

Physico-chemical properties of rubber particles-polyester composite using FTIR analysis

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ABSTRACT

The tire rubber particles are treated by microwave energy. The effect of treatment on tire rubber particles is studied after they have been exposed to different microwave energies. The rubber particles is examined and evaluated using an FTIR and a digital microscope. To investigate the impact of treatment on the mechanical properties of composites, polyester rubber composites were prepared. The FTIR analysis demonstrates the effectiveness of the limited devulcanization process, which improves the polymers interface adhesion. The microscopic analysis shows an increased roughness in the particle after treatment which may enhance the adhesion with polymers. The morphological analysis is also performed on the composite and shows good wettability of treated rubber particles and matrix in the composite. Treated polyester rubber composites have a higher ultimate tensile strength than untreated ones, but they have a lower Young's modulus and impact strength.

KEYWORDS

Waste tire rubber particles, Recycling, Microwave treatment, Tensile properties, Impact strength.

INTRODUCTION

Waste tire rubber is an economical, social, and environmental problem nowadays. The waste tire rubber has a large volume which requires a huge area of landfills and dumps. The huge volume and shape of wasted tires can be considered a reason for environmental problems. It can be a good place for the proliferation of mosquitos and many diseases [1]. Also, it can cause soil contamination because of the existence of chemical substances[2]. There is another significant impact on people's general health, which is reflected in the increase in health care costs. Moreover, using nonefficient recycling methods will result in low properties and low economic benefit material from this resource^{[3],[4]}. Even though a new method of recycling tire rubber that retains its good properties, such as using it in a polymeric and butamine composite, has been developed [5],[6],[7],[8],[9]. Several methods were developed for treatment through chemical or physical ways, which aim to break the links and chains of polymers[10],[11],[12],[13]. Ground tire rubber could be exposed to variant types of devulcanization [14].

The aim of this approach is to split C-S and/or S-S crosslinks while preserving the hydrocarbon structure. As elastomers are crosslinked structures so devulcanization of elastomers will make them easily recycled as thermosets cannot be melted. Devulcanization is not an easily controlled process as the energy used is high, which results in a C-C breaking bond [15]. It must be considered that degradation occurs due to breaking the polymer chains [14] [15]. For the waste tire treatment process, researchers show many methods by using chemical, mechanical, thermomechanical, microbial, ultrasound, and microwave treatments [1],[10],[11],[16],[17]. The microwave treatment method or partial devulcanization shows an efficient way to apply a considerable amount of energy without using chemicals in the process [18]. This method has a uniform and fast effect on applying energy to rubber, making it eco-friendly [18]. It enables the breaking of sulfur cross-links [16]. Rubber elastomers are

weak in absorbing microwave energy, so this method requires adding materials such as carbon black to help absorbing energy.

However, this is not required for waste tire rubber as it is rich in carbon black, which supports and helps the use of microwave treatment[16]. Carbon black can absorb electromagnetic waves, and this is attributed to Maxwell-Wagner polarization [18],[19],[20]. Researchers show that the microwave is used in the process of devulcanization of many elastomers [19]. However, devulcanization through microwave has been used, but only a few researchers focus on studying waste tire rubber particles after applying microwave radiation with different energies. Many ways have been developed to recycle waste tire particles. One of the most promising ones is to combine particles with polymers to produce polymeric composites, which can be easily shaped into many products. There are three main targets to use waste tire rubber particles. The first one is to reduce the products' cost by using an economical filler [21]. The second one is to enhance properties as mechanical, physical, and thermal properties of the polymers by adding the waste tire rubber particles as a filler [22]. This research aims to study the effect of using waste tire rubber to prepare polymeric composites and how to enhance the polymer's properties.

MATERIALS AND METHODS

Materials

The ground tire particles were sieved to a size of 2.5 mm of the measured density of 0.4 g/cm³. HOPPEC Company provided the ground tire rubber particles; ground tire rubber generally contains various types of rubber, styrene-butadiene rubber, polybutadiene, carbon black as a filler, and other chemicals additives applied during tire manufacturing. Polyester resin is bought from SUNPOL Company, Turkey.

