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Article

Improving the Mechanical Properties and Durability of Cold Bitumen Emulsion Mixtures Using Waste Products and Microwave Heating Energy

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Abstract: Scientists have effectively demonstrated that the introduction of a waste product comprising cementitious chemical compositions can enhance the mechanical properties and durability of cold bitumen emulsion mixes (CBEMs). On the other hand, the high air void content of the CBEM mix remains a challenge that is considered unsatisfactory by paving engineers. As a result, this investigation highlights two major changes that were made. The first is the use of waste paper sludge ash (PSA) as a filler in CBEM instead of the conventional mineral filler (CMF). The second change was made to further improve the mixture by reducing the amount of CBEM air voids using microwave (MW) heating energy as a post-treatment method. When compared to typical hot mix asphalt (HMA), the new CBEMs showed great mechanical properties and durability. Moreover, the proposed method, using CBEMs, has lower environmental risks, is safer, and is more cost-effective than existing paving mix technologies. This study presents a method for controlling air voids within pavement specifications without affecting mechanical behaviour or generating additional environmental or economic considerations. When compared to typical mixtures, laboratory test results showed that MW-heating can enhance both the stiffness modulus and the air void content. Furthermore, these results revealed a minor reduction in creep stiffness and water sensitivity. Nevertheless, in terms of mechanical, volumetric, and economic properties, the suggested post-mix treatment was comparable to HMA. The findings point to the need to adopt CBEM post-heating approaches, particularly the MW treatment procedure.

Keywords: cold bituminous emulsion mixtures; creep stiffness; paper sludge ash; MW-heating



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1. Introduction

In recent years, Europe's annual output of hot asphalt mixture (HMA) has been about 309 million tons [1]. It has been reported that 21 kg of CO₂ is generated for each 1 ton production of HMA [2]; in other words, the asphalt manufacturing sector in Europe emits about 6.5 million tons of CO₂ each year. In comparison to HMA, cold bituminous emulsion mixtures (CBEMs) emit considerably less CO₂ during the manufacturing process, producing just 3 kg of CO₂ per metric ton [3]. This means that if HMA is replaced with CBEM, carbon emissions in the asphalt production sector may be decreased by more than 75%. Additionally, and especially in light of the ongoing increase in energy costs, the usage of CBEMs has some energy efficiency benefits over HMA; i.e., one ton of CBEM requires about 13% of the energy required to produce the same amount of HMA [2]. CBEMs also offer several additional advantages over the HMA mixes such as the reduction in

dust and gaseous emissions during the heating of mixtures, the ability to be prepared in distant locations, the use of virgin and recycled aggregates, and improved safety during preparation [4–6]. As a result, appropriate CBEMs are in high demand as a viable alternative to HMA.

Suitable CBEMs can be introduced as a highly promising and effective solution instead of HMA. Unfortunately, surface treatment and applying bond coats are the only applications of such technology today [7,8]. Consequently, CBEMs that are suitable alternatives to HMA are in great demand. Until now, this method has been restricted to bond coat application and surface treatment [9,10].

Many studies have focused on improving the engineering characteristics of CBEMs. They have experimented with a variety of additives and methods to accomplish this purpose. As additives, polymers [11,12], fibres [13–15], fly ash [16,17], fluid catalytic cracking catalyst [18], ground granulated blast-furnace slag [19] and ordinary Portland cement [20–23] have been utilized to enhance the mixture's performance, and several assistance techniques were utilized to prepare CBEM such as double mixing, initial coating with asphalt and two mixing phases [24]. Nevertheless, the majority of these efforts were unsatisfactory and reflected either negative financial or environmental consequences.

To make use of waste or by-product resources, many researchers have recently investigated the possibility of substituting a certain amount of such waste materials with virgin materials to achieve greater sustainability [25–27]. Although most of these research works improved the strength characteristics of CBEMs, obtaining an acceptable strength in a short curing period was still an issue (i.e., fewer than 7 days). Furthermore, compacted mixes were still suffering from an excessive level of air void content according to the standards.

Previous researchers had conducted a feasibility study and reported that a novel CBEM, paper sludge ash (PSA), could be utilized in substitution of the traditional filler material [28]. Such material can significantly improve the mechanical and durability characteristics of CBEMs. It was claimed that PSA's hydraulic properties contribute significantly to the generation of a new adhesive component in addition to that of the bituminous binder. In addition, the hydration process generated by the existence of PSA in the mix consumes the trapped moisture between the bituminous binder and the aggregate and simultaneously strengthens the asphalt binder. Nevertheless, the detrimental features have been eliminated entirely, as the new CBEMs demonstrated comparable performance to conventional HMA within a shorter curing time. However, the high content of air voids was determined to be outside of engineering limitations. As a result, a new procedure should be adopted to overcome the mixture air voids issue. It is well known that the heating of an asphalt mixture promotes the backing of the mix components by reducing bitumen viscosity; accordingly, the compacted mix will include a few air voids [29]. It was suggested in this study that MW-heating technology could be a viable and long-term solution for the reduction of the mixture's air voids.

MW technology has been shown to be an effective alternative in heating materials over the past 70 years. Industrial applications of such technology have grown extensively since the 1980s, particularly in the food, rubber sectors, textile and paper. The MW-heating process is dependent on the polarization of the molecules of the heated materials' dielectric [30]; this process is independent of the thermal conductivity of the materials. As a result, the previous heating method offers certain benefits over traditional heating in terms of energy usage, heating homogeneity, time consumed and the material's volumetric properties.

Finally, the development and enhancement of asphalt mixtures to associate with the global context of sustainable materials production at low cost are highlighted in this research work as a main aim. Using waste materials with the capability of microwave processing (a low power technique) to improve asphalt mixtures is the main hypothesis to sustain the aim. However, many limitations need to be mentioned in this research work such as the fact that the study was conducted using a non-industrial scale microwave, the lab process could be different on field application and specific material is used (different

materials have different responses to microwave processing), etc. These points and more need to be covered in detail for more understanding of the benefits of the microwave processing technique. Nevertheless, this attempt is a part of the required efforts to disclose the potential of the microwave technique. Therefore, the following sections aim to disclose the experimental programme undertaken to achieve such an aim.

2. Experimental Programme

2.1. Materials Characteristics

In this laboratory scale research work, crushed green granite was used as coarse aggregate, whereas the fine aggregate used was virgin sand. The physical characteristics of both coarse and fine aggregates were determined following British Standards for bituminous mixes used in roads and airfields BS EN 13043 [31]. The aggregates were rated as 0/10 mm close-graded for surface course application [32], Table 1. The bulk specific gravity, apparent specific gravity and water absorption for the coarse aggregate were 2.79 g/cm³, 2.82 g/cm³ and 0.4%, respectively. These values were 2.74 g/cm³, 2.77 g/cm³ and 0.4% for the fine aggregate, respectively.

