

The Development of a Novel, Microwave Assisted, Half-Warm Mixed Asphalt

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Abstract

Global warming is an imminent threat that the world and its inhabitants have to confront. As such, it is the duty of the pavement industry as a contributor of greenhouse gas emissions, to pave the way by lowering its carbon footprint. It is vital that new measures are adopted in order to do so such as employing the use of emulsion-based mixtures (EBM). The problem with this is that the performance properties of such mixtures are inferior to that of traditional hot mix asphalts (HMAs). The air void content of EBM is very high and considered unacceptable by road engineers for application as a surface layer. That said, these mixtures are not only environmentally friendly but also boast ecological and economic advantages. An innovative approach was applied in this research by using a pre-compaction microwave processing technique to develop a novel, half-warm mix asphalt mixture (H-WM). This new mix was

shown to have improved mechanical properties and lower air void content. EBM mixtures comprised of cementitious binary blended filler, were prepared using microwave heating applied over different lengths of time. Stiffness modulus, air voids content and temperature were used to establish the optimum microwave radiation time. The results indicated that 1.5 minutes of microwave processing decreased air void content from 8.92% to 7.12%. A 7% improvement in stiffness modulus was also found and the temperature was within lower limits (43 °C). Hydration was accelerated by the microwave radiation, and the demulsification of bitumen emulsion was promoted. Microwave processing was found to have a positive impact on permanent deformation at elevated temperatures in comparison to the two reference HMAs used. It also proved to be an adequate technique to produce a fast-curing H-WM with lower air voids content. Water damage resistance for the microwaved H-WM (99%) is better than the reference HMA mixes. The findings of this study show that the novel half-warm asphalt mixture has superior properties in comparison to EBM.

Keywords:

Cold mix asphalt; half-warm mix asphalt; microwave heating; stiffness modulus; sustainability; waste fillers; wheel track test.

1. Introduction

Global warming has raised concerns regarding the use of hot mix asphalt throughout the asphalt industry because of its negative environmental impact. Asphalt mixtures are categorized into four categories according to the mixing temperature level, namely: Hot Mix Asphalt (HMA), Warm Mix Asphalt (WMA), Half-Warm Mix Asphalt (H-WM) and Cold Mix Asphalt (CMA). The main differences between these technologies are laying and compaction temperatures. These mixtures are manufactured and laid at the following temperatures: 150-190 °C, 100-140 °C, 60-100 °C and 0-40 °C, respectively. As such, the production and placement of H-WM mixtures are lower than those used for HMA by around 50-130 °C [1-4]. There is some debate about the upper limits of CMA, some researchers suggested that the term CMA has been applied to the production of bituminous mixtures at a temperature up to 60 °C [5-7]. It is significant to note that the consumption of energy when producing CMA is around 95% less than both HMA and WMA [8]. H-WM consumes 50% less energy in comparison to HMA, WMA consuming 10-30% less [9, 10].

Emulsion-based mixtures (EBMs), are a distinctive type of CMA offering an alternative and promising technique of pavement construction, whilst being environmentally sustainable and economically viable. EBM is a combination of asphalt emulsion, fine and coarse aggregates, mineral filler and water (external), prepared and compacted at ambient temperatures. Some researchers have suggested the use of additives to upgrade its properties, i.e. cement, fly ash, polymer, crushed glass and a variety of waste and by-products [11-15]. Theoretically, these mixtures could see widespread use as structural layer materials [16, 17]. However, their performance is poor and considered inferior to HMA due to shortcomings such as long curing times, weak early-life strength, lower stability and high air voids content in the mixtures when compacted [11, 18-20]. To date, such mixtures have seldomly been used for structural layers in heavy-duty pavements, being limited to reinstatement work and surface treatment on low-

trafficked roads [12, 16, 21]. Similarly, EBM is commonly used for maintenance and other small construction works such as utility reinstatement in the form of cold recycled asphalt [8].

Despite this, additives have been shown to increase EBM's durability and mechanical properties [22]. Various researchers have used cement as an enhancer, this enhancing the bond between the emulsion and aggregate [23-28]. However, cement manufacturing inversely impacts the environment [29, 30] as significant amounts of CO₂ (5%-7%) are released into the air during production.

In order to encourage sustainable development, waste and by-product materials from different types of industries can be recycled and used as sustainable cementitious fillers in EBM. Hydration products consume water and speed up the emulsification of bitumen emulsion [31, 32]. For example, the indirect tensile stiffness modulus of EBM is improved significantly by using a spent catalytic cracking catalyst and calcium-rich fly ash [33]. Glass and hemp fibres have also been used by Shanbara et al. [34] to improve the water sensitivity of EBM, these mixtures successfully reinforced with natural fibres (jute and coir) to mitigate permanent deformation [35].

However, the high levels of air voids content in EBM mixtures are far from ideal [33, 36, 37]. It has been established that reductions in the service life of untreated EBM for stone matrix asphalt and asphalt concrete, was due to high air voids content in such mixes in comparison to their HMA counterparts [38]. Ibrahim [39] reported that the stiffness modulus of bituminous mixtures was increased by reducing the air voids content, the resulting strain lower at a given stress level, leading to an extended fatigue life.

WMA technology involves reducing asphalt binder viscosity, this enabling the binder to achieve optimal viscosity to coat the aggregate [40, 41]. WMA technologies can be divided into three categories defined by the use of chemical additives, organic additives, and water-

based or water-containing foaming processes which eventually affect the degree of temperature decrease [3]. However, additional costs created by the use of additives, can be compensated to a certain extent, by reductions in the manufacture temperature [42]. Decreasing the production temperature of WMA reduces energy consumption, this in turn, lowering the production of greenhouse gases [43, 44]. As a result, better working conditions can be provided for workers during paving operations [45, 46].

Unlike CMA which is restricted to the repair of damaged pavements on low trafficked roads, the mechanical performance of both WMA and H-WM is comparable to that of HMA, so their use is on the rise [21, 47]. H-WM has significantly reduced combustion gases in ratios varying from 58% to 99.9% for SO₂ [21]. Such mixtures use bitumen emulsions and foamed bitumen [46, 48-50], where the aggregate is mixed with emulsion after heating each to 90-95 °C and 60-65 °C, respectively. Atmospheric emissions decrease significantly when bituminous mixes are produced at lower temperatures [51]. H-WM reduces the mixing temperature to 60 °C through the inclusion of foamed bitumen. Chomicz-Kowalska et al. [48] found that foamed bitumen concrete modified with 2.5% FT synthetic wax, met the requirements for water resistance for one freeze cycle. Aggregates do not have to be fully dry to be used for foamed bitumen [50, 52], as there is a positive impact on the additional foaming process due to a thin water film on the surface of the aggregates [50]. Lizárraga et al. [49] demonstrated that a half-warm mix made with reclaimed asphalt can be compared to that of HMA by means of resistance to permanent deformation and fatigue, this further increasing potential ecological and economic advantages. Van de Ven et al. [50] reported that H-WM can offer similar fatigue properties in addition to comparative monotonic properties at elevated temperatures. As such, H-WM provides a wide range of ecological, technical, and economic advantages. With lowered production, laying and compacting temperatures, H-WM provides an array of benefits

including, but not limited to: better working conditions, reduced GHG emissions, lower energy consumption, an extended paving window, and greater hauling distances [49].

The three primary methods of heating asphalt are: induction heating (coil) [53, 54], microwave heating [55-57] and infrared heating [58]. Microwaves are a type of electromagnetic wave comparable to radio waves but with lower wavelengths ranging from 0.003 to 3 m and frequencies which range from 100 MHz to 100 GHz. Microwave heating has a higher heating rate for asphalt concrete, the heating process itself more homogeneous than that of the other two methods [58, 59].

The application of microwave technology has received a lot of attention recently regarding the maintenance of bitumen pavements [60]. Wang et al. [61] reported that microwaves have many benefits when compared to traditional heating, including a fast thermal response, higher thermal efficiency, no direct contact with liquids and phase selective heating. In contrast to conventional heating, microwave heating is energy-saving, has fast and high heating rates and is less damaging to the environment [62, 63].

An important point to note is that oil-in-water emulsions can be effectively separated using microwave processing [64]. Al-Kafaji et al. [15] described how microwave irradiation facilitates and speeds up emulsion breaking due to the lineup of molecule charges which decrease emulsion stability. Microwave heating of asphalt concrete in the laboratory has a higher rate of heating and is more homogeneous in comparison to infrared and induction heating (coil) [57].

