

# Stiffness, Creep Properties and XRD Analysis of a New Fast-Curing Cold Mix Asphalt for Use in Highway Pavement

Hassan Al Nageim<sup>1, a</sup>, Shakir Al-Busaltan<sup>2, b</sup>

<sup>1</sup> School of Built Environment, Liverpool John Moores University, Liverpool, United Kingdom

<sup>2</sup> Civil Engineering Department, University of Kerbala, Kerbala, Iraq

<sup>a</sup> h.k.alnageim@ljmu.ac.uk

<sup>b</sup> shakerfa2003@yahoo.com

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## ABSTRACT

*The paper reports the stiffness and creep properties and XRD analysis of a new cold mix asphalts developed for use in highway pavement surface layers.*

*The high voidage, low stiffness modulus and long curing time which normally spans approximately from two months to 24 months has restricted road engineers to allow these materials for use in road experiencing low traffics.*

*To remove the above restriction, the main filler in the traditional Cold Bituminous Emulsified Mixtures CBEM's is replaced with treated biomass waste fly ash materials.*

*The new filler has increased the stiffness and creep properties of the new CBEM's and hence provide new CBEM's product possessing the following advantages compared with traditional Hot Mix asphalt (HMA);*

- 1. reduced energy of production,*
- 2. reduced emission of pollutants,*
- 3. reduced pavement total construction costs*

*As well as providing a use for what would otherwise become a hazardous land fill waste products. The fly ash used in this research project is available in more than 250,000 tonne annually in the UK and more than this quantity in Europe, USA, and worldwide.*

*This new products will contribute in an outstanding percentage of reducing CO<sub>2</sub> emission when the new products finds its way to replace HMA prepared and constructed at 120C<sub>0</sub> to 170C<sub>0</sub>.*

*A treated biomass fly ash, which is waste or by-product material, was incorporated in the cold bituminous emulsion mixtures with five percentages of replacement to mineral traditional filer ranging from 0 to 5.5% of aggregate weight in the mixture. The results have shown outstanding comparative improvement in the stiffness and creep properties of the new cold mixtures compared with traditional cold mix asphalt. The reason for achieving these results was explained in this paper by using XRD analysis of the fine mineral-emulsified mortar used in this study.*

**Keywords:** Additives, Cold Asphalt, Emulsions, Fibres, Stiffness

## 1. Introduction

The use of Cold Bituminous Emulsion Mixtures (CBEMs) for road construction, rehabilitation and maintenance is gaining interest day by day, as these mixtures offer advantages over traditionally hot mixtures in different terms such as; environmental impact, energy saving, cost effectiveness, safety and cheap production processes. In the UK, today's the use of cold mix takes less interest compared with Hot Mix Asphalt, as this mixtures show low earlier strength to resist the different traffic loads and low resistance to water damage especially rainfall. Other countries such as USA, European countries, and Australia showed more interest in the uses of the materials due to the above advantages.

Cold mix asphalt bituminous materials normally prepared at ambient temperatures. In the UK, the use of CBEM is largely restricted to surface treatment such as surface dressing slurry surfacing, and reinstatement work on low trafficked and walkways (HAUC, 1992; Read & Whiteoak 2003), due to the long curing time required for such materials to reach its full strength after paving, especially in the UK climate. In addition, such mixes are highly sensitive to rainfall at early life due to the high voidage within the compacted mix.

Mechanical properties of CBEMs' including; stiffness modulus, permanent deformation and fatigue resistance are affected by many factors such as; based binder grade and characteristics, mixture void content, curing time, aggregate characteristics, and additives (Needham, 1996; Thanaya, 2003). Attempts to improve cold mixes mechanical properties have been investigated. Early study conducted by Head in (1974) indicated that Marshall Stability of modified cold asphalt mix is increased by 250-300 % with the addition of 1 % Portland cement compared with un-treated mix. Oruc et al., (2007) conducted experiments to evaluate the mechanical properties of emulsified asphalt mixtures having 0-6 % Portland cement. The test results showed significant improvement with high Portland cement addition percentage; moreover they suggested based on their study test results, that the cement modified asphalt emulsion mixes might be used as structural pavement layers.

Thanaya et al., (2009) showed that the addition of 1-2 % rapid-setting cement accelerates the earlier strength as well as improves the mechanical performance of the modified cold mixes.

Pouliot et al.(2003) conducted a study with the aim of understanding the hydration process, the microstructure, and the mechanical properties of mortars prepared with a new mixed binder made of a cement slurry and a small quantity of asphalt emulsion (SS-1 and CSS-1, i.e. anionic and cationic emulsion). They proved that presence of a small quantity of emulsion had an effect on the cement hydration. Their test results also indicated that the launch of asphalt droplets inside a cement mortar matrix leads to a considerable reduction in compressive strength and elastic modulus in addition to a slight decrease in flexural strength. Also, they found that the cationic emulsion (CSS-1) in contrast with anionic emulsion (SS-1) shows higher mortars strengths and elastic modulus. Other study by Wang & Sha (2010) indicated that the increase of cement and mineral filler fineness has a positive impact on micro hardness of the interface of aggregate and cement emulsion mortar.

