# PREPARATION AND CHARACTERISATION OF KENAF BAST FIBRE REINFORCED RECYCLED POLYPROPYLENE/RECYCLED POLYAMIDE 6 COMPOSITES

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

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To my beloved mother (Noresah Md Seh), late father (Suradi Musa), and husband (Muhamad Ridhwan Abdullah) for your wonderful spirit, encouragement, and love

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#### ABSTRACT

The purpose of this study was to investigate the effects of kenaf bast fibre (KBF) reinforced recycled polypropylene (rPP)/recycled polyamide 6 (rPA6) composites on mechanical, morphological, thermal and rheological properties. KBF was treated with alkaline treatment and then rPP and rPA6 were prepared by extruding using single screw extruder. Alkaline treated KBF was then reinforced with rPP/rPA6/ maleic anhydride grafted polypropylene (MAPP) blends and the mechanical, morphological, thermal and rheological properties were analysed. The amount of MAPP was fixed at 5 wt. % for all rPP/rPA6 blends. The blends ratios for rPP/rPA6/MAPP were 65/30/5, 45/50/5 and 25/70/5. The best mechanical properties of rPP/rPA6/MAPP blends ratios (25/70/5) then added with various amount of KBF compositions from 0 to 30 parts per hundred resin (phr). The addition of KBF caused the flexural strength and flexural modulus increased proportionally with increasing in its compositions in the composites. The tensile strength, Young's modulus, and hardness of composites remain unaffected with the increase in KBF composition. However, the elongation at break and impact strength of composites decreased with the increased in KBF compositions. From scanning electron microscope (SEM) micrograph, it was observed that the increase in the KBF compositions caused better dispersion, less interfacial gaps and void spaces. The melting temperature (T<sub>m</sub>), crystallization temperature (T<sub>c</sub>), degradation temperature (T<sub>deg</sub>), and onset temperature (Tonset) decreased with the increase in KBF compositions in the composites. The melt flow index and rheological properties decreased with the increasing of KBF compositions in the composites. As for conclusion, KBF successfully functioned as reinforcement for rPP/rPA6/MAPP composites.

#### ABSTRAK

Tujuan kajian ini adalah untuk mengkaji kesan gentian basta kenaf (KBF) yang bertetulang komposit polipropilena kitar semula (rPP)/poliamida (rPA6) kitar semula dari aspek mekanikal, morpologi, sifat terhadap haba dan sifat reologi. Dalam kajian ini, KBF telah dirawat dengan rawatan alkali dan kemudiannya rPP dan rPA6 diproses menggunakan penyemperitan skru tunggal. KBF yang telah dirawat kemudiannya digunakan untuk memperkukuh campuran rPP/rPA6/ maleik anhidrida yang dicangkuk dengan polipropilena (MAPP) dan aspek mekanikal, morpologi, sifat terhadap haba dan sifat reologi telah dianalisis. Penambahan ditetapkan pada 5 pada semua campuran rPP/rPA6. wt% MAPP Nisbah campuran bagi rPP/rPA6/MAPP dikaji adalah 65/30/5, 45/50/5 dan 25/70/5. Campuran rPP/rPA6/MAPP dengan sifat-sifat mekanikal terbaik (25/70/5) kemudiannya ditambah dengan komposisi KBF yang berbeza dari 0-30 bahagian dari seratus jumlah keseluruhan resin (phr). Dari kajian ini, penambahan KBF menyebabkan kekuatan lenturan dan lenturan modulus meningkat berkadaran dengan peningkatan komposisi KBF dalam komposit. Kekuatan regangan, modulus Young dan kekerasan komposit tidak berubah dengan peningkatan komposisi KBF dalam komposit. Bagaimanapun, sifat pemanjangan ketika putus dan kekuatan hentaman berkurangan dengan peningkatan komposisi KBF dalam komposit. Berdasarkan mikrograf mikroskop electron pengimbas (SEM), diperhatikan penambahan KBF menyebabkan penyebaran yang lebih baik, kurang ruang antara muka dan ruang lompang yang kelihatan. Suhu takat lebur (T<sub>m</sub>), suhu penghabluran (T<sub>c</sub>), suhu kemusnahan (T<sub>deg</sub>), dan suhu permulaan (T<sub>onset</sub>) berkurangan dengan peningkatan komposisi KBF dalam komposit. Indeks aliran leburan dan sifat-sifat reologi menurun dengan peningkatan komposisi KBF dalam campuran. Kesimpulannya, KBF berjaya bertindak sebagai pengukuh bagi campuran rPP/rPA6/MAPP.

