# MECHANICAL AND THERMAL PROPERTIES OF KENAF FIBER AND MONTMORILLONITE REINFORCED RECYCLED POLYETHYLENE TEREPHTHALATE/RECYCLED POLYPROPYLENE COMPOSITES

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# ABSTRACT

The feasibility of developing kenaf fiber (KF) reinforced recycled polyethylene terephthalate (rPET) and recycled polypropylene (rPP) with comparison to two different reinforcing fillers, KF and montmorillonite (MMT) reinforced rPET/rPP was studied. In addition, the compatibilizer of ethylene vinyl acetate grafted maleic anhydride (EVA-g-MA) at composition 0-10 phr was used. Composites were prepared using twin-screw extruder and followed by injection molding. The optimum blend ratio of rPET/rPP was observed at 90 wt% rPET and 10 wt% rPP. Thermogravimetric analysis data showed that thermal stability of uncompatibilized rPET/rPP blend with ratio of 90/10 had maximum degradation temperature at 399.3 $^{o}$C. Differential scanning calorimetry data revealed that rPET/rPP blend had two melting temperatures. The incorporation of 5 phr EVA-g-MA improved tensile and impact strength of the blends. Besides, the maximum decomposition temperature of rPET/rPP blend also increased. Scanning electron microscopy (SEM) micrographs revealed that by adding EVA-g-MA, uniform particles sizes of rPP was observed, indicating an interaction between both of tertiary carbon of rPP and ester group of rPET with EVA-g-MA. The addition of KF into compatibilized blend decreased mechanical and thermal properties. The maximum value of tensile and impact strength of the blends was obtained at 43.9 MPa and 43.4 J/m respectively, when 1 phr of MMT was added into the rPET/EVA-g-MA/rPP/KF blend. However, SEM micrograph showed that the addition of 4 phr MMT led to filler agglomeration which decreased tensile strength of rPET/EVA-g-MA/rPP/KF/MMT composite.

# ABSTRAK

Komposit daripada gentian kenaf (KF) memperkuat polietilena terephthalate kitar semula (rPET) dan polipropilena kitar semula (rPP) dengan perbandingan dua pengisi penguat berbeza, KF dan montmorillonite (MMT) diperkuat rPET/rPP telah dikaji. Penserasi etilena vinil asetat tercangkuk malik anhidrida (EVA-g-MA) dengan komposisi 0-10 phr telah digunakan. Komposit disediakan menggunakan adunan skru berkembar dua dan diikuti dengan pengacuan suntikan. Nisbah campuran optimum rPET/rPP diperhatikan pada 90% rPET dan 10% rPP. Analisis termogravimetri menunjukkan bahawa kestabilan haba campuran rPET/rPP dengan nisbah 90/10 mempunyai suhu degradasi maksimum pada 399.3 $^{o}$C. Data pengimbasan perbezaan kalorimetri mendapati bahawa campuran rPET/rPP mempunyai dua suhu lebur. Penambahan 5 phr EVA-g-MA meningkatkan kekuatan tegangan dan kekuatan hentaman kesan campuran. Selain itu, suhu penguraian maksimum campuran rPET/rPP juga meningkat. Mikrograf dari pengimbasan elektron mikroskop (SEM) menunjukkan bahawa dengan menambahkan EVA-g-MA, saiz seragam rPP diperhatikan, menunjukkan interaksi antara EVA-g-MA dengan karbon tersiar rPP dan ester rPET. Penambahan KF ke campuran rPET/rPP berpenserasi menurunkan sifat mekanikal dan terma. Nilai maksimum tegangan dan kekuatan hentaman kesan campuran diperoleh pada 43.9 MPa dan 43.4 J/m, apabila 1 phr MMT ditambah ke dalam campuran rPET/EVA-g-MA/rPP/KF. Mikrograf SEM menunjukkan bahawa penambahan 4 phr MMT menyebabkan penggumpalan pengisi yang mengurangkan kekuatan tegangan komposit rPET/EVA-g-MA/rPP/KF/MMT.

