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Ultrasonic Characterisation of Epoxy Resin/Polyethylene Terephthalate (PET) Char Powder Composites

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This study is carried out in order to determine the elastic properties of the Epoxy Resin (ER) / Polyethylene terephthalate (PET) Char Powder Composites by ultrasonic wave velocity measurement method. Plastic waste was recycled as raw material for the preparation of epoxy composite materials. The supplied chars were mixed with epoxy resin matrix at weight percentages of 10 %, 20 % and 30 % for preparing ER/PET Char Powder (PCP) composites. The effect of PET char powder on the elastic properties of ER/PCP composites were investigated by ultrasonic pulse-echo method. According to the obtained results, the composition ratio of 80:20 is the most appropriate composition ratio, which gave the highest elastic constants values for ER/PCP composites. On the other hand, the best electrical conductivity value was obtained for 70:30 composition ratio. It was observed that ultrasonic shear wave velocity correlated more perfectly than any other parameters with hardness.

Keywords: PET, recycling, polymer composites, elastic properties, ultrasound.

1. INTRODUCTION

Ultrasonic, which is a subcategory of acoustics, deals with acoustics beyond the audio limit (20 kHz). The ultrasonic techniques are used for thickness gauging, flaw detection and acoustic imaging and can also be used to define and quantify mechanical properties such as density and elastic modulus, and to characterize internal structural features of parts. Ultrasonic velocity has a wide range of application in the field of material characterisation. The characterisation of materials by ultrasound has some advantages in relation to traditional methods. Ultrasonic measurements can be made on actual components without destroying the samples [1].

Plastics offer features such as light weight, high strength, convenience, and low cost, making them attractive for businesses and consumers. Leaving the waste materials to the environment directly can cause many environmental problems. Hence, many countries have been working on how to recycle the waste materials to reduce hazards to the environment [2].

Over 78 wt.% of all plastic waste produced corresponds to thermoplastics and the remaining to thermosets [3]. The thermoplastics can be recycled and one alternative way to plastics waste disposal is recycling them. However, recycling of plastics is so important, only a small percentage of postconsumer plastics waste is recycled around the world. For this reason, attention is being focused on the development of recycling technologies. However, the tendency towards recycling has increased [4], the problem of recycling polymeric materials has not been solved at a satisfactory level. Recently, environmental, legislative, and consumer pressures have led to an increased interest in using polymeric wastes [5]. The suitable treatment of plastic wastes is one of the key questions of the waste management and is important from energetic, environmental, economic and political aspects [6].

The types of plastics most used are polyethylene, polypropylene, polyvinylchloride, polystyrene, and polyethylene terephthalate (PET) in Europe [7]. The PET is one of the most frequently used raw materials for the manufacture of soft-drink bottles [8]. Suebsaeng et al. [9] reported that in the pyrolysis of PET, newly formed functional groups can react with the surface of the polymer to produce a more thermally stable, less volatile char. Gil et al. [9] obtained about 22 % char in their final product by using pyrolysis of PET. Using the char of PET in epoxy resin can help to obtain new kind of conductive composite materials. In this case, the pyrolysis is considered to be an effective technology for recycling the PET wastes.

In related literature, several studies were carried out on using char in different kind of composites [10 – 12]. There are limited studies related with usage of PET char with epoxy resin (ER) and also ultrasonic characterisation of these materials. Therefore, the present study aims to determine the elastic properties of Epoxy Resin (ER) / Polyethylene terephthalate (PET) Char Powder composites by ultrasonic wave velocity measurement method.

2. EXPERIMENTAL

2.1. Materials

The commercially available undiluted ER-NPEL 128 (EEW 190 g equiv., Konuray Chemical Co.) was a bisphenol A-type ER. The curing agent was a cycloaliphatic polyamine Epamine PC17 (Konuray Chemical Co., Turkey). The epoxy embedding medium accelerator (2, 4, 6-tris [dimethylaminomethyl] phenol) was obtained from Sigma-Aldrich Co., USA. The char of

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plastic waste as raw carbon material was obtained by the pyrolysis of polyethylene terephthalate (PET) at 450 °C, respectively.

