



WASTEWATER SLUDGE ASH IN THE PRODUCTION OF A NOVEL COLD MIX ASPHALT (CMA): DURABILITY, AGING AND TOXICITY CHARACTERISTICS

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ABSTRACT

New Sustainable with no CO₂ emission cold mix asphalt (CMA) for road and highways surfacing is successfully developed from wastewater sludge ash normally sent to landfills. The Paper reports; durability in terms of water sensitivity, aging and contamination levels of heavy metals of the new developed novel CMA containing upgraded fly and bottom ash secondary cementitious materials from United Utilities fly and bottom ashes. Road engineers currently restricted the use of conventional CMA containing conventional limestone fillers (LF) for use in walkways and light trafficked roads only, as these mixtures needs from 2 to 24 months for curing. Conventional CMA mixtures also have high voidage contents and thus low traffic bearing capacity. The durability of these mixes is low especially in the UK and European countries where the climate is predominantly wet and cold.

In Germany and France, ordinary Portland cement (OPC) replaced the filler used in traditional CMA. Although this allows the new CMA to be used in roads for light and heavy trafficked loads. The materials are not environmentally friendly, as one tone of cement releases approximately one tone of CO₂ emission during their manufacturing process.

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The results of the new developed CMA are satisfying the British and European codes of practices requirements. The new novel mixtures are also; highly cost-effective compared with the use of Hot Mix Asphalt (HMA), has no CO₂ emission during mixing construction, and has no health and safety issues compared with the HMA manufacturing and application process.

KEYWORDS: Ageing of asphalt, cold mix asphalt, high metal contamination, toxicity characteristics, water sensitivity.

1. INTRODUCTIONS

Cold mix asphalt (CMA) is a mixture of bituminous materials mixed and compacted at ambient temperature (Al Nageim et al., 2012; Al-Busaltan et al., 2012a; Dulaimi et al., 2020). Its main binding agent is a bituminous emulsion that normally contains approximately 20% of its dry contents water. CMA mixtures normally take 2 to 24 months to cure and produce a material with low strength, high voids and low durability (Head, 1974; Al-Busaltan et al., 2012b; Al-Nageim et al., 2019). Therefore, road engineers only allow such mixtures to be used in low trafficked pavements such as car parks and walkways.

In Germany and France, Ordinary Portland cement (OPC), has been used as a replacement to the 6% of the conventional limestone fillers within the ingredients of CMA. The results have shown a fast curing cold mix asphalt and used in road pavement construction (Al-Busaltan et al., 2012a; Dulaimi et al., 2016a; Dulaimi et al., 2016c). These cold asphalt mixtures are normally used in light and heavy trafficked loads in the said countries. However, CMA containing OPC as a filler is not environmentally friendly construction materials as every tone of cement releases approximately one tone of CO₂ emission during their manufacturing process (Al-Hdabi et al., 2014; Dulaimi et al., 2016b). The new cold mix asphalt studied in this research work is made by replacing traditional limestone fillers in conventional CMA with secondary cementitious materials (SCM) made from new novel upgraded fly and bottom ashes from incinerated wastewater sludge.

1.1. Testing Samples for Water Sensitivity

Cohesion and bonding properties of bituminous emulsions experience a reduction in the presence of water within the mixtures. This will reflect on the reduction in both the durability and mechanical characteristics of asphalt mixtures made from these emulsions.

In this project the procedures in the UK, BS EN 12697-12 (European Committee for Standardization, 2008) were used to find the water sensitivity of the bituminous emulsions mixtures. Two groups of specimens prepared. Specimens in group 1 were made in the asphalt

laboratory, left in the sample molds for 24 hours before removing from the molds, then these samples were left 7 days in the laboratory at a room temperature of 20°C. Whereas the specimens in group 2, were subjected to the following process. At age 24 hours, the samples were removed from their molds, then remained in the asphalt laboratory for curing for additional 4 days. The laboratory temperatures was 20 °C. At a temperature of 20 °C, the samples were then and at an absolute pressure of 6.7 kPa subjected to a vacuum, followed by immersing them in water for 30 minutes, see Fig. 1, and a temperature of 40 °C soaked the samples for 3 days in water. EN 12697-26 (European Committee for Standardization, 2012) test procedures then need to be followed to calculate ITSM of conditioned sample and SMR should be calculated from the following formula;

$$\text{SMR} = (\text{Wet ITSM} / \text{Dry ITSM}) \times 100$$



Fig. 1. Water sensitivity apparatus and samples preparations.