Table 1. Aggregate grading for 0/10 mm size close-graded surface course (BS EN 13108-1).

Test Sieve Aperture Size mm	% by Mass Passing Mid	% by Mass Passing Specification Range
14	100	100
10	97.5	95–100
6.3	65	55–75
2	28	19–37
1	20	10–30
0.063	5.5	3–8

The penetration (25 °C), softening point (°C), kinematic viscosity (at 135 °C), and density (at 25 °C) for the bituminous binder 100–150 were 143, 43.6, 175 and 1.00, respectively. The values were 43, 52.4, 325 and 1.01 for the bituminous binder 40–60, respectively.

Cationic slow-setting bitumen emulsion was used in CBEM to provide excellent adhesion between aggregate particles [3]; the emulsion properties are according to the requirement of BS EN 13808 [33]. The boiling point (°C), relative density (at 15 °C g/mL), and residue by distillation (%) were 100, 1.05 and 56, respectively. Whereas, 53 pen and 143 pen grades of the bituminous binder were used for hard and soft HMA, respectively; the binders' properties are according to the requirements of BS EN 12591 [34]. Conventional mineral filler in addition to the PSA was utilized as filler in this study. Limestone filler is widely used as a commercial filler in both HMA and CMA. PSA is generated from power plants that burn waste at temperatures between 850 °C and 1100 °C by the use of a fluidized bed combustion technology. It is a promising sustainable material to use as a filler in bituminous mixes because it has a chemical structure similar to ordinary Portland cement (OPC), consisting of silica, calcium oxide, and alumina [35,36]. Table 2 demonstrates the chemical composition of the used fillers.

Table 2. Chemical composition of fillers.

Element	Concentration	
	Mineral Filler	PSA
Al ₂ O ₃	9.221	3.471
CaO	5.58	60.93
SiO ₂	53.597	28.178
Fe ₂ O ₃	7.368	0.202
MgO	4.984	3.554
K ₂ O	3.123	0.354
TiO ₂	0.831	0.556

2.2. Mix Design and Sample Preparations

Universally and to date, there is no unified design method for CBEMs. Several previous researchers proposed mix design procedures for CBEMs to achieve their local requirements, which were mainly derived from the suggested mix design producer of the MS-14 manual. Therefore, all CBEM specimens in this study were prepared according to the Asphalt Institute (MS-14) method [37] with some modifications. The modifications were that the indirect tensile strength test (ITS) was utilized instead of the Marshall stability test to determine the optimum emulsion content. Further, a density test was followed to decide the optimum liquid contained within a mixture. It was reported that poor initial moisture of aggregates promotes balling of the bituminous binder with the fine aggregate particles, resulting in an undesirable coating degree [24]. According to the characteristics of the aggregates, the optimum prewetting water content (PWwc), optimal emulsion content (OEC), and optimal total liquid content (OTLC) values were 4%, 11.5%, and 14.5% of the aggregates' weight, respectively. After determining the optimum mixing values, different laboratory trial mixes were prepared in which the virgin mineral filler (VMF) was replaced with four percentages of PSA, ranging from 0% to 5.5% by weight of total aggregate. In addition, conventional hot mix asphalt (HMA) specimens with two different binder grade types were prepared to utilize the same CBEM aggregate gradation. It is worth mentioning that each specific mix required three specimens with 1100 g, for both HMA and CBEMs. Figure 1 shows the microwave process.



Figure 1. Microwave process.

2.3. The Adopted Methodology for MW-Treated Mixes

After following the MS-14 procedure of mixing the proportions, the CBEM samples were exposed to two different heating methods, namely, traditional and MW-heating. The latter heating method was performed utilizing a home-type MW device with a frequency capacity of 2.45 GHz. Three different power levels were performed; as detailed in Table 3. In addition, four durations were adopted for the mixes post-mix microwaving; they are, 2.5, 5, 7.5, and 10 min, in addition to a control sample that was made without MW-heating for comparison. The traditional heated specimens were exposed to temperatures the same as the MW-heated specimens across a range of time periods. The temperature of the specimens after microwaving for 2.5, 5, 7.5, and 10 min was recorded to be 76 °C, 82 °C, 90 °C, and 101 °C, respectively.

Table 3. Microwave power levels.

Level	Category	Actual Power within 30 s (KW)	Power Setting %	Actual Power on Time (s)
1	low	5.784	20.00	6
2	medium	14.124	53.33	16
3	high	25.800	100.00	30

The duty-cycle control approach is normally used to regulate the output power of the MW device. The MW magnetron device, which is responsible for generating high

electromagnetic waves, was operated at full power and fragmented periods; that is, during a certain cycle, the magnetron is switched on for a predetermined amount of time, followed by an off interval; this on/off cycle is repeated within the allotted heating time. Typically, full power signifies that the magnetron operates continuously, whereas lesser levels have set on/off periods within the cycles. Three separate power levels were employed to explore the influence of varying power levels at different topic times; the power levels utilized are shown in Table 3. The MW duty cycle utilized in this research was 30 s, and the total magnetron output was 860 watts, as recorded by the power meter. The power level is limited by the ratio of on time to total cycle time. The effective time of heating will be the accumulation of the on times.

2.4. Samples Conditioning

In general, curing temperature and curing time are two major factors that influence the mechanical strength of CBEMs. Thus, according to the previous research suggestions and recommendations, particular curing methods were adopted to characterize the mechanical characteristics of various CBEMs. Jerkins' [38] procedure was used for ITSM test specimens, while Thanaya's [24] procedure was used for UCCT test specimens. Additionally, a methodology for curing durability test specimens was developed following BS EN 12697-12 [39]. The three mentioned procedures are detailed in Table 4.

Table 4. CBEMs curing protocol.

Test	First Stage Curing	Second Stage Curing	Time Testing (Days)	Recommended by
Indirect tensile Stiffness modulus	20 °C for 1 day	40 °C for 1 day	2, 7, 14, 28, 90, 180 and 360 days	Jenkins [38]
Uniaxial Compression cyclic	20 °C for 1 day	40 °C for 14 days	15	Thanaya [24]
Stiffness modulus Ratio (durability test)	20 °C for 1 day	20 °C for 9 days	10	BS EN 12697-12 [39]
	20 °C for 1 day	20 °C for 6 days, then soaked 3 days at 40 °C		

2.5. Methods

This research work covers two mechanical fundamental tests, in addition to one durability test. It is worth mentioning that three replicates were performed in each test. The tests are detailed as follows:

2.5.1. Indirect Tensile Stiffness Modulus (ITSM)

The test was done utilizing the Cooper Research Technology HYD 25 testing apparatus per BS EN 12697-26 [40]. Table 5 clarifies the adopted testing conditions.

