Characterisation of Cold Bituminous Emulsion Mixtures Using Microwave Heating Process

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Abstract: A previous study conducted by the authors proved that the inferiority of the mechanical properties of Cold Bituminous Emulsion Mixtures (CBEMs) could be overcome by incorporating a waste or by-product material, namely Paper Sludge Ash (PSA). The new CBEMs have demonstrated comparative mechanical and durability properties compared to conventional Hot Mix Asphalt (HMA). Furthermore, the new CBEMs have less impact on the economy, environment, and safety. However, the air void content of the new CBEMs is still high – to a stage unacceptable by pavement engineers. Thus, this study introduces a treatment method to reduce the air void without affecting the improvement achieved in mechanical properties and other environmental and economic issues. This study presents microwave energy treatment as a unique post-mix treatment method to overcome high air void content in CBEMs. Test results showed that microwave energy improves the stiffness modulus and air void content. However, new post-mix microwave treatment CBEMs still have comparative mechanical, volumetric, economic, and environmental characteristics to HMA.

Keywords: Cold bituminous emulsion mixture; microwave treatment; paper sludge ash; stiffness modulus.

1. Introduction

Annual production of Hot Mixture Asphalt (HMA) in recent years in Europe is around 309 million tones [1]. Generally, each metric ton produces 21 kg of CO₂ [2]; in other words, approximately 6.5 million tons of CO₂ have been produced in Europe annually from the asphalt production industry. In contrast with HMA, Cold Bituminous Emulsion Mixtures (CBEMs) have significantly lower CO₂...
emission during the production process, i.e., each metric ton produces only 3kg of CO$_2$. However, carbon emission could be reduced by more than 75% within the asphalt industry if the HMA is replaced by CBEM. Furthermore, and taking into account the continual rise in energy prices, the use of CBEMs offers certain advantages over HMA in terms of the total energy required; i.e., the production of each metric ton of CBEM consumes approximately 13% of the energy compared with HMA [2]. CBEMs offer further advantages such as there is no dust or gaseous emission from the binder due to the production of heat, they can be produced in remote areas, virgin and recycled aggregate can be used, and increased safety during production [3-6]. Accordingly, suitable CBEMs are in great demand as a relevant alternative for HMA. However, at present, such mixtures are unfortunately restricted to surface treatment and bond coat application [7]. Mostly, CBEMs have shown inferior mechanical properties, especially in early life, such as high air void and long curing time needed to gain ultimate strength [8-12].

Upgrading CBEMs’ engineering properties characteristics has attracted many researchers to overcome the disadvantages in such materials [13]. They have tried different additives and techniques to achieve this goal. Portland cement [14, 15], rapid setting cement [16], fibers [17-20], and polymers [21] have been used as additives, while double mixing, two mixing stages, and pre-coating with asphalt have been used as assistance techniques [22]. Unfortunately, most of the said attempts either fail to satisfy the required goals, or they have cost or environmental impact.

Utilizing waste or by-product materials is becoming increasingly important in this field and, in recent years, many researchers have studied the possibilities of replacing some of the virgin materials with waste or by-product materials [23-27]. Although such studies achieved advances in strength characteristics of CBEMs, none of them achieved the ultimate strength of the CBEMs in a short curing time, i.e., less than seven days [28]. Additionally, compacted mixtures still have high air void to unacceptable ranges according to the valid specifications.

A feasibility study achieved by the authors suggested a new CBEM, domestic fly ash, namely Paper Sludge Ash (PSA) could be used as a replacement for the conventional mineral filler [29]. This ash provided CBEMs with a significant improvement in mechanical and durability properties. It is believed that the hydraulic characteristics of the PSA play a significant role in generating a new binding resource besides the primary binding of the bituminous binder. Also, the hydration process due to the presence of PSA and water in the mixture leads to the absorption of trapped water between the aggregate and bituminous binder, whereas this product strengthens the bituminous binder itself. However, the inferior characteristics have been solved completely as the new CBEMs offered comparative mechanical properties to traditional HMA within an acceptable curing time. Nevertheless, it was recognized that the air void content was still within an unacceptable range. Thus, a new process should be introduced to the new CBEMs to control the air voids. Heating of the bituminous mixture facilitates the backing of the mixture’s ingredients as it minimizes the bitumen’s viscosity; consequently, the compacted mixture will show low air voids. The authors suggest the examination of microwave heating as a sustainable alternative for reducing the air voids of the new CBEMs, as the heating by microwave needs less power than traditional heating [30].

