Investigation into the stiffness improvement, microstructure and environmental impact of a novel fast-curing cold bituminous emulsion mixture

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Digital Object Identifier (DOI): dx.doi.org/10.14311/EE.2016.425

ABSTRACT

Cold bituminous emulsion mixtures (CBEMs) have environmental advantages over conventional hot mix asphalt as they are cold products and are safe to use whilst also reducing energy. However, their low early strength and long curing time have been cited as the main barriers for their wide utilisation. This study describes the development of a new fast-curing CBEM incorporating binary blended cement filler (BBCF) containing high calcium fly ash (HCFA) and high aluminosilicate waste material (HASW) to replace the commercial limestone filler. In addition, a waste alkaline NaOH solution was used as a replacement for the pre-water. The new CBEM needs only ambient temperature curing in order to achieve strengths comparable to the traditional hot mixture in a very short time (less than one 1 day). Water sensitivity of the mixtures was assessed by the Stiffness Modulus Ratio (SMR). Scanning electron microscopy (SEM) was employed to characterise the surface morphologies. Furthermore, the environmental impact of using these waste materials in CBEMs was assessed by using a toxicity characteristic leaching procedure (TCLP).The results indicated a significant improvement in the indirect stiffness modulus (ITSM) test and water sensitivity compared to hot mix asphalt. Moreover, SEM analysis confirmed the formation of hydration products after various curing ages. Furthermore, the TCLP test for environmental impact revealed that the heavy metal concentration in the leachates satisfied the required criteria.

Keywords: Cold Asphalt, Emulsions, Health Safety and Environment, Leaching, Waste

1. INTRODUCTION

Cold asphalt mixtures are defined as bituminous materials mixed utilising cold aggregates and binder (Jenkins, 2000). Cold bitumen emulsion mixtures (CBEMs) are one of the general types of cold asphalt mixtures. However, they are usually considered inferior to hot mix asphalt (HMA) as a result of their weak early life strength, long curing times necessary to accomplish the optimal performance and the high air-void content that remains after compaction (Thanaya et al., 2009). Cement has been used widely in order to reduce these disadvantages (Terrel and Wang, 1971, Schmidt et al., 1973, Brown and Needham, 2000, Oruc et al., 2007, Al-Hdabi et al., 2014, Fang et al., 2015). However, cement production is an intensive activity and has negative environmental impacts in relation to both the material and energy produced. In addition to this, waste materials usage in road pavement construction has become an essential subject recently due to the decrease in natural resources in addition to ecological concerns.

Chemical activation shows that some chemicals can be used to activate the possible reactivity of cementitious constituents (Sajedi and Razak, 2011). Alkali-activated materials have been shown to have improved mechanical characteristics at higher levels than cement. Accordingly, alkali activation of fly ash offers a potential financial and environmental cost saving when used as a cement substitution (Davies, 2011).

The environmental impact of including waste materials in pavement layers is a significant issue that must be considered for a number of reasons. For example, these pavement layers are in continuous direct contact with the surface and also with underground water, which increases the hazard of water pollution through leaching of harmful materials such as heavy metals into underground water resources (Modarres et al., 2015a). Leaching can be defined as the mechanical or chemical process of removal of a waste constituent from a solidified matrix into a solution, by the passage of a solvent (Ling and Poon, 2014). A waste can be categorised as a hazardous waste or not by using TCLP as the main tool to decide that (Xue et al., 2009). Xue et al. (2009) considered the effects of municipal solid incineration ash in a stone mastic asphalt mixture by performing a TCLP test for controlling the leachate quality. They found that there could be a high risk of water pollution from leaching of dangerous ingredients such as heavy metals into underground water resources.

The goal of this study is to develop a fast-curing CBEM mixture by incorporating an alkali-activated binary blended cement filler generated from two waste materials (HCFA and HASW) for binder course materials in heavily trafficked road pavement, with the target of decreasing the disposal of wastes and ensuring that raw materials will be conserved, which will assist in sustainable development. This study focuses on the ITSM of such mixtures, which could affect the CBEM performance. The microstructure was studied on fracture surfaces from the binder pastes by using a scanning electron microscope. In addition, the toxicity characteristic leaching procedure (TCLP) test can be considered, according to the literature, as one of the major leaching procedures that can be employed to examine the possible of heavy metal leachability from stabilised layers (Xue et al., 2009, Modarres et al., 2015a). The leaching test is the prime and most commonly used pointer of chemical stability and therefore the possible environmental effects of the treated layer (Ilyas et al., 2014).

2. MATERIALS

2.1 Aggregate

The gradations of the granite aggregates used in this research are presented in Table 1. As for the asphalt mixtures, asphalt concrete [BS EN 13108-1 specifications (European Committee for Standardization, 2006)] was considered as the binder course. The physical specifications of the crushed coarse and fine aggregates are listed in Table 2.