1.2. Aging test

Cold mix asphalt is normally prepared at ambient temperature without heating. Therefore, the short-term oven heating that is normally applied for hot mix asphalt mixtures is not relevant to cold mix asphalt aging. Instead, long term oven aging simulating the aging of cold mix asphalt mixtures when using in services is selected, adopting the procedures set by the Strategy Highway Research Program (SHRP) (Bell et al., 1994). Kliewer et al., (1995) indicated that samples cured for 2 days or 5 days in the oven at 85 °C simulate 5 or 10 years of bitumen ageing in the field respectively.

To indicate the aging effects on the new CMA compared with cold mix asphalt containing OPC as replacement of conventional limestone filler (CMA-OPC) and conventional Hot Mix Asphalt (HMA). The procedure involves preparing two groups of samples. Group one named

unconditioned samples (or dry samples) kept in the lab at a temperature of 20°C without curing in the oven. Group two samples, subjected to conditioning in the oven at 85 °C and for 5 days. Then the samples were left in the lab for curing for one day to a temperature of 20 °C before testing for ITSM. ITSM tests were based on the details of the procedure in the BS EN 12697-26 ([European Committee for Standardization, 2012](#)) to determine SMR,

$$\text{SMR} = [\text{ITSM (conditioned)} / \text{ITSM (un-conditioned)}] \times 100$$

where

SMR = the Stiffness Modulus ratio.

1.3. Toxicity Characteristic Leaching Procedure (TCLP) Test

Leaching of heavy metals from pavement layers to the soil, underground waters, and surface waters due to slow flow of solvents and metals contaminated water is referred in literatures as leaching ([Dulaimi et al., 2020](#); [Ling and Poon, 2014](#); [Halim et al., 2003](#)). The ambient environment, birds, animals, peoples can seriously affected by heavy metals. [García-Lara et al., \(2009\)](#) and [Wan et al., \(2011\)](#) reported that skin cancer and serious damage to central nervous can happen system due to drinking contaminated water.

United States Environmental Protection Agency (USEPA) TCLP test is used in this research project to determine the concentration of heavy metals, namely [copper (Cu), cadmium (Cd) barium (Ba), chromium (Cr lead (Pb), zinc (Zn), nickel (Ni) strontium (Sr))] leaching from the tested pavement mixtures. Following the successful use of this test by other researchers like [Modarres and Ayar, \(2016\)](#), [Dulaimi et al., \(2017\)](#) and [Xue et al., \(2009\)](#), to determine the concentration of heavy metals from inorganic wastes and organic wastes to the environment.

[Fig. 2](#) and [Fig. 3](#) show the Rotary extractor and Atomic adsorption spectrophotometer used to complete the tests.



Fig. 2. Rotary extractor.



Fig. 3. sample under test using Atomic Adsorption Spectrophotometer.

2. MATERIALS, MIX DESIGN AND TESTING:

2.1. Aggregate

For appropriate interlocking between aggregates in cold mix asphalt, crushed 0/10 mm surface course close graded granite aggregate with sieve analysis complying with the requirements of BS EN 933-1 ([European Committee for Standardization, 2012](#)) and BS EN 13108 ([European](#)

Committee for Standardization, 2015) were used in this study. Colas Ltd, UK, supplied the aggregates. The aggregates grading is shown in Table 1. Whereas Table 2 shows the aggregate physical properties.

This gradation of dry mix aggregates shown BS EN 4987-1 for 0/10 mm size close graded surface course according Dulaimi et al., (2017), shown in Table 1 below is commonly used in the manufacturing of bituminous mixtures normally selected by road engineers for use in road pavement surfacing of heavy trafficked highways and roads, (Al Nageim et al., 2012).

