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DEVULCANIZATION AND CHARACTERIZATION OF GROUND TIRE RUBBER WITH MICROWAVE METHOD

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ABSTRACT

The increase in transportation vehicles worldwide has also led to an increase in the production of tires, which are an important part of the vehicle and are used in these vehicles. Industrially, vehicle tires are produced through a process called vulcanization and acquire their high elasticity in this way. However, the sulfur crosslinks formed by the vulcanization process make it very difficult to recycle tire rubber. In the evaluation of vehicle tires that become waste after their service life is completed, direct evaluation, use as an energy source and pyrolysis are used in recycling methods. However, none of these methods aim to recycle tire rubber as a material, that is, to turn it into a re-moldable rubber. In recent years, studies have stated that devulcanization, that is, the breaking of crosslinks, is possible with thermal, mechanical and chemical effects.

In this study, ground tire rubber was devulcanized by the microwave method. The aim of this study is to examine the effectiveness of the process by characterizing the ground tire rubber exposed to different microwave durations. For this purpose, the characterization of devulcanized rubber was carried out by soluble fractions, crosslink densities, FTIR analyses and SEM imaging.

Keywords: Rubber, Vulcanization, Waste vehicle tires, Microwave devulcanization, Recycling.

1 INTRODUCTION

The increasing usage rate of vehicles leads to an increase in tire production and therefore waste tires. Approximately 2.47 billion tires were sold globally in 2023 [1].

Natural and synthetic rubbers are used as the main material in tire production. As in other rubber products, the high elastic properties expected from vehicle tires are obtained due to the sulfur crosslinks formed in the rubber with the vulcanization process. Vulcanization can be defined as the formation of a three-dimensional crosslinked network structure between rubber macromolecules with the help of sulfur, activators, accelerators and other additives as a curing agent and with the effect of heat (Figure 1) [2], [3]. However, these crosslinks prevent rubber-based products from being melted and recycled or remolded by being transformed into another way after their service life has ended [4], [5].

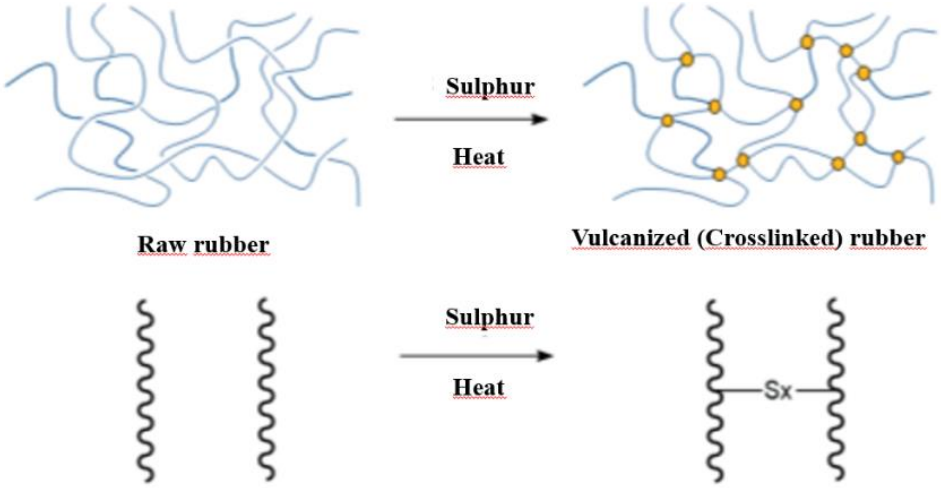


Figure 1. Sulphur crosslinks in vulcanized rubber [3].

Disposal of end-of-life tires is both an environmental and economic problem all over the world. Leaving waste tires in nature in an unorganized manner causes fires, insect reproduction, and environmental pollution with chemicals leaking from their structure [6]-[8]. In recent years, increasing environmental sensitivity and the search for efficient use of raw material resources have led to legal regulations all over the world. In this context, the Waste Tire Regulation was published in Turkey in 2006 and the processes were defined for the collection and evaluation of waste tires [9].