Ground waste tire rubber particles processing

Ground tire rubber particles were treated by a conventional microwave. A motorized rotational mechanism was connected to the microwave. Using the microwave is agreed with other researchers [23],[24]. In this paper, the microwave used is “Galanz D90N30Atp-Zj Digital Microwave with Grill, 900 W, 30 Liter” with 240 mm height, 354 mm width, and 358 mm depth. The power used in the treatment process was set to 900 Watt. Twenty grams of ground tire rubber particles were placed in a flat ceramic bowl with a constant rotation speed of 60 rpm. Eleven samples of the same mass of particles were exposed to certain periods that result in different energies. This is based on the equation below.

$$T = E \cdot m / P$$

Where: **T** stands for the exposure time in seconds, **m** is the mass in grams, **P** is the power in watt and **E** is the energy in J/g.

The microwave energy and exposure time for different rubber particle samples are tabulated as shown in

Table 1.

Rubber particle treatment procedure

- Twenty grams of waste tire rubber is placed in a flat ceramic bowl.
- The bowl is then placed in the microwave oven for a certain time, as shown in
- Table 1.
- The mass of particles is measured before and after treatment for each sample.

Table 1. Microwave energy and exposure time for different rubber particle samples

Sample	Time in sec	Energy in J/g
T0	0	0
T1	24	1080
T2	48	2160
T3	72	3240
T4	96	4320

T5	120	5400
T6	144	6480
T7	168	7560
T8	192	8640
T9	216	9720
T10	240	10800

Composite samples preparation

Rubber particles/polyester composite of rubber volume fraction of 50%-as agreed with other researchers [25]-was prepared.

- 1) A silicon rubber mold is used to prepare the composite.
- 2) The needed mass of polyester and rubber particles (either treated or untreated) is calculated from the volume fraction at 50 vol%.
- 3) Both polyester and ground tire rubber particles are stirred by a wooden stick together at ambient temperature.
- 4) A vacuum chamber is used for degassing the mixture at -0.8 bar for 5 min.
- 5) The degassed mixture is then poured into the mold.

Chemical analysis

In the EPRI central labs, a Fourier transforms infrared (FTIR) spectroscopy study was performed to determine the effect of treatment on the chemical composition of ground tire rubber. The spectra were obtained on a PerkinElmer Spectrum one

Physical measurements

Mass analysis was done on the ground tire rubber; the mass was measured before particles were exposed to the microwave and after. This test is done on a 0.01 g digital scale.

Morphological examination

The surface of the particles was analyzed using a digital microscope with a magnification of up to 1000x. An average of 5 samples is examined to compare between the treated and untreated samples.

Mechanical testing

- 1) On a LLOYD universal testing machine-30 ton, a tensile test was performed with a crosshead speed of 5 mm/min. In accordance with ASTM D 3039/D 3039M-00, the test samples have dimensions of 250*25*6 mm.
- 2) According to ASTM D 6110-04, a notched impact test is conducted with an Izod & Charpy Impact XJJU-5.5/50J.

RESULTS AND DISCUSSION

Morphological study of rubber particles

Treated and untreated rubber particles surfaces were examined by a digital microscope. It was observed that the treatment had affected the surface of the rubber particles. Treated rubber particles looked rougher, as shown in Figure 1, which agrees with other research [26]. This promotes mechanical adhesion as the surface roughness has increased, which means that the surface area of contact has increased, which increases the interfacial adhesion. Therefore, a deep analysis was required to assess the changes that had happened on the surface of rubber particles.

