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Experimental study on the Microwave processing of waste tyre rubber aggregates to enhance their surface properties for their use in rubberised bituminous mixtures

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Abstract

This experimental study utilises the microwave technology to process the waste tyre rubber aggregates to enhance the interaction properties of waste rubber and bitumen by smoothing its surface. This was to make the waste rubber less reactive to the bitumen sample at the elevated temperatures of around 140-170°C. Parameters such as the microwave power, the height of the sample inside the processing chamber and the processing time have been considered and optimised. The results show that the microwave processing technique designed in this study has significantly smoothed the surface of waste rubber. Scanning Electron Microscope (SEM) surface analysis has been carried out to confirm the changes and smoothness of the rubber surface in comparison to the untreated waste rubber aggregates. In addition, Microwave spectroscopy analysis has been carried out to confirm the reduction in the absorption properties of waste rubber. The microwave curves of the bitumen samples extracted from the treated rubber-bitumen blend are comparable to the pure bitumen sample with minute shifts in the resonant peaks. The results show the potential of the technique and recommends designing a dedicated processing unit to further improve it through parameters such as volumetric and uniform surface treatment, penetration depth of the microwaves.

Keywords: Microwave processing; waste rubber aggregates; robust processing; microwave spectroscopy; microwave analysis; surface properties; rubber absorption properties.

1. Introduction

1.1. Background of the Scrap tyres arising

The tyres after their service life can be categorised as either 'partly-worn' or 'End-of-Life' tyres (ELTs) [1]. The partly-worn tyres can be reused after re-treading or re-processing, whereas, the ELTs are non-reusable waste and enters into waste management system for further treatment or disposition [1-4]. The introduction of legislations in 2006 and onwards that defines the landfill management, End-of-life (ELV) Vehicles salvage and waste incineration eliminate the option of landfill. The emphasis is on introducing new end use markets applications and sustainable recovery options for the waste tyres. Although the market for recycled tyres and its material has been established, specifically in wide range of civil engineering and industrial products and applications, the quantity of waste tyres arising worldwide still significantly exceeds the end use market for these materials. However, the UK is meeting its 100% recovery target through a combination of reuse, recycling and recovery options [5]. The waste rubber from scrap tyres is usually processed into crumb or powder rubber of various sizes to suit a particular application [5, 6].

1.2. Statement of the problem & brief literature

There is a tremendous increase over the last decade in the use of both the fine and coarse waste rubber aggregates as a binder modifier and within the bituminous mixture as aggregates for pavement engineering applications. This has improved engineering properties such as durability, fatigue resistance, skid resistance, deformation, etc. up to a certain extent. It has also positively impacted the environment in terms of both reducing the surface noise pollution as well as the threat posed by the scrap tyres to the environment [7]. However, research [8-16] has shown that to achieve the durability and long-term performance of the rubberised bituminous mixtures the key parameter to consider is the bitumen-rubber interaction. This interaction of bitumen-rubber results in two mechanisms, namely; particle swelling and dissolution. The swelling takes place when bitumen is partly absorbed by the rubber particles. This is due to the migration of light fractions (aromatic oils, resins and saturates). This absorption results in a physical reaction than chemical reactions and the rubber particles swells two to three times its original size. In addition to rubber swelling, dissolution is caused by the depolymerisation and devulcanisation of the rubber at high mixing temperatures of around 140-170°C. Hence, the adhesion and bonding between the

bitumen and rubber particles break. The causes loose particles of rubber being distributed and dispersed in the bitumen in the case of fine particles and on the surface of pavements in the case of crumb rubber [7, 15, 16].

It was found from the literature that properties such as surface roughness, hydrophobicity, adhesion and cohesion are some of the important properties for waste rubber to show better interaction effect with bitumen. Large number of treatment methods have been utilised to improve these properties. Some of these include the use of Sodium Hydroxide (NaOH) saturated aqueous solution [17], use of epoxy and polyurethane adhesives [18], chlorination of waste rubber [19], graft polymerisation [20], Ultraviolet (UV) surface modification [21], Partial modification by oxidation process [22], Plasma treatment using oxygen and microwave plasma at atmospheric and low pressure [23, 24], bio-process using Thiophilic micro-organisms [25], chemical modification using carbon black [26], etc. However, none of the techniques were focused on controlling the absorption properties of waste rubber aggregates. In addition all of these modifications of the surface properties of rubber was carried out for its applications other than in rubberised bituminous mixtures & pavements.

