

Thermal, Morphological Analysis of Epoxy/Unsaturated Polyester Blended Nanocomposite with Wollastonite Powder as a Particulate

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Received: 8 April 2019;

Accepted: 3 May 2019;

Published online: 31 July 2019;

AJC-19499

In the proposed research article, polymer nanocomposites on the basis of thermoset polymer resultant blends (epoxy/polyester) having wollastonite powder are prepared by high shear mixer (mechanical), followed by an ultra-sonicator for uniform dispersion of wollastonite particulates in the final blend matrix. Wide range percentages of modified wollastonite nano-particulates with 0, 1, 2, 3, 4 and 5 wt. % were incorporated into the epoxy/polyester blend matrix in order to evaluate the effect of nanofillers on nanophase morphological structure and to study material properties. Various thermal parameters of the nanocomposite were evaluated using thermogravimetric analysis and differential scanning calorimetry techniques. Further, the scanning electron microscopy images have been employed to know fractured surfaces of the titled compound. From the experimental data it was found that uniform miscibility of epoxy/polyester blend mix and homogenous dispersion of wollastonite nano particulates in the blend matrix was noticed. From the TGA studies, 8 % weight loss and 4 °C rise in decomposition temperature was observed with addition of 5 wt. % wollastonite when compared with the 0 wt. % wollastonite combination of epoxy/unsaturated polyester resin blend. DSC results further revealed that the modified wollastonite nanoparticles is major responsible for the glass transition temperature (T_g) of the nanocomposites.

Keywords: Nanocomposites, Blends, SEM, TGA, DSC.

INTRODUCTION

Superior properties, affordable cost and multiple use of polymers are needed to meet the current requirement of the society and therefore by particularly in polymer industries. Polymers are generally amorphous or semi-crystalline in nature. A semi-crystalline polymer has majorly an amorphous and lower in crystalline portion. As a result, lower portion of crystalline part, has more or less ordered structure wherein the chains of the polymer are often folded in a non-random form. Thermal properties of the semi-crystalline polymers are strongly come to know by the crystallites, which normally improve their stiffness and thermal stability (for example in polypropylene). Polymers having amorphous in nature are either brittle (polystyrene) or high in toughness (polycarbonate). It is very tough to judge the mechanical properties of a semi-crystalline material keeping in view of its determined by many parameters like its percentage of crystallites. Therefore, it is very easy to examine

the mechanical properties of an amorphous polymer. Unfortunately as the current requirements in different research areas could not meet the expected properties by the existing single polymer and hence look for enhanced properties containing materials. To meet these requirements, one of the methods to be adopt to get desired product by mixing two or more polymers with different compositions. Mixing two or more polymers to get desired resultant properties of blends or alloys is a well established path to attain a certain feasible physical and thermal properties, without any synthesise route for specialized polymer resins. Thermal analysis is one of the versatile techniques to measure various physical properties of the polymer blends at different temperatures. Various studies had been conducted on the thermal properties of polymers, polymer blends and nanocomposites. Louis *et al.* [1] indicated that rubber and polysulfone blends exhibited partial miscibility with good mechanical and thermal properties. The behaviour of the new materials and their quantitative measurement of the

physical change with respect to temperature is a great agreement for helpful information with respect to the nature of materials. Therefore, thermal techniques played an important role in many research fields of basic and applied research specifically in production and quality control in the preparation of materials particularly in polymer composites. Lee *et al.* [2] indicated that the addition of clay and wood flour to polypropylene increased the mechanical properties as well as the decomposition temperature and melting temperature of the hybrid composite. Chen *et al.* [3] highlighted on the thermal properties and flame retardancy of an ether-type UV-cured polyurethane coating materials. In all the polymeric material, temperature is unique parameter which influences to vary the physical and mechanical properties significantly that enhances its useful application and end-use performance. Several research articles reveals that number of authors has been devoted on various experimental works in this direction [4-9]. The principle aim of the proposed research work is to explore the influence of wollastonite on blended polymers to achieve thermal stability. In the present study epoxy/polyester blended nanocomposites were fabricated and characterized as a function of wollastonite composition.

EXPERIMENTAL

The materials used in the present study such as epoxy (Araldite-LY 556) along with hardener (Aradur HY-951) supplied by M/S Huntsman, Sree industrial composite products, Hyderabad and unsaturated polyester (Ecmalon-4413) supplied by M/S Ecmas India Pvt Ltd, Hyderabad. The reinforcement filler wollastonite powder (Kemolit-KF4) received from M/S Wolkem industries India Ltd, Rajasthan. The epoxy resin along with hardener mixed with unsaturated polyester were used for the preparation of composite with 2 % cobalt naphthanate as an accelerator, 2 % methylethyl ketone peroxide (MEKP) as a catalyst in 10 % dimethyl acetamide solution as a promoter. The ratio of epoxy: unsaturated polyester resin: accelerator:catalyst:promoter will be 85:15:2:2:2 in weight percentage. The test specimens were fabricated by adopting wet hand-layup technique. In addition, wollastonite powder (Kemolit-KF4) surface was modified (treated) by using 25-30 % trimethyl stearyl ammonium as a nano filler material.

Fabrication of blended nanocomposites: Initially, wollastonite nanomaterial kept in an oven at 100 °C for 2 to 3 h in order to eliminate the moisture content. Then pre-calculated amount of epoxy/unsaturated polyester (*i.e.* 85:15 ratios) was mixed in a suitable beaker. Wollastonite powder (nano particulate material) is mixed to a specific amount of epoxy/polyester by using a known ratio and mixed vigorously with the help of mechanical stirrer for about 1 h duration at room temperature. Then the epoxy/polyester and wollastonite mix was placed in a high intensity ultrasonicator with duration of 90 min under the pulse mode of 15 min on/15 min off. In order to avoid the internal temperature development during the ultra-sonication process, the reaction mix was kept in water bath. Consequently, hardener/accelerator/catalyst/promoter in the proportionate of 100:10:2:2:2 parts by weight were mixed to the tailored epoxy/polyester ratio. A glass mould was prepared on par with ASTM standards for making of desired laminates and mould releasing

agent is applied on the glass surface with wax in order to remove the laminate specimens without any dimensional fractures after the curing process. For obtaining of uniform size and smooth surface, enough pressure is applied by using the hand roller to impregnate the nanocomposite systematically. Then, an appropriate load was applied with a pressure for duration of 24 h at room temperature. In order to confirm the complete curing process, the blended nanocomposite samples were post cured at 70 to 90 °C for 1 h and the test specimens were prepared as per the ASTM standards to evaluate for different mechanical properties using various tests procedures. Samples of pure blend and wollastonite mixed blend were prepared.

Thermal analysis: The epoxy/polyester composite is synthesized by using surface treated wollastonite powder as nano filler. The resultant composite material is characterized by using both thermogravimetric analysis (TGA) (TGA Q500 model) and differential scanning calorimeter (Netzsch -Model DSC 204 F1 Phenoix) techniques. TGA technique was employed to examine thermal decomposition nature of the nanocomposite blend. Further, differential scanning calorimeter utilized to find the glass transition temperature (T_g) of the investigated composite material. The experimental tests were performed in inner atmosphere by using nitrogen at a scan rate of 10 °C/min in a wide range of temperature with definite intervals from 30 to 500 °C. A sample with definite weight of 5 to 10 mg employed for each measurement and weight change was measured and recorded as a function of temperature.

Scanning electron microscopy analysis: A model of JEOL JSM-6400, scanning electron microscope (SEM) at 15 kv accelerating voltage combined with energy dispersive spectroscopy (EDS) was employed to evaluate the dispersion of wollastonite particles and miscibility nature of the resultant blend. In order to enhance the conductance of the fractured sample surfaces, gold coated thin film was used.

RESULTS AND DISCUSSION

Thermal analysis: Thermogravimetric analysis technique was employed to observed weight/mass loss curves for five different blended nanocomposite materials samples (*i.e.* with 0, 1, 2, 3, 4 and 5 wt. % wollastonite) and resultant curves are depicted in Fig. 1. For neat blend (without filler) weight loss was constant up to 200 °C and then decomposition starts at 400 °C. From the experimental results, it is clear that the decomposition temperature of the blended nanocomposite moved towards the upper temperatures as wollastonite amount increased up to 5 wt. %. Weight loss was constant for 5 wt. % wollastonite blended up to 350 °C. The weight loss is mainly due to elimination of moisture content in the tested 1 wt. % wollastonite. Therefore, it is obvious that the decomposition temperature of the investigated composite moved towards upper temperature shows better thermal stability of the polymer up to 5 wt. % wollastonite. The presence of an inorganic material as filler in the polymer matrix, generally, considerably improves the thermal stability of the polymer nanocomposite. Based on the weight-loss temperature curve indicates that the non-decomposable polymer blend beyond 450 °C is on par with the inorganic filler material each sample. These observations proves that a better interface and chemical bonding

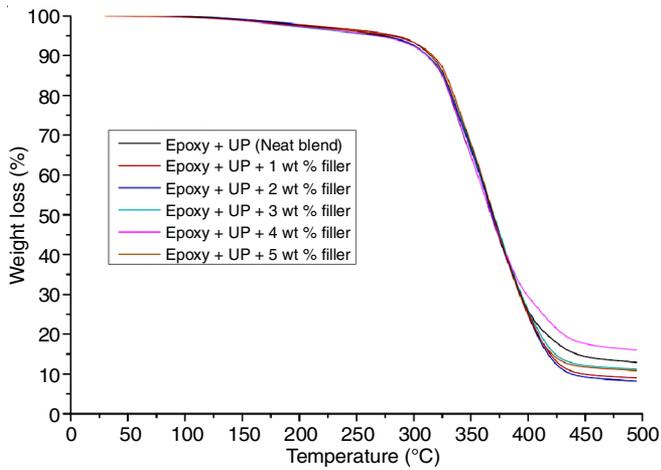


Fig. 1. TGA results of epoxy/unsaturated polyester blend as a function of wollastonite

between epoxy/polyester resin mixed with nano filler led to the enhanced thermal stability. Addition of 5 wt. % wollastonite in a resultant polymer blend shown an 8 % weight loss and 4 °C rise in decomposition temperature as of pure blend.

From differential scanning calorimeter, typical thermograms are obtained for epoxy/polyester blended nanocomposites with five different loadings *viz.* 0, 1, 2, 3, 4 and 5 wt. % wollastonite was shown from Fig. 2. The glass transition temperature (T_g) are 150.8, 149.7, 145.2, 144.8, 147 and 147.8 °C and decomposition temperature of blended nanocomposites are 348.6, 350, 350.4, 351.1, 349.6 and 349.1 °C was found to be various amounts of wollastonite *viz.* for 0, 1, 2, 3, 4 and 5 wt. % of wollastonite. As a result, the highest heat flow and lower in glass transition temperature was recorded. Further, with addition of wollastonite the T_g value does not shifted. From thermal graphs depicted in Fig. 3, clearly observed that the

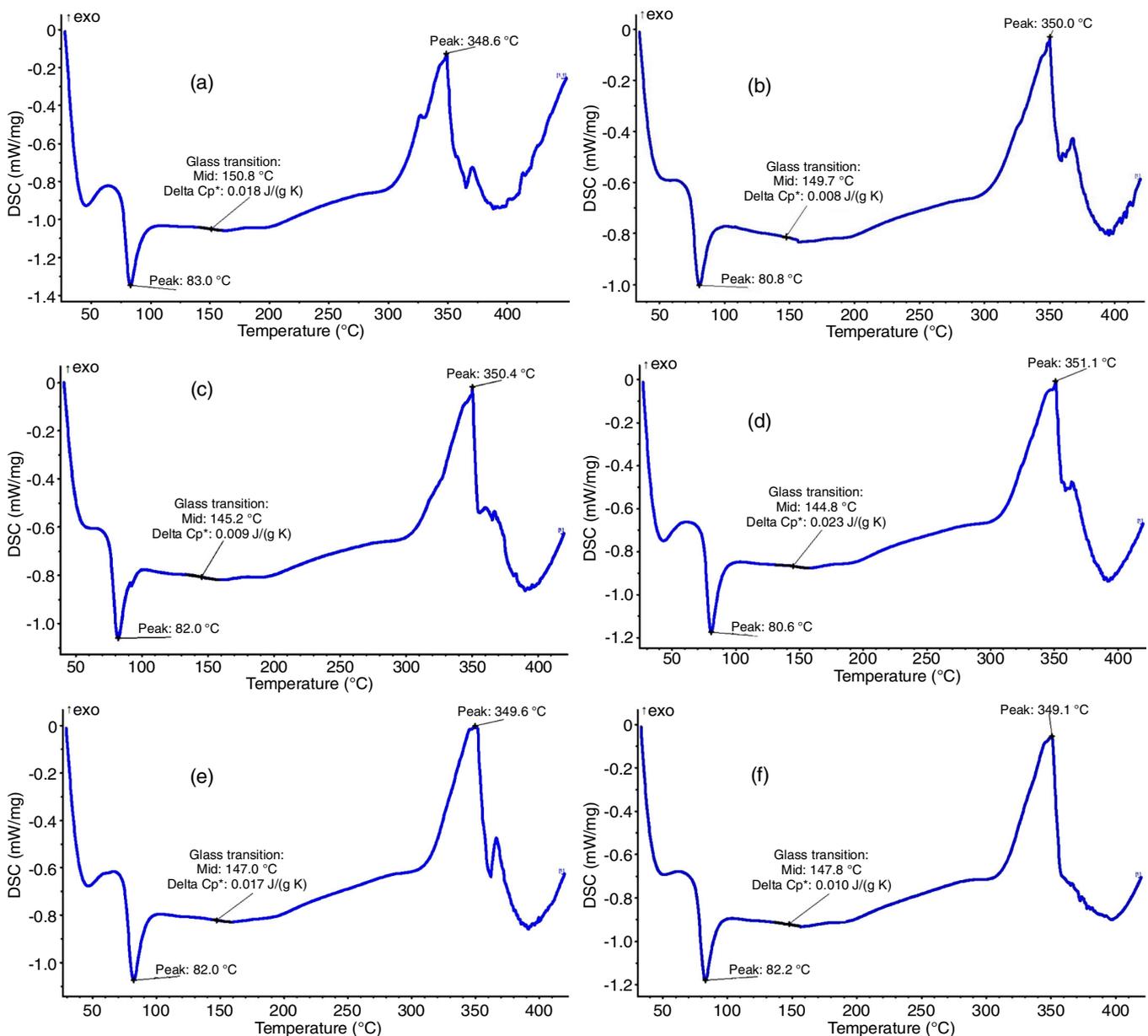


Fig. 2. DSC graph of (a) pure blend composite, (b) 1 wt. %, (c) 2 wt. %, (d) 3 wt. %, (e) 4 wt. %, (f) 5 wt. % wollastonite in blended nanocomposite

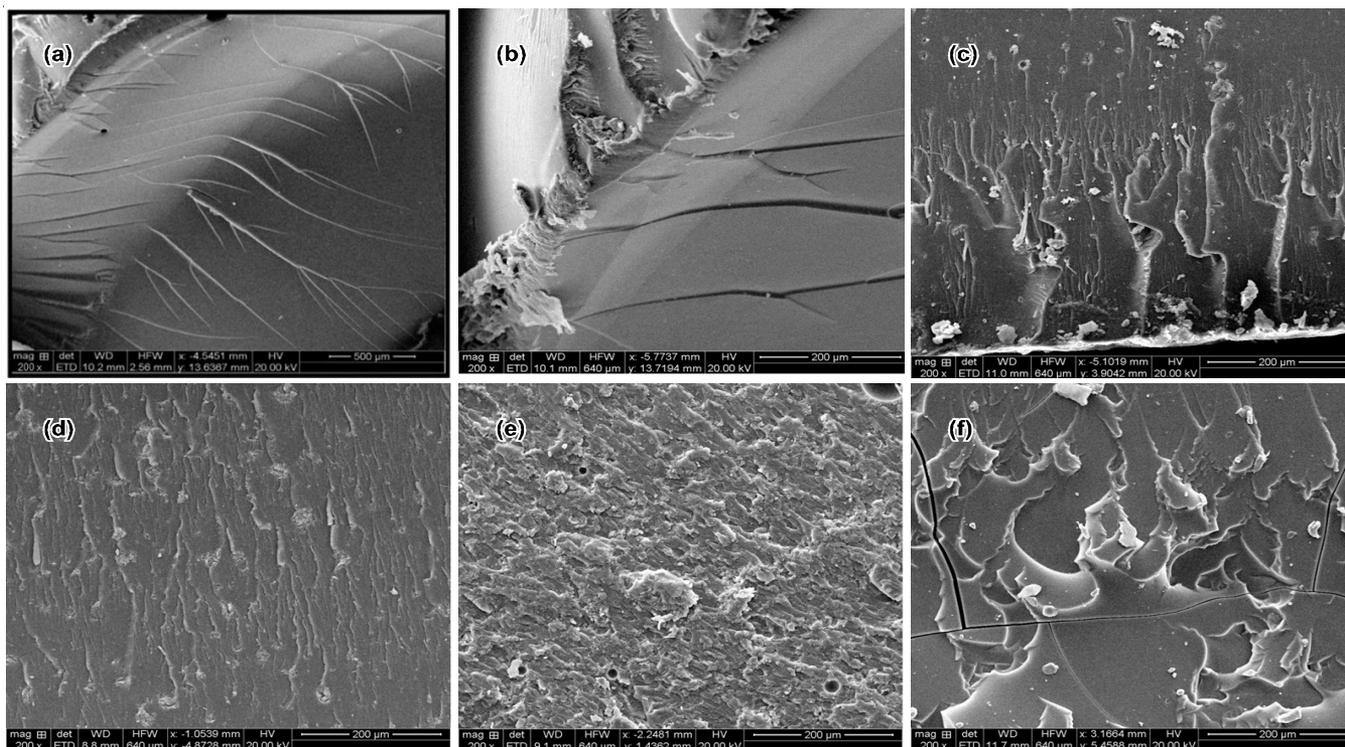


Fig. 3. SEM micrograms showing the fractured surfaces of (a) neat blend, (b) 1 wt. % filler, (c) 2 wt. % filler, (d) 3 wt. % filler, (e) 4 wt. % filler, (f) 5 wt. % filler

disintegration temperature of 4 °C lowered with the addition of 5 wt. % wollastonite when compared with 3 wt. %, whereas little change was observed between the 2 and 3 wt. % wollastonite contents. An endothermic maxima is at 351.1 °C is noticed for the resultant blended nanocomposites.

Morphological observations: SEM images of epoxy/polyester blends having various amounts of wollastonite are shown in the Fig. 3. The pure blend sample shows brittle fracture surface, indicative of miscible characteristics between the epoxy and polyester in the Fig. 3a. Further, the addition of wollastonite improved the homogeneity of the blend as increased wollastonite content turned out brittle fracture surface to ductile fracture surface up to 3 wt. % wollastonite that can be found by observing the SEM micrograms as represented in Fig. 3b-d. From Fig. 3e it was observed that there are microvoids, blow holes are observed. In Fig. 3f the crack initiation was found at 5 wt. % wollastonite content that indicates due to the increase in viscosity, causing entrapment of the air voids in the investigated composite. The dispersion particles appeared to be enriched in the matrix and are barely detectable at the magnification level employed. At 3 wt. % wollastonite blends there was strong indication of superior adhesion among the matrix blend and wollastonite due to the improved dispersion. On other hand, poor adhesion was observed beyond 3 wt. % wollastonite blend nanocomposites.

Conclusion

In the present work epoxy/polyester blended nanocomposites were prepared as a function of wollastonite particulates. The thermal properties of the wollastonite modified blend are studied. Addition of 5 wt. % wollastonite causes to increase 8 % weight loss and 4° C rise in disintegration temperature

was observed when compared with pure blend. The investigated polymer blend of DSC data revealed that glass transition temperature (T_g) was increased up to 3 wt. % wollastonite content, but corresponding improvements were not found at 4 and 5 wt. % wollastonite. The SEM micrograms showed the better distribution of wollastonite particulates in epoxy/unsaturated polyester blended composite at 3 wt. %.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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