Heating materials using microwaves have proved efficient within the last 70 years. The industrial application of microwave has gained a lot of interest since the 1980s, especially in the food, textile, paper, and rubber industries. The heating mechanism in a microwave is independent of materials’ thermal conductivity; it is dependent on the polarization of the dielectric of the molecules of the heated materials. Consequently, the said heating technique suggests some advantages over conventional heating in terms of time required, total energy consumption, heating uniformity, and volumetric material properties. The main aim of this study is to characterizing the significant of microwave processing in improving the CBEM, in other word introducing sustainable method in paving industry.
2. Experimental Program

2.1. Materials
The coarse aggregate used in this study was crushed green granite, while the fine aggregate was sand. Their physical properties were tested according to British Standards BS EN 13043 [31] test procedures for aggregates used for bituminous mixtures for highways and airfields. The results can be seen in Table 1. The aggregates’ gradation were selected as 0/10 mm close-graded surface course according to the BS EN 13108-1 [32] requirements for the asphalt concrete mixtures; the gradation can be seen in Table 2.

| Table 1. Physical properties of the aggregate according to BS EN 13043 |
|---------------------------------------------------------------|
| **Properties** | **Value** |
| Coarse aggregate | | |
| Bulk specific gravity, g/cm³ | 2.79 |
| Water absorption, % | 0.4 |
| Apparent specific gravity, g/cm³ | 2.82 |
| Fine aggregate | | |
| Bulk specific gravity, g/cm³ | 2.74 |
| Water absorption, % | 0.4 |
| Apparent specific gravity, g/cm³ | 2.77 |

| Table 2. Aggregate grading for 0/10 mm size close-graded surface course (BS EN 13108-1) |
|-----------------------------------------------|
| **Test sieve aperture size mm** | **% By mass passing specification range** | **% By mass passing mid** |
| 14 | 100 | 100 |
| 10 | 95-100 | 97.5 |
| 6.3 | 55-75 | 65 |
| 2 | 19-37 | 28 |
| 1 | 10-30 | 20 |
| 0.063 | 3-8 | 5.5 |

| Table 3. Bituminous binder and bitumen emulsion properties |
|----------------------------------------------------------|
| **Bitumen emulsion** | **Bituminous binder 100-150** | **Bituminous binder 40-60** |
| **properties** | **value** | **properties** | **value** | **properties** | **value** |
| Appearance | Black to dark brown liquid | Appearance | Black | Appearance | Black |
| Boiling Point, °C | 100 | Penetration, 25 °C | 143 | Penetration, 25 °C | 43 |
| pH | 5 | Softening point, °C | 43.6 | Softening point °C | 52.4 |
| Relative Density at 15 °C g/ml | 1.05 | Kinematic Viscosity at 135°C | 175 | Kinematic viscosity at 135°C | 325 |
| Residue by distillation, % | 56 | Density at 25 °C | 1.00 | Density at 25°C | 1.01 |
Table 4. Chemical composition of fillers

| Element | Concentration | Mineral filler | PSA |
|---------|---------------|----------------|-----|
| CaO     | 5.58          | 60.93          |     |
| Al₂O₃   | 9.221         | 3.471          |     |
| SiO₂    | 53.597        | 28.178         |     |
| Fe₂O₃   | 7.368         | 0.202          |     |
| MgO     | 4.984         | 3.554          |     |
| K₂O     | 3.123         | 0.354          |     |
| TiO₂    | 0.831         | 0.556          |     |

2.2. Mix design and sample preparations

To date, there is no UK or universally accepted design mixture for the CBEMs. Consequently, all samples produced were prepared following the method adopted by the Asphalt Institute (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design (MS-14)) [33]. Only one change has been made to the said method by replacing Marshall test with the indirect tensile strength. The design method and sample preparations were achieved through the following steps:

- Determine the aggregate gradation: the selection of the aggregate gradation has been done as mentioned in section 2.1.
- Determine the initial emulsion content: an empirical equation that depends on the aggregate gradation was used as recommended by the Asphalt Institute manual, MS-14.
- Determine pre-wetting water content: coating ability of the bitumen emulsion to the aggregates is highly sensitive to the pre-wetting water content, especially when the aggregate gradation contains a high percentage of materials passing through a 63 µm sieve. Thanaya [22] reported that inadequate pre-wetting water content results in balling of the binder with a fine portion of the aggregate and thus unsatisfactory coating. Different pre-wetting water contents were investigated to find the lowest percentage to ensure adequate coating.
- Optimum emulsion content: indirect tensile strength test is used to determine the optimum emulsion content according to BS EN 12697-23 [34].
- Determine the optimum total liquid content: the mix density test was used to determine the optimum total liquid content at compaction (i.e. emulsion plus pre-wetting water contents, which give the highest mix indirect tensile strength and density); the test was conducted according to BS EB 12697-6 [35].

Therefore, according to the selected materials’ characteristics, pre-wetting water content was observed to be 4%, the optimum bitumen emulsion was 11.5%, and the optimum total liquid content at compaction was 14.5% of the aggregate weight.