Table 1. Aggregate grading for 0/10 mm size, close graded surface course according to BS EN 4987-1 (Dulaimi et al., 2017).

sieve size (mm)	Passing Mass Specification range (%)	Mid Mass Passing (%)
14	100	100
10	100	100
6.3	62-68	65
2	25-31	28
1	14-26	20
0.063	6	6

Table 2. Physical properties of aggregates.

Properties	Value
Coarse aggregate/ Bulk specific gravity (Mg/m^3)	2.60
Apparent specific gravity (Mg/m^3)	2.66
Water absorption (%)	0.8
Fine aggregate/Bulk specific gravity (Mg/m^3)	2.52
Apparent specific gravity (Mg/m^3)	2.58
Water absorption (%)	1.6

2.2. Properties of Asphalt and Emulsion:

K3 cationic slow setting bituminous emulsion properties are shown in Table 3. This emulsion is used to provide strong bonding between the aggregate particles in the mass of cold mix asphalt. While Table 4 shows the mix types, filler contents and bitumen used in this research work. 40/60 and 100/150 hard and soft binders respectively are used in the preparations of hot mix asphalt.

Table 3. Asphalt binder and asphalt emulsion properties.

Asphalt emulsion		Asphalt binder [40–60] pen		Asphalt binder [100–150] pen	
Properties	Value	Properties	Value	Properties	Value
Appearance	Brown liquid	Appearance	Black	Appearance	Black
Base bitumen, 1/10	50	Penetration 25° C	49	Penetration 25° C	131
Boiling Point (C)	100	Softening point (C)	51.5	Softening point (C)	43.5
Relative density at 15 C g/ml	1.05	Density at 25° C	1.00	Density at 25° C	1.05
Bitumen content, %	50				

3. MIX TYPES, FILLERS AND SAMPLE PREPARATIONS FOR SENSITIVITY AND AGING TESTS

Cold mix asphalt specimens were mixed and compacted at a laboratory temperature of 20 °C following the procedures of the Asphalt Institute (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design-MS-14) ([Asphalt Institute, 1997](#)). Indirect Tensile Modulus test was used instead of the Marshall test to performed testing according to the procedures of BS EN12697-26 ([European Committee for Standardization, 2012](#)). Mix type, filler amount, type and contents are shown in [Table 4](#). 150-170 °C mixture temperatures was used to mix and compact HMA (Hot Mix Asphalts) specimens.

Table 4. Mix type, percentages of the filler and filler contents.

Mix type	Description/Filler
CMA-LF	Cold mix asphalt with 6% Control limestone filler
CMA-OPC	Cold mix asphalt with 6% as control OPC filler
Mix1-CMA	CMA with [2.1% OPC+ 3.9% fly ash (FA)] filler1
Mix2-CMA	CMA with [2.1% OPC+3.3%FA+0.6%BA] filler2
CMA+ SF [silica fume]	
Mix1+SF	CMA with [2.1% OPC+ 3.9%FA+4%SF] filler1+SF
Mix2 +SF -	CMA with [2.1% OPC+3.3FA+0.6%BA+4%SF] filler2+SF
HMA (soft)	Traditional HMA with [100/150] pen. asphalt binder
HMA (hard)	Traditional HMA with [40/50] pen. asphalt binder

The Fly ash (FA) and Bottom ash (BA) are a product of the incineration process of water sludge at the united utility power generation plant in the UK. The two ashes are normally sent from the production sites as a waste to a landfill. The FA and BA are ground to the same size then mixed with the percentage of the OPC defined in [Table 4](#) above to form filler 1 and filler 2 respectively. 20 –22 °C is the temperature range for the mixing and compaction of the specimens of CMA, while 150-170 °C is the range of the temperatures for mixing and compaction of the specimens of HMA.

The CMA and HMA mixtures were manufactured following the procedures provided by the Asphalt Institute ([Asphalt Institute, 1997](#)). CMA strength properties are very sensitive to both temperature and curing time, therefore the samples conditioning and testing were completed in two stages as detailed in [Table 5](#).

