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Gamma-Alumina as Reinforcement in Study on Mechanical Performance of TPU/EPDM Based Nanocomposites

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ABSTRACT

Gamma-Alumina (γ - Al_2O_3) reinforced thermoplastic polyurethane (TPU)/Ethylene–propylene–diene monomer rubber (EPDM) blends have been developed. Thermoplastic polyurethane (TPU)/Ethylene–propylene–diene monomer rubber (EPDM) based nanocomposites having 1, 2, 3, and 4 Phr γ - Al_2O_3 were synthesized via co-rotated intermeshing twin-screw extruder. Structure and properties of TPU/EPDM based nanocomposites are composed. The surface functionalised by aminopropyl triethoxysilane, and interactions between nanomaterial and polymers observed by SEM. The upgraded dispersion and interfacial bonding that produced the best significant enhancement in properties of Gamma-Alumina (γ - Al_2O_3) nanocomposite were found at 2 Phr loading.

Keywords: TPU, EPDM, Nanoclay, γ - Al_2O_3, Nanocomposite, Morphology, Mechanical properties

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INTRODUCTION

Addition of nano fillers such as carbon, nanofibers, layered silicate clays, nanotubes, and silica nanoparticles play an important role to modify the properties of polymers and their blends, including high moduli, improved Strength and heat resistance [1-9]. Qianping et al studied the thermoplastic MMT nano composites [10]. Dilmani et al [11] prepared EPDM polymer layered silicate nano-composites in melt by reactive extrusion. PP-g-MA and poly (maleic anhydride-alt-1-octadecene)-g-PEO (poly (MA-alt-OD)-g-PEO) were used as functionalized polymer compatibilizer-internal plasticizer, octadecylamine-montmorillonite (ODA-MMT) and dimethyldidodecyl ammonium-MMT (DMDA-MMT) were added as reactive and non-reactive nano-fillers respectively. The formation of nano structural fragments and properties of polymer blend composites were studied. It was found that intercalation of EPDM depended on the origin and content of organoclay . Presence of reactive organo-filler (ODA-MMT) and PEO grafted alternating copolymer gave better results. The glass-transition (T_g), melting (T_m) and recrystallization (T_c) temperatures depended on the origin and content of organoclay and PEO-grafted copolymer-compatibilizer respectively.

Among the researchers, use of alpha nano alumina is very popular as metal oxide reinforcing material with wide range of applications particular in the area of textile, cosmetics, footwear and many industrial purposes including high thermo-mechanical properties [12-16]. There is no work found earlier on γ-alumina reinforced TPU /EPDM blend. In this present work, through the blending of EPDM with thermoplastic polyurethane (TPU) an optimized composite is produced at 8 wt. % of EPDM [16] and surface modified Gamma-Alumina (γ- Al_2O_3) was added as reinforcing nano filler in different quantities of 1,2,3 and 4 Phr. Composites were prepared by using a co-rotated intermeshing twin-screw extruder. The surface was functionalized by aminopropyl triethoxysilane, and interactions between γ- Al_2O_3 and polymer blend is observed by using SEM. The functionalized alumina nanoparticles showed enhanced mechanical properties. The fractured surface showed an interfacial effect with the addition of the functionalized nanoparticles.

MATERIALS AND METHODS

Materials

Polyester based- Thermoplastic polyurethane (Texin® 255) having a specific gravity of 1.21 g/cm^3 and glass transition temperature (T_g = -26^{0}C) was obtained from Bayer Material Science, Chennai, India. EPDM (KEP-960) had specific gravity of 0.87 and a mooney viscosity of 49 (ML 1+4 at 125^{0}C) with ethylene content = 70 wt. %, propylene content = 23.85 wt. % and ethylene norbonenen of 5.7 wt. % was provided by Kumho Polychem. Co. Ltd., Korea. Spherical particle shaped Gamma-Alumina (γ- Al_2O_3) nanoparticles were supplied by Advance Technology Materials; Mumbai, India having 15-20 nm averages particle size and 85 – 100 m^2/g with minimum 98.8 % of purity.

Melt-Compounding

Prior to compounding, the granules of thermoplastic polyurethane (TPU) were pre dried at 100^0C in electric oven for 2 hours. The flexibility of twin screw extruder allows this operation to be designed specifically for the formulation being processed. After pre drying the TPU, EPDM rubber and Gamma -Alumina was mixed in different weight ratio. The uniform mixture of TPU/EPDM/ γ- Al_2O_3 was fed into high performance co-rotating intermeshing twin screw extruder (diameter of screw = 21 mm, L/D ratio=40, model ZV20, manufactured by Specifiq Engineering, Vadodara, India) for melt extrusion. The melt temperature was 190^0C to 220^0C and the screw speed was maintained at 240 rpm. The ingredients were weighed at a prescribed ratio and manually mixed before being loaded into the extruder for melt blending. The compound was passed through the different zones of the extruder and cooling water bath were finally collected in pellet form.