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### LIST OF ABBREVIATIONS

APS	-	3-aminopropyltriethoxysilane
ASA	-	Acrylate-styrene-acrylonitrile
DSC	-	Differential scanning calorimetry
DTG	-	Derivative thermogravimetry
EPR-g-MA	-	Maleic anhydride-grafted-ethylene-propylene rubber
F-ASA	-	Functioned group acrylate-styrene-acrylonitrile
$H_2SO_4$	-	Sulphuric acid
KBF	-	Kenaf bast fibres
KCF	-	Kenaf core fibres
KF	-	Kenaf fibres
LNR	-	Liquid natural rubber
MAH	-	Maleic anhydride
MAPP	-	Maleic anhydride grafted polypropylene
MFI	-	Melt flow index
MFR	-	Melt flow rate
MVR	-	Melt volume rate
NaOH	-	Sodium hydroxide
NBR	-	Nitrile butyl rubber
OH	-	Hydroxyl
PA	-	Polyamide
PA6	-	Polyamide 6
PE	-	Polyethylene
phr	-	Part of resin
PP	-	Polypropylene
QReC	-	Quality Reagent Chemical
rPA6	-	Recycled polyamide 6
rpm	-	Rotation per minutes

rPP	-	Recycled polypropylene
SEBS	-	Styrene ethylene-butylene-styrene
SEBS-g-MA	-	Maleated styrene-ethylene-butylene-styrene
SMA	-	Styrene-co-maleic anhydride
T <sub>c</sub>	-	Crystalline temperature
T <sub>deg</sub>	-	Degradation temperature
TG	-	Thermogravimetry
Tg	-	Glass transition temperature
TGA	-	Thermogravimetric analysis
T <sub>m</sub>	-	Melting temperature
Tonset	-	Onset temperature
UTM	-	Universal testing machine
v/v	-	Volume per volume
vol.%	-	Volume percents
W/V	-	Weight per volume
w/w	-	Weight per weight
wt.%	-	Weight percents
X <sub>c</sub>	-	Crystalline percentages
X <sub>c</sub> -rPA6	-	Crystallinity of rPA6
X <sub>c</sub> -rPP	-	Crystallinity of rPP
$\Delta H_{m}$	-	Heat of fusion

## LIST OF SYMBOLS

°C	-	Degree Celsius
°C/min	-	Degree Celsius per minutes
μm	-	Micrometre
cm	-	Centimetre
g	-	Grams
g/10 min	-	Grams over 10 minutes
g/cm <sup>3</sup>	-	Gram over cubic centrimetres
GPa	-	Giga Pascal
J	-	Joule
J/g	-	Joule per grams
J/m	-	Joule per metres
kg	-	Kilogram
m	-	Metre
mg	-	miligram
mm	-	Millimetre
mm/min	-	Milimetre per minutes
MPa	-	Megapascal
Ν	-	Newton

### LIST OF APPENDICES

#### APPENDIX

#### TITLE

#### А Kenaf Bast Fibres (KBF) Characterization 88 В Bulk Density Characterization for KBF 89 С Melt Flow Index (MFI) Test for rPP and rPA6 90 **KBF** Before and After Alkaline Treatment 91 D E Publication 1: Journal of Polymer Engineering (Impact factor: 0.408) 92 Publication 2: Technical paper presented in F The Asian International Conference on Materials, Minerals, and Polymer (MAMIP 93 2012) G Publication 3: Technical paper presented in The University Malaysia Terengganu Annual Seminar (UMTAS 2012) 94

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#### **CHAPTER 1**

#### **INTRODUCTION**

#### 1.1 Background of Study

Over the past few years, natural fibres have gained the interest of the researchers as substitute for the synthetic fibres (Yousif *et al.*, 2012). Natural fibres have many advantages over synthetic fibres such as good relative mechanical properties, lighter weight, and lower cost (Kassim *et al.*, 2012). Other than that, natural fibres are abundant and renewable. Nowadays, the use of natural fibres is common in automotive, construction and furniture industries (Yousif *et al.*, 2012).

Kenaf fibre is included in the natural fibres category. Due to attractive features of kenaf fibres as reinforcing materials over others natural fibres, comprehensive studies of kenaf fibres were done (Ren *et al.*, 2012). However, usually kenaf fibres face compatibility problems with the polymeric materials (El-Shekeil *et al.*, 2012b). The hydrophilic nature of kenaf fibres is incompatible with hydrophobic polymeric materials (Kassim *et al.*, 2012). Hence, researchers favoured the use of surface treatment on kenaf fibres to solve this problem. It was found that the alkaline treatment enhances matrix-fibres adhesion by increasing roughness and also removing impurities of the fibres (Yousif *et al.*, 2012).

Kenaf fibres have two main components, the core and bast. In terms of mechanical properties, kenaf bast fibres (KBF) are found to have higher tensile, flexural, and impact properties than kenaf core fibres (KCF) (Ishak *et al.*, 2010). In

this study, KBF has been chosen over KCF for its ecological advantages and its mechanical properties.

Kenaf fibre reinforced polymer composites are studied intensively by researchers (El-Shekeil *et al.*, 2012b; Lee *et al.*, 2012). This is due to the increasing interest in the development of environmentally friendly material. Kenaf fibres has the potential to replace synthetic fibres due to its low cost, low density, acceptable specific strength properties, ease of separation, carbon dioxide sequestration and biodegradability.