Aging test: Long-term aging of all cold mix asphalt mixtures made with filler 1 or filler 2 with and without SF together with the two hot mix asphalts, was achieved by preparing two groups of samples. Group one named unconditioned samples were kept in the lab at temperature 20°C without curing in the oven. Group two samples were subjected to conditioning in the oven at 85 °C and for 5 days. Then the samples were left in the lab for curing for one day to a temperature of 20 °C before testing for ITSM. ITSM tests were based on the details of the procedure in the BSEN 12697-26 ([European Committee for Standardization, 2012](#)) to predict the SMR. Where

$$\text{SMR} = [\text{ITSM (conditioned)} / \text{ITSM (un conditioned)}] \times 100$$

In this project the procedures in the UK, BS EN 12697-12 ([European Committee for Standardization, 2008](#)) were used to find the water sensitivity of the bituminous emulsions mixtures. Two groups of specimens prepared. Specimens in group 1 were made in the asphalt laboratory, left in the sample molds for 24 hours before removing from the molds, then these samples were left 7 days in the laboratory at a room temperature of 20°C. Whereas the specimens in group 2, were subjected to the following process. At age 24 hours, the samples were removed from their molds, then remained in the asphalt laboratory for curing for additional 4 days. The laboratory temperatures was 20 °C. At a temperature of 20 °C, the samples were then and at an absolute pressure of 6.7 kPa subjected to a vacuum, followed by immersing them in water for 30 minutes, see [Fig. 1](#), and a temperature of 40 °C soaked the samples for 3 days in water. EN 12697-26 ([European Committee for Standardization, 2012](#)) test procedures then need to be followed to calculate ITSM and SMR, using the following formula;

$$\text{SMR} = \frac{\text{Wet stiffness}}{\text{Dry stiffness}} \times 100$$

Table 5. Curing conditions for the tests used.

Test	Curing in days Stage 1	Stage 2	Testing at age (days)	BS EN
Water sensitivity test	Curing at 20 C° for 1 day	Soaked in water at 40C° for 7 days	8	12697-12
Aging test		Heated in the oven at 85C° for 4 days	5	(SHRP) A003A

4. RESULTS ANALYSIS AND DISCUSSION

4.1. Water sensitivity test results and discussion:

Water sensitivity tests of all cold mix asphalts and control hot mix asphalts were carried according to BS EN 12697-12 (European Committee for Standardization, 2008) to examine the influence of both filler 1 and filler 2 as replacements for the LF(limestone filler). In Fig. 4 and Table 6, it is clearly shown that ITSM values for the mixtures Mix 1 (conditioned) and Mix 1 (unconditioned) confirmed that these results are more than their values for the tested HMA bituminous mixtures with (143pen). Also, this finding hold true for conditioned mix2- CMA. The improvement of the values of in ITSM (conditioned) is because of the hydration process of the secondary cementitious materials [SCM] enhanced with the existing water within the mix and the water due to the sample conditioning for testing. Adding SF to the SCM in these mixtures has added more improvements to the hydration mechanisms as shown in the results of the mixture reported in Fig. 4. The results for both unconditioned and conditioned specimens containing SF shows a close comparison of the conditioned specimens and unconditional specimens with the control mix OPC as well as the HMA.

Also, it is clearly seen in Fig. 4 and Table 6, that SMR ration for the two mixes both Mix 1 and Mix 2 are above 100%. These results for conditioned samples were close to those for the HMA specimens and thus reached the requirements of road engineers for bituminous mixtures, namely, 1500 MPa for soft HMA and 300 MPa for hard HMA.

When the new fillers namely filler 1 and filler 2 are used to replace LS (limestone filler), test results have proven that they have improved the cohesion and produce stronger bituminous mass. This is attributed to the fact that FA & (FA+BA) with the addition of OPC, produces more hydration products in the presence of water within the emulsion contents, and when specimens are submerged in water. the resulting CMA mixtures is less susceptible to moisture damage. Therefore, a cold mix asphalt with FA & (FA+UBA) plus the added percentages of OPC and or SF improve materials specification requirements for use in roads and pavements.

