iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 7 Issue: V Month of publication: May 2019

DOI: https://doi.org/10.22214/ijraset.2019.5598

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.177
Volume 7 Issue V, May 2019- Available at www.ijraset.com

The Effect of Processing and Clay Modifications on Properties of Pet/Nano Clay Nanocomposites

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Abstract: Well dispersion or intercalation/exfoliation of nanoscale fillers (such as nano-$CaCO_3$ and nano-clay) in polymer matrix is currently not easy to be achieved. Supercritical carbon dioxide ($ScCO_2$) has been reported to have great potential for facilitating the dispersion or intercalation/exfoliation of the nanoscale fillers in polymer matrix. Supercritical fluids have a unique and valuable potential for the enhanced processing of many materials. In this study poly (ethylene terephthalate) (PET)/clay nanocomposites with three different clays with different weight percentages of montmorillonite (MMT) have been processed by scCo2 technique. The X-ray diffraction (XRD) patterns and transmission electron microscopic (TEM) images reveal the formation of intercalated nanocomposites. In present study, three different organically modified montmorillonite (MMT) clays of Cloisite 10A, 20A and 30B (5%) were added into polyethylene terephthalate (PET) polymer. Nanoclay and PET polymer were combined with melt blending method in terms of twin screw extruder and ScCo2 processing Internal and morphological properties of PET/clay nanocomposites were analyzed with transmission electron microscopy (TEM) images and XRD curves. The findings and results of nanocomposites were compared with that of PET polymer. At the end of the study, the changes in PET polymer and the effect of clay type on material properties were determined. Compared to the nanocomposites prepared without the aid of $ScCO_2$, the nanocomposites with $ScCO_2$ addition appear to have higher degree of nano-filler dispersion or intercalation/exfoliation.
Keywords: Poly (ethylene terephthalate) (PET), clay, nanocomposites, Nanoclay, supercritical carbon dioxide

I. INTRODUCTION

Polymer nanocomposites are constituents of reinforced polymers with lower proportion of nanoscale fillers (<5wt%). These tiny fillers, when well dispersed into polymer matrices, have at least one dimension smaller than 100nm (1nm=10-9m). This research focuses on organomodified montmorillonite (MMT)There is a great interest developed in recent years on polyester/clay nanocomposites especially on poly(ethylene terephthalate) (PET) nanocomposites due to their vast range of applications [1-8]. Semicrystalline Poly(ethylene terephthalate) (PET) and its composites are widely used in packaging,construction, automobile, household, electrical and textile industries.Considerable efforts have been devoted to improve its physical, mechanical and barrier properties through mixing it with nanoclays to produce layered clay-incorporated PET composites.

A. *Material Experimental Equipment and Sample Preparation*

The equipment mainly included a co-rotating twin screw extruder and a CO_2 injection system.The PET used for this study was supplied by coca cola Company. In the present study, the fillers, montmorillonite, types of Cloisite 10A, 20A and 30B, were provided from the Southern Clay Products Proper choice of organoclay chemistry is critical and hence we aimed to analyse the different clay types on nanocomposite properties. Organoclays with said referred to aspect ratios starting from 10 to 300.In this study, we develop a process to help exfoliate and disperse the nanoclay into PET matrix with the aid of supercritical CO_2. Organic clays and PET powder had been primarily dried in vacuum at 90^{o}C for 12h before dealing with. Then resins of PET with diverse kinds of clay (scCO2 processed or as-received), modification (20A,30B and 10A) and fraction (1, 3 and five wt%) had been closed in a foil bag made of aluminium and blended mechanically by the usage of a "Thumler's tumbler" for 12h before extrusion.[14]

PET/clay NC have been compounded with the aid of the use of a micro-compounder of laboratory scale ("Thermo Haake Minilab") with a recirculation channel which permits the processing of batches of usually 5–40 g, a barrel temperature of 265^{o}C and screws of twin counter-rotations at a velocity of 50rpm. At last, the strands which have been extruded cooled down to the temperature of the room and are made into pellets. PET sample which is pure turned into made following the similar tactics as manage. Thin (~0.2mm) samples of pure PET films and NC had been made via pellet melt pressing right into a cupper mold which is custom-made ("diameter=56mm") among foil sheets coated with Teflon.[14]

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II. CHARACTERIZATION TECHNIQUES