At present, KBF has been used as reinforcement for conventional polymers such as polypropylene. Polypropylene (PP) is an important polymer in packaging, medical instrumentation, and automotive industries (Ezat *et al.*, 2012). PP exhibits good strength, is easily processed, lightweight and economical. The mechanical properties of PP were improved with small amounts of filler (Khanjanzadeh and Tabarsa, 2012).

On the other hand, polyamide 6 (PA6) is well known as an engineering thermoplastic with excellent strength and stiffness, low friction, and chemical resistance (Liu *et al.*, 2012). However, PA6 has drawbacks such as poor dimensional stability and high water absorption (Liao and Tjong, 2011). These drawbacks limit the industrial use of PA6. Hence, numerous efforts have been made to overcome these drawbacks such as the addition of inorganic filler.

PA6 are usually blended with PP to improve the properties of final products (Golfazani *et al.*, 2012). The poor processability and high moisture absorption of PA6 was compensated by the good processability and low moisture absorption of PP. Previously, researchers have suggested that the combination of PP and PA6 balances the properties of the final product (Chow *et al.*, 2005). Due to the excellent properties of PP/PA6 blends, researchers favour to use of these polymer combinations for their study (Wang *et al.*, 2012; Golfazani *et al.*, 2012). However,

due to the polarity differences and crystalline structure, the PP and PA6 were chemically immiscible (Jung *et al.*, 2007). Therefore to overcome the immiscible problems, the addition of maleic anhydride grafted polypropylene (MAPP) compatibilizer is necessary. The compatibilizer was use to reduces the interfacial tension of PP and PA6. Hence, it improves the interfacial adhesion between PP and PA6.

The better mechanical properties of composites show with MAPP rather than others compatibilizer (Jung *et al.*, 2007). It is due to the compatibilizing effects between maleic anhydride (MA) with amide group in PA6. The applications of MAPP in the PP/PA6 system was not a new issue since it has been studies previously by Takahashi (2002) and Li *et al.* (2010). Even though, PP/PA6 system has been studied intensively, but the studies on recycled PP/PA6 system remain unexplored.

At present, the applications of non-biodegradable PP and PA6 become common. The widespread consumption of these plastics in industries has implications to the environment (Khanjanzadeh and Tabarsa, 2012; Lee *et al.*, 2012). Environment problems have been created environmental problems due to excessive consumption of these polymers (Lee *et al.*, 2012). Finally, the excessive consumption of these polymers ends up in landfills. In response to these problems, the industrial applicator added a small amount of recycled polymers with virgin polymers in their industrial process. However, this practice is not sufficient to overcome these problems. Hence, instead of virgin PP and PA6, recycled PP and PA6 were chosen in this study. The purpose of this study is to suggest a solution of these environmental problems.

### **1.2** Problems Statement

Even though intensive studies on PP/PA6 system have been carried out, but the problems caused by these non-biodegradable polymers are still not resolved. Hence, the use of virgin PP and PA6 was replaced with recycled polymers to reduce these environmental problems. Since, PP/PA6 system is incompatible, MAPP compatibilizer was added to ensure optimum compatibilization effects of recycled PP (rPP) /recycled PA6 (rPA6) blends. KBF was added as reinforcement agents. The research questions that need to be answered are as follows;

- i. What is the rPP/rPA6 ratio that provides the best mechanical and thermal properties?
- ii. What are the effects of the KBF ratio on mechanical, morphological, thermal, and rheological properties of composites?
- iii. What is KBF ratio that provides the best mechanical, morphological, thermal, and rheological properties of composites?

#### 1.3 Objectives of Study

The objectives of this study are to produce composites of rPP /rPA6/MAPP reinforced KBF.

- i. To determine rPP/rPA6 ratio that gives the best mechanical and thermal properties.
- ii. To investigate the effects of KBF ratio on mechanical, morphological, thermal, and rheological properties of composites.
- iii. To determine the best KBF ratio that gives the best mechanical, thermal, and rheological properties of composites.

#### **1.4** Scope of Study

- i. Literature reviews were done on the related study of KBF, chemical modification of KBF, properties of PP and PA6.
- ii. The study was involved the following steps:
  - a) Alkaline treatment of KBF.
  - b) Preparation of rPP and rPA6, MAPP was fixed at 5 phr for all rPP/rPA6/MAPP blends.
  - c) Blending of KBF reinforced rPP/rPA6/MAPP, maximum KBF ratio fixed at 30 parts per hundred (phr) based on total polymer blend weight.
  - d) Sample characterizations, by comparing the properties with and without KBF using:
    - Mechanical characterisations using tensile test, flexural test, hardness test, and izod impact test.
    - Thermal characteristic using Differential scanning calorimetric (DSC) and thermogravimetric analysis (TGA) methods.
    - Morphological characterization using scanning electron microscope (SEM).
    - Rheological characteristic were investigated using Melt flow index (MFI) and capillary rheometer.
  - e) Data analysis by comparing mechanical, thermal, morphological and rheological study.

- iii. The best KBF ratio that gives the best mechanical, thermal, and rheological properties of composites were determined by analyst and verified the best blends compositions.
- iv. Report writing.

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