There are various methods currently applied all over the world for the evaluation and recovery of waste tires. These include using granulated tire rubber as a ground covering in parks, burning it as an energy source in factories, or separating it into various raw materials through the pyrolysis method [10]-[12]. However, since the amount of waste is very large, the recovery of waste rubber products as a reusable rubber material should be the basis. This is possible with devulcanization, which is the reverse of vulcanization. In devulcanization, it is

aimed to break the sulfur crosslinks formed by vulcanization without damaging the polymer main chain structure, thus making the waste rubber remoldable (revulcanization). It is seen that devulcanization is a subject on which many studies have been conducted and is still being worked on intensively. There are methods developed and studied on devulcanization such as chemical, mechano-chemical, thermomechanical, ultrasonic, microwave and biological devulcanization. [13]-[16]. Among these methods, microwave and ultrasonic devulcanization stand out in terms of their potential to be used on an industrial scale and the quality of the devulcanized rubber obtained, provided that the process parameters are controlled [12],]17].

In the microwave technique, the energy required to break down the C-S and S-S bonds in vulcanized rubber is provided by using microwave energy at a certain frequency and level. For the microwave technique to be applied, there must be polar components in the structure. Heating is provided by dipole rotation under the influence of microwave energy. Rubber materials are essentially apolar, but heating occurs due to the polar structure of the carbon black in their structure [18].

In this study, an experimental study was carried out for the recycling of waste vehicle tires, which is very important in terms of both environmental effects and raw material recovery by microwave devulcanization method. To determine the properties of devulcanized rubber obtained from devulcanized waste vehicle tires and to demonstrate the effectiveness of devulcanization, it was aimed to determine the parameters where high rates of devulcanization were obtained by soluble fractions, crosslink densities, SEM and FTIR analyses.

2 MATERIAL AND METHOD

The study was carried out in the form of supply of ground tire rubber, characterization of the powder, microwave devulcanization studies and characterization of devulcanized rubber.

2.1 Supply and Characterization of Ground Tire Rubber

In the study, ground tire rubber (GTR) in the size range of 0-1 mm, produced by mechanically shredding waste truck tire at room temperature, supplied by Unsal Rubber Co. Ltd (Adana, Turkey)., was used. Powder size measurement, thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR), scanning electron microscope (SEM) analyses were performed to characterize the untreated powder.

2.1.1 Particle Size Measurement

The size measurement of the rubber powder was made by sieve analysis method according to ASTM D 5644-01. This analysis was made by vibratory sieve shaker in Aksaray University Civil Engineering Laboratory. For size measurement, 100 g sample was passed through vibrating sieve for 10 minutes and the obtained results are given in Figure 2. According to the sieve analysis results, it is seen that the particle size of ground tire rubber is mostly in the range of 0.45-1 mm and 0.25-0.45 mm.

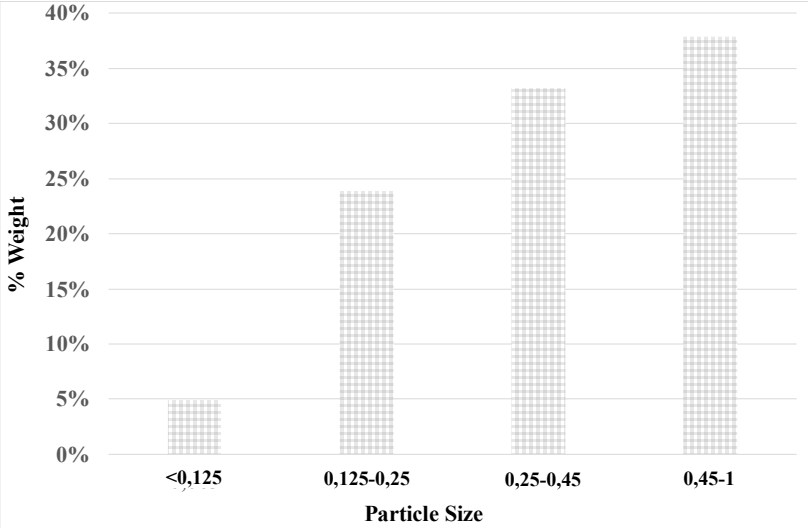


Figure 2. Particle size distribution of ground tire rubber.