2. Proposed waste rubber aggregates treatment

This experimental study was focused on utilising an extensive novel microwave processing of the surface of waste rubber aggregates by controlling various parameters. The aim was to reduce the absorption properties of rubber through surface modification. Studies [13, 27, 28] have shown that smoothening the surface of waste rubber can be helpful in reducing the reactive properties of rubber. This surface change property of waste rubber, if utilised effectively, can be used to reduce the absorption of the light fractions from bitumen to rubber at elevated mixing temperature of around 140-170 °C. Hence, the focus of this research paper was to process the coarse waste rubber aggregates to smooth their surface that would make it less reactive to the bitumen at elevated temperatures. The potential shown by the outcome of the treatment could have significant impact on improving the waste rubber and bitumen properties as well as the service life of pavements. The surface properties of the processed waste rubber was analysed and tested using Scanning Electron Microscope (SEM). In addition to the surface properties, the interaction properties of the modified rubber with the bitumen was also studied using a novel Microwave Spectroscopy (MS) technique. The analysis of the bitumen was also carried out after its reaction with the treated waste rubber to study changes

in the bitumen properties (due to migration of light fractions). This was to indirectly analyse the absorption properties of the treated rubber.

3. Materials and methods

3.1. Material & sample preparation

The crumb rubber of the size 2-6 mm was used and supplied by H. Allcock & sons based in Manchester, UK. The rubber was recycled from trucks scrap tyres and was mechanically shredded to the desired size by the company. The waste crumb rubber is shown in Fig. 1.



Fig. 1. Crumb rubber of the size 2-6 mm obtained from truck tyres

The sample preparation was carried out by weighing up to 15 grams of waste rubber sample in a petri dish at a time for processing. Beakers were used to process the sample in the microwave processing chamber.

4. Microwave processing theory

At present there is a need of a cutting edge technology to process the engineering materials. There is a demand of a new and improved processing method that can yield better control and is more efficient [29]. This research article has proposed a novel treatment method based on the microwave processing technology. Microwaves (MW) are a part of Electromagnetic (EM) Spectrum and cover the frequency range of 300 MHz to 300 GHz within the spectrum. They are the alternating current signals with an electrical wavelength of 1 metre to 1 mm [30]. In microwave heating & processing of materials, the heat is injected through the surface. The

heating of the material depends on it being neither a perfect conductor nor a perfect insulator [31].

In conventional heating/processing of materials, the heat is transferred into the surface and then into the material based on the conduction, convection and radiation phenomena. In comparison, the microwave heating and processing is carried out at the molecular level providing volumetric heating in the processed component. The efficiency of the processing depends on the physical properties of the material [29]. Materials can be categorised into three main groups, namely; i) the conductor or opaque materials that reflects back the microwaves and do not allow them to penetrate through the material ii) Insulators or transparent materials that allow the microwaves to pass through them without having any interaction with them, and iii). The absorbers that are categorised as high dielectric loss materials and absorb microwaves up to a certain degree depending on the dielectric loss factor [29, 32, 33]. The properties that cause the absorption of microwaves by the material includes complex relative permittivity ε^* and loss tangent $\tan\delta$ represented by the equations 1 and 2 below:

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \quad \text{Equation 1}$$

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \quad \text{Equation 2}$$

Where ε' and ε'' in Equation 1 signifies the dielectric constant and dielectric loss of the material that defines the capacitive and conductive components of the dielectric response. The $\tan\delta$ in Equation 2 defines the ability of the material to convert microwave energy into heat [34, 29].

5. Experimental design & setup

A commercial microwave oven, model MHO/340 with a stainless steel cavity operating at 2.45 GHz was utilised for the treatment of waste rubber. It was a conventional style microwave with a cavity/chamber, door and upper panel controls. To gain more control for the processing of materials, additional controls were embedded to provide range of functionalities. This include variable 0-1 kW power control, infrared temperature detector as well as process control loop. The microwave processing system and the front control panel is shown in Fig. 2 and 3 respectively.

Since the aim of the microwave treatment was to smooth the surface of crumb rubber reducing its absorption properties, the following was the procedure and parameters used.

