A Novel Emulsion-Based Mixture (EBM) Containing Ground Granulated Blast-Furnace Slag and Waste Alkaline Ca(OH)$_2$ Solution

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Abstract. Emulsion-based mixtures (EBM) attract less interest to use as a structural layer due to the weak mechanical performance, although it has environmental, economic, energy-saving benefits. Ground granulated blast-furnace slag (GGBS) activated by waste alkaline Ca(OH)$_2$ solution is used in the present investigation to replace the conventional limestone filler in a new Emulsion-based mixture (EBM). The mechanical properties by means of indirect tensile stiffness modulus test of the EBM with the activated GGBS are examined and compared with those of EBM contained traditional limestone filler. The results indicate that the activated GGBS by waste alkaline Ca(OH)$_2$ solution can enhance the ITSM in both early and mature ages. The hydration products can improve the bond strength inside the new EBM.

Keywords: Emulsion-base mixture; ground granulated blast-furnace slag; indirect tensile stiffness modulus; waste alkaline Ca(OH)$_2$ solution.

1. Introduction

Emulsion-based mixtures (EBM) provide benefits including a decrease in both economic and environmental impacts. Furthermore, EBM offers extended working time for both hauling and placement. So, EBM that make can be considered as a promising alternative to conventional bituminous mixes such as hot bituminous mixes (HMA) and warm mix asphalt (WMA) [1-5]. Such EBM is cold mix asphalt (CMA) that used asphalt emulsion as a binder. The total energy needed to prepare a tonne of CMA is 457 MJ, while HMA requires 680 MJ for each tonne. The consumption of energy in case of EBM is about 33% lesser in comparison to the HMA [6]. Besides, they found that 54 kg of pollutants...
and greenhouse gas emissions generated by each tonne of HMA, while CMA generates 36 kg for each tonne. Nevertheless, there are restrictions to the widespread use of said mixture due to the challenges faced by their mechanical performance. The poor strength at early ages for EBM and the prolonged time needed for curing to accomplish an optimal performance make this mixture type of low quality in comparison to HMA [7, 8].

Many techniques have been used to improve the performance of EBM. It was found by Brown et al. [9] that the degree of emulsion coalescence increased as compaction increased. Al Nageim et al. [10] performed research to evaluate the performance properties of emulsion-based bituminous mixes comprising 0 to 6% Ordinary Portland cement (OPC) substitution for traditional filler. OPC showed substantial enhancements in the indirect stiffness modulus test especially at higher content which was better than the hard and soft HMA at 14 days curing time. Also, incorporating natural and synthetic fibres into EBM technology appeared to improve the performance properties and reduce the curing time of such mixture [11, 12]. Also, Different finite element analysis has been performed to investigate the behaviour of EBM [13, 14].

The use of waste and industrial by-products in emulsion-based mixtures has been evaluated by many studies. Al-Busaltan et al. [15] and Al-Busaltan et al. [16] used waste cementitious materials in cold graded EBM. It was established that water susceptibility stiffness modulus, resistance to rutting were enhanced specifically when more than 50% of the new cementitious materials made from a waste domestic fly ash were used to substitute the limestone filler. A new developed binary blended filler formed from the fluid catalytic cracking catalyst and fly ash has been used in EBM [2, 17, 18]. It was found that using such materials providing cold asphalt concrete with fast-curing properties due to the enhancements in microstructural integrity.

Recently, a waste alkaline NaOH solution was used to produce fast curing and environmentally friendly EBM by activating the binary blended filler. Significant improvements were achieved in both water sensitivity and mechanical properties [2]. A ternary blended filler is generated when silica fume is added to the binary blended filler. The findings demonstrated that the ternary blended filler attained more enhancements in mechanical and durability properties in contrast to binary filler [19]. It was found that the cationic emulsifiers and charge on bitumen droplets are quickly broken down due to increasing the pH value causing coalescence and rapid flocculation of the emulsion [20].