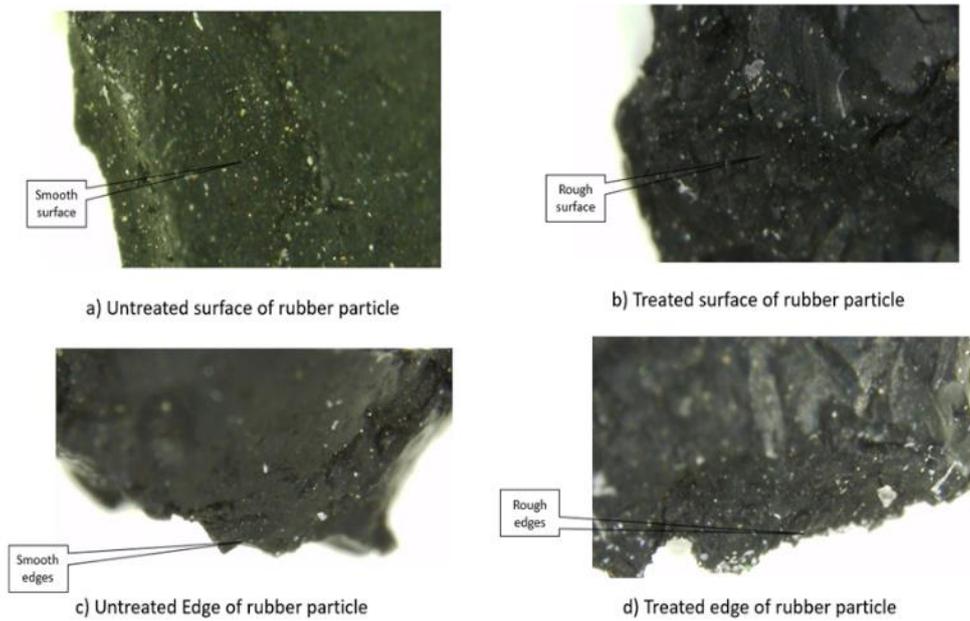


Figure 1. Microscopic examination of untreated (a, c) and treated (b, d) rubber particles – 120X

Chemical analysis

The microwave treatment results in a devulcanization, which results in a change in the surface structure. The breaking of C-S and S-S bonds is enhanced by the microwave. A detailed examination of the FTIR spectrum reveals that several wavelength bands have a peak transition. The researchers showed the wavelengths for bands of functional group as shown in Table 2.

Table 2. Wavelength and cross bonding functional group.

<i>Wavelength cm^{-1}</i>	<i>Functional group</i>
<i>1537 cm^{-1}</i>	<i>Methyl-assisted conjugated double bond for natural rubber and styrene-butadiene rubber. [27]</i>
<i>1435 and 1376 cm^{-1}</i>	<i>CH_2 and S-O bonds [27]</i>
<i>1076 cm^{-1}</i>	<i>C-S-C group. [26]</i>
<i>1029 and 1006 cm^{-1}</i>	<i>C-S bonds [26]</i>
<i>962 cm^{-1}</i>	<i>: -CH=CH- of butadiene present in styrene-butadiene rubber. [28],[29]</i>
<i>828 cm^{-1}</i>	<i>C-H from natural rubber. [27]</i>
<i>3490 and 2925 cm^{-1}</i>	<i>O-H bond. [8]</i>

Figure 2a shows a peak change in bands mentioned above which confirms the structural change in treated rubber particles. A change in intensity of the peak versus the amount and time of ground tire rubber exposure to microwave is also displayed, as shown in Figure 2b. This was done by focusing on each spectrum individually and get the peak intensity difference from its baseline. The peak intensity is referring to the number of bonds per unit volume. So, increasing the peak intensity means the increase of existence of related bonds and vice versa. Figure 2a also shows the full spectrum zone for all microwave treatment superposition. The figure shows similar behavior for most microwave energies. It shows that the existence of hydroxyl groups for all microwave treatment energy with the highest value at T3, as shown in Figure 2b. The peak for C-H does not have a significant difference concerning all microwave treatments, which confirms that C-H bonds did not degrade. The decrease in C=C and

C=O peak intensity for microwave energies after T3 confirms the degradation of these bonds. For C-O, S-O-C, C-C, and S-O bonds, a decrease in intensity is observed, which confirms the devulcanization. As a summary, the analysis shows that two mechanisms are happening at the same time of devulcanization, which are and degradation and deoxidation [16].