TESTING

Surface Modification

There are several approaches which are used in modifying surfaces of nano reinforcements. Functionalization of Gamma -Alumina (γ- Al_2O_3) nano particles with 3- Aminopropyl triethoxy Silane, firstly 100 ml of deionized water with 5.5 – 7.5 P^H and 50 gm. of nanoparticles was taken. The substrate is treated with a mixed solution of 0.2 - 2 % silane coupling agent and 98% diluent as deionized water. The dilution could be applied by spray or immersion procedures followed by drying at 120 - 180^0C for 2 - 5 minute. An aq. solution of silane is most effective because the silane hydrolyses to form silanol.

Tests of mechanical performance are as follows:

Phase Morphology

Samples of the TPU/EPDM/ Gamma -Alumina nanocomposite were platinum coated to make the surface conductive. The morphology of the fractured surfaces was observed with scanning electron microscopy (SEM; JEOL JSM- 6390, Tokyo, Japan).

Mechanical properties

Test specimens for analysing the mechanical properties are prepared by high performance injection moulding machine (Model - Endura, Clamping tonnage = 90 Ton, L/D ratio = 20 manufactured by Electronica Plastic Machine Ltd. Pune, India). The barrel temperatures of different zones used were 190, 200, 210, 230^0C from hopper to nozzle and an injection velocity of 25 $mm\text{-}s^{-1}$. The test samples are initially conditioned according to ASTM- D 618 using 23 ± 2^0C and 50 ± 5 % RH for 24 hrs. prior to testing.

Tensile strength

Tensile properties are determined as per ASTM- D638 using dumb-bell shaped specimens for tensile testing, allowing the samples at least 24 hours after moulding, the test performed by INSTRON Universal testing machine model 3342.

Flexural modulus

Flexural properties are evaluated as per ASTM- D790 using an INSTRON universal testing machine, model 3342.The dimensions of the test specimen were 127mm in length, 12.7mm in width and 3mm thickness.

Hardness

The hardness property was measured according to ASTM- D2240 standard using **Durometer** (shore-D) hardness tester.

Abrasion

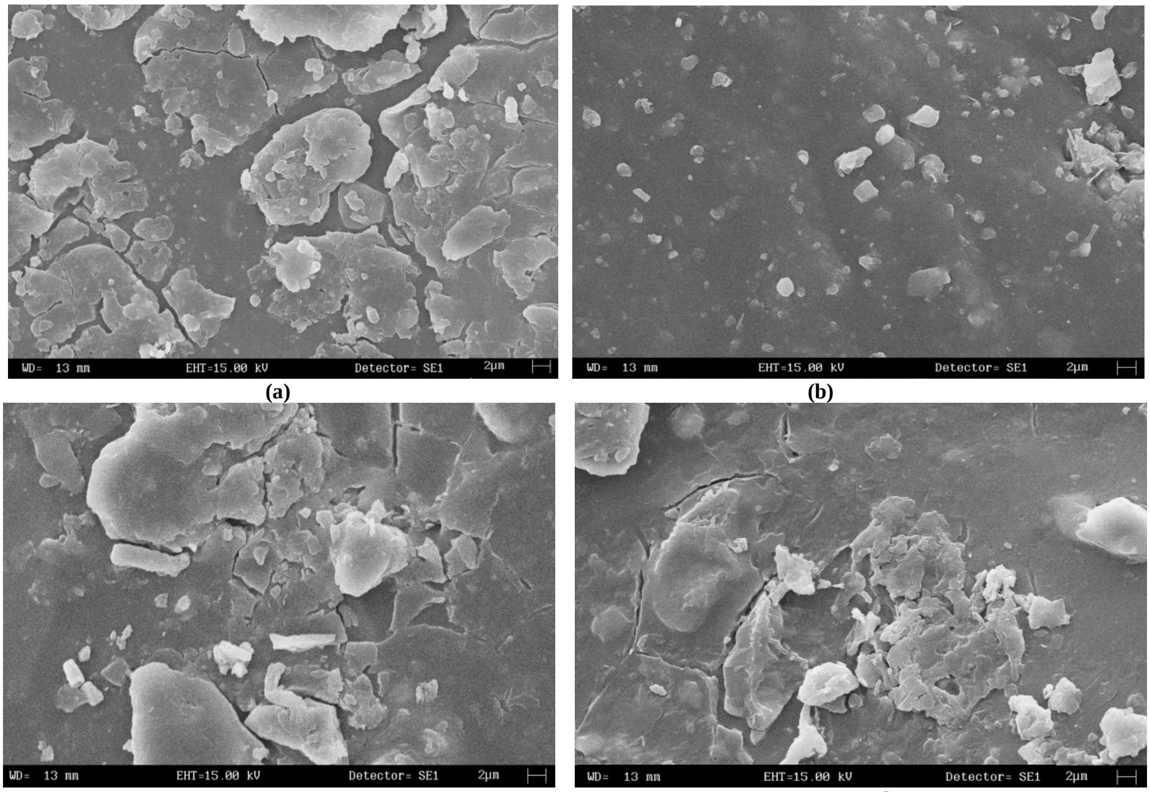
The abrasion resistance was determined according to ASTM- D1044 with the test machine (ABRASER 5131 by Taber® Industries. N.Y., USA). Test samples of 100 mm diameter disc and thickness 3 mm is used. Samples are drilled with a hole of 8 mm at the centre and fixed on the turn table under a load of 1000 g. Test specimens are conditioned at a standard test temperature and humidity of 23±2^0C and 50±5% RH for a specified period of time.

