INFLUENCE OF MONTMORILLONITE AS SECONDARY FILLER ON THE PROPERTIES OF CARBON NANOTUBE / HIGH DENSITY POLYETHYLENE NANOCOMPOSITES

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"Special dedications to my beloved parents and family
Thanks for the love, support and encouragement"

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ABSTRACT

In this study, the influence of secondary filler on the dispersion of carbon nanotube (CNT) reinforced high density polyethylene (HDPE) nanocomposites (CNT/HDPE) was investigated. In order to understand the mixed-fillers system, montmorillonite (MMT) in addition with maleic anhydride grafted high density polyethylene (PE-g-MA) was added to CNT/HDPE nanocomposites. It was followed by investigating their effects on the thermo-mechanical, rheological and morphological properties of the aforesaid nanocomposite. Incorporation of 3 wt% each of MMT and PE-g-MA into CNT/HDPE nanocomposites resulted in the increase values of the tensile and flexural strength (32% increase in both), as compared to that of pure HDPE matrix. Thermal analysis results showed an improvement in thermal stability of the formulated nanocomposites. The initial decomposition temperature (Ti) for nanocomposite with 9 wt% of MMT and 3 wt% of PE-g-MA increased from 265 ^{o}C (Ti for neat HDPE matrix) to 296 ^{o}C. Addition of MMT into CNT/HDPE nanocomposites had also increased the rheological properties, indicating a dominant elastic response. Significant increase in loss modulus, storage modulus and complex viscosity were observed upon addition of PE-g-MA, whereas Tan δ was found to be reduced. This might be due to better interfacial adhesion between MMT and HDPE phases that attributed to the elastic dominance. Improvement in dispersion of CNT upon addition of MMT and PE-g-MA was further supported by the morphological analysis. Transmission electron microscopy (TEM) images revealed that larger aggregates of CNTs were disappeared upon addition of these two components leading to the enhancement of thermo-mechanical properties for such nanocomposites.

ABSTRAK

Dalam kajian ini, pengaruh pengisi kedua terhadap penyerakan tiub karbon nano (CNT) yang memperkukuhkan polietilena berketumpatan tinggi (HDPE) komposit nano (CNT/HDPE) telah dikaji. Bagi memahami sistem pengisi bercampur, montmorilonit (MMT) yang dicampurkan bersama malik anhidrat cantuman polietilena berketumpatan tinggi (PE-g-MA) telah dimasukkan ke dalam komposit nano CNT/HDPE. Ini diikuti dengan mengkaji kesannya terhadap sifat-sifat termo-mekanikal, reologi dan morfologi komposit nano tersebut. Penambahan 3% berat setiap MMT dan PE -g -MA ke dalam komposit nano CNT/HDPE telah meningkatkan nilai kekuatan tegangan dan lenturan (peningkatan sebanyak 32% untuk kedua-duanya), berbanding dengan matriks HDPE tulen. Hasil analisis terma komposit nano menunjukkan peningkatan kestabilan haba yang lebih baik. Suhu penguraian permulaan (Ti) untuk komposit nano dengan 9% berat MMT dan 3% berat PE-g-MA meningkat dari 265 ^{o}C (Ti untuk matriks tulen HDPE) kepada 296 ^{o}C. Penambahan MMT ke dalam CNT/HDPE komposit nano juga meningkatkan sifat-sifat reologi yang menunjukkan tindak balas dominasi elastik anjal modulus penyimpanan. Peningkatan yang ketara dalam modulus penyimpanan, modulus kehilangan serta kelikatan kompleks diperhatikan menerusi penambahan PE-g-MA, manakala Tan δ didapati berkurangan. Ini mungkin disebabkan oleh lekatan yang lebih baik di antara permukaan MMT dan HDPE yang berkaitan dengan dominasi anjal. Peningkatan dalam penyebaran CNT dengan penambahan MMT dan PE-g-MA turut disokong oleh analisis morfologi. Imej mikroskop penghantaran elektron (TEM) mendedahkan bahawa agregat CNT yang lebih besar telah hilang menerusi penambahan kedua-dua komponen utama yang membawa kepada peningkatan sifat termo-mekanikal komposit nano tersebut.