In the studies, it has been stated that the small sample size has a positive effect on increasing the homogeneity of devulcanization and that better mechanical properties are obtained after revulcanization [5], [19]-[23].

2.1.2 SEM Imaging

SEM images of the ground tire rubber used have been given in Figure 3. It is seen that it consists of particles of varying sizes and has a rough surface structure. This shows that it was obtained through mechanical disintegration at room temperature.

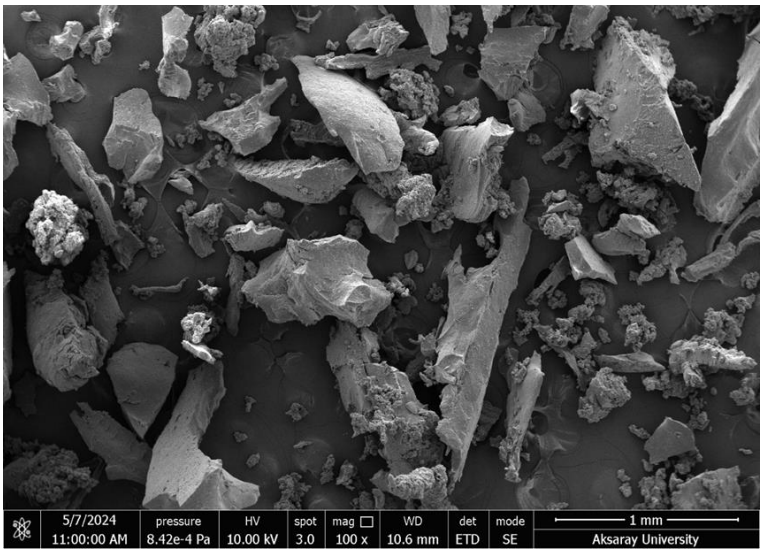


Figure 3. SEM image of used ground tire rubber.

2.1.3 Thermogravimetric Analysis

With TGA, the change in the mass of a material is measured depending on temperature or time. As a result of the chemical reaction caused by temperature, the material decomposes and a gradual change in its mass occurs. As a result of this change, both the thermal behavior of the material is characterized and an idea about the material composition is obtained. In the study, thermogravimetric analysis of ground tire rubber was performed in accordance with the ASTM E1131 standard, by heating approximately 10 mg of the sample in the Aksaray University Central Research Laboratory (ASUBTAM) at a heating rate of 10 °C/min in a nitrogen atmosphere to a temperature of 800 °C.

The TGA/DTG curves of ground tire rubber are given in Figure 4. The peak at 382.6 °C indicates the presence of polymer in GTR and its decomposition temperature. According to the results obtained from the TGA analysis, it is seen that the first mass loss starts around 165 °C, and between 165–300 °C, the mass decreases due to the decomposition of oils and other non-polymeric additives added to the rubber material as process facilitators. This mass loss was determined to be approximately %9.3. The mass loss accelerated between 300–500 °C due to the decomposition of polymeric phases in rubber. It is seen that the curve becomes horizontal where the mass loss decreases above 500 °C. In the TGA curve, it is seen that the GTR composition consists of %54.2 polymer and %35.8 carbon black and the non-decomposed part (consisting of ash and other inorganic additives).

