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Influence of Graphite Waste Incorporation on the Properties of Epoxy Matrix Composites

Calis Acikbas N[1*], Ozcan S[2]

[1*]Department of Metallurgical and Materials Engineering, Bilecik Seyh Edebali University, Bilecik, Turkey
[2] Department of Chemical and Process Engineering, Bilecik Seyh Edebali University, Bilecik, Turkey

***Corresponding author**
Calis Acikbas N

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Abstract: The addition of filler materials to a polymer is a common practice to improve strength, stiffness, toughness, hardness, conductivity, wear resistance, as well as reducing the processing cost significantly. In this study the effect of waste graphite as a cost effective reinforcement in epoxy matrices for improving the mechanical properties was investigated. As the graphite content increased bending modulus, hardness and bulk density increased and bending strength and total porosity decreased.

Keywords: characterization, graphite waste, mechanical properties, composite, epoxy.

INTRODUCTION

Polymer matrix composites have a number of advantages due to their high specific strength and modulus, light weight, and ease of processing to name a few [1, 2]. Polymer matrix composites with such enhanced properties constitutes 90% of commercial applications with a wide range from sanitary wares to aerospace materials [2, 3]. Various kinds of materials have been suggested to reinforce composites as well as to decrease the production cost. Especially, waste materials are convenient candidates. In literature, glass fiber, urea formaldehyde, marble, porcelain, boron oxide, walnut shell, rubber, and gypsum containing wastes have been shown to reinforce polymer matrix composites [4-12].

Graphite is an alternative filler material for polymer matrix composites. Due to its weak bonds between the crystalline sheet layers it is an excellent dry lubricant [13], and due to its high electrical conductivity graphite based polymer composites also provides high electrical conductivity for electromagnetic shielding, batteries, light emitting devices, biosensors etc. [14,15]. On the other hand graphite reinforced composites have exceptional mechanical properties such as high stiffness, high elastic modulus, enhanced flexural strength, and light weight [16]. Thermal properties were also enhanced as the glass transition temperature was increase with the increasing amount of the graphite phase [18, 19]. The tribological behavior of these kinds of composites has showed that the graphite addition decreased friction and wear loss [17]. Hybrid composites of graphite with other materials like glass fiber were also investigated [20-23]. In the literature, the graphite content of polymer matrices studied were in the range of 0.5 to 5wt%.

In this study, a graphite obtained from a waste heating element was evaluated to be used as filler in the epoxy matrix, and the mechanical properties of the composites with high graphite content (30-60 wt%) were determined.

MATERIALS AND METHODS

Materials

The matrix material is formed by the reaction of the epoxy resin with the hardener. The epoxy resin and the hardener were supplied from Smooth-On Limited, Canada. A gas pressure sintering furnace waste heating element was used to obtain the graphite. Waste heating element material was milled, dry sieved of particles size below 45µm. Phase analysis of this material was carried out by XRD method (Panalytical Empyrean model). The density of waste material was measured by He-gas pycnometer. Particle size distribution of graphite waste was determined by the laser diffraction method.

Preparation of composites

The schematic presentation of the preparation of composites with different graphite loadings is given in Figure-1. Graphite filler amount in the composite was changed between 30 to 60 wt% (Table-1). The graphite weight percentages in the composite were taken into account in the specimen coding.

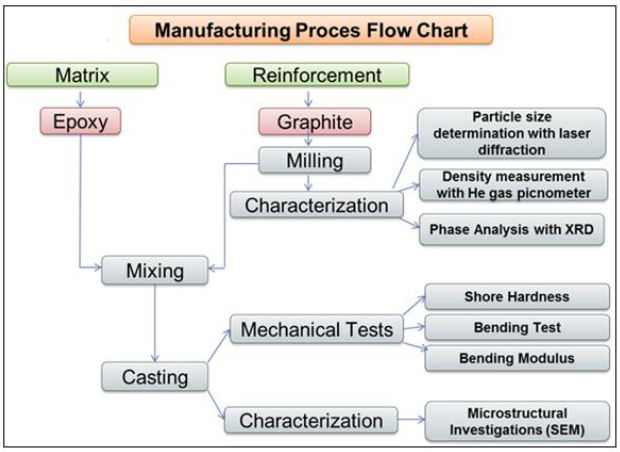

Fig-1: The composite manufacturing process flow chart and characterization steps.