Specimens of CBEMs were prepared using different amounts of PSA as a replacement for the mineral filler ranging from 0 to 5.5% of the total aggregate weight. Furthermore, conventional HMA samples were prepared with the same aggregate type and gradation; 5.3% binder content was used. Both cold and hot mixes were prepared in quantity to produce three 1100 g specimens for each specific mix. Mix type and the percentages of the filler used are shown in Table 5.

Table 5. Mixtures matrix

| Mix type   | Filler                  |
|------------|-------------------------|
| CBEM-1     | 5.5% conventional mineral filler |
| CBEM-1F    | 1.375% PSA              |
| CBEM-2F    | 2.75% PSA               |
| CBEM-3F    | 4.125% PSA              |
| CBEM-4F    | 5.5% PSA                |
| HMA-1      | HMA 143 pen             |
| HMA-2      | HMA 53 pen              |
2.3. Apply the microwave to the samples

After mixing of the materials according to the said MS-14 method, the CBEM samples were subjected to two types of post-mix heating, namely conventional heating and microwave heating. Microwave heating was achieved by using a home-type microwave with a frequency of 2.45GHz. Three microwave power levels were used; the details can be seen in Table 6. In addition, four post-mix microwave times were used (2.5, 5, 7.5 and 10 min), and a non-microwaved control sample was prepared. The conventional heating samples were heated to the same temperatures initiated by microwave heating for different lengths of time. It was found that the samples’ temperature due to microwave subjection for 2.5, 5, 7.5 and 10 minutes are 76, 82, 90 and 101 ºC, respectively.

The power level in a home-type microwave is controlled through a process called duty-cycle control. In this process, the microwave magnetron is operated at full power but with segmented periods; i.e. within a specific cycle the magnetron is on for a specific period and then off for another period; after that this cycle of on/off is replicated within the selected heating time. Normally, the full power level implies the continuous operation of the magnetron, while for other levels, there are specific on/off periods within the cycles depending on the required level. Accordingly, the on time over the total cycle time limits the power level. However, the actual heating time is the accumulation of on times. In this study, the duty cycle of the microwave used is 30 sec, and the full magnetron power is 860 watts, which is measured by a power meter. Three power levels were used to investigate the effect of different power levels within different subjected times; the details of the power levels used can be seen in Table 6. After post-mix microwave subjection, impact compaction (Marshall Hammer) was applied with 50 blows to each face of the specimens.

| Level | Category | Actual power on time (sec) | Power setting % | Actual power within 30 sec (KW) |
|-------|----------|-----------------------------|-----------------|-------------------------------|
| 1     | low      | 6                           | 20.00           | 5.784                         |
| 2     | medium   | 16                          | 53.33           | 14.124                        |
| 3     | high     | 30                          | 100.00          | 25.800                        |

2.4. Samples’ conditioning

CBEMs’ mechanical properties are very sensitive to curing period and curing temperature. Therefore, specific curing protocols were selected according to previous research to characterize the mechanical properties of different CBEMs. For ITSM test specimens Jenkins’ [36] protocol was adopted, while for UCCT specimens Thanaya’s [22] protocol was adopted. On the other hand, the curing protocol for durability test samples was achieved according to BS EN 12697-12 [37]. The details of the three protocols can be seen in Table 7.

| Test                                      | First stage curing | Second stage curing | Time testing (days) | Recommendation |
|-------------------------------------------|--------------------|---------------------|---------------------|----------------|
| Indirect tensile stiffness modulus (ITSM) | 20 ºC for 1 day    | 40 ºC for 1 day     | 2,7,14,28,90, 180 and 360 days | [38] |

2.5. Methods

Mechanical properties of CBEMs were explored through the ITSM fundamental test. The test was conducted in accordance with BS EN 12697-26 [39], using Cooper Research Technology HYD 25 testing apparatus. The samples were conditioned for 4hr before testing. The test was conducted at 20 ºC.
3. Results and discussion

3.1. Apply the microwave to the samples

Results of ITSM for samples subjected to conventional and microwave post-mix heating are shown in Figure 1. Generally, for conventional post-mix samples, the ITSM decreased due to heating compared to the non-heated sample, but with an increase in heating temperature, the ITSM started to increase. For the microwave heating samples, there is a decrease in ITSM value associated with 2.5 min microwaving, then an increase with the increase in time; after that, ITSM dropped when subjected to more than 7.5 min microwaving. On the other side, the air void of the samples subjected to conventional post-mix heating showed a continuous decrease due to an increase in heating temperature; while samples subjected to microwaving displayed a turning point of increase in air void after a decrease at 7.5 min microwaving, as can be seen in Figure 2.