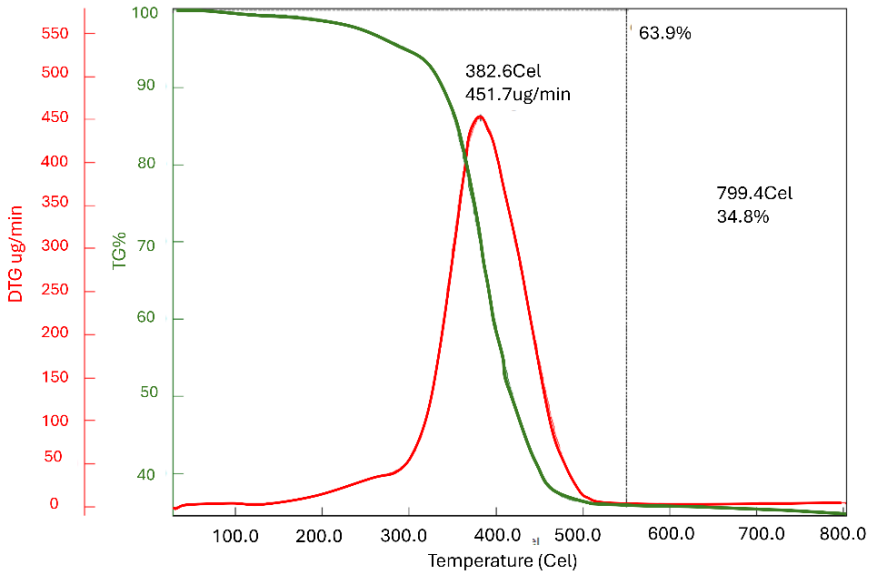


Figure 4. TGA analysis of used ground tire rubber.

In this study, TGA analysis of ground tire rubber was performed not only to characterize the material but also to determine the appropriate temperature and time for microwave devulcanization. The composition of the ground tire rubber used as a result of the interpretation of the TGA curves is given in Table 1.

Table 1. Composition of GTR (%wt).

Sample	Process oil, humidity and other additives %	Rubber %	Carbon black and ash %
GTR	9.7	54.2	36.1

2.2 Microwave Devulcanization

For the devulcanization process, a Samsung brand household type microwave oven operating in the 300-800 W power range has been used (Figure 5). The material was placed in a 1000 ml glass container inside the oven.



Figure 5. Microwave oven.

Studies have shown that the sample should be mixed in the oven to ensure homogeneous heating and to prevent hot spots [24]-[26]. For this purpose, a stainless-steel apparatus was fixed to the oven ceiling and the sample was mixed while the oven tray was rotating (6 rpm).

A 100 g sample has been used for microwave devulcanization. To determine which temperature was reached at which time for the oven, the samples were heated at 800 W microwave power for 4 to 7.5 minutes. Sample temperatures were measured with a probe surface thermometer immediately after the process. The change in temperature depending on time is shown in Figure 6. The results of TGA analysis were used when determining the appropriate time and temperature for devulcanization. Since the aim of devulcanization is to break only the crosslinks without damaging the polymer chains of the rubber, the temperature was kept below 300 °C, which is the temperature at which the rubber begins to decompose.

