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Effect of Microwave Treatment On The Properties of Waste Tire Rubber Particles–Polyester Composites

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***Abstract*: *Waste disposal of solid materials has become a major issue in the last years. Waste tire rubber is one of the major issues in solid waste disposal. Waste tires are stored in landfills which is an environmental hazard. One of the ways for safe disposal is to use it as particles in polymeric composites. Unsaturated polyester resins have been one of the best polymers used as a matrix in polymeric composites because of their availability and desirable properties. To increase the interfacial adhesion between the particles and the polymer matrix, ground tire rubber is treated. Microwave energy is used to treat tire rubber particles, which is an environmentally friendly process. The effect of this type of treatment on the mechanical properties of polyester/rubber composites is studied. Interfacial adhesion is verified through mechanical testing and morphological analysis. Tension and impact tests were performed to compare the properties of composites before and after rubber particles treatment. The morphological examination reveals an increase in adhesion, which affects the composites' mechanical properties.*

Keywords: *Waste tire rubber particles; Recycling, Microwave treatment; Tensile properties; Impact strength.*

I. INTRODUCTION

Waste tire rubber management is a big issue that needs a lot of attention these days. Waste tire rubber management creates a lot of environmental problems. There are many methods to utilize this waste [1]. The annual volume of the waste tire is estimated to be 800 million tires globally. It is subjected to a 2% increase each year [1]. The annual production of tires globally is reported to be around 1.4 million tires, equivalent to 17 tons of waste rubber [2]. Landfilling is the most common method for dumping tires today. Landfilling method is considered the worst way of disposal as it creates environmental problems. The shape of the tire allows it to preserve water, which allows the breeding of insects and mosquitoes that are the main reason for diseases such as malaria.[3]. One of the promising ways of recycling tire rubber is grinding it into particles[4]. The ground tire rubber particles can be utilized as a filler in polymeric composites that improve polymers' properties and reduce the cost [4].

In order to use ground tire rubber particles with polymer matrix, the bond between them must be maintained. Previous researchers have studied interfacial adhesion and tried to increase it [5]. Ground tire rubber treatment can be carried out through physical or chemical methods. The surface of ground tire rubber was treated with radiation and gas. [6]. The goal of these methods is to create polar groups on the surface of rubber particles, which will improve the interface by interacting with polymer polar groups [6]. Microwave can also be used as a treatment for ground tire rubber. The microwave method is promising due to its uniform energy and high productivity [7]. The microwave is a physical method that does not include any chemicals; it also provides the amount of energy needed for treatment in short periods and is considered an eco-friendly process [7].

Polymers have been used widely as a matrix material in composites. Unsaturated polyester resins have been one of the best polymers used as matrix material in fiber-reinforced composites. The automotive industry consumes a lot of these composites. The thermoset resins have a problem which is low impact strength compared to thermoplastics. In automotive, these parts suffer from damages even at low impact events. Many studies show the aim to improve this inability by adding liquid rubber to the resin [8] or by adding impact modifiers as waste tire rubber particles and marble dust[9] [10][11]. Rubber from waste tires can also be used as a composite element in cement and concrete. [12]. The modifiers aim to increase the impact strength of this resin resulting in high-impact composites. The addition of modifiers shows deterioration in mechanical properties due to bad interfacial adhesion [10]. Making composites using thermoset polymer and waste tire rubber particles is easy in manufacturing and can use conventional methods such as compression molding and hand lay-up.

Meanwhile, there are two objectives of using waste tire rubber particles as a filler with polymeric composites. The first objective is the cost, as this is a waste and cheap material compared to raw materials. The second objective is to modify the mechanical properties of the polymer[14]. Generally, it can be concluded that these are the main objectives of using waste tire rubber in composites with polymers. The price of ground tire rubber is lower than resins. The ground tire rubber particles' price can cost about 120 €/ton for a size of 1-3 mm; this cost increases to 300 €/ton for a size of 0.8 mm [15]. On the other side the prices for thermosets, which promote the research in this field, are high. Table 1 shows the costs of resins and ground tire rubber.

Table 1 Costs of resins and waste tire rubber [15].