2.2. Preparation of ER/PCP composites

The PET char was ground to obtain char powder before being mixed with epoxy. The PET char powder (particle size < 250 µm) obtained in 10 wt.% and epoxy resin matrix in 90 wt.% were mixed with mechanical stirring during 3 h at 1200 rpm speed. Afterwards, 30 wt.% of Epamine PC17 hardener and 1 wt.% of (2, 4, 6-tris [dimethylaminomethyl]) phenol catalyst were added and the mixture was outgassed in an ultrasonic bath for 1 h at room temperature and then transferred into the stainless steel moulds (20 x 20 x 20 mm^3). The curing procedure occurred in an oven at 40 °C for 24 h and then post cured, while increasing the temperature from 60 °C to 120 °C within 48 h. The epoxy resin (ER)/PET Char Powder (PCP) composite obtained was labelled as ER/PCP-1.This process was done for ER/PCP addition at 80:20 and 70:30 ratios as well. The composites obtained were denoted as ER/PCP-2 and ER / PCP-3, respectively. Only one sample, which has 20 x 20 x 20 mm^3 dimensions, was obtained for each kind of composites.

2.3. Density, thickness and ultrasonic wave velocity measurements

For the calculation of the elastic constants of each sample, initially their densities were calculated using Archimedes' principle using an analytical balance (Radwag AS220/C/2, capacity 220 g, readability 0.1 mg, Poland) and a density kit (Radwag 220, Poland). All the densities were measured at same temperature as 25 °C. The estimated error of density was about 0.02 %. The thicknesses of the specimens were measured using an analog micrometer and the samples were found to possess plane parallelism to an accuracy of ± 0.002 mm.

The ultrasonic wave velocities measurements were done by pulse-echo method at room temperature. A flaw detector (Epoch-XT-Panametrics Olympus, USA) was used to measure the ultrasonic velocities. 20 MHz longitudinal (V116-Panametrics Olympus, USA) and 5 MHz shear (V155-Panametrics Olympus, USA) contact transducers were used. The glycerin (BQ-Panametrics Olympus, USA) was used for the longitudinal wave velocity measurements, and honey was used for the shear wave velocity measurements as the coupling mediums.

The knowledge of the transit time through the thickness of the sample allows the determination of the wave velocities by Eq. 1:

$$V = \frac{2d}{\Delta t}, \tag{1}$$

where V, d, and Δt are the velocity of sound, the thickness of the sample, and the time-of-flight between subsequent backwall signals on the oscilloscope, respectively. The measurements are repeated ten times to check the reproducibility of the data. The percentage error of longitudinal wave velocity measurements was about 0.2 % and the percentage error of shear wave velocity measurements was about 0.1 %.

2.4. Determination of elastic constants

The elastic properties of composites are calculated according to the following equations, which are valid for isotropic materials [13].

$$L = \rho \cdot V_L^{\,2}; \tag{2}$$

$$G = \rho \cdot V_s^2; \tag{3}$$

$$K = L - \frac{4}{3}G; \tag{4}$$

$$\mu = \frac{L-2G}{2(L-G)}; \tag{5}$$

$$E = 2G(1+\mu); \tag{6}$$

$$H = \frac{(1-2\mu)E}{6(1+\mu)}; \tag{7}$$

$$Z = \rho V_L; \tag{8}$$

where V_L, V_S, L, G, K, μ, E, H, Z, and ρ are respectively the longitudinal wave velocity, shear wave velocity, longitudinal modulus, shear modulus, bulk modulus, Poisson's ratio, Young's modulus of elasticity, ultrasonic micro-hardness, acoustic impedance, and density of the samples. The estimated percentage error for L, G, K and E were respectively 0.4 %, 0.2 %, 0.4 % and 0.2 %, for acoustic impedance, it was 0.2 %, for Poisson's ratio measurements was 0.08 % and for ultrasonic micro-hardness was 0.2 %.

2.5. Conductivity and morphological measurements

Conductivity of composites was measured in the range of 1 – 10 Volts. Conductivity was measured by means of the standard in-line four point probe method using an Entek FPP-470 instrument, Entek Electronics Co., Ankara, Turkey. An average of 10 measurements were taken to check the reproducibility of the data.

Scanning electron microscopy (SEM) was performed to investigate the interface between ER and PET char powder (Philips XL30 SFEG, Eindhoven, Netherlands).