Somaratna et al. [65] reported that the major difference between microwave heating and conventional heating techniques is that microwaves can penetrate the material and promote heating from the inside, this resulting in volumetric heating. In contrast, conventional heating generates non-uniform heating thermal gradients in the material that could lead to less than

ideal properties. Furthermore, microwaves have also been used to promote the self-healing of asphalt mixtures , whilst also establishing that the ideal steel wool content is about ten times smaller in comparison to that suggested by electromagnetic induction, thus reducing costs [66].

Microwave application has been investigated by Nieftagodien [67] as an energy-efficient method to generate an H-WM using crushed aggregates and heat reclaimed asphalt mixes. This technique provided high and fast volumetric heating meaning that productivity can be improved when using appropriate materials. Finally, microwave heating is one of the three major ways to accelerate the process of self-healing in bituminous mixes, along with electromagnetic induction and microcapsule technology [66, 68, 69]. There is an industrial microwave system which uses two types of processing; batch and continuous operations, such systems commonly used in paper, food, and paint industries. However, in the USA, microwave heating has been used for pavement maintenance processing from the 1970s [70]. It goes without saying that making use of microwaves as part of field asphalt technology will raise some challenges, but ongoing research, testing and development will overcome these.

At present, knowledge about the manufacture of H-WM by microwave processing is limited to a few pieces of research investigating microwave heating of bituminous mixtures. This research has attempted to develop an innovative, original, and novel product: a half warm mix asphalt that will combat the drawbacks of emulsion-based mixes, specifically the stiffness modulus and air voids content. To achieve this, two sustainable approaches have been starting with the replacement of mineral filler with waste and by-product materials followed by processing of the mix using microwave irradiation during the production stage. Such an H-WM represents a cleaner manufacturing technology for bituminous mixes.

2. Materials

2.1. Aggregate

Crushed granite aggregates were used in the manufacture of the bituminous mixes. The gradation used, was one that met the recommendations for close graded HMA surface course, complying with BS EN 13108-1 specification [71], as shown in Figure 1. The water absorption and apparent particle density for the coarse aggregates were found to be 0.7% and 2.65 Mg/m³, respectively. For the fine aggregate, water absorption was 1.5% with an apparent particle density of 2.64 Mg/m³.

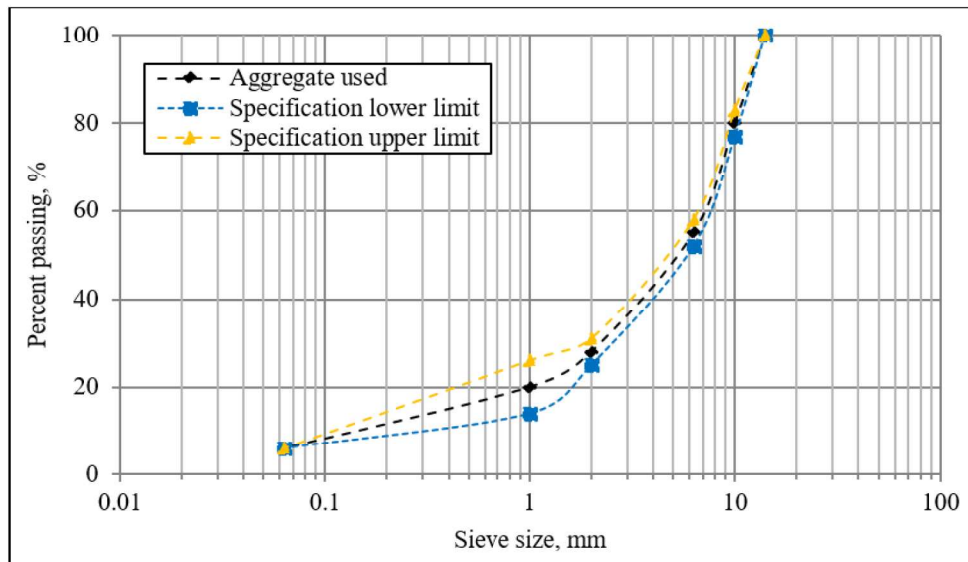


Figure 1. Aggregate gradation chart

2.2. Bitumen Emulsion and Asphalt

Cationic, slow-breaking bitumen emulsion with a 50% base asphalt content was chosen. Classified following BS EN 13808:2013 [72] as C50B4, it is commercially known as SPRAYCO CAB 50 and is designed to be applied as a binder for onsite, cold mixed asphalt. This was used to confirm the improved adhesion between aggregate particles for all cold emulsion mixtures. The two predominant parameters of this emulsion are high stability and adhesion. Asphalt emulsion has to maintain the mixtures' flexibility and to provide water for

the hydration process. Table 1 lists the properties of C50B4, provided by Jobling-Purser, Newcastle, UK, in large containers (25 kg).

For comparison, two grades of asphalt, a soft asphalt of penetration grade 100/150 that has a penetration of 142 and a hard asphalt of penetration grade 40/60 with 49 penetration, were selected to manufacture the HMAs.

Table 1. Bitumen emulsion properties

| Essential characteristics and standard | Value |
|--|--------------------|
| Appearance | Thick Black |
| Breaking Value (EN13075-1) | 110-195 / class 4 |
| Viscosity (Efflux time 2 mm at 40 °C) (EN 12846-1) | 15-75 s / class 3 |
| Penetration (EN 1426) | ≤ 50 dmm / class 2 |
| Bitumen content (%) (EN 1428) | 50 |
| Softening point (EN 1427) | ≥ 55 °C / class 3 |

2.3. Fillers

Limestone filler (LSF), ground granulated blast furnace slag (GGBS) and calcium carbide residue (CCR), were used as filler materials. The LSF was provided by Francis Flower, UK; the GGBS supplied by Hanson, UK, and the CCR obtained from BOC, UK & Ireland. Large, wet blocks of CCR had to be broken into tiny parts and oven-dried at 110 °C for 24 hr. The resultant pieces of CCR were then ground using a mechanical grinder. A pestle and mortar was also applied for 15 min to prevent particle agglomeration.

LSF is widely used as a commercial filler in HMA and CMA. GGBS is a cementitious material generated as a by-product of the iron production process. It has a comparable chemical composition (silica, calcium oxide, and alumina) to ordinary Portland cement (OPC) making it a promising sustainable material to use as a filler in bituminous mixes [14, 73, 74]. However,

the hydration process of GGBS is slow, so chemical activators are usually used to speed up its hydration [75, 76]. It has been reported that alkali-activated GGBS performs with satisfactory mechanical strength, the major hydration product being calcium silicate hydrate which has a strong binding capacity [77-79]. CCR is an industrial waste created through the generation of acetylene and leads to environmental pollution if disposed of inappropriately. It has been found that CCR can successfully accelerate the hydration of GGBS because of its Ca(OH)_2 , this leading to the generation of more hydration products [73].

The X-ray diffraction (XRD) of the fillers were examined using a Rigaku Miniflex diffractometer. There was no evidence of the existence of amorphous phases for the CCR which comprises mainly of Portlandite and calcite: GGBS does have an amorphous nature. LSF is composed of quartz and calcite, as can be seen in Figure 2.

The Particle size distribution (PSD) of the selected fillers are displayed in Figure 3. The PSD of the GGBS powder shows that around 90% of the particles are smaller than 40 μm . The average grain size (D50) was approximately 24.6 μm for the CCR (ground for 15 mins), the dominant particle size falling below 25 μm . The PSD of the LSF shows that 90% of the particles pass through a sieve size 80 μm .

An X-ray fluorescence spectrometer (Shimadzu EDX 720, energy dispersive) was used to chemically analyse the selected fillers. Lime and silica are the main oxides found in CCR, while GGBS is comprised of lime, silica, alumina, sodium and magnesium. These findings are in common with those found by Du [17] and Hanjitsuwan et al. [80]. LSF has a high amount of CaO in its structure (Table 2).

