lura [20] revealed that the behaviour of bitumen emulsion in the presence of cement and filler is still unclear and may slightly retard or accelerate cement hydration, but has no significant effect on the degree of cement hydration. These research studies have demonstrated developments in the properties of CAEMs by using different types of filler. However, this raises an important question as to how these fillers (cement/artificial by-products) interact with bitumen emulsion and affect the hydration and microstructural characteristics of CAEMs.

2. Research significance

This study aims to develop special types of CAEM by using artificial by-products such as FA, GGBS and SF in combination with OPC. Accordingly, it is hoped that wider utilization of CAEMs in the construction of highway and pavement materials (with both environmental and economic impacts) can be derived from the current research. Meanwhile, this work is designed to contribute to a deeper understanding of the microstructure and internal composition of the mixes concerned.

3. Material and testing program

3.1. Material characteristics

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 was within the limits of 0/14 mm size close graded surface course, according to BS EN 13108-1. This selection was made in order to ensure an appropriate interlock between the aggregate particles in the mixtures as recommended by the European Standard [21]. The gradation of the aggregate is shown in Fig. 1. The amount of mineral filler (limestone-passing sieve 0.063 mm) was selected to be 5% of the total weight of the aggregate.

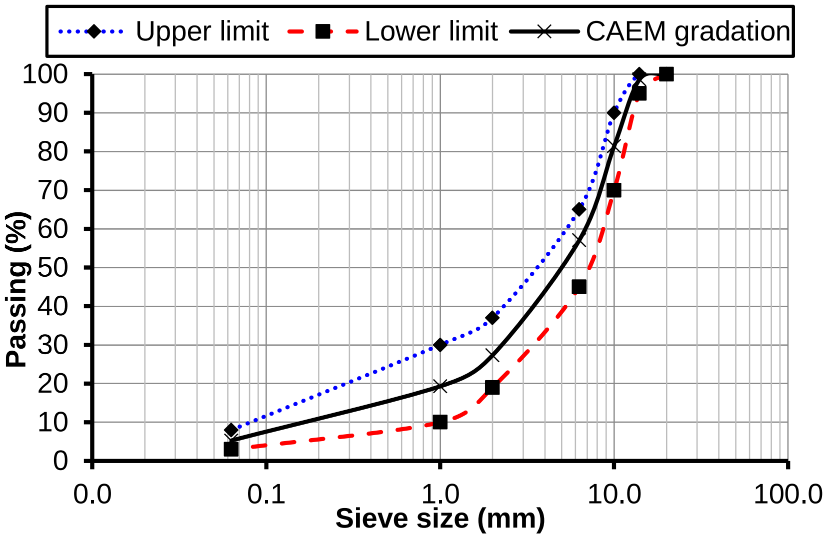

Fig. 1. Limestone aggregate gradation of 0/14 mm size close graded surface course according to BS EN 13108-1.

A commercial cationic slow setting bituminous emulsion, C60B5, was used to manufacture the CAEMs. This type of emulsion contains 60% residual bitumen content of 40/60 pen base binder with a softening point of 52 ^{o}C. The high stability and high adhesion of such cationic emulsion were the reason for selection as recommended by Nikolaides [22] and Thanaya [23].

Ordinary Portland cement (OPC) CEM I, 42.5 N, obtained from Cemex, was used in this study. A combination of OPC, fly ash, (class F) according to ASTM C-618, and GGBS obtained from Cemex and Hanson respectively, were used for the BBF mixtures. Densified silica fume (SF) produced by Elkem Microsilica was used as an additional mineral filler for the TBF mixtures.

A detailed characterization was carried out to investigate the chemical and physical properties of the selected fillers. The chemical composition of fillers (major oxidises) was analysed by EDX, energy dispersive X-ray fluorescence spectrometer. The results are presented in Table 1. Scanning electron microscopy (SEM) was used for determining the morphology of fillers, as shown in Fig. 2. SEM analysis was implemented under a resolution of 3-4 nm and an accelerated voltage of 15 kV.

Table 1. Chemical composition of fillers and cement

	OPC	**FA**	**GGBS**	**SF**	**LF**
Na_2O	0.56	1.27	0.28	0.17	--
MgO	2.04	1.76	6.89	0.36	--
Al_2O_3	4.11	25.14	11.21	0.39	--
SiO_2	19.84	51.32	35.65	97.95	1.97
SO_3	5.41	1.90	2.43	--	--
K_2O	1.06	4.24	0.64	0.89	--
CaO	64.35	2.57	41.42	0.24	98.03
TiO_2	0.25	1.29	0.63	--	--
Fe_2O_3	2.39	9.77	0.26	--	--
P_2O_5	--	0.74	--	--	--
MnO	--	--	0.60	--	--