3. RESULTS AND DISCUSSIONS

3.1. Density, sound velocity and conductivity

As seen from Table 1, the actual density of the ER/PCP composites increases with increasing of the filler content as compared to epoxy resin (ER) and the densities ranged between 1164 – 1182 kg/m^3 for composites of ER/PCP. The highest density value was obtained for ER/PCP-2. The density of solid materials depends upon many factors such as structure, coordination number, cross-link density, and dimensionality of interstitial spaces [14]. Therefore, the increase in densities is supposed to due to addition of the PET char powder.

The longitudinal ultrasonic velocities of ER/PCP composites ranged from 2717 to 2753 m s^{-1} and the shear ultrasonic velocities ranged from 1218 to 1262 m s^{-1}. As can be seen in Fig. 1, the ultrasonic velocity in all the composites increased with the increase in PET char powder content.

Table 1. The density (ρ), ultrasonic wave velocities (V_L and V_S) and electrical conductiviy of the pure ER and ER/PCP composites

Sample ID	Composition ratio of ER/PCP, wt.%	ρ, kg/m^3	V_L, m/s	V_S, m/s	Electrical conductivity, *S*/cm
ER	100:0	1160	2693	1206	$1.03\text{x}10^{-14}$
ER/PCP-1	90:10	1164	2717	1218	$1.35\text{x}10^{-7}$
ER/PCP-2	80:20	1182	2742	1262	$3.72\text{x}10^{-4}$
ER/PCP-3	70:30	1168	2753	1244	$2.81\text{x}10^{-3}$

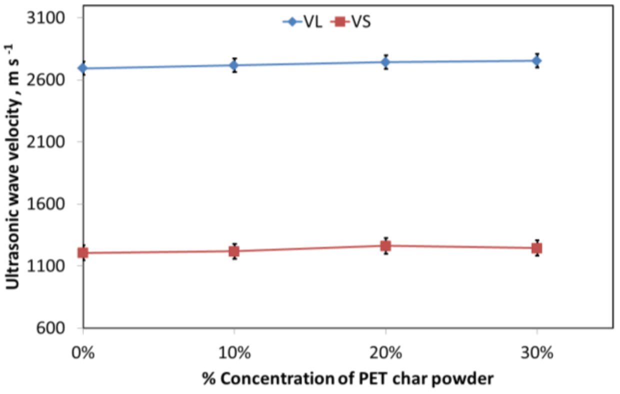


Fig. 1. Variation in ultrasonic wave velocities (V_L, V_S) with wt.% amount of PET char powder in ER/PCP composites

The both ultrasonic longitudinal and shear wave velocities have showed the similar behaviour until 30 % PET char addition in composites. However, the longitudinal wave velocity was showed a linear increase with increasing the PCP in ER/PCP composites, the shear wave velocity has showed a decrease after increasing PCP mount from 20 wt.% to 30 wt.%. On the other hand, the highest velocity values for both longitudinal and shear waves were seen by PCP addition into pure ER at 20 wt.%.

The ultrasonic velocity is related to the elastic constants and density of materials. Hence, it gives the information about the mechanical and elastic properties of medium through it passes. As it is known that the increase of ultrasonic wave velocity is generally related to the decrease in inter-atomic spacing of the material. The increase in ultrasonic velocities may be attributed to decrease in the inter-atomic spacing. The decrease, which was accoured at 20 wt.% addition of PCP, can be explained by effect of PET char powder on the transversal bonds' attractive forces.

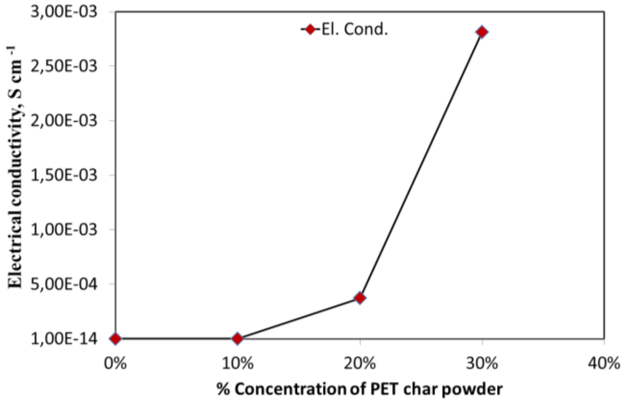


Fig. 2. Variation in electrical conductivity for wt.% amount of PET char powder in ER/PCP composites

The conductivity of ER has increased with the increasing PCP amount. The conductivity of ER/PCP composites increases by increasing concentration of PET char powder in composites (see Fig. 2). The conductivity of composites in different contents was in the range of 1.35 x 10^{-7} to 2.81 x 10^{-3} S cm^{-1}. This means that the PET char powder addition in ER has formed semi-conductive composites and this is a very important result, which shows that it is possible to obtain semi-conductive material from recycled PET wastes. These conductivity results of the research are in agreement with related literature [15]. Char is a porous material rich in carbon (up to 50 %) in a highly stable form [16]. The presence of sp^2 carbon structures enhances the possibility of using these carbon materials for wider applications involving electrical conductivity [17]. Therefore, the increase in electrical conductivity is attributed to increase in carbon source.

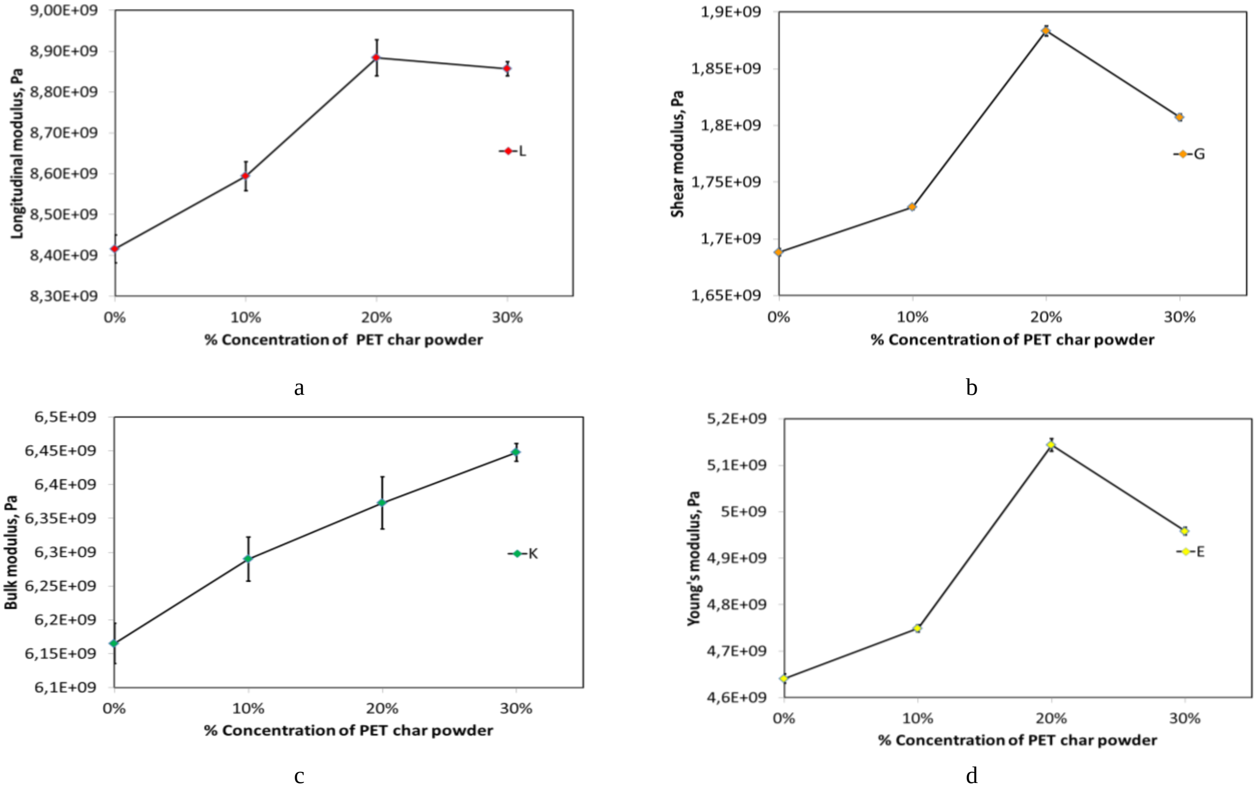
3.2. Elastic modulus

The elastic moduli namely longitudinal modulus, shear modulus, bulk modulus and Young's modulus determined in this study are the real parts of the complex modulus. Because that the attenuation coefficient was not determined in this study. The imaginery part of the complex modulus, which is related with the viscosity of the samples was not carried out and the viscosity of the samples was not discussed.