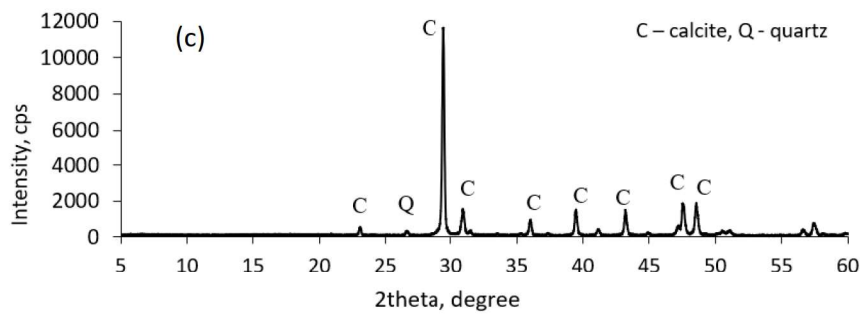
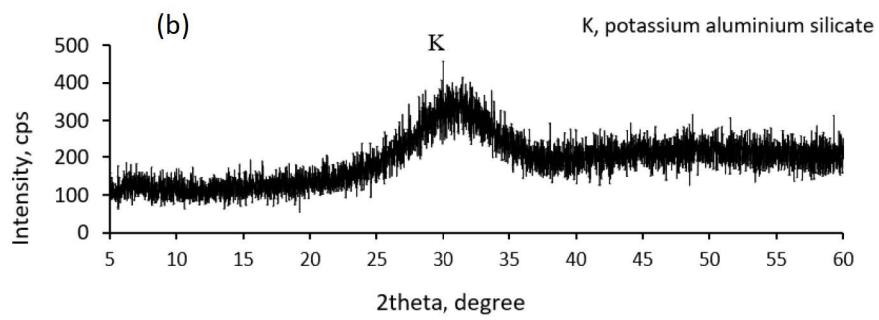
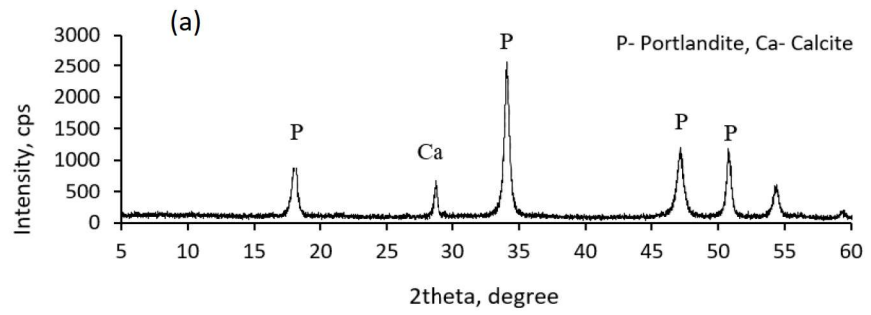


Figure 2. Powder XRD pattern of (a) CCR, (b) GGBS and (c) LSF

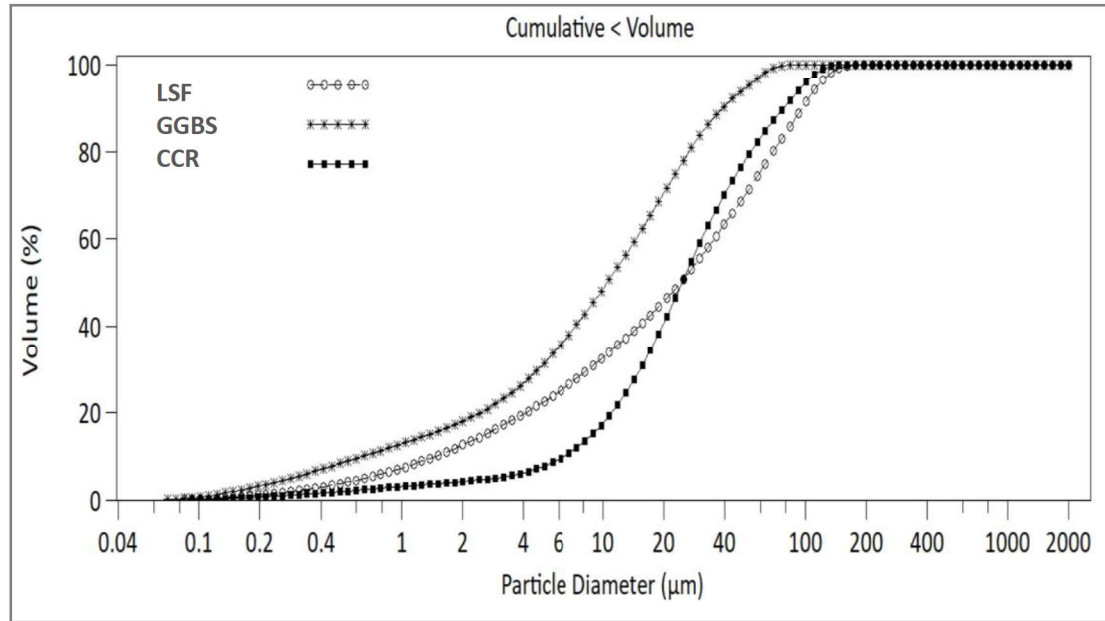


Figure 3. Particle size distribution for the selected fillers

Table 2. Chemical properties of CCR, GGBS and LSF

| Chemical composition | CCR | GGBS | LSF |
|------------------------------------|-------|-------|-------|
| CaO, % | 81.84 | 43.78 | 77.82 |
| SiO ₂ , % | 14.08 | 37.31 | 17.21 |
| Al ₂ O ₃ , % | 0.90 | 5.83 | 0.0 |
| MgO, % | 0.77 | 4.89 | 0.89 |
| Fe ₂ O ₃ , % | 0.00 | 0.83 | 0.0 |
| SO ₃ , % | 0.77 | 0.71 | 0.01 |
| K ₂ O, % | 0.20 | 1.56 | 0.35 |
| TiO ₂ , % | 0.12 | 0.11 | 0.19 |
| Na ₂ O, % | 1.32 | 2.86 | 2.27 |

2.4. Manufacture of Specimens

The emulsion-based mixtures were designed using the Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design (MS-14) [81]. The optimum asphalt emulsion content was 12.5% with a pre-wetting water content of 3%. GGBS was used to substitute conventional LSF, while CCR was used to replace some of the GGBS. The role of the CCR is to activate the GGBS by providing an alkaline medium.

Two sets of specimens were made for the indirect tensile stiffness modulus requirements, the first made using the aforementioned fillers without microwave heating, the second set treated by microwave heating. The pre-heating process applied before compaction lowers the air voids content in this new mix. Other advantages include energy-saving and volumetric heating in comparison to conventional heating.

Initially, the aggregates and fillers were combined in a Hobart mixer. Pre-wetting water was introduced to improve the bond between the aggregate and the bitumen emulsion which was gradually poured in later. All the mixtures in the second set were heated by placing the samples in a domestic microwave oven (Figure 4) which generated microwaves of up to 800 W, with a frequency of 2.45 GHz, over different times. Compaction was immediately conducted by applying 50 blows of a standard Marshall hammer to both sides. The compacted specimens were stored in their moulds at lab temperature for 24 hrs, then extruded. The specimens remained at room temperature for 3, 7, 14 and 28 days.

At the same time, samples of HMA were made with 5.1% optimum bitumen content by weight of aggregate, as per the requirements of PD 6691:2010 [82]. These samples were mixed and compacted, the 100/150 and 40/60 HMAs mixed at 150-160 °C and 160-170 °C, respectively. The equivisous procedure suggested by the Asphalt institute MS-2 [83], was used to determine the mixing and compaction temperatures for the HMAs. Table 3 displays the proportions of all mixes.



Figure 4. Microwave process

Table 3. Specifics of mix proportions.

| Mix type | Fillers type | Bitumen emulsion, % | Microwave time, min |
|-------------|------------------|----------------------|---------------------|
| LFS | Limestone filler | 12.5% | / |
| CBBF | 4% GGBS + 2% CCR | 12.5% | / |
| H-WM | 4% GGBS + 2% CCR | 12.5% | 1.5 |
| HMA 100/150 | Limestone filler | 5.1% bitumen 100/150 | / |
| HMA 40/60 | Limestone filler | 5.1% bitumen 40/60 | / |

3. Laboratory Testing Programme

The laboratory performance tests included an indirect tensile stiffness modulus test, wheel tracking test and fatigue resistance to calculate the mechanical properties of the mixes, while air voids content were determined to evaluate the volumetric properties. The microstructure was analysed and examined by scanning electron microscopy.