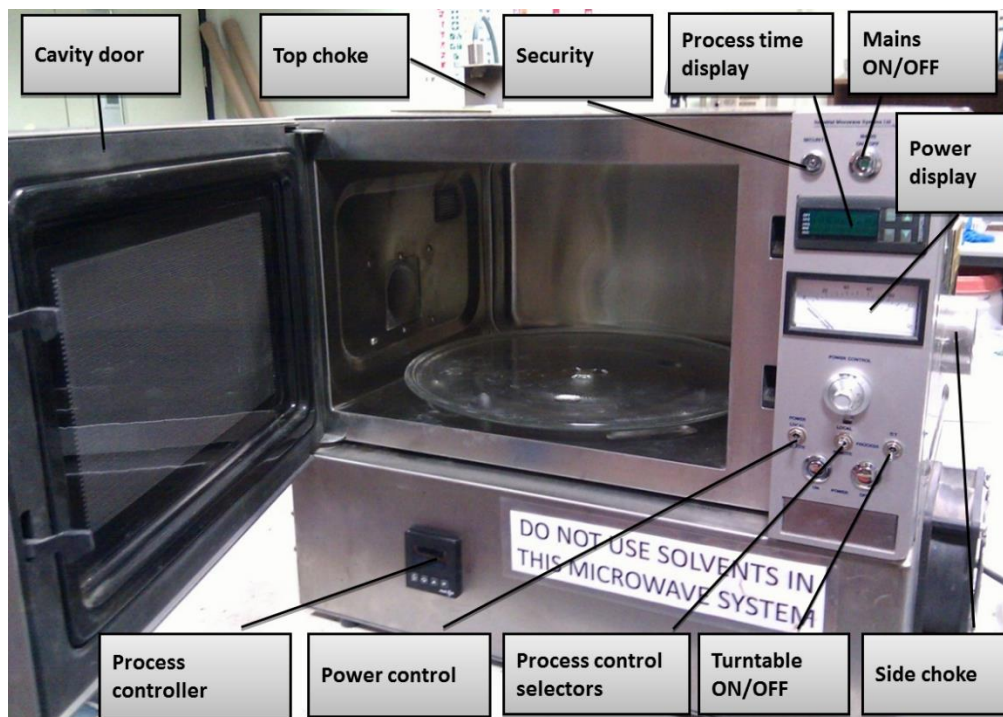


Fig. 2. Industrial microwave unit for material processing



Fig. 3. Front panel control and display of the microwave system

- Five different power levels were selected to find out the effective power for the treatment. These included 1 kW (100%), 750 W (75%), 500 W (50%), 375 W (37.5%) and 250 W (25%) of power.
- With each power level, five different height levels were selected. These included sample on the turntable, 4.3 cm high, 5 cm high, 6 cm high and 8.5 cm high. The height were selected to find the effect of the distance of the sample from the radiation source. This was to obtain the optimum combination of power with the height factor.
- The effect of the above parameters on the sample temperature was observed. It was useful to find out the maximum bearing temperature of rubber aggregates.
- Treatment time for the combination of given power and height without being adversely affected by the microwave radiations. The time range was between 0-80 minutes to monitor the effect of short-to-long term processing of rubber sample and its impact on the surface properties. Specifically, it was interesting to see this impact in the case of low power processing.
- The weight of the sample before and after treatment was also carried out to observe the change in waste rubber aggregates.

Graphical depiction of the generalised treatment process is shown in Fig. 4.

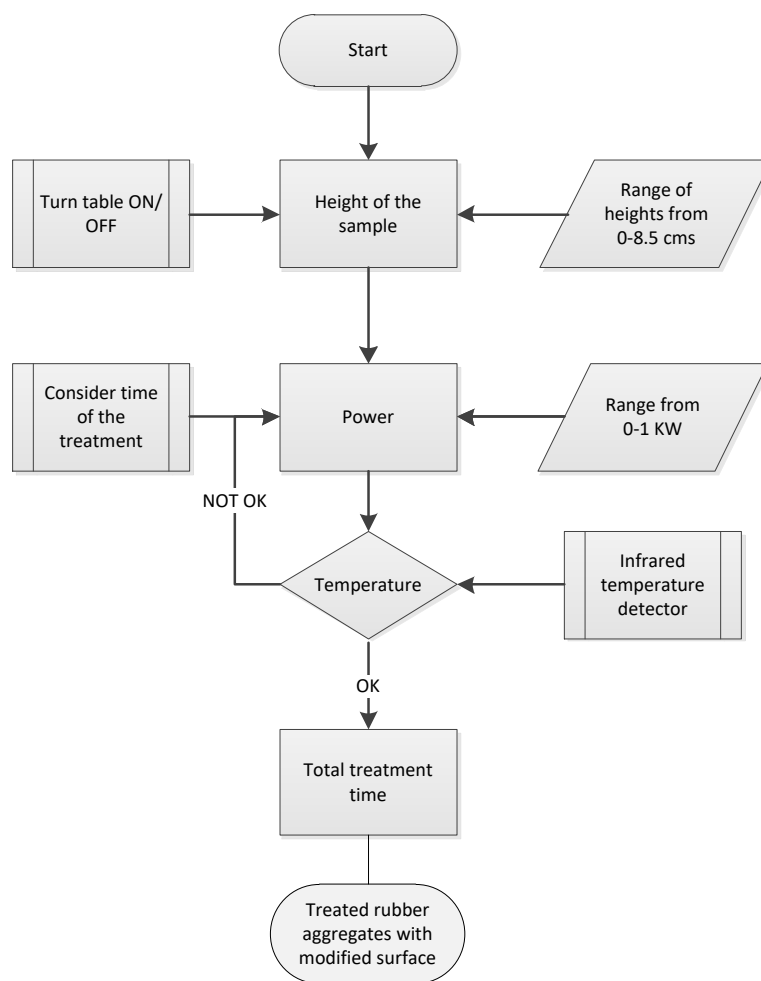


Fig. 4. Graphical representation of the microwave processing/treatment of the waste rubber surface

To illustrate how the processing was carried out inside the microwave system, two of the samples inside the microwave system is shown in Fig. 5.



