COMPATIBILISER EFFECTS ON PROPERTIES OF POLYAMIDE-6/ACRYLONITRILE-BUTADIENE-STYRENE AND POLYAMIDE-6/ACRYLONITRILE-BUTADIENE-STYRENE/SHORT GLASS FIBRE THERMOPLASTIC COMPOSITES

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‘For my lovely wife Amaliah Othman, my kids – Afifah, Afif, Afwan and Afrina for supporting me – together we are going to achieve our dream’

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ABSTRACT

Polyamide-6 (PA6), acrylonitrile-butadiene-styrene (ABS) and their blends are an important class of engineering thermoplastics that are widely used in electronic and automotive industries. Many efforts have been taken to improve the properties of both pure components and the blends. It was for this reason that the dynamic mechanical and rheological properties of PA6/ABS blend systems compatibilised by acrylonitrile-butadiene-styrene–maleic anhydride (ABS-g-MAH) was studied. The compatibiliser levels were kept up to 5wt. % in the blends. Short glass fibre (SGF) was used to improve the stiffness of the compatibilised blends and the fibre contents were from 10 to 30 wt. %. Therefore, the reason behind blending the PA6/ABS blends with short glass fibre was to balance the toughness and stiffness. Both the blends and corresponding composites were compounded using a counter-rotating twin screw extruder. Tensile, flexural and impact properties were determined using the injection moulded test samples according to ASTM standards. The mechanical properties of the blends and the composites were investigated in both static and dynamic modes. Rheological properties were investigated using rotational and capillary rheometer. In general, the mechanical strength either dynamic (refer to dynamic mechanical properties) or static conditions improved by incorporation of compatibiliser to the PA6/ABS blends. The incorporation of SGF into the PA6/ABS blends enhanced the mechanical strength but reduced the toughness of the composites. The rheological measurements confirmed the interaction between the blend components with the incorporation of compatibiliser has been improved. However, the compatibiliser has no favourable effect on the mechanical properties of the composites although it has significant effect on the blends of PA6/ABS. The compatibiliser increased the melt viscosity of the blends. The SGF increased the rheological properties especially viscosity and flowability of the composites. The optimum ratio of compatibiliser and SGF concentration were determined using power law, *n* and consistency index, *K* analyses. From the analysis, the optimum ratio obtained was 1.5 wt. % for 50/50 and 60/40 PA6/ABS blends and 3 wt. % for 70/30 PA6/ABS blends. The *n* values drastically decreased, when concentration of the SGF was about 20 wt % indicating more pseudoplastic nature for the composites and suggesting that, 20 wt % is the optimum SGF concentration.

ABSTRAK

Poliamida-6 (PA6), akrilonitril-butadiena-sterina dan adunannya merupakan satu bahan kejuruteraan termoplastik yang penting dan sangat luas penggunaanya dalam industri elektronik dan automotif. Pelbagai usaha telah diambil untuk memperbaiki sifat-sifat kedua-dua komponen dan adunannya, Ini menjadikan alasan kajian bagi sifat-sifat dinamik mekanikal dan reologi adunan PA6/ABS yang telah diserasikan oleh akrilonitril-butadiena-sterina-melaik anhadrida (ABS-g-MAH). Kandungan penserasi dalam adunan PA6/ABS telah ditetapkan sehingga 5 wt. %. Gentian kaca pendek (SGF) yang digunakan untuk mempebaiki kekakuan adunan yang diserasikan dan kandungannya diubah dari 10 hingga 30 wt. %. Oleh yang demikian, alasan disebalik campuran adunan PA6/ABS dengan gentian kekaca pendek adalah untuk mengimbangi sifat-sifat kekakuan dan kekukuhan adunan. Kedua adunan dan komposit diadun menggunakan penyemperit skru berkembar arah berlawanan. Sifat-sifat mekaniknya telah dikaji dalam keadaan mod static dan dinamik berdasarkan piawaian ASTM. Analisis dinamik mekanikal (DMA) telah dilakukan untuk mengkaji kelakukan dinamik mekanikal adunan dan komposit. Sifat-sifat reologi telah dikaji menggunakan alatan reologi rerambut dan pengayun. Secara umumnya, kekuatan mekanikal telah dipertingkatkan dengan penambahan penserasi ke dalam adunan PA6/ABS. Penambahan SGF ke dalam adunan juga telah mempetingkatkan kekuatan mekanikal bahan, tetapi menurunkan kekukuhan kompositnya. Keputusan reologi menunjukkan peningkatkan interaksi antara komponen adunan dengan penambahan SGF. Penserasi tidak mempunyai kesan terhadap sifat-sifat mekanik komposit, tetapi ada kesan yang ketara terhadap adunan PA6/ABS. Penserasi meningkatkan kelikatan leburan adunan. SGF pula meningkatkan sifat-sifat reologi komposit terutamanya kelikatan dan keboleh-alirannya. Nisbah kandungan penserasi dan SGF yang optimum telah ditentukan dengan menggunakan analisis indek hukum kuasa, *n* dan ketetapan, *K*. Dari analisis tersebut, kandungan optimum telah didapati adalah 1.5 wt % untuk adunan PA6/ABS 50/50 dan 60/40 PA6 dan 3 wt % pulak untuk adunan PA6/ABS 70/30. Penambahan SGF sebanyak 20 wt %, nilai indek hukum kuasa menurun secara mendadak menunjukkan komposit mempunyai sifat-sifat pseudoplastik yang jelas dan disimpulkan bahawa 20 wt % adalah kepekatan optimum bagi komposit PA6/ABS 60/40.

