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

AGUS BIN ARSAD

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

AGUS BIN ARSAD

A thesis submitted in fulfilment of the requirements for the award of the degree of Doctor of Philosophy (Polymer Engineering)

Faculty of Chemical and Natural Resources Engineering
Universiti Teknologi Malaysia

JUNE 2010
‘For my lovely wife Amaliah Othman, my kids – Afifah, Afif, Afwan and Afrina
for supporting me – together we are going to achieve our dream’
ACKNOWLEDGEMENTS

I am grateful to my supervisor, Assoc. Prof. Dr. Abdul Razak Rahmat for his encouragement, guidance, criticism and academic freedom during the working in this project. The high standards set by him have not only greatly contributed to the quality of this work but also helped me become a better person.

I thank Prof Dr. Azman Hassan and the late Prof. Dr. Mohd Nasir Zainal Arif for skilfully co-supervising my PhD. This work would not have been possible without their research support and useful suggestions. I also thank Assoc. Prof. Dr. Mat Uzir Wahit for his support during my hard time and sharing happiness together. I also extend my appreciation to Pn Munirah Mokhtar for helping me to be my proof reader of my thesis.

I also wish to express my appreciation to the technical members of Polymer Engineering, especially Ms. Zainab Salleh, Mr. Suhee Tan and Mr. Nordin Ahmad, the Quality Control staffs at Poly-Star Compounds Sdn. Bhd. and Nuclear Malaysia, Bangi for their help and support in my research.

I wish to thank Mr. Shaiful Nizam Iskandar, Polyethylene (M) Sdn. Bhd. for helpful discussions and exchange of ideas regarding rheology of the polymer blends. Special thanks, all the staff of Technical Service Laboratory, Polyethylene (M) Sdn Bhd, Kertih, for giving me opportunity to use their capillary and dynamic rheology, without them, my rheological study could not be materialised.

I would like to acknowledge Ministry of Higher Education (MOHE) grant 78142 and UTM Initial Research Student (IRGS) Grant 75223 throughout my studies.

I would also like to extend my gratitude to my family, especially mom-in-law for their inspiration and continuing support. Most of all, I thank my wife Amaliah Othman and my children, Afifah, Afif, Afwan and Afrina who have been a source of strength and motivation for success. I hope that by now they have forgiven me for all the weekends and long hours spent away in the laboratory.
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

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<thead>
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<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>ABS</td>
<td>Acrylonitrile butadiene styrene polymer</td>
</tr>
<tr>
<td>ABS-g-MAH</td>
<td>Acrylonitrile butadiene styrene-grafted-maleic anhydride</td>
</tr>
<tr>
<td>CaCO₃</td>
<td>Calcium carbonate</td>
</tr>
<tr>
<td>CPE</td>
<td>Crosslinked polyethylene</td>
</tr>
<tr>
<td>DMA</td>
<td>Dynamic mechanical analyser</td>
</tr>
<tr>
<td>DSC</td>
<td>Differential scanning calorimetry</td>
</tr>
<tr>
<td>EPR-g-MA</td>
<td>Ethylene propylene rubber-grafted-maleic anhydride</td>
</tr>
<tr>
<td>FRP</td>
<td>Fibre reinforced polymer</td>
</tr>
<tr>
<td>FTIR</td>
<td>Fourier transforms infrared</td>
</tr>
<tr>
<td>GMA-MMA</td>
<td>Glycidyl methacrylate/methyl methacrylate</td>
</tr>
<tr>
<td>HLMI</td>
<td>High load melt index</td>
</tr>
<tr>
<td>IA</td>
<td>Imidized acrylic</td>
</tr>
<tr>
<td>LCP</td>
<td>Liquid crystalline polymer</td>
</tr>
<tr>
<td>MA</td>
<td>Maleic anhydride</td>
</tr>
<tr>
<td>MAP</td>
<td>Maleic anhydride-grafted-polypropylene</td>
</tr>
<tr>
<td>MFI</td>
<td>Melt flow index</td>
</tr>
<tr>
<td>MMA-GMA</td>
<td>Poly(methyl methacrylate-co-glycidyl methacrylate)</td>
</tr>
<tr>
<td>MMA-MA</td>
<td>Poly(methyl methacrylate-co-maleic anhydride)</td>
</tr>
<tr>
<td>PA</td>
<td>Polyamide or nylon</td>
</tr>
<tr>
<td>PA/ABS</td>
<td>Polyamide/acrylonitrile butadiene styrene</td>
</tr>
<tr>
<td>PA6</td>
<td>Polyamide 6</td>
</tr>
<tr>
<td>PA6,6</td>
<td>Polyamide 6,6</td>
</tr>
<tr>
<td>PA6/ABS</td>
<td>Polyamide 6/acrylonitrile butadiene styrene</td>
</tr>
<tr>
<td>Par</td>
<td>Polyarylate</td>
</tr>
<tr>
<td>PB</td>
<td>Polybutadiene</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Full Name</td>
</tr>
<tr>
<td>--------------</td>
<td>-----------</td>
</tr>
<tr>
<td>PB-g-MA</td>
<td>Polybutadiene-grafted-maleic anhydride</td>
</tr>
<tr>
<td>PBT</td>
<td>Poly butylene terephtalate</td>
</tr>
<tr>
<td>PC</td>
<td>Polycarbonate</td>
</tr>
<tr>
<td>PE</td>
<td>Polyethylene</td>
</tr>
<tr>
<td>PEEK</td>
<td>Poly(ether ether ketone)</td>
</tr>
<tr>
<td>PEI</td>
<td>Poly(ether imide)</td>
</tr>
<tr>
<td>POE-g-MA</td>
<td>Metallocene polyethylene-grafted-maleic anhydride</td>
</tr>
<tr>
<td>PP</td>
<td>Polypropylene</td>
</tr>
<tr>
<td>PPO</td>
<td>Polyphenylene oxide</td>
</tr>
<tr>
<td>PPS</td>
<td>Poly(phenylene sulphide)</td>
</tr>
<tr>
<td>PVC</td>
<td>Polyvinyl chloride</td>
</tr>
<tr>
<td>SAN</td>
<td>Styrene acrylonitrile</td>
</tr>
<tr>
<td>SANMA</td>
<td>Styrene/acrylonitrile/maleic anhydride</td>
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<tr>
<td>SEBS</td>
<td>Styrene ethylene butylene styrene</td>
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<tr>
<td>SEBS-g-MA</td>
<td>Styrene ethylene butylene styrene-grafted-maleic anhydride</td>
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<tr>
<td>SGF</td>
<td>Short glass fibre</td>
</tr>
<tr>
<td>SMA</td>
<td>Styrene maleic anydride</td>
</tr>
<tr>
<td>UV</td>
<td>Ultraviolet</td>
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## LIST OF SYMBOLS