Material type	**Manufacture**	**Average cost €/kg**
Epoxy	Organika Sarzyna S.A.	4.9
Polyester	Organika Sarzyna S.A.	3
Polyurea	Neotex S.A.	9.3
Polyurethane	Dow	3
Waste tire rubber average size	Grupa Recykl S.A.	0.2

Using the ground tire rubber with resins is proved to have better properties, mainly for impact and toughness [16]. This is due to the high elasticity of rubber, which enhances these properties in composites. But it also results in deterioration in other mechanical properties due to lack of good interfacial adhesion[17]. There are many methods of enhancing interfacial adhesion. One of the methods is to use a compatibilizer with non-polar polymers that do not have functional groups. Another one is to use maleic anhydride or glycidyl methacrylate that have good interaction with polymer and could also result in good interaction with ground tire rubber[18]. Still, this way is only suitable for thermoplastics. In the case of thermosets, another methodology is used, which is the modification of tire rubber particles surface. As can be seen in Figure 1., thermosets have a high functional group content. Untreated ground rubber, on the other hand, is non-polar and has few surface functional groups. On the rubber particle's surface, the aim is to have a high content of functional groups. Various compounds are grafted on the surface of rubber particles in this process [19]. In devulcanization, the process is accompanied by breaking the network structure of rubber. This is a summarization of the different ways of enhancing the interfacial adhesion between ground tire rubber particles and polymers. This study on waste tire rubber particles aims to thoroughly analyze the tire rubber thermoset composites after applying the microwave treatment.

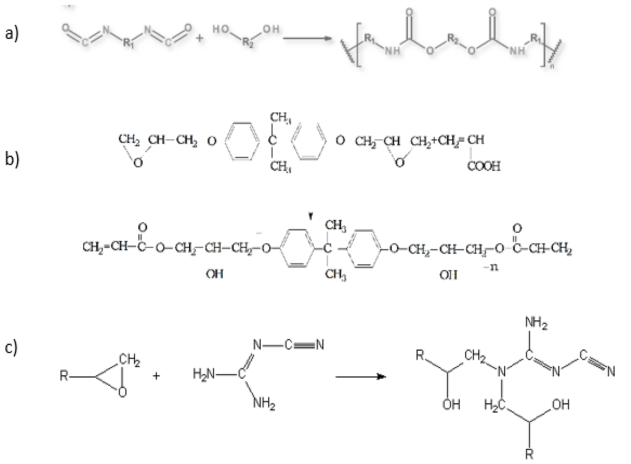

Figure 1 General reaction (a) polyurethane, (b) polyester, and (c) epoxy [19]

II. MATERIALS AND METHODS

A. Materials

HOPPEC Company supplies rubber waste particles, Egypt, and then the particles were sieved to get a particle size of 2.5 mm. The density of these particles is 0.4 g/cm$^{3.}$ Unsaturated Polyester resin and its hardener are supplied by SUNPOL Company, Turkey. The density of this matrix is 1.2 g/cm^{3}.

B. Ground waste tire rubber particles treatment

Microwave energy is obtained from a conventional microwave and is used to treat waste tire rubber particles [20] [21]. The microwave used is "Galanz D90N30Atp-Zj Digital Microwave, 900 W, 30 Liter with dimensions of 240 mm *354 mm *358 mm. The microwave rotational speed is 60 rpm. The used power was 900 Watt. The treatment was done using an energy of 3240 J/g, which is the optimum energy obtained in a previous study[22]. This is according to the following equation.

Time (seconds) = Energy (J/g) *mass (g)/Power (watt)

Treated rubber particles are prepared as follows:

a. Waste tire rubber is placed in a flat ceramic bowl.
b. The bowl is then placed in the microwave oven for a certain time to obtain the required energy according to the mass needed in the sample.

C. Composites preparation

Composite polyester/rubber particles are prepared the following way:

a. Figure 2 shows how the mold is shaped to suit the dimensions of the test samples.
b. The polyester and rubber particle volume fractions are determined, and the corresponding mass is weighed and prepared.
c. According to the volume fraction of each sample, both polyester and rubber particles are stirred by a wooden stick together at ambient temperature.
d. A 2.5 percent hardener is applied to the mixture and stirred for 2 minutes.

e. The blend is degassed for 5 minutes at -0.8 bar in a vacuum.
f. The mixture was then poured into the ready silicon rubber mold and allowed to dry for 1 hour, based on the sample size needed.