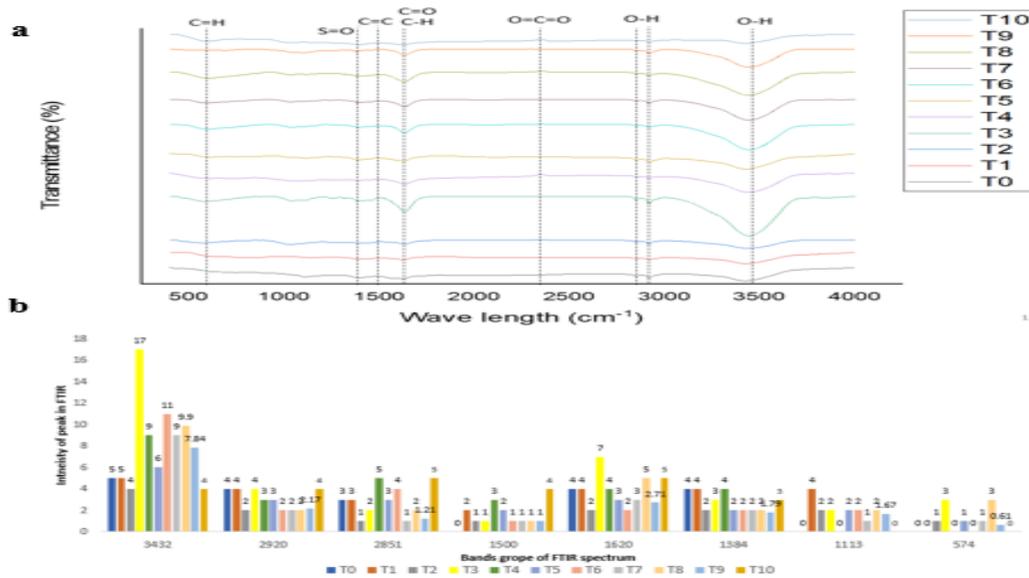


Figure 2. a) FTIR spectrum for untreated rubber particles (T0) and treated rubber particles (T1 – T10), and **b)** Bands group FTIR intensity

Figure 3a displays a detailed area on the FTIR spectrum band from 4000 to 3000 cm^{-1} where the hydroxyl groups (OH) groups can be found, which confirms the increase in hydroxyl groups. These hydroxyl groups represent evidence that the hydrophilicity of the rubber increases. Figure 3b shows a detailed area on the FTIR spectrum band from 3000 to 2850 cm^{-1} . At this band, the C-H bond appears. This group does not have a significant difference which confirms the preservation of the hydrocarbon's chains in rubber and that it does not degrade using microwave treatment.

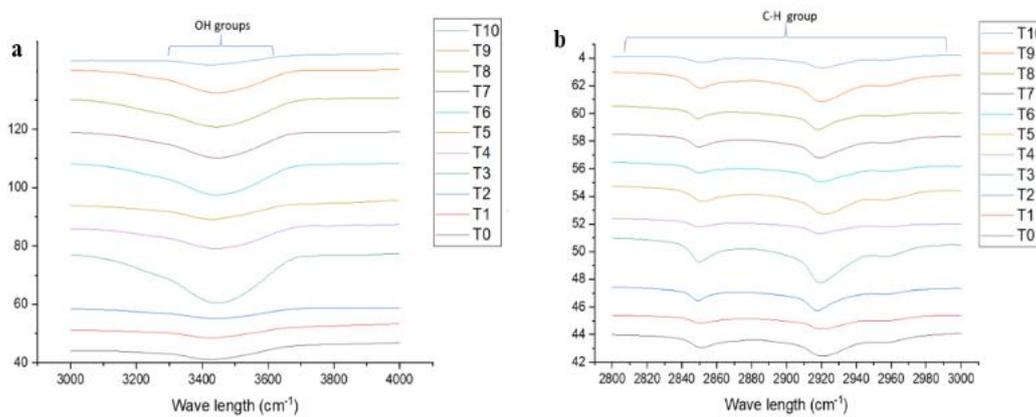


Figure 3. a) FTIR Band from 4000 - 3000 cm^{-1} , and **b)** FTIR band 3000 - 2850 cm^{-1}

