EFFECT OF NANOCLAY ON MECHANICAL AND THERMAL PROPERTIES OF KENAF RECYCLE POLYETHYLENE WOOD PLASTIC COMPOSITES

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INDOK NURATIKAH BINTI NIZAMUDIN

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DEDICATION

I dedicated this entire work for my beloved family, friends and who always be my side..

For all their love, support, encouragement and inspiration..

Thank you very much..

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ABSTRACT

Recycle wood plastic composites (WrPC) was prepared using kenaf wood flour, recycle polyethylene (rPE) and maleic anhydride polyethylene (MAPE) in the presence of nanoclay filler. Kenaf wood flour, rPE, MAPE and nanoclay were premixed manually and fed into single-screw extruder. Universal Tensile Machine (UTM) and Izod Impact were used to study the mechanical properties. Differential Scanning Calorimeter (DSC) and Thermal Gravimetric Analysis (TGA) were used to study the thermal properties. Scanning Electron Microscope (SEM) was used to observe the morphology. The effect of nanoclay on the mechanical properties, thermal properties and morphology were studied. Tensile strength of WrPC was decreased from 14.67 MPa to 14.07 MPa due to the agglomeration of nanoclay layer. Impact strength was increased from 6.07 kJ/m^2 to 8.22 kJ/m^2 due to the effectiveness distribution of applied stress to overcome the crack. Only one peak of glass transition temperature, T_g appeared at 133.5 °C as a result of miscible blending. Decomposition of WrPC was slightly affected by the addition of nanoclay. The internal structure of WrPC showed that the addition of nanoclay had filled the voids and lead to smooth morphology.

ABSTRAK

Komposit kayu plastik kitar semula (WrPC) telah disediakan dengan menggunakan serbuk kayu kenaf, polietilena kitar semula dan maleic anhidrida polietilena dengan kehadiran pengisi nanoclay. Serbuk kayu kenaf, rPE, MAPE dan nanoclay telah dipracampurkan secara manual dan dimasukkan ke dalam extruder screw tunggal. Mesin Penguji Universal (UTM) dan Impak Izod telah digunakan untuk mengkaji sifat-sifat mekanikal. Kalorimeter Imbasan Perbezaan (DSC) dan Analisis Gravimetrik Terma (TGA) telah digunakan untuk mengkaji sifat haba. Mikroskop Imbasan Elektron (SEM) telah digunakan untuk melihat morfologi. Kesan nanoclay ke atas sifat mekanikal, sifat haba dan morfologi telah dikaji. Kekuatan tegangan WrPC telah menurun daripada 14.67 MPa kepada 14.07 MPa disebabkan oleh penggumpalan lapisan nanoclay. Kekuatan impak telah meningkat daripada 6.07 kJ/m^2 to 8.22 kJ/m^2 disebabkan oleh keberkesanan pengagihan tekanan untuk mengatasi retak. Hanya satu puncak suhu peralihan kaca, T_g yang ada pada 133.5 °C akibat terlarut campur. Penguraian WrPC telah terjejas sedikit dengan penambahan nanoclay. Struktur dalaman WrPC menunjukkan bahawa penambahan nanoclay telah memenuhi lompong dan membawa kepada morfologi yang licin.

TABLE OF CONTENT

CHAPTER 3 METHODOLOGY

CHAPTER 4 RESULTS AND DISCUSSIONS

CHPATER 5 CONCLUSIONS AND RECOMMENDATIONS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

~	Approximately
T_c	Crystallization temperature
°C	Degree Celsius
°C/min	Degree Celsius per minute
ε_t	Elongation at break
F	Force
T_g	Glass transition temperature
kg	Kilogram
kJ/m^2	Kilojoule per meter square
kV	Kilovolt
MPa	Mega Pascal
m^2/g	Meter square per gram
µm	Micrometer
mm	Millimeter
mL/min	Milliliter per minute
nm	Nanometer
phr	Part per hundred
%	Percent
psi	Pounds per square inch
rpm	Revolutions per minute
σ_t	Tensile strength
E_t	Tensile modulus
wt %	Weight percent

LIST OF ABBREVIATIONS

DMTA	Dynamic Mechanical Thermal Analysis
DSC	Differential Scanning calorimeter
HDPE	High-density polyethylene
LDPE	Low-density polyethylene
MAPE	Maleic anhydride polyethylene
MAPP	Maleic anhydride polypropylene
MSW	Municipal solid waste
PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl chloride
rHDPE	Recycle high-density polyethylene
rPE	Recycle polyethylene
rPS	Recycle polystyrene
SEM	Scanning Electron Microscope
TGA	Thermal Gravimetric Analysis
UTM	Universal Testing Machine
WPC	Wood plastic composites
WrPC	Wood recycle plastic composites

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

The study of wood plastic composite (WPC) become one of essential products in the industry nowadays. WPC can simply defined as a mix of material consists of natural wood and thermoplastic. Furthermore, WPC is eco-friendly product, which the durability achieved without using toxic chemicals (Alireza, 2008). Wood recycle plastic composite (WrPC) is a product that use recycle plastic in order to minimize environmental impact and consumption of virgin plastic. Most of industry claimed that the process of making WrPC is elementary and the products are commercialize (Alireza, 2008).