As it is seen in Table 2 and Fig. 3, the values of elastic moduli of ER/PCP composites are higher than pure ER. The longitudinal modulus of ER is measured 8.42 GPa. However, it is increased from 8.42 to 8.88 GPa by PET char powder addition into pure ER at 20 wt.%. The longitudinal modulus ranged from 8.76 to 8.88 GPa, the shear modulus ranged from 1.68 to 1.88 GPa, the bulk modulus ranged from 6.16 to 6.44 GPa and the Young's modulus ranged from 4.74 to 5.14 GPa for ER/PCP composites. The highest elastic constants values were seen for ER/PCP composite, which prepared at ratio of 80:20. Based on the findings from Table 2 and Fig. 3, it was figured out that addition of PET char powder in different amount has increased all the elastic moduli at different percentages. For example, the variation in longitudinal modulus values ranged between 4 to 5.5 %, the variation in shear modulus values ranged between 2.3 to 11.9 %, the variation in bulk modulus values ranged between 2.1 to 4.5 % and the variation in Young's modulus values ranged between 2.1 to 10.7 %. Also, it is clear that L, G and E moduli values were increased until 20 wt.% of PCP addition in pure ER and then decreased slightly for 30 wt.% addition. However, bulk modulus has showed a linear increase in PET char powder addition. It is a well-known fact that the reinforcing materials at appropriate quantities can have a considerable effect on elastic properties of materials used as matrix. Therefore, the increase in the modulus attributed to the PET char powder addition.

Table 2. The elastic properties (*L*, *G*, *K*, *E*, *H*, *μ*, *Z*) of the pure ER and ER/PCP composites

Samples' ID	*L*, GPa	*G*, GPa	*K*, GPa	*E*, GPa	*H*, GPa	*μ*	*Z*, 10^6kg $m^{-2}s^{-1}$
ER	8.42	1.68	6.16	4.64	0.141	0.375	3.12
ER/PCP-1	8.76	1.72	6.29	4.74	0.145	0.374	3.16
ER/PCP-2	8.88	1.88	6.37	5.14	0.169	0.374	3.24
ER/PCP-3	8.85	1.80	6.44	4.95	0.154	0.362	3.22



a

b

c

d

Fig. 3. Variation of elastic moduli with wt.% amount of PET char powder in ER/PCP composites

As a summary of results related with elastic constants in Table 2, it can be stated that the best combination ratio for pure ER and PCP is ratio of 80:20 in wt.%.

A modulus of elasticity is the mathematical description of a material or component's tendency to be deformed elastically when a force is applied to it and is related to interatomic forces, the fracture, porosity, crystal growth and microstructural factors.Thus, it is evident that the minor changes in elastic modulus of the material are very sensitive to any minor compositional changes, and can be effectively monitored by ultrasonic methods. Therefore, monitoring such changes with the help of nondestructive methods would be useful.

3.3. Poisson's ratio

Poisson's ratio, defined as the lateral contraction per unit breadth divided by the longitudinal extension per unit length in simple tension [18], is reported to provide more information about the character of the bonding forces than any of the other elastic coefficients[19].

It has been reported that the Poisson's ratio decreased with increasing Young's and shear moduli, and it was maximum when elastic moduli were minimum [19]. Therefore, it can be stated that the decrease in Poisson's ratio is an indication of improved durability for materials against the external forces.

As it is seen in Table 2 and Fig. 4, the Poisson's ratio value is 0.375 for pure ER and Poisson's ratio values for ER/PCP composites ranged between 0.362 – 0.374 for composites of ER/PCP. It is clear that the Poisson's ratio of composites are lower than pure ER. As explained above, these results related with Poisson's ratio shows that the PET char powder addition into pure ER has improved the durability of ER against external impacts.