A detailed area on FTIR spectrum band from 1850 to 1500 cm^{-1} that is shown in Figure 4a is referring to carbonyl (C=C), (C=O). After T3, which represents the highest peak, the peaks start to decrease with the increase of microwave energy, reaching the lowest value at T10. This result proves the existence of degradation as the main polymer chain is broken [16]. Figure 4b displays a detailed area on the FTIR spectrum band from 1500 to 400 cm^{-1} . This band can be split into two bands. First, from 1500 to 750 cm^{-1} , C-O, S-O-C, C-O-C, S-O, and C-C are all represented by this band. The peaks intensities decrease, as shown in Figure 2b that means the disappearance of groups. A special band at 1050 cm^{-1} decreases, which refers to sulfonyl groups as sulfone -R-SO₂-R- and sulfoxide R-S-O. This disappearance can be explained by the reaction that happened between these groups and the air

forming gases. This confirms the formation of oxidative degradation, and this increases with the increase of microwave treatment energy [16]. Second, the band from 750 to 400 cm refers to C-S, and S-S shows a decrease in intensity, which confirms the break of bonds, proving the success of the devulcanization process [16].

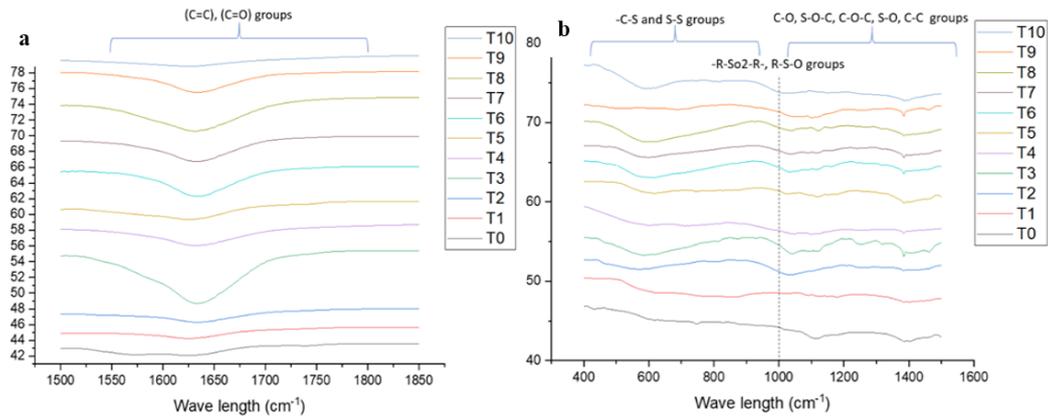


Figure 4. a) FTIR band from 1850 to 1500 cm^{-1} , and b) FTIR band from 1500 to 400 cm^{-1}

The surface treatment was able to increase the wettability at the surface by increasing the existence of functional groups. It's also possible that this would improve the wettability of waste tire rubber particles on the polymers. The tendency of the Polymer Matrix to bind to the rubber particle is referred to as wettability. One of the changes that happened after the treatment is the hydrophilicity of the material due to the higher existence of functional groups (OH:). This enhances the interfacial adhesion [30]. Microwave energy at T3 has the highest intensity, which means higher existence of functional groups on the surface. This shows that the optimum energy to be used by microwave is for T3, in aim to have the best interfacial adhesion.

Physical properties

The mass - of a certain amount of tire rubber at a certain level of energy - was measured twice for both treated and untreated particles. Data on variations in mass for different samples are shown in Figure 5a. Mass loss percentage for each sample was calculated and is shown in Figure 5b. These losses confirm the degradation of rubber and this degradation increases with increasing microwave energy. These results comply with the FTIR results that there is a degradation associated with microwave treatment. Further to T3, the polymer chain starts to degrade, as shown in FTIR.