A research works implemented by Thanaya et al (2006) indicated that the Pulverised Fly Ash (PFA) can be used as suitable filler in cold mixes at full curing conditions. Also they found the stiffness of cold mix achieved is very comparable to hot mixtures.

From the above research works and other attempts tried the use of waste and by-products materials to improve cold mixes, four main benefits can be achieved when utilizing the by-product materials on CBEM's. These are:

- i) improving mechanical properties, in general there will be an enhancement of ultimate mechanical and strength properties due to the cementitious properties.
- ii) gaining economic benefit as the pozzolanic and cementitious materials used are mostly industrial by-products.
- iii) Thirdly, trapped water could be reacted with these materials to complete the hydration process and the result is getting rid of this water which is the main reason of increasing the curing period in CMA's, and lastly,
- iv) the ecological benefit factor.

The central theme of this work reported her concentrated on the use of biomass treated fly ash as filler in cold bitumen emulsion mixtures to overcome the low early stage mechanical strength and reduced the cold mix asphalt curing time.

Biomass fly ash incorporated as filler in CBEM's in this study at a percentage of replacing filler from 0.0 to 5.5% of the aggregate weight. The improvement in mechanical properties determined using the indirect tensile stiffness modulus

and Uniaxial compressive cyclic test, are reported the improvement in the stiffness properties of the new developed were explained by using XRD analysis to provide an understanding of the early stage stiffness and strength developments.

## 2. Materials and sample preparation method

### 2.1 Materials

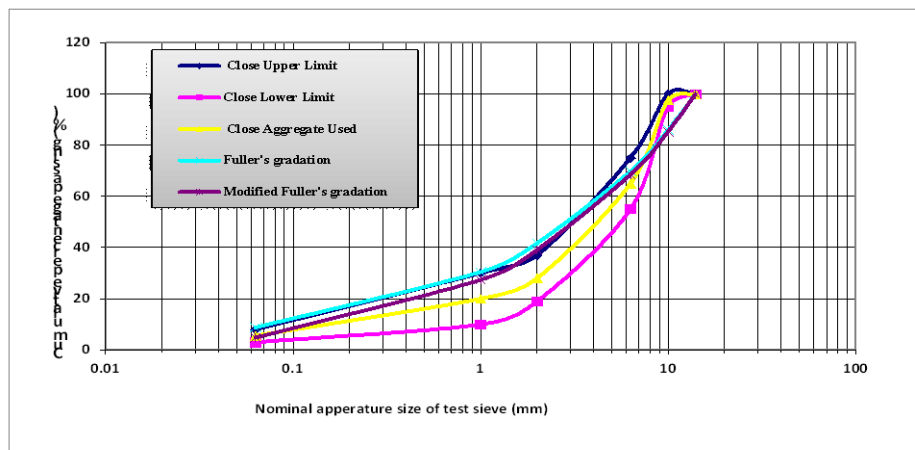
In order to ensure appropriate interlocking between cold mix asphalt ingredient and to provide high surface strength pavement layer, 0/10 mm Close graded surface course with mix gradation according to BS EN 4987-1 was selected for this research work. The aggregate used in this study is crushed green granite from Cliffe Hill quarry and the aggregate gradation is given in Table 1. Physical properties of the aggregates are given in Table 2. The aggregate were dried, riffled and bagged with sieve analysis achieved in according with BS EN 933-1 and BS EN 12697-28. The grading of the 0/10 mm mix is shown in Figure 1.

**Table 1: Aggregate grading for 0/10 mm size close graded surface course BS EN 4987-1**

Test sieve aperture size mm	% by mass passing specification range	% by mass passing mid
14	100	100
10	95-100	97.5
6.3	55-75	65
2	19-37	28
1	10-30	20
0.063	3-8	5.5

**Table 2: Physical properties of aggregates**

properties	value
Coarse aggregate	
Bulk specific gravity, gm/cm <sup>3</sup>	2.79
Apparent specific gravity, gm/cm <sup>3</sup>	2.82
Water absorption %	0.4
Fine aggregate	
Bulk specific gravity, gm/cm <sup>3</sup>	2.74
Apparent specific gravity, gm/cm <sup>3</sup>	2.77
Water absorption %	0.4



### Figure 1: Grading 0/10 mm aggregate

The selection of this gradation was done with consultation of our industrial partners and based on: firstly this gradation has been used successfully in the heavy traffic surface coarse hot coated macadam (BS EN 4987:1, 2005) and secondly, the dense gradation has more coarse aggregates percentage compared with close graded hot mix coated macadam, see, Doyle et al. (2010) and not easily compacted.

