



DEVELOPMENT OF A PREMIUM COLD MIX ASPHALT

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ABSTRACT

Cold mix asphalt produced using conventional emulsion and limestone filler has three main disadvantages, namely low early strength due to high amount of water within the mix, long curing time ranging from 2-24 months and high air voids content.

In this study, the effect of a novel technology for producing high-quality cold mix asphalt using modified bitumen emulsion has been investigated in terms of indirect tensile stiffness modulus. the effect of a novel technology for homogenizing a bitumen emulsion on its viscosity, particle size and particle size distribution has been investigated. Ultrasound technology has been used to reduce the viscosity and the size of the bitumen droplets. Different samples of cationic bitumen emulsion (C50B4) have been prepared with different treatment durations (0, 5, 7, 10, 12.5, 15, 30, 45 and 60) min. The results have shown a reduction in the viscosity of the bitumen emulsion at treatment times from 5-15 min. The viscosity has been decreased by 28% after 10 min treatment for the emulsion compared to the untreated sample. This reduction indicated a uniform droplet size distribution. In addition, the particle size measurements revealed that the 7 min treatment showed a significant reduction in the D₅₀, which was 84.5%, D₁₀, was 85.89%, and D₉₀ was 90.28% compared to the untreated bitumen emulsion. The results reported contains a comparison between the viscosity of the emulsions, and the indirect tensile stiffness modulus of a control conventional cold mix asphalt and the new high quality cold mix asphalt with low viscosity. Interestingly, the reduction of the 28% in the viscosity of the new emulsion causes an improvement of the indirect tensile stiffness modulus to 216%, which indicates an outstanding achievement.

KEYWORDS: Ultrasound technology, bitumen emulsion, viscosity, indirect tensile stiffness modulus, cold mix asphalt.

1. INTRODUCTION

The most popular application of bitumen emulsion is producing the cold asphalt mixtures for paving roads. Cold bitumen emulsion mixtures (CBEMs) technology allows the mixing, laying and compaction of mixtures with no heat. CBEM experiences some drawbacks including low early life strength, high air voids and long curing time, thus it needs from 2-24 months to reach its ultimate strength. Accordingly, the applications for CBEM have been limited to low/medium trafficked roads and footways (Ojum, 2015). This problem has been solved by replacing limestone filler with ordinary Portland cement (OPC) (Oruc et al., 2007). However, the use of OPC has economic and ecological drawbacks (Schneider et al., 2011). Having said the above, the emulsion in CBEM has the durability, viscosity, bonding associate problems due to the non-uniform droplet size (Salomon, 2006).

Bitumen emulsions have different applications in the pavement industry such as surface dressing, prime and tack coats, bond coat, chip seals, micro-surfacing and cold asphalt mixtures. Regardless of its application, there are four essential physical parameters controlling the overall performance of bitumen emulsions: viscosity, stability, coating and breaking. These parameters are strongly affected by the bitumen particle size distribution (PSD) which also plays a significant role in the interactive bond between the cement and bitumen emulsion in the asphalt mixture (Hu et al, 2009). Macro emulsions are inherently in an unbalanced state, attempting to achieve a state of equilibrium over time through sedimentation, flocculation and coalescence. As a result of these destabilising processes, the droplet size distribution will alter, this eventually leading to complete separation of the phases (Sjoblom, 2001). Emulsions with small globule size are more kinetically stable against sedimentation because of the increase in the repulsive forces between the particles due to the absorbed layer of surfactant and also the movement of the droplets due to gravitational forces is hindered by their neighbours; thereby given sufficient time before separate into their phases. This leads to an increase in the repulsive forces between particles, reducing the effect of destabilising actions (Tadros et al, 2004). In general, smaller particle size produces noticeable enhancements in the performance of bitumen emulsion in both mix and spray applications (Deneuvillers and Samanos, 2000).

Recently, interest has grown in the development of technological processes for the production of bitumen emulsion in order to control its physical and performance properties by adjusting the particle size and particle size distribution during the manufacturing process. Currently, ultrasound technology is the most promising and is applied in various industrial sectors, offering several improvements in performance compared to normal manufacturing processes.

This study has therefore investigated, for the first time, the influence of ultrasound technology on the operational characteristics and performance of a bitumen emulsion, by changing the particle size and particle size distribution of the bitumen droplets. This has had an outstanding influence on the viscosity of the new emulsion (ultrasonic emulsion). Furthermore, the mechanical performance of the CBEM in terms of indirect tensile stiffness has been improved by incorporating the developed bitumen emulsion.

2. MATERIALS

2.1. BITUMEN EMULSION

In this study, a slow setting cationic emulsion (C50B4) comprising 50% residual bitumen designed for using in road pavement and general maintenance applications has been used. Jobling Purser, Newcastle, UK, supplied the emulsion. [Table 1](#) shows the main properties for the bitumen emulsion.