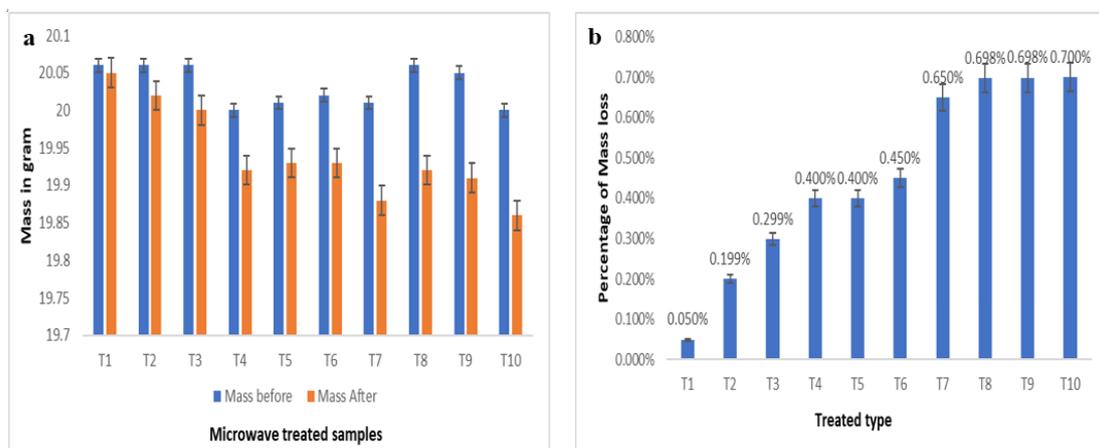


Figure 5. a) Mass of treated and untreated samples of rubber particles, and b) Mass loss percentage vs treatment type rubber particles

Mechanical properties

Both treated (at optimum energy, T3) and untreated rubber particle/polyester composites samples of 50% rubber volume fraction were tested to fracture under static tensile loading and Charpy impact loading

Tensile Test

The stress-strain behavior of pure polyester, processed, and untreated particles/polyester composite is shown in Figure 6. Both treated and untreated rubber composites exhibit the same action, as shown by the curve. The fracture strain of the treated rubber particles composite is higher than the untreated one, as shown by the curve. Furthermore, as opposed to pure polyester, composites have lower tensile strength and fracture strain.

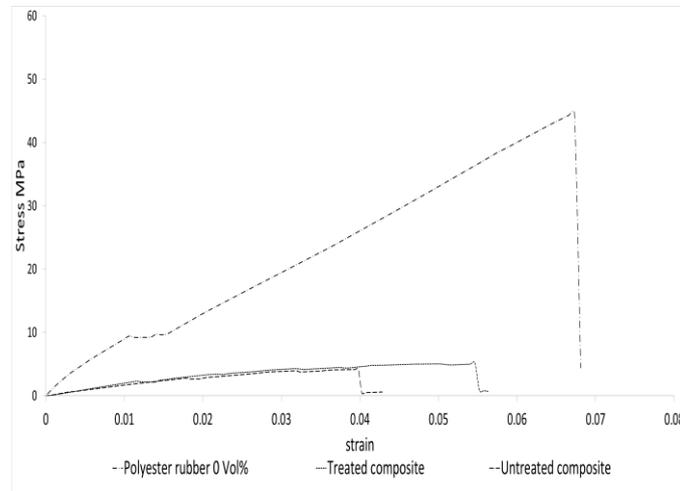


Figure 6. Stress-strain curve of treated and untreated rubber/polyester composites and pure polyester

Table 3 shows a deterioration in tensile strength upon adding treated and untreated rubber particles. This may be attributed to structural defects in the composite that existed when rubber particles were inserted, as well as the high-volume fraction of tire rubber particles. The treated rubber/polyester composites have a higher value of UTS compared to untreated tire rubber particles polyester composites. Figure 2 displays an FTIR analysis that demonstrates this., This reinforces the need to increase the number of functional groups on the surface of the particles in order to improve wettability between the rubber particle and the polymer. Figure 7a also proves the good adhesion between the treated particle and polymer matrix. While depicts the poor adhesion of untreated rubber particles to the polymer matrix, which may explain why treated rubber composites have better mechanical properties than untreated rubber composites.

Table 3. Ultimate Tensile Strength of treated and untreated rubber/polyester composites and pure polyester

Ultimate tensile strength	Value (MPa)
Pure polyester (Rubber particles 0vol.%)	42.2
Untreated composite (Rubber particles 50vol.%)	4
Treated composite (Rubber particles 50vol.%)	4.3