Table 1: Aggregate grading for AC 20 mm dense binder course BS EN 13108-1

Test sieve aperture size, mm	Mass passing specification range, (%)	Mass passing mid, (%)
20	99-100	100
10	61-63	62
6.3	47	47
2	27-33	30
0.250	11-15	13
0.063	6.0	6.0

Table 2: Physical characteristics of the granite aggregate

Material	Property	Value
Coarse aggregate	Bulk particle density, Mg/m^3	2.62
	Apparent particle density, Mg/m^3	2.67
	Water absorption, %	0.8
Fine aggregate	Bulk particle density, Mg/ m^3	2.54
	Apparent particle density, Mg/ m^3	2.65
	Water absorption, %	1.7
Traditional mineral filler	Particle density, Mg/ m^3	2.57

2.2 Bitumen emulsion and asphalt:

Cationic slow-setting bitumen emulsion (C60B5) was utilised in all the cold asphalt mixtures. The relevant properties of the chosen bituminous emulsion are shown in Table 3. In addition, 100/150 and 40/60 penetration grade bitumen was used in this research for the hot asphalt binder course. Table 4 illustrates the binder characteristics.

2.3 Fillers:

HCFA used in this study was derived from a power station in the UK while HASW is a waste material from the industrial sector. Table 5 shows the chemical composition of the two materials.

Table 3: Properties of (C60B5) bitumen emulsion

Description	(C60B5) bitumen emulsion
Type	Cationic
Appearance	Black to dark brown liquid
Base bitumen	100/150 pen
Bitumen content, (%)	60
Particle surface electric charge	Positive
Boiling point, (˚C)	100
Relative density at 15 ˚C, (g/ml)	1.05

Table 4: Properties of 40/60 and 100/150 penetration grade bitumens

Bituminous binder 40/60		Bituminous binder 100/150	
Property	Value	Property	Value
Appearance	Black	Appearance	Black
Penetration at 25 (°C)	49	Penetration at 25 °C	131
Softening point, (°C)	51.5	Softening point, °C	43.5
Density at 25 °C	1.02	Density at 25 °C	1.05

Table 5: EDXRF analysis of the chosen filler materials, %

Properties	CaO	SiO_2	Al_2O_3	MgO	Fe_2O_3	SO_3	K_2O	TiO_2	Na_2O
HCFA	70.417	26.003	2.552	2.988	0	0.357	0.279	0.497	1.918
HASW	0.047	35.452	44.167	0.684	0.368	0	0.049	0	0

2.4 Waste sodium hydroxide (NaOH) alkali

Sodium hydroxide (NaOH) alkali waste solution produced from an acid neutralisation plant containing ≤ 8% NaOH in water was used as the alkali activator.

3. PREPARATION OF SPECIMENS

The method adopted by the Asphalt Institute (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design (MS-14)) (Asphalt Institute, 1989) was used to design the cold asphalt concrete binder course bituminous emulsion mixtures. Various pre-wetting water contents were examined to find the lowest ratio and, as a result, adequate coating will be confirmed. Moreover, indirect tensile stiffness modulus tests were used to decide the optimum emulsion content, and the mix density test was used to determine the optimum total liquid content at compaction. According to this procedure, pre-wetting water content, optimum total liquid content at compaction and optimum residual bitumen content were 3.5%, 14% and 6.3%, respectively. Incorporation of the HCFA was carried out with replacement of the conventional mineral filler with (6%) of HCFA by total mass of aggregate, while HASW was used as the activator in four percentages (1%, 2%, 3% and 4%) by dry aggregate weight. The final stage is replacing the pre-water with the waste alkali solution to make an alkali-activated binary blended cement filler (ABBCF).

The materials were mixed in a Hobart mixer. Aggregate, filler and pre-wetting water content were added and mixed for 1 min at low speed. After that, bitumen emulsion was added progressively throughout the next 30 seconds of mixing, and the mixing was continued for the next 2 min at the same speed. In addition, the samples were mixed and placed in the mould, and after that they were directly compacted with 100 blows of the Marshall hammer, 50 on each side, using a standard Marshall Hammer (impact compactor). The specimens were then left in their moulds at room temperature (20±1°C) for 1 day. After this, they were extruded and left in the room for the required curing time.

An indirect tensile stiffness modulus (ITSM) test was conducted to examine the effect of replacement of conventional mineral filler with HCFA and the effect of HASW addition. Furthermore, the effect of the alkali activated of the BBCF was studied by means of ITSM test.