Fig. 5. Two samples of varying heights (a) on the turntable (b) 8.5 cm higher on the turntable

6. Results and discussions

The microwave treatment of waste rubber was carried out in phases. This was required because the industrial microwave system used doesn't have a capability to focus the microwave energy at a single point. The first phase was to determine the suitable heights out of the samples in combination with the power levels used and the processing times. Optimum values of the above were chosen based on the careful inspection and analysis of the treated surface of the rubber to monitor the smoothness of the surface. Other factors such as weight of the sample before and after treatment, temperature of the samples were also considered. The final treatment was carried out with the following parameters.

- Power level of 500 W with sample on the turntable and 6 cm high.
- Power level of 750 W with sample on the turntable, 6 cm high and 8.5 cm high.
- Processing times between 7 and 80 minutes, whereby up to 50 minutes was used for the 750 W and up to 80 minutes was used for the 50 W treated samples.

All the processing was repeated to verify the results and the effect of the parameters. The SEM analysis was carried out to study the properties of treated surface in comparison to the untreated rubber. In addition to the surface analysis, a novel Microwave spectroscopy analysis technique was also used to study the effect of the treatment on the absorption properties of waste rubber. This was verified by analysing various samples of binders from the bitumen and treated/untreated rubber mixes. Microwave spectroscopy analysed the dielectric properties of binder samples extracted from the bitumen-treated/untreated rubber mixes to indirectly monitor changes in the absorption properties of waste rubber aggregates. Results of the sample placed on the turntable and at a height of 8.5 cm inside the microwave processing unit with the processing power of 500 and 750 W respectively is discussed below.

6.1. SEM results and discussion

SEM analysis was carried out to examine the surface of the untreated and treated waste rubber aggregates at approximately 3500x magnification. After examining the results of the SEM analysis of all the samples using varying microwave power, heights inside the cavity of the microwave oven and processing times, promising results were achieved with:

- 500 W of microwave power with the sample on the turntable and processed for approximately 80 minutes.
- 750 W of microwave power with the sample placed at the height of 8.5 cm inside the chamber of the industrial microwave oven. The processing was carried out for approximately 50 minutes.

The SEM micrograph of the untreated waste rubber is presented in Fig. 6. The results of the processed rubber aggregates with the 500 W and 750 W power is shown in Fig. 7 and 8 respectively for the comparison purposes.

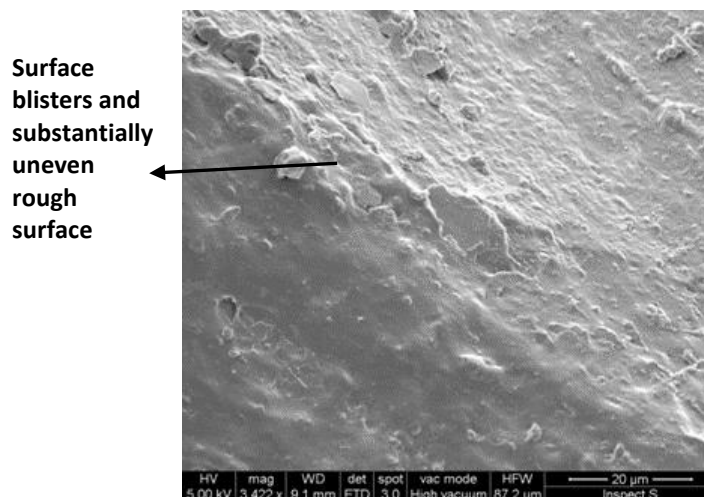


Fig. 6. SEM surface micrograph of the untreated crumb rubber at approximately 3500x magnification

- The untreated waste rubber surface appears to be uneven along with a lot of roughness on the surface.
- The surface also appears to be blistered as indicated in the graph.

In Fig. 7, the waste rubber is processed using 500W of microwave power and the sample placed on the turntable inside the cavity of the microwave oven. Fig. 8 on the other hand shows the micrograph of the processed rubber using 750 W of microwave power along with the sample height of 8.5 cm and the processing time of around 50 minutes.