In addition, and as the results from these test, provide proofs that the new cold mix asphalt containing the new cementations fillers made from FA and OPC and (FA+BA) plus OPC as defined in Table 4 above, with or without the 4% of added SF as a conventional limestone filler replacement in CMA remove the expected moisture damage in the new CMA when it comes in contact with water.

Table 6. Water sensitivity test results.

No.	Type of mix and filler	ITSM conditioned (MPa)	ITSM unconditioned (MPa)	SMR
1	Mix 1-CMA	3155	1749	191.32
2	Mix 2-CMA	3211	1123	285.93
3	Mix 1-+ SF -CMA	4086	2651	154.13
4	Mix 2 +SF- CMA	4149	3801	109.16
5	OPC-CMA	4220	3903	108.12
6	L.F HMA (40/60 Pen)	3178	4442	71.54
7	L.F HMA (100/150 Pen)	1046	1527	68.50

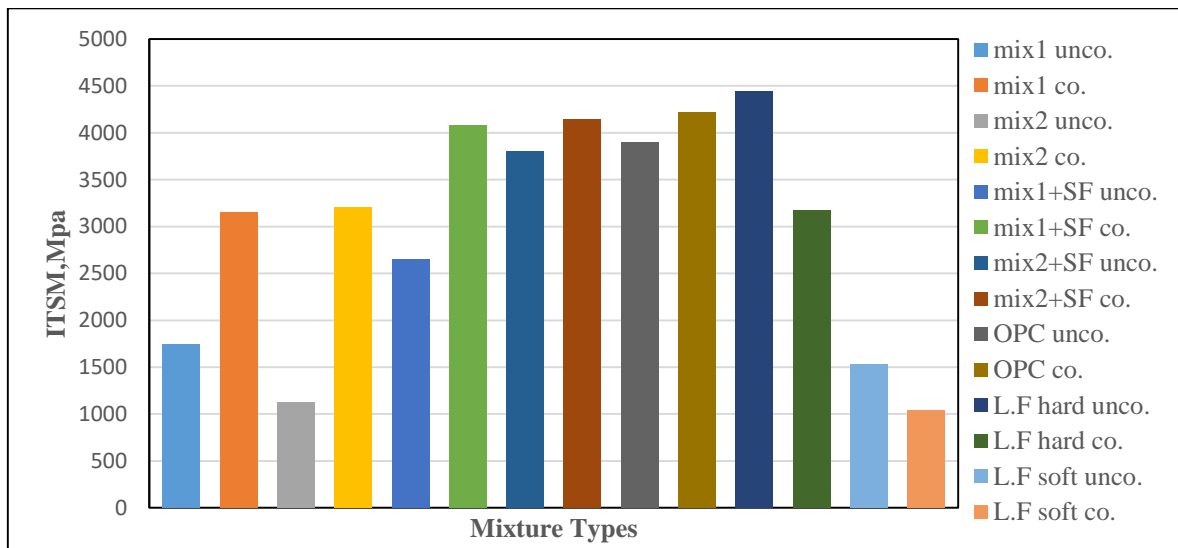


Fig. 4. ITSM water sensitivity results.

4.2. Aging test results and discussion

Long-term aging of all cold mix asphalt mixtures made with filler 1 or filler 2 with and without SF together with the two hot mix asphalts, were achieved by conditioning the samples in the oven at 85 °C and for 5 days. Then the samples were left in the lab for curing for one day to a temperature of 20 °C before testing for ITSM. ITSM tests were based on the details of the procedure in the BSEN 12697-26 (European Committee for Standardization, 2012). Table 7 and Fig. 5 shows the results of these laboratory tests. Where the SMR is the ratio of the unconditioned ITSM test values to conditioned ITSM tests values. The results of all the cold

mix asphalts are higher than the corresponding control hot mix asphalt. From Table 7 and Fig. 5, also, it can be seen that cold mix asphalts containing filler 1 or filler 2 provided outstanding improvement in their ITSM after conditionings. This can be explained due to the increase in the hydration products of the secondary cementitious materials [filler 1, filler 2 with and without SF] in these mixtures at high-temperature curing. Together with losing excessive water and in addition to the fact that the growth of hydration products produces a significant additional bonding for the bitumen bonding the CMA mass. This additional bonding agent may contribute to preventing the loss of volatile fractions and oxidation during the aging process. The ITSM for these mixtures after ageing is considered a promising result. Heating the CMA samples at 85 °C for 5 days provided enhanced cohesion and bonding characteristics, which provided a better ageing performance.