Table 5. ITSM Test Conditions.

Item	Range
Specimen diameter(mm)	100 ± 3
Rise time	124 ± 4 ms
Loading time	3–300 s
Transient peak horizontal deformation	5 µm
No. of test plus	5
No. of conditioning plus	10
Poisson's ratio	0.35
Test temperature (°C)	20 ± 0.5
Compaction	Marshall 50 × 2
Specimen temp. conditioning	4hr before testing
Specimen thickness mm	63 ± 3

2.5.2. The Uniaxial Compressive Cyclic Test (UCCT)

This test was performed per BS EN 12697-25 [41], using Cooper Research Technology HYD 25 testing apparatus. The test conditions are shown in Table 6.

Table 6. UCCT Conditions.

Item	Range
Loads	100 ± 2 KPa
Frequency	0.5 Hz
Rest period	1 ± 0.05 s
Loading pulse	1 ± 0.05 s
Poisson's ratio	0.35 for 20 °C
Pre loading	10 KPa for 10 min
Test temperature (°C)	40 ± 0.5
No. of test plus	3600
Specimen thickness	60 ± 2 mm
Specimen diameter	148 ± 5

Both of the previously mentioned tests are introduced to evaluate the mechanical characteristics (elastic modulus and fatigue life of the mixture), respectively.

2.5.3. Durability (Water Sensitivity) Test

The test was conducted per BS EN 12697-12 [39]. The indirect tensile strength has been replaced with the Stiffness Modulus ratio (ITSR) for evaluating the mixture's sensitivity to water damage using the Cooper Research Technology HYD 25 testing device. This test was conducted under comparable conditions to the indirect tensile stiffness modulus test.

3. Results and Discussion

3.1. Effect of Post-Mix Heating Methods

The ITSM for samples heated using traditional and MW post-mix methods is shown in Figure 2. Overall, when traditional post-mix samples were heated, the ITSM reduced in comparison to the unheated sample but increased when the heating temperature was increased. For MW treated samples, a noticeable reduction in the ITSM value was observed after 2.5 min of microwaving, followed by an improvement with higher durations until 7.5 min, where the ITSM value decreased when microwaving exceeded that limit. On the other hand, treated specimens with conventional post-mix heating demonstrated a continuous decrease in air void as heating temperature increased; whereas, MW-treated specimens demonstrated a turning point of increased air void at 7.5 min of microwaving, as shown in Figure 3. The behaviour of these results is in agreement with the findings of Dulaimi, Al-Busaltan and Sadique [29]. Moreover, the trendlines for the ITSM with post-heating confirm the behaviour of the materials under different processing techniques, where the optimum for MW processing is at 7.5 min; whereas, conventional heating needs extra time to reach the optimum.

However, these findings indicate that heating temperatures had a significant influence on calculating the air void and stiffness modulus characteristics of post-mix heating CBEMs. MW energy produces two processes, namely, heating and polarization. The heating of CBEM components increased the rate of bitumen emulsion breaking and caused an increase in free water evaporation, in addition to a reduction in binder viscosity, which reflected a higher coating homogeneity of the aggregate particles with bitumen. However, the polarization of the charges in aggregates and bitumen emulsions improved their adhesion rates. Therefore, although workability is improved due to a rise in mix temperature and the release of trapped water, air voids in the samples are reduced due to a decrease in base bitumen viscosity. MW post-treated CBEM specimens had the greatest stiffness modulus and low air void content. Thus, it can be concluded that this post-mix treatment procedure is more beneficial for improving the mechanical properties of CBEMs. However, some wetting water should be retained to act as a lubricant throughout the compaction process. This amount of water is needed for the hydration process of PSA. As a result, post-mix heating needs to be controlled to provide the best mechanical properties at the lowest air void content. At the same trend of ITSM, the trendlines for the void content with post-heating confirm the behaviour of the materials under different processing techniques,

where the optimum for MW processing is at 7.5 min (or 90 °C), whereas the conventional heating shows continuing reduction in the air voids.

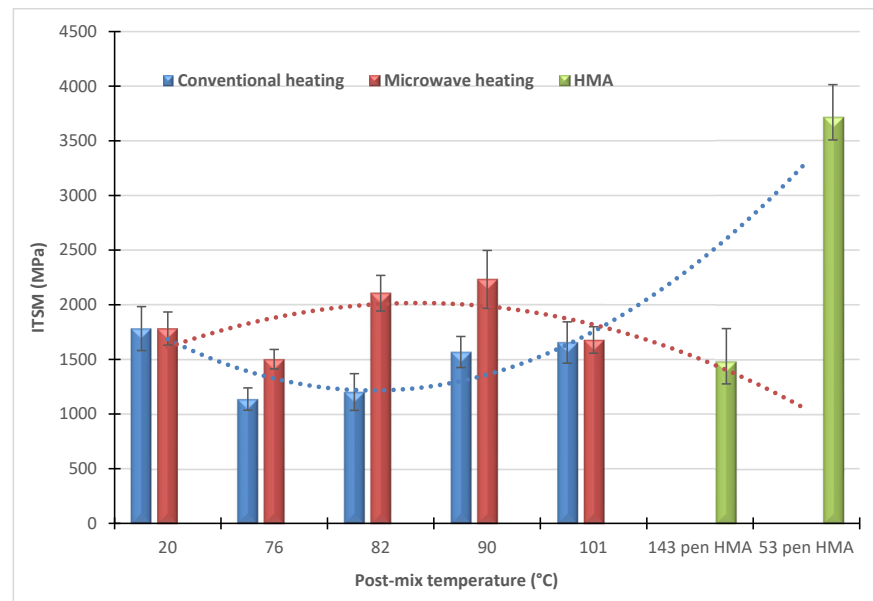


Figure 2. Impact of post-mix time on ITSM of CBEMs containing 5.5% PSA after 2 curing days.