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# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| AA | - | Acrylic acid |
| ABS | - | Acrylonitrile butadiene styrene |
| ABS-g-MA | - | Acrylonitrile butadiene styrene grafted maleic anhydride |
| AF | - | Alfa fiber |
| ASTM | - | American standard testing and material |
| BC | - | Bamboo carchoal |
| BHET | - | Bis(hydroxyethyl) terephthalate |
| CNW | - | Cellulose nanowhiskers |
| CRH | - | Chopped rice husk |
| DSC | - | Differential scanning calorimetry |
| EG | - | Ethylene glycol |
| EVA | - | Ethylene vinyl acetate |
| EVA-g-MA | - | Ethylene vinyl acetate grafted maleic anhydride |
| FTIR | - | Fourier transform infrared |
| GMA | - | Glycidyl methacrylate |
| HDPE | - | High density polyethylene |
| IV | - | Instrinsic viscosity |
| KF | - | Kenaf fiber |
| LDPE | - | Low density polyethylene |
| MA | - | Maleic anhydride |
| MFI | - | Melt flow index |
| MH | - | Magnesium hydroxide |
| MMT | - | Montmorillonite |
| M-TDSC | - | Modulated-temperature differential scanning calorimetry |
| OIT | - | Oxidation induction time |

| | | |
|---|---|---|
| OIT | - | Oxidation induction time |
| OMMT | - | Organically modified montmotrillonite |
| PA11 | - | Polyamide11 |
| PALF | - | Pinneaple leaf fiber |
| PBT | - | Polybutylene terephthalate |
| PC | - | Polycarbonate |
| PET | - | Polyethylene terephthalate |
| PLA | - | Polylactic acid |
| POE | - | Polyolefin |
| POE-g-GMA | - | Polyolefin grafted glycidyl methacrylate |
| PP | - | Polypropylene |
| PP-g-AA | - | Polypropylene grafted acrylic acid |
| PP-g-MA | - | Polypropylene grafted maleic anhydride |
| PVC | - | Poly(vinyl chloride) |
| rPET | - | Recycled polyethylene terephthalate |
| rPP | - | Recycled polypropylene |
| SEBS | - | Styrene ethylene butylene styrene |
| SEBS-g-GMA | - | Styrene ethylene butylene styrene grafted glycidyl methacrylate |
| SEBS-g-MA | - | Styrene ethylene butylene styrene grafted maleic anhydride |
| SEM | - | Scanning electron microscopy |
| SSP | - | Solid state polymerization |
| TEM | - | Transmission electron microscopy |
| TGA | - | Thermogravimetric analysis |
| TPA | - | Terephthalic acid |
| vPET | - | Virgin polyethylene terephthalate |
| vPP | - | Virgin polypropylene |
| WAXD | - | Wide angle X-ray diffraction |
| WAXS | - | Wide angle X-ray scattering |

# LIST OF SYMBOLS

| | | |
|---|---|---|
| % | - | Percent |
| $\Delta Cp$ | - | Heat capacity at constant pressure |
| $\Delta H_m$ | - | Enthalpy of melting |
| $\Delta H^o_m$ | - | Enthalpy of melting formation |
| µm | - | Micrometer |
| Å | - | Angstrom |
| $cm^{-1}$ | - | Centimeter power -1 |
| dL/g | - | deciliter per gram |
| *f* | - | Weight fraction |
| $g/cm^3$ | - | Gram per centimeter cube |
| g/min | - | Gram per minute |
| GPa | - | Giga Pascals |
| h | - | Hour |
| J/m | - | Joule per meter |
| kJ/m | - | Kilo Joule per meter |
| mg | - | Miligram |
| min | - | Minute |
| MPa | - | Mega Pascals |
| $N/mm^2$ | - | Newton per milimeter square |
| nm | - | Nanometer |
| $^oC$ | - | Degree Celcius |
| $^oC$/min | - | Degree Celcius per minute |
| phr | - | Part per hundred |
| rpm | - | Revolutions per minute |
| $T_{10}$ | - | Temperature at 10% weight loss |

| | | |
|---|---|---|
| $T_{10}$ | - | Temperature at 10% weight loss |
| $T_c$ | - | Crystallization temperature |
| $T_g$ | - | Glass transition temperature |
| $T_m$ | - | Melting temperature |
| $T_{max}$ | - | Maximum temperature |
| $T_{on}$ | - | Onset temperature |
| Tonnes/ha | - | Tonnes per hectare |
| wt% | - | Percentage weight |
| wt/v | - | Weight per volume |
| %Xc | - | Degree of crystallinity |

# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

The vast increment of packaged water consumption is followed by the rise of environmental concerns. Since the material of packaged water is mainly bottle and made from plastic, so that, it is uneasy to degrade in nature (Awaja and Pavel, 2005; Martin and Brandau, 2012). The most favorable material for packaged water is polyethylene terephthalate (PET) because of its transparent, lightweight, and shatter-resistant, while polypropylene (PP) often makes up the caps due to its excellent moisture barrier, pliability and toughness (Ferrier, 2001; Boonstra and van Hest, 2017). However, most of packaging bottles were used only once. The changing in people way of living to get better and healthier quality of water also increases the demand for packaged water usage. As a consequence, the large amount of post-consumer packaged water containers increase annually, making these materials the main target for recycling. Nevertheless, the recycled materials will be decomposed and degraded in recycling processes which drives to the decrement in intrinsic viscosity. As a result, the chemical, mechanical and thermal properties of materials will also deteriorate (Awaja and Pavel, 2005; Karayannidis and Achilias, 2007; Lou *et al.*, 2007; Inoya *et al.*, 2012).