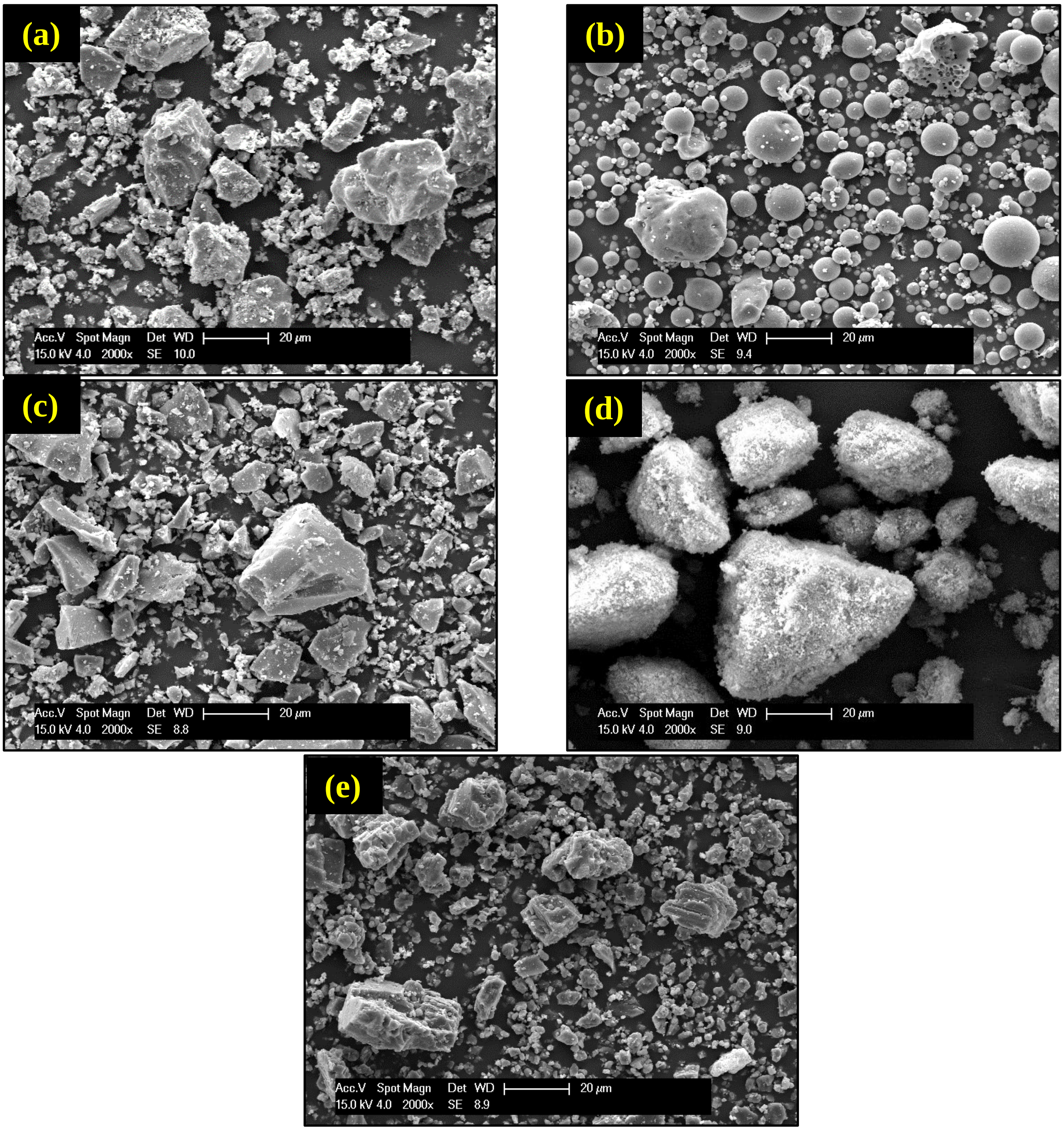

Fig. 2. Morphology of fillers and cement: (a) OPC; (b) FA; (c) GGBS; (d) SF; (e) LF

3.2. Mix proportions and sample preparation

In this study, OPC, FA, and GGBS were used to fully replace the conventional limestone filler in CAEMs, while SF was used as an additive as recommended in the literature [6, 7]. BBFs containing 80% FA/GGBS and 20% OPC (C-FA and C-GGBS) were used for BBF-mixtures. Meanwhile, TBFs consisted of 80% FA/GGBS and 20% OPC with additional SF of 20% and 40% of combined (FA/GGBS+OPC) weight were used for TBF-mixtures (C-FA-20SF, C-FA-40SF, C-GGBS-20SF and C-GGBS-40SF).

A performance-based mix design approach was adopted to optimize the mix proportions for CAEM using a statistical technique described in a previous research work [24]. According to this procedure, pre-wetting water and optimum bitumen emulsion contents were 2.12% and 6.75% of total weight of aggregate, respectively.

The mixture proportions by mass of Marshall specimens are summarised in Table 2. The mixing process was carried out using a Sun and Planet mixer. After that, impact compaction (Marshall Hammer) was utilized to compact the specimens; 75 blows were applied to each face. The Marshall compaction was selected due to it is convince to produce a suitably dense mixture After compaction, the specimens were cured in their moulds (in a sealed condition) for 24hrs and then they were extruded. All the specimens were then conditioned in a thermostatically controlled air chamber at 20°C. This curing temperature was selected as an approximation of the actual condition of CAEMs on site as well as to avoid any early ageing of the binder [25-27].

Table 2. Details of the mix proportions of CAEMs

Mixture Types	Mixtures (g)	Aggregate (g)	Filler combinations (g)					Bitumen Emulsion (g)	Pre-wetting Water (g)
			OPC	FA	GGBS	SF	LF		
BBF mixes	C-FA	878.2	8.8	35.1	-	-	-	59.3	18.6
	C-GGBS	878.2	8.8	-	35.1	-	-	59.3	18.6
TBF mixes	C-FA-20SF	878.2	8.8	35.1	-	8.8	-	59.3	18.6
	C-GGBS-20SF	878.2	8.8	-	35.1	8.8	-	59.3	18.6
	C-FA-40F	878.2	8.8	35.1	-	17.6	-	59.3	18.6
	C-GGBS-40SF	878.2	8.8	-	35.1	17.6	-	59.3	18.6
Reference mixes	LF-mix	878.2	-	-	-	-	43.9	59.3	18.6
	OPC-mix	878.2	43.9	-	-	-	-	59.3	18.6
	HMA	878.2	-	-	-	-	43.9	45.18g base binder (40/60)	

Three additional reference mixes were tested for comparison purposes. The first was untreated CAEM with conventional LF (LF-mix) having a similar design to other CAEMs. The second was CAEM fully treated with 5% OPC only (OPC-mix). The third was standard hot mix asphalt (HMA). This mix contained the same aggregate type, gradation and base binder (40/60) as used in the other CAEMs; the bitumen content in the HMA was 4.9% as recommended by the British

Standard [21]. All CAEMs were mixed and compacted at ambient temperature (20°C) while the HMA was mixed and compacted at 165°C using 75 blows applied to each face.

4. Experimental program and tests performed

4.1. Indirect tensile stiffness modulus (ITSM)

The stiffness gain (curing trend) of reference CAEMs (LF-mix and OPC-mix) and treated CAEMs (BBF and TBF) were monitored over a period. The non-destructive stiffness test, ITSM, was selected for assessing the stiffness modulus over a period of approximately three months. It was performed at different ages, i.e. 3, 5, 7, 14, 28, 45 and 84 days. Three specimens per mix were tested under the same conditions. The ITSM was chosen in order to carry out the test 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. The test was carried out according to BS EN 12697-26 [28], as shown in Table 3.

Table 3. ITSM test configuration based on BS EN 12697-26

Item	Range
Specimen diameter	100±2 mm
Transient peak horizontal deformation	3 µm
Rise time	124±4 ms
Poisson’s ratio	0.35
Test temperature	20°C
Specimen thickness	40-80 mm

4.2. Repeated Load Axial Test (RLAT)

In the UK, the RLAT is the most widely used standard mechanical test for assessing the permanent deformation characteristics of bituminous mixtures. The test applies a repeated pulse load to simulate traffic and measures the permanent deformation after each repeated load. The protocol for the RLAT test was performed based on BS EN 12697-25. Table 4 shows the test configuration [29]. After compaction, the specimens were cured at 20°C for 28 days; after that they were cured in a forced draft oven for a further of 7 days at 40°C. This curing protocol was selected to ensure that a fully cured condition was achieved, as recommended by Needham [9] and Oke [30]. The two faces of the specimen were coated with a thin layer of silicone grease with graphite powder before running the test. Graphite powder is used to eliminate the influence of unevenness of the specimen face on the test results.

Table 4. RLAT test configuration based on BS EN 12697-25

Item	Range
Specimen diameter	100±2 mm
Conditioning stress	10kPa
Conditioning period	600 s
Test stress	100kPa
Test Duration	3600 cycles
Test Cycle	Square wave pulses 1s on, 1s off
Specimen thickness	40-80 mm
Test temperature	40°C

4.3. Durability tests

Durability is a feature directly related to the effect of environmental conditions on the performance of asphalt mixtures during their service life. The durability of CAEMs was evaluated against both moisture and frost damage. This was to present a wide overview regarding the performance of CAEMs in warm and cold climates. Both damage modes are considered as potentially serious problems in a climate such as in the UK. They were evaluated based on BS EN 12697-12 [31] and AASHTO T283 [32], respectively as presented in Table 5.