To cut the cost and municipal solid waste (MSW), plastics material have been used in the production of wood composite by blending it together with wood powder. For instance, recycled materials such as recycled high-density polyethylene become a value added in the manufacturing of WrPC without slacking off the properties. Wood powder is renewable resources and abundance in nature. Thus, it possesses high potential that can be used in the making of WrPC as it can show better performance of mechanical strength, wettability and good thermal insulation (Saba *et al.*, 2015).

Besides, WPC can be used in wide range of applications such as decking, building and structural components. Generally, wood powder comprise between 50 to 70 percent of the production wood plastic composite. The most common type of wood that been used is kenaf. This kind of wood is easily obtain and low in cost. Many research proved that kenaf possessed excellent mechanical strength and thermal properties compare to other type of natural fiber (Saba *et al.,* 2015).

In the awareness of green technology, recycle plastic such as polyethylene is use to produce WrPC. A study had shown that the properties of recycle polyethylene (rPE) not largely different from virgin plastic and suitable for many applications. Moreover, nanoclay has its own special properties and perfect atom arrangement that suitable as reinforced polymer-based composites (Kin *et al.,* 2006). According to Kin et al. (2006), it is one of the good mineral filler to overcome the drawback or failure of WPC.

The process of making WrPC been done by blending fine wood particles with recycle polyethylene in order to make firm, smooth sample for convenient handling and further processing. The addition of nanoclay can enhance the mechanical properties of material. Moreover, to improve the compatibility and bonding between wood flour and recycle polyethylene, maleic anhydride polyethylene (MAPE) was used as coupling agent.

1.2 Problem Statement

Recently, the market of wood plastic composites had grown higher and it becomes trending, as the product is environmentally safer with better performance in any applications. Virgin thermoplastic become one of the materials used in WPC. Recycle plastic that can melt and process below the degradation temperature of wood or any lignocellulos fillers suitable in production of WPC (Nourbakhsh *et al.,* 2009). In the meantime, most industry had changed the manufacturing of wood plastic composite (WPC) to wood recycle plastic composite (WrPC) because the used of virgin plastic was exceeded and the cost was high. Accordingly, recycle plastic polyethylene was applied in this study to prepare wood recycle plastic composite in the basis of kenaf to minimize the cost.

Besides reducing the cost, recycle plastic polyethylene (rPE) offers an excellent dimensional stability as compared to virgin plastic polyethylene (Hua *et al.,* 2012). The mechanical properties such as tensile and flexural strength of recycle polyethylene wood plastic composite are equivalent to virgin polyethylene composite. Moreover, recycle plastics are cheaper and can reduce the environmental impact. However, the study of WPC based on recycle polyethylene is very limited. According to Yam et al. (1990), the performance of recycle high-density polyethylene (rHDPE) and wood fiber composites is good as virgin plastic composites. The advantage of natural fiber such as kenaf is it shows superior flexural strength and modulus of elasticity but have some drawback such as fiber degradation can occur at high temperature (Yam *et al.*, 1990).

Mineral filler like nanoclay employed in this research was to address the issue of WrPC such as poor impact strength, tensile strength and flexural strength. This filler met the expectation of improving mechanical properties of resulting product. In this study, a WrPC was prepared using kenaf wood flour and recycle polyethylene. This study is to improve mechanical properties of WrPC for structural and non-structural applications.

1.3 Objectives of Research

This work aimed to produce kenaf wood flour, recycle polyethylene, maleic anhydride polyethylene and nanoclay with outstanding mechanical and thermal properties. Thus, several objectives listed as below:

1. To prepare wood recycle plastic composite using kenaf powder, recycle polyethylene, maleic anhydride polyethylene and nanoclay filler.
2. To characterize mechanical properties, thermal properties and morphology of the resulting WrPC.
3. To compare the mechanical properties of WrPC without the addition of nanoclay and WrPC in the presence of nanoclay filler.

1.4 Scope of the Study

To achieve the goals of this work, the scopes listed as below:

1. Manually premix kenaf wood flour, recycle polyethylene, maleic anhydride polyethylene and nanoclay filