

PREPARATION AND CHARACTERIZATION OF COMPATIBILIZED
POLYPROPYLENE / POLY (ACRYLONITRILE-BUTADIENE-STYRENE)
BLENDS AND NANOCOMPOSITES

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UNIVERSITI TEKNOLOGI MALAYSIA

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'To my beloved parents and friends'

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ABSTRACT

Polypropylene (PP)/poly(acrylonitrile-butadiene-styrene) (ABS) copolymer blends containing organophilic layered silicates montmorillonite (MMT) compatibilized with polypropylene-grafted-maleic anhydride (PP-g-MA) were prepared by means of melt extrusion using twin screw extruder followed by injection molding in order to examine the mechanical, morphological and thermal properties of the blends and nanocomposites. For PP/ABS blends, the loadings of ABS were varied from 20 to 50 wt% and the optimum formulation of 40 wt% of ABS was chosen based on the balance of stiffness and toughness which then compatibilized with 2, 4 and 6 phr of PP-g-MA. The nanocomposites were produced by the addition of 3 and 5 phr MMT into PP/ABS/PP-g-MA. The results showed that the incorporation of MMT in PP/ABS nanocomposites leads to higher stiffness but at the expense of toughness and strength. Meanwhile, the incorporation of PP-g-MA increased the stiffness and strength but the toughness was reduced. Morphological study revealed the two-phase morphology which was clearly visible for PP/ABS blend system. The dispersed phase of ABS was decreased as the amount of PP-g-MA increased. X-ray diffraction (XRD) and transmission electron microscopy (TEM) results showed the formation of nanocomposites as the MMT was intercalated by PP macromolecules. Incorporation of PP-g-MA has improved the degree of intercalation and hence resulting in better dispersion in PP/ABS. Thermogravimetric analysis (TGA) showed that the thermal stability significantly increased with the incorporation of MMT and further improvement in thermal stability was observed as the PP-g-MA was incorporated in PP/ABS nanocomposites. Differential scanning calorimetry (DSC) results showed that the degree of crystallinity (X_c) increased substantially with MMT content while maintaining its melting temperature (T_m). Further improvement was observed upon addition of PP-g-MA. Dynamic mechanical analysis (DMA) showed that the storage modulus increased with MMT content for compatibilized PP/ABS system compared to uncompatibilized system.

ABSTRAK

Adunan kopilimer polipropilena (PP)/poli(akrilonitril-butadiena-stirena) (ABS) yang mengandungi montmorilonit (MMT) diubahsuai secara organofilik serta diserasikan dengan polipropilena-tercangkuk-maleik anhidrida (PP-g-MA) disediakan melalui penyemperitan leburan menggunakan mesin penyemperit skru berkembar dan diikuti proses acuan suntikan bagi mengkaji sifat-sifat mekanikal, morfologi dan terma nanokomposit. Bagi adunan PP/ABS, komposisi ABS diubah dari 20 sehingga 50 wt% dan komposisi optima 40 wt% ABS dipilih berasaskan sifat kekakuan dan kekukuhan yang seimbang kemudian diserasikan dengan 2, 4 dan 6 phr PP-g-MA. Nanokomposit pula dihasilkan melalui penambahan MMT 3 dan 5 phr MMT ke dalam PP/ABS/PP-g-MA. Hasil menunjukkan bahawa penambahan MMT ke dalam nanokomposit menyebabkan peningkatan kekakuan tetapi menyebabkan penurunan kekukuhan dan kekuatan bahan. Sementara itu, gabungan PP-g-MA pula meningkatkan kekakuan dan kekuatan dengan penurunan kekukuhan bahan tersebut. Kajian morfologi pula menunjukkan pembentukan dua fasa morfologi untuk campuran PP/ABS dan pengurangan saiz partikel melalui pertambahan PP-g-MA. Keputusan pembelauan sinar-X (XRD) dan mikroskop pancaran elektron (TEM) menunjukkan pembentukan nanokomposit melalui interkalasi MMT dengan makromolekul PP. Gabungan PP-g-MA telah meningkatkan darjah interkalasi seterusnya menyebabkan penyebaran yang lebih baik di dalam PP/ABS. Analisa termogravimetrik (TGA) menunjukkan kestabilan terma meningkat apabila MMT dimasukkkkan dan ianya meningkat dengan lebih ketara dengan gabungan PP-g-MA ke dalam nanokomposit PP/ABS. Kalorimeter pengimbasan pembezaan (DSC) pula menunjukkan darjah pengkristalan (X_c) meningkat dengan kandungan MMT sementara suhu leburannya (T_m) pula dikekalkan. Peningkatan ketara dapat dilihat terhadap penambahan PP-g-MA. Analisa mekanikal dinamik (DMA) pula menunjukkan modulus simpanan meningkat dengan peningkatan kandungan MMT bagi sistem yang diserasikan berbanding dengan sistem yang tidak diserasikan.