Cationic slow setting emulsion (K3) was used in the experimental program to ensure high adhesion between aggregates particles. Table 3 show the properties of the selected emulsion.

**Table 3: Bitumen Emulsion Properties (K3)**

properties	value
Appearance	Black to dark brown liquid
Boiling Point (° C)	100
PH	5
Relative Density at 15 ° C gm/ml	1.05
Residue by distillation,%	56

## 2.2 Samples preparation

All samples produced for this study was prepared according to the method adopted by the Asphalt Institute (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design) (MS-14, 1989). According to the selected materials characteristics, pre-wetting water content was observed to be 4%, the optimum bitumen emulsion was 11.5 % and optimum total liquid content at compaction was 14.5 %.

Specimens of cold bitumen emulsion mixtures were prepared using different ratios of biomass treated fly ash (0.0 - 5.5 %) as a replacement of mineral filler. Impact compacting (Marshall Hammer) was applied with 50 blows to each face of the specimens. Moreover, conventional hot mixture samples were prepared with the same aggregate type and gradation, 5.3% binder content was used to match the BS EN 4987 (2005) with 0/10 mm size close graded surface course of the grading described above. Both cold and hot mixes were prepared in quantity to produce three 1100 gm specimens for each specific mix. The cold mix specimens were mixed and compacted at lab temperature (20 - 25 °C), while hot mix specimens were compacted at (135-140 °C).

## 2.3 Samples conditioning

Cold mixtures strength characteristics are very sensitive to curing time and temperature. Therefore, samples conditioning for indirect tensile test were achieved at two stages; stage one with 20 °C for 24 hours as the sample needs to be left in mould before extruded to prevent specimen disintegration. Second stage conditioning was achieved with three curing temperatures (20, 40, 60 °C) and tested at age of (i.e. 2, 7, 14, 28, 90 and 180 days). The selections of two stage sample conditionings criteria based on the past curing criteria adopted by various researchers, see Jenkins (2000).

## 3. Testing, results and discussion

### 3.1 Indirect tensile stiffness modulus

The Indirect Tensile Stiffness Modulus (ITSM) is a non-destructive test used mainly to evaluate the stiffness modulus of hot mix asphalt. ITSM at 20 °C was used to evaluate the effect of the new filler on stiffness modulus. The test was conducted in accordance with BS EN 12697-26:2004, the Cooper Research Technology HYD 25 testing apparatus was used. The test conditions as in Table 4.

### 3.2 Uniaxial compressive cyclic test

The Uniaxial compressive cyclic test (UCCT) is a destructive test used mainly to evaluate the permanent deformation characteristics of hot mixes. UCCT at 20 °C was used to evaluate the effect of the TBFA on creep stiffness. The test was conducted in accordance with BS EN 12697-25:2005, the Cooper Research Technology HYD 25 testing apparatus was used. The test conditions as in Table 5.

**Table 4: ITSM Test Conditions**

item	range
Specimen diameter mm	100±3
Rise time	124±4 ms
Transient peak horizontal deformation	5 µm
Loading time	3-300 s
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 50×2
Specimen temp. conditioning	4hr before testing

**Table 5: UCCT Conditions.**

item	range
Frequency	0.5 Hz
Loads	100±2 KPa
Loading pulse	1±0,05 s
Rest period	1±0,05 s
preloading	10 KPa for 10 min
Poisson's ratio	0.35 for 20 °C test tem.
No. of test plus	3600
Test temperature °C	40 ±0.5
Specimen diameter	148±5
Specimen thickness	60±2 mm

### 3.3 Results and Discussion

#### 3.3.1 Indirect tensile stiffness modulus (ITSM)

All cold bitumen emulsion mixture specimens for ITSM test were tested at age of 2,7,14, 28, 90 and 180 days. The first curing time was 24 hours at 20 °C, then 24 hours curing at three curing temperatures (20,40, and 60 °C) in order to identify the effect of replacing mineral filler with the new filler (TBFA) on the mixtures hydration mechanism and thus on the improvement in ITSM strength. The results of these tests are shown in figures 2 to 4.