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LIST OF ABBREVIATIONS

ASTM	-	American Standard of Testing and Method
CNT	-	Carbon Nanotube
SWCNT	-	Single-Walled Carbon Nanotube
MWCNT	-	Multi-Walled Carbon Nanotube
CB	-	Carbon Black
PNCs	-	Polymer Nanocomposites
IPP	-	Isotactic Polypropylene
PE	-	Polyethylene
HDPE	-	High Density Polyethylene
TPU	-	Thermoplastic Polyurethane
MMT	-	Montmorillonite
PE-g-MA	-	Polyethylene Grafted Maleic Anhydride
PP-g-MA	-	Polypropylene Grafted Maleic Anhydride
MA-g-PO	-	Maleic Anhydride Grafted Polyolefins
DSC	-	Differential Scanning Calorimeter
TGA	-	Thermo Gravimetric Analysis
XRD	-	X-Ray Diffraction
SEM	-	Scanning Electron Microscopy
TEM	-	Transmission Electron Microscopy
SAXS	-	Small Angle X -Ray Scattering
WAXD	-	Wide Angle X -Ray Diffraction

LIST OF SYMBOLS

T_i	-	Initial Decomposition Temperature
T_c	-	Crystallization Temperature
T_m	-	Melting Temperature
T_D	-	Decomposition Temperature
ΔH_c	-	Crystallization Enthalpy
ΔH_m	-	Melting Enthalpy
E	-	Young's Modulus
η^*	-	Complex Viscosoty
G'	-	Storage Modulus
G"	-	Loss Modulus

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CHAPTER 1

INTRODUCTION

1.1 Background

In last couple of decades, polymer nanocomposites have generated great interests among researchers as a type of composite material because of their superior thermo-mechanical properties when compared with the pure polymer or composites having conventional fillers [1]. Addition of nanoparticles as fillers in the polymer matrix not only improves the polymer strength, rigidity, and flexibility, but also facilitates the improvement of the polymer light transmission, barrier property, thermal resistance and electrical conductivity [2-5]. Nanocomposites are the combination of nanotechnology and nanoparticles, the future of composite materials.

Among the choice of polymer matrix, polyethylene is widely used due to its attractive properties. It is one of the most common volume thermoplastic with applications in packaging, consumer goods, pipes, cable insulation, etc. [6-11]. Polymer blends composed of high-density polyethylene (HDPE) has attracted considerable interest both in the research community and in industry. As a widely used polyolefin, HDPE possesses excellent chemical resistance, tensile properties, and hardness [12-16].

Several nanoparticles have been used as fillers in the polymer matrix so as to achieve the aforesaid functionalities of polymer nanocomposites. Off late carbon nanotube (CNT) has been widely explored as a potential filler owing to its larger surface area available to interact with matrix, high aspect ratio, excellent mechanical strength, and electrical and thermal conductivity ,.

Another nano-filler that has always attracted lots of interest among researcher is Montmorillonite (MMT). MMT attracts researchers mainly due to its availability and being "green" filler [7, 17-20]. The addition of MMT to polymer matrix can modify rheological properties and provide improved barrier properties and fire retardancy to the nanocomposite [21-23]. It also offers industrial significance since the use of small amounts of MMT is enough to improve the overall physical properties of a polymer matrix at a relatively low cost [24].

To achieve a high interfacial area (i.e. low interfacial tension) between nanotubes and polymers, their compatibility has to be enhanced which can be done using different methods such as functionalizing the sidewalls of CNT [19, 25, 26], grafting technique [23, 27] and also by addition of polymeric compatibilizer such as maleic anhydride grafted polymers [28-31]. It is widely accepted that the polymer nanocomposites prepared with the incorporation of a compatibilizer performs better [28, 31, 32]. Yang et al. [29] enhanced the compatibility and dispersion of CNTs in polyethylene matrix by melt blending of amine–functionalized nanotubes with maleic anhydride modified high density polyethylene (PE-g-MA). Pollanen et al. [28] enhanced the thermal and mechanical properties of HDPE by melt mixing it with CNT/PE-g-MA master batch.

Also, recently a strategy has been proposed involving the addition of a second filler/nanofiller working in synergy with CNT, and is defined as mixed (nano-) fillers system [21, 33, 34]. Addition of secondary filler can be interesting from an application view point since the selection of secondary filler may not only assist the CNT dispersion but might also provide additional functionalities to the polymer matrix. [24]. Bao et al. [35] studied the effect of montmorillonite (MMT) on the dispersion of carbon black (CB) into polymer matrix. Liu et al. [36] studied the effect of addition of nanoclay to single-walled carbon nanotubes (SWCNT)/ epoxy composite.

Different processing techniques have been reported to fabricate polyolefin nanocomposites such as; *in situ* polymerization [9], solution mixing [37, 38], surfactant modification [39], polymer wrapping [40], polymer absorption [40] and melt blending [38, 41]. Our interest lies in melt blending technique as it is simple, fast and economical method compatible with current industrial techniques [7, 41, 42]. The idea of using melt blending technique in combination with nanoclay and compatibilizer is to achieve good dispersion of the nanofillers through shear forces as well as strong coupling/interface interaction between nanofiller and matrix [3, 7, 41].

Although, the above mentioned techniques and methods are not new but still there is no data available in literature which focuses on the enhancement of Mechanical, thermal, electrical and other related properties of HDPE based polymer nanocomposites using mixed-filler system. The focus of this study is HDPE because of its ease of availability, low price and more importantly it can serve the high-end applications (automobiles, electronic caskets, EMI/RF shielding etc) as engineering polymer.