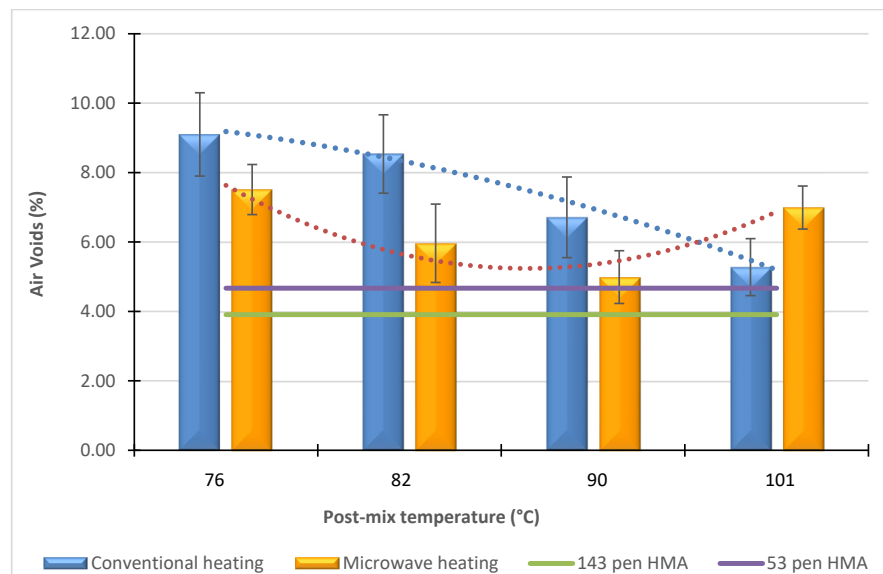


Figure 3. Impact of post-mix time on air void of CBEMs containing 5.5% PSA.

3.2. Effect of MW Power Level on Air Void and Stiffness Modulus

Figures 4 and 5 illustrate the test results for various post-mix MW power levels. The test results revealed that the ITSM values varied significantly owing to variations in power level and treatment duration. There was a general improvement in ITSM value with increasing time of MW treatment for low-power levels; whereas, the best value was noticed at medium- and high-power levels. Furthermore, low-power MW treatment improved the ITSM value more than non-MW-treated CBEM, which is mainly because of the MW's polarisation impact; whereas, treatment at medium- and high-power levels results in the optimal ITSM value because of the MW's polarisation and heating actions. Additionally, a decrease in stiffness was observed at a particular treatment duration and power level; this

decrease is attributed to polarisation action, which causes agglomeration of the bitumen emulsion, and reflected a poor bitumen coating.

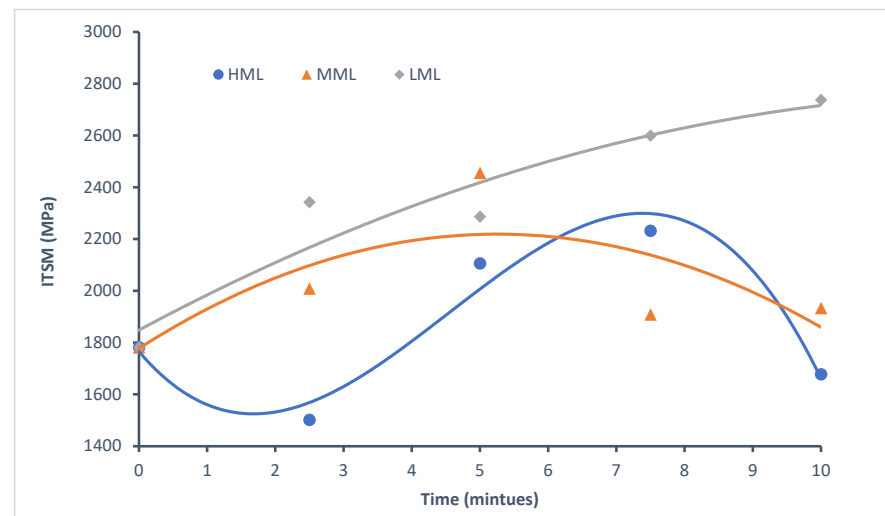


Figure 4. Effects of varying MW energy and time on the ITSM of 5.5% PSA-containing CBEMs. HML: High microwave level; MML: Medium microwave level; LML: Low microwave level.

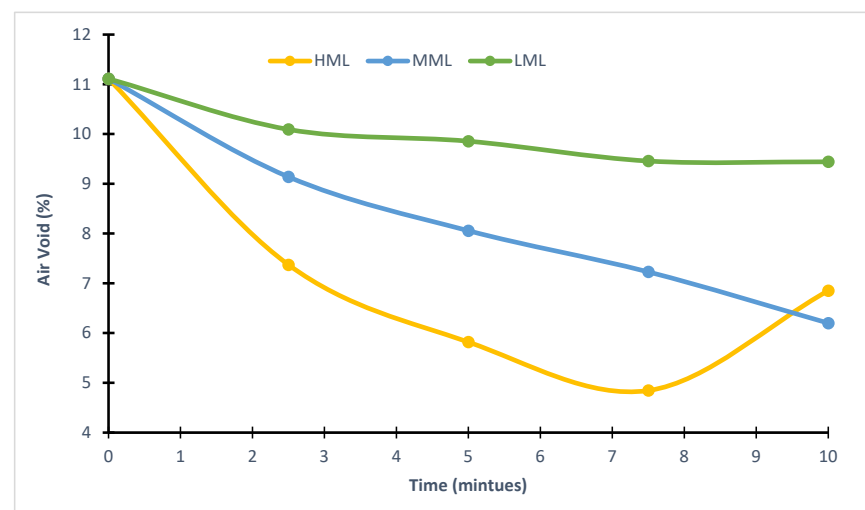


Figure 5. Effects of varying MW power and duration on air voids of CBEMs containing 5.5% PSA.

The resulting air void depicted in Figure 5 showed a continual decrease at low- and medium-power levels of MW-treated specimens, but a noticeable change was observed at high power levels, after which the air void content rose again with increased MW treatment. Additionally, the results clearly demonstrated that the air void reduction rates rise when the power level is raised. However, it is asserted that the dielectric permittivity and heat transfer properties of the MW cause MW-heating to raise aggregate and water temperatures and that this heating subsequently transfers to the bituminous material, reducing its viscosity. As a result, the mixture was more compatible and the final air void was smaller. Additionally, the coating of the aggregate with bituminous material was more effective.

Water is a significant lubricant in CBEM and is also necessary for hydrating the filler. Eliminating a certain amount of water will affect stiffness and air void. Figure 6 demonstrates that when MW treatment time increases, the rate of increase in stiffness modulus reduces. It was revealed that after 90 days, there was significant progress in stiffness: 272%, 223%, 184%, and 166% for MW durations of 2.5, 5, 7.5, and 10 min, respectively. In com-

parison to non-MW-treated specimens, MW-treated specimens consistently demonstrated the lowest values as curing time increased due to the elimination of water needed for the hydration process. Nevertheless, such values are equivalent to both hard and soft HMA at appropriate MW treatment power and time, demonstrating that MW treatment of CBEMs containing hydraulic filler significantly improves the stiffness modulus and reduces the air void.