Uniform flat
and
smoothened
surface with
reduced
blistering

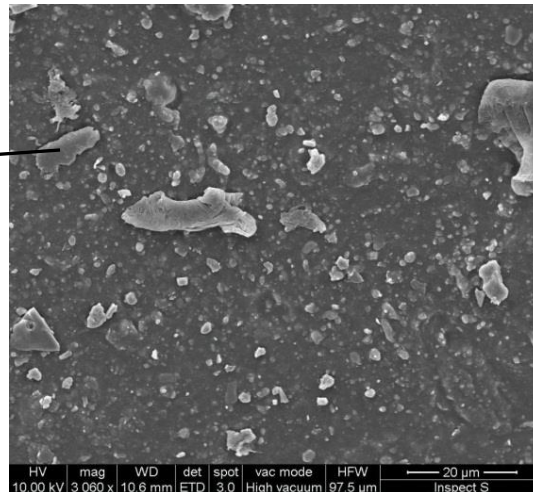


Fig. 7. SEM surface micrograph of the treated crumb rubber at approximately 3500x magnification using 500W of microwave power, on the turntable and processed for 80 minutes

In comparison to the Fig. 6 showing the untreated waste rubber surface, both the Fig. 7 and 8 shows that:

- Waste rubber treatment with Microwave radiations had a significant effect on the texture of the surface. The surface appears to be more uniform without any ridges and smoothened surface area as well as patches.
- Small number of blisters can be seen on the surface in the case of both the low and high power microwave processing. However, this is more in the case of 500 W processing compared to the 750 W microwave processing. Also, the sample processed with higher power was closer to microwave source in comparison to the waste rubber treated with low power and away from the source. This shows that high power microwave processing had a significant positive impact in comparison to the low power microwave processing on the surface of waste rubber.
- The results shows that microwave has significantly changed the surface's physical properties. The absorption affect needed to be verified and Microwave spectroscopy analysis was carried out for this purpose, the results of which are presented in the next section.
- The SEM micrographs also shows that the microwave processing technique does have a potential to smooth the surface. This process can be significantly improved if a more dedicated waveguide is designed to apply microwave on the waste rubber surface.

This will also help in controlling the parameters such as penetration depth of microwave radiation into the waste rubber surface, uniform volumetric heating, etc.

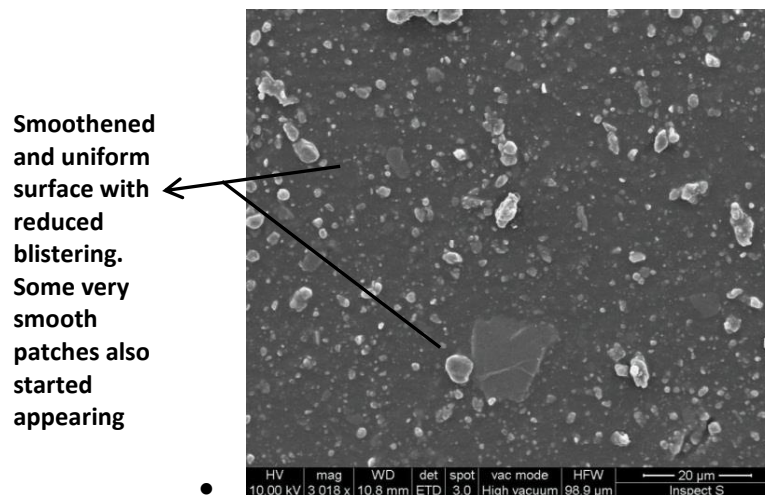


Fig. 8. SEM surface micrograph of the treated crumb rubber at approximately 3500x magnification using 750 W of microwave power, the height of 8.5 cm and processed for 50 minutes

6.2. Microwave spectroscopy analysis of the bitumen

To further analyse the effectiveness of the microwave surface enhancement, microwave spectroscopy analysis was utilised. Bitumen samples were analysed in their pure form, cured and un-cured form, extracted from the rubber-bitumen mixes with and without a treated rubber. Microwave analysis technique has been successfully utilised in previous studies carried out by the same author [7, 35, 36] to show its effectiveness in assessing the properties of bitumen with and without its interaction with rubber. It showed promising results in terms of its accuracy, repeatability and validity, hence used in this study. The curing was considered because it is an important factor to analyse the effectiveness of the microwave surface processing over a longer period of time when the bitumen and rubber are left after mixing. This is to see how much of the swelling of waste rubber aggregates can take place after the treatment. If the treatment is not very effective, there would be a significant shift of the microwave spectrum away from the pure bitumen sample after the curing of the treated rubber and bitumen blend. Curing is important and is usually the case in real world situations where the mix is left at an elevated temperature for up to 6 hours before its application to the pavement surface. The analysis also made the experimental study innovative and novel in terms of both the processing and analysis.

Fig. 9 shows the case of microwave treated rubber with 500 W of microwave power. The Figure shows two spectrums of pure bitumen (with and without 6 hours curing), two spectrums of bitumen from the mix of pure (untreated) rubber-bitumen blend mixed for 1 hour and further cured for 6 hours respectively, bitumen from the mix of treated rubber-bitumen blend kept for 1 hour and cured for 6 hours. The results are very interesting and can be summarised as follows:

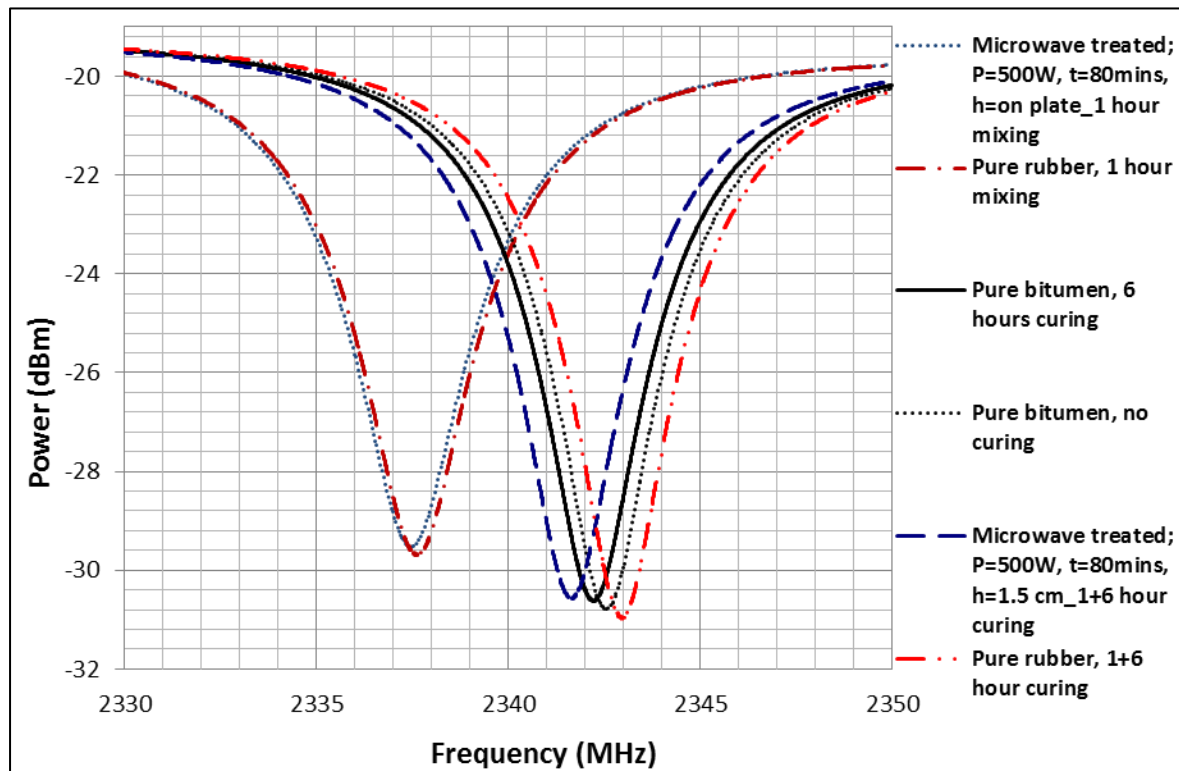


Fig. 9. Microwave spectrums of bitumen samples after the Microwave surface treatment of waste rubber aggregates; $P = 500 \text{ W}$, $H = \text{on turntable}$, treatment time = 80 mins, curves after 1 hour mixing and further 6 hours curing of rubber-bitumen blends (10:100)

- The microwave resonant peaks for the bitumen with and without curing were observed at the frequency of around 2.3421 GHz and 2.3425 GHz respectively.
- When comparing the bitumen spectrums with the spectrums of bitumen from the untreated (pure) rubber-bitumen blend interesting trend was observed. The peak of the bitumen extracted and analysed after 1 hour is at 2.3376 GHz which is a shift of approximately 45 MHz and 49 MHz to the left from the cured and un-cured pure bitumen samples. The significant and sudden shift in the frequency could be attributed to the fast changes that took place within 1 hour of mixing, possibly in the light fractions of bitumen as well as the absorption of light fractions into rubber from

bitumen. The absorption was not stable in the first hour and hence the abrupt change observed in the microwave spectrum.

- After an additional 6 hours of curing, the bitumen extracted from the untreated rubber-bitumen blend shows a shift back to the right of the pure bitumen signatures. This shows the stability achieved after a long term interaction between the two. The peak is observed at around 2.3430 GHz. It is a shift of around 5 MHz and 9 MHz from the un-cured and cured bitumen respectively. This shift clearly showed the measureable decrease in the dielectric property of the binder after 6 hours of curing in comparison to the pure bitumen samples. Since, the curing was carried out at a temperature of 160-170°C, the light fractions would be affected and stabilised which is represented by the decrease in the dielectric value.
- Similar trend in the shifts was observed when bitumen samples were extracted and analysed from the mix of treated rubber and bitumen blend mixed for 1 hour and further cured for 6 hours. It can be seen that the bitumen mixed for 1 hour in the treated rubber mix has a frequency peak identified at approximately 2.3374 GHz which is a significant shift to the left of the pure bitumen sample. It is also to the left of the bitumen sample from the untreated rubber-bitumen blend. However, when the mixing conditions were stabilised the microwave spectrum shifted back towards the right closer to the pure bitumen. The peak was observed at approximately 2.3416 GHz. This peak is still to the left of both the pure bitumen samples and the bitumen sample from the untreated rubber-bitumen blend. The stability shows that the migration significantly reduced after an initial absorption and could be attributed to the surface enhancement of the waste rubber.
- The shift observed in the curve of the bitumen from the 6 hours cured treated rubber and bitumen blend is at around 5 MHz from the cured bitumen sample and 9 MHz from the un-cured pure bitumen sample. Also, the shift is around 14 MHz to the left from the bitumen off the cured untreated rubber-bitumen blend.
- The microwave spectroscopy showed that there is a measureable and significant difference between the spectrums of various samples. Especially the shift is significant between the bitumen from the untreated rubber-bitumen and treated rubber-bitumen blend. The spectrum of the treated sample is reasonably closer to the