Reprocessing of recycled PET (rPET) bottle materials and/or recycled PP (rPP) cap bottle will arise another problem since both rPET and rPP has low chemical, mechanical, and thermal properties (Torres *et al.*, 2000; Awaja and Pavel, 2005; Martin and Brandau 2012). Hence, in order to improve rPET and rPP properties after recycling process, blending rPET with rPP has gained more attraction since the ease of fabrication and more cost-effective. Nonetheless, immiscibility in chemical nature and polarity of rPET and rPP make rPET/rPP blend exhibits two-phase morphology with poor interfacial adhesion and thus shows poor properties (Oromiehie and Meldrum, 1999; Lee and Han, 2000; Chiu and Hsiao, 2006; Inoya *et al.*, 2012; Dikobe and Luyt, 2016). Therefore, in order to resolve the major drawbacks of rPET/rPP blend, many approaches have been adopted, such as adding compatibilizer, organic and/or inorganic filler into rPET/rPP blend.

The introduction of compatibilizer into the immiscible blends can improve the compatibility of the blends by increasing adhesion and minimizing interfacial tension which leads to better chemical, mechanical, and thermal properties as compared to uncompatibilized blends (Koning *et al.*, 1998; Chiu and Hsiao, 2006; Inuwa *et al.*, 2017). Several studies have been reported in the literatures on the compatibilizer usage to compatibilized two immiscible polymer blends. Kang *et al.* (1999) reported that the thoughening effect of ethylene vynil acetate (EVA) grafted maleic anhydride (MA) increased the impact strength of polybutylene terephthalate (PBT) and low density polyethylene (LDPE) without significantly decreased tensile and flexural strength. van Bruggen *et al.* (2016) reported styrene ethylene butylene styrene (SEBS) grafted glycidyl methacrylate (GMA), SEBS-g-MA, and polyolefin (POE)-g-GMA increased the viscosity ratio and mechanical properties of PP/PET blends. The high flexibility of EVA-g-MA is expected to enhance impact resistance, provide good dispersion, and improve the compatibility of rPET/rPP blends (Kang *et al.*, 1999; Kim *et al.*, 2003; Wang *et al.*, 2007). However, the presence of a rubber-like polymer often decreased other properties such as tensile, flexural, and heat distortion temperature (Li *et al.* 2002). In order to overcome this problem, the incorporation of natural fiber in the post-consumer plastic blend can improve performance characteristics of the blends.

Natural fiber (NF) reinforced composites have gained a great attention compared to synthetic fiber in relation to their ecological friendly and sustainability. One example of promising natural fiber used as composite filler is kenaf fiber (KF). The chemical structure of KF consists of 45-57% cellulose (Akil *et al.* 2011). KF presents the easiness of processing, low density with high specific strength and modulus, biodegradable, and low cost because it is abundantly available (Akil *et al.*, 2011; Basri *et al.*, 2014). According to Akhtar *et al.* (2014), the addition of 40 wt% KF improved mechanical properties of PP. Mohammad and Arsad (2013) reported that by incorporating 5 wt% of KF into rPET/ABS composite improved Young's modulus, flexural modulus and viscosity of the composites. Nevertheless, since the majority of KF is cellulose, it has high moisture absorption (Carllson, 2005), so that, the hydrophilic properties of KF become the main problem in combining KF with polymer matrix. Some researchers suggest to add two different reinforcements into two incompatible polymer blends, such as NF and clay, to get more remarkable improvement of mechanical, chemical, and thermal properties of the blend (Azmi *et al.*, 2012; Arjmandi *et al.*, 2016; Suharty *et al.*, 2001).

As reported by Azmi *et al.* (2012), mechanical properties of PP/polylactic acid (PLA)/KF had been shown to increase when 1 wt% of montmorillonite (MMT) clay was added. MMT is layered smectite clay mineral which has attained huge consideration as filler in polymer/clay composites due to its large surface area and high aspect ratio (Ray and Okamoto, 2003; Calcagno *et al.*, 2008; Parvinzandeh *et al.*, 2010). Moreover, the addition of MMT in polymer blend improves barrier properties and flame retardation of composites. Besides that, the existence of MMT together with KF will help in holding up the bond even if KF is breakage, so the compatibility between matrix and filler will improve. Some supporting evidences and agreement have been given by some researchers, for example de Lima *et al.* (2015) reported an improvement of PET thermal stability with 1%, 3%, and 5% MMT due to the differences of entropy and entanglement density near the clay surface. Besides, Arjmandi *et al.* (2015) reported the addition of 5 parts per hundred (phr) MMT and 1 phr cellulose nanowhiskers (CNW) resulted the highest tensile strength.