More recently, A new binary blended filler, produce from calcium carbide residue and ground-granulated blast-furnace slag (GGBS) [21]. The production of the cementitious products including calcium silicate hydrate gel, ettringite and Portlandite confirmed substantial variations in the microstructure of components that have been noticed by the scanning electron microscopy during the curing period. This new binary blended filler made totally from waste materials, so it offers many advantages for the environment in comparison to a binary blended produced from fluid catalytic cracking catalyst residue and ordinary Portland cement [22]. Aziz et al. [23] stated that GGBS can be applied as an alternative cement, which provides significant technologies, economics, and ecological benefits for the concrete industry. This GGBS is formed when molten blast furnace slag is quickly cooled, through submersion in water in the generation of iron in the steel industry. It was found that the early strength of GGBS has been improved by accelerating its hydration through activated by carbide slag and wet-basis soda residue [24].

Alkali activation of waste and by-products through a chemical process can support the conversion of glassy structures into very compact and well-cemented composites. It was reported that some alkaline materials such as cement kiln dust could have a positive effect on the mechanical performance of CMA [25]. In theory, the material that can be alkali-activated should be comprised of silica and alumina. Comrie et al. [26] stated that once silica comes into contact with an alkali solution, it begins to dissolve. Researchers have used the above approaches to enhance the performance of EBM, other researchers used self-healing, polymer capsules, high-grade bitumen, fly ash to improve the performance and maintenance of bituminous mixes whether they were used in hot or cold mix asphalt [1, 27-32].

Developing EBM with acceptable early age strength with minimal time delay requirements and improved resistance to water ingress would be considered as a breakthrough in EBM research.
2. Materials

2.1 Mineral Aggregate
Aggregates used in this research are crushed granite with its fine and coarse components. AC14 close-graded surface course with 14 mm grading was chosen for this research complying with the British standard BS EN 13108-1 [33]. Table 1 shows the aggregate grading which is appropriate for all traffic conditions and all pavement layers.

Table 1. AC14 surface course -aggregate gradation

| Test sieve aperture size (mm) | % by the mass passing range | % by mass passing mid |
|-------------------------------|-----------------------------|----------------------|
| 20                            | 100                         | 100                  |
| 14                            | 100                         | 100                  |
| 10                            | 77-83                       | 80                   |
| 6.3                           | 52-58                       | 55                   |
| 2                             | 25-31                       | 28                   |
| 1                             | 14-26                       | 20                   |
| 0.063                         | 6                           | 6                    |

2.2 Fillers
Two fillers have been used through this study, the first is a commercial limestone filler (LSF) (obtained from Francis Flower Ltd) as reference material, while ground granulated blast-furnace slag (GGBS) (obtained from Hanson Cement Group) which is a by-product of the iron manufacturing industry was used as a replacement for the LSF. GGBS is generated from the grinding of blast-furnace slag. Both economic and environmental gains in terms of lowering CO₂ emissions could be achieved by incorporating industrial waste and by-product materials. Furthermore, reducing the impact on landfills can be achieved.

In this research, the main oxides and trace components for the chosen filler materials were examined using the Shimadzu EDX 720 energy dispersive X-ray fluorescence spectrometer. A Rigaku Miniflex Diffractometer was also used to detect the phase structure for the fillers. Besides, images of the materials after activation have been taken by a Quanta 200 Scanning Electron Microscope. Furthermore, the particle size distribution of the fillers evaluated by a Beckmen Coulter Laser diffraction particle size analyser.

2.3 Waste alkalinity Ca(OH)₂ solution
Waste alkalinity Ca(OH)₂ solution is discharged from the dewatering process of the calcium carbide residue. It has a pH value of 13.5 was used to activate the GGBS by making the hydration mediums with a high pH concentration to enhance breaking and dissolution in the glassy phases of the GGBS. This waste alkalinity Ca(OH)₂ solution replace the 3% pre-wetting water content in the EBM.

2.4 Bitumen & Bitumen Emulsion
The bitumen emulsion used in this study is (C50B4) which is a slow setting cationic bitumen emulsion provided by Jobling Purser, Newcastle, UK. The said emulsion comprising 50% residual bitumen and is based on 40/60 penetration grade base bitumen. It was approved by Thanaya [34], Nikolaids [35] and Dulaimi et al. [21] that this type of emulsion has high adhesion and high stability. On the other hand, bitumen binder grades of 100/150 pen and 40/60 pen were used to produce hot asphalt mixtures for comparison purposes.