3.1. Indirect Tensile Stiffness Modulus (ITSM) Test

In general, stiffness is an essential index of bituminous paving materials performance as it identifies the capacity of each pavement layer to dispense loads to the layer below. Load frequency and material temperature are impacted by the stiffness magnitude of bituminous materials [84]. In common with other bituminous mixtures, EBM mixes have been successfully

evaluated and ranked by their ITSM [36, 85, 86]. The specimen in this test was subjected to five transient load pulses through its perpendicular axis. Two Linear Variable Differential Transducers (LVDTs) were mounted on the horizontal axis to measure the resulting indirect deformation. The specimens were stored at 20 °C for 4 hours prior to testing, the test performed following the standard procedure BS EN 12697-26 [87].

3.2. Permanent Deformation Resistance test

Repeated traffic loading causes a longitudinal depression in the wheel path, this known as permanent deformation or rutting. Increased axle loads and tyre pressure cause the most significant distress i.e., rutting. A wheel tracking test was used to identify the permanent deformation of the emulsified asphalt mixes as it can simulate field conditions effectively. A small device procedure following BS EN 12697-22 [88], was conducted at 45 °C as this represents moderate to heavily stressed sites based on the British Standard PD 6691:2010 [89]. The loose H-WM mixture was treated for 1.5 minutes by microwave heating before compaction via roller compactor, according to BS EN 12697-33 [90], the resulting sample a slab of dimensions 400 x 305 x 50 mm. The test itself is the application of a forward and backward movement of a single wheel on the surface of the slab, the perpendicular deformation along the wheel path measured using LVDT. The moving wheel has a rubber tyre of diameter 200 mm and width 50 mm. For accuracy, two specimens were created for each mixture type.

3.3. Fatigue test

The site performance of any pavement is impacted by the presence of fatigue cracking [91]. HMA's resistance to fatigue cracking is mainly influenced by the properties of asphalt mixtures [92], therefore investigating the ability of materials to resist fatigue is essential to prevent such failure. A four-point bending test (4 PB), as specified by BS EN 12697-24 [93], is commonly used to identify fatigue characteristics. Common fatigue failure criterion is defined by fatigue

life (Nf), which is the number of cycles associated with a 50% reduction in the initial stiffness of the asphalt mix. It is commonly reported that up to 200 microstrains occur in a pavement structure, but this is dependent on variables like subgrade bearing capacity, layer thickness, load magnitude and the mixture type [94]. In this research, the experiments were performed at a testing temperature of 20 °C and a frequency of 10 Hz with a controlled strain mode, under a sinusoidal waveform of two strain levels, namely 150 $\mu\epsilon$ and 200 $\mu\epsilon$. Prismatic shape samples of 400 x 50 x 50 mm, were made by sawing compacted slabs to the required dimensions.

3.4. Water Sensitivity Test

When asphalt mixes absorb water, bitumen is stripped away. This causes premature asphalt distress and may result in failure of the pavement due to inadequate adhesion between the aggregate and the bitumen binder. This in turn causes a lack of cohesion and a reduction in the stiffness of the bitumen film [95]. Resistance to water damage for all the mixes was calculated by the stiffness modulus ratio (SMR) following BS EN 12697-12 [96].

A Marshall hammer was used to manufacture the samples which were then divided into two sets, each set comprising of three specimens. After 1 day in their moulds, the samples for the dry (unconditional) set were kept for seven days in the laboratory and then subject to an ITSM test. The samples for the wet set (conditional) were extruded from their moulds and saturated in a water bath at 40 °C for 72 hours after being vacuumed for 30 minutes at 6.7 kPa. They were then stored for four days in the laboratory. Both the conditioned and the unconditioned specimens were examined for ITSM at 20 °C. SMR represents the ratio of stiffness modulus after conditioning, over stiffness modulus before conditioning.

3.5. Microstructure analysis

The paste samples of the new cementitious binary blended filler were prepared and examined at 3 and 28 days. Small, thin pieces were broken from the paste samples, each piece

approximately 5 mm diameter and 1 mm thickness. The samples were subjected to microwave processing after preparation, their microstructure analyzed at 3 and 28 days. The microstructure was analysed by a Quanta 200 scanning electron microscope with a 10 kV applied accelerating voltage, at a testing temperature of 20 °C.

4. Results and Discussion

4.1. Performance of EBM comprising a cementitious binary blended filler

A new cementitious binary blended filler (CBBF), composed of ground-granulated blast-furnace slag and a calcium carbide residue, has been developed and found to be remarkably effective at generating an improved EBM [14]. The findings indicate that there is a substantial improvement in ITSM, as can be seen in Figure 5. It is suggested that this is due to the creation of hydration products. CCR provides the medium needed to activate the latent hydraulic material GGBS, and because of its high alkaline nature (pH=13.1), it accelerates gains in ITSM, as a consequence of the broken cationic asphalt emulsion. It has previously been reported that changing the pH of emulsions, destabilizes them [97].

The generation of ettringite needles, Portlandite and calcium silicate hydrate (C-S-H) gel, supports stiffness enhancement at both early and later stages. Previous investigations have revealed that the optimum blend (cementitious binary blended filler - CBBF) of GGBS and CCR is 4% and 2% by the total mass of aggregate, respectively [14].

Figure 5 illustrates the evolution of the ITSM test results across different curing times (3, 7, 14 and 28 days) for the mixtures containing traditional LSF, CBBF and the two references hot mixes. A maximum stiffness modulus of 1678 MPa was measured in the CBBF after three days curing, achieving nine times the improvement seen in the LSF mix. There were considerable improvements in ITSM for the CBBF mix across all tested curing times, exceeding the ITSM of HMA 100/150 within 3 days normal curing. Its performance was 2 times better than that of the HMA 100/150 at 7 days. The CBBF mixture has almost 90% of ITSM found in hard HMA

40/60 at 28 days normal curing. The ITSM tests were conducted at a temperature of 20 °C as per the standard BS EN 12697-26 [87].

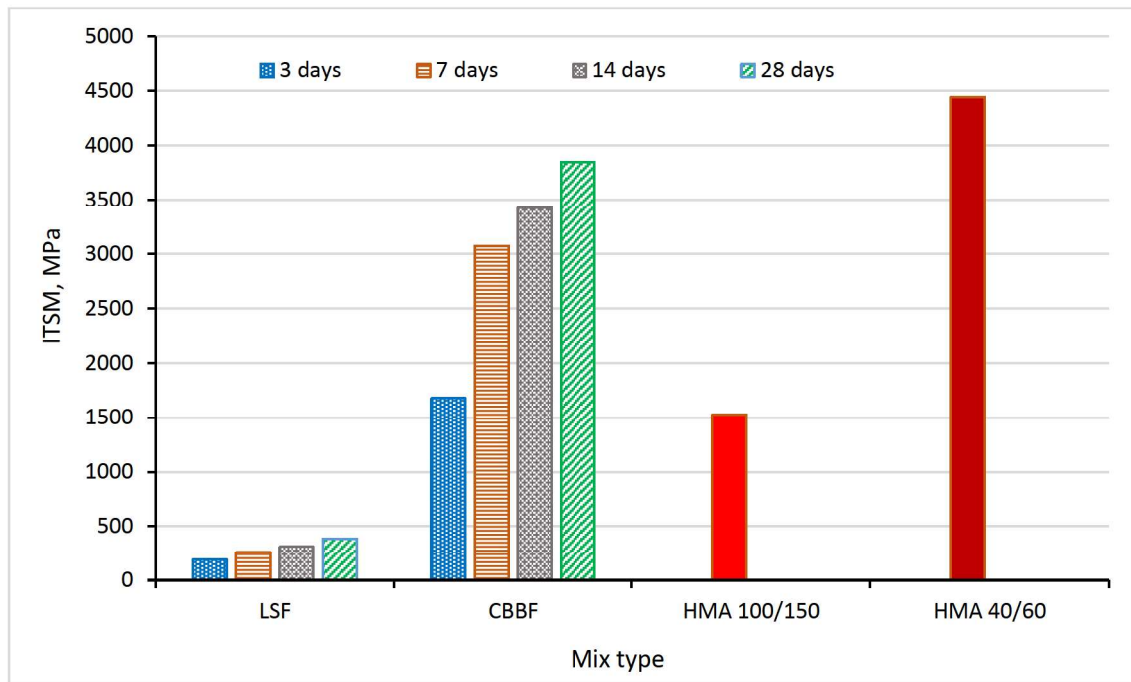


Figure 5. Influence of curing time on ITSM

4.2. Air voids content-ITSM optimization through microwave heating

Air voids content are a vital factor affecting the water sensitivity of asphalt mixes [95]. Volumetric properties were calculated as per the recommendations of the Asphalt Institute [81]. The EBM specimens were heated in a microwave oven before compaction over different times: 0, 1.5, 3, 4.5 and 6 minutes. The temperatures of the specimens were taken immediately after removing the loose mixtures from the microwave, prior to the compaction process. The average temperature was determined after the temperature was recorded 3 times at random. Air voids content for the samples were measured as well as the ITSM, after 3 days of curing to identify the optimum microwave processing time. The optimized ITSM and air voids content are shown in Figure 6, whereas Table 4 gives details of volumetric properties in terms of air voids content and ITSM results.