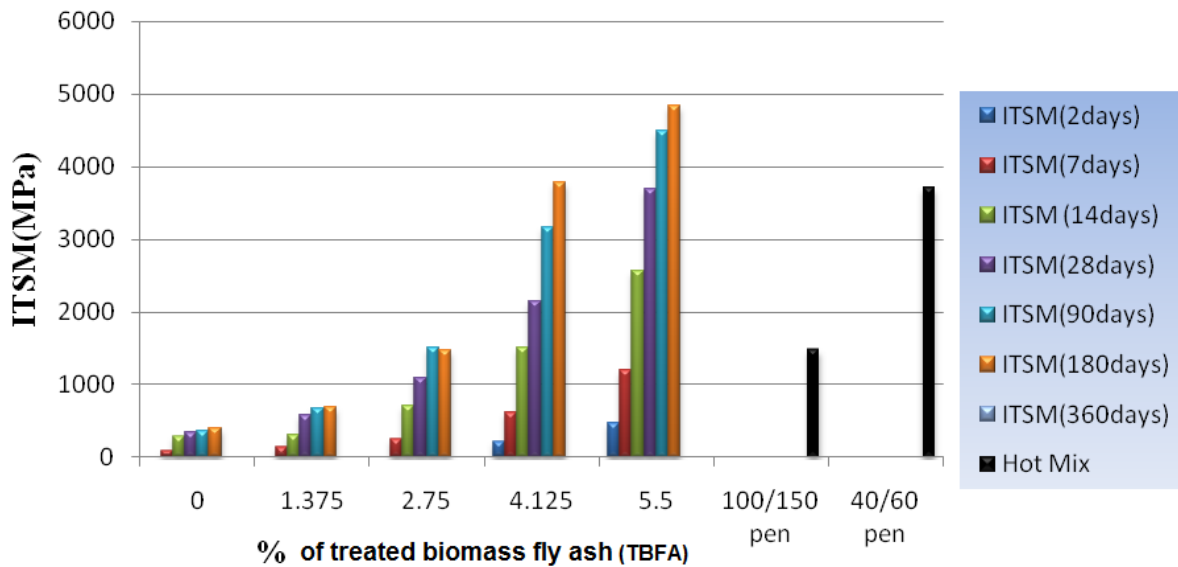


Figure 2: ITSM (MPa) of cold mix asphalt

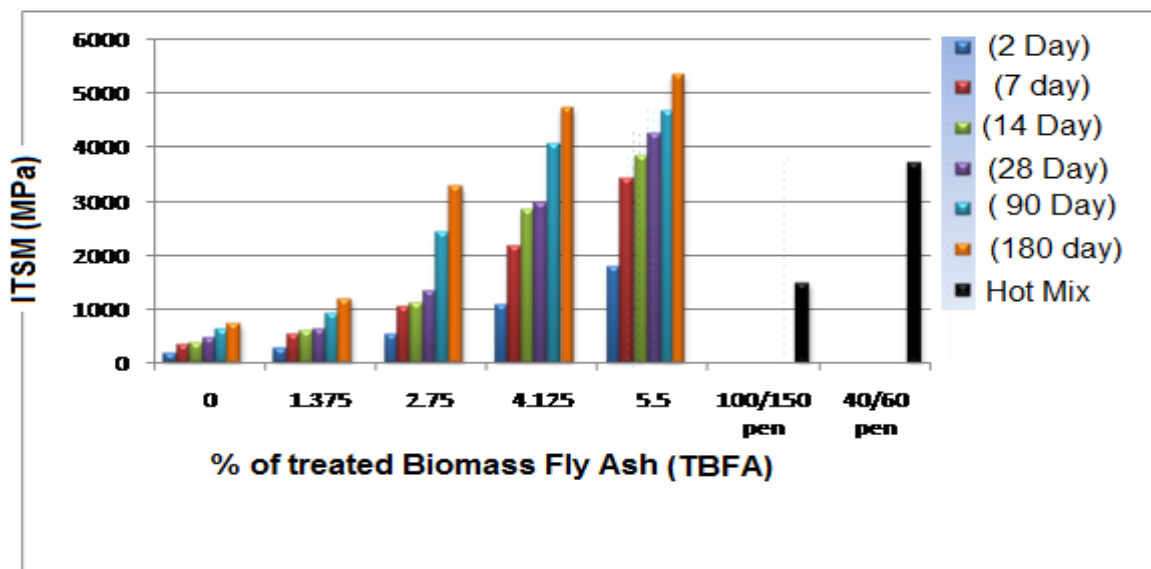


Figure 3: Effect of TBFA % on ITSM (24hrs, 20 C<sup>0</sup> + 24 hrs. 40 C<sup>0</sup>)

Results of ITSM tests shown in Figure 2, indicate that the stiffness modulus of CBEM's increased dramatically with the increased percentage of the replacing new filler, and reached its ultimate values when all the mineral fillers is replaced with the new filler. Additionally, the ITSM were increased significantly with time.

An outstanding gain in ITSM values were experienced at the other curing methods (i.e. stage two with 40, and 60 °C) with the increase in curing time and TBFA percentage. At the same time the HMA show unnoticeable changes in ITSM with time. With no or low percentage of the replacing new filler, no results after two days are shown in figure 2 as the specimens couldn't withstand the testing load.

