IOSR Journal of Applied Chemistry (IOSR-JAC)
e-ISSN: 2278-5736.Volume 9, Issue 2 Ver. I (Feb. 2016), PP 26-29
www.iosrjournals.org

Development of Bio-Based Nanocomposite from Epoxidized Castor Oil, Layered Silicate and Their Characterization

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Abstract: *In recent years, polymeric materials from renewable resources have received significant research attention because of their importance in environmental and economic view points. Inexpensive triglyceride plant oils have been used for the production of biodegradable polymeric materials. Polymeric nanocomposites were synthesized from epoxidized castor oil-based polymer and montmorillonite (MMT) clay using an in situ polymerization reaction. The resultant nanocomposites were characterized using FTIR, XRD and SEM analysis. It was found that the desired polymer nanocomposite structure was achieved when the OMMT was loading at 1, 2and 3 wt%. All the nanocomposites were found to have improved mechanical properties as compared with ordinary polymer, which have been confirmed by mechanical analysis.*

Keywords: *Epoxidized castor oil, organically modified montmorillonite, bio-based nanocomposites, mechanical properties*

I. Introduction

Recently, a new class of materials represented by bio-nanocomposites (biopolymer matrix reinforced with nanoparticles) has proven to be a promising option in improving mechanical and barrier properties of biopolymers derived from renewable sources. Modification of clays with organic modifiers and to intercalate such clays in polymers is the need of us. Development of such polymers using clay is an interdisciplinary science, fundamentally applicable to physics, chemistry, polymer science and material science. These developments are being done by various scientists to overcome basis drawbacks like low mechanical and thermal properties, electrical and optical properties of the polymers. Cetyltrimethyl amine like compounds can also be taken for such modifications. The extremely large surface area and high aspect ratio (between 30 and 2000) of the clay makes it possible for property improvements resulting from the formation of nanocomposites. Therefore, the current study was undertaken with the main objective of developing bio-nanocomposite films based on epoxidized castor oil and montmorillonite (MMT) with enhanced mechanical and barrier properties.

II. Material And Methods

2.1 Materials

4,4-Diaminodiphenylmethane(DDM), Montmorillonite(MMT) are sigma products. Amberlite XAD-4, BisphenolA Diglycidylether(DGEBA) are Alfa aesar products and Castor oil, N-cetyl-N,N,N-Trimethylammoniumbromide are loba products. All other solvents were of analytical grade and used for the synthesis.

2.2 Preparation of Na^+ Montmorillonite clay and Intercalation of the alkyl ammonium ions

Na MMT clay was prepared as per the report given by M. Aboobucker Sithique *et.al.* 100ml of cetyl trimethylammonium bromide solution and 3.7g of MMT clay was added and stirred well using magnetic stirrer for 2 days. A white precipitate formed was isolated by filtration and washed several times with a hot water/ethanol (1:1) mixture until no bromide was detected in the filtrate by one drop of 0.1 N $AgNO_3$ solutions. The cetylammonium ion exchanged montmorillonite was then dried for 2 days at 75^{o}C.

2.3.1 Synthesis of Epoxidized Castor Oil

Castor oil (45.9 g, 0.07 mol), glacial acetic acid (8.4 g, 0.14 mol), Amberlite (12.5 g), and toluene (20 g) were placed in a 500 mL four- neck round-bottom flask equipped with a magnetic stirrer, a thermometer sensor, and a reflux condenser. The mixtures were heated to a constant temperature of 55^{o}C. Then, 30% H_2O_2 (28.4 g, 0.25 mol) was added slowly *via* a separating funnel and allowed to react at 55^{o}C for 7h. The solution was subsequently filtered and washed with a saturated solution of sodium carbonate and distilled water. The toluene was removed by distillation under vacuum, and the product was further dried under vacuum at 80^{o}C for 2hours.

2.4 Preparation of Castor-based epoxy matrix

Castor -based epoxy matrix was prepared by mixing castor epoxy resin with base DGEBA resin at 30/70 wt % at 60^{o}C with vigorous stirring. The homogeneous solution thus obtained was mixed with stoichiometric amount of DDM at 90^{o}C. The mixture was degassed to remove the trapped air bubbles and poured into a preheated mould. The casting was cured at 150^{o}C for 3 hours and post cured at 190^{o}C for 2 hours and finally removed from the mould and characterized.