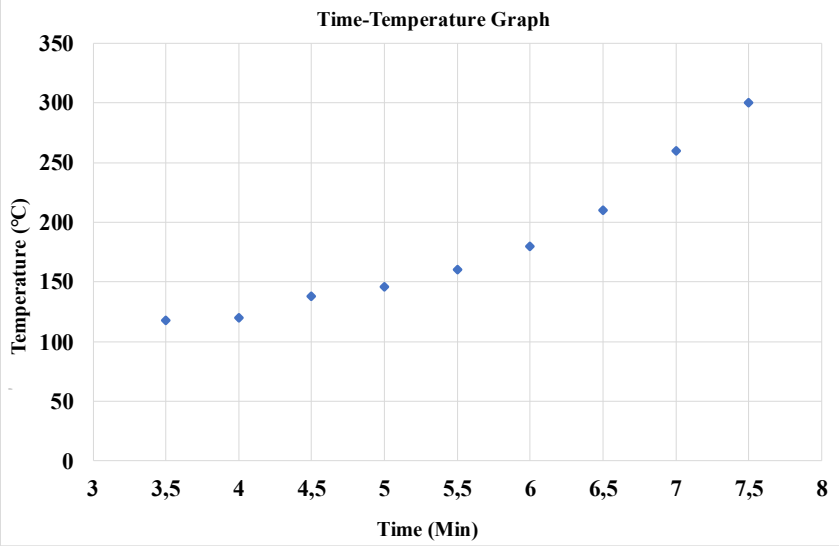


Figure 6. Microwave heating-time graph.

2.3 Characterization of Devulcanized Rubber

2.3.1 Determination of Soluble Fraction

Typically, a vulcanized rubber material contains a large amount of insoluble fraction (gel fraction) that is insoluble in any solvent and a small amount of soluble fraction (sol fraction) that can be dissolved and removed from the structure. These fractions can be determined by extracting the rubber in a suitable solvent. In this study, the soluble fractions of both devulcanized and unvulcanized ground tire rubber were measured using the Soxhlet extraction apparatus shown in Figure 7 according to the ASTM D 297 standard. Toluene has been used as the solvent. The sample amount was approximately 2 g, and the extraction time was 24 hours.

After extraction, the sample was dried in a 70 °C oven for 24 hours. The following equation was used to calculate the soluble fraction of the sample.

$$\%\, Soluble\ Fraction = \frac{W_i - W}{W_i}\ x100 \quad (1)$$

In this equation;

W: weight of dried sample after extraction (g)

Wi: weight of sample before extraction (g)



Figure 7. Soxhlet extraction apparatus.

2.3.2 Crosslink density measurement

With vulcanization, crosslinking occurs, which transforms the linear molecular chain structure of the rubber into a network structure. The degree of crosslinking within the network structure of the rubber molecular chains can be characterized by the crosslink density [27]. The Flory-Rehner equilibrium volume swelling equation is used to calculate the crosslink density of vulcanized rubbers. With this calculation method, the amount of solvent absorbed by the vulcanized rubber is evaluated and the crosslink density is calculated. The low penetration of the solvent into the rubber and the swelling of the rubber mean that the crosslink density is high [28]-[30].

In this study, for the calculation of the crosslink density of the devulcanized and undevulcanized samples, 0.2 g of the sample was inflated by waiting in 10 ml of toluene at room temperature for 72 hours and then removed from the toluene and the excess solvent was

immediately removed and its weight was measured. Afterwards, the sample was dried for 24 hours and its weight was measured again. Crosslink density is calculated from the Flory-Rehner equation, which was developed by considering the Kraus correction factor with filled rubber, with the following equation [31], [32];

$$\rho_c = \frac{1}{2Vs} \frac{\ln(1 - v_r) + v_r + X(v_r)^2}{v_r^{\frac{1}{3}} - \frac{v_r}{2}} \quad (2)$$

In this equation:

ρ_c: Crosslink density (mol/m^3)

V_s: Molar volume of toluene (1.069 x 10^{-4} m^3/mol)

v_r: Volumetric ratio of rubber in the swollen sample (calculations were made by considering the Kraus correction factor)

X: Rubber-toluene interaction parameter (0.3795 for ground tire rubber-toluene) [31].

After finding the crosslink density of the samples, the degree of devulcanization for each sample was calculated with the following equation:

$$\%Devulcanization = \left[1 - \frac{vf}{vi}\right] \quad (3)$$

In this equation;

v_f : Crosslink density of devulcanized rubber

v_i : Crosslink density of ground tire rubber.