2.5 Mix Design
The EBM mixtures have been designed following the (MS-14) procedure adopted by the Asphalt Institute (1989) (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design) [36]. The
indirect tensile stiffness modulus (ITSM) test has been used instead of the Marshall test. In order to mimic the manufacture, placing and compaction of the said mixes in field conditions, it was important to adopt a lab temperature of 20°C as the standard curing temperature for all the manufactured ITSM samples. The specimens were kept in their moulds for 24 hours and then extruded and kept at room temperature until testing at the ages of 3, 7, 14 and 28 days. So, this normal curing temperature was adopted to prevent any premature aging of the bitumen and simulate the manufacture, laying and compaction conditions of these mixtures in the field [7, 37, 38].

3. Experimental program
To investigate the mechanical performance of the EBM, a testing programme was set up for the evaluation of the effect of both GGBS and waste alkaline Ca(OH)₂ solution. The mechanical performance has been evaluated through the indirect tensile stiffness modulus test as below.

3.1 Indirect Tensile Stiffness Modulus Test (ITSM)
Stiffness modulus is related to the capacity of the material to distribute traffic loads, so it is an indicator of the structural performance of bituminous mixes. Many researchers have used the ITSM test as an indicative test to rank EBM [16, 18, 39-41]. This test was performed following the BS EN 12697-26 [42] utilizing the HYD-25 Cooper Research Technology testing machine displayed in Figure 1. According to BS EN 12697-26, a target horizontal deformation of 5±2 μm has been achieved by an impulsive load with a rise-time of 124±4 ms. In this test, ten conditioning pulses were applied to the samples before five test pulses. The equation below (Equation 1) has been used to calculate the stiffness modulus the test data load and the horizontal strain was measured and kept. The calculated stiffness is the average value after measurements were repeated along two diameters. At a minimum of three specimens have been used for each mix type.

$$S_m = \frac{L}{D \times t} \times (v + 0.27)$$  \hspace{1cm} (1)

Where:
- $S_m$ = ITSM, MPa.
- $L$ = applied vertical load, N.
- $D$ = horizontal diametric deformation.
- $t$ = the mean thickness of the samples, mm.
- $v$ = Poisson’s ratio.

![Figure 1. Cooper HYD-25 testing apparatus.](image-url)
4. Results and discussion

4.1 Characterisation of the selected mineral fillers

The role of the filler in EBM; their performance in the hydration process could be influenced by their chemical composition. It was revealed through the energy dispersive X-ray fluorescence spectrometer (EDXRF) that the chief oxides for the limestone filler (LSF) are calcium oxide (CaO) and silicon dioxide (SiO$_2$). It can be seen that LSF has a high ratio of calcium oxide, around 78%, however, no hydration occurs when in contact with water due to that calcium oxide occurs in a non-hydrated state, so it is considered an inert material. Silica, calcium, aluminium and magnesium oxides comprise the major components in GGBS. Table 3 summarizes further details regarding the physical properties of the fillers. It is essential to characterize the surface texture and the shape of the tested fillers, so it can be seen from the SEM micrographs that the particles for the LSF and GGBS are irregular in shape and the surface texture is sharp, Figure 2. The X-ray diffraction pattern revealed that the major crystal peaks of the LSF were calcite and quartz, while an amorphous nature has been detected for the GGBS with a halo between the 25° and 35° (2$\theta$) range, Figure 2. The particle size distribution of fillers displayed in Figure 4. It can be seen that the two fillers are fine and well-graded, this could make an improvement in the mix packing properties.