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LIST OF ABBREVIATIONS

ABS	Acrylonitrile butadiene styrene polymer
ABS-g-MAH	Acrylonitrile butadiene styrene-grafted-maleic anhydride
$CaCO_3$	Calcium carbonate
CPE	Crosslinked polyethylene
DMA	Dynamic mechanical analyser
DSC	Differential scanning calorimetry
EPR-g-MA	Ethylene propylene rubber-grafted-maleic anhydride
FRP	Fibre reinforced polymer
FTIR	Fourier transforms infrared
GMA-MMA	Glycidyl methacrylate/methyl methacrylate
HLMI	High load melt index
IA	Imidized acrylic
LCP	Liquid crystalline polymer
MA	Maleic anhydride
MAP	Maleic anhydride-grafted-polypropylene
MFI	Melt flow index
MMA-GMA	Poly(methyl methacrylate-co-glycidyl methacrylate)
MMA-MA	Poly(methyl methacrylate-co-maleic anhydride)
PA	Polyamide or nylon
PA/ABS	Polyamide/acrylonitrile butadiene styrene
PA6	Polyamide 6
PA6,6	Polyamide 6,6
PA6/ABS	Polyamide 6/acrylonitrile butadiene styrene
Par	Polyarylate
PB	Polybutadiene

PB-g-MA	Polybutadiene-grafted-maliec anhydride
PBT	Poly butylene terephtalate
PC	Polycarbonate
PE	Polyethylene
PEEK	Poly(ether ether ketone)
PEI	Poly(ether imide)
POE-g-MA	Metallocene polyethylene-grafted-maleic anhydride
PP	Polypropylene
PPO	Polyphenylene oxide
PPS	Poly(phenylene sulphide)
PVC	Polyvinyl chloride
SAN	Styrene acrylonitrile
SANMA	Styrene/acrylonitrile/maleic anhydride
SEBS	Styrene ethylene butylene styrene
SEBS-g-MA	Styrene ethylene butylene styrene-grafted-maleic anhydride
SGF	Short glass fibre
SMA	Styrene maleic anydride
UV	Ultraviolet

LIST OF SYMBOLS

τ_a	Apparent shear stress [Pa]
$\eta*$	Complex viscosity [Pa.s]
ρ	Density [m^3/kg]
D_f	Desirability factor [-]
X_i	Percentage of component, *i*, in blend [%]
P_i	Price of component *i*
$\dot{\gamma}$	Shear rate [s^{-1}]
$\phi_{polymer}$	Mass fraction of polymer [%]
$\dot{\gamma}_w$	Wall shear rate/actual shear rate [s^{-1}]
τ_w	Wall shear stress/actual shear stress [Pa]
τ	Stress [Pa]
ω	Frequency [s^{-1}]
γ	Strain [s^{-1}]
ΔH_c	Heat of crystallisation [J/kg]
ΔH_f	Heat of fusion [J/kg]
ΔH_{mix}	Heat of mixing [J/kg]
ΔH_m	Heat of melting [J/kg]
ϖ, Ω	Rotational velocity, angular velocity [s^{-1}]
ΔG_{mix}	Gibbs energy of mixing [J/kg]
η_w	Wall shear viscosity/actual shear viscosity [Pa.s]
	Apparent viscosity [Pa.s]
	Apparent shear rate [s^{-1}]