RESULTS AND DISCUSSION

The surface morphology of TPU/EPDM blends with different loading amount of modified γ- Al_2O_3 can be seen in Fig.1 (a-d) by SEM micrographs.

Fig.1 (a-d) shows the fracture surfaces of TPU/EPDM/ Gamma –Alumina of TPU/EPDM (92/8) blend with 1, 2, 3 and 4 Phr of Gamma –Alumina respectively. Fig.1 a, clearly shows the uniform surface of two phase structured polymer blend with 1 Phr dispersion of modified Gamma -Alumina throughout the matrix. The SEM images of the TPU/EPDM/ Gamma -Alumina nanocomposite displays that the EPDM is present as second phase in the blend along with stress focus points, which can evoke a shear zone in the matrix, Fig.1b therefore, the outside energy exerted to the blends can be dissipated through the formation of a shear zone. A further increase in the amount of Gamma -Alumina leads to a different shape of the blend in Fig.1(c- d).

The series of nanocomposites based on TPU/EPDM/ γ- Al_2O_3 were prepared via co-rotated twin-screw extruder with varying compositions viz., 0, 1, 2, 3and 4 Phr of Gamma-Alumina. The results from Table-1 clearly showed effects of addition of modified Gamma-Alumina nanofiller at 2 Phr loading, greatly improved mechanical properties of TPU/EPDM blends occurs due to addition of silane treated Gamma-Alumina nanofiller. The tensile strength is increasing from 17.06 to 24.57 Mpa at 2 Phr nanofiller content then goes down. The improvement in the tensile strength by the incorporation of the Gamma-Alumina is due to reinforcing character of nano alumina filler. At higher loading of nano alumina the decrease in properties occurs due to agglomeration of Gamma -Alumina particles.



(c) **(d)**

Fig.1 SEM micrographs images of fractured surfaces of Gamma-Alumina reinforced TPU/EPDM nanocomposite, a) 1Phr Nanofiller, b) 2 Phr Nanofiller, c) 3 Phr Nanofiller and d) 4 Phr Nanofiller

Table-1 Mechanical Properties of TPU/EPDM blends on adding Gamma-Alumina (γ- Al_2O_3) nanofillers.

Sample Code	PU/EPDM/ γ-Al_2O_3 (Phr)	Hardness (Shore-D)	Abrasion Loss (mg)	Flexural Strength (Mpa)	Tensile Strength (Mpa)	Elongation at break (%)
C0	92/8/0	52.0	32.0	12.58	17.06	471.70
C1	92/8/1	58.8	31.0	18.14	19.02	453.83
C2	92/8/2	60.2	28.0	23.04	24.57	434.33
C3	92/8/3	61.7	27.0	19.58	21.45	446.67
C4	92/8/4	62.8	25.0	17.23	18.60	468.35

Elongation at break of TPU/EPDM/Gamma- Alumina nanocomposite decreases at different loading of Gamma-Alumina content due to brittle character of filler. Flexural Strength of TPU/EPDM/Gamma-Alumina nanocomposite determined at different loading of Gamma- Alumina content shows that C2 composite from table-1 having 2 Phr nano gamma alumina filler gives the best results. Shore-D hardness of Gamma-Alumina filled nanocomposites at different loading of nano Gamma- Alumina nanofiller content (Phr) is calculated. It is observed from results in Table-1 that the shore-D hardness of the nanocomposites increases with the increase in the Gamma-Alumina nanofiller content linearly. The increase in the hardness may be due to the Gamma-Alumina particle presence on the outer surface of the polymer matrix which resists the crack propagation of the outer surface of the polymer blend material. The abrasion loss of TPU/EPDM/ γ- Al_2O_3 nanocomposites with different amounts of Gamma -Alumina content is shown in table 1. Abrasion loss of TPU/EPDM blend reduced on adding Gamma -Alumina from 32 mg related to that of the pure TPU. Abrasion resistance decreases due to increase in the hardness of the matrix. In this study abrasion loss gradually decreases with the increase of Gamma -Alumina because of improved resistance offered by between polymer matrix and Gamma -Alumina. Therefore, the abrasion resistance was best when the Gamma -Alumina content was 4 Phr .These results are similar to the mica filled polymer composite in which mica improved its rigidity, strength, and thermal stability, but dramatically decreased the elongation at break.

CONCLUSIONS

Addition of Gamma- Alumina nano-particles to TPU/EPDM (92/8) blends exhibited that the optimum concentration for Gamma-Alumina was found to 2 Phr, after which there was a decrease in properties, caused by particle agglomeration.

- It is noted that nano Gamma-Alumina filled composite have high tensile strength and flexural strength at 2 Phr.
- The Abrasion loss of TPU/EPDM/Gamma-Alumina blends reduced gradually on increasing Gamma-Alumina.

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