The results were compared with a standard AC 20 hot dense binder course. Consequently, two types of hot binder course mix, namely AC 20 dense binder course 100/150 and AC 20 dense binder course 40/60, were used throughout the research with the same aggregate type and gradation. 4.6% optimum binder content by weight of aggregate was used according to the PD 6691:2010 (European Committee for Standardization, 2010) for the AC 20 dense binder course. Asphalt samples were fabricated and compacted at lab temperature (20 ºC), whereas the 100/150 and 40/60 hot mixtures were mixed at (150–160 ºC) and (160–170 ºC), respectively. In addition, a cold asphalt concrete binder course containing limestone was used for comparison. Every indirect stiffness modulus test value is the average of 3 specimens to ensure the reliability of the results.

Conversely, microstructural analyses were carried out by using Scanning Electron Microscopy (SEM) on the chosen paste samples taken from the crushed samples to identify the changes in the materials at different curing ages. Appropriate pieces were taken off the paste at certain ages, e.g., 3 and 28 days, for SEM examination. These fragments were undisturbed and taken from the centre of the paste specimens to guarantee that they would suitably characterise the paste samples.

4. METHODS

4.1 Indirect tensile stiffness modulus (ITSM) test

The ITSM test is a non-destructive test; indirect tensile stiffness is commonly recognised as a very important performance property of bituminous paving materials and it can be utilised as an indication of the load-spreading ability of bituminous paving layers. The test was conducted in accordance with BS EN 12697-26 (European

Committee for Standardization, 2012) using Cooper Research Technology HYD 25 testing apparatus, as shown in Figure 1. The test conditions were as shown in Table 6.

Table 6: ITSM test conditions

Item	Range
Specimen diameter, (mm)	100 ± 3
Rise time, (ms)	124 ± 4
Transient peak horizontal deformation, (µm)	5
Loading time, (s)	3-300
Poisson's ratio	0.35
No. of conditioning plus	10
No. of test plus	5
Test temperature, (°C)	20 ± 0.5
Specimen thickness, (mm)	63± 3
Compaction	Marshall 50 blows/face
Specimen temperature conditioning	4hr before testing

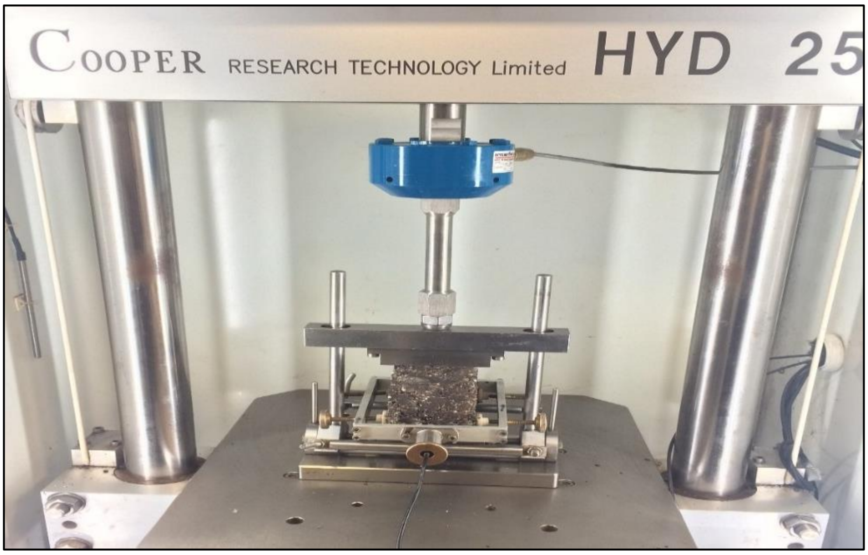

Figure 1: ITSM apparatus

4.2 Moisture damage resistance

A water susceptibility test was carried out following a standard procedure in EN 12697-12 (European Committee for Standardization, 2008) to evaluate the susceptibility of the study mixtures under the effect of moisture. This test determines the influence of saturation and accelerated water conditioning on the ITSM of cylindrical samples of the mixtures, which are tested following EN 12697-26 (European Committee for Standardization, 2012). Tested samples were divided into two sets; the first set of specimens were left in the mould for 1 day before extraction and then cured at 20 °C for 7 days before the ITSM test was carried out at 20 °C. These specimens were then left to cure at ambient temperature and thus represent a dry condition. Conversely, the other samples were left in the mould for 1 day before extraction and cured at 20 °C for 4 days before being exposed to a vacuum (with 6.7 kPa pressure for 30 minutes) and left submerged in the glass jar for an additional 30 minutes. Next, they were conditioned at 40 °C for 3 days before testing, which represents a wet condition. The water susceptibility of the mixtures was measured by finding the stiffness modulus ratio (SMR), which is determined as the ratio of the stiffness of the conditioned samples to the stiffness of the dry samples.