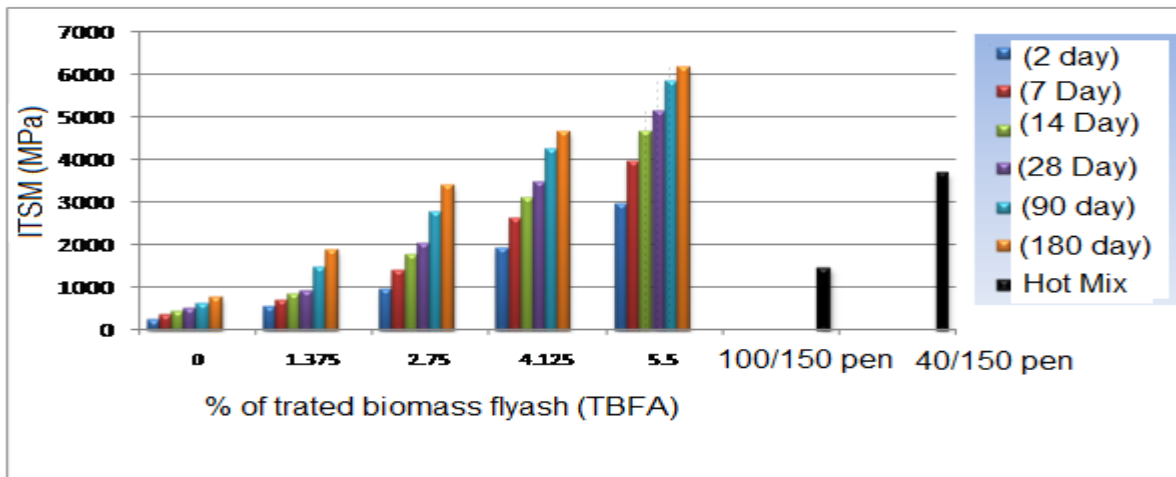


Figure 4: Effect of TBFA % on ITSM (24hrs, 20 C<sup>0</sup> + 24 hrs. 60 C<sup>0</sup>)

### 3.3.3 Uniaxial compressive cyclic test

The results of the uniaxial compressive cyclic tests are given in Figs. 5 and 6. Figure 5 present the accumulated strain versus pulse counts, whereas figure 6 illustrates the creep stiffness of mixtures with different TBFA content. These figures show the general trend of the five different TBFA content within the mixes as replacement of mineral conventional filler. The figures demonstrate the positive effect of TBFA on the creep properties of CBEM's; specimens with higher TBFA content had considerably longer life under cyclic load creep tests when compared to control specimens as well as with Hot Asphalt Mixtures. However, before the end of the uniaxial compressive cyclic test, the control specimens gain a total collapse, while all specimens with different TBFA contents withstand the 3600 pulses. This reflects that the TBFA modified specimens would show a longer life than the control specimens would.

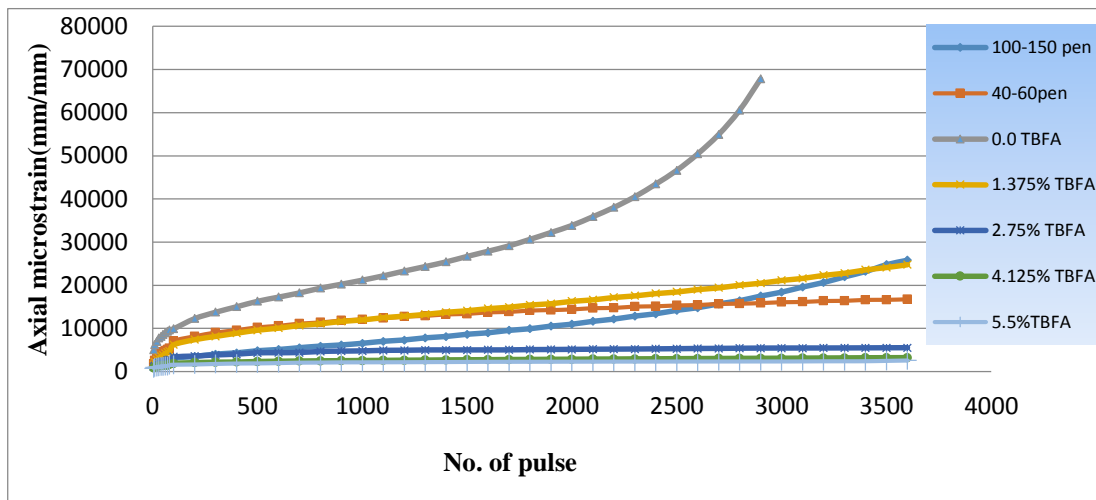
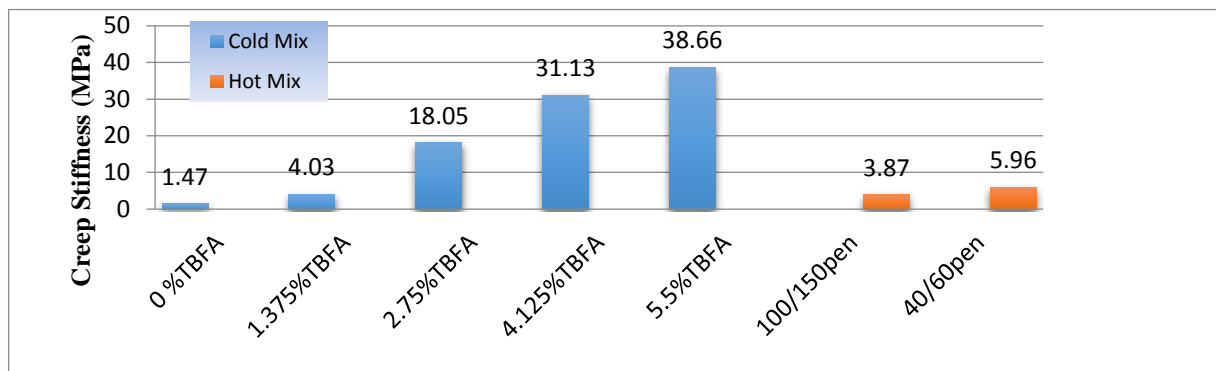