A	Pre-exponential factor [-]
a_T	Temperature shift factor [-]
c_p	Specific heat [J/kg.K]
E'	Dynamic mechanical storage modulus [Pa]
E''	Dynamic mechanical loss modulus [Pa]
E_a	Energy of activation for viscous flow [J/kg]
F	Force [N]
F_d	Dynamic or oscillatory force [N]
Fs	Static or clamping force [N]
G'	Rheological elastic or storage modulus [Pa]
$G*$	Complex modulus [Pa]
G''	Rheological viscous or loss modulus [Pa]
G_z	Graetz number [-]
Hz	Hertz
K	consistency index [$Pa.s^n$]
L_f	Flow length [m]
M	Torque [N.m]
m,wt.	weight [kg]
n	Power law index [-]
P_d	Driving pressure [Pa]
Q	Volumetric flow rate [m^3/s]
R	Universal gas constant [8.3144 J/kg.K]
T	Temperature [°C]
Tan δ	Loss tangent delta [-]
T_c	Crytallisation Temperature [°C]
T_g	Glass transition temperature [°C]
T_m	Melting temperature [°C]
V_e	Mean velocity [m/s]
X_c	Degree of crystallisation [%]
λ	Thermal conductivity [$W.K^{-1}.m^{-1}$]
η_o	Zero-shear viscosity [Pa.s]

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Polyamides (PA)s are particularly attractive class of polymers due to their good strength and stiffness, low friction and excellent chemical and wear resistance. The benefits of these polymer properties have led to the wide range of usage especially in automotive, electrical and mechanical applications. However, PAs have some disadvantages associated with their processing instability – high mould shrinkage and dimensional stability – due to their inherent properties of rapid crystallisation (Jang and Kim, 2000) and high moisture sensitivity because of their hygroscopic nature (Acierno and Puyvelde, 2004). These characteristics significantly limited their utility. Fortunately, the inherent chemical functionality of PA makes them suitable for modification. Therefore, several efforts have been proposed to minimise these drawbacks by blending with appropriate polymers or material such as nanomaterials and fibres (Wahit *et al.,* 2005).

Polyamide 6 (PA6), a thermoplastic and crystalline polymer, is widely used in many applications, such as electronic and automobile industries. PA6 is the most popular type of commercial available polyamide. PA6 is often blended with suitable elastomers with chemical functionality that can react with PA chain ends. PA6 also can be blended with other copolymers to reduce water absorption ability. Many authors also discussed various approaches for improving the toughness of PA6 by reacting polymers which contain appropriate chemical functionalities with acid or amine end groups of the PA during melt processing and also blending with elastomers such as acrylonitrile butadiene styrene (ABS) (Kudva *et al.,* 2000; Araujo *et al.,* 2002; Araujo *et al.,* 2003), ethylene-propylene-diene (EPDM), poly(phenylene oxide), polyolefin elastomer (Wahit *et al.,* 2006), ethylene copolymer (Triacca *et al.*, 1991) and natural rubber (NR) (Carone *et al*., 2000).

The main reasons behind the blending of ABS with PA6 is the relatively lower price of ABS compare to PA6, good processibility, low water absorption and high impact strength (Howe and Wolkowicz, 1987). ABS is also stronger in terms of impact properties than PA6 and has low mould shrinkage; even other mechanical and thermal properties are not as good as PA6. However, blends of PA6 and ABS are immiscible throughout the whole range of compositions and exhibit low impact toughness because large butadiene particles formed during the melt blending process reduce the interfacial adhesion (Tjong *et al.,* 2002). In the absence of compatibilisers, such blends lack the interfacial adhesion and generally exhibit poor mechanical properties. Therefore, reactive compatibilisation is the most promising way to enhance the interfacial adhesion and improve the compatibility of PA6 and ABS blends. Reactive compatibilsation is based on in-situ formation of block-or graft copolymer at the interface between the phases of the polymer blend during melt blending (Dedecker and Groenicky, 1998). In other word, the compatibilisers are miscible with one of the blend components and reactive with the other and will be forming a compatibilizing polymer at the interface. Few types of compatibilisers have been used in the previous studies of PA6/ABS blends, however, to the author's knowledge; there is limited literature available on using ABS-grafted-maleic anhydride (ABS-g-MAH) as compatibiliser for PA6/ABS blends. Therefore, in this research, a desirable combination of toughness of ABS and rigidity of PA6 will be realised by adding ABS-g-MAH as compatibiliser to enhance the phase adhesion of the blends.