4.3 Scanning Electron Microscopy (SEM) analyses

The technique of SEM imaging has been used commonly in the cement research area for petrographic analysis of hardened products and to assess the degree of hydration (Sarkar et al., 2001, Sánchez de Rojas et al., 2006, Kar et al., 2012).

The scanning electron microscopy (SEM) approach, which is a technique for high-resolution imaging of surfaces illustrating the microstructure morphology of the particles and surface characterisation of materials, was used to study the microstructure of the new ABBCF paste.

SEM images were collected with the aid of an Inspect scanning electron microscope. The SEM is a type of electron microscope that provides photographs of the surface of the paste specimens by scanning them with a high-energy beam of electrons across the sample surface.

SEM applies a concentrated beam of high-energy electrons to produce a diversity of signals at the surface of solid test samples. The signals arising from electron-sample interactions provide information about the sample, containing outside morphology (texture), chemical structure, and crystalline structure and direction of materials.

4.4 Toxicity characteristic leaching procedure (TCLP) test

TCLP is one of the major leaching technique tests used to investigate the risk of leachability of heavy metals from stabilised layers (Xue et al., 2009). In this study, a TCLP test has been applied to determine the leached concentrations of cadmium (Cd), chromium (Cr), lead (Pb), arsenic (As), barium (Ba), nickel (Ni), copper (Cu), and zinc (Zn) from the new CBEM according to the procedure adopted by Xue et al. (2009) and Modarres and Ayar (2014) to analyse the possibility of heavy metal leachability from the CBEM mixtures. This procedure is based on the US Environmental Protection Agency method, SW846-1311.

In this research, the TCLP test was first conducted on both the HCFA and HASW samples. In addition, the test was performed on ABBCF samples containing the waste materials. To achieve the targeted plan, ABBCF specimens were prepared in the laboratory and these specimens in addition to the original two waste materials were then tested using the toxicity characteristic leaching procedure (TCLP). A stock of TCLP leachant was prepared by mixing stoichiometric amounts of deionised water and acetic acid (pH 2.88) supplied by Hach-Lang and used as supplied. Weighted amounts, 10 grams, of the crushed specimens were placed in bottles that contained 200 mL of the TCLP leachant. These bottles, which contained the crushed specimens and the TCLP leachant, were agitated using a rotary extractor (type: Labent 222 DS) at 30 rpm for 18 h (Figure 2). All experiments were conducted at a temperature of 20 °C, which was precisely controlled by the rotary extractor precisely. After the extraction process, the solutions were filtered using a 47 mm glass fibre filter supplied by Hach-Lang. Then, the filtrates were acidified using acetic acid to pH below 2. The concentrations of the heavy metals were measured using an atomic adsorption spectrophotometer (type: Thermo, Model: ICE 3300).

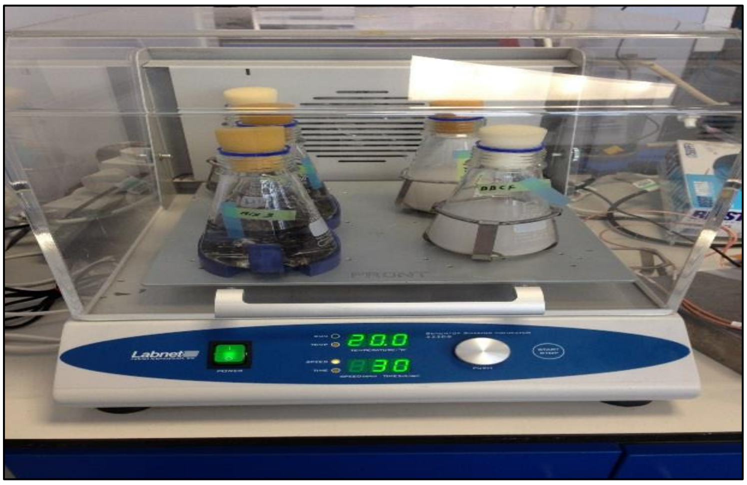

Figure 2: Rotary extractor

5. RESULTS AND DISCUSSION

5.1 ITSM performance

The first stage of the research considered the influence of the replacement of the commercial mineral filler with HCFA on the stiffness modulus of the cold asphalt mixtures. HCFA was introduced at 6% by dry weight of aggregate. The ITSM test was run in accordance with BS EN 12697-26 (European Committee for Standardization, 2012) at age 3 days.

The results of the ITSM tests are shown in Figure 3; they reveal that the ITSM for 6% HCFA replacement after 3 days is around 17 times the reference for untreated cold mix asphalt. This enhancement is due the generation of a new binder by the hydration activity of HCFA as well as the absorption of the trapped water by this filler.