Table 7. Aging test results.

No.	Type of mix and filler	ITSM Co.	ITSM UnCo.	SMR
1	Mix 1-CMA	5437	1798	302.39
2	Mix 2-CMA	5550	1123	494.22
3	Mix 1-+ SF -CMA	8660	3452	250.87
4	Mix 2 +SF- CMA	8021	3156	254.15
5	OPC-CMA	8156	3724	219.01
6	L.F HMA (40/60 Pen)	2683	4442	60.40
7	L.F HMA (100/150 Pen)	846	1527	55.40

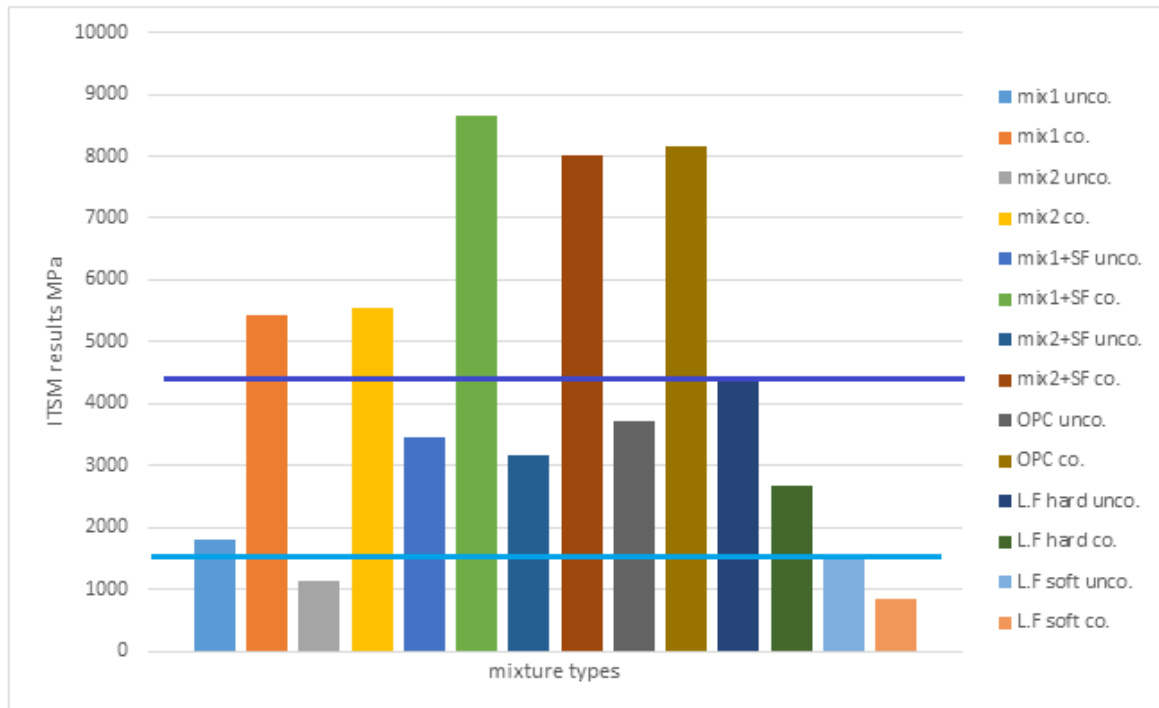


Fig. 5. Aging test results.