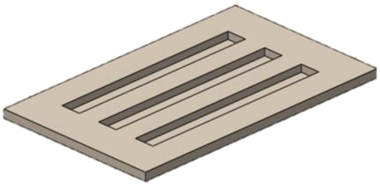

Figure 2 Silicone rubber mold for preparing test samples

A universal testing machine LLOYD 30-ton is used. The crosshead speed of the test is 5 mm/min. Figure 3 shows the dimensions of the tensile test sample according to ASTM D 3039/D 3039M-00. The material's behavior is studied based on the obtained stress-strain curve from the tensile test. Ultimate strength is obtained as the maximum stress, offset yield strength is obtained at 0.1% offset. Toughness is calculated from the area under the stress-strain curve. Yield strength, ultimate tensile strength (UTS), and toughness were normalized by dividing their values by the composites' density, which is 1.15 g/cm^3 [23]. As shown in Figure 4, the impact strength was determined using an XJJU-5.5/50J Izod & Charpy Impact tester and a standard Charpy impact sample according to ASTM D 6110-04. Also, the impact strength was normalized.

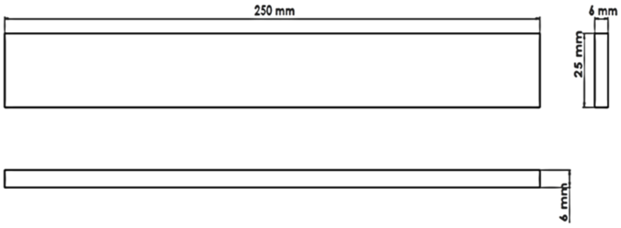

Figure 3. Sample of the Tension Test

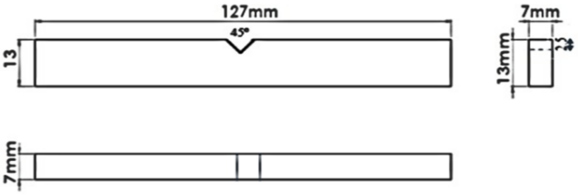

Figure 4 Sample for impact testing

D. Morphological analysis

The interfacial adhesion of rubber particles with the polymer in the composites was assessed using fracture analysis of the samples' surface morphology that underwent impact testing. The Leo Supra 55 field emission scanning electron microscope was used at the AUC labs in Cairo. In addition, the fracture surface was examined using a digital microscope.

III. RESULTS AND DISCUSSION

A. Mechanical properties

Samples for tensile and impact tests were prepared according to the standard dimensions shown in Figure 3 and Figure 4. The treated rubber particles of corresponding energy of 3240 J/g are used in preparing tensile and impact samples. The Untreated rubber particle composites result for tensile and impact strength was taken from previous research (Elenien et al.2020) [23].

a) Tensile Test

Figure 5 demonstrates the stress-strain curves obtained from the tension test for different volume fractions of rubber particles. The behavior under tensile loads is almost identical regardless of the volume fraction of rubber particles. Compared with Polyester resin (0% vol. fraction), the composites' stress and strain values have decreased. This can be explained as the matrix loses its continuity by adding particles. The curves also show that the strain increases with the increase of rubber volume fraction while the stress decreases and agrees with previous researchers [24].

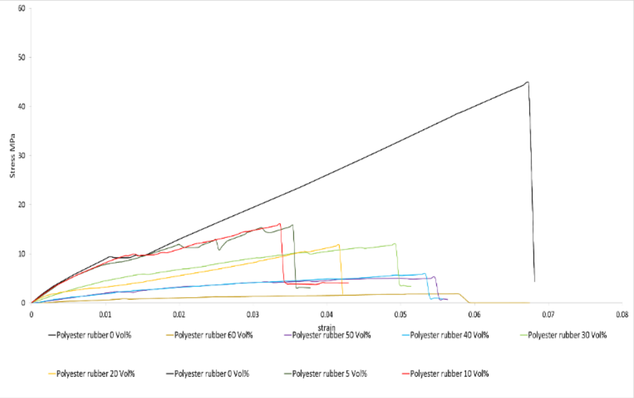

Figure 5 Stress-Strain curves of treated rubber particles/polyester composites and pure polyester

The increase of fracture strain can be explained by the elastomeric characteristic of rubber compared to thermoset polyester polymer [23]. According to other researchers, the tensile strength obtained from tensile tests for untreated waste tire rubber particles decreases as the volume fraction of particles increases. [10] [11].

The offset yield strength of treated particle composites increased as the volume fraction of waste tire rubber particles increased, with the maximum value at 10%, beyond 10 vol. %, offset yield strength begins to decrease but remains higher than pure polyester; beyond 30 vol. %, offset yield strength is lower than pure polyester resin.