2.4.1 Preparation of OMMT Castor-based epoxy nanocomposites

Castor-based epoxy resin (30/70 wt %) mixed with different organophilic montmorillonite clay concentrations (1 wt%, 3 wt%, 5 wt% and 7 wt% based on the total weight of the resin mixture) at 70°C for 24 hours using a mechanical stirrer. A stoichiometric amount of the amine curing agent corresponding to epoxy equivalents was added. The product was subjected to vacuum to remove the trapped air and then cast and cured at 120°C for 3 hours. The castings were then post cured at 180°C for 2 hours and finally removed from the mould and characterized.

2.5 Measurement and Techniques

IR spectroscopy measurements are carried out in the range from 4000 cm^{-1} to 400cm^{-1} with a fully computerized SHIMADZU FT-IR TRACER-100 spectrometer

XRD patterns were obtained using an X-ray diffract meter (BRUKER ECO D8 ADVANCE) equipped with Cu, Kα radiations.

The fracture surfaces of materials were observed with scanning electron microscopy (SEM). A JEOL JSM 6360 SEM with field emission filament was used to collect SEM images of all the samples.

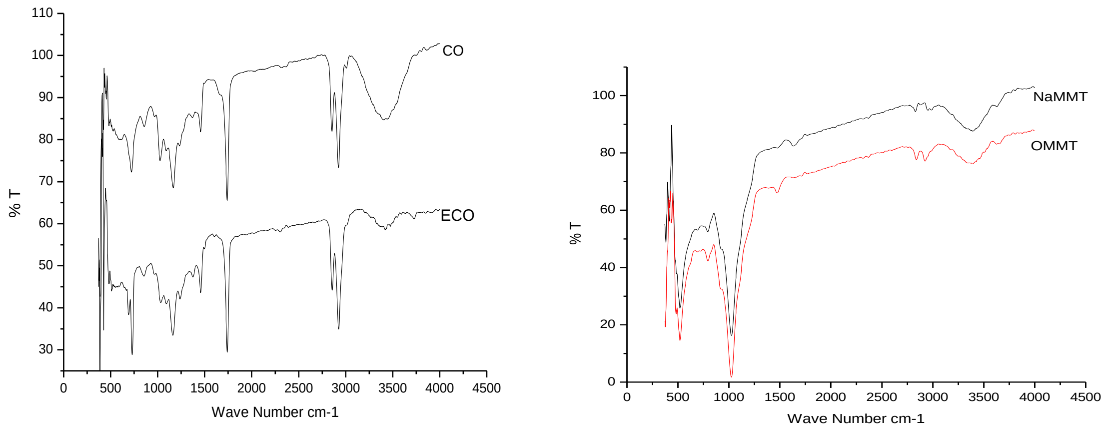
The Izod impact strength was measured for the composite materials. Izod impact specimens with the same dimension as prescribed in ASTM-D-256 standard were tested with a 453 g pendulum.

Water absorption studies were carried out by immersing the specimens of size 10mm x 10mm x 3mm in water for about 24 hours at 30°C.

III. Results And Discussion

Fourier Transform-Infrared Spectroscopy

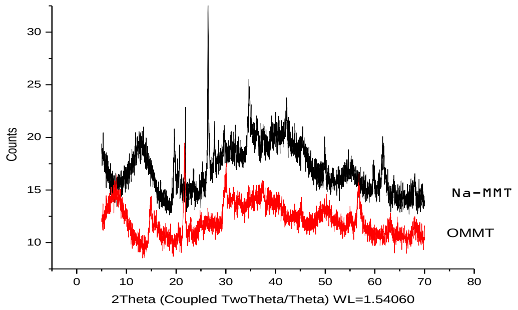
In FT-IR spectrum Fig.1, absence of the peak for an epoxidized castor oil at 3500 cm^{-1} indicate that the epoxidation process were occurred on the -CH=CH- of the castor oil. In Fig.2 presence of the peak at 3000-3100 cm^{-1} indicate that amines are successfully intercalated in the Na-MMT galleries.