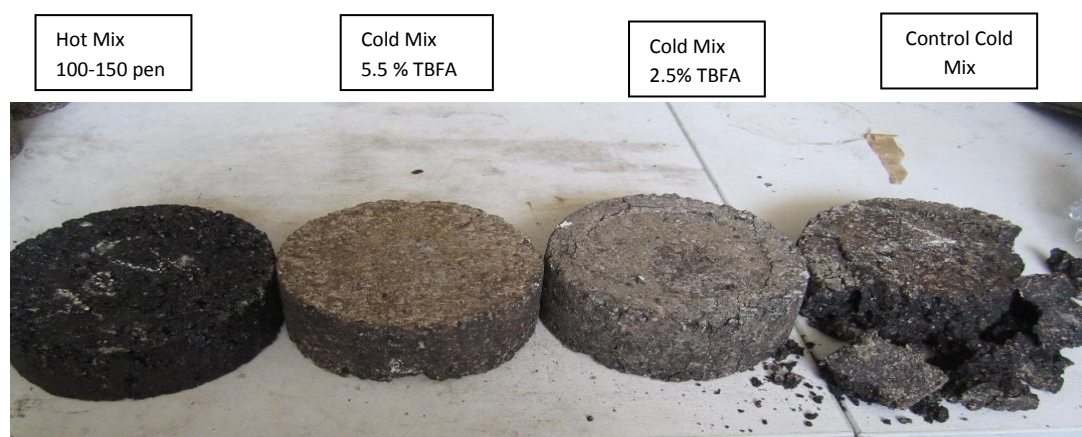
Figure 5: Accumulative creep strain versus pulse count of specimens with different % of TBFA

The specimen with 5.5 % TBFA, had creep stiffness approximately 26 times bigger than the control specimens under the same testing conditions figure 6; additionally, the same specimen had creep stiffness approximately 9 and 6 times bigger than 100-150 pen, and 40-50 pen hot asphalt mixtures respectively. This is a significant modification viewing the positive effect of TBFA in CBEM's.



**Figure 6: Effect of TBFA % on creep stiffness**

Figure 7 shows the final conditions of control and specimens containing the new replacing filler. Control specimens shown a total collapse, where the specimen with 5.5 % replacing filler does not show any sign of collapse. However, specimen with 2.5 % TBFA shown partial sign of collapse, whereas hot mix asphalt shown a mark of the upper loading plate.



**Figure 7: Shows the final conditions of control and TBFA modified specimens**

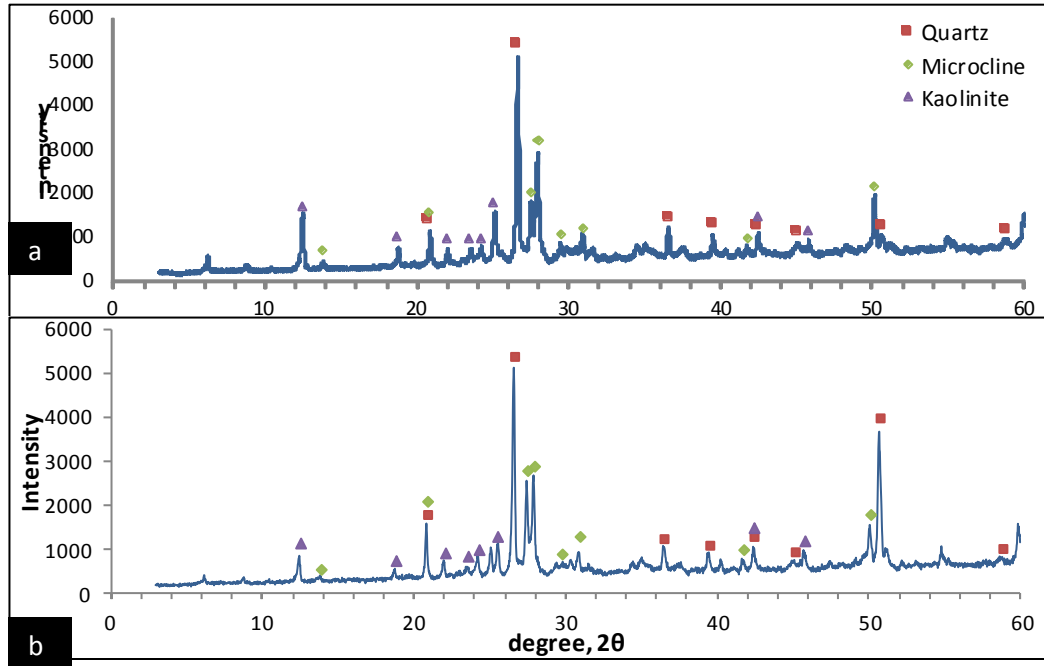
### 3.3.2 Microanalysis of Cold Mix Filer (CMF) and TBFA