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LIST OF ABBREVIATIONS

ABS	-	Acrylonitrile-butadiene-styrene
ASTM	-	American Society for Testing and Materials
CNT	-	Carbon nanotubes
<i>D</i>	-	Spacing between diffractive lattice plane (interspacing)
DCP	-	Dicumyl peroxide
DSC	-	Differential scanning calorimetry
DMA	-	Dynamic mechanical analysis
E'	-	Storage modulus
HNT	-	Halloysite nanotubes
MA	-	Maleic anhydride
MMT	-	Montmorillonite
OMMT	-	Organically modified montmorillonite
PE	-	Polyethylene
PET	-	Polyethylene terephthalate
Phr	-	Part per hundred resin
PP	-	Polypropylene
PP-g-AA	-	Polypropylene grafted acrylic acid
PP-g-MA	-	Polypropylene grafted maleic anhydride
PP-g-2-HEMA	-	Polypropylene grafted-2-hydroxyethyl methacrylate
PP-g-GMA	-	Polypropylene grafted glycidyl methacrylate
PVC	-	Polyvinyl chloride
SAN	-	styrene-co-acrylonitrile
SEM	-	Scanning electron microscope
Tan δ	-	Damping factor (tangent delta)
TEM	-	Transmission electron microscope
THF	-	Tetrahydrofuran

TGA	-	Thermogravimetry analysis
TPO	-	Thermoplastic polyolefin
T _g	-	Glass transition temperature
T _m	-	Melting temperature
T _c	-	Crystallization temperature
WAXS	-	Wide angle X-ray scattering
Wt%	-	Percentage by weight
XRD	-	X-ray diffraction
X _c	-	Degree of crystallinity
θ	-	Diffraction angle
ΔH _m	-	Heat of fusion
μm	-	Micrometer

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Polymer nanostructured material or more inclusively, polymer nanocomposite has created a key revolution in the 21st century exploiting the new properties and functionalities providing novel characteristics for newly developed materials. Two decades ago, researchers from Toyota Central Research and Development produced a new group of polymer-clay complexes or composites, which was called polymer layered silicate nanocomposites or polymer nanocomposites [1]. Today, various nanofillers are being used in nanocomposites. Cost and availability continue to change as the field is relatively new and several of these fillers are still being developed. These nanofillers may be halloysite nanotubes (HNT), carbon nanotubes (CNT), montmorillonite (MMT), inorganic particles, etc. Incorporation of these nanofillers into polymer matrices is a popular strategy used to develop new materials with specific beneficial properties, and this method has attracted considerable interest as indicated by numerous publications [2-5]. The property enhancements have allowed these materials to commercially compete with traditional materials. For that reason, polymer/clay systems hold great promise for industrial applications due to their ability to display synergistically advanced properties with relatively small amounts of clay loading [4]. To further utilize the unique performance of these nanoparticles, numerous researches have incorporated clay into PP matrix, one of the widely used nonpolar polyolefin thermoplastics [3].