376 Heating samples in the microwave for 1.5 mins, resulted in a drop in air voids content from
377 8.92% to 7.12%, the ITSM of the EBM mix, reaching its peak value at that same radiation time.
378 There was an increment in the stiffness modulus due to the enhancement in the consistency of
379 the base bitumen, this achieved by decreasing its viscosity. The workability of the mixtures has
380 been improved, leading to wider coverage and better bonding between mixture constituents.
381 Another reason for the improvement in ITSM at early ages is suggested as due to microwave
382 heating producing more hydration products through the hydration process. This result is in
383 agreement with the results of Kong et al. [98] who indicated that the compressive strength of
384 cement mortar at initial ages could be improved if heated in a microwave.

385 It is worth mentioning that the air voids content of the untreated LSF mix decreased from 10.12%
386 to 8.92% when replacing the LSF with CBBF. Pore size, and their continuity, is a vital factor
387 regarding the evaluation of the mechanical and durability properties of the mix. Noticeably, the
388 progression of hydration products due to the existence of CBBF, is facilitated by blocking or
389 minimizing pore size and/or their continuity. This prevents the free movement of attached
390 water and air, as confirmed by both the air void content results mentioned above, and as seen
391 in Figure 11 which will be discussed later. In consequence, blocking also produces a solid
392 structure, which possesses higher strength and durability. In other words, both mechanisms
393 (i.e., the prevention of movement of water and air, and a solid structure) positively alter strength
394 and durability enhancement. Nassar et al. [99] reported that the use of OPC and fly ash in cold
395 asphalt emulsion mixes can improve its volumetric properties due to the presence of hydration
396 products, for example Ettringite, in the in the capillary voids of such mixes.

397 A rise in microwave heating time of up to 3 minutes, softened the specimens, resulting in a
398 noticeable decrease in ITSM. This can be explained by the loss of trapped water in the EBM
399 samples. Trapped water is essential for the hydration process so when the temperature of the
400 mix rises with the increase in microwave time, faster CBBF hydration occurs. Emulsion

breaking can be accelerated in a higher temperature environment. Under such low asphalt viscosity, the CBBF particles can be wrapped by an impermeable membrane which prevents hydration. Consequently, microwave heating offers both positive and negative impacts on the ITSM, these results consistent with those of Wang et al. [32]. Air voids content in the samples are reduced due to the decrease in base bitumen viscosity, while workability is enhanced due to the increase in the mix temperature and the loss of trapped water.

Increasing microwave time to 4.5 minutes, slightly improved the ITSM. The reduction in air voids content also continued, this generated from the evaporation of trapped water (compaction preventor). Improvements in workability were also seen due to the increase in the temperature of the mix and reduction in base asphalt viscosity. Microwave heating for 6 minutes generated a further increment in the ITSM because of extra mixture densification, this leading to an improvement in the interlock of the mix particles. There was also a greater reduction in the viscosity of the bitumen, resulting in extra asphalt coverage. This produced an improvement in the bond reaction between the aggregate particles and bitumen film. The decline in air voids content is from 8.92% to 5.24%. With an increase in microwave heating time from 0 to 6 minutes, a gradual escalation in the mix temperature was created, rising from 20 °C to 101 °C.

Looking at Figure 6 and Table 4, the mix receiving microwave conditioning for 1.5 minutes at 43 °C, represents a new half warm asphalt mixture (H-WM) which has the following characteristics: i) an ITSM of 1794 MPa which performs better than HMA 100/150, ii) air voids content sharply reduced from 8.92% to 7.12%, and iii) a low heating compaction temperature of 43 °C because of the microwave heating. This definition of a half warm mix is adopted based on the fact that EBM's are generated and laid at temperatures between 0 and 40 °C [1, 2]. This means that H-WM lowers the production temperature by about 50-130 °C in comparison to HMA [2]. As such, H-WM can be manufactured and spread at lower temperatures, in comparison to traditional HMA.

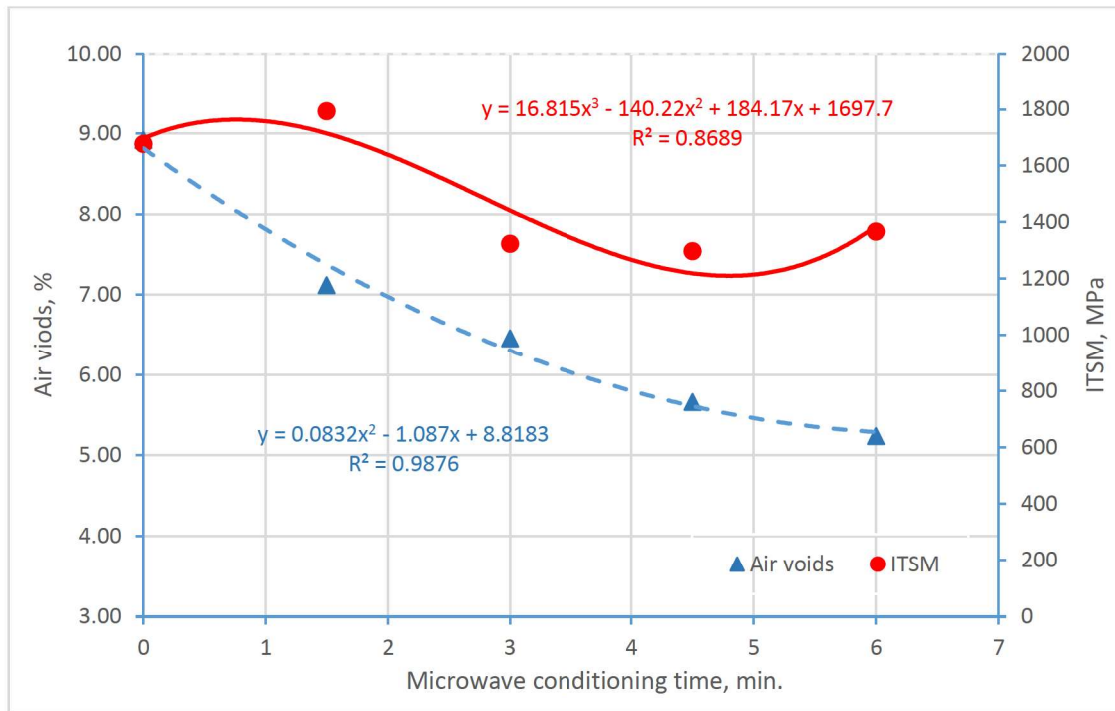


Figure 6. Air voids contents and ITSM optimization for microwave treated mixtures

Table 4. Summary of air voids content and ITSM results of the microwave mixtures

| Microwave irradiation time, min. | Air voids content, % | Temperature, °C | ITSM after 3 days, MPa |
|----------------------------------|----------------------|-----------------|------------------------|
| 0 | 8.92 | 20 | 1678 |
| 1.5 | 7.12 | 43 | 1794 |
| 3 | 6.46 | 65 | 1426 |
| 4.5 | 5.66 | 87 | 1298 |
| 6 | 5.24 | 101 | 1367 |

The ITSM test results over various curing times (3, 7, 14 and 28 days) when using microwave heating, are shown in Figure 7. This figure reveals that the new H-WM offers about a 20% improvement in ITSM compared to HMA 100/150, after only 3 days. A microwave heating temperature of 43 °C, has caused a reduction in the bitumen viscosity, enhancing both the workability of the mix and the mixture densification. The results also indicate that the ITSM of the new H-WM increases in the first three days. After three days, the ITSM rate of increment starts to reduce, this a similar trend as recognised by Kong et al. [98] who clarified this as a

437 result of the enhancement of the hydration process at an early age, this facilitating and
438 accelerating the production of fine and dense C-S-H gels. However, this process will impede
439 and affect later hydration products. The increase in curing temperature also enhanced the
440 hydration of the cement and bitumen emulsion demulsification, leading to an increase in early-
441 stage strength [100, 101]. Reductions in retraction water to continue the hydration process and
442 extra coverage of base asphalt to CBBF, also contribute to increases in ITSM.