#### XRD analysis of CMF

XRD analysis was achieved to investigate the changes in chemical phases of CMF after mixing with water and bitumen emulsion. The X-ray diffraction pattern of CMF powder is shown in Figure (8-a), while Figures (8-b,) illustrate the XRD patterns of the CMF mastic (after mixing with water and bitumen emulsion) at age of 28 days. From the Figure, the following can be concluded:

The crystalline components of the CMF powder are more or less Quartz ( $\text{SiO}_2$ ) with some feldspar Microcline ( $\text{K}_{0.92}\text{Na}_{0.08}\text{Al}_{0.99}\text{Si}_{3.01}\text{O}_8$ ) and Kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ). The analysis proved no significant change in crystalline phases between the CMF powder and the CM mastic. Whereas, the same phases of quartz, feldspar microcline and kaolinite still represent the crystalline phases after mixing with water and bitumen emulsion over the analysis periods. However, there is only limited release in the kaolinite phase to an amorphous phase, as can be seen when comparing Figure (8-a) with (8-b). This was expected, as the traditional mineral filler mineralogy has no hydraulic or pozzolanic compositions; it has very low contents of calcium and aluminium oxides. CMF mineralogical phases are inert phases, and thus they did not interact with water under used mastic preparation conditions. Pauwels et al.(1989) reported the feldspar with quartz could dissolute under condition of 180 °C and after few days to another phases of kaolinite and quartz. Nevertheless, these phases' are also inert phases.





**Figure 8: XRD patterns of CMF powder and CMF mastics**

### XRD analysis of TBFA

Figures (9a) show the XRD analysis of the dry TBFA, whereas figure (9, b-d) demonstrates the XRD patterns of powder produced from new filler mastic (after mixing TBFA with emulsion) which analysed after 2, 14 and 28 days of curing, respectively. The results shows significant changes occurred within the new filler mastic phases with time. Some phases were released and either completed at an early age like Lime, or with time like Mayenite and Gehlenite which took more time. Simultaneously, Portlandite ( $\text{Ca}(\text{OH})_2$ ) and Hydrotalcite ( $(\text{Mg}_4\text{Al}_2)(\text{OH})_{12}(\text{CO}_3)(\text{H}_2\text{O})_{3,5}$ ) appeared progressively, and the Calcite ( $\text{CaCO}_3$ ) increased too, see Figures (9, a-d)

Appearance of Portlandite and Hydrotalcite give the evidence of hydration process, which in turn prove that the new treated waste filler is a hydraulic material not pozzolanic. On the other hand, the new filler clearly contribute to increase the release of Kaolinite in contrast to CMF of figure 9a-d. This is mainly due to increase in the alkalinity of the mix.

Based on the above analysis, the authors can state that producing hydration products during the hydration process of the new treated filler are the main reason for the strength increase of the new cold mix asphalt developed in this research work.