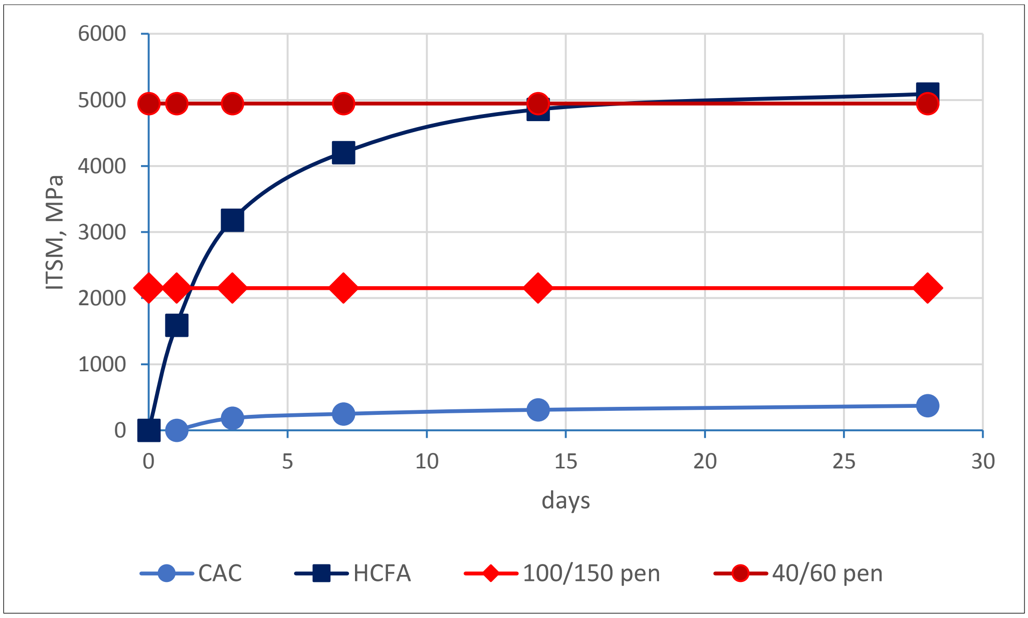

Figure 3: Effect of HCFA time on ITSM at 20 ºC after 3 days

The second stage employed the HASW as supplementary cementitious material to activate the HCFA filler. HASW was introduced at 1%, 2%, 3% and 4% by dry weight of aggregate, as can be seen in Figure 4. A new binary blended cement filler (BBCF) was generated with 4.5% HCFA and 1.5% HASW and reached maximum stiffness at 3 days. The reason for the improvement is that the pozzolanic constituents of HASW react with $Ca(OH)_2$ released during the hydration operation of HCFA and speed up the hydration of the HCFA particles and more hydrated products are generated. A balanced oxide composition was expected to be formed at this composition within the BBCF.