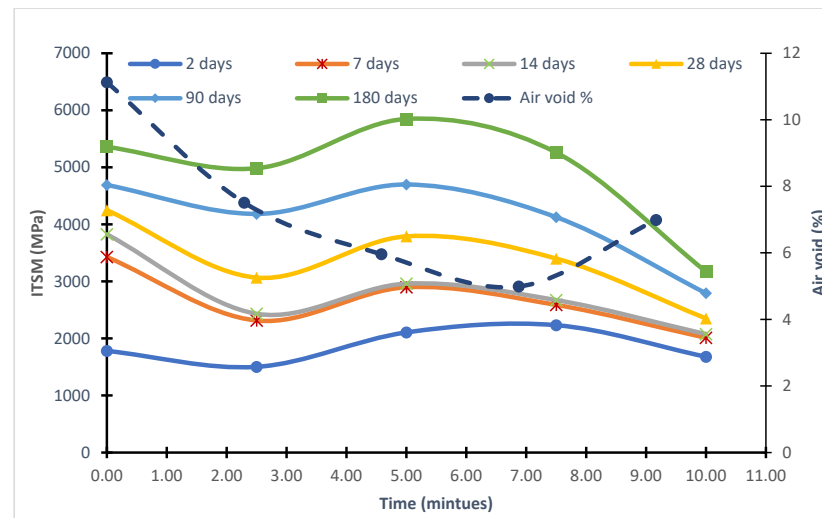


Figure 6. Effect of MW time on ITSM of CBEMs containing 5.5% PSA in terms of various curing times.

Figure 7 illustrates the results of conventional CBEM specimens. At different points throughout the process, full MW post-mixing power was applied. The test results indicate that ITSM improves continuously as MW treatment duration increases; furthermore, the ideal air void content is reached after 7.5 min of heating. Additionally, it should be noted that the ITSM value of 10 min achieved the HMA value after 28 days, which is a remarkable accomplishment considering that untreated traditional CBEMs often need 2–24 months to reach such progress. However, as conventional CBEMs have no hydraulic filler, removing the free water aids is important to the ongoing improvement of ITSM. However, it is thought that a specific amount of water is needed to achieve a low air void content.

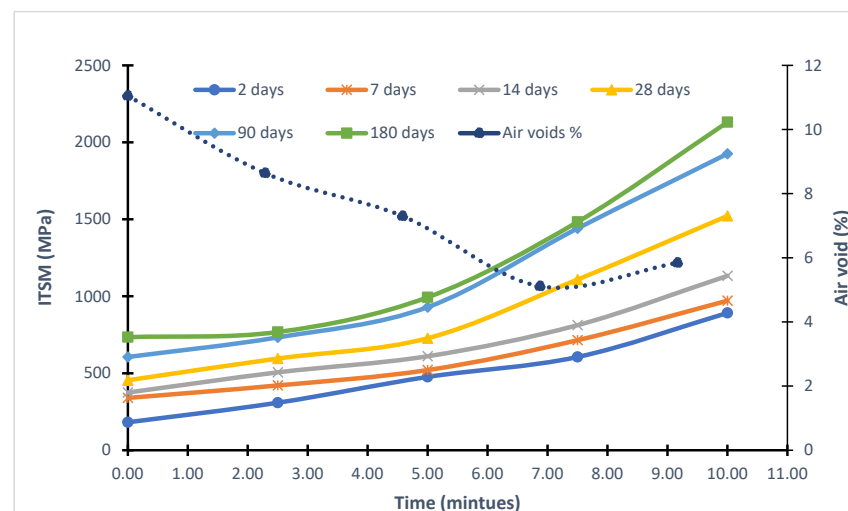


Figure 7. Impact of MW time on ITSM of traditional CBEM.

3.3. Effect of MW Treatment on CBEMs Permanent Deformation

Test results from the uniaxial compressive cyclic test show that CBEMs comprising 5.5% PSA and treated with the MW had a higher resistance to permanent deformation in contrast to soft and hard HMA, as can be seen in Figure 8. Nevertheless, in contrast to un-MW-treated CBEMs, they showed lower resistance to permanent deformation. To better understand permanent deformation resistance characteristics, a comparison of the creep stiffness and creep rate of the said mixtures showed that post-mix MW treatment minimises the creep stiffness of the same CBEM to a fifth of their stiffness and increases the creep rate seven times, as can be seen in Figures 9 and 10. However, that does not mean the MW treatment eliminates the gain in permanent deformation resistance due to incorporating PSA; in fact, it reduces this improvement, whereas the MWd samples still had a superior creep stiffness and creep rate compared to soft and hard HMA. It is suggested that this reduction is due to the removal of the water required to continue PSA hydration, not only due to MW treatment but also due to curing protocol when the sample is cured for 14 days at 40 °C.

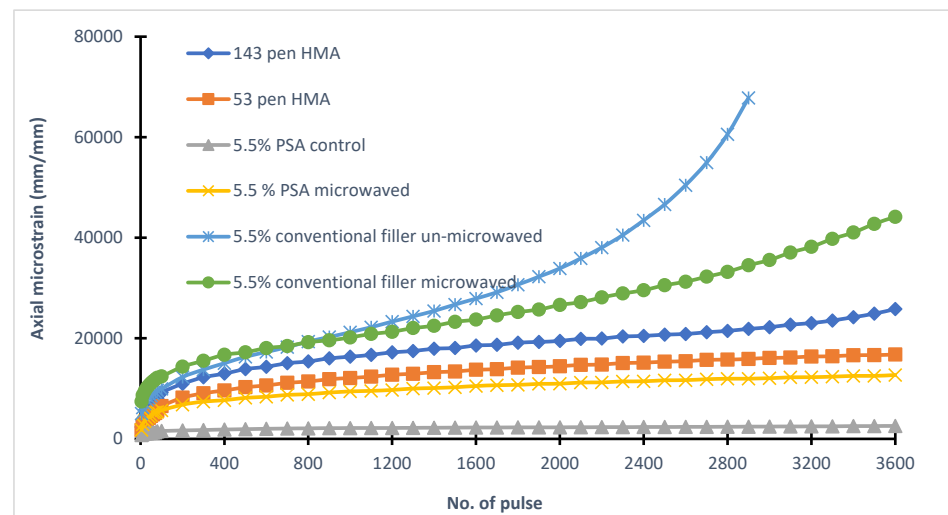


Figure 8. Correlation between accumulated creep strain and pulse count of various CBEMs.