Table 1. Properties of the bitumen emulsion.

Material	Property	Value
Bitumen emulsion	Type	Cationic
	Appearance	Black to dark brown liquid
	Base bitumen, 1/10 mm	50
	Bitumen content, %	50
	Boiling Point, °C	100

2.2. MINERAL AGGREGATE

The aggregate used in this study is crushed granite for both coarse and fine aggregate obtained from Carnsew Quarry at Mabe, Penryn, UK (operated by Colas). These materials are normally used for producing asphalt pavement mixtures. The aggregates are washed, dried, riffled and bagged with the sieve analysis achieved according to BS EN 933-1 ([European Committee for Standardization, 2012a](#)). A close graded surface course with 10 mm gradation has been selected for this study according to the standard BS EN 13108-1 ([European Committee for Standardization, 2006](#)). The grading of the 0/10 mm mix is shown in [Fig. 1](#). The physical properties of the coarse and fine aggregates with conventional limestone filler are listed in [Table 2](#). This selection was made in order to ensure an appropriate interlock between the aggregate particles in the mixtures. Add to that, this gradation has been used successfully in

heavy traffic surface course hot coated macadam (European Committee for Standardization, 2015).

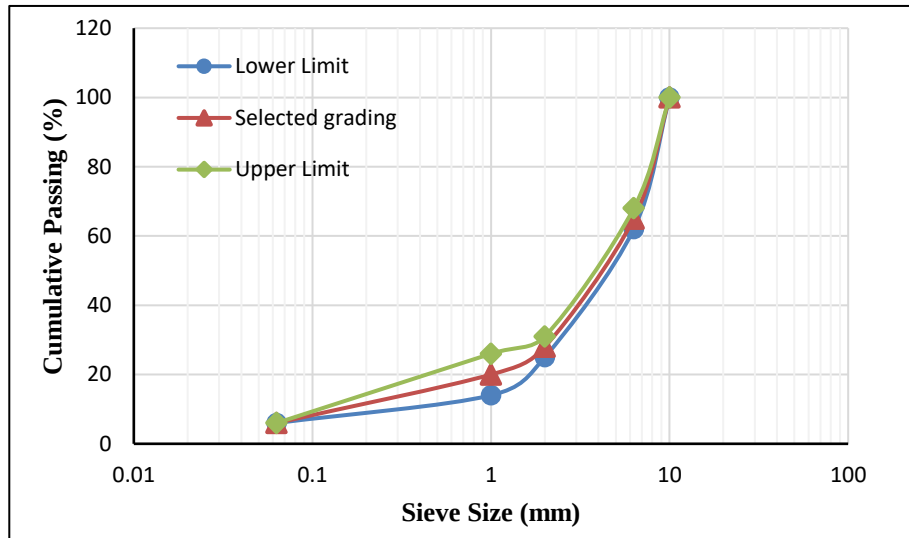


Fig. 1. The gradation of AC 10mm close graded surface course.

Table 2. Physical Characteristics of the Granite Aggregate.

Material	Property	Value
Coarse aggregate	Bulk Particle Density, Mg/m ³	2.60
	Apparent particle Density, Mg/m ³	2.66
	Water Absorption, %	0.8
Fine aggregate	Bulk Particle Density, Mg/m ³	2.52
	Apparent particle Density, Mg/m ³	2.58
	Water Absorption, %	1.6
Conventional mineral filler	Particle Density, Mg/m ³	2.64

2.3. FILLER

A commercial limestone filler was obtained from Francis Flower Ltd, achieving the following parameters: CaCO₃ content greater than 70%; loose bulk density in kerosene between 0.5-0.9 g/ml; more than 70% of particle size is passing 63μm, and a moisture content of less than 0.1%, used in this study.

3. SAMPLE PREPARATION METHOD

All samples produced for this study was prepared according to the method adopted by the Asphalt Institute (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design) ([Asphalt Institute \(MS-14\), 1989](#)). According to the selected materials characteristics, pre-wetting water content was observed to be 3% and the optimum bitumen emulsion was 12.5%. Specimens of cold bitumen emulsion mixtures were prepared using different phases of treated emulsions using ultrasound apparatus. Impact compacting (Marshall Hammer) was applied with 50 blows to each face of the specimens. The cold mix specimens were mixed and compacted at lab temperature (20 – 25° C).

4. LABORATORY TESTING PROGRAMME

4.1. SONICATION TREATMENT OF THE BITUMEN EMULSION

Ultrasound has been shown its ability to produce emulsions with smaller particle sizes thanks to the cavitation phenomenon responsible for ultrasonically induced effects ([Jayasooriya et al, 2004](#)). The propagation of acoustic waves through aqueous media causes pressure fluctuations in simple sound waves which result in the formation and violent collapse of small bubbles during the expansion cycles. This breaks coarse particles down into smaller sizes.