Table 5. Summary of durability test protocols used

	Moisture damage (BS EN 12697-12)	Frost damage (AASHTO T283)
Unconditioned sets	Curing for 7 days at 20^{o}C	Curing for 7 days at 20^{o}C
Conditioned sets	Curing for 7 days at 20^{o}C+ Vacuum saturation at 6.75kPa for 30mins+ Soaking for 3 days at 40^{o}C+ 2hrs at 20^{o}C	Curing for 7 days at 20^{o}C+ Vacuum saturation at (13-67)kPa+ Freezing for 16hrs at -18^{o}C+ Thawing 24hrs at 60^{o}C +2hrs at 20^{o}C

The durability of asphalt mixtures can be defined by the loss of strength due to the impact of the exposure conditions. The evaluation of durability was determined as a ratio of the indirect tensile strength (ITS) of conditioned specimens to those of unconditioned specimens, expressed in percent (%). The ITS test involved applying a diametric compression load with a constant deformation rate of (50 ± 2) mm/min on the samples between two uniform loading strips. This enables tensile stresses to develop along the diametric vertical plane to cause a splitting failure. The test was conducted at 20°C using Instron test equipment.

4.4.Mineralogy and microstructure tests

X-ray diffraction analysis was utilised to study the hydration of blended cementitious materials and the mineralogical patterns of the crystalline solids within the CAEMs. After 28 days of curing at 20^{o}C, CAEM specimens were crushed into small parts. For XRD examination, some of these parts (from the middle of the specimens) were ground to a powder and passed through a 63μm sieve. The grinding process was continued until all parts passed through the sieve. This was to detect all the crystalline hydration phases in the XRD patterns. The powder samples were positioned and flattened carefully in the sample holder of the XRD machine. A scanning speed of 2° per minute and a step of 0.02° were used in the range of 10 to 60° using a Bruker –AXS D8 Advance XRD equipment. For examining the morphology and the internal microstructure (the associated changes to the hydration products due to the use of binary and ternary blended fillers in CAEMs), other pieces were fixed on small SEM stubs and exposed to high vacuum. Then, a platinum coating was applied to the fractured specimen before capturing the SEM images (secondary electron mode) using a Philips XL 30 SEM instrument. Fig. 3 summarizes the methodology to investigate the internal microstructure of CAEM.

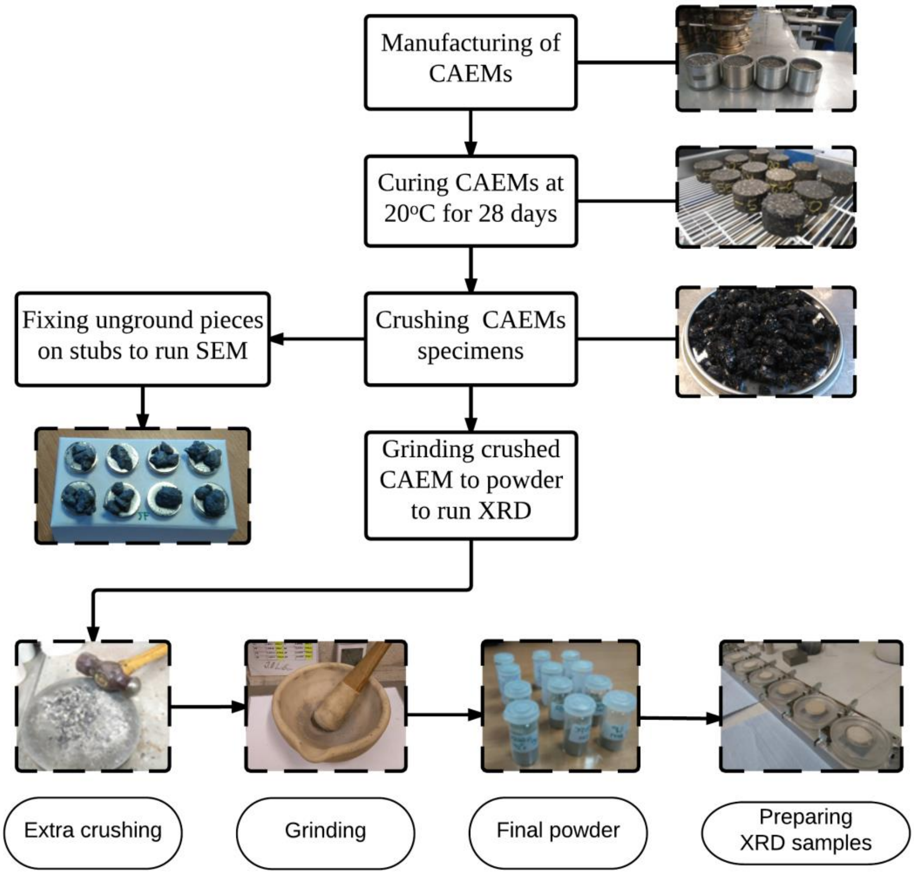

Fig. 3. Methodology to investigate the internal microstructure of CAEM

5. Results and discussion

5.1. Indirect Tensile Stiffness Modulus

Fig. 4 presents the average stiffness modulus of CAEMs for three specimens for each mix with a maximum and minimum coefficient of variation of 13.06% and 0.94%, respectively. It can be observed that the stiffness modulus differences are markedly increased when either BBF or TBF has replaced the conventional filler in the LF-mix. In CAEMs containing TBF, stiffness modulus is generally higher than in those with BBF, indicating a positive effect of adding SF, see section 5.5. However, SF addition improved the stiffness of the mixes containing FA much more than those containing GGBS. The values of stiffness modulus for TBF treated CAEMs are only slightly lower than those for the OPC-mix, indicating the positive effect of this kind of cement replacement.