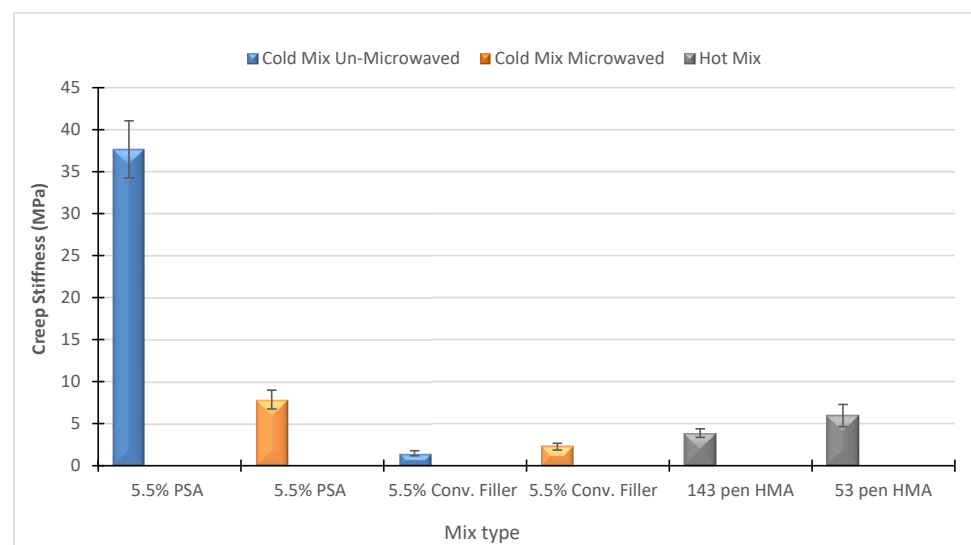


Figure 9. Effect of MW treatment on creep stiffness.

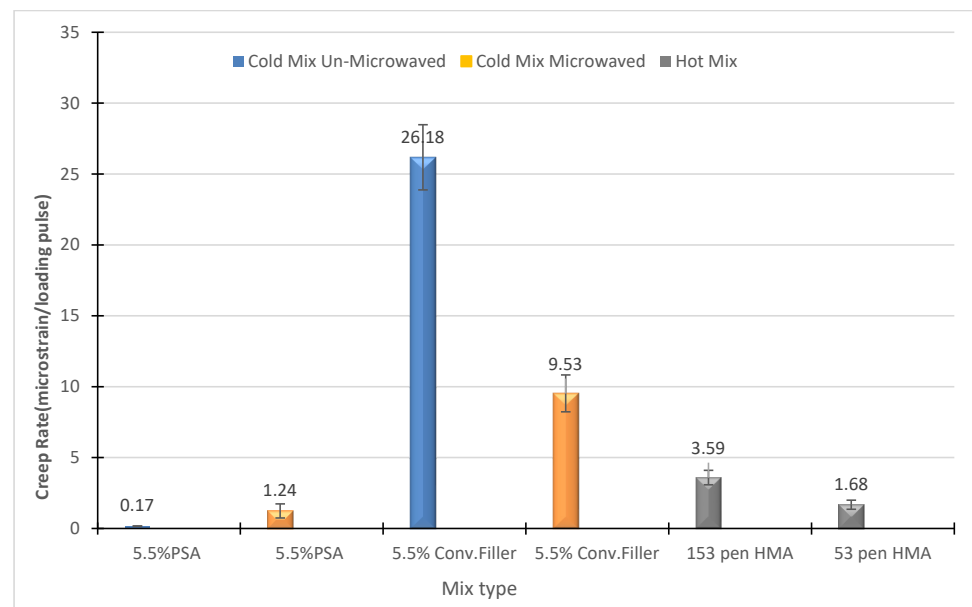


Figure 10. Effect of MW treatment on creep rate.

On the other hand, the MW treatment for CBEMs composed of 5.5% conventional filler showed a 50% increase in creep stiffness and a 64% reduction in creep rate. However, there is an upgrading in permanent deformation resistance for conventional CBEMs, but not to the levels of the HMA.

3.4. Effect of MW Treatment on CBEM Durability

The indirect tensile stiffness ratio (SMR) test's results are presented for the CBEMs to check the mixture's adequacy to water sensitivity. SMR is the ratio of ITSM of the conditioned sample over ITSM of the un-conditioned sample. Unconditioned specimens were cured at 20 °C for 10 days and specimens were cured at 20 °C for seven days; they were put in a vacuum for one hour prior to being submerged in a water bath at 40 °C for three days.

Figure 11 demonstrates the outcome results of the water sensitivity testing. The results show the significant effect of the post-mix MW treatment whereby CBEMs have been less sensitive to the presence of water. For mixtures composed of conventional mineral filler, a significant improvement in SMR was achieved; whereas, SMR jumped from 45.5% to 88.3% when the mixture was treated with MW. In addition, MW treatment preserved the gain in water sensitivity resistance of CBEM composed of PSA; the SMR was still more than 100% and proved to have better durability characteristics compared with soft and hard HMA. The reduction in air void and the improvement in the adhesivity between aggregate and bituminous material (mainly caused by polarisation action) both led to improved water sensitivity. It is correct that the reduction in air void and densification of CBEM constrain the development of the hydration of the PSA but to an acceptable limit as the presence of water does not prevent hydration occurrence.

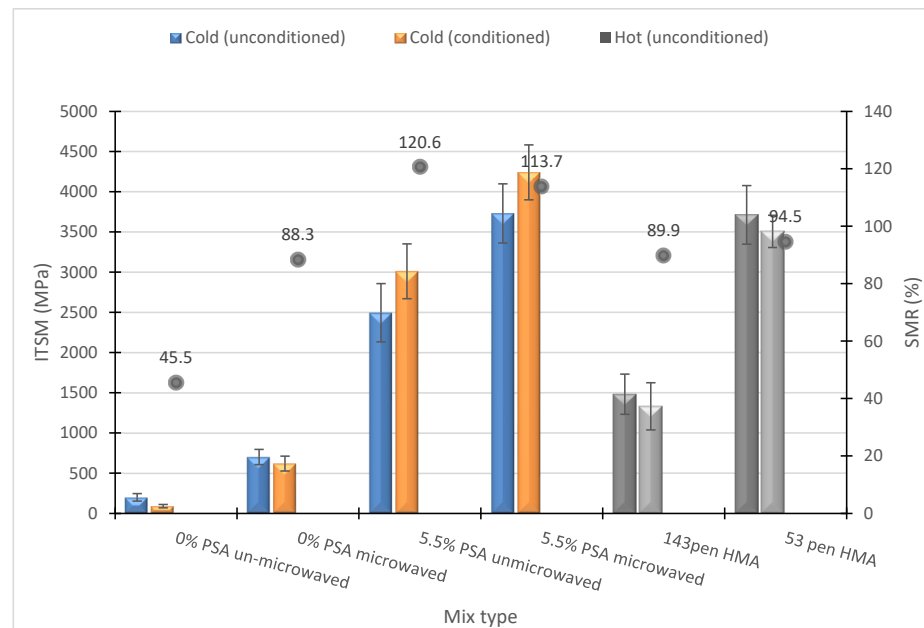


Figure 11. Effect of MW treatment on stiffness modulus ratio.