443 Processing the emulsion with microwave irradiation causes molecules to lineup in the direction
444 of the radiation. Because of the continuous rotation of the microwave base and sample, there
445 is also rotation of the molecules. This process results in ion conduction in the dispersed aqueous
446 phase (i.e., water) without variations in molecular structure, alongside a rise in temperature due
447 to inter-molecular friction, resulting in a decreased oil phase viscosity (asphalt). The lineup and
448 rotation of water droplets have facilitated contact between asphalt droplets because of
449 disturbances in the charges of the emulsifier. As a consequence, the demulsification and phases
450 separation have occurred faster. In other words, continuous molecular rotation leads to internal
451 heating, neutralization of the internal phase of the zeta potential and weakening of the hydrogen
452 bonds between water molecules and the surfactant [32]. This thermal effect is the main
453 mechanism that microwave processing enhances in emulsion demulsification [102], although
454 charge neutralization changes asphalt molecules back to their base nature, forming a thin
455 bonding film.

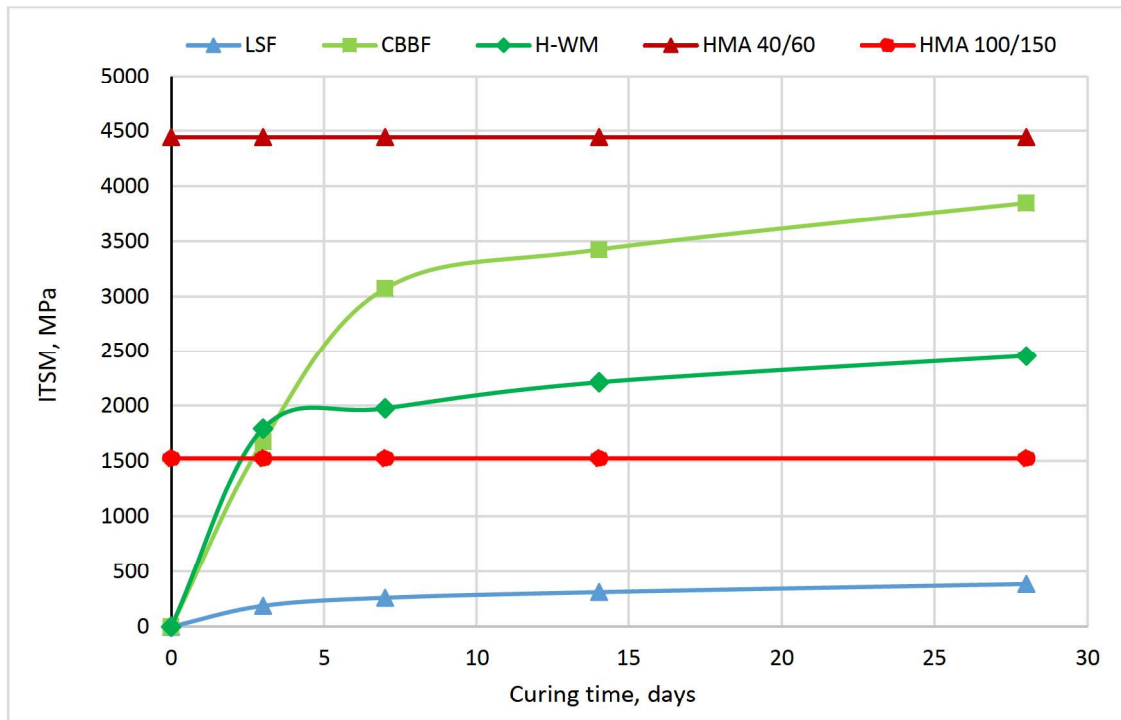


Figure 7. ITSM outcomes for all mixtures

4.3. Wheel track test results

The rutting resistance of the new H-WM was calculated by a wheel-tracking test as per the procedure recommended in BS EN 12697-22 [88]. This test was used to determine the deformation resistance of all mixes at 45 °C. From Figure 8, it can be seen that the permanent deformation resistance of the H-WM microwave mixture was better than the control mixes; LSF and both types of hot mixes. This is because of the positive effect of increasing the particle interlock with stable mastic. The hydration products have 2 functions: binding materials together and growing a network within the asphalt binder that strengthens the mastic. However, the accumulated rutting in the H-WM mix is larger than that of the CBBF, which is a result of the improved area coverage of the bitumen binder. As such, there is a slight increase in irrecoverable strain. Regarding EBM containing CBBF, the strength of the network created by hydration products, withstands loading and behaves like brittle materials with a high elastic modulus.

In essence, these findings confirm the validity of the use of microwave heating to produce an H-WM mixture comparable to HMA. The H-WM mix asphalt reduced rutting depths by approximately 275%, 200% and 24% in comparison to conventional LSF, HMA 100/150 and HMA40/60, respectively. H-WM's permanent deformation behaviour shown in Figure 8, suggests that vertical pressure wheel loading caused the consolidation of the mixtures, so that there is a rapid rise in rutting at the initial stage of the test, a low rutting rate in the second and no phase three failure. This has been confirmed by other researcher findings [15, 103].

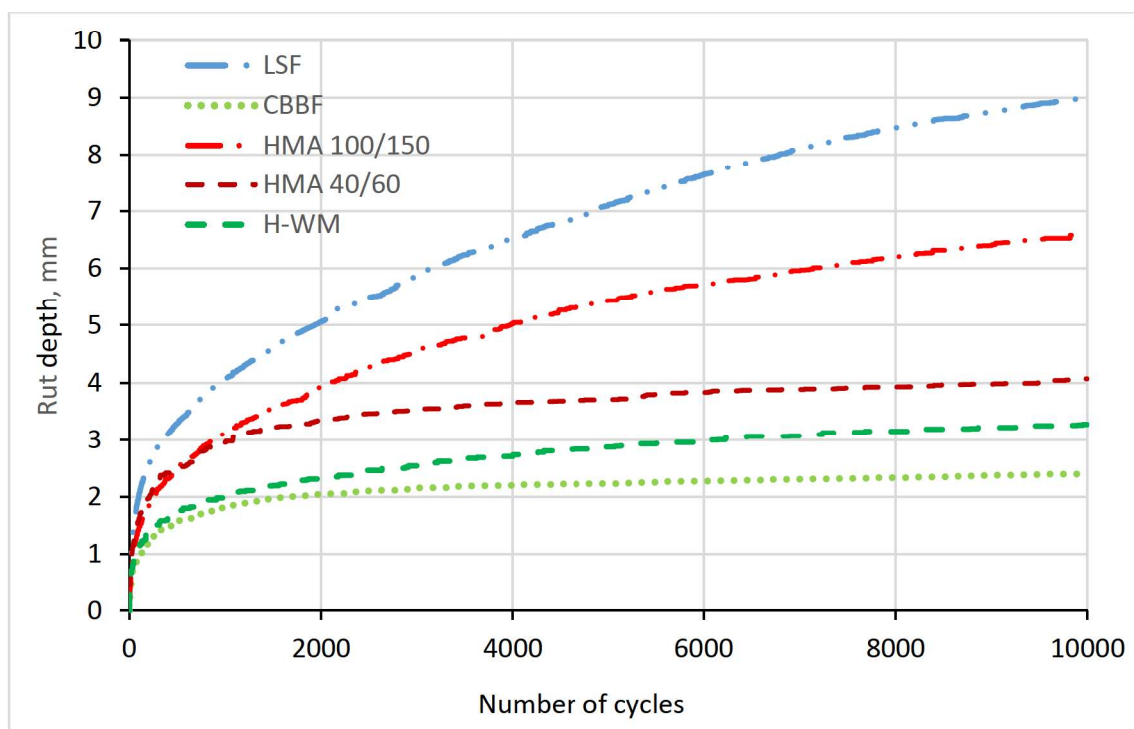


Figure 8. Permanent deformation results

3.4. Fatigue test

Figure 9 shows the fatigue life for both 150 and 200 $\mu\epsilon$ controlled strain criteria for the bituminous mixes. These results illustrate the significant impact of H-WM on fatigue life behaviour compared to the control LFS, CBBF, HMA 100/150 and HMA 40/60 mixes. Based on these outcomes, the use of both microwave heating and the CBBF filler, have a substantial impact on fatigue performance. Both techniques increased the fatigue life of H-WM by approximately 80% and 12% for the samples tested at 150 $\mu\epsilon$, in comparison to that of HMA

100/150 and HMA 40/60, respectively. The increment was around 150% in comparison to the LFS mix. Similarly, improvements of approximately 150% and 25% were noted in comparison to HMA 100/150 and HMA 40/60 for samples tested at 200 $\mu\epsilon$, respectively. A 500% improvement was recorded in fatigue life when compared to that of EBM comprising LSF. These enhancements can be attributed to the air voids content reduction due to microwave heating which increased the consistency of the asphalt, enhancing the interlock between mix ingredients. Thicker asphalt film can be formed, which further enhances aggregate particle bonding.

