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THERMAL AND MORPHILOGICAL BEHAVIOR OF MECHANOCHEMICAL DEVULCANIZED GROUND TIRE RUBBER (GRT) FILLED EPOXY COMPOSITES

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ABSTRACT

In this study mechanochemical devulcanized ground rubber tire powder (GRT) was used to prepare the composites of epoxy by varying the GRT content from 1-12 phr. The structural characterization of the composites was done with FT-IR. Thermal characterization by DSC and TGA was carried out for all the compositions. The increased thermal stability of these composites with increase in GRT content was observed with dynamic thermogravimetry in Air. The fractured surfaces of these cured composites were examined by scanning electron microscopy (SEM). These studies reveal good dispersion and interaction of GRT in the epoxy matrix.

Keywords: Epoxy/GRT Composites; FT-IR; DSC; TGA; SEM

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INTRODUCTION

Epoxy resins are well known class of thermosetting resins. These polymers have an excellent physio-chemical, bonding, mechanical and thermal characteristic[1] which makes them available in a variety of applications such as coatings, encapsulating materials, adhesives, casting, molding resins, and matrices in reinforced polymer composites[2]. The processability becomes difficult once the epoxy resins are cured[3] and its mechanical and thermal characteristics are not sufficient to meet the widespread applications. To improve its thermal stability and working performance further, modification of epoxy resins becomes essential. One of the effective approaches is the use of different types of filler material including silica[4], minerals[4], carbon fiber[4], fiberglass[5] and ground tire rubber[6]. The ground rubber tire (GRT) finds a great attention for its applications as filler for rubber[7], thermoplastics[8] and thermosets[9]. The particle size, surface characteristics and chemical composition of GRT have a significant effect on the curing of thermosets and other properties of GRT filled composites[10]. Although the use of GRT as filler in polymer composites is a potentially attractive approach but it is fraught with a number of difficulties which arises due to its cross-linked and compositionally complex nature which can contribute to the final properties of the particulate composites. In this work we attempt a study on the effect of addition of mechanochemical devulcanized GRT particles on the thermal and morphological behavior of composites of GRT filled DGEBA epoxy resin. The structural characterization of the composites was done with FT-IR. The thermal stability and thermal transition of epoxy GRT composites were analyzed using Differential scanning calorimetry (DSC) and Thermal gravimetric analysis (TGA). SEM studies were undertaken on the cryofractured surfaces of the epoxy composites to investigate the morphology and interaction.

EXPERIMENTAL

MATERIALS

The epoxy resin, diglycidyl ether of bisphenol-A (DGEBA, FLOROPOXY 4805) was supplied by FLOROCK. GRT, PolyDyne (80 Mesh) were obtained from by Lehigh Technologies.

COMPOUNDING AND CURING OF EPOXY RESIN-GRT

Compounding of epoxy with GRT in different Composition (Table I) in parts per hundred parts of resin (Phr) was carried out in a round bottom flask at 120 °C/ 3h, at a constant stirring speed. The composites were cured at 220 °C.

INSTRUMENTATION

Infrared (FT-IR) Spectra were recorded on Nicolet FT-IR-100 using KBr salt plate. Mettler Instruments Toledo TGA/SDTA 851 was used with a heating rate of 10 °C/min from 25 °C to 700 °C under an air flow. The glass transition temperature (T_g) of the samples was determined by using Differential Scanning Calorimetry (DSC) performed with a TA Instruments Q1000 in the temperature range of 25 °C to 250 °C at a heating rate 10 °C/min under a nitrogen flow. SEM based on Hitachi S-3400 was used to do the Scanning Electron Microscopy on cryofractured surfaces coated with silver.

RESULTS AND DISCUSSION

FOURIER TRANSFORM INFRARED (FT-IR) SPECTROSCOPY

The FT-IR spectroscopy measurements (% Transmittance Vs Wave number cm^{-1}) helped in monitoring the chemical reaction i.e. cross linking and functionality changes during the curing process. The spectra obtained from neat epoxy and representative composites are shown in Figure 1. Neat epoxy shows peak at 971 and 915 cm^{-1} due to oxirane functionality which disappearance in the GRT filled Epoxy composites show the chemical reaction between epoxy and GRT. The effect of these interactions can be seen on the thermal stability of the composites.

THERMAL GRAVIMETRIC ANALYSIS (TGA)

The TGA thermograms of epoxy and composites are shown in Figure 2. Table II shows the T_{onset}, T_{max} and T_f values of neat epoxy and composites. All the composites showed two steps decompositions while single step degradation was observed in neat polymer (Figure 2, Table II). There is observable enhancement in thermal stability of composites. T_{onset}, T_{max} and T_f values are higher than those of the neat epoxy matrix as well as the neat GRT. T_{onset} increases with GRT content in the composites. At 12 Phr, the T_{onset} is 166°C higher than that of epoxy. The increase in T_{onset} indicates a thermal stability in the composites which can be attributed to the chemical interactions such as cross linking, which increase the average effective bond strength and/or the intermolecular attraction and thus stabilize the composites against thermal degradation.

DIFFERENTIAL SCANNING CALORIMETRY (DSC)

The DSC thermograms obtained for neat epoxy and GRT filled composites are shown in Figure 3. An increase in glass transition temperature, T_g is observed with increase in GRT composition. An increase in T_g can be attributed to the chemical interactions such as cross linking taking place in the composites matrix. The T_g of the polymers is controlled by the inherent chemical structure of the resin and the degree of crosslinking[11].

SCANNING ELECTRON MICROSCOPY (SEM)

The SEM photomicrographs of cryofractured samples of epoxy and Epoxy/GRT composites are shown in Figure 4. No phase contrast was observed in the GRT and epoxy in the composites. The fracture surface patterns of the composites appear to have local shear deformation. Although few definite cut edges were seen at scattered points at low concentration epoxy/GRT composites but at higher concentration, the local yielding and shearing seems to be prevalent to some extent[12].

CONCLUSION

The Mechanovulcanized GRT rubber powders can be utilized in getting composite materials with improved thermal stabilities and curing characteristics. The thermal stability of epoxy/GRT composites shows a higher thermal stability over epoxy resin with a margin of about 166 ºC. The higher T_g of epoxy/GRT composites was observed by DSC. The interaction of epoxy matrix in epoxy/GRT composites was confirmed by spectral technique. SEM photomicrographs of cryofractured surface suggest the local shear yielding of matrix which is an indication of the effective interaction of the GRT with epoxy matrix.

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TABLE I

FORMULATION USED FOR COMPOUNDING

Sample Code	Epoxy Resins, (g)	GRT, (g)
PP0	100	0
PP3	100	3
PP5	100	5
PP9	100	9
PP12	100	12

Table II

DECOMPOSITION TEMPERATURES OF NEAT EPOXY AND EPOXY/GRT COMPOSITES

Sample	$T_{Onset,}$ (˚C)	$T_{max,}$ (˚C)	$T_{final,}$ (˚C)
PP0	164.6	320.1	363.3
PP3	298.1 (445.4)[a]	367.5 (499.6)	392.0 (537.7)
PP5	307.4 (446.1)	381.2 (514.2)	407.7 (555.8)
PP9	320.0 (448.0)	383.7 (518.6)	412.1 (557.1)
PP12	330.1 (456.9)	397.6 (527.5)	423.4 (569.7)

( )[a] : TGA parameters for second step degradation

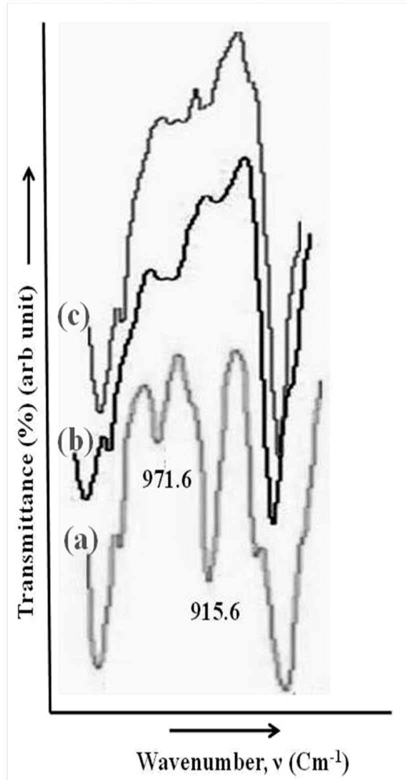

FIG. 1. -- FT- IR spectrum of epoxy (a) epoxy /GRT composites, (b) (PP5); (c) (PP12).