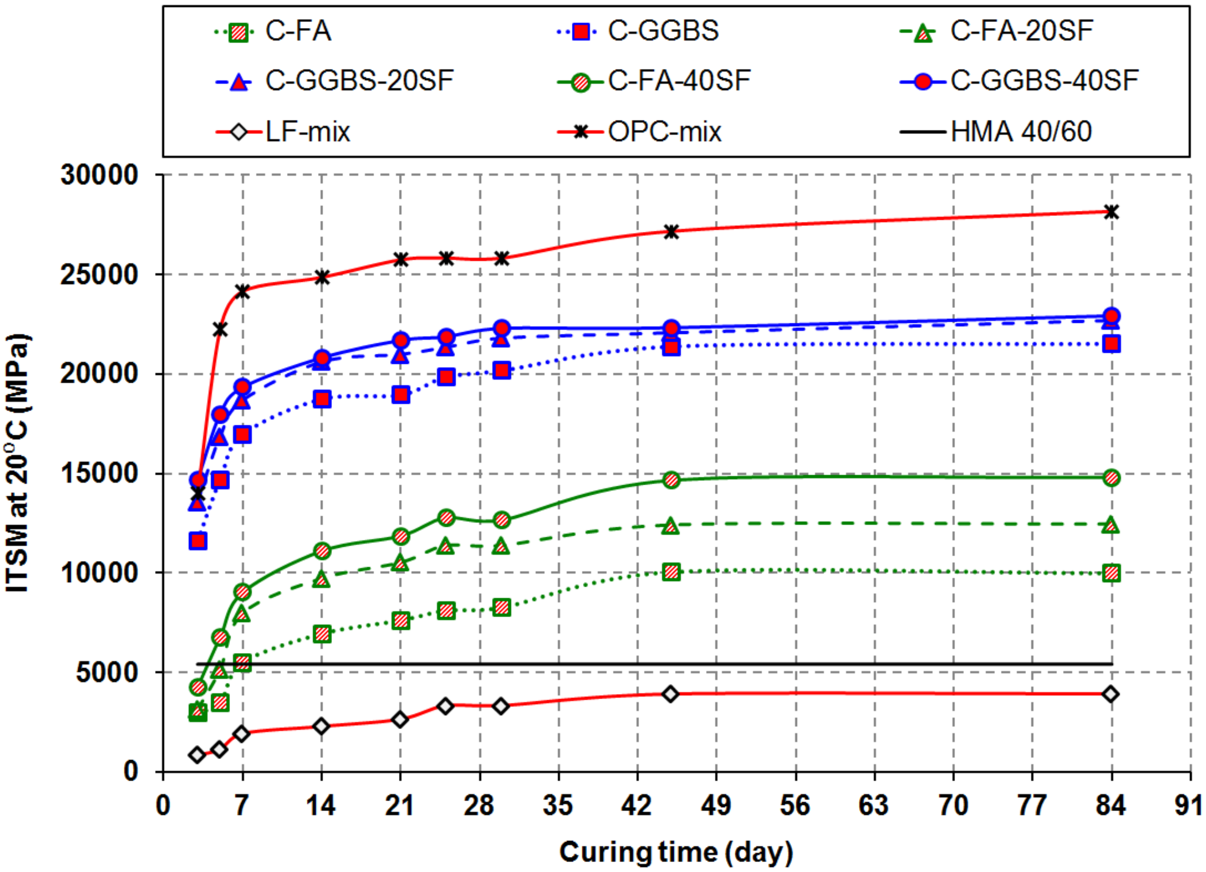

Fig. 4. The stiffness modulus development of studied mixtures

Low early stiffness is one of the main drawbacks of cold asphalt mix containing LF, as is clear in Fig. 4. However, a remarkable stiffness increase was achieved within only one week of curing using both BBF and TBF. The obtained stiffness for these mixes can contribute effectively to minimizing the curing time in the field. For example, the early stiffness of C-FA, C-FA-20SF and C-FA-40SF are approximately 3 to 5 times higher than untreated LF-mix, and these become 13 to 17 times in C-GGBS, C-GGBS-20SF and C-GGBS-40SF mixes during the first week. These results might be attributed to the pozzolanic reaction caused by FA, GGBS and SF in combination with low cement content to produce another hydraulic binder (see section 5.5). These findings are consistent to those obtained by Al-Busaltan, Al Nageim, Atherton and Sharples [6] and Al-Hdabi, Al Nageim, Ruddock and Seton [7]. However, the stiffness values were much higher than those found by Al-Busaltan, Al Nageim, Atherton and Sharples [6] and Al-Hdabi, Al Nageim, Ruddock and Seton [7]. This might be related to different factors such as the aggregate type, bitumen emulsion content and compaction method.

The results also demonstrated that the HMA 40/60 mix did not show any stiffness development during the curing period. The stiffness of this mix was constant from an age of 3 days until the end of the curing period. In comparison, the stiffness modulus of HMA 40/60 (5400 MPa) was surpassed by the OPC-mix and all the CAEMs including GGBS within the first three days. C-FA, C-FA-20SF and C-FA-40SF mixes achieved this value after 5-7 days whereas the untreated LF-mix never reached this stiffness value during the curing period. The stiffness of the blended mixes could, therefore, lead to reduced curing time in the field.

It is worth mentioning that the air voids of the treated CAEMs varied between 8.50% for OPC-mix to 10.30% for C-GGBS-40SF. However, the control LF-mix had 10.34%. The results demonstrate an improvement of volumetric properties for treated CAEMs which may affect positively on their performance. This improvement will be discussed later in section 5.5.

5.2. The effect of temperature on stiffness modulus

Stiffness modulus of CAEMs after 28 days of curing was evaluated at different testing temperatures, namely 5, 20 and 40^{o}C, as shown in Fig. 5. The stiffness modulus decreased with the increase of temperature. This trend is very strong in both HMA 40/60 and LF-mix. However, in treated CAEMs, the reduction in the stiffness depends on the type of filler. For example, stiffness reductions of only 28% and 43% resulted when heating C-GGBS and C-FA-40SF respectively from 5 to 40^{o}C. In contrast, HMA 40/60 and LF-mix lost 95-98% of their stiffness under the same condition. This might be considered as an excellent advantage in terms of the pavement performance in hot weather. This could potentially make the asphalt material less prone to cracking at low temperatures and less prone to rutting at high temperatures. These results are comparable to those published by other authors [14, 33, 34].

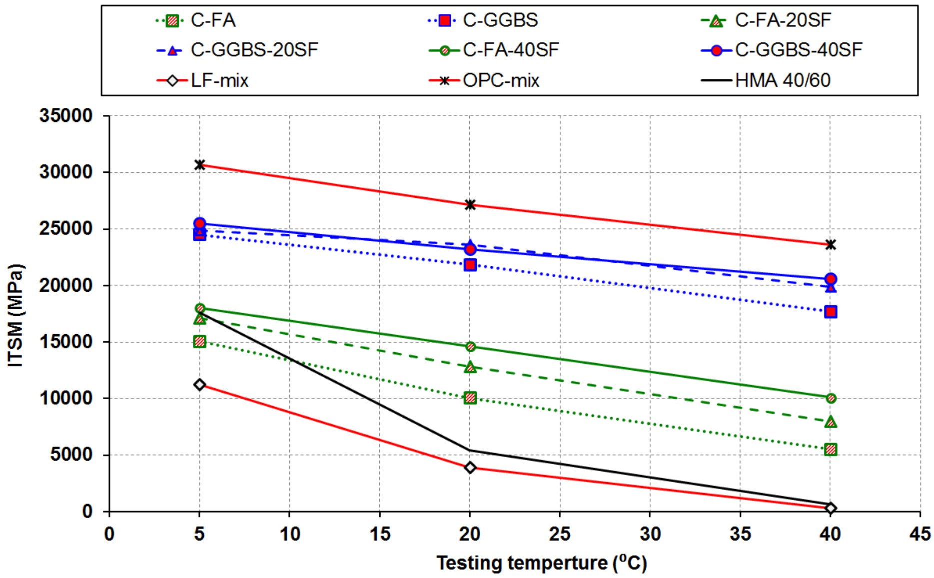

Fig. 5. Stiffness modulus under different testing temperatures

5.3. Resistance to permanent deformation

The results of the repeated load axial test are shown in Fig. 6 while the creep modulus of CAEMs and the percentage reduction in permanent strain relative to the LF-mix are displayed in Fig. 7.