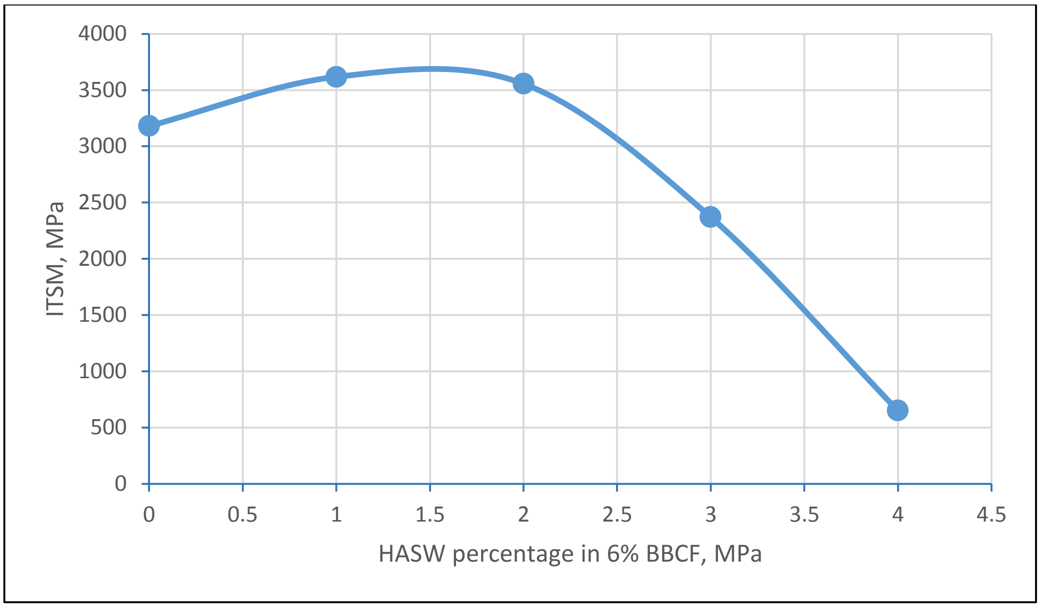

Figure 4: Effect of HASW on BBCF at 20 ºC after 3 days

The third step aimed at applied alkali activation for further improvement of the BBCF by using waste alkali sodium hydroxide (NaOH) as a replacement for the 3.5% of the pre-wetting water content. Alkali-activated binders offer the opportunity to employ waste materials, because the properties of materials based on alkali-activated binders are often greater than those of concrete and mortar prepared from standard Portland cement (Frantisek Skvára et al., 2003). Within an alkaline environment, glass phases of the particles of fly ash are expected to break and to react with $Ca(OH)_2$, consequently generating C-S-H gel. Figure 5 shows that CBEM with alkali-activated binary blended cement filler (ABBCF) developed a higher stiffness (approximately 33%) than BBCF mixtures after 3 days with 100% pre-water replacement by the waste NaOH solution.

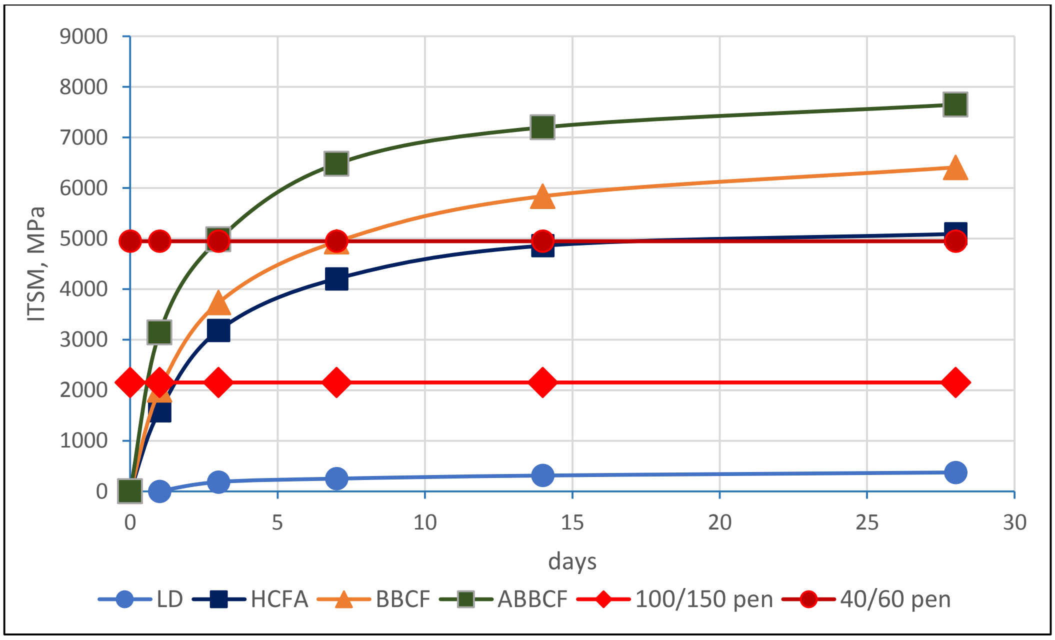

Figure 5: Effect of curing time on ITSM at 20 ºC after 3 days

5.2 Durability performance

The durability of the mixtures in terms of moisture susceptibility was measured by determining the SMR following a standard procedure in BS EN 12697-12 (European Committee for Standardization, 2008). It is remarkably shown in Figure 6 that the SMR for the ABBCF-treated mixture is more than 100%. The results are better than for the hot asphalt concrete binder course specimens and meet the requirements for bituminous mixtures. This positive result can be clarified with the presence of HCFA, HASW and NaOH, which produce strong adhesion bonds that develop inside the mixtures. Furthermore, submerging the samples in water activates the hydration process for the mixtures, and conditioning the samples at high temperatures also activates the hydration process.