There was a reduction in fatigue life of around 40% and 45% when compared to EBM comprising CBBF for the 150 $\mu\epsilon$ and 200 $\mu\epsilon$, respectively. This difference might be due to the stiffness of such mixes, something that depends on the strength of the secondary binder, i.e. the hydration products. The CBBF mix had a higher ITSM in comparison to the H-WM mix at later ages. Consequently, the CBBF mix could be relatively more brittle due to this higher stiffness modulus.

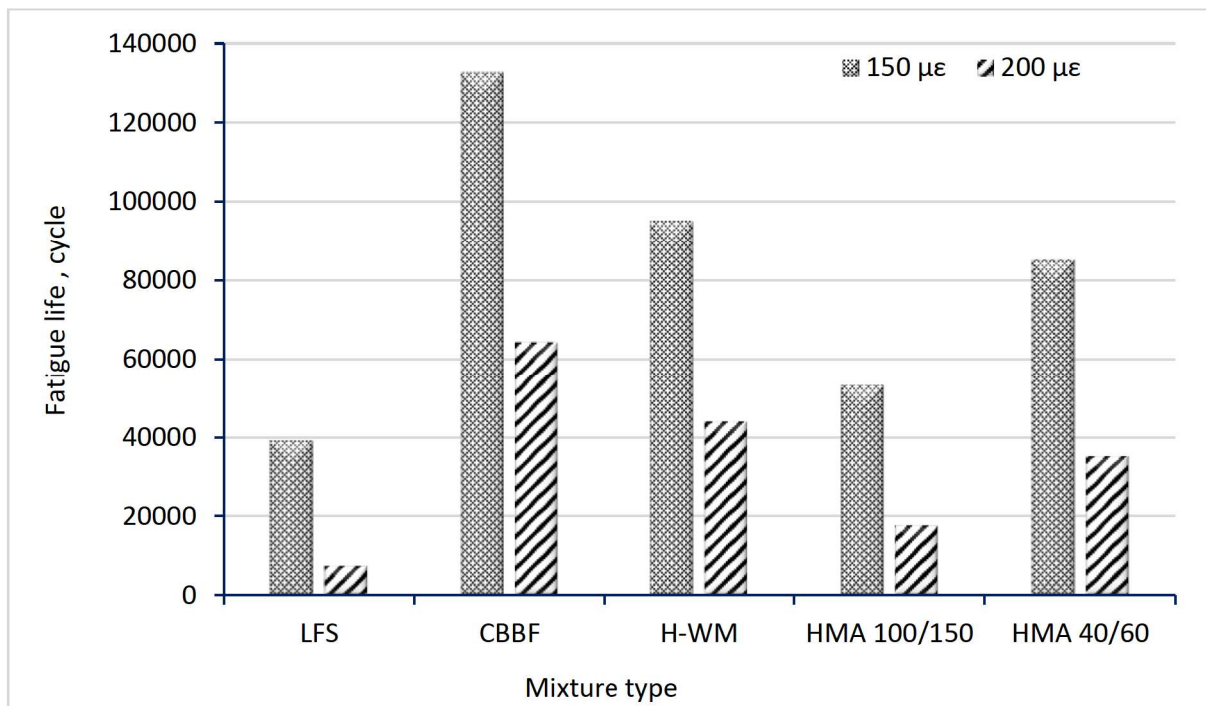


Figure 9. Fatigue results

4.6. Moisture susceptibility test result

The durability of the H-WM mix needs to be investigated to assess the effect of water damage on its mechanical properties. Pavement distresses in the field have been reported due to the impact of water susceptibility [104]. The results of the water damage tests for H-WM compared with the control mixtures, are shown in Figure 10. The SMR for H-WM is superior to that of HMA 100/150 and HMA 40/60 mixtures, this indicating an improvement in resistance to water damage. The improved asphalt coverage area, asphalt cohesion, and reduce air voids content due to microwave possessing, are the primary factors enhancing water sensitivity.

In the case of the CBBF mix, CBBF hydration products provide the adhesion required between aggregates. Fine aggregates, hydration products and emulsion, fill the voids between coarse aggregates. Regarding the CBBF mix, when there was no application of microwave radiation, external water could not penetrate the mix. This can be explained by the fact that most of the voids that exist among the aggregates, are filled with hydration products that increase the bond among the aggregates leading to improve water damage resistance. These results are consistent with Lyu et al. [105] who stated that cement hydration products enhance the water stability of cold recycled asphalt emulsion mixtures. The increase in temperature of H-WM after microwave irradiation, can promote the hydration process (at early ages) and asphalt film. This result is in agreement with the findings of Wang et al. [32]. However, coating cementitious particles with asphalt film limits the hydration process, even in the presence of water, during SMR testing. Therefore, the SMR of H-WM is inferior to that of CBBF.

