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Full Length Article

Effect of functionalized metal oxides addition on the mechanical, thermal and swelling behaviour of polyester/jute composites

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ABSTRACT

The unsaturated polyester composites were fabricated in hand lay-up method by reinforcing with jute fibre along with alumina or zirconia particles in different filler loading *viz.* 5, 10, 15 and 20 wt%. It was observed that with incorporation of fillers, the microhardness value of the resulting composites increases and reaches its maximum at 20 wt% filler content. Characterizations were performed on the composites fabricated with overall 20 wt% filler content (18 wt% fibre and 2 wt% metal oxide particles). Various characterizations like Vicker's microhardness testing, scanning electron microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDS), X-ray Diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, thermogravimetric (TG) analysis, differential scanning calorimetry (DSC), limiting oxygen index (LOI) testing and water absorption test were performed. Tensile, Flexural testing were also performed on the normal and water absorbed samples. SEM analysis ensured good dispersion of filler within the polymer matrix. EDS and XRD were performed to identify the filler in the composites. FITR spectroscopy revealed the bonding of fillers with the matrix. TG analysis showed that thermal stability, degradation temperature of jute-ZrO_2 composites were best over the others. LOI testing also shows similar trend, showing better fire resistant property of jute-ZrO_2 composites than the Al_2O_3 dispersed. Water absorption test indicates the stability of different composite in various atmospheres (normal, boiling, simulated marine, alkali and acid water).

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1. Introduction

Unsaturated polyester (UP) resin are widely used as a low cost, room temperature curable thermosetting industrial resin for fibre reinforced composites which encompasses affluent processability and also possesses excellent mechanical properties. Styrene is often used as a reactive diluent for UP resin which one hand serves to wet the reinforcing fibre well by lowering the viscosity of the mixture and on the another hand helps to crosslink C=C bonds in different UP chains. Thus, use of styrene enhances mechanical properties of the resin effectively [1]. Environmental concerns, energy issues, reduction in the use of petroleum products have forced the researchers to think for an alternative reinforcing resource instead of the traditional synthetic fibres for the composites. In this context natural fibres have drawn the attention due to their low cost and extensive availability. Moreover surface modification of these fibres assists in achieving superior mechanical properties of the composites as a whole [2–5]. Many investigations have carried out by reinforcing nanometer range or micron range metal, metal oxide particles which improve many physical and thermal properties of the thermosetting polymer composites. The overall properties of the composite increase monotonically with the addition of particles [5–7]. Mohammed et al. [8] reported the effect of sun flower and water-melon seed shell powders on the hardness of UP matrix. Hardness of the composites increases with the incorporation of K_2CO_3 particle within the UP matrices. Apart from that many studies on the increment of hardness with filler addition are reported [9]. Doan et al. have studied that chemical treatment of jute fibre improves thermal property of the reinforced polypropylene composites [10]. Addition of boric acid increases the limiting oxygen index (LOI) of glass fibre reinforced polyester composites and thus flame retardancy of the composites also increases [11]. UP resin reinforced with ceramic particles ($CaCO_3$, CaO, $MgCO_3$, MgO) have

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found to be with enhanced hardness and water resistance property compared to the virgin polymers [12]. Another approach for improvement in properties of the composites is by incorporation of particle and natural fibre within polymer matrix [13]. M.M. Kabir et al. studied the properties of two types of laminates i.e., sisal fibre /glass fibre/polyester resin and sisal fibre/ glass fibre/red mud with polyester resin. It was established that above composition composites are superior in the properties than sisal/polyester composite or pure UP [14]. Widely used toughening agent for brittle thermosets were rubbers particles. Proper dispersion of rubber particle enhances material toughness significantly. But, it has been widely established that the toughening of brittle thermosetting matrices with rubber particle ends up in reducing composites overall modulus and glass transition temperature (*Tg*). So, several studies reported that rigid metal oxide incorporation in the thermoset matrices increases their toughness [15,16]. The objective of the present investigation is to fabricate polymer composites with superior properties by incorporating natural fibre (i.e., jute) along with metal oxide (Al_2O_3 or ZrO_2) particles as filler within UP matrix. The composites are fabricated considering it as a potential candidate for low structural and packaging applications with optimum thermal and weathering behaviour.

2. Materials and methods

2.1. Materials

The unsaturated polyester (UP) resin of GP grade, methyl ethyl ketone peroxide (MEKP, catalyst) and cobalt naphthenate (accelerator) were bought from M/s A K B Agencies, Kolkata. Carded jute fibres were collected from National Institute of Research on Jute and Allied Fibre Technology (NIRJAFT). Then the fibres were treated with an alkaline solution and finally, cut into 3.5–4 cm lengths. Al_2O_3 (~99% pure approx. 50 μm) and ZrO_2 (~99.5% AR fine mesh 11–39 μm) particles were purchased from Loba Chemie. Styrene was used as a diluent for UP resin. NaOH and trimethoxymethylsilane were procured for Sigma Aldrich. Wax and 5% poly vinyl alcohol (PVA) solution were used as lubricating agents for the mould surfaces. A metal mould of specific dimension was used for composite fabrication. Mylar polyester sheet was used on top and bottom faces of the mould for smooth surfaces of the composites.

2.2. Chemical treatment of fibre

Jute fibres were treated with 5% NaOH solution for 2 h maintaining a liquor ratio of 20:1 at ambient temperature. Then fibres were washed several times with normal water for removal of additional NaOH, sticking to the fibre surfaces. Fibres were neutralized with dilute acetic acid solution and finally they were washed with distilled water with ensuring pH = 7. The fibres were then dried at room temperature for 48 h followed by oven drying at 80 °C for 6 h.

2.3. Surface treatment of particle

Al_2O_3 and ZrO_2 particles were treated with dilute silane solution in order to ensure better bonding with the matrix. 1% silane was added in a solution of 95% methanol and 5% distilled water. Then the metal oxides were added in the solution and stirred for 5 min. Then, the particulate filler added silane solution was dried at 93 °C for 30 min in order to evaporate the alcohol.

2.4. Fabrication of the composites

The fabrication of UP composites was done by hand lay-up technique. A metal mould with dimensions $140 \times 120 \times 5$ mm^3 was used for fabricating the composites of different compositions. Wax and 5% PVA solution were used as a release agent. A thin layer of wax and PVA were applied to the inner surfaces of the mould for quick and easy release of the fabricated composites. Diluted PVA solution were commercially marketed as synthetic glue hence, it was used as a lubricating agent. 5% PVA (95% pure) was added in warm water and the PVA solution was prepared by continuous stirring for 4 h. After thin layer of wax coating a layer of thin glue was also applied. After setting of the glue composites were fabricated in the mould. The prevention of moist environment was ensured by imitating the technique of commercial compression moulding. A top and lower stainless steel plate was used to cover the mould. A polyester mylar plastic sheet was added on top and bottom surface of the mould and on top of the sheets stainless steel plate was placed. A load of 10 kg was applied on top of the sandwich system and it was covered with a chamber to avoid the moist environment. At first UP resin was diluted with styrene 30% on the weight. Viscosity of the mixture was evaluated with standard B-4 ford cup. The viscosity of the commercial UP resin (starting material) was found to be 3960 centipoises which reduced up to 600 centipoises after addition of 30 wt% styrene. Then measured amount of filler (18 wt% fibre and 2 wt% metal oxide particles) was added to the modified resin. For uniform blending of fillers with UP resin, mechanical stirring at 2500 rpm for 5 min was performed. Then the resin was mixed with accelerator and catalyst (1% by wt of the resin each). 0.1% silane on the weight of resin was also added in this modified resin for better adhesion with matrix. It was then poured uniformly over the pre-laid fibres in the mould. Then the system was allowed for curing at room temperature for 24 h, followed by 2 h post curing in an air oven at 80 °C.

2.5. Characterizations

The microhardness test was conducted using LECO LM 100 Micro Vickers Hardness Tester. The dwell time used in this test was 15 s and the indentation was done 50 gf. The microstructural features of the composites were examined by using scanning electron microscope (Hitachi S-3400 N) with operating voltage of 15 kV. Identification of elemental composition of the composites were examined using the energy dispersive X-ray spectroscopy (EDS, Horiba EX-250) attached with the SEM facility. X-ray diffraction (XRD) technique was performed for phase identification of the fabricated composites with overall 20% filler content. The Bruker axs (D8 advance) diffractometer, armed with the position-sensitive detector, has been used with Ni-filtered Cu-Kα ($\lambda = 0.154$ nm) radiation generated at 40 kV/ 40 mA. Scan speed was maintained at 0.5 s for step of each 0.02° from angular range (2θ) of 10–90°.

Fourier transform infrared (FTIR) spectroscopy was done using Bruker Tensor 27 with maximum resolution of 1 cm^{-1}. It has a DTGS detector, mid-IR source (4000 to 400 cm^{-1}) and a KBr beam splitter. The decomposition process of the composites was studied by using a NETZSCH instrument TG 209 f1 (Germany) Thermo gravimetric analyzer in N_2 atmosphere from 30 to 600 °C with a heating rate of 10 °C/min. From the TGA data, the differential thermogravimetric analysis (DTGA) data was obtained directly from the software embedded within the instruments. DSC measurements were performed by NETZSCH DSC 200 PC instrument.

Limiting oxygen index (LOI) was done according to ASTM D2863. The test sample was positioned vertically in a glass chimney, and an oxygen/nitrogen environment was generated with a flow from the bottom of the chimney. While burning the top edge of the sample; concentration of oxygen was gradually decreased until the flame extinguishes. Oxygen index, in percent, was calculated from the final oxygen concentrations.

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