Table 2. Chemical properties of the LSF and GGBS

| Chemical structure | LSF   | GGBS  |
|--------------------|-------|-------|
| CaO, %             | 77.82 | 40.35 |
| SiO$_2$, %         | 17.21 | 37.23 |
| Al$_2$O$_3$, %     | 0.0   | 5.73  |
| MgO, %             | 0.89  | 4.22  |
| Fe$_2$O$_3$, %     | 0.0   | 0.01  |
| K$_2$O, %          | 0.35  | 0.0   |
| SO$_2$, %          | 0.01  | 0.0   |
| TiO$_2$, %         | 0.19  | 0.63  |
| Na$_2$O, %         | 2.27  | 0.0   |

Figure 2. SEM image for dry powder of both LSF and GGBS.
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4.2 Indirect Tensile Stiffness Modulus Test (ITSM)

In order to recognize the optimum composition in EBM which provides the highest ITSM value at 3 days, GGBS was used as a full substitution for the LSF, introduced in 6% by weight of dry aggregate. Figure 5 demonstrates the influence of GGBS when replaced the LSF, with no significant increase in ITSM values by this replacement.
The second stage has been done by introducing an alkaline activator which is the waste alkalinity Ca(OH)$_2$ solution to the GGBS by increasing the pH concentration of the hydration mediums. The waste alkalinity Ca(OH)$_2$ solution replaced the 3% pre-wetting water content. The ITSM for the new alkaline GGBS activated mixture (AAGG) increased by around 7 times in comparison to the same mixture with water for pre-wetting.

This developed AAGG mix has almost a similar ITSM compared to the 100/150 HMA at 3 days. The AAGG mix also surpassed conventional LSF and GGBS mixes by around 8 and 7 times at 3 days of age, Figure 6. There was also a continued increment in the ITSM for the AAGG mix over time, Figure 7.
It was revealed that the reaction kinetics of the GGBS is directly impacted by the addition of the alkaline activator. This highly alkaline activator attack GGBS particles and induce them to continue to dissolve. Also, the high pH of the aqueous solution supports accelerating the breaking of bitumen emulsion in the AAGG. This represents a remarkable achievement from both ecological and commercial perspectives. This AAGG mix performed better than 100/150 HMA at 28 days by increasing the ITSM to around 2.5 times. So, a remarkable achievement from both economic and ecological perspectives has been achieved. Besides, the new AAGG mix has more than 80% of the ITSM of the 40/60 HMA at 28 days.

4.3 Scanning Electron Microscope Observation

The observation of a scanning electron microscope has been used by many researchers to confirm the generation of cementitious products [18, 21, 39]. Figure 8 presented the microstructure of the AAGG pastes at 3 and 28 days of curing after being examined. This figure reveals a dense microstructure that has increased considerably over time when water was replaced by a waste calcium hydroxide solution. The micrograph of the AAGG paste at 3 days of age demonstrates the production of an essential product which is calcium silicate hydrate (C-S-H) gel where most of the engineering properties of the cement paste are related to this product. It can be observed that the GGBS particles have been replaced by hydration products, C-S-H gel. The surface enriched by the hydrates and the microstructure of the AAGG has become denser at 28 days. The improvements in the ITSM results in Figures 6 and 7 proved that the said hydration products are not affected when incorporated into the AAGG mix. This means that there are two binders works together, the cementitious products and bitumen, inside the AAGG mix. Rapid flocculation and coalescence of the emulsion have been generated by the fast broken down of the charge and cationic emulsifiers on bitumen droplets due to the increasing the pH value. The presence of the waste alkalinity Ca(OH)₂ solution quickening setting of the cationic asphalt emulsion as a result of the increases in the pH and this permitting quickly flocculation and coalescence of the bitumen emulsion, as confirmed by Wang et al. [20].
5. Conclusions
   1. Waste calcium hydroxide solution was used to form a high alkali environment for the activation of GGBS which is rich in calcium oxide (CaO). This has increased the hydraulic reactivity of the cementitious elements and improved the ITSM.
   2. The weak early strength of the LSF mix in terms of ITSM has been improved by around 8 times at just 3 days of normal curing by using GGBS activated by the waste calcium hydroxide solution. The increase in the pH value causes a rise in the coalescence rate of the bitumen emulsion. Besides, a substantial enhancement was achieved in ITSM at mature ages (28 days) by around 9 times.
   3. Hydration products have been formed such as calcium silicate hydrate gel, ettringite and portlandite (CH) has been approved through the SEM observations at various curing ages. ITSM has been improved throughout these products and the bitumen binder by building up a dense microstructure.
   4. The long curing time in the case of traditional EBM which is required to achieve ultimate strength has been reduced to less than 3 days through this superior performance of AAGG mix.

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