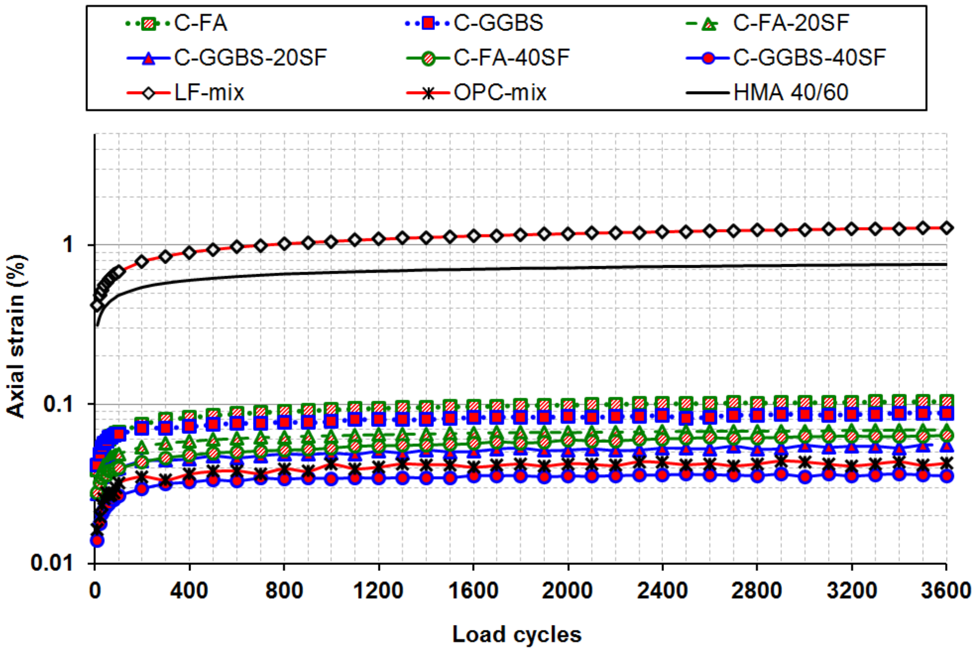

Fig. 6. The permanent strain in CAEMs under 100kPa stress

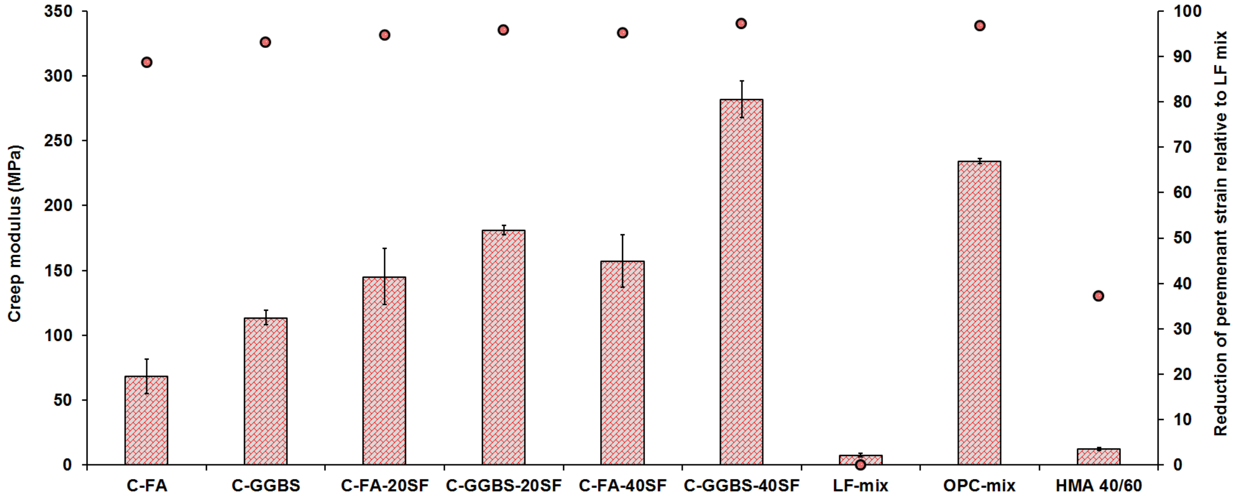

Fig. 7. The creep stiffness modulus of CAEMs and the percentage of reduction in permanent strain

The results, in Fig. 6, show that the incorporation of BBF and TBF resulted in a remarkable decrease in the permanent strains relative to the untreated LF-mix and HMA 40/60. These two mixes exhibited much higher strain values throughout the test. In mixtures containing BBF, the use of this type of filler combination resulted in a reduction in a permanent strain of 89-93 % relative to the LF-mix while the reduction was about 95-97 % for mixtures containing TBF, see Fig. 7.

The maximum achieved values of creep stiffness modulus (181.3 and 282.1 MPa) were recorded for the CAEM mix containing TBF (OPC plus GBBS) with 20%SF and 40%SF additions respectively as shown in Fig. 7. This may be attributed to the ability of GGBS mixtures to maintain a higher amount of trapped water inside the mixture in comparison to FA replacement [35]. This is more likely to enhance the hydration of cement. More interestingly, the resistance to permanent deformation for the C-GGBS-40SF mix is quite high compared to OPC-mix. This also might be related to the role of highly densified SF in producing a dense microstructure, particularly with the GGBS combination. Overall, it can be concluded that CAEMs treated with BBF and TBF have significantly decreased susceptibility to permanent deformation, indicating the potential benefit of using this material on heavily trafficked roads.

5.4. Durability against moisture and frost damage

Fig. 8 illustrates the ITS test results in both conditioned and unconditioned states (see Table 5). From this figure, the possible benefits gained from incorporating BBF and TBF fillers into CAEMs are clear.