Table-1: Composition of ingredients

Sample Code	Epoxy (wt%)	Graphite (wt%)
G30	70	30
G40	60	40
G50	50	50
G60	40	60

Casting method was used to shape and produce epoxy matrix composites. Polyvinyl alcohol (PVA) was used as a lubricant for the easy removal of the final product from the mold. Epoxy resin, hardener and graphite particles were mixed together at 500, 1000 and 1500 rpm for 5 min respectively. The composite mixture was vacuum desiccated removing any air bubbles formed during the mixing, in order to prevent due pores in the composite. The epoxy resin-filler blends were left for 24 hours in the initially lubricated silicon molds at room temperature for curing, after which the composite specimens were removed from the molds.

Physical, mechanical and microstructural characterizations

The theoretical densities of the composites were calculated using the theoretical density values and the volume fractions of the graphite waste material and the epoxy resin as given by by Eq. 1 [24]. The volume fractions were calculated from the known weight fractions.

$$\rho_c = (\rho_m * V_m) + (\rho_g * V_g) \quad \text{(Eq.1)}$$

where, ρc is theoretical density of composite samples, ρ_m is theoretical density of matrix, ρ_g is the theoretical density of waste graphite filler, while V_m is the volume fraction of the matrix, and V_g is the volume fraction of the respective filler.

The Archimedes principle was used to determine the density and the porosity of the specimens. Bulk density, % theoretical density and % total porosity were calculated by Eqs 2-4.

$$\text{Bulk Density}(\text{B. D.}) = \frac{W_1}{W_3 - W_2} * \rho_{water} \quad \text{(Eq.2)}$$

$$\%\text{Theoretical density} = \frac{\text{B.D.}}{\text{T.D.}} * 100 \quad \text{(Eq.3)}$$

$$\%\ \text{Porosity}(\text{Total}) = (100 - \%\ \%\text{Theoretical density}) \quad \text{(Eq.4)}$$

Where, W_1 is the dry weight, W_2 is the wet weight suspended in water, W_3 is the wet weight, B.D. is the bulk density, T.D. is the theoretical density of the samples.

The ShoreD hardness was measured for the 5cmx5cm specimens. The average of the five measurements was taken. Three point-bending strength tests of the samples were done in compliance with ASTM D 790 / ISO 178 standard. The average of five measurements was used to calculate the bending strength and bending modulus by Eqs 5-6.

$$\sigma_{bs} = \frac{3.P.L}{2.W.D^2} \qquad \text{(Eq.5)}$$

$$E = \frac{L^3.m}{4.W.D^3} \qquad \text{(Eq.6)}$$

Where, σ_{bs} is the bending strength (N/mm^2-MPa), P is the loading force (N), E is the bending modulus (GPa), m is the slope, L is the distance between the span (mm), W is the width (mm) and D is the test sample thickness (mm).

The scanning electron microscope imaging of the polished and fracture cross-section of the composite surfaces were used for the microstructural analysis. Secondary electron scanning electron microscopy images of composite materials were taken with FEG-SEM (Zeiss Supra 40 VP).

RESULT AND DISCUSSION

Figure 2 showed that waste material is graphite with 2H crytallographic structure. The density of the graphite was found to be 2,11 g/cm³ which was taken as the theoretical density. Figure-3 showed that the analysis average particle size is ~13 µm as determined by the laser diffraction technique.

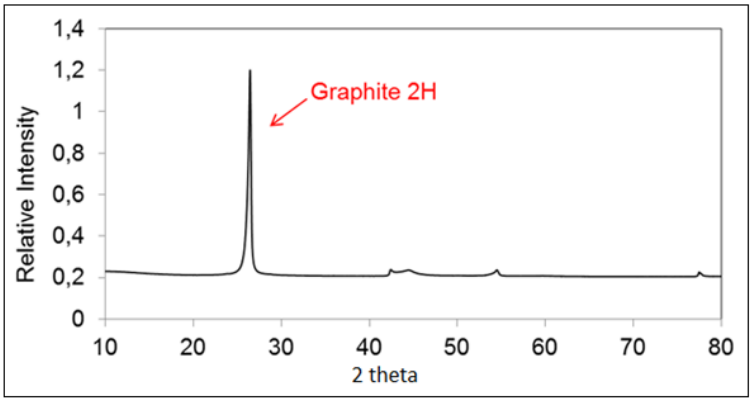

Fig-2: XRD spectra of graphite waste

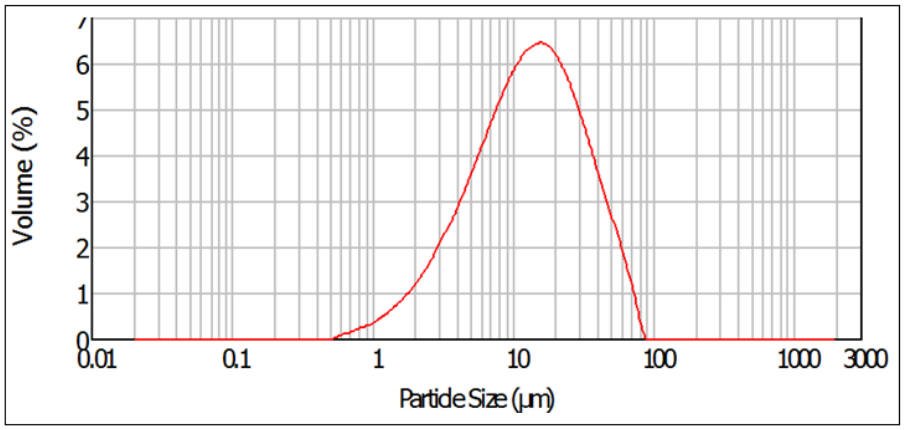

Fig-3: Particle size distribution graph of graphite waste

Determination of mixable epoxy: filler ratio

For the determination of optimum epoxy: waste ratio for casting, varying amounts of waste (epoxy: waste ratio 70:30, 60:40, 50:50, 40:60) was added into epoxy resin in order to observe processability of mixture (epoxy and waste). Observations related to the casting behavior of composite mixture were given in Table 2. For the composite with 30wt% waste the mixing was easy with little or no bubble formationand hence with little porosity evolution, there was no lamination problem, the viscosity was low and hence the casting was trouble free. When the waste amount was increased from 30 to 40wt%, the blend was still mixable with little porosity formation, without lamination problem, and the viscosity was low enough to obtain castable mixtures. Further increase in the waste amount to and above 50wt%, mixing was harder and caused high porosity in the specimens. While there was still no lamination problem, the viscosity became too high for a smooth casting. Due to the significant loss of fluidity, filler additions over 40 wt% were not efficient. Therefore, it was concluded that 60 epoxy: 40 waste filler ratio was an optimum in achieving low cost and processable composites. A longer time of mixing or sonication processes might have assisted in providing a more homogeneous distribution of the waste in the polymer matrix for all of the compositions.

Table-2: Observations for the preparation of composite mixture of various epoxy: waste ratios

	G30	G40	G50	G60
Observations	Easy mixing Low porosity Low viscosity Easy casting	Optimum mixing Low porosity Medium viscosity Optimum casting	Difficult mixing Medium porosity High viscosity Difficult casting	Very difficult mixing High porosity Very high viscosity Very difficult casting

Physical properties of composite materials

The physical properties of the composites in terms of bulk density, theoretical density, %theoretical density and %total porosity is given in Table 3. The bulk densities of composite were changed between 1.31 gr/cm^3 for the G30 to 1.54 gr/cm^3 for the G60. Bulk density was affected by the existence of open and closed pores. So for the materials with high bulk density, total pore amount is expected to be low. Accordingly, the bulk density, theoretical density, % theoretical density values increased and total porosity level decreased with the increasing filler waste graphite content. Representative SEM images of composites are given in Figure-4. As seen from the SEM images, low concentration graphite containing composite (G40) had a higher porosity than the higher amount of graphite containing composite (G60). The results was in compliance with the total porosity values which were obtained by Archimedes principle.

Table-3: Theoretical density, bulk density, %total porosity %theoretical densities of the composite samples

	G30	G40	G50	G60
Bulk Density (g/cm³)	1,31	1,38	1,45	1,54
Theoretical Density (g/cm³)	1,85	1,88	1,92	1,96
% Theoretical Density	70,50	73,10	75,17	78,60
% Total Porosity	29,50	26,90	24,83	21,40

Mechanical Properties of Composites

Bending strength values of the graphite waste based composites ranged from 82 MPa to 48 MPa and bending modulus ranged from 3.86 GPa to 5.22 GPa. Bending strength value of epoxy matrix was 106 MPa. Addition of waste particles into epoxy resin led to a decrease in bending strength due to the brittleness induced by the filler (Fig. 5). The processability of the composite mixtures was increasingly getting harder with higher graphite concentrations (over 50wt.%). Therefore, a decrease in bending strength was more pronounced in composite that contained 60wt% graphite waste. Since the interface between the matrix and the filler phase was weakened as the filler amount increased, the bending strength decreased accordingly.. Similar results were given for the polymer matrix composites in the literature [25, 26].