2.3.3 Fourier Transform Infrared Spectrophotometer Analysis

To see the chemical modifications after devulcanization, FTIR analyses of devulcanized and undevulcanized samples were performed in accordance with ASTM D 3677 standard using Perkin Elmer FTIR analyzer at Erciyes University Technological Research and Application Center (TAUM). Infrared spectra of the samples were recorded in the range of 450–4000 cm-1 with 4 cm-1 resolution.

3 RESULTS AND DISCUSSION

3.1 Soluble Fraction of Microwave Devulcanized Samples

Soxhlet extraction is a good method for measuring the effectiveness of the devulcanization process. The increase in the soluble fraction in devulcanized rubber indicates that the sulfur crosslinks are broken and devulcanization has occurred [33].

The soluble fractions of ground tire rubber devulcanized at different temperatures are given in Table 2. While the soluble fraction of ground tire rubber is %6.15, it is seen that the soluble fractions increase with the increase in temperature because of microwave devulcanization, and the highest increase is obtained at 240 °C. It was observed that the soluble fraction decreased when the temperature increased to 270 °C. This situation can be explained by the production of free radicals by breaking the bonds and the re-crosslinking of some of the produced macro radicals after the breaking of these bonds. [34], [35].

In the literature review, soluble fraction in microwave devulcanization studies were reported as %14-31 depending on time by Garcia et al. [36], %19.8-21.2 between 190-210 °C by Simon et al. [37], %13.23 to %25.85 for 3-5 min by Musidin [38], %18.2 for 200 °C by De Sousa [33], %26.2 for 220 °C by Molanorouzi and Mohaved [39] and %19.05 to %25.1 depending on time. It is seen that the results obtained from this study are compatible with literature.

Table 2. Soluble fraction depending on temperature.

Sample	Soluble fraction (%)
GTR	%6,15
150±10	%10,08
180±10	%15,44
210±10	%20,89
240±10	%28,40
270±10	%22,08

3.2 Crosslink Densities of Microwave Devulcanized Samples

Crosslink densities were calculated by applying swelling tests to devulcanized and non-devulcanized samples at different temperatures. The calculated crosslink density values are given in Table 3. It is observed that the crosslink density values of devulcanized samples decrease compared to the non-devulcanized sample up to 210 °C and devulcanization occurs. It is observed that the broken crosslinks tend to reconnect with increasing temperature.

Table 3. Crosslink densities.

Sample	Temperature (°C)	Crosslink density (mol/dm^3)	Devulcanization rate (%)
GTR		0,260	-
MD180	180±10	0,221	%15,08
MD210	210±10	0,157	%39,62
MD240	240±10	0,179	%31,30
MD270	270±10	0,393	-

**Sample names are coded with md representing microwave devulcanization and numbers representing temperature.*

In the literature, it is seen that the devulcanization rates in microwave devulcanization studies are given as %40, %35 and %43 by Rajendran [40], %42 by Khavarnia and Mohaved [41], %42.5 by Molanorouzi and Mohaved [39], between %12.16 to %29.43 by Vahdatbin and Jamshidi [42], and %28 by Shabani and Jamshidi [43]. It is seen that the results obtained are consistent with the literature.

Since the crosslinks are broken by the effect of heat during devulcanization, the crosslink density decreases. The increase in the soluble fraction in the extraction of devulcanized rubber is also an indicator of the decrease in crosslink density.

3.3 FTIR Analysis of Microwave Devulcanized Samples

The spectra of the untreated sample and the MD210 sample with the lowest crosslink density obtained by FTIR analysis are given in Figure 8-a.