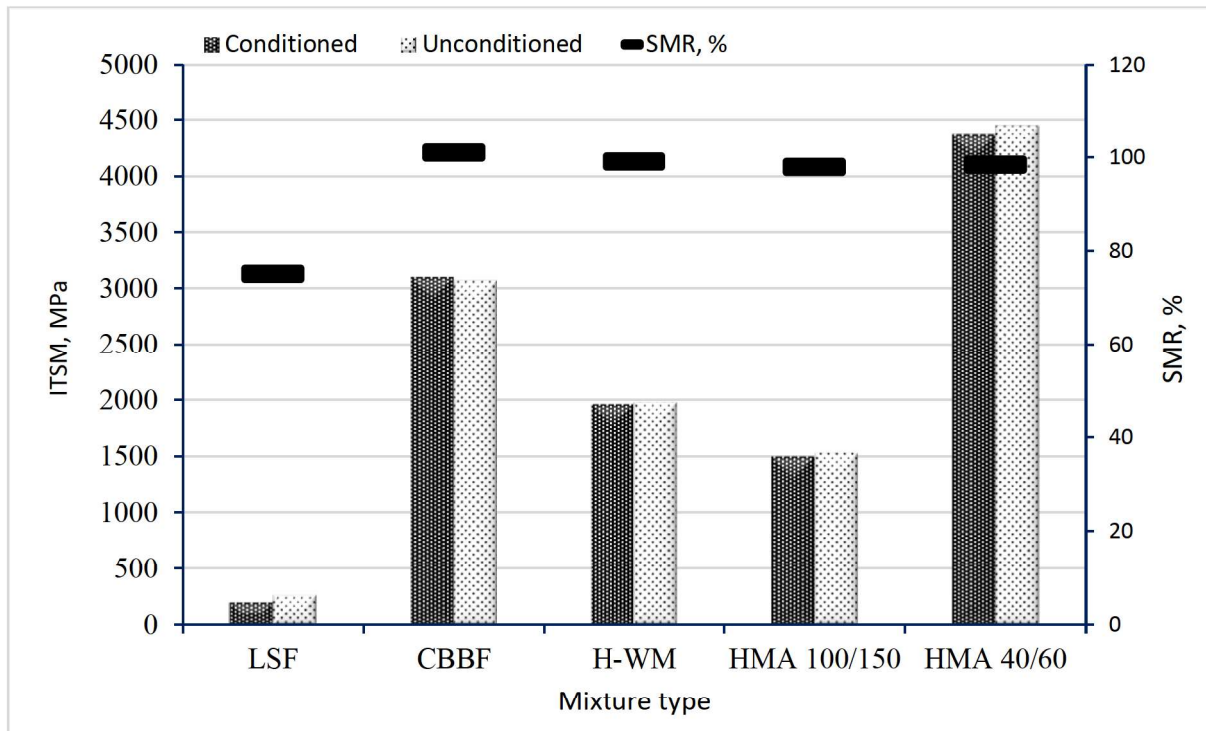


Figure 10. Water sensitivity results

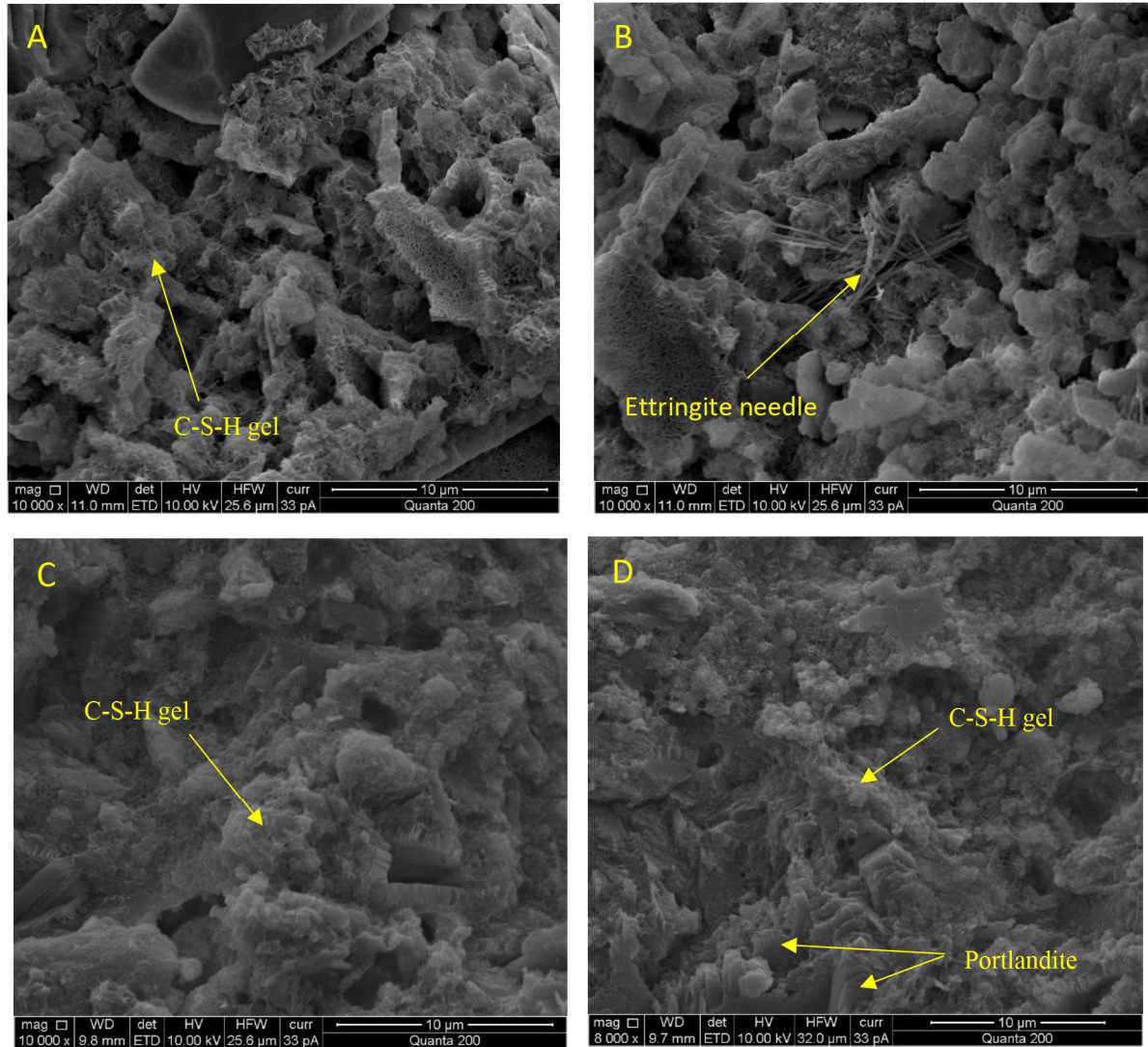
4.7. Microstructure analysis

Microstructure analysis helps to understand the role of the filler in CBBF and H-WM. Figure 11 details observations of the hydration products on the surface of the paste samples at 3 and 28 days, for normal paste and microwaved samples. At 3 days, the fillers have grown and separated inside the sample and the surface is rough. Such noticeable hydration products are present even after microwave irradiation, as seen in Figures 11 A and C. Hydration products can fill the voids left because of water evaporation or consumption by the hydration process. Consequently, more solids and a sound morphology are created, resulting in enhanced binder strength. The rough products from the CBBF particle surface seem to play a vital role in reinforcing the asphalt membrane from an early-stage, facilitating gains in strength.

The strength gained by the mechanical properties in the early-stages is due to acceleration of the hydration process by microwave radiation, as can be seen when comparing Figure 11 A with B. The crisscross morphology facilitates a solid, dense and uniform structure, this

enhancing the mechanical and durability performance of the H-WM. This result is in agreement with the findings of Lin et al. [106].

The proportion of hydration products reflects CBBF performance during the hydration process. They are a rigid material influencing the whole mix stiffness and reducing the temperature sensitivity of EBM [107]. Distributing hydration products in an asphalt binder enhances adhesion between the binder and aggregates, as double binding is generated, primarily by the asphalt, secondly by CBBF hydration products. Therefore, the rutting resistance and moisture damage resistance of the EBM is improved by the CBBF, as confirmed by Figure 11 D, there being more solids and fewer voids compared with Figure 11 C. The C-S-H phase is represented by gel structure and the Portlandite (C-H) crystals appear in many various shapes and sizes, starting from massive, platy crystals with distinctive hexagonal prism morphology or large thin elongated crystals (as shown in Figure 11 D), based on the study by Sarkar et al. [108]. In summary, there is an increase in density of hydration products in the early stage due to microwave processing, but at a later age (28 days), there are fewer products present than in the microwaved sample. Consequently, both the mechanical and durability properties are strongly related to these types and densities of the hydration products.



* Ettringite (needle shape); C-S-H (gel-like shape); Portlandite (platy crystals)

Figure 11. Morphology details of microstructures: A) microwaved CBBF at 3 days, B) CBBF at 3 days, C) microwaved CBBF at 28 days, D) CBBF at 28 days.

5. Conclusions

In this study, a novel, half-warm asphalt mix has been developed using microwave heating. This mixture will overcome the problems associated with the use of hot mix asphalts including greenhouse gas emissions. The conclusions based on the research findings can be summarised as follows:

1. The substitution of traditional limestone filler with a combination of by-product filler (including GGBS and CCR), considerably improved the stiffness modulus. The new cementitious binary blended filler helps to generate secondary bonding inside the mix in addition to that initiated by the bitumen emulsion residue, a primary binder.
2. Microwave heating was used to develop a novel H-WM mixture that offers a stiffness modulus approximately ten times greater after 3 days, than a mix with traditional limestone filler. Improvements in the novel mix were confirmed by the improved ITSM in comparison to the conventional HMA 100/150.
3. Microwave heating is approved as a suitable technique to reduce air voids content in emulsion-based mixtures. 1.5 minutes of optimum microwave conditioning time, depending on the air voids content, ITSM and the pre-compaction temperature of the mix, is an adequate application time.
4. The longer the microwave heating time, the better reduction in air voids content. Nevertheless, excessive microwave heating time results in a lower ITSM. The temperature at which CBBF achieves its peak ITSM is 43 °C.
5. The air voids content in the H-WM mixture decreased from 8.92 % to 7.12%. The workability and consistency of the bitumen were enhanced due to the reduction in primary binder viscosity as a result of temperature rise. Furthermore, the bonds between mixture constituents were also improved because the hydration process was activated at an early age using microwave heating.
6. Microwave radiation enhances resistance to permanent deformation at a higher temperature for H-WM in comparison to the corresponding control limestone mixture and the two reference hot asphalt mixtures. This was because of the resultant binding properties created by the hydration products, in addition to a reduction in air voids content leading to an increase in particle interlock.

7. The resistance to water damage in the novel H-WM is better than that of LSF and both HMA mixes.

8. Although the fatigue life of H-WM after microwave heating is enhanced, it is still less than that of the CBBF mix due to the increased brittleness of hardened hydration products in this mix.

This research is laboratory scale work meaning that it is recommended to work on a construction site to identify on site challenges. A portable industrial microwave can be suggested as the main processing tool to produce the H-WM in situ, similar to the microwave used to de-ice pavements.

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