In this study, a pre-prepared, cationic bitumen emulsion (C50A4), containing 50% residual bitumen content with macroscale bitumen droplets, was subject to ultrasonic agitation treatment using a high power ultrasonic device. A probe system ultrasonic processor manufactured by Hielscher Ultrasound Technology, Model UP 400S ([Fig. 2](#)), was used to modify the bitumen emulsion. The horn tip of a 22 mm diameter cylindrical titanium probe was dipped in the conventional bitumen emulsion, the sonication process was then turned on to the highest power (100% amplitude), at a constant frequency of 24 kHz.

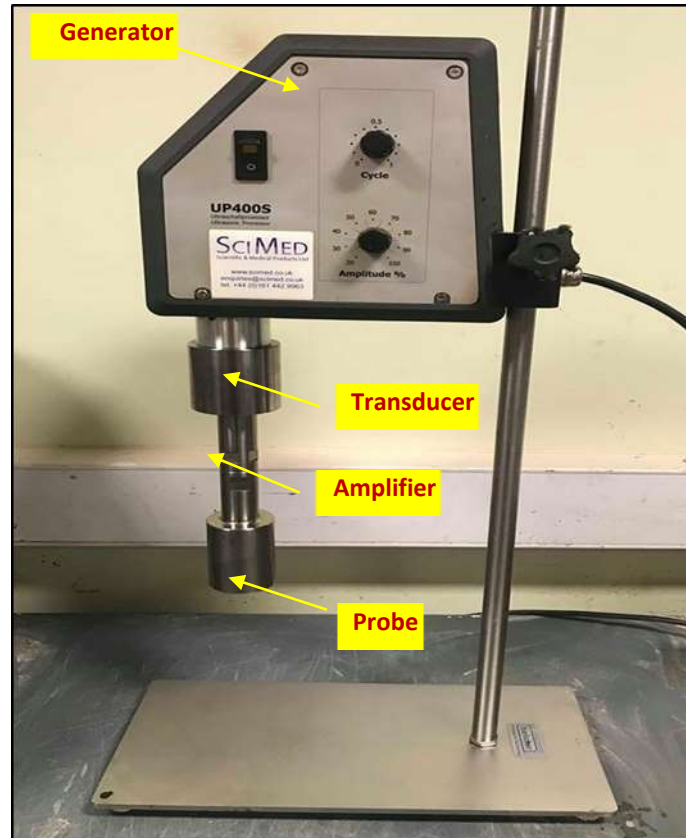


Fig. 2. Ultrasound apparatus.

4.2. VISCOSITY MEASUREMENT

Viscosity can be defined as a measurement of a fluid's resistance to flow. This test has been conducted according to BS EN 13302 (European Committee for Standardization, 2010) using a rotating spindle apparatus, as shown in Fig. 3. The apparatus used in this study is Brookfield DV-II+ Pro Viscometer, which basically consists of two parts- a head unit with a motor and a spindle that is driven by the motor. Principally, DV-II+ Pro operates by means of a disk shape spindle that is immersed into the liquid sample for measuring the resistance of the substance at a selected known speed. This resistance results are the measurement of the viscosity according to the flow characteristics of the reference spindle, the instrument calculates the result and directly displays the viscosity that is reported in centipoise (cP) using the following formula:

$$\text{Full Scale Viscosity Range (cP)} = \text{TK} * \text{SMC} * 10,000/\text{RPM} \quad (1)$$

Where:

TK = DV-II+Pro Torque Constant (for the RV model = 1)

SMC = Spindle Multiplier Constant (for the spindle entry code 07 = 400)

RPM = Revolution per minute

The viscosity for all the bitumen emulsion samples treated by ultrasound at different phases have been measured at ambient temperature, as well as the untreated emulsion sample for comparison purposes. The viscosity result for each sample is the average of three measurements.



Fig. 3. Viscometer Apparatus.

4.3. DROPLET SIZE MEASUREMENTS

Particle size (PS) and particle size distribution (PSD) were characterized by a Beckman Coulter Laser Diffraction Particle Size Analyser LS 13 320 apparatus, shown in Fig. 4, for different lengths of ultrasonic treatment. Five measurements were carried out for each sample at lab temperature, using three samples for each treatment time. PSD was presented as a volume percentage vs. droplet diameter. The most commonly used metrics when describing particle size distributions are D-Values (D_{10} , D_{50} & D_{90}) which are the intercepts for 10%, 50% and 90% of the cumulative mass. In addition, the width or ‘span’ of the PSD was calculated from the following equation:

$$\text{Span} = [D_{90} - D_{10}] / D_{50} \quad (2)$$

where

D_{50} : is the size in microns that splits the distribution with half above and half below this diameter.