To see the changes in functional groups after devulcanization in detail, the 1700-900 cm-1 region of the spectrum is given in Figure 8-b. The peaks formed at 1370 cm-1 and indicated the presence of methyl groups characterize natural rubber. Similarly, the peaks formed at 700 cm-1 characterize SBR rubber [44]-[46]. The preservation of both peaks after devulcanization shows that there is no regeneration, i.e. breakage in the main chain structure. The preservation of the peak formed at 1500 cm-1 and characterizing the C=C bond again shows that the polymer main chain structure is preserved. The deepening of the C-O and S=O peaks seen at 1000-1100 cm-1 is a situation that characterizes oxidative decomposition that may occur in the event of thermal devulcanization and was also observed here [47]. The decrease in the peak characterizing carbon black at 1020 cm-1 also occurred for a similar reason [48]. The peak formed at 1538 cm-1 indicates the –C=O peak in the presence of Zn stearate in rubber recipes and the weakening of the peak is an indication of the decrease in crosslink density [49], [50].

The 900-400 cm-1 region of the spectrum is given in Figure 8-c. The most important indicator showing that devulcanization has occurred is the weakening observed in the S-S peaks formed at 460 and 550 cm-1 [38], [45], [50]-[54].

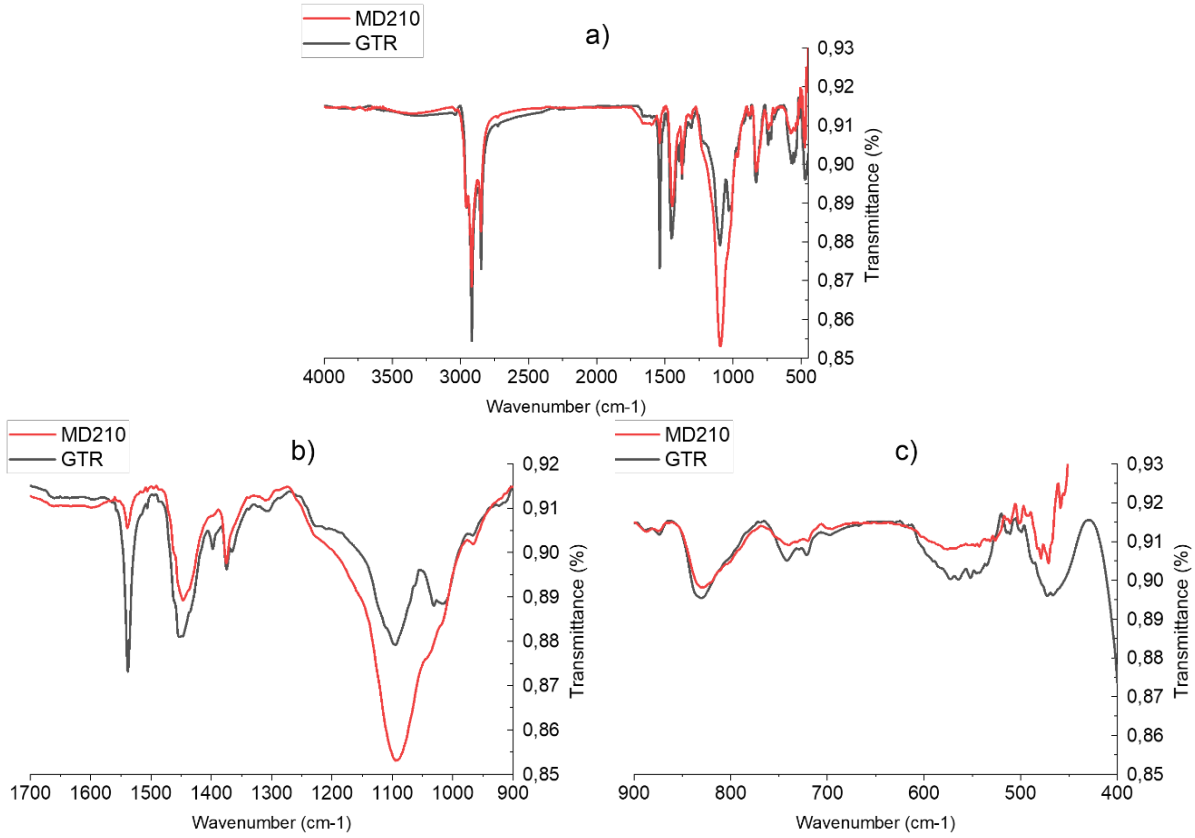


Figure 8. a) FTIR spectra of ground tire rubber and devulcanized sample; b) Spectra of ground tire rubber and devulcanized sample in the range of 1700-900 cm-1; c) Spectra of ground tire rubber and devulcanized sample in the range of 900-400 cm-1.