4.3 Leaching Procedure (TCLP) Test: CMA Toxicity Characteristic

In the new CMA's mixtures, the authors used TCLP test to find the percentages of nickel (Ni), copper (Cu), chromium (Cr), strontium (Sr), barium (Ba), zinc (Zn), lead (Pb), cadmium (Cd), in the pavement tested mixtures (U.S. Environmental Protection Agency, 1986). The equipment used are the Rotary Extractor, Fig. 3, and Atomic Adsorption spectrophotometer, Fig. 4, which are available at LJMU chemical laboratory.

Table 8 include the concentration of these heavy metals. The concentration of Zn is found high in the ground filler contains FA, BA and mix 1 with SF. However, this concentration was less than the regulatory level of TCLP specified by USEPA. Interestingly the TCLP test results in mg/L on the fillers in all the mixtures studied were found considerable lower than their contents in OPC and significantly lower than the regulatory level reported by TCLP of USEPA. See the work of Modarres and Nosoudy, (2015) and Modarres et al., (2015). We can therefor conclude that these metals within the ground FA, BA and CMAs' mixtures have been stabilised and or solidified and therefore the new CMA tested in this project is environmentally friendly. TCLP regularity levels are shown in Table 8 which proofs that the heavy metals in the mixes labeled, mixes 1 and 2, Mix 1 with SF, and Mix 2 with SF satisfy the limits for classifying hazardous materials.

Table 8. TCLP test results (mg/L) on the filler of the mixes.

Heavy metal concentration (mg/L)	OPC	FA	BA	LF	Mix1 filler	Mix1+SF filler	Mix2 filler	Mix2+SF filler	TCLP regulatory level
Nickel (Ni)	0.0	0.001	0.002	0.0	0.0	0.0	0.0	0.0	25
Copper (Cu)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	25
Lead (Pb)	0.004	0.005	0.006	0.006	0.005	0.006	0.006	0.006	5
Chromium (Cr)	0.009	0.003	0.003	0.003	0.007	0.007	0.009	0.009	5
Zinc (Zn)	0.024	1.898	1.352	0.307	0.490	1.645	0.314	0.711	25
Strontium (Sr)	0.309	0.176	0.142	0.253	0.094	0.047	0.019	0.155	-
Barium (Ba)	0.008	0.004	0.004	0.002	0.007	0.007	0.007	0.007	100
Cadmium (Cd)	0.009	0.013	0.025	0.012	0.004	0.011	0.006	0.006	1

5. CONCLUSIONS

The following conclusions can be withdrawn from our study reported in this paper:

ITSM values for the developed CMA tested, namely; Mix1 and Mix2 without and with SF resulted from water sensitivity and ageing tests are promising results, because, they all show higher results compared with conventional HMA containing either (100/150pen) or (40/60 pen) binders. These results are in agreements with our previous studies in ([Al Nageim et al., 2012](#); [Al-Busaltan et al., 2012](#) and [Dulaimi et al., 2020](#)). Very interesting and high-value results, which necessitate the need for further work on-site trials to show the performance of the new asphalt mixtures in real interaction between traffic and environmental conditions.

The concentrations of heavy metals, Copper (Cu), Nickel (Ni), Strontium (Sr), Chromium (Cr), Cadmium (Cd) and Barium (Ba) in the new developed United Utility SCM fillers used in the mixes [Mi1, Mix2, Mix1+SF, and Mix2+SF] identified using TCLP test were within the regularity level accepted by USEPA.

The high values of ITSM reported in this paper, can be explained due to the increase in the hydration products within the CMA at an early age of curing. The amount of water available within the emulsion and CMA both provide excellent environment for improving the hydration process. The growing of hydration bonding products within the mixes causes an increase in the ITSM of the new CMA products, as well as a significant reduction in the water available in the CMA, and thus fast curing time. This is in agreements with the findings in (([Al Nageim et al., 2012](#); [Al-Busaltan et al., 2012](#) and [Dulaimi et al., 2020](#)) and ([Al-Nageim et al.,2019](#); [Dulaimi et al., 2016b](#); [Al-Hdabi et al., 2014](#))). The increase of hydration products produced also partial cover for the aggregates between and inside the

mixtures microstructures. This has contributed to preventing oxidation and the loss of volatile fractions from bituminous binders during aging of the new mixtures.

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