D_{10} : is the diameter at which 10% of the sample's mass is comprised of particles with a diameter less than this value.

D_{90} : is the diameter at which 90% of the sample's mass is comprised of particles with a diameter less than this value.



Fig. 4. Laser diffraction particle size distribution analyser.

4.4. INDIRECT TENSILE STIFFNESS MODULUS (ITSM) TEST

To assess the load bearing capacity of the layer manufactured from the asphalt mix, the indirect tensile stiffness modulus was determined. The ITSM tests were carried out at 20°C and were conducted on cylindrical specimens following the BS EN 12697-26 ([European Committee for Standardization, 2012b](#)) using a Cooper Research Technology HYD 25 testing machine as shown in [Fig. 5](#). Many researchers such as ([Al-Hdabi et al., 2013](#)), ([Nassar et al., 2016](#)), ([Dulaimi et al., 2016](#)), ([Al-Busaltan et al., 2012](#)) have adopted ITSM in order to evaluate the stiffness modulus of CMAs. Three samples have been used for each mixture type. The test conditions were as shown in [Table 3](#).

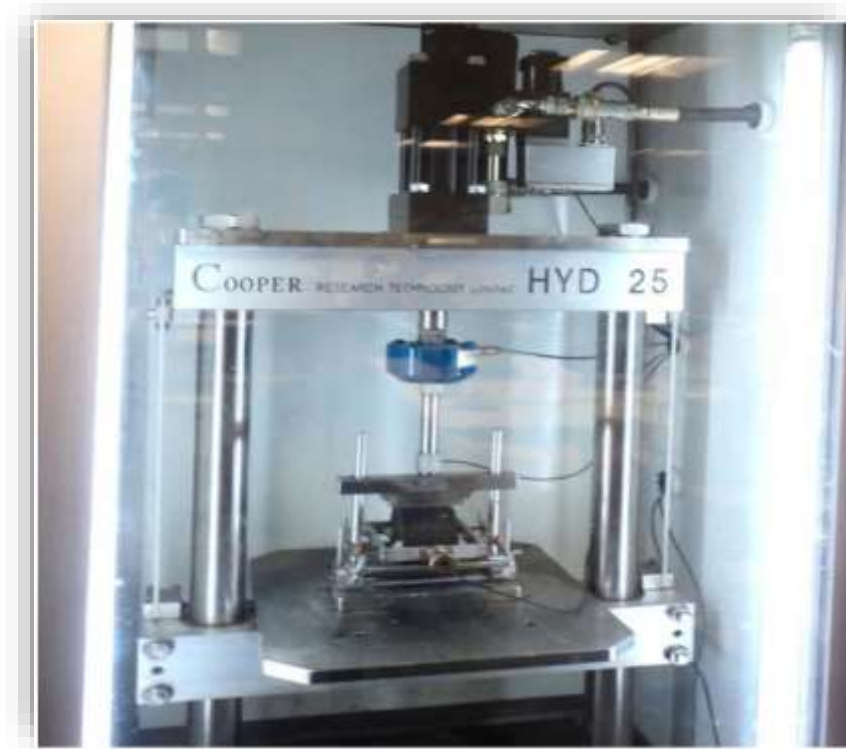


Fig. 5. Indirect tensile stiffness modulus apparatus.

Table 3. Conditions of the ITSM test.

Item	Range
Specimen diameter, (mm)	100 ± 3
Rise time, (ms)	124 ± 4
Transient peak horizontal deformation, (mm)	5
Loading time, (s)	3–300
Poisson's ratio	0.35
No. of conditioning plus	10
Specimen thickness, (mm)	63 ± 3
Compaction	Marshall 50 $\times$ 2
Specimen temperature conditioning	4 hrs. before testing

5. RESULTS AND DISCUSSION

5.1. SONICATION TREATMENT

As the field of emitted sound is usually heterogeneous in ultrasound instruments, it is important to recirculate the emulsion sample so that all bitumen droplets experience the same shear rate

and achieve a reasonably uniform particle size distribution. During the sonication process, the temperature of the treated emulsion was raised gradually because of the cavitation phenomenon. A digital thermometer was put on the side of the beaker to measure the temperature of the emulsion sample at the end of treatment. Fig. 6 shows the elevation in temperature by the increase in processing time. The temperature at 0 to 20 min sonication processing time, increased linearly by approximately 275% compared to the normal (untreated) sample. After 20 minutes, the elevation in temperature was only 7% up to 60 min treatment. At the beginning of the sonication process, a violent turbulence in the emulsion sample was noticed, this accompanied by a loud sound which was attributed to the occurrence of cavitation. As the treatment time increased, the processing action became much steadier and calmer. This explains the considerable elevation in the sample's temperature at the beginning of treatment. In order to eliminate the impact of temperature rise on the properties of the treated bitumen emulsion, all treatments were performed in a cold water bath.