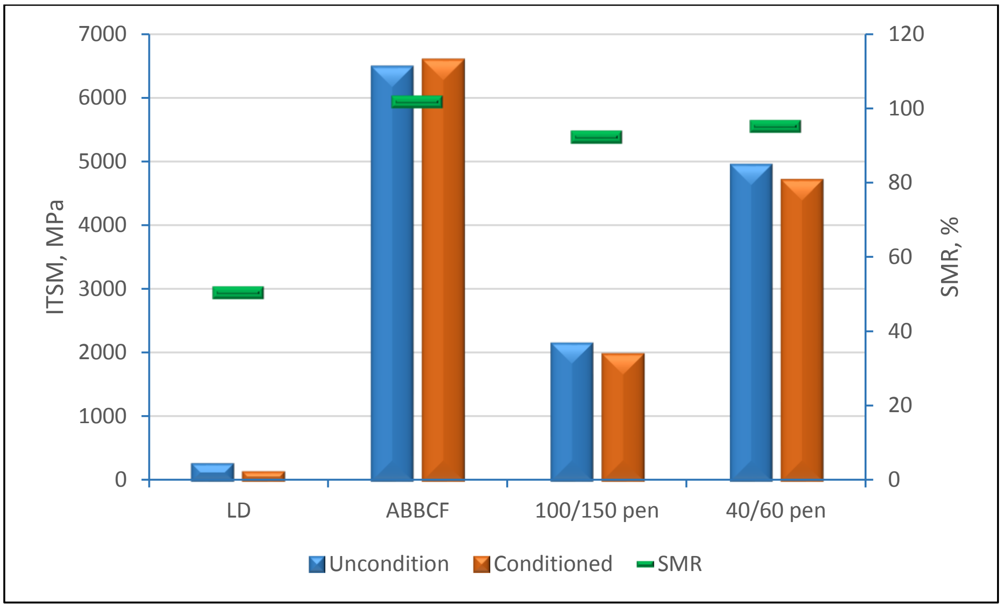

Figure 6: Water sensitivity results

5.3 SEM observation

The microstructures of the representative fractured surfaces of the ABBCF samples and the raw materials present information about the chemical reaction achieved. Figure 7 illustrates the microstructures of the original raw materials and ABBCF samples after 3 and 28 days. It can be seen from the 3 days photographs that the ABBCF particles have started to react, and the particles were found to have dissolved and to have produced good quantities of hydrated products and generated a dense material. On the other hand, the SEM photographs after 28 days reveal that the surfaces of the ABBCF particles had changed into C-S-H gel. This is the result of the reaction of the active BBCF and NaOH. Accordingly, the ABBCF showed the improved properties.

5.4 TCLP results

Heavy metal concentrations in the leachates of the TCLP test are shown in Table 7. It can be observed that there was a high concentration of Sr in the HCFA leachate. This concentration was lower in the BBCF as the HCFA amount was reduced in this filler. On the other hand, the concentrations of heavy metals in the leachate of the HCFA-, BBCF- and ABBCF-treated mixtures were considerably reduced; they were several times less than those of the HCFA and BBCF ones, and met the requirements of the standard limits and will be within the regulatory levels (Modarres and Nosoudy, 2015, Modarres et al., 2015b). According to the atomic adsorption, Sr concentration in the raw materials was higher than the concentration of the other heavy metals, which explains the high Sr leachate concentration compared to the other heavy metals. In addition, this result highlights that the Sr-capturing capacity of the prepared samples was not enough to hold all the Sr. However, the Sr leachate concentration was not an issue.

This means that these mixtures could stabilise and solidify the heavy metals found in both the HCFA and the BBCF.

Based on the results, it can be established that not only did the application of the three waste materials in the new CBEM have technical advantages, it also decreased the harmful effects on the environment.

It should be noted that the regulatory requirement relating to Sr levels was not stated in the TCLP test