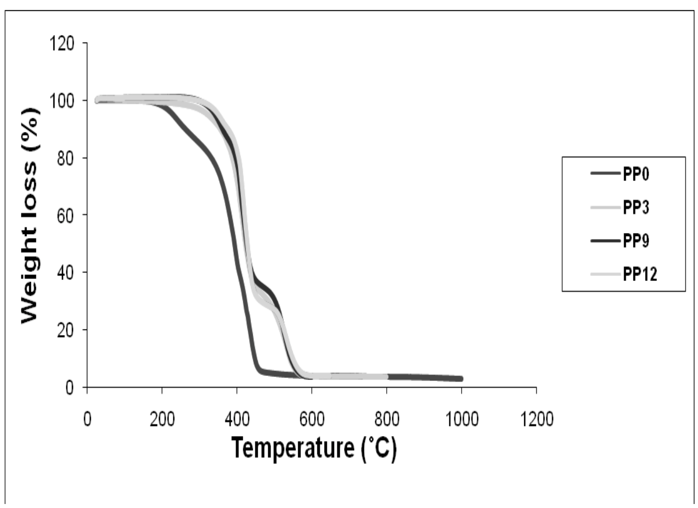

FIG. 2. -- TGA scans for neat epoxy (PP0) and epoxy/GRT composites by varying GRT composition.

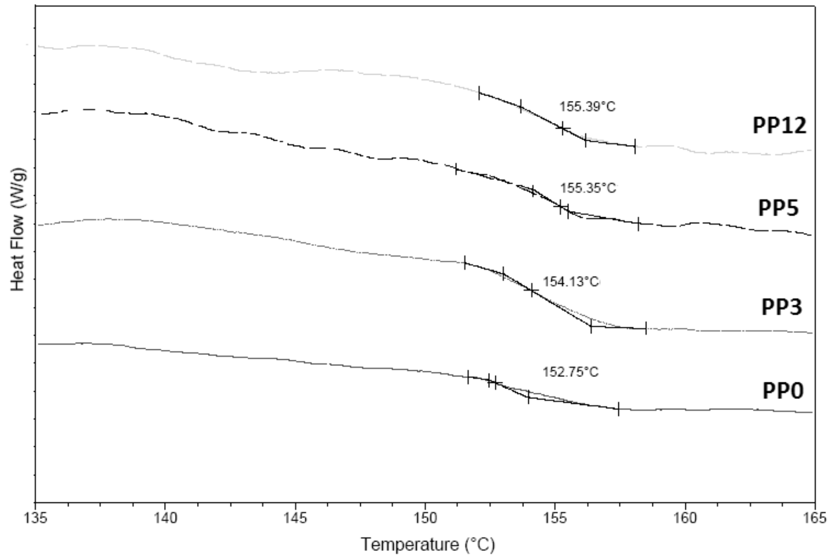

FIG. 3. – DSC scans showing Glass Transition temperature (T_g) for the neat epoxy (PP0) and different GRT composition

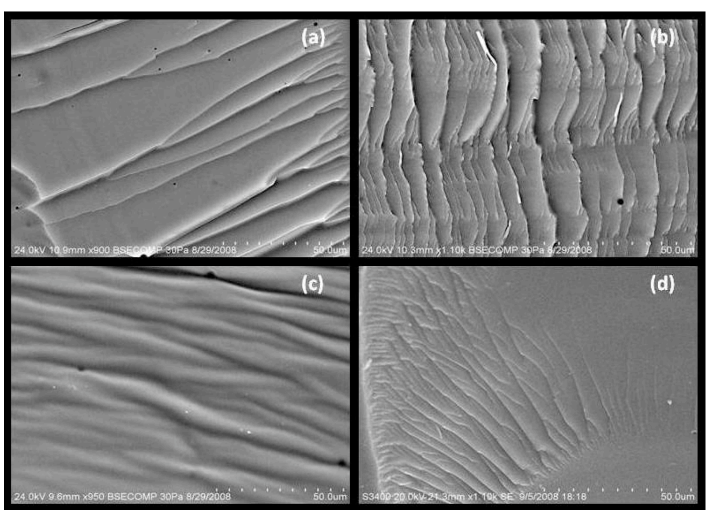

FIG. 4. --SEM photomicrographs of epoxy GRT composites at different Phr a) PP3 b) PP5 c) PP9 d) PP12.