1.2 Problem statement

In this study the focus is to enhance the mechanical and thermal properties of HDPE, so that it can serve in high-end applications. In order to achieve this CNT is used as fillers. But, to exploit all the features and properties of CNTs, one needs to ensure good dispersion and distribution of CNTs in polymer matrix along with strong interfacial interaction. In general, the mechanical properties of carbon nanotubes reinforced polymer do not improve significantly mainly due to the weak polymer-

CNT interfacial adhesion that prevents the efficient stress transfer from the polymer matrix to CNT [19, 23, 40, 43]. As a result, nanocomposite having properties much inferior to theoretical expectations is obtained [3, 19, 28]. Recent study by Bao et al. [35] suggested that carbon black (CB) aggregates could adsorb onto MMT platelets thus modifying both the level of CB dispersion and flow properties. A study by Liu et al. [36] explained that upon incorporation of nanoclay, SWCNT become more exfoliated and form better networks in such composites, mainly because of its affinity with nanoclay. But there's no literature available about mixed filler system wherein CNTs and nanoclay are used as fillers and HDPE is used as polymer matrix. Thus, present study deals with the mixed-filler system, where CNT is used as primary filler and MMT is used as secondary fillers. Here, attempts have been made to understand the effect of secondary filler on the CNT/HDPE nanocomposite.

i) What improvements will be achieved by incorporating MMT as secondary filler to CNT/HDPE nanocomposite in terms of thermal and mechanical properties?
ii) Does the incorporation of MMT as secondary filler to CNT/HDPE nanocomposite will have effect on the morphology of the nanocomposite?
iii) What will be effect of MMT incorporation on the microstructural changes of CNT/HDPE nanocomposites?
iv) What optimizations can be made for the formulated nanocomposites in terms of thermo-mechanical and morphological properties.

1.3 Objectives of the Study

One of the most important aspects of mixed filler system is to enhance the thermo-mechanical properties of CNT/HDPE nanocomposite without affecting the processability. The present study aims to explain the influence of secondary filler (MMT) to CNT/HDPE nanocomposite on processability, thermo-mechanical and morphological properties of the formulated nanocomposites. The primary objectives can further be classified as:

i) To determine the effect of MMT as secondary filler to CNT/HDPE nanocomposite in terms of thermal and mechanical properties.
ii) To study the effect of incorporation of MMT as secondary filler to CNT/HDPE nanocomposite on the morphology of the nanocomposite.
iii) To investigate the effect of MMT incorporation on the microstructural changes of CNT/HDPE nanocomposites using XRD technique.
iv) To optimize the formulated nanocomposites using compatibilizer in terms of thermo-mechanical and morphological properties.

1.4 Scope of Research

In order to achieve the objectives of this research, following procedures were carried out.

a) Sample preparation via melt extrusion.

i) Drying of nanofillers and neat HDPE resin for removal of any moisture content.
ii) Blending of HDPE with different nanofillers using twin screw extruder in a single extrusion step.
iii) Blend fabrication into test specimens via injection molding according to ASTM standards.

b) Thermal Analysis

i) **Differential Scanning Calorimeter (DSC):** A DSC analysis was carried out to find the crystallization, melting and onset temperatures. Also, it was used to find the enthalpies of the nanocomposites.

ii) **Thermogravimetric Analysis (TGA):** TGA analysis was carried out to find the thermal stability of the nanocomposites.

c) Mechanical Analysis

i) **Tensile test:** Tensile test provides the tensile strength and young's modulus of the nanocomposite.

ii) **Flexural test:** Flexural test provides the flexural strength and its modulus of the nanocomposites.

iii) **Izod impact test:** Impact test provides the impact strength and the energy required.

d) Rheological Measurements

Rheological tests were used to find the viscosity, modulus (both loss and storage) and tangent delta for the nanocomposites.

e) X-ray diffraction (XRD)

XRD was used to find the distribution of fillers in polymer matrix.

f) Transmission Electron Microscopy (TEM)

TEM analysis was used to find the morphology of the nanocomposites.

1.5 Significance of Study

Carbon nanotubes are considered to be the strongest and stiffest material among all the other existing materials. But in general transferring the properties of CNTs to polymer nanocomposites is very difficult. Hence, the practical values obtained for CNT reinforced polymer nanocomposites are found to be way less than that found theoretically. One way to exploit all the features and properties of CNTs, one needs to ensure good dispersion and distribution of CNTs in polymer matrix along with strong interfacial interaction. Different techniques have been reported in literature on how to improve the dispersion of CNTs in polymer matrix. But, these techniques lack something or the other, which are discussed in detail in chapter 2.

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