3.4 SEM Imaging of Microwave Devulcanized Samples

The SEM image of devulcanized ground tire rubber is shown in Figure 9. When compared with the structure of the ground tire rubber given in Figure 3, it was observed that the surface roughness of the devulcanized rubber increased. It has been stated in some studies that this situation is an indication of devulcanization [10], [48], [52], [55].

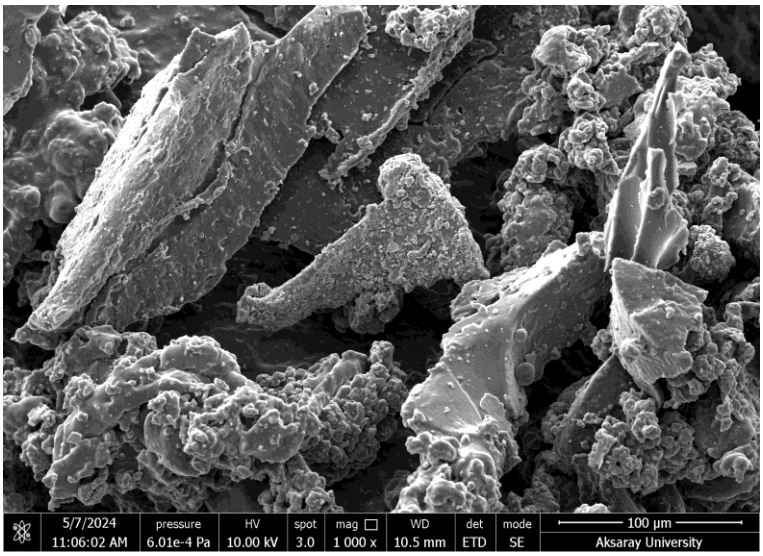


Figure 9. SEM image of devulcanized rubber.

4 CONCLUSION AND SUGGESTIONS

In this study, ground tire rubber with a particle size of less than 1 mm obtained by shredding waste tires at room temperature was subjected to microwave devulcanization. The results obtained because of the study are listed below:

- The soluble fractions obtained by extraction of devulcanized rubber changed depending on the devulcanization time and temperature. While the soluble fraction, which is an important parameter in terms of revealing the effectiveness of devulcanization, was %6.15 in ground tire rubber, this rate increased to %28.40 in the sample devulcanized at 240 °C.
- In the measurement of the crosslink densities of devulcanized rubber, it was observed that the crosslink density values in the samples devulcanized at 210 and 240 °C decreased significantly compared to the ground tire rubber and the devulcanization rate increased in these samples.
- The results obtained from the FTIR analysis performed after devulcanization revealed that the main polymer chain structure was not damaged and the sulfur crosslinks were broken as targeted.
- All the results obtained revealed that microwave treatment at appropriate temperatures is a suitable process for the devulcanization of waste tires.

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Conflict of Interest Statement

There is no conflict of interest between the authors.

Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

Artificial Intelligence (AI) Contribution Statement

This manuscript was entirely written, edited, analyzed, and prepared without the assistance of any artificial intelligence (AI) tools. All content, including text, data analysis, and figures, was solely generated by the authors.

Contributions of the Authors

Conceptualization İ.E. and F.K., Methodology İ.E. and F.K., Writing İ.E. Review and editing F.K. Supervision F.K. All authors have read and agreed to the published version of the manuscript.

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