Compatibilised PA6/ABS blends still have a few weaknesses, even though other properties could be improved. It has been shown that the strength of PA6/ABS blend especially tensile strength is lower than the virgin PA6 (Meincke *et al.*, 2004; Kudva *et al.*, 2000) and depending on the ratio of PA6 added into the blends which is about 50–70 wt % of ABS (Cho and Paul, 2001), impact property became poorer when the proportion of PA6 in the system was decreased (Chiu and Hsiao, 2004). The PA6 blends can be ‘supertough’ that is, having Izod impact strength higher than 800 J/m (Cho and Paul, 2001) however; it is believed that, the incorporation of a rubber phase in PA6 reduces the strength and stiffness relative to virgin PA6. Consequently, the blends of PA6 with ABS are still not the right answer to become an alternative material and will not contribute synergistic effects for the both of PA6 and ABS properties. Reinforcement by inorganic filled (Tjong and Xu, 2001) or short glass fibre (Nair *et al.*, 1997) can restore the required strength and stiffness of rubber toughened PA6s, leading to the formation of ternary or hybrid composites.

The addition of rubber or elastomeric materials such as ABS to PA6 leads to a reduction of strength and stiffness of the obtained blend. In contrast, reinforcing thermoplastics by short glass fibre (SGF) is known to improve both strength and mechanical stiffness (Tjong *et al*., 2000; Fu *et al*., 2000), however high content of glass fibres (GF) are necessary to achieve high strength and high stiffness (Fu and Lauke, 1998; Fu *et al*., 2000; Bader and Collins, 1983; Biolzi *et al*., 1994). Unfortunately, height content of GF leads considerable loss in toughness and ductility when these short glass fibres incorporated to the composite (Ahn and Paul, 2006). Therefore, the combination of reinforcement and blending ABS are expected to balance the impact and stiffness of the materials.

Some critical questions would arise; will this composite be easily processed through injection moulding or any other thermoplastics processing methods? Will this composite material be easily moulded to form small and critical parts? Most of the composite materials containing fibres are difficult to be produced by injection moulding due to their high viscosity. The composite materials are then processed either by using compression moulding or extrusion. Thus, in order to investigate the processibility, the rheological properties of the composite have to be thoroughly investigated by using rheological apparatus such as dynamic and capillary rheometers.

The dynamic mechanical properties study of PA6/ABS either composite or blends are also very limited in the literatures (Aoki and Watanabe, 1992). It is because many researchers thought that, a static mechanical investigation is enough to understand the mechanical behaviour of the blends and composites (Sun *et al.*, 2005; Lai *et al.*, 2005; Jang and Jim, 2000). However, in reality, these materials are being used in dynamic application. Therefore, the dynamic mechanical properties are to be investigated thoroughly in this study in order to understand the dynamic mechanical behaviour during commercial application such as interior parts of automobile, motor housings and covers.

Additionally, many authors have investigated the rheological properties of constituent of PA6 or ABS (Lee *et al.*, 2002) and its blends (Gao *et al.*, 1999; Kudva *et al.*, 1999) and only a few on its composites (Benderly *et al.*, 1999; Cho and Paul, 2001). Therefore, the rheological information of the PA6 composites is very limited. Therefore, the development of a GF reinforced PA6/ABS composite and characterization of rheological properties were emphasised. Then, the rheological data can be used to predict the performance and behaviour of the final product of the composites (Braun and Rosen, 2000).

1.2 Problem Statements

The important reason in polymer blend either reinforced or non-reinforced development is to achieve a good set of combination properties in addition to processibility. Since, only a few literature reviews have been reported on short glass fibre reinforced PA6/ABS composites, it is the objective of this research to investigate specifically the mechanical and rheological properties of the composites. Until now, the rheological properties of non-reinforced PA6/ABS blends have only been studied for a narrow range of compositions (Jafari *et al.*, 2002). In this study, the following three new areas which are not being discussed by previous researchers will be presented:-

i. The use of ABS-g-MAH as compatibiliser

ii. The use of DMA as a tool to study the dynamic mechanical properties of PA6/ABS blends and composites

iii. The use of rheometer (rotational and capillary) to study the rheological properties of PA6/ABS composites.

While prior research has been performed on the rheology and dynamic mechanical properties of PA6/ABS blends, more extensive analysis on the glass fibre reinforced PA6/ABS composites is still quite necessary due to many questions still unanswered. This study deals with the dynamic mechanical, processing, thermal, and morphology of the PA6/ABS blends and compositions.

The resulting balance of crystalline of PA6 and amorphous of ABS properties reportedly makes the alloys suitable for automotive interior moldings (like consoles), consumer products, appliance housings and sporting goods. Also, the PA6/ABS composites are expected to be suitable for electronic application such as vacuum cleaner housings and for automotive applications.