bitumen samples. This shows the change in the absorption properties of the treated rubber. Effectively the shift to the left shows increase in the dielectric properties. In other words higher light fractions.

- The results demonstrated the potential of using microwave processing technology to enhance the waste rubber surface properties and to reduce their absorption properties.

Fig. 10 shows the results of the treatment using 750 W of microwave power. The spectrums again show the cured and uncured bitumen samples. A detailed analysis of Fig. 10 shows that:

- The results of the microwave spectroscopy analysis of all the samples are pretty consistent with the results in Fig. 9.
- The resonant peaks of the pure bitumen samples (cured and uncured) are at 2.3421 and 2.3425 GHz respectively. These values are very consistent with the curves obtained in Fig. 9. This verifies that the results from the microwave spectroscopy measurements are reasonably accurate and repeatable.
- In comparison to the pure bitumen samples, the bitumen sample extracted from the untreated rubber-bitumen blend, mixed for 1 hour, was analysed and the resonant peak was identified to the left of the pure bitumen spectrums. The peak is at 2.3377 GHz which represents a shift of around 44 and 48 MHz from the cured and uncured bitumen samples respectively.

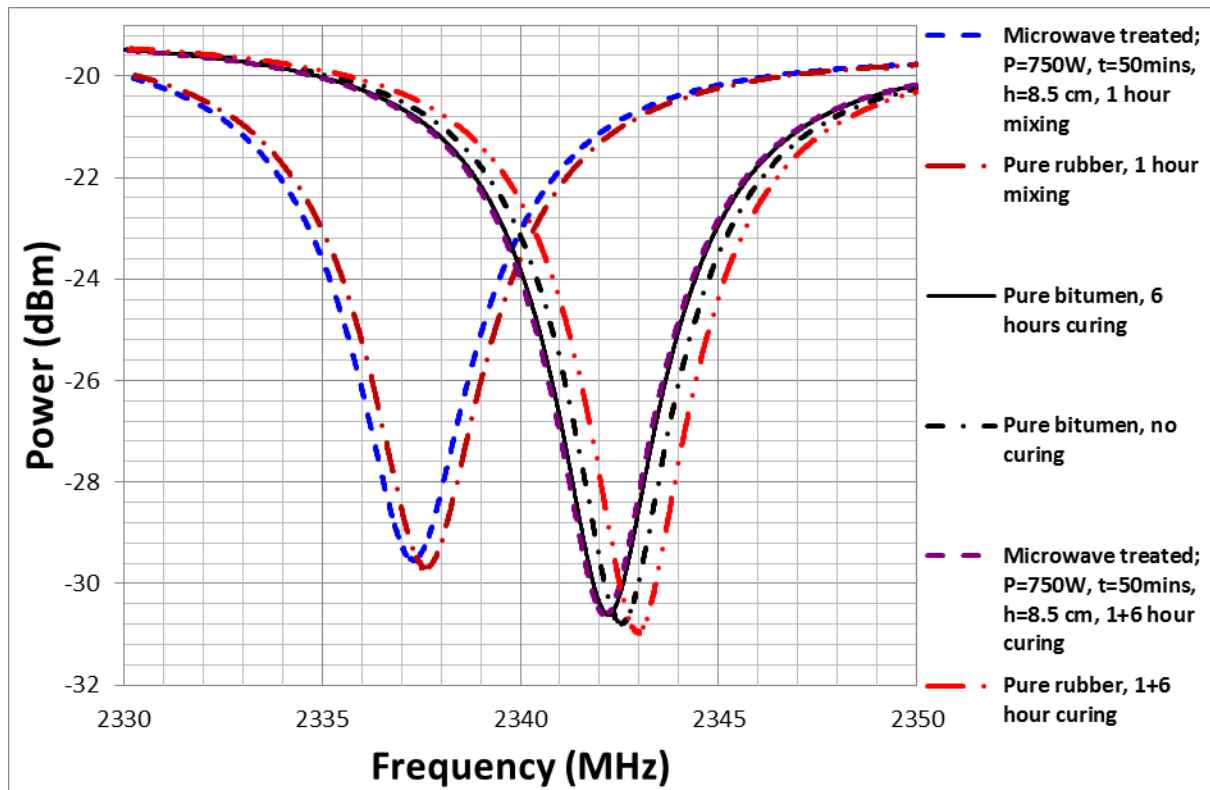


Fig. 10. Microwave spectrums of bitumen samples after the Microwave surface treatment of waste rubber aggregates; $P = 750 \text{ W}$, $H = 8.5 \text{ cm}$, treatment time = 50 mins, after 1 hour and further 6 hours curing of rubber-bitumen blends (10:100)