FTIR Spectrum of CO and ECO

X-ray Diffraction Studies (XRD)

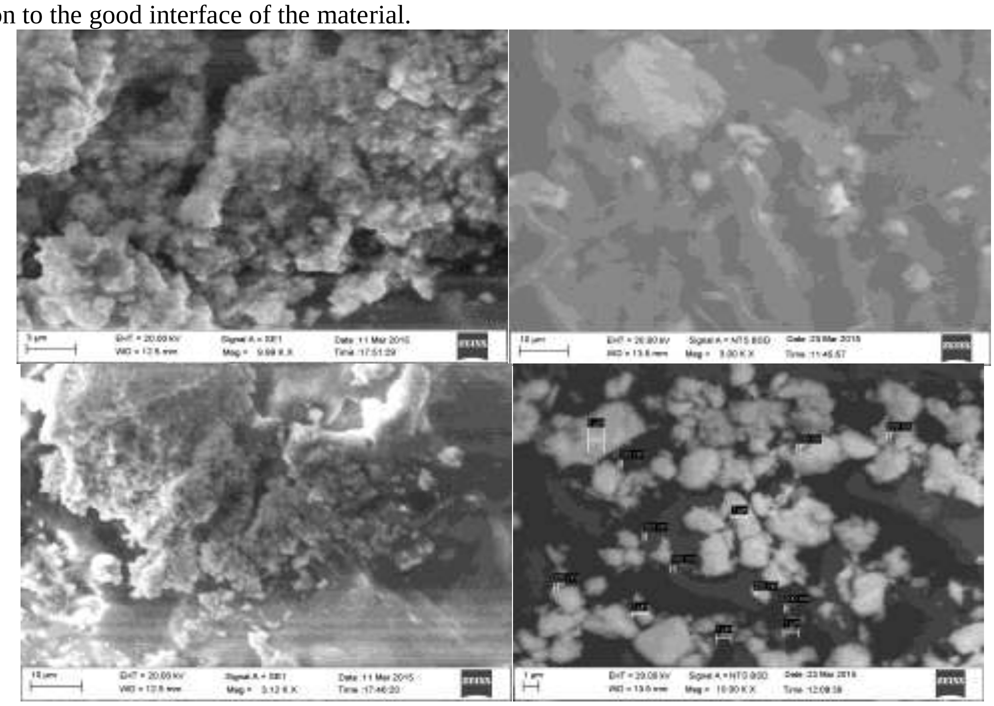
For the following Fig.3 shows the XRD curves of Na-MMT and OMMT. It can seen that the intense and sharp peak of Na-MMT at 2θ=25.70 shifted to 26.90 in OMMT indicate the intercalation of montmorillonite clay.



XRD Spectrum Of Na-MMT and OMMT

SEM analysis

The SEM micrographs indicate that there is no phase separation between silicate layers and the castor-based epoxy matrix. The SEM analysis of the composites exhibits relatively uniform dispersion of MMT layers in addition to the good interface of the material.



SEM Images of OMMT Castor- based epoxy nanocomposites

Mechanical Properties

Analysis of the impact testing finds that the incorporation of castor- epoxy in the DGEBA improved the impact strength.The values of both toughness and the impact strength of castor-based epoxy nanocomposites are increased appreciably according to the concentration of OMMT.

Water Absorption Behavior

The incorporation of castor epoxy into DGEBA matrices greatly reduces the percentage of water uptake. This is due to the hydrophobic nature of epoxidized castor oil and increased cross-linking density.

Table 1: Mechanical properties of neat DGEBA, Castor-based epoxy and OMMT Castor-based epoxy nanocomposites.

Composition (Wt. %)			Impact Strength (J/m)	Water Absorption(%)
DGEBA	castor epoxy	OMMT		
100	0	0	120.1±6	0.120
70	30	0	189.4±2	0.102
70	30	1	210.3±4	0.095
70	30	3	231.5±3	0.082
70	30	5	243.0±4	0.063
70	30	7	261.8±5	0.051

IV. Conclusions

Organophilic modification of Na-MMT clay was carried out with a new and totally renewable intercalant, ECO. From this organically modified clay, ECO-based polymeric nanocomposites with various degrees of loading were prepared by *in situ* polymerization of ECO and DGEBA monomer mixture. The thermal stabilities and dynamic mechanical properties of the nanocomposites were improved, compared to neat castor based epoxy matrix, with addition of suitable organophilic clay. Strengthening of the polymer matrix was also observed. As a result, it can be safely concluded that OMMT Castor-based epoxy nanocomposites, with enhanced thermal and dynamic mechanical performance, can be prepared using equal amounts of a renewable ECO and DGEBA monomers and a renewable intercalant, even with 1–3 wt% clay loadings.

Acknowledgement

We wish to express our sincere thanks to Tamilnadu State Council for Science and Technology, Tamilnadu for financially supporting this work under the grant TNSCST student project scheme.

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