The automotive industry pioneered the use of polymer nanocomposites. The first commercial application of these materials was the use of the nylon 6/clay nanocomposite as a timing belt covers for Toyota cars, for which a very small amount of nanofiller loading resulted in a pronounced improvement of thermal and mechanical properties [6]. Nearly 80% of polymer nanocomposites are consumed by the automotive and packaging industries. In addition to automotive applications, polymer/clay nanocomposites have been used to improve barrier resistance in beverage applications [7]. More recently, Mitsubishi Gas Chemical and Nanocor have co-developed nylon-6 nanocomposites for multi-layered polyethylene terephthalate (PET) for bottle applications [8]. All of this development demonstrated a significant improvement in a wide range of engineering properties by reinforcing polymers with clay on the the nanometer scale. Since then, extensive research in this field has been carried out globally. At present, development has been widened into almost all engineering polymers including polypropylene, polyethylene and poly(acrylonitrile-butadiene-styrene) [6].

In the last 50 years, polypropylene (PP) has become one of the most widespread commodities, employed in the industry due to its low cost, huge commercial market, wide range of applications and good mechanical performance. Today, PP is competing successfully with other environmentally less friendly and less versatile plastics. In order to improve PP's competitiveness in engineering resin applications, it is an important objective in PP compounding to simultaneously increase dimensional stability, stiffness, strength and impact resistance without sacrificing easy processability [3]. The preparation of PP based nanocomposites has been extensively studied [1, 2, 6, 9].

The reason behind blending PP with ABS is to combine the good process ability of PP with the high impact properties of ABS resin, eventually producing a polymer blend with improved properties. The large diversity of end uses makes ABS copolymer one of the most successful of the engineering thermoplastics available. Its position in terms of both properties and price between the more highly priced, high performance engineering plastics and the lower priced commodity plastics makes ABS the material of choice for many applications and the largest volume engineering thermoplastic sold [10]. However, blends of PP and ABS are immiscible throughout

the whole range of compositions and exhibited low impact toughness as large butadiene particles formed during the melt blending process reduce interfacial adhesion [11]. It does not mean that immiscible polymer blends are of little use and that the only solution is to search for miscible ones. In fact, immiscible polymer blends are much more interesting for commercial development [12]. This is because immiscibility allows the polymers to preserve the good features of each of the base polymer components of the blends. The challenge is to develop techniques that allow control of the properties when the polymer is subjected to the mechanical load. Such techniques are called compatibilization.

Considering the role of nanoclays in the enhancement of mechanical performances of polymers, it is expected that the addition of nanoclays into a PP/ABS blend system may further improve its mechanical and thermal properties. In this context, a recently published report on ABS/PP nanocomposites focused mainly on the study of morphology of ABS/clay nanocomposites blended with PP, its viscosity ratio, and dynamic mechanical properties [13]. Sung *et al.* [13] found that the intercalated structure of clay was obtained since the interlayer distance was increased and most of the clay existed in ABS continuous phase because of the affinity between the ABS and clay.

PP nanocomposites are relatively difficult to produce because PP does not contain any polar groups in its backbone chain, hence homogenous dispersion of the polar hydroxyl groups of organoclay in non-polar PP cannot be easily obtained [2, 3]. On the other hand, by introducing a polar functional group into the system, which is reactive toward the polymer matrix to the clay surface, higher degrees of dispersion of the clay layers could be achieved, thereby improving the mechanical properties of the nanocomposites [14]. The polar functional group most commonly used in this method is maleated PP or PP-g-MA. It is believed that the polar character of anhydride causes an affinity for the silicate surface such that the maleated PP or PP-g-MA can serve as a compatibilizer between the matrix and the filler.

Numerous studies on compatibilized PP/clay nanocomposites have been conducted including studies on the PP/PP-g-MA/clay which focused on the mechanical, morphological and rheological properties [15], optical transmittance and

mechanical properties of PP/PP-g-MA/clay [16], different types of compatibilizer on PP/clay [15] and recently, the mechanical and fracture behaviors of PP/PP-g-MA/clay [4] and so far, an improvement shown in most of the properties evaluated including mechanical and thermal properties. However, the study on PP/ABS/clay nanocomposites in the presence of compatibilizer has not yet been explored.