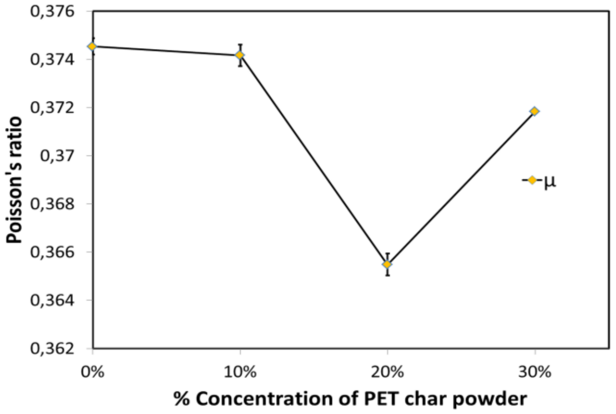


Fig. 4. Variation in Poisson's ratio for wt% amount of PET char powder in ER/PCP composites

In conclusion, the increase in elastic moduli and decrease in Poisson's ratio are in good agreement in this study and the results of Poisson's ratio are in good agreement of results for elastic moduli.

3.4. Acoustic impedance

Fig. 5 illustrates the acoustic impedance of ER/PCP composites as a function of the PET char powder. The acoustic impedance ranged between 3.16 x 10^6 to 3.24 x 10^6 kg.m^{-2}.s^{-1} for ER/PCP composites.

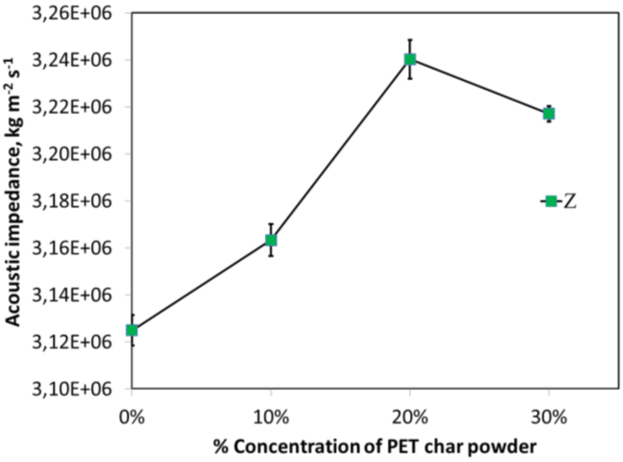


Fig. 5. Variation in acoustic impedances for wt% amount of PET char powder in ER/PCP composites

Acoustic impedances of all composites are higher than of pure ER. This result is in agreement to ultrasonic velocities and elastic constants. The composite labelled as ER/PCP-2 has the highest acoustic impedance value (3.24 x 10^6 kg.m^{-2} s^{-1}).

3.5. Ultrasonic micro-hardness

As it is seen from Table 2 and Fig. 6, the ultrasonic micro-hardness of ER/PCP composites ranged from 0.145 to 0.169 GPa. Also, forming ER/PCP composites have caused an increase in micro-hardness of pure ER. For example, the micro-hardness of pure ER has increased from 0.141 to 0.169 GPa by PET char powder addition ratio of 20 wt.%. The ultrasonic micro-hardness of ER/PCP composites increased with increasing PCP amount in ER/PCP composites until 20 wt.% and then decreased slightly by increasing PCP amount from 20 wt.% to 30 wt.%.

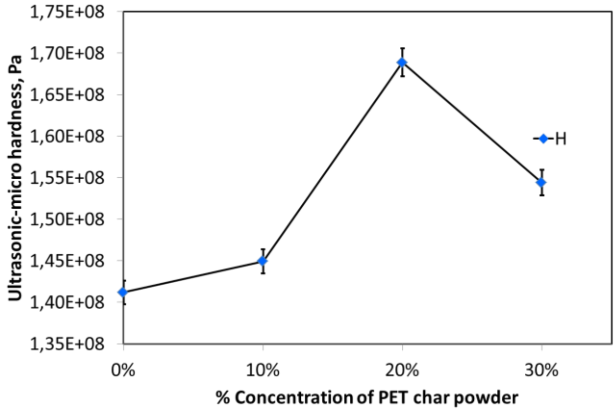


Fig. 6. Variation in ultrasonic micro-hardness for wt.% amount of PET char powder in ER/PCP composites

This result is also in agreement to the ultrasonic velocity values and elastic constants values obtained. Afifi and Hasan [20], have compared the ultrasonic-micro hardness values and mechanical hardness (Vickers) values of poly methyl metha crylate (PMMA). The difference between ultrasonic-micro hardness values measurements and mechanical hardness (Vickers) values measurements was seen about 9 %. This result prove that ultrasonic micro-hardness measurement is reliable.