## 1.2 Problem Statement

PET and PP become the most used plastic in daily life. This is due to both PET and PP are used as favorable material for packaged water particularly bottle. The increment amount of rPET (bottle itself) and rPP (cap of bottle) as post-consumer packaged water also cause a problem. The large amount of disposable bottles presently produced which are not biodegradable, makes imperative the search for alternative procedures for recycling or reuse these materials. Previous studies have been carried out to recycle rPET and rPP waste by blending those materials together. Nevertheless, due to the immiscible properties between rPET and rPP, the rPET/rPP blend will form two-phase morphology and result in poor mechanical, chemical, and thermal properties.

The interest in using the rubbery compatibilizer which has toughening effect, such as EVA-g-MA, is expected to not only makes rPET/rPP blend becomes a compatible blend but also enhances impact resistance of the blend without sacrificing other properties. Thus, the addition of KF into compatibilized rPET/rPP blend is expected to improve the materials properties. Nonetheless, KF has high flammability and low thermal stability. The incorporation of MMT together with KF into compatibilized rPET/rPP blends is presumed to improve mechanical properties and thermal behaviour of the blends, and also decreased the flammability of the blends.

To the best of our knowledge, there are many studies about blending PET/PP, natural fiber reinforced composite and polymer/clay nanocomposites with and without compatibilizer. However, the study of either KF or KF/MMT reinforced rPET/rPP blend with addition of EVA-g-MA compatibilizer have not been reported. In this study, the optimum loading ratio of rPET/rPP blend was observed and was used as basis formulation for the next step, the effect of addition of EVA-g-MA compatibilizer on rPET/rPP was also investigated, and the effect of different KF loading and hybrid KF/MMT incorporation into compatibilized rPET/rPP blend on mechanical and thermal properties were also studied.

## 1.3 Objectives of the Study

The aim of this study is to develop new material based on KF and MMT reinforced compatibilized rPET/rPP composites with better mechanical and thermal properties. The main objectives can be further divided into:

1. To determine the effect of different ratio of rPET and rPP on MFI value, mechanical, thermal, and morphological properties of rPET/rPP blend.

2. To investigate the effect of EVA-g-MA compatibilizer incorporation on the compatibility of rPET/rPP blend based on MFI value, mechanical, thermal, and morphological properties.

3. To study the effect of different KF and MMT loading on mechanical, thermal, and morphological properties of compatibilized rPET/rPP blends.

## 1.4 Scope of the Study

In order to achieve the goals, this research involved development of rPET/rPP blends with the incorporation of KF and MMT with and without compatibilizer EVA-g-MA. The following steps were carried out :

1. Sample preparation and melt processing

   In this study, sample preparation and melt processing by using twin screw extruder followed by injection molding were conducted. The content of rPP was varied from 0, 10, 15, 20, and 100 wt% each to find the optimum ratio of

rPET/rPP blend. The incorporation of EVA-g-MA into the optimum ratio was varied from 0, 3, 5, 7, and 10 phr each. KF with ratios of 0, 1, 3, 5, 7, 10, and 15 phr was added into the observed maximum result of compatibilized rPET/rPP blend. Meanwhile, 0, 1, and 4 phr MMT were used together with 1 phr KF as another reinforcement in polymer matrix.

2. Characterization and Analysis

   Melt flow index (MFI) measurement was done based on American standard testing and material (ASTM) D1238 to investigate flow rates of polymer. Fourier transform infrared (FTIR) was carried out to characterize the functional group exist in the blends. Scanning electron microscopy (SEM) was done to examine the surface morphology of the blends. Mechanical properties was observed through tensile (ASTM D638) and impact test (ASTM D256). Thermal properties was analysed by using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC).

## 1.5 Significance of the Study

Polymer blending has been a useful technique to enhance properties or reduced cost. It can directly be made by added at least two polymers together to create a new material with different properties from the individual components. Simply combining rPET and rPP will result in incompatible polymer blend. Adding EVA-g-MA compatibilizer should overcome the compatibility problems and enhanced rPET/rPP blends properties. The incorporation of KF and MMT is expected to enhance mechanical and thermal properties. This research was conducted in order to develop acceptable properties in term of tensile, impact and thermal properties of KF/MMT reinforced rPET/rPP composites from post-consumer bottle with EVA-g-MA compatibilizer. However, the result obtained are found to be way less than that found theoretically, which are discussed in detail in Chapter 4.

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