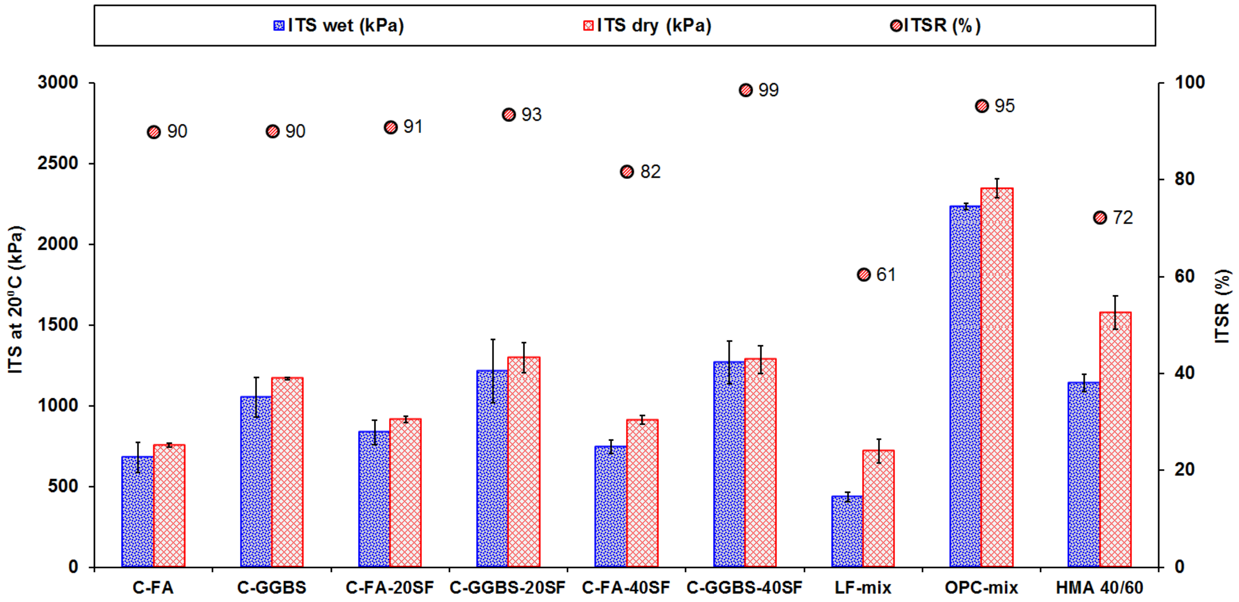

Fig. 8. The results of moisture damage for CAEMs

The ITSR values (ratios of wet to dry ITS) of treated CAEMs (with BBF and TBF) were higher than that for the untreated LF-mix and the equivalent HMA (HMA 40/60). Moreover, these values were comparable with the OPC-mix. These higher values of ITSR (the ability of the mix to maintain its strength after damage) may be related to the formation of new hydration products due to the incorporation of pozzolanic materials with cement. In addition, the temperature rises due to heating which would accelerate the hydration process of these active fillers. The results are consistent with trends noticed in concrete [36, 37]. This leads to the conclusion that CAEMs with these multi-blended fillers are less susceptible to moisture damage.

The ITS values before and after exposure to freeze-thaw cycles and corresponding ITSR values are shown in Fig. 9. Overall, the trend of the results is approximately similar to those of moisture damage presented in Fig. 8. It can be seen that the ITSR values of HMA 40/60 and CAEMs treated with BBF, TBF and 5% of OPC are in the range from 77% to 105%. However, LF-mix shows lower ITSR value of 53%. Thus, it can be concluded that incorporating waste by-product fillers had a significant positive effect on making CAEMs less susceptible to frost damage and stripping problems. Furthermore, the C-GGBS-40SF mix showed the highest resistance to exposure to both moisture and freeze-thaw damage which might be attributed to the same reason suggested in the discussion relating to Fig. 4. Further, it should be noted here that the microstructural evaluation in section 5.5 supported the positive impact of adding the (OPC+GGBS and SF) on the internal structure of TBF-CAEMs.

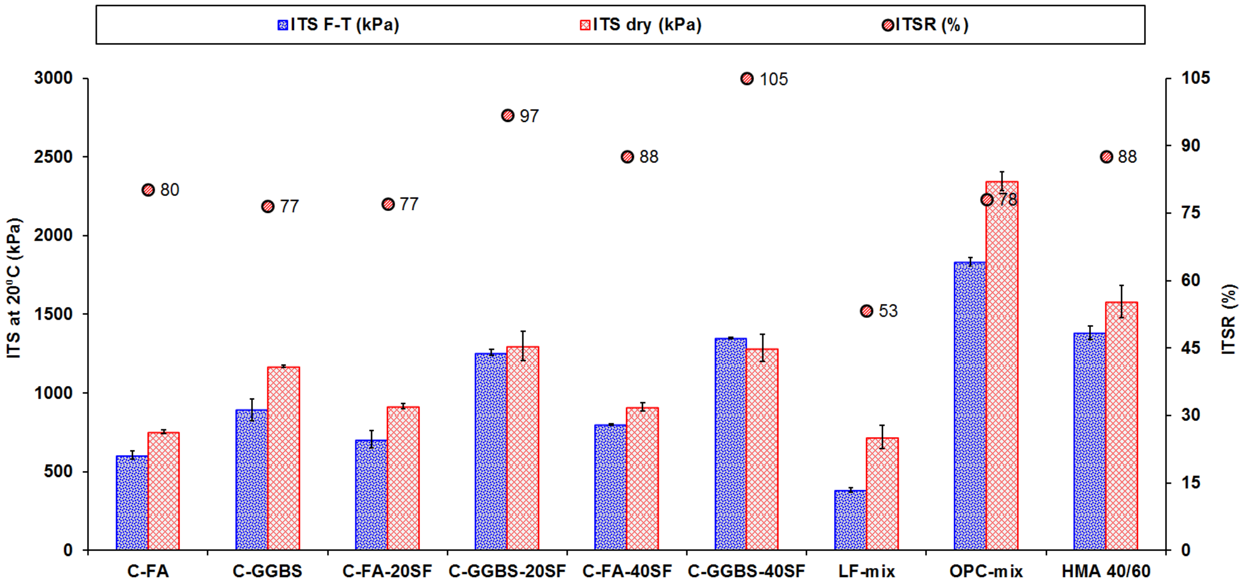

Fig. 9. The results of frost damage for CAEMs

There is no universally accepted minimum level of ITSR to define a CAEM as a durable material for structural purposes in a pavement exposed to severe conditions. However, the authors would suggest that CAEM should be considered a sufficiently durable material against moisture and frost damage if the values of ITSR for both types of damage are more than 70%. A similar suggestion of minimum acceptable ITSR is found in the literature for HMA [38, 39]. Under this proposed criterion, all the produced CAEMs in the present study are relatively resistant to moisture and frost damage except the untreated LF-mix.

5.5. XRD analysis and SEM examination

Fig. 10 shows the XRD patterns for CAEM mixtures while Fig. 11 illustrates the morphology of their internal microstructure in detail.

The XRD analysis demonstrated a presence of $Ca(OH)_2$ (Portlandite) with a high reference intensity peak in the OPC-mix indicating a lower degree of hydration compared to binary and ternary CAEMs. The $Ca(OH)_2$ in all the inspected specimens was identified by a reference intensity peak at 34.1° 2-theta scale [40]. The intensity values at 34.1° were reduced significantly in each C-GGBS, C-FA, C-GGBS-40SF and C-FA-40SF compared to OPC-mix. This reduction might be explained by the reaction of $Ca(OH)_2$ with pozzolanic materials. Another potential reason is that BBFs and TBFs, which contain low cement content, lead to reduce the $Ca(OH)_2$ availability. Overall, these results demonstrate a reduction in the concentration of undesired hydration products such as large crystals of $Ca(OH)_2$).