A. *Wide-Angle X-Ray Diffraction (WAXD)*

“A Rigaku Rotaflex Powder Diffractometer with a Cu Kα X-ray source λ=1.54 Å (accelerating voltage= 44 kV, cutting-edge=120 mA)” became utilized to appraise the intergallery spacing of clay. Samples of Composite films had been arranged in a zero-history background, custom-made sample holder this is 0.9 mm in deepness. Various scans have been acquired from distinct places within the specimen and tested to be reproducible when patterns of diffraction had been superimposed on each another. “The 2θ angle was determined the use of the JADE software that accompanies the diffractometer, and the d001 spacing for the clays was calculated the use of Braggs’ diffraction Law”. .[9-15]

B. *Transmission Electron Microscopy (TEM)*

NC Thin sections (70-100nm) had been made the use of a “Leica Ultramicrotome with a diamond knife (knife angle $=35^o$)” and positioned on two hundred mesh grids made of copper protected with carbon. With the help of “Hitachi H7600 Transmission Electron Microscope operated at 80 kV” all samples were tested. Numerous (10-15) pix have been gathered for all specimens to make certain distinct illustration of the dispersion inside the matrix of the polymer and clay morphology.[9-15]

III. RESULTS AND DISCUSSION

Figure 1 shows the XRD patterns for PET/clay nanocomposites prepared with 5 wt% ScCO2 and without scCO2. These patterns similar to the previous studies results of Fengyuan Yang [14]

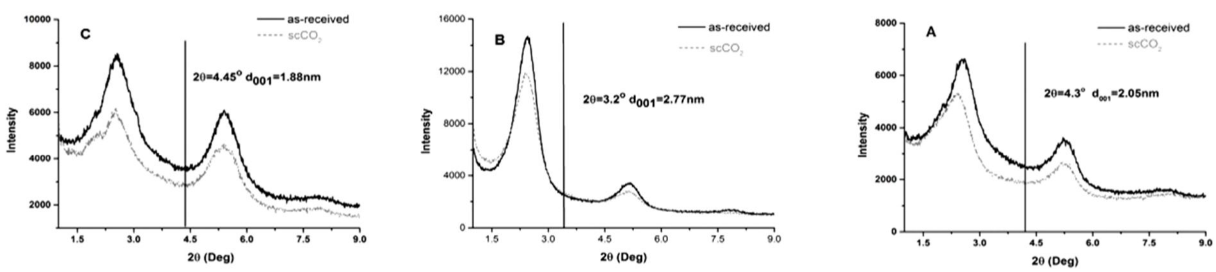

FIG1: NC’s with 5wt% clay (A) 30B (B) 20A and (C) 10B WAXD patterns

All NC showed properly-described both d001 and d002 diffraction peaks, no matter their surface chemistry or processing of scCO2, advising the existence of sure exceeding clay structures which are ordered in all NC. In addition, all of NC confirmed shifts of the d001 peaks to especially lower 2θ compared with corresponding pristine clays, advising that the polymer have been embedded into the galleries of clay. In particular, 10A, 20A and 30B strengthened PET NC displayed d001 diffraction peaks at 2θ equal to 2.54°, 2.4^o and 2.55^o which corresponding to increases of inter gallery spacing from 1.05 to 2.47 nm, 1.77 to 2.68nm and 0.88 to two.46nm respectively. The boom of inter gallery spaces for 30B, 20A and 10A suggesting that 30B and 10A might possess more favorable polymer-clay interaction than 20A did.

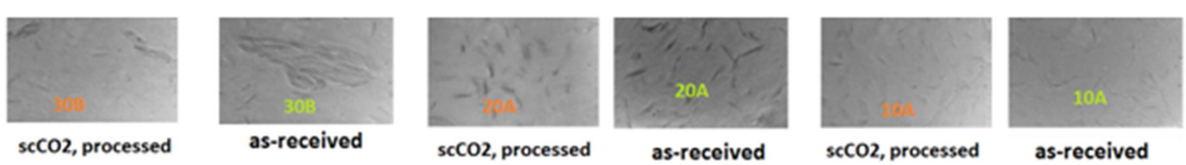

FIG2:(5 wt %) clay/PET NC TEM images

Figure 2 affords illustrative TEM snap shots of PET/clay NC with 5wt% of various clays.Both 10A and 20A showed especially uniform dispersion interior PET, even though as-acquired 20A displayed positive degree of aggregation. Enormous improvement on clay dispersion becomes discovered in scCO2 processed samples, although partially aggregations nevertheless exist. Most of the tactoids were in the dispersed shape, which elevated clay density (range of debris according to unit place) and improved clay particles homogenous distribution in evaluation of clay of as-received [15]