![Figure 1. Effect of post-mix time on ITSM of CBEMs comprised of 5.5% PSA 2days’ curing](image1)

![Figure 2. Effect of post-mix time on air void of CBEMs comprised of 5.5% PSA](image2)

From these results, however, it is suggested that heating temperatures were not only the main role in identify stiffness modulus and air void characteristics of post-mix heating CBEMs; whereas microwave energy generates two mechanisms, namely heating and polarization. The heating of CBEM
components led firstly to increase evaporation of free water and increase the rate of bitumen emulsion breaking, and secondly to decrease the viscosity of the base bituminous material, accordingly better coating the aggregate particles by bituminous materials. On the other hand, the polarization of the charges in both aggregates and bitumen emulsion led to an increase in the rate of adhesiveness between them [30]. In the results for the same post-mix temperatures, CBEMs treated with the microwave showed the highest stiffness modulus and fewer air voids; hence, such a post-mix technique is more beneficial for improving the mechanical properties of CBEMs. Nevertheless, a specific percentage of wetting water should be kept to work as a lubricating agent during the compaction stage; in addition, this percentage of wetting water is required for the hydration process of the hydraulic filler. So, post-mix heating should be optimized to specify the highest mechanical properties with the lowest air void.

3.2. Effect of microwave power level on stiffness modulus and air void

Test results of different post-mix microwave power levels are shown in Figures 3 and 4. Stiffness modulus results showed that there was a major difference among the ITSM values due to differences in power level and treated time. For low power level generally, there was an increase in ITSM value with an increase in post-mix microwave treatment time, while for medium and high-power levels there was an optimum value. In addition, low power levels increase ITSM value more than non-microwave-treated CBEM, normally due to the polarization effect of the microwave, while the polarization and heating actions in medium and high-power levels lead to optimum ITSM value. Besides, it was noted that there is a drop in stiffness at a specific treatment time and power level; it is suggested that this drop is due to polarization action whereas leads to agglomerate of the bitumen emulsion result in bad bitumen coating.

Results of the air void indicated a continuous reduction for low and medium power levels, while for high power level, there is a turning point after which with an increase in microwave treatment, the air void starts to increase again, as can be seen in Figure 4. Also, the results showed that the rates of reduction in air void are increased with increasing power level. However, it is suggested that microwave heating increases the aggregate and water temperature as the dielectric permittivity and heat transfer characteristics dominate the microwave power, then this heating transfers to the bituminous material resulting in a reduction in its viscosity. Consequently, the compatibility of the mixture increased and the final air void decreased; also, the coating of the aggregate by bituminous material improved. However, removing the water to a specific percentage will affect both the stiffness and air void, as water is a major lubricating agent in the cold mix; also, water is required for the hydration process of the filler. Figure 5 demonstrates the above as it can be seen that the rate of the increase in stiffness modulus is decreased as microwave treatment time increases. For example, gains in stiffness after 90 days are 272, 223, 184 and 166% for a microwave treatment time of 2.5, 5, 7.5 and 10 min, respectively. In addition, in contrast with non-microwave-treated samples, microwave-treated samples generally showed degradation values with an increase in curing time, which is because of the removal of water that is required for the hydration process. However, these values are still comparative to soft and hard HMA at optimum microwave treatment power and time, which suggests that the microwave treatment of CBEMs comprising hydraulic filler is significantly benefited in terms of reducing the air void while preserving the gain in stiffness modulus at acceptable levels.
Figure 3. Effect of different microwave energy and time on ITSM of CBEMs comprised of 5.5% PSA

Figure 4. Effect of different microwave energy and time on air void of CBEMs comprised of 5.5% PSA

Figure 5. Effect of microwave time on ITSM and air void of CBEMs with 5.5% PSA with different curing times
The results for the conventional CBEMs are shown in Figure 6. Full power microwaved post-mix at various times was used. Test results indicate that there is a continuous improvement in ITSM with increase in microwave treatment time; at the same time, air void content showed an optimum value around 7.5 min. In addition, it can be recognized that ITSM value of 10 min reached soft HMA value within 28 days, which is an outstanding achievement as the non-treated conventional CBEMs normally need 2-24 months. However, as there is no hydraulic filler incorporated in conventional CBEMs, the removal of the free water helps in the continuous improvement of ITSM; while it is clearly shown that still there is a specific amount of water required to obtain low air void content.

Figure 6. Effect of microwave time on ITSM and air void of conventional CBEM

4. Conclusions

Based on the experimental results of this study, the following can be concluded:
1. Post-mixing treatment is a valid method to upgrade CBEMs. When comparing a conventional heating method to microwave treated, the second has proven better.
2. The microwave generally reduces the air void content to acceptable levels; 7.5 minutes with full power energy was found to be an optimum treatment condition.
3. The microwave treatment introduces two mechanisms of treatment for CBEM, namely heating and polarization.
4. Microwave treatment modifies stiffness modulus and air void to levels acceptable to the pavement engineer.

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