However, SEM examination (Fig. 11e) demonstrated a tiny presence of hexagonal crystals of $Ca(OH)_2$ compound in the BBF-FA mixture. The absence of this compound in the XRD pattern of the reference LF-mix is not an indicator of a higher degree of hydration, but it is certainly due to the absence of cement in this mixture. The higher degree of hydration obtained in the BBF and TBF mixtures as deduced from XRD patterns and SEM observations led to enhancement of most engineering features. This might be as a result of the pozzolanic activity of the FA, GGBS and the very high reactivity of SF. Active fillers and mineral admixtures can efficiently consume the $Ca(OH)_2$ phase in the cement matrix and produce a higher degree of hydration and dense internal microstructure with less porosity. This might be achieved by producing an additional CSH (cement gel) in the presence of water. Both Ca/Si ratio and the available water molecules in the new CSH products might give different features compared to the original CSH hydration product [37]. However, the growth and development of the hydration products in CAEMs, due to the presence of bitumen emulsion, might differ from those of pure cement or cement-filler matrices [18]. Thus, it would be interesting to identify the changes in the formation of the hydration products in CAEMs as well as these due to the addition of active fillers in these mixtures.

From this perspective and for a pure cement-bitumen matrix, the SEM examination in Fig. 11a-d suggested that the presence of the bitumen might not affect the hydration of the silicates in cement. The formation of additional CSH was observed due to the replacement of conventional LF by cement and active fillers. However, it seems that it can cause a delay in the formation of other hydration products (Ettringite) resulted from the hydration of aluminates in cement (as shown in Fig. 11d). Further research is needed to explain the change in cement hydration mechanism in the presence of bitumen more deeply.

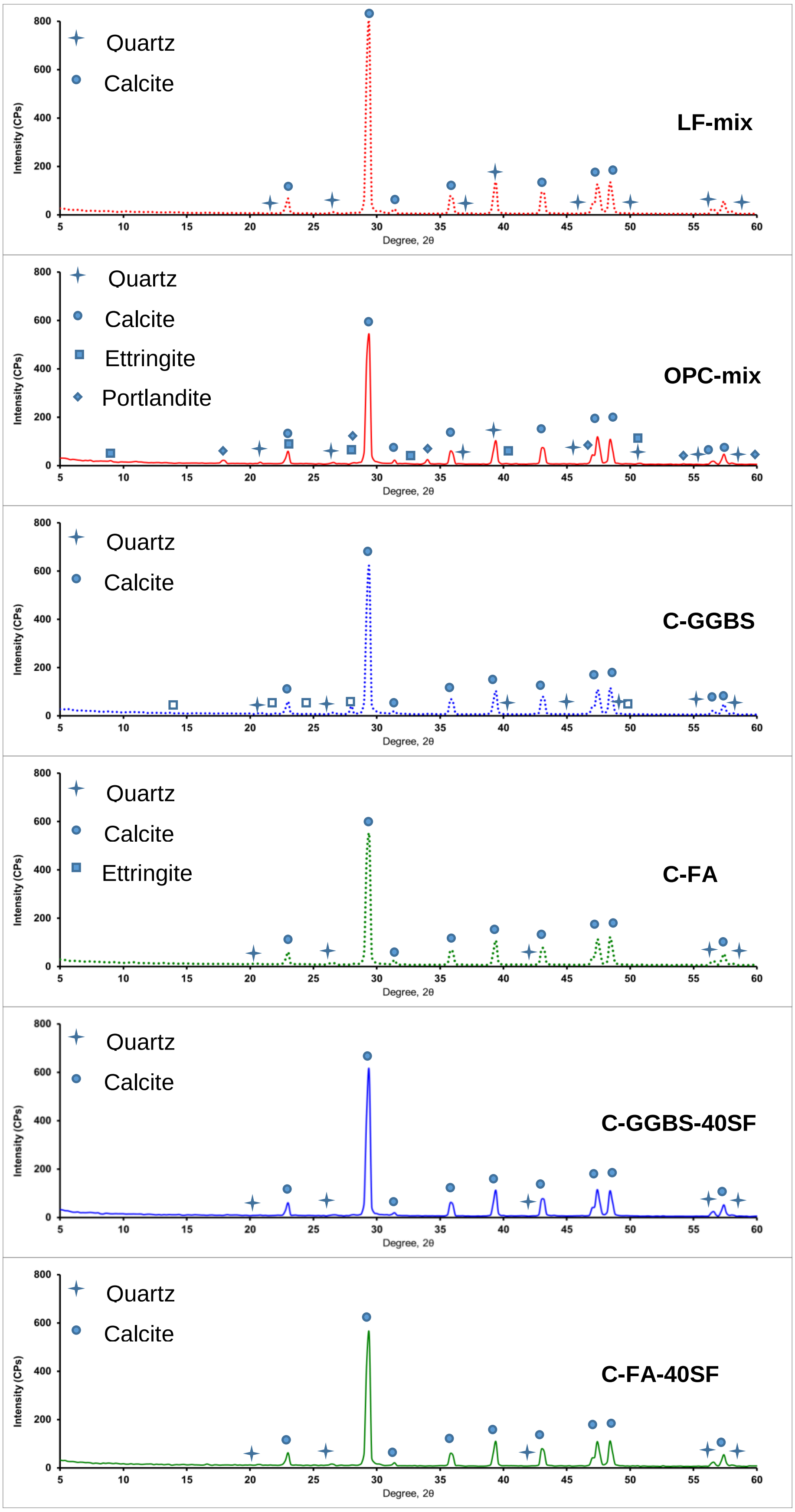

Fig. 10. XRD results of CAEMs

The SEM examination (Fig. 11c, d and e) showed that, even after 28 days of curing, the presence of Ettringite with needle shape (AFt) was clearly observed in many places in the binary blended matrices. This is especially in the capillary voids of the FA-BBF mixture, in contrast to the reference LF-mix and OPC-mix. The presence of the AFt phase at later ages indicates to a delayed reaction, and the resulting AFt phase is not an expansive hydration phase at ambient temperature [41]. Thus, it can improve the volumetric properties (less porosity) of the CAEM by minimising both the pore sizes and their continuity, see section 5.1. This consequently inhibits the movement of water and other types of aggressive fluid into the mixture and can thus effectively improve the durability of cold asphalt mixtures.

However, when exposed to heat due to hot/warm weather in the presence of water, AFt transforms to the second expansive form which is monosulfate with prismatic shape (AFm). This form had an increase of 2.3 times in volume [41]. The formation of internal stresses inside the capillary pores of the bitumen-cement matrix due to this volume increase and the susceptibility of AFt to dissolve in the presence of water might be a potential reason for less durable mixtures.