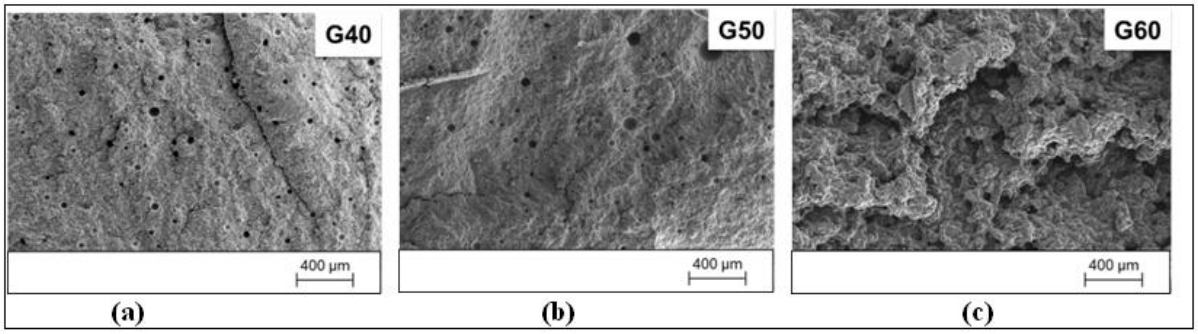

Fig-4: Representative SEM-SE images of fracture surface of composites (a) G40, (b) G50 and (c) G60

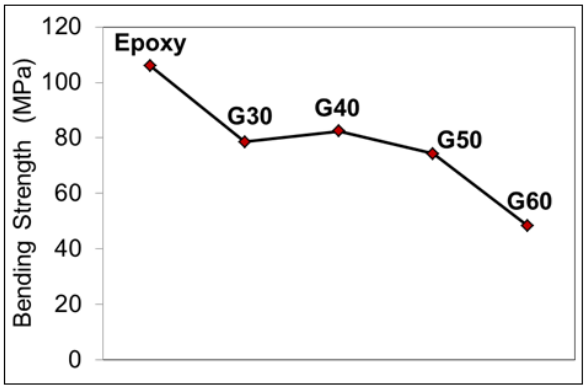

Fig-5: Change in bending strength values with graphite amount in the composite

However the bending modulus improved with the addition of the graphite waste particles into the epoxy resin. Besides, with the increase in the waste ratio bending modulus showed small increases (Fig.6). Therefore, it can be concluded that the waste graphite filler led to an increase in rigidity as revealed by the enhancing bending modulus. The bending modulus was also affected by the porosity. A decreasing porosity (29.5% for G30 and 21.4% for G60) in the microstructure caused an increase in the bending modulus.

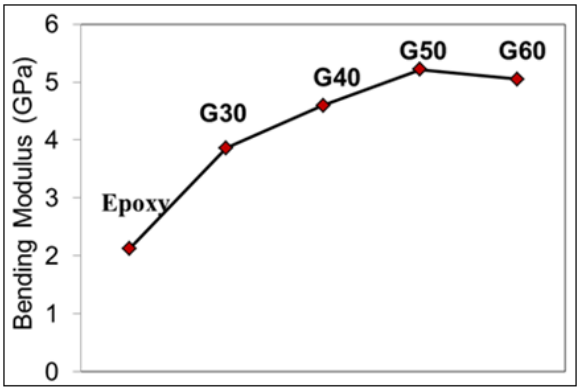

Fig-6: Change in bending modulus values with graphite amount in the composite

Incorporation of graphite filler into polyester matrix led to an enhancing hardness (Fig-7). The hardness was affected by the reinforcement phase hardness, porosity and the matrix-reinforcement connectivity. As the porosity values decreased with increasing filler content, the hardness values increased with the increase in the filler content.

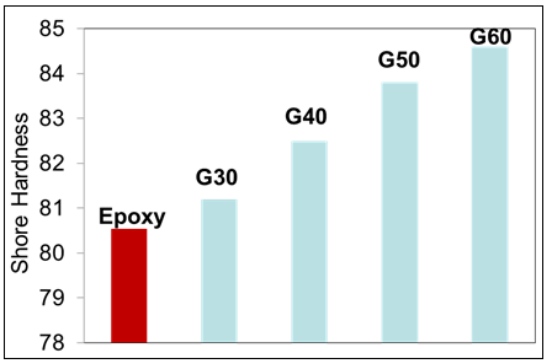

Fig-7: Change in hardness values with graphite amount in the composite

CONCLUSIONS

Optimum pourable epoxy: graphite ratio was found to be 60: 40 wt%. With a higher graphite content while the total porosity was reduced, the bulk density and the theoretical density attained higher values. Addition of graphite particles led to an increase in hardness. Bending modulus values increased up to 50wt% graphite particle loading. Further increase in the graphite content induced a decrement in the flexural modulus due to the inherent faults in the microstructure. The best bending strength value was obtained with 60:40 wt% epoxy: graphite ratio as 85MPa.

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