From 0 vol. % till 5 %, the particle's effect as a stress raiser is low, which has no significant effect on yield strength. From 10 vol. % till 30 vol. %, the particle's effect to act as stress raiser was high, resulting in increasing the offset yield strength. This can be due to the good continuity of composites structure. After 30 vol.%, the particle's effect to act as a stress raiser is not effective due to the high-volume fraction, which increases the defects in composites' structure.

Figure 6 shows a comparison between the mechanical properties of untreated rubber [23] and microwave treated rubber/polyester composites. The treated rubber particle composites show higher offset yield strength for all volume fractions. This proves that microwave treatment has improved the interfacial adhesion resulting in higher-yielding strength. There has been an operative transfer of the load from –the matrix to the particles.

The offset yield increased by 35 % at 10 vol.% compared to pure polyester. This is due to physicochemical adhesion that happened at the interface of treated rubber particles and matrix by the functional groups, as agreed with Aoudia et al. [25]. This was created on a rubber surface during microwave treatment. This can be verified by the FTIR analysis, which indicated the existence of high content of functional groups on rubber particles' surface, which increases the interfacial adhesion between rubber and matrix [22].

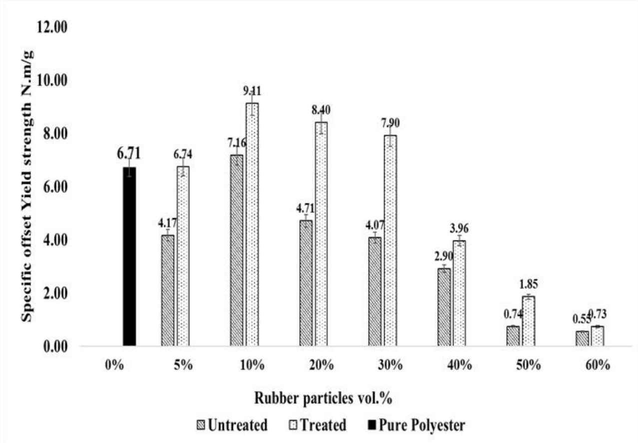

Figure 6 Specific offset Yield strength of treated and untreated rubber/polyester composites

Figure 7 shows that as the volume fraction of treated tire rubber particles increases, the ultimate tensile strength decreases, which is consistent with previous studies. It's possible that this is due to the added particles' lower ultimate tensile strength. The ultimate tensile strength of treated particles composites shows no significant effect than untreated, as shown in Figure 7.

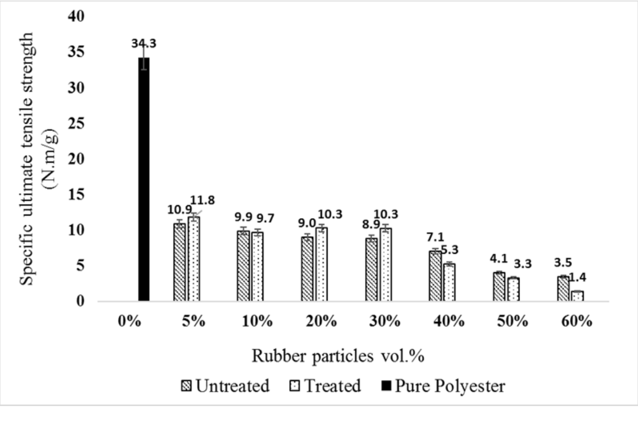

Figure 7 Specific ultimate tensile strength of treated and untreated rubber/polyester composites

Figure 8 shows that the strain at fracture decreases significantly by adding treated rubber particles volume fraction compared with pure polyester. This can be explained as the matrix loses its continuity by adding particles. It is obvious that the strain at fracture increases by increasing the rubber particles volume fraction. This can be explained as the ductility of rubber particles is more than that of polyester resin which promotes the increases in the composites' ductility.

The strain at fracture of treated particles composites shows higher values compared to that of untreated composites. The reason is that the rubber particles' treatment increases the functional groups on the surface of rubber particles, which increases the interfacial adhesion as discussed in the analysis of FTIR [22].