4. Conclusions

The following may be inferred based on the experimental findings of this study:

1. Post-heating MW treatment is an acceptable way of enhancing CBEMs. Both ITSM and air void content are significantly improved using both conventional and MW-heating methods. However, the MW treatment results in higher ITSM and lower air void content when the heating temperature treatment process is up to 100 °C.
2. The curing process improves as it allows the water trapped between the aggregate and the bitumen to be absorbed due to the hydraulic properties of the PSA and its high content of calcium oxide.
3. There is an association between treatment timing and produced mixture's temperature that is responsible for mixture characteristics modification.
4. The ITSM of treated CBEMs by MW is improved with curing time, where the timing of the MW is limiting the stage of improvement. Although the conventional heating method shows continuous improvement, the MW has an optimum timing value.
5. Post-mixing treatment has an inversed effect of creep stiffness or rutting resistance in contrast to that of controlled CBEM, but the inferiority is insignificant.
6. Post-mixing treatment has a slightly inversed effect of stiffness modulus ratio or water resistance in contrast to that of controlled CBEM, but the SMR reduction is limited.

This study is based on a laboratory scale; however, it is highly recommended that an in situ pavement section is carried out to detect additional challenges of MW technology because the utilization of the MW method in the field requires quite different parameters. A portable industrial MW, similar to that used for de-icing pavements, could be recommended as the principal processing tool for producing CBEM in situ. Since the 1970s, microwave heating has been applied to processing for pavement maintenance in the USA [42]. The use of microwaves in the field of asphalt technology will inevitably present some challenges but these will be resolved via continued research, testing, and improvement.

The results reveal the need to move toward post-heating techniques of CBEM, especially the MW treatment process. Post-heating limits the gap in the required properties of paving material in contrast to that of HMA, where the developed treated CBEM by post-treatment can represent a new era of sustainable paving materials, but the treatment process has to be developed in a parallel way. In other words, there is a significant need to develop a low-energy MW processor that accommodates the industrial-sized need.

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References

1. EAPA. *Asphalt in Figures*; European Asphalt Pavement Association: Brussels, Belgium, 2010.
2. Bouteiller, E.L. Asphalt Emulsion for Sustainable pavements. In Proceedings of the First International Conference on Pavement Preservation, Newport Beach, CA, USA, 13–15 April 2010; pp. 627–640.
3. Shanbara, H.K.; Dulaimi, A.; Al-Mansoori, T.; Al-Busaltan, S.; Herez, M.; Sadique, M.; Abdel-Wahed, T. The future of eco-friendly cold mix asphalt. *Renew. Sustain. Energy Rev.* **2021**, *149*, 111318. [\[CrossRef\]](#)
4. Needham, D. Developments in Bitumen Emulsion Mixtures for Roads. Ph.D. Thesis, University of Nottingham, Nottingham, UK, 1996.
5. Wang, Z.; Sha, A. Micro hardness of interface between cement asphalt emulsion mastics and aggregates. *Mater. Struct.* **2010**, *43*, 453–461. [\[CrossRef\]](#)
6. Al Nageim, H.; Dulaimi, A.; Ruddock, F.; Seton, L. Development of a new cementitious filler for use in fast-curing cold binder course in pavement application. In Proceedings of the The 38th International Conference on Cement Microscopy, Lyon, France, 17–21 April 2016; pp. 167–180.
7. Read, J.; Whiteoak, D. *The Shell Bitumen Handbook*; Thomas Telford Publishing: London, UK, 2003.
8. Dulaimi, A.; Al Nageim, H.; Ruddock, F.; Seton, L. Microanalysis of Alkali-Activated Binary Blended Cementitious Filler in a Novel Cold Binder Course Mixture. In Proceedings of the The 38th International Conference on Cement Microscopy, Lyon, France, 17–21 April 2016; pp. 189–205.
9. Ibrahim, H.E.; Thom, N.H. The Effect of Emulsion Aggregate Mixture Stiffness on Both Mixture and Pavement Design. In Proceedings of the Second European Symposium on Performance and Durability of Bituminous Materials; University of Leeds: Leeds, UK, 1997; pp. 351–368.
10. Brown, S.F.; Needham, D. A Study of Cement Modified Bitumen Emulsion Mixtures. *AAPT* **2000**, *69*, 92.
11. Forbes, A.; Haverkamp, R.; Robertson, T.; Bryant, J.; Bearsley, S. Studies of the microstructure of polymer-modified bitumen emulsions using confocal laser scanning microscopy. *J. Microsc.* **2001**, *204*, 252–257. [\[CrossRef\]](#) [\[PubMed\]](#)
12. Xu, S.F.X.; Zhao, Z.C.; Xu, Y.; Wang, X.X. Mixture Design and Performance Evaluation of Cold Asphalt Mixture Using Polymer Modified Emulsion. *Adv. Mater. Res.* **2015**, *1065–1069*, 760–765.
13. Benedito, S.B.; Wander, R.S.; Dario, C.L.; Enivaldo, M. Engineering Properties of Fibers Reinforced Cold Asphalt Mixes. *ASCE J. Environ. Eng.* **2003**, *129*, 952–955.
14. Shanbara, H.K.; Shubbar, A.; Ruddock, F.; Atherton, W. Characterizing the Rutting Behaviour of Reinforced Cold Mix Asphalt with Natural and Synthetic Fibres Using Finite Element Analysis. In *Advances in Structural Engineering and Rehabilitation*; Springer: Berlin/Heidelberg, Germany, 2020; pp. 221–227.
15. Ferrotti, G.; Pasquini, E.; Canestrari, F. Experimental characterization of high-performance fiber-reinforced cold mix asphalt mixtures. *Constr. Build. Mater.* **2014**, *57*, 117–125. [\[CrossRef\]](#)
16. Dulaimi, A.; Al Nageim, H.; Ruddock, F.; Seton, L. Assessment the Performance of Cold Bituminous Emulsion Mixtures with Cement and Supplementary Cementitious Material for Binder Course Mixture. In Proceedings of the The 38th International Conference on Cement Microscopy, Lyon, France, 17–21 April 2016; pp. 283–296.
17. Al-Busaltan, S.; Al Nageim, H.; Atherton, W.; Sharples, G. Green Bituminous Asphalt relevant for highway and airfield pavement. *Constr. Build. Mater.* **2012**, *31*, 243–250. [\[CrossRef\]](#)
18. Dulaimi, A.; Nageim, H.A.; Ruddock, F.; Seton, L. Laboratory studies to examine the properties of a novel cold-asphalt concrete binder course mixture containing binary blended cementitious filler. *J. Mater. Civ. Eng.* **2017**, *29*, 04017139. [\[CrossRef\]](#)
19. Lu, D.; Wang, Y.; Leng, Z.; Zhong, J. Influence of ternary blended cementitious fillers in a cold mix asphalt mixture. *J. Clean. Prod.* **2021**, *318*, 128421. [\[CrossRef\]](#)