IV. CONCLUSION

In PET/clay NC, the dispersion of clay particles follow the trend that 30B<20A<10A .ScCO2 processing outcomes in progressed polymer-clay interface because of scCO2 processed clay uncovered more available surface to polymeric matrix and the exfoliated shape formed strong polymer-clay interaction. The improved polymer-clay interface may want to correctly boom the powerful aspect ratio which in flip improves the barrier properties of resultant NC scCO2 processing bring about stepped forward clay dispersion with the aid of exfoliating the clay layers from the tactoids, similarly, the processed clay lead to more homogenous clay dispersion due to smash down huge clay particles. The advanced homogenously clay dispersion ought to efficaciously growth the aspect ratio which in flip improves the barrier properties of resultant NC

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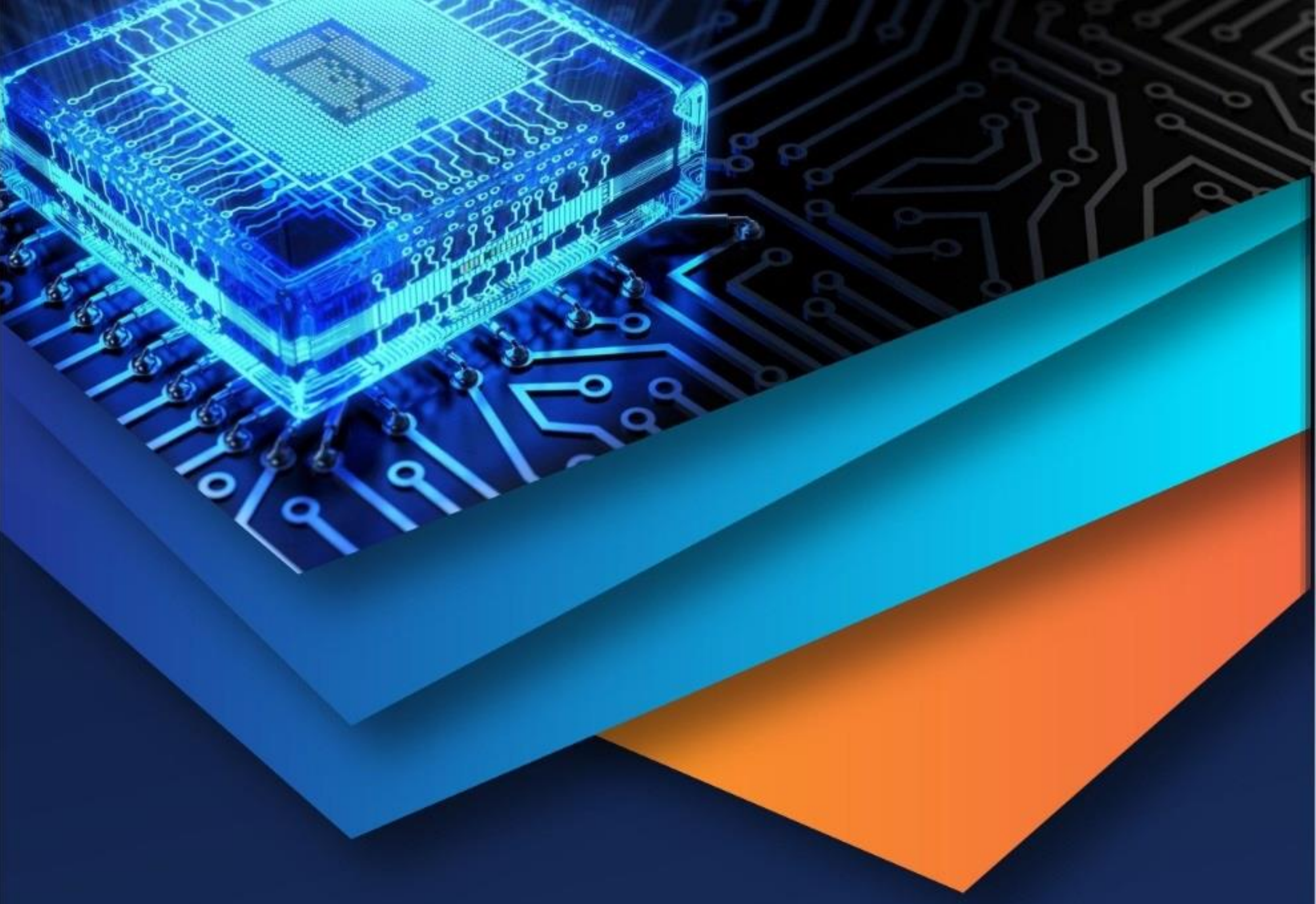

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