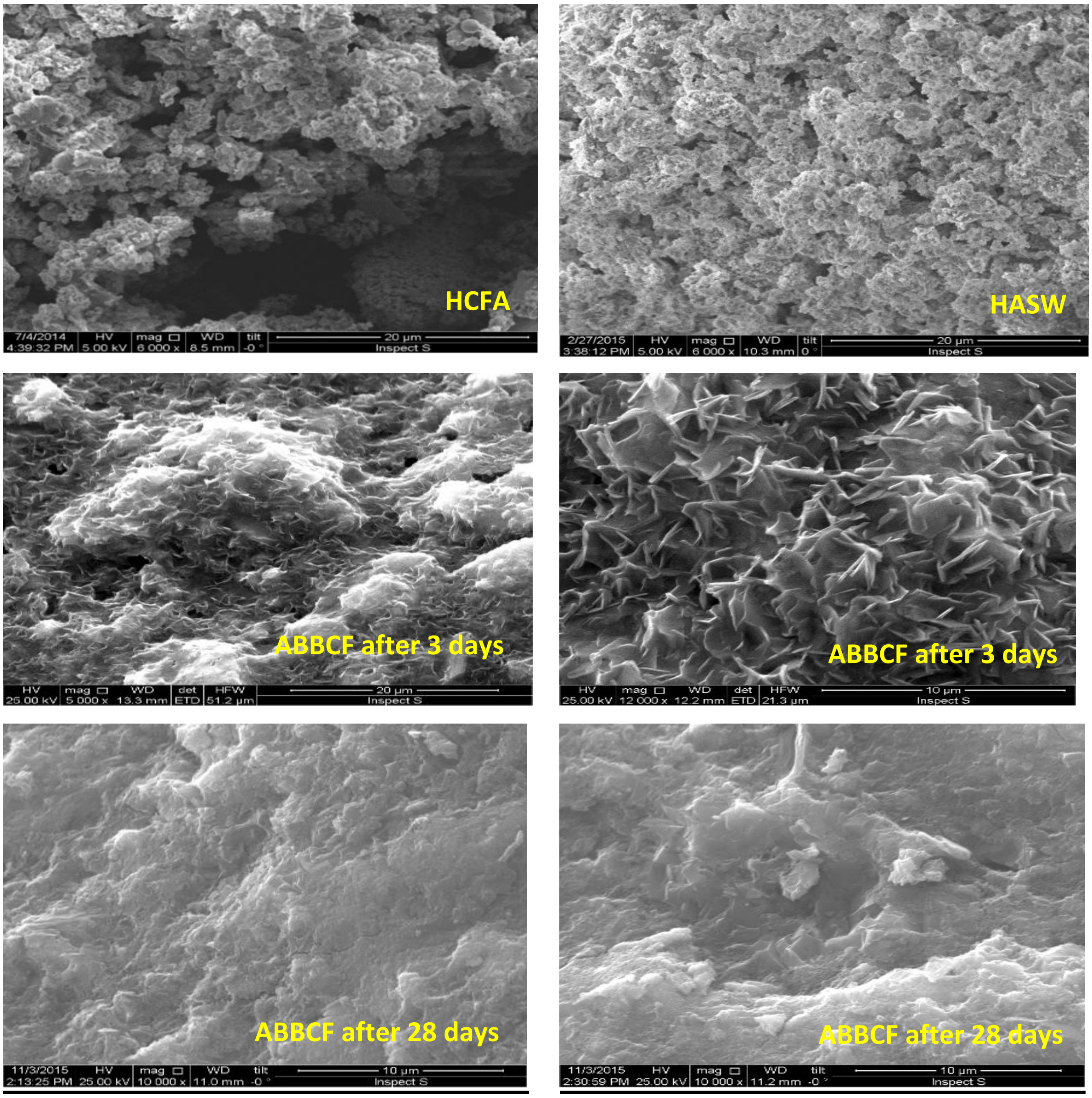

Figure 7: SEM photos

Table 7: TCLP test results (mg/L)

Heavy metal concentration (mg/L)	Reference water quality	HCFA filler	BBCF filler	HCFA mixture	BBCF mixture	ABBCF mixture	TCLP regulatory level
Nickel (Ni)	0.0	0.007	0.004	0.0	0.0	0.0	25
Copper (Cu)	0.0	0.0	0.0	0.0	0.0	0.0	25
Lead (Pb)	0.0	0.0	0.0	0.0	0.0	0.0	5
Chromium (Cr)	0.0	0.020	0.017	0.009	0.008	0.008	5
Zinc (Zn)	0.0	0.0	0.0	0.0	0.0	0.0	25
Strontium (Sr)	0.0	5.841	3.616	0.367	0.257	0.087	-
Barium (Ba)	0.0	0.0	0.0	0.0	0.0	0.0	100
Cadmium (Cd)	0.0	0.0	0.0	0.0	0.0	0.0	1

6. CONCLUSION

The aim of this research is to develop an environmentally friendly cold bituminous emulsion mixture including waste materials as a replacement for the conventional limestone powder. The experimental study focused on both mechanical properties in terms of ITSM and environmental impacts, while SEM shows the microstructure characteristics. The conclusions can be summarised as follows:

1. The use of HCFA and HASW not only leads to desired ITSM, but also decreases the quantity of the pollutant waste deposits and their harmful effects on the environment.
2. A mixture of CBEM that incorporates the BBCF and is activated by a waste alkali solution can produce a cementitious material that is suitable to replace the limestone filler.
3. Water sensitivity of the ABBCF-treated mixture is more than two times that of the reference cold mixtures with limestone. Moreover, there is a significant enhancement of the ABBCF mixture where the stiffness modulus for the conditioned specimens is better than that of the unconditioned samples and accordingly the SMR is above 100%.
4. SEM provides indication of the presence of hydrated products that account for the ITSM development in the CBEM mixture that incorporates the ABBCF.
5. No detrimental effect on the surrounding environment was revealed through the TCLP test.

ACKNOWLEDGEMENTS

The financial support from the Ministry of Higher Education and Scientific Research and Kerbala University in Iraq for the first author is highly appreciated. Additionally, the authors wish to express their sincere gratitude to Jobling Purser, Colas and Francis Flower for the bitumen emulsion, aggregate and limestone filler that were kindly donated for the current study.

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