Although numerous studies concerning the effect of compatibilizers on PP nanocomposites have been reported, the purpose of this study is to better understand the influence of compatibilizer and filler content on the structure-property relationship of polymer blend. The focus will be on the changes occurring in mechanical, morphological and thermal properties of compatibilized PP/ABS blends and nanocomposites due to the incorporation of PP-g-MA.

1.2 Problem Statement

As mentioned earlier, the studies on PP/ABS blends and nanocomposites have received less attention and there are very limited reports on the production and the analysis of PP/ABS blends and its nanocomposites [11, 13, 17, 18]. The previous studies on ABS/PP nanocomposites concentrate mainly on the use of PP as the added polymers, with ABS as the main component [13]. The present study will focus on the effect of adding ABS into PP. However, the reason behind polymer blending in the materials development is to achieve a good combination set of properties and processability at moderate cost. As far as mechanical properties are concerned, the main target is to strike the balance of strength, stiffness and toughness. In order to achieve the goal, a filler such as MMT could be incorporated since MMT are extensively used in most of the polymer blends to produce a nanocomposites, but unfortunately, exfoliated or intercalated structure are not easily obtained without using compatibilizer [19]. Due to the immiscibility between PP and ABS, compatibilization is required. Although some of the researchers have established their work in this area but only a few of them used PP-g-MA as a compatibilizer. The effect of adding silicate layers nanofiller (MMT) into PP/ABS blends in the presence of compatibilizer (PP-g-MA) has not yet been explored. Therefore, it is interesting to

look at the system where compatibilized PP/ABS blends are combined with nanofiller thus affecting its mechanical, morphological and thermal properties.

1.3 Objectives

The overall objective of the study is to prepare and characterize PP/ABS blend and nanocomposites compatibilized with PP-g-MA via extrusion and injection molding to obtain the composite that has superior properties which suitable to be used in various application such as automotive application.

The objectives of this work can be divided into:

- 1) To determine the optimum PP/ABS blend formulations in order to achieve a good balance of mechanical properties.
- 2) To examine the effect of PP-g-MA as compatibilizer on PP/ABS blend and PP/ABS/MMT nanocomposites at optimum ratio of PP-g-MA.
- 3) To determine the effect of incorporating MMT into compatibilized and uncompatibilized PP/ABS blends at different ratios of MMT.

1.4 Scope of Study

The scope of this study comprised the following:

- (a) Sample preparation through melt blending method
 - i. Blending of PP with ABS to determine the optimum formulation based on mechanical properties using twin screw extruder.

- ii. Blending of PP-g-MA with PP/ABS blends to produce compatibilized PP/ABS blends using twin screw extruder.
- iii. Blending of PP-g-MA with PP/ABS nanocomposites to produce compatibilized PP/ABS nanocomposites using twin screw extruder.
- iv. The blends and nanocomposites fabricated into test specimens according to standard test method ASTM via injection molding for analysis.

(b) Mechanical, morphological, chemical characterization, and thermal analysis

- i. The entire samples were tested in order to study the mechanical properties of the blends and nanocomposites.
 - Tensile test (ASTM D638)
 - Flexural test (ASTM D790)
 - Impact test (ASTM D256)
- ii. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were carried out to evaluate the morphology of the blends and nanocomposites.
- iii. X-ray diffraction (XRD) was employed to characterize the formation of the nanocomposites.
- iv. Differential scanning calorimeter (DSC) was used to investigate the compatibility of the sample by obtaining thermal properties such as the glass transition temperature, melting temperature and degree of crystallinity.
- v. Thermogravimetric analysis (TGA) was done to determine the thermal stability of the nanocomposites.
- vi. Dynamic Mechanical Analysis (DMA) was conducted to determine the dynamic modulus and tan delta.

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