3.6. Relationship of ultrasonic wave velocity, electrical conductivity and ultrasonic micro-hardness

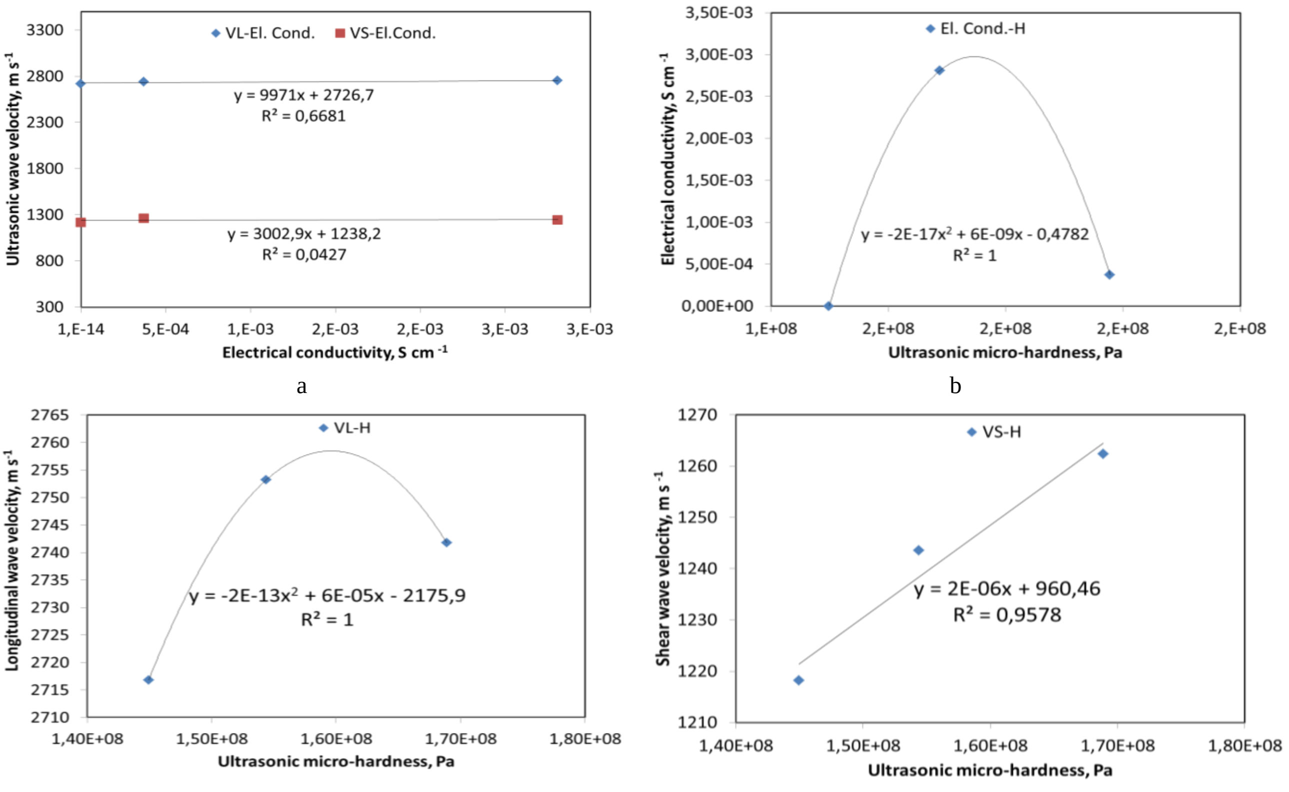
The linear correlation between electrical conductivity and ultrasonic wave velocities (V_L, V_S) was existed (Fig. 7 a). The correlation coefficient R^2 of about 0.66 was obtained between longitudinal wave velocity and electrical conductivity of ER/PCP composites.