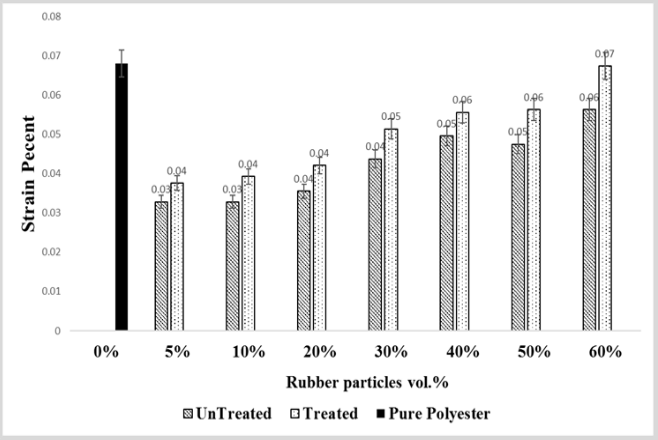

Figure 8 Strain at fracture of treated and untreated rubber/polyester composites

Figure 9 shows that as the volume fraction of treated rubber particles increases, the toughness of the material decreases. This is consistent with the fact that, despite increasing the strain to fracture, ultimate tensile strength has decreased significantly, resulting in lower toughness. The toughness for treated particles is higher than untreated, and this is because of good adhesion. This proves that microwave treatment has improved interfacial adhesion.

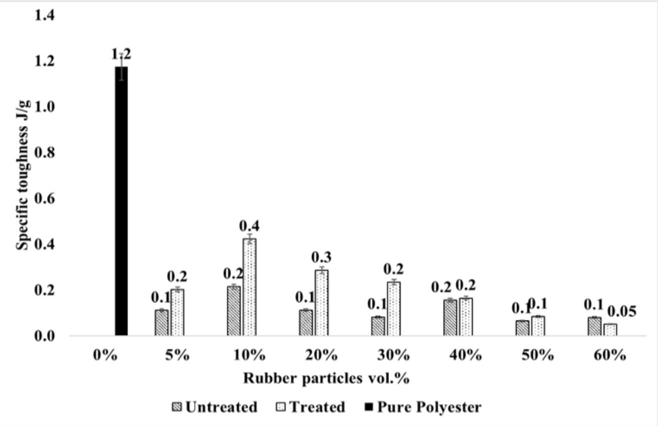

Figure 9 Specific toughness of treated and untreated rubber/polyester composites

b) Impact Test

Figure 10 shows the impact strength for treated and untreated rubber/polyester composites. The impact strength of treated composites decreases as the volume fraction of tire particles increases from 0 vol % to 10 vol %. Then, it shows a significant increase of up to 50 vol. %.

The Charpy impact shows how easily the crack initiates and how fast it propagates at low volume fractions up to 20 vol. %, the crack is easily initiated in the matrix, and the low amount of rubber cannot block the propagation. At higher volume fractions up to 50 vol. %, the particles delay the initiation of the crack and slow the crack's propagation, increasing the impact strength above 50 vol. %, the matrix volume fraction, which acts as an adhesive for the particles, decreases, and the particles become looser and cannot withstand the impact.

In comparison to the untreated rubber composites, the treated particles composites displayed less impact strength as the volume fraction of rubber particles increased. This can be explained as the devulcanization process breaks the formed 3-dimensional network in the material, and this decreases the elastic property of the elastomer. The rubber becomes of less mechanical resistance [23].

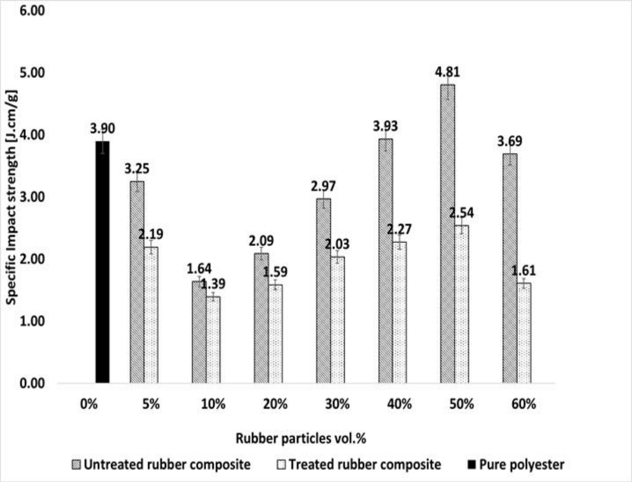

Figure 10 Specific Impact strength of treated and untreated rubber/polyester composites

B. Morphological analysis of the composites

Untreated tire rubber particle composites were examined using a scanning electron microscope (SEM). For SEM, all samples were gold-sputtered. The poor adhesion of waste tire rubber particles to polyester is shown in Figure 11 and Figure 12. It also shows the river marks pattern happened due to the brittle mode of failure of polyester.