20. Head, R.W. An informal report of cold mix research using emulsified asphalt as a binder. In Proceedings of the Association of Asphalt Paving Technologists Proceeding (AAPT), Lino Lakes, MN, USA, 18 October 1975; 43, pp. 110–131.
21. Du, S. Effect of curing conditions on properties of cement asphalt emulsion mixture. *Constr. Build. Mater.* **2018**, *164*, 84–93. [\[CrossRef\]](#)
22. Wang, Z.; Dai, N.; Wang, X.; Zhang, J.; Guo, H. Laboratory investigation on effects of microwave heating on early strength of cement bitumen emulsion mixture. *Constr. Build. Mater.* **2020**, *236*, 117439. [\[CrossRef\]](#)
23. Fang, X.; Garcia-Hernandez, A.; Winnefeld, F.; Lura, P. Influence of Cement on Rheology and Stability of Rosin Emulsified Anionic Bitumen Emulsion. *J. Mater. Civ. Eng.* **2016**, *28*, 1454. [\[CrossRef\]](#)
24. Thanaya, I. Improving The Performance Of Cold Bituminous Emulsion Mixtures Incorporating Waste Materials. Ph.D. Thesis, The University of Leeds, Leeds, UK, 2003.
25. Thanaya, I.; Forth, P.; Zoorob, S. Utilisation of coal ashes in hot and on cold bituminous mixtures. In Proceedings of the International Coal Ash Technology Conference, Birmingham, UK, 15–17 May 2006. Paper ref.A9.
26. Sharma, R.L.; Pandey, S.P. Influence of mineral additives on the hydration characteristics of ordinary Portland cement. *Cem. Concr. Res.* **1999**, *29*, 1525–1529. [\[CrossRef\]](#)
27. Shunsuke, H.; Fuminori, T.; Makoto, K.; Kwang, R.H. Effects of water/powder ratio, mixing ratio of fly ash, and curing temperature on pozzolanic reaction of fly ash in cement paste. *Cem. Concr. Res.* **2001**, *31*, 31–39.
28. Al-Busaltan, S. Development of New Cold Bituminous Mixtures for Road and Highway Pavements. Ph.D. Thesis, Liverpool John Moores University, Liverpool, UK, 2012.
29. Dulaimi, A.; Al-Busaltan, S.; Sadique, M. The development of a novel, microwave assisted, half-warm mixed asphalt. *Constr. Build. Mater.* **2021**, *301*, 124043. [\[CrossRef\]](#)
30. Al-Busaltan, S.; Nageim, H.A.; Dulaimi, A.; Shanbara, H.K.; Kadhim, M.A.; Al-Kafaji, M. Characterisation of Cold Bituminous Emulsion Mixtures Using Microwave Heating Process. *J. Phys. Conf. Ser.* **2021**, *1973*, 012239. [\[CrossRef\]](#)
31. BS EN 13043; Aggregates for Bituminous Mixtures and Surface Treatments for Roads, Airfields and Other Trafficked Areas. British Standard Institution: London, UK, 2013.
32. BS EN 13108; Part 1. Bituminous Mixtures Materials Specification-Asphalt Concrete. British Standard Institution: London, UK, 2016.
33. BS EN 13808:2005; Bitumen and Bituminous Binders—Framework for Specifying Cationic Bituminous Emulsions. British Standard Institution: London, UK, 2005.
34. BS EN 12591:2009; Bitumen and Bituminous Binders—Specifications for Paving Grade Bitumens. British Standard Institution: London, UK, 2009.
35. Dulaimi, A.; Al-Busaltan, S.; Kadhim, M.A.; Al-Khafaji, R.; Sadique, M.; Al Nageim, H.; Ibrahim, R.K.; Awrejcewicz, J.; Pawłowski, W.; Mahdi, J.M. A Sustainable Cold Mix Asphalt Mixture Comprising Paper Sludge Ash and Cement Kiln Dust. *Sustainability* **2022**, *14*, 253. [\[CrossRef\]](#)
36. Sadique, M.; Al Nageim, H.; Atherton, W.; Seton, L.; Dempster, N. A new composite cementitious material for construction. *Constr. Build. Mater.* **2012**, *35*, 846–855. [\[CrossRef\]](#)
37. Asphalt Institute. *Asphalt Cold Mix Manual, Manual; Series No.14 (MS-14)*, 3rd ed. Asphalt Institute: Baltimore, MA, USA, 1989.
38. Jerkins, K.J. *Mix Design Considerations for Cold and Half-Warm Bituminous Mixes with Emphasis on Foamed Asphalt*; University of Stellenbosch: Stellenbosch, South Africa, 2000.
39. BS EN 12697: Part 12; Bituminous Mixtures-Test Methods for Hot Mix Asphalt-Determination of the Water Sensitivity of Bituminous Specimens. British Standards Institution: London, UK, 2008.
40. BS EN 12697: Part 26; Bituminous Mixtures-Test Methods for Hot Mix Asphalt-Stiffness. British Standard Institution: London, UK, 2012.
41. BS EN 12697-25; Bituminous Mixtures Test Methods for Hot Mix Asphalt Part 25: Cyclic Compression Test. British Standard Institution: London, UK, 2005.
42. Bosisio, R.G.; Spooner, J.; Grønger, J. Asphalt Road Maintenance with a Mobile Microwave Power Unit. *J. Microw. Power* **1974**, *9*, 381–386. [\[CrossRef\]](#)

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