The questions addressing the current problems to be investigated or discussed and explained in the present study are as follow:

i. How the composition of SGF affects the thermal, rheological, static as well as dynamic mechanical properties of PA6/ABS composites?

ii. Does the compatibiliser effect on thermal, rheological, static as well as dynamic mechanical properties of PA6/ABS composites?

iii. What are the correlate changes in structure and morphology of the compatibilised composites with changes in thermal, rheological, static as well as dynamic mechanical behaviour?

iv. What is the optimum composition of short glass fibre, referring to the dynamic mechanical and rheological properties?

1.3 Objectives

The objectives of the study have been achieved by carried-out the study into three stages. First is to study the effect of ABS in PA6 blends without compatibiliser. This study focuses on mechanical properties and thermal properties. The study on the effect of compatibiliser in the blends will be conducted in the second stage.

The study on the dynamic mechanical and rheological properties of polymer blends is of great theoretical and practical importance that will help to understand the dynamic mechanical behaviour of the blends and the rheological properties of polymer blends and composites.

Overall, the objectives of this study are:

i. To study the effect of SGF composition on thermal, rheological, static as well as dynamic mechanical properties of PA6/ABS composites.

ii. To study the effect of compatibiliser on thermal, rheological, static as well as dynamic mechanical properties of PA6/ABS composites.

iii. To correlate changes in structure and morphology of the compatibilised composites with changes in thermal, rheological, static as well as dynamic mechanical behaviour.

iv. To determine the optimum composition of SGF, referring to the dynamic mechanical and rheological properties.

1.4 Scopes of Study

The scopes involved the preparation of PA6/ABS blends using the melt intercalation method in a twin screw extruder. The composites were reinforced with varying compositions reinforced with SGF of a specified length using injection moulding. The thermoplastic composites were then evaluated for thermal, rheological, static and dynamic mechanical properties with and without incorporation of a stabilizer, maleic anhydride. These tests were: thermal (T_g, T_m and degree of crystallinity) rheological (viscosity and effect of temperature on viscosity), static (tensile, flexural and impact strength) as well as dynamic mechanical (storage modulus, loss modulus and tan δ).

The observations obtained from tests were explained and correlated to the structural changes by using FTIR and for morphology by using scanning electron microscopy.

In order to achieve the objectives, the scopes covered are as follow:

1. Blending of PA6/ABS with and without compatibiliser

    - In this work, sample was prepared using melt intercalation method which was carried out using a twin-screw extruder over the set range of compositions between ABS, PA6 and ABS-g-MAH. This was followed by the injection moulding process to prepare test specimen according to the ASTM testing standard.

    - There were two set of samples with the set of range composition prepared for testing and analysing: uncompatibilised PA6/ABS blends and compatibilised PA6/ABS blends.

- The PA6 contents in PA6/ABS blends range from 70% - 50% weight ratio. While, the ABS-g-MAH percentage as compatibiliser was varied from 1, 3 and 5 wt. %.

2. Blending of PA6/ABS composites

   - The composite samples were prepared using melt intercalation method.

   - The PA6, ABS and ABS-g-MAH compositions were selected based on the optimum ratio, which was obtained from the study of polymer blends.

   - The amounts of SGF were added into PA6/ABS blends gradually from 0 to 30 wt %.

3. The entire samples specimens were tested in order to study the mechanical and dynamic mechanical properties: - tensile, flexural and impact according to ASTM standard as well as dynamic mechanical analysis (DMA).

4. Differential scanning calorimetery (DSC) was used to investigate the compatibility of the sample by obtaining thermal properties; the glass transition temperature, melting temperature and degree of crystallinity.

5. Rheological studies – capillary and rotational rheometers were used to investigate the rheological parameters of polymer composites and blends.

6. Scanning electron microscopy analysis was carried out to evaluate the morphological structure of the blends and composites.

7. Fourier transforms infrared analysis was carried out to confirm the reaction during melt intercalation process.

1.5 Thesis Outline

This thesis includes five chapters. Chapter 1 is an introduction of the study which is discussing about the background, problems statement, objectives and scope. Chapter 2 contains a literature review which is discussing the theoretical studies reported and published regarding the polymer blending, polymer composite and the detail of the related study of PA6/ABS blends and PA6/ABS composites by previous researchers. Chapter 3 discusses the materials used in this study and also the testing methods involved such as the mechanical testing, thermal investigation, morphological analysis and rheological analysis of PA6/ABS blends and composites. Chapter 4 describes the results of experiments and discusses the reasons and findings. Chapter 5 is the general conclusion and a summary of the key results of this thesis and a list of recommendations for future works.

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