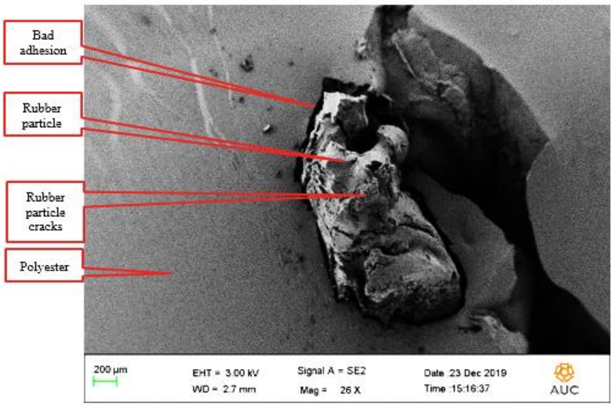

Figure 11 SEM of untreated rubber/polyester composites with impact fractured surfaces at 25X magnification.

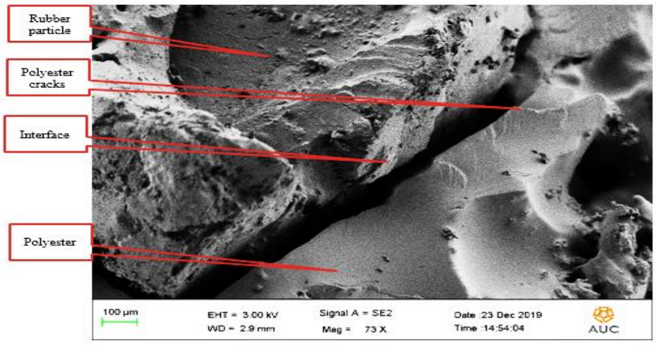

Figure 12 SEM of untreated rubber/polyester composites of impact fractured surfaces-73X

The fracture surface of treated rubber particle composites was investigated using a digital microscope. Figure 13 and Figure 14 show a good adhesion between treated tire rubber and polyester. Also, the polyester shows a fracture pattern named river marks pattern so that cracks start at the rubber particle and propagate[26]

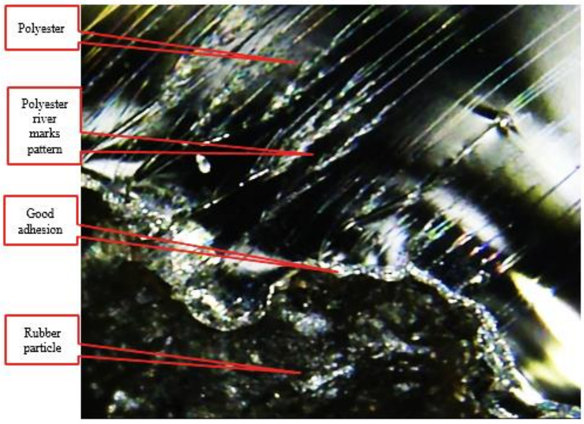

Figure 13 Fractography of impact surface of treated rubber/polyester composites-127X

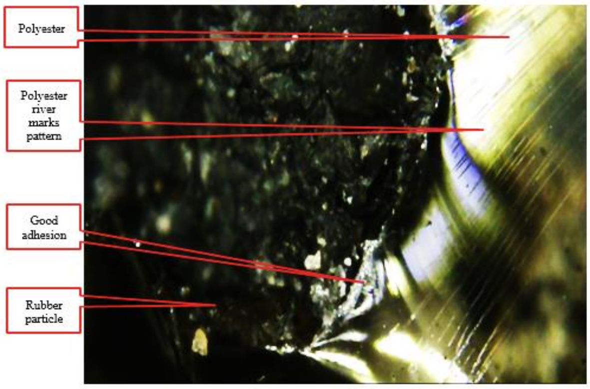

Figure 14 Fractography of impact surface of treated rubber/polyester composites-90X

IV. CONCLUSION

The following conclusions can be drawn from this research:

- Microwave treatment of waste tire rubber particles improves interfacial adhesion between the particles and the matrix.
- The offset yield strength for treated composites increases with increasing the rubber particles volume fraction up to 10 vol. %.
- The offset yield strength for treated composites is greater than untreated.
- The strain at fracture increases by increasing the rubber particles volume fraction. The strain at fracture of treated particles composites shows higher values compared to that of untreated composites.
- The impact strength of untreated rubber particle composites is higher than a treated one.

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