- $\tau_a$: Apparent shear stress [Pa]
- $\eta^*$: Complex viscosity [Pa.s]
- $\rho$: 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$^{-1}$]
- $\phi_{\text{polymer}}$: Mass fraction of polymer [%]
- $\dot{\gamma}_w$: Wall shear rate/actual shear rate [s$^{-1}$]
- $\tau_w$: Wall shear stress/actual shear stress [Pa]
- $\tau$: Stress [Pa]
- $\omega$: Frequency [s$^{-1}$]
- $\gamma$: Strain [s$^{-1}$]
- $\Delta H_c$: Heat of crystallisation [J/kg]
- $\Delta H_f$: Heat of fusion [J/kg]
- $\Delta H_{\text{mix}}$: Heat of mixing [J/kg]
- $\Delta H_m$: Heat of melting [J/kg]
- $\omega$, $\Omega$: Rotational velocity, angular velocity [s$^{-1}$]
- $\Delta G_{\text{mix}}$: Gibbs energy of mixing [J/kg]
- $\eta_w$: Wall shear viscosity/actual shear viscosity [Pa.s]
- $\eta_a$: Apparent viscosity [Pa.s]
- $\eta_{\text{app}}$: Apparent shear rate [s$^{-1}$]
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Unit</th>
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<tbody>
<tr>
<td>$A$</td>
<td>Pre-exponential factor</td>
<td>[-]</td>
</tr>
<tr>
<td>$a_T$</td>
<td>Temperature shift factor</td>
<td>[-]</td>
</tr>
<tr>
<td>$c_p$</td>
<td>Specific heat</td>
<td>[J/kg.K]</td>
</tr>
<tr>
<td>$E'$</td>
<td>Dynamic mechanical storage modulus</td>
<td>[Pa]</td>
</tr>
<tr>
<td>$E''$</td>
<td>Dynamic mechanical loss modulus</td>
<td>[Pa]</td>
</tr>
<tr>
<td>$E_a$</td>
<td>Energy of activation for viscous flow</td>
<td>[J/kg]</td>
</tr>
<tr>
<td>$F$</td>
<td>Force</td>
<td>[N]</td>
</tr>
<tr>
<td>$F_d$</td>
<td>Dynamic or oscillatory force</td>
<td>[N]</td>
</tr>
<tr>
<td>$F_s$</td>
<td>Static or clamping force</td>
<td>[N]</td>
</tr>
<tr>
<td>$G'$</td>
<td>Rheological elastic or storage modulus</td>
<td>[Pa]</td>
</tr>
<tr>
<td>$G^*$</td>
<td>Complex modulus</td>
<td>[Pa]</td>
</tr>
<tr>
<td>$G''$</td>
<td>Rheological viscous or loss modulus</td>
<td>[Pa]</td>
</tr>
<tr>
<td>$G_z$</td>
<td>Graetz number</td>
<td>[-]</td>
</tr>
<tr>
<td>$Hz$</td>
<td>Hertz</td>
<td>[Hz]</td>
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<tr>
<td>$K$</td>
<td>Consistency index</td>
<td>[Pa.s^n]</td>
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<tr>
<td>$L_f$</td>
<td>Flow length</td>
<td>[m]</td>
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<tr>
<td>$M$</td>
<td>Torque</td>
<td>[N.m]</td>
</tr>
<tr>
<td>$m_{wt.}$</td>
<td>Weight</td>
<td>[kg]</td>
</tr>
<tr>
<td>$n$</td>
<td>Power law index</td>
<td>[-]</td>
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<tr>
<td>$P_d$</td>
<td>Driving pressure</td>
<td>[Pa]</td>
</tr>
<tr>
<td>$Q$</td>
<td>Volumetric flow rate</td>
<td>[m$^3$/s]</td>
</tr>
<tr>
<td>$R$</td>
<td>Universal gas constant</td>
<td>[8.3144 J/kg.K]</td>
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<tr>
<td>$T$</td>
<td>Temperature</td>
<td>[°C]</td>
</tr>
<tr>
<td>Tan $\delta$</td>
<td>Loss tangent delta</td>
<td>[-]</td>
</tr>
<tr>
<td>$T_c$</td>
<td>Crystallisation Temperature</td>
<td>[°C]</td>
</tr>
<tr>
<td>$T_g$</td>
<td>Glass transition temperature</td>
<td>[°C]</td>
</tr>
<tr>
<td>$T_m$</td>
<td>Melting temperature</td>
<td>[°C]</td>
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<tr>
<td>$V_e$</td>
<td>Mean velocity</td>
<td>[m/s]</td>
</tr>
<tr>
<td>$X_c$</td>
<td>Degree of crystallisation</td>
<td>[%]</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>Thermal conductivity</td>
<td>[W.K$^{-1}$.m$^{-1}$]</td>
</tr>
<tr>
<td>$\eta_0$</td>
<td>Zero-shear viscosity</td>
<td>[Pa.s]</td>
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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 compatibilisation 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

   o 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.

   o 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.
o 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

o The composite samples were prepared using melt intercalation method.

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

o 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 calorimetry (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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