On the other hand, the correlation coefficient R^2 of about 0.04 was obtained between shear wave velocity and electrical conductivity of ER/PCP composites. This result suggested that correlation between ultrasonic longitudinal wave velocity and electrical conductivity is higher than the correlation between shear wave velocity and electrical conductivity.

The polynominal correlation, which have correlation coefficient R^2 of 1, was seen between electrical conductivity and ultrasonic micro-hardness of ER/PCP composites (Fig. 7 b). From Fig. 7 b, it was clearly seen that the relatively strong relationship existed among hardness and electrical conductivity (R^2 of 1). It was observed that up to 0.154 GPa of hardness, the electrical conductivity shows rapid increase and after that point a rapid decrease was observed in electrical conductivity.

As it is known that linear regression assumes that the relationship between a dependent variable and a set of independent variables are additive, or linear. If the relationship is actually non-linear, the R^2 for the linear model will be lower than it would be for a better fitting non-linear model.

Therefore, it can be stated that the polynomial model fits the data quite good with an R^2 value of 1. Correlation coefficient R^2 of 1 was obtained between longitudinal wave velocity and ultrasonic micro-hardness hardness through 2nd order polynomial fit, as seen in Fig. 7 c. So, the graph between longitudinal wave velocity and ultrasonic micro-hardness was non-linear. The polynomial line depicts a rapid increase in longitudinal wave velocity up to 0.154 GPa of hardness and after that point a rapid decrease in longitudinal wave velocity, as shown in Fig. 7 c. Linear relationship of hardness with correlation coefficient of about 0.95 was calculated for shear wave velocity (Fig.7d). It was observed that ultrasonic shear wave velocity correlated more perfectly than any other parameters and could be used for hardness prediction with reasonable accuracy. The analysis of results suggested that correlation between ultrasonic longitudinal wave velocity and electrical conductivity is higher than any of other parametres, while the correlation between shear wave velocity and ultrasonic micro-hardness is much better than any of other variables.



a b

c d

Fig. 7. Correlation of conductivity, ultrasonic wave velocities (V_L, V_S) and micro hardness: a – conductivity- ultrasonic wave velocities; b – conductivity-micro hardness; c – longitudinal wave velocity- micro hardness; d – shear wave velocity- micro hardness

3.7. Morphological results

Popa et al. [21] emphasized that the interface between the filler or reinforcing material and the polymeric matrix is essential in polymeric composites.

According to the SEM image in Fig. 8, the char powder particles were homogeneously distributed throughout the surface at a higher magnification, which results in enhanced mechanical properties.Fig. 8 also shows a random distribution and partial clusters of char powder particles in the epoxy matrix, but they are well connected to the polymer matrix. However, Akcaozoglu et al. [22] have stated that the PET waste aggregate addition causes reduction at compressive strength, flexural-tensile strength and splitting tensile strength of cement-based composites. Because that elastic modulus is sensitive to minor chemical compositional changes it can be monitored accurately by ultrasonic methods. Therefore, the ultrasonic velocity measurement method is one of the best method to determine the elastic properties of the ER/PCP composites.

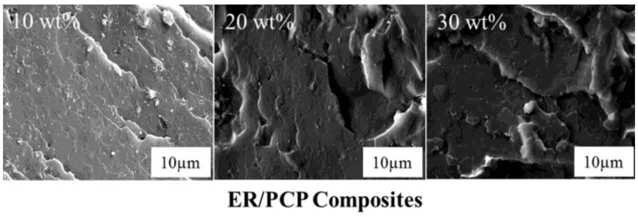


Fig. 8. Fracture surface images of the ER/PCP composites

4. CONCLUSIONS

From the above study the following conclusions can be drawn:

1. The highest ultrasonic velocities and elastic constants values were observed for composite sample labelled as ER/PCP-2.
2. According to the obtained results, the composition ratio of 80:20 is the most appropriate composition ratio for the ER/PCP composites.
3. It was observed that ultrasonic shear wave velocity correlated more perfectly than any other parameters with hardness and it can be used for hardness prediction with reasonable accuracy.
4. Elastic modulus is sensitive to minor chemical compositional changes and it can be monitored accurately by ultrasonic velocity measurement.
5. It can be stated that the pyrolysis of PET wastes is one of the best way to recycle the PET wastes.

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