- The 6 hours cured sample of the bitumen from the untreated rubber-bitumen blend has its resonant peak shifted to the right of the pure bitumen samples. It is identified at the frequency of around 2.3430 GHz, similar to Fig. 9. This is approximately a shift of 5 and 9 MHz from the uncured and cured bitumen samples respectively.
- When comparing the spectrum of the bitumen from the treated rubber and bitumen blend, both after 1 hour mixing and 6 hours curing the peaks are found at approximately 2.3372 and 2.3420 GHz respectively. The latter is the peak of the cured sample which is almost overlapping the cured pure bitumen sample which is at 2.3421 GHz.
- The results of both the 1 hour mixed and 6 hours cured bitumen samples from the treated rubber blends show similar behaviour as in Fig. 9. However, the processing/treatment of the rubber surface seems to be more successful. This is demonstrable from the results in Fig. 10 where the spectrum almost overlap the pure bitumen sample showing similar attributes in terms of the properties of bitumen.

- The overlap of the spectrum also aligns with the SEM analysis of the processed rubber surface in Fig. 8 which shows improvement to the surface of the high power microwave processed rubber in comparison to the low power processed as well as the untreated rubber in Fig. 7 & 6 respectively. The results show reduced absorption properties and bitumen exhibiting similar properties to the pure bitumen after 6 hours of curing.
- The consistency and repeatability of the microwave spectroscopy analysis technique to indirectly assess the processed rubber absorption properties from the extract of the rubber-bitumen blend is promising, as in Fig. 9 and 10. All the bitumen samples analysed are showing repeatable trends. The only change observed is in the bitumen spectrum of the sample extracted from the treated rubber and bitumen blend. It shifted in Fig. 10 in comparison to Fig. 9 due to the change in the microwave processing parameters. This clearly shows the impact of the processing parameters on the surface and absorption properties of the crumb rubber. It is also useful to further investigate and optimise the microwave treatment technology in terms of controlled processing of the surface of waste rubber aggregates.

7. Conclusions and recommendations

Waste tyre rubber aggregates of size 2-6 mm were processed using a microwave processing system at the standard 2.45 GHz frequency. Various microwave powers were used to treat the rubber surface in combination with other parameters to see the effect of the microwave processing on the surface properties of rubber aggregates. The results showed some promising improvement in terms of changing the surface of the crumb rubber. Although, the microwave processing method utilised didn't use a controlled microwave radiation system such as a microwave reactor to control the processing depth, extensive set of experiments with important parameters such as height of the sample inside the microwave chamber, processing time and microwave power helped in identifying efficient processing parameters to obtain desirable surface changes in rubber. The results showed a potential to utilise microwave radiations and encourage to further develop a dedicated microwave processing system. The following are briefly some of the conclusions from the processing experimentation and results:

- The SEM results and microwave spectroscopy analysis shows that the waste rubber treated with 750 W of microwave power at a height of 8.5 cm for 50 minutes reasonably changed waste rubber surface properties to achieve the desirable absorption properties. These results are presented in Fig. 8 and 10 respectively.
- The processing enhanced the surface appearance as in Fig. 8 and smoothened the waste rubber surface up to a measureable degree by fine tuning the processing parameters.
- The absorption properties of waste rubber were measured indirectly through the analysis of bitumen extracted from the untreated rubber and bitumen blend as well as the treated rubber and bitumen blends. The extraction was carried out at two occasions, i.e. after 1 hour of mixing and 6 hours of further curing of the blend. It was to see both the short and long term absorption of light fractions of bitumen into rubber. The key parameter to study was to compare the microwave spectrum of the treated bitumen sample to the pure bitumen curve to observe how close the spectrums are. The results showed the reduction in the migration of the light fractions into rubber surface as the properties were similar to the pure bitumen in terms of the spectrum obtained.
- The result for the microwave spectroscopy analysis demonstrated the effectiveness of the treatment. However, knowing the potential of the technique it is proposed that this processing method can be improved further in terms of the treatment time and uniformity of surface processing.

It is recommended that the current measurements results shows a potential of further development of this technique to substantially improve the processing time as well as the quality of the processing. Further research work in the development of the dedicated processing system is recommended. This could be in the form of a microwave reactor with controlled depth of surface processing and improved volumetric processing along with a reduced treatment time.

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