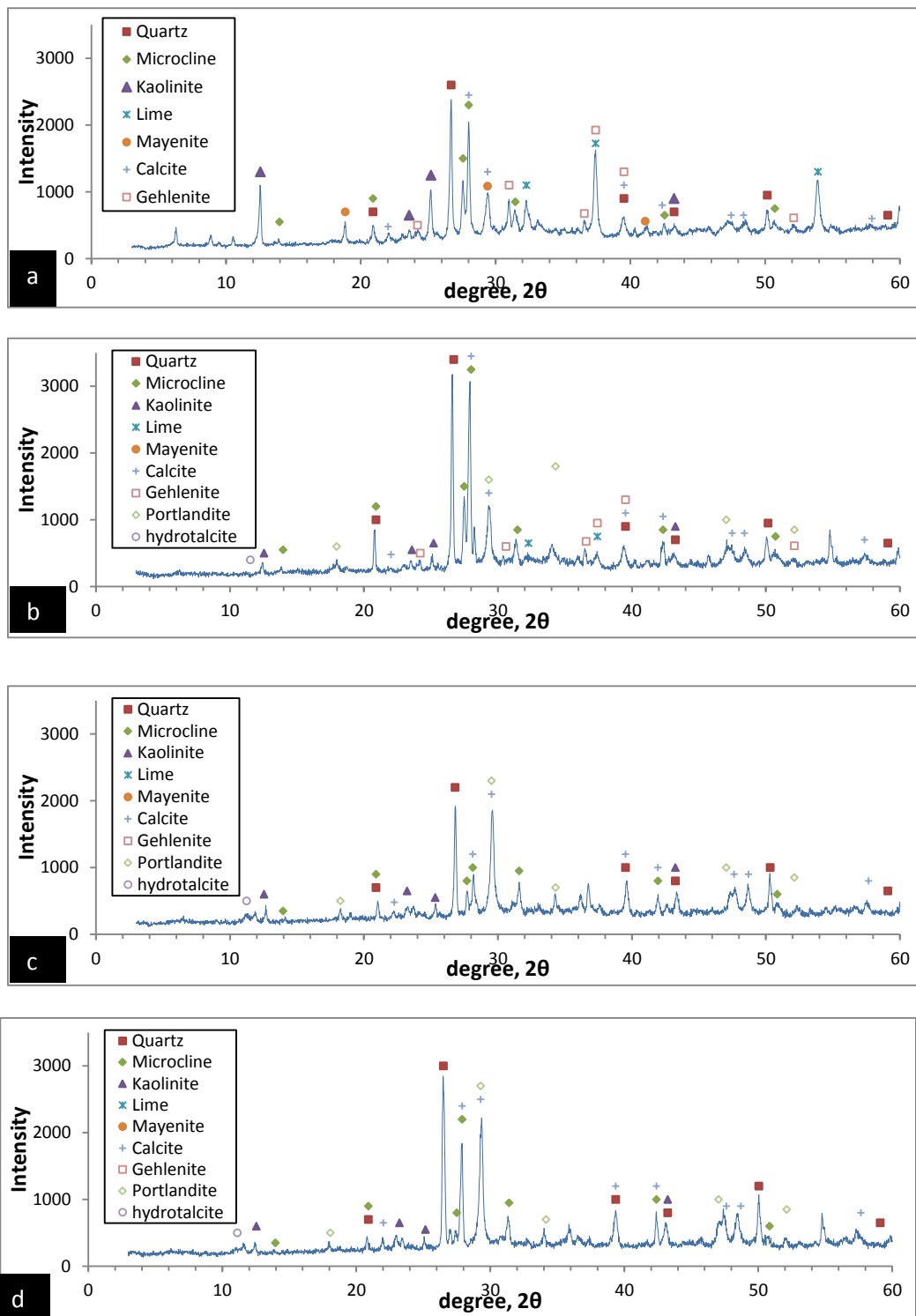


Figure 9: XRD patterns of the new treated biomass fly ash powder and mastics

#### 4. Conclusions

1. This experimental study has focused on studying the effect of a new treated biomass fly ash (TBFA) on improving the Engineering properties of close graded surface course Cold Bitumen Emulsion mixtures (CBEM's) in terms of stiffness modulus and creep stiffness.

2. Previous research works have shown that adding cementitious based fine materials such as Ordinary Portland Cement, Rapid Setting Cement, etc to CBEM's have shown successfully improvement in engineering properties. Unfortunately, these materials have a significant carbon foot print, and not cost effective. Whereas this study concentrate on the use of waste materials with significant low or non-carbon print.
3. The test results confirmed that there is a significant improvement in the stiffness modulus with use of TBFA, especially when 50% and more of the mineral filler is replaced with TBFA. More than 9 times the value of the traditional cold mix (non-treated mixtures) stiffness was achieved when all the mineral filler (i.e.5.5%) was replaced with TBFA. Furthermore, the result showed that cold mix stiffness modulus achieved is more than the HMA after 14 day at 20 °C curing. On the other hand, it was clearly shown that the curing temperatures played a considerable influence on stiffness modulus values.
4. The improvements in stiffness modulus due to the use of TBFA were due to two valuable TBFA's characteristics. Firstly, the TBFA water high absorptive ability which is obviously shown during mixtures preparation process especially in mixing. This property did not affect coating of the aggregates with emulsion. Secondly, the TBFA cementitious property clearly shown through the increment in the stiffness modulus with time of curing at all curing temperature.
5. The test results proved that there is a significant improvement in the permanent resistance of mixture containing TBFA, More than 26 time improvement in the creep stiffness in mixture containing 5.5% TBFA.

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