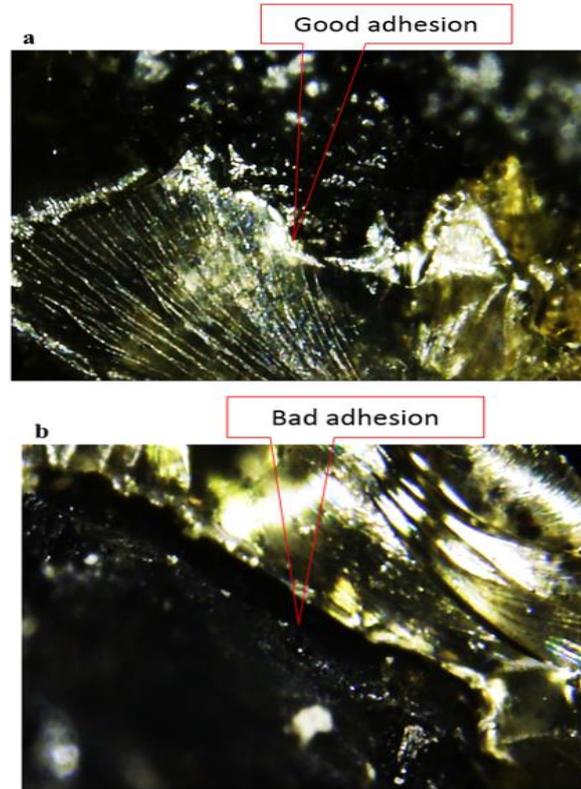


Figure 7. a) Fractography of untreated rubber/polyester composite-300X, and b) Untreated Sample with magnification 240 X

Error! Reference source not found. shows Young's modulus for pure polyester, treated and untreated rubber polyester composite. A deterioration in Young's modulus upon adding treated and untreated tire rubber particles is observed. This could be due to the structural defects that happened in the composite on adding rubber particles. The high-volume fraction of tire rubber particles also contributes to this. As compared to untreated rubber polyester composites, treated rubber polyester composites have a lower value. This could be reasoned by the elastic behavior of rubber caused by the treatment that decreases rubber rigidity. This also can be explained by the good adhesion between treated particles and matrix in the composite [16].

Table 4. Young's modulus of treated and untreated rubber/polyester composites and pure polyester

Young's modulus	Value (MPa)
Pure polyester (Rubber particles 0vol.%)	782
Untreated composite (Rubber particles 50vol.%)	230
Treated composite (Rubber particles 50vol.%)	208

Impact Test

Table 4 shows the impact strength for pure polyester, treated and untreated tire rubber particles polyester composite, and untreated tire rubber particles polyester composite at 50 vol%. The figure shows a deterioration in

Impact strength	Value (J/cm ²)
Pure polyester (Rubber particles 0vol.%)	4.8
Untreated composite (Rubber particles 50vol.%)	5.3
Treated composite (Rubber particles 50vol.%)	2.8

impact strength on adding treated tire rubber particles composite while for untreated tire rubber particles composite, there is a significant increase. This is because the untreated rubber particles have higher sulfur cross-linking. The sulfur cross-linking, which can also be referred to as vulcanization in rubber, increases the cohesive strength and results in higher mechanical properties allowing the particle to absorb more impact energy [25].

Table 4. Impact Strength of treated and untreated rubber/polyester composites and pure polyester

Impact strength	Value (J/cm ²)
Pure polyester (Rubber particles 0vol.%)	4.8
Untreated composite (Rubber particles 50vol.%)	5.3
Treated composite (Rubber particles 50vol.%)	2.8

CONCLUSION

The following conclusions can be drawn from this research:

1. The microscopic analysis shows an increased roughness in the rubber particle after treatment which may enhance the adhesion with polymers
2. According to the FTIR report, surface treatment will improve the wettability of waste tire rubber particles when combined with polymers.
3. The FTIR shows the success of the partial devulcanization process caused by microwave treatment.
4. The optimum energy to get optimum devulcanization with the least degradation for treatment of rubber particles by microwave method is 3240 J/g.
5. Treated tire rubber particles polyester composites have a 7 percent higher ultimate tensile strength than untreated tyre rubber particles polyester composites.
6. Untreated tire rubber particles polyester composites have a ten percent higher Young's modulus than treated tyre rubber particles polyester composites.
7. Untreated tire rubber particles polyester composites have an impact strength that is 89 percent greater than treated tire rubber particles polyester composites.
8. Microwave treatment resulted in a mass loss of rubber particles.

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