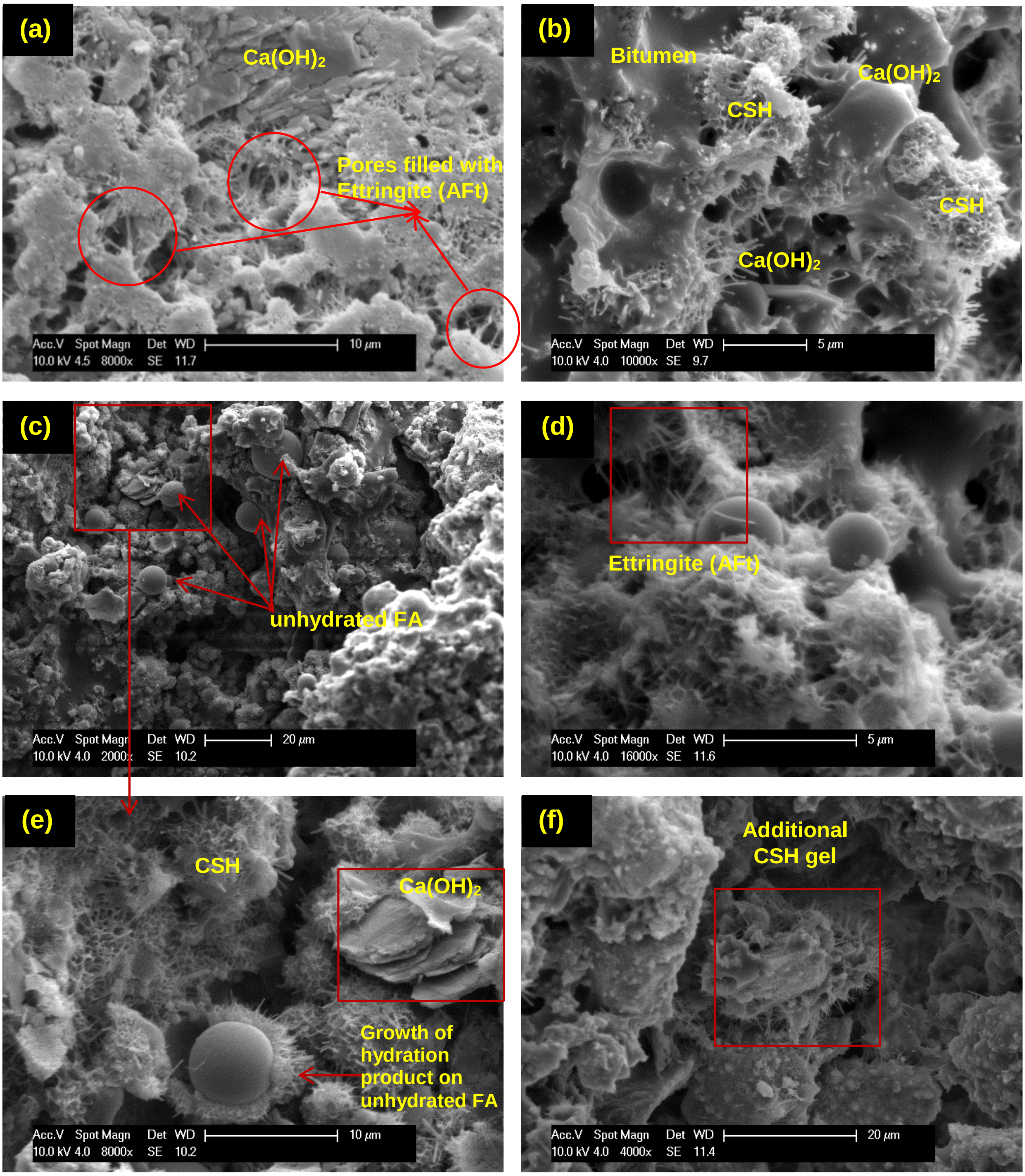

Fig. 11. Morphology details of the microstructure of CAEMs: (a) and (b) morphology of OPC-mix, (c) morphology of BBF mixture (C-FA) at low magnification, (d) and (e) morphology of BBF mixture (C-FA) at high magnification and (f) additional hydration products formation in TBF mixture (C-GGBS-40SF)

The SEM observation indicated that the deposition of AFt phase inside the capillary pores was more pronounced in the FA binary mixture in comparison with GGBS. However, it completely disappeared in the presence of SF addition for the TBF mixtures (both FA and GGBS). Instead, a more CSH gel formation was deduced as shown in Fig. 11f. Compared to other CAEMs, a very high intensity of the reference peak of $CaCO_3$ (calcite) was detected in the XRD pattern for the LF-mix (Fig. 10). This might indicate the non-reactivity of this filler and its inability to enhance the internal microstructure and chemical composition as the microstructural observation revealed. The low values obtained for most of these properties for the LF-mix, as presented previously, might support this hypothesis.

6. Conclusions

The current work aimed to investigate the impact of incorporating binary and ternary blended fillers (BBF and TBF) on both the engineering and microstructure properties of cold asphalt emulsion mixtures (CAEMs). The conclusions of the study can be summarized as follows:

- Significant enhancements were obtained in engineering properties due to the incorporation of by-product fillers, ground granulated blast furnace slag (GGBS) and fly ash (FA), in combination with cement in BBF mixtures, while further substantial improvements of such features were achieved by adding silica fume (SF) for TBF mixtures.
- The impact of the temperature increase, from 5 to 40°C, on the stiffness modulus was much greater in HMA 40/60 and LF-mix than in the other CAEMs investigated. After heating to 40°C, stiffness reduced by 96% and 93% for HMA 40/60 and LF-mix respectively whereas reductions of 28% and 43% were recorded for the BBF mix with GGBS and TBF with FA-40SF, respectively.
- The replacement of LF with TBF (OPC+GGBS+SF) showed lower axial strain than the other type of TBF (OPC+FA+SF) in the repeated load axial test.
- Treatment of CAEMs with BBF and TBF significantly decreased its susceptibility to permanent deformation by producing higher creep stiffness modulus, indicating the potential benefit of using this material on heavily trafficked roads. The maximum creep stiffness modulus was achieved in the TBF with both GGBS and SF.

- All the CAEMs produced in the present study were considered to be durable as the control hot mix (HMA 40/60) except the LF-mix; maintaining more than 70% of their strength after exposure to both moisture and frost damage.
- The combined results of stiffness modulus and the mineralogical and microstructural assessments suggested that the SF addition to BBF- CAEMs can eliminate any delay in hydration product formation (aluminates in cement) caused by bitumen emulsion in the CAEMs. However, the effect on the other hydration products (silicates in cement) might be much close to the pure cement matrix without bitumen emulsion.
- Further investigations are needed to examine the hydration products in CAEM at nano-scale to understand the effect or presence bitumen emulsion on the structure of hydration products.

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