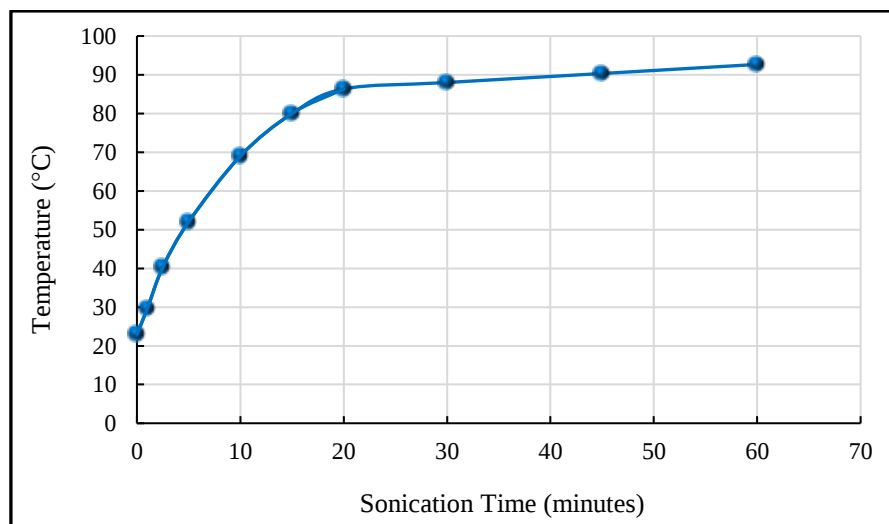


Fig. 6. Effect of sonication treatment time on temperature.

5.2. VISCOSITY RESULTS

All the experiments were implemented in 600 mL glass beaker at lab temperature in triplicate. From the results, shown in Fig. 7, the viscosity of the emulsions decreased with increasing the sonication treatment time phase, which indicates that samples exhibit shear-thinning behaviour at these treatment levels. After phase 7 of treatment, the viscosity has been increased gradually. Phase 0 represents the untreated emulsion.

From the results shown in Fig. 7, the viscosity of the emulsions initially decreased with increasing treatment time, which indicates that samples exhibited shear-thinning behaviour at

these ultrasonic duration levels. The reduction in the viscosity at 7 min treatment, was approximately 28% compared to the normal sample. Similar findings were presented by Wang et al (2006) in that the effect of ultrasonic treatment on petroleum coke oil slurry is not limited to simply decreasing the apparent viscosity, but also markedly improves its stability. Another study conducted by [Kaltsa et al \(2014\)](#) found that increasing the time or amplitude reduces the viscosity and droplet size in olive oil model emulsions containing xanthan, while also improving its stability. This reduction can be explained by the fact that when applying ultrasonic waves, the bitumen emulsion was more dispersed as the diameters of the bitumen droplets had decreased. Consequently, the amount of emulsifier adsorbed on the surface of the bitumen droplet decreased, i.e. the total bitumen-water interfacial surface area increased while the emulsifier film thickness decreased. Following this stage, sonicated bitumen emulsion will reach a mono-disperse state where the viscosity has decreased compared to that of the normal emulsion. Several researchers working on the physico-chemical characteristics of emulsions have found that the increase in the thickness of an emulsifier in an emulsion, leads to an increase in its viscosity ([Napper, 1983](#); [Karpenko and Gureev, 1998](#)).

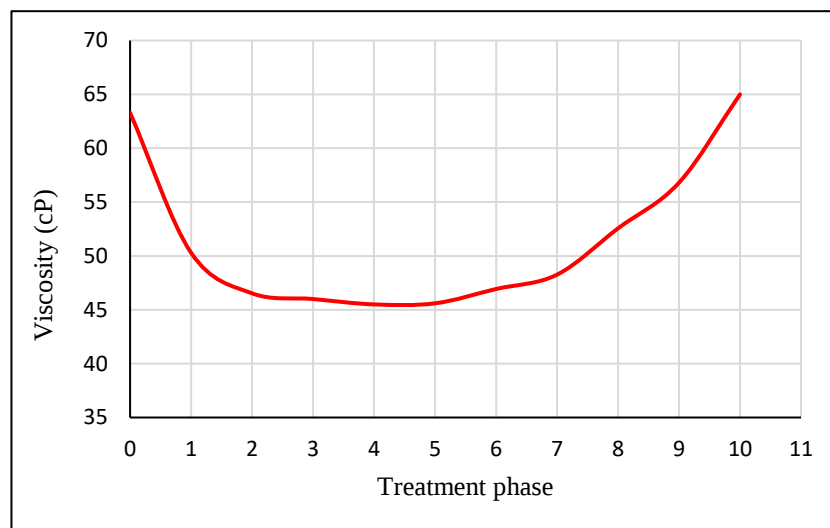


Fig. 7. The change in the viscosity of the treated bitumen emulsion samples.

After 10 minutes of sonication, viscosity increases gradually. As treatment time increases, dispersion and coagulation of the bitumen droplets start to compete because the temperature has risen due to the vigorous mechanical mixing of the emulsion (over-processing). Coagulation of the bitumen globules will occur after a certain period of sonication treatment because of the increase in the number of globules, leading to increasing collisions through Brownian motions. The coalescence of bitumen particles will occur when the amount of emulsifier at the interface drops to a level where it loses its function as a barrier to prevent

contact and coalescence of droplets. Therefore, the optimal time for ultrasound treatment should be used to avoid shear-induced coalescence and to prepare a more monodispersed bitumen emulsion with lower viscosity.

5.3. PARTICLE SIZE AND PARTICLE SIZE DISTRIBUTION

Fig. 8 shows the differential PSD for all bitumen emulsion samples treated with different sonication durations, while Fig. 9 presents the cumulative PSD. The emulsion samples were treated for 5, 7, 10, 12.5, 15, 20, 30, 45 and 60 minutes and their PSDs were compared with the PSD of the normal sample. The results show that the diameters of the particles decreased on an increase in sonication time. An explanation for this is that as the treatment time increased, the applied energy also increased leading to more intensive cavitation and the production of smaller particles. These differences were more obvious in the case of D10, D50 and D90, as seen in Table 4. This Table also demonstrates how the width of distribution became closer with increasing treatment time. 7 min sonication treatment gives approximately 85% reduction in D₅₀, 86% reduction in D₁₀ and 90% reduction in D₉₀, compared to the normal sample. In addition, the PSD became narrower by approximately 40% when compared with before sonication treatment.

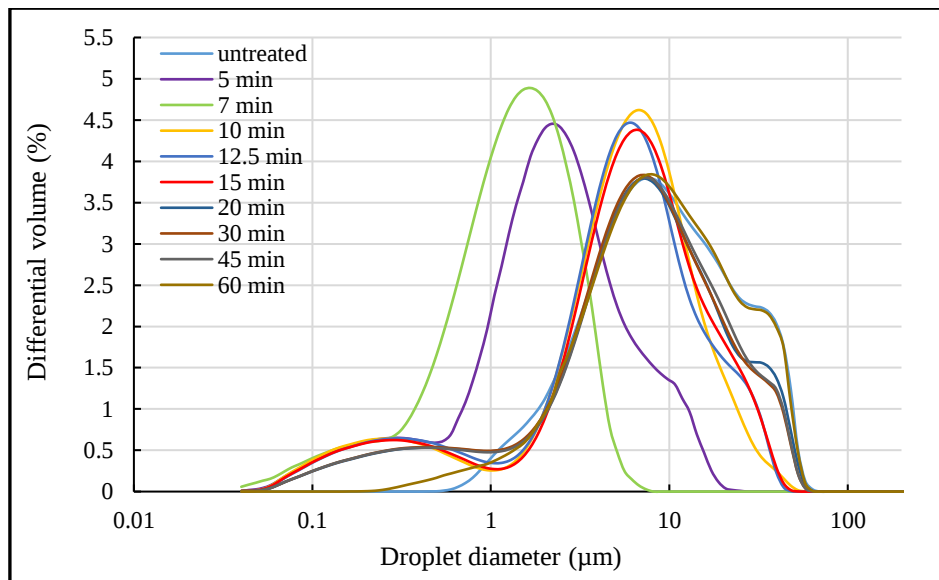


Fig. 8. Effect of sonication time on PSD.

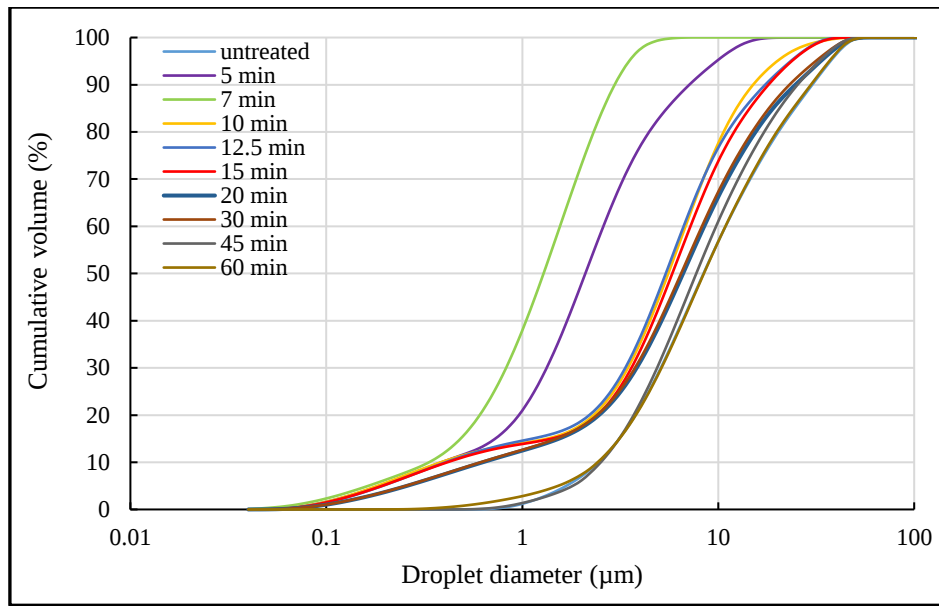


Fig. 9. Effect of sonication time on cumulative distribution.

Table 4. Effect of sonication time on PS.

<i>Treatment time (min.)</i>	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)	<i>Span</i>
0	2.423	8.146	30.068	3.393
5	0.375	2.207	7.421	3.192
7	0.341	1.261	2.919	2.044
10	0.375	5.609	15.651	2.723
12.5	0.412	5.609	17.181	2.989
15	0.412	6.158	18.861	2.995
20	0.598	6.760	24.951	3.602
30	0.598	6.760	24.951	3.602
45	2.423	7.421	27.391	3.364
60	2.423	8.943	30.068	3.091

However, this is apparently limited to a specific duration (7 min); after that, PS starts to increase. This phenomenon, where the particle size increases with an increase in input energy, can be referred to as “over-processing” as stated by [Desrumaux and Marcand \(2002\)](#). This can be attributed to poorer functioning of the emulsifiers and an increase in Brownian motion, hence a higher probability of collision and coalescence with higher energy input. In these conditions, the droplet size distribution of the emulsion, is a result of the competition between

two opposite processes; droplet disruption and drop-drop coalescence. A fresh interface is created whenever a droplet is formed from the disruption of an original droplet. Between its formation and its subsequent encounter with other droplets, some surfactant will be adsorbed onto this fresh interface. If the timescale of collision is shorter than the timescale of adsorption, the fresh interface of the newly formed droplets will not be fully covered with surfactant, such conditions leading to coalescence. Since the newly formed droplets are not completely covered by the surfactant, a higher rate of collision represents a greater coalescence rate, leading to an increase in the droplet size (over-processing).

From the PS and PSD results above, it is clear that 7 min represents the optimum time (threshold time) for sonication treatment of bitumen emulsion to achieve smaller PS and more uniform PSD.

5.4. INDIRECT TENSILE STIFFNESS MODULUS RESULTS (ITSM)

CBEMs made from LF and treated bitumen emulsion subject to a range of sonication times (5, 7, 10, 12.5, 15 and 20 minutes), were tested and compared with the control CBEM and HMAs at various curing ages. The results of ITSM testing for the prepared specimens after 3 days' age are presented in [Fig. 10](#), where 0 time refers to the control CBEM including untreated emulsion. The results show that 7 minutes' sonication treatment achieved the best ITSM value over other treatment times. The improvement in ITSM at 3 days of age for CBEM made from 7 minutes treated emulsion, is approximately 70% in comparison to the mix with the untreated emulsion at the same age. This improvement is most probably due to the greater specific surface area provided by smaller particles in the sonicated bitumen emulsion which achieved a better coating quality between the aggregates and bitumen binder. As the sonication time increased over 7 minutes, ITSM values decreased gradually. This can be attributed to recoalescence of the newly formed bitumen droplets due to them being insufficiently covered with surfactant. This also explains the increase in emulsion viscosity, as discussed in [Fig. 7](#).

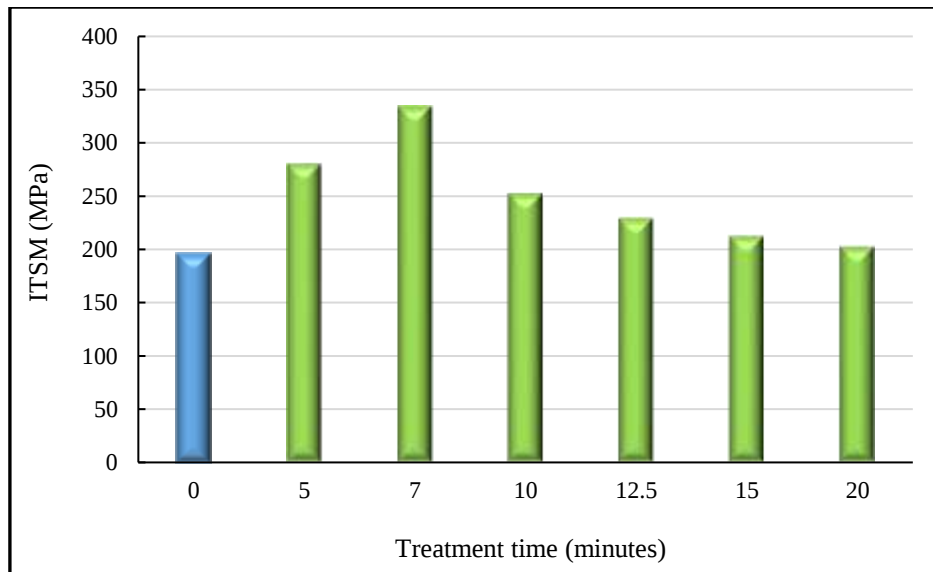


Fig. 10. Effect of sonication time on the ITSM of CBEMs at 3 days.

From the results above, it is clear that 7 minutes represents the optimum time (threshold time) for the sonication treatment of the bitumen emulsion used in this study. Accordingly, this treatment time was selected to prepare a modified bitumen emulsion with smaller PS and more uniform PSD, to be used to develop a new CBEM.

The CBEM with 7 minutes treated bitumen emulsion at different curing ages, was subjected to ITSM testing and compared with the control mixes, as seen in Fig. 11. Similar to the control CBEM with untreated bitumen emulsion, the 7 minutes treated CBEM gained the most improvement in ITSM during the first 28 days, compared to longer age spans.

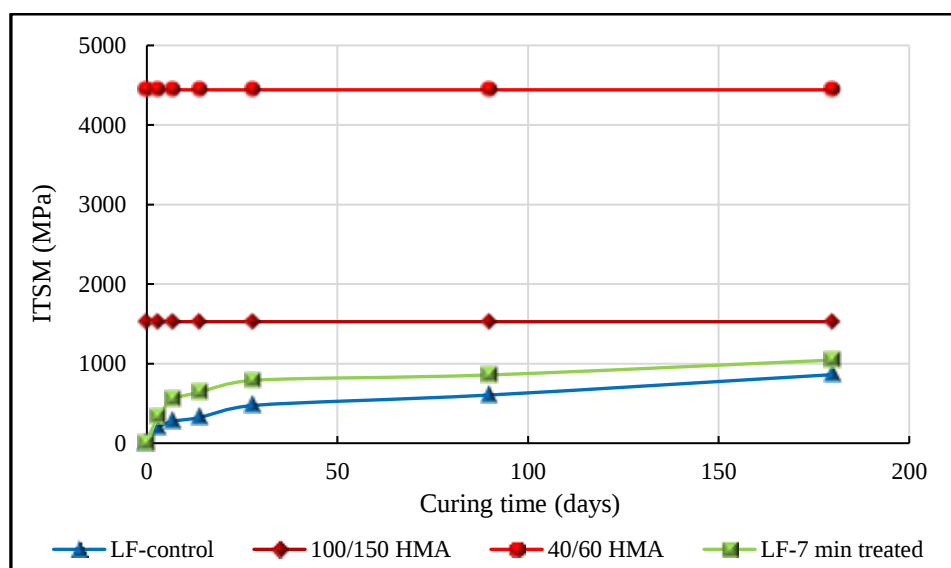


Fig. 11. ITSM values of 7 minutes treated CBEM with curing times.

6. CONCLUSIONS

1. The ultrasonic technique is very effective in reducing bitumen droplet size and the viscosity of the bitumen emulsion. This is apparent between 5 and 15 min time of sonication. The reduction in the viscosity at 7 min treatment, was approximately 28% compared to the untreated sample. This indicates that within this duration, the samples experience a reduction in the solution shearing resistance.
2. As sonication time increases (more than 7 min), the viscosity of the bitumen emulsion also increases. This can be attributed to the recoalescence of bitumen droplets because of over-processing.
3. The optimum sonication time was found to be 7 min. This resulted in an approximate 85% reduction in D_{50} , 86% reduction in D_{10} and 90% decrease in D_{90} . The PSD curve also become closer to the mean value and more uniformly distributed. This technology is providing a better coating quality and this has since been confirmed by the ITSM test.
4. There is an improvement in the ITSM at 7 min. treatment. The increment is 70% for 3 days, 100% for 7 days, 98% for 14 days and 66% for 28 days compared to the untreated emulsion.
5. This innovative technology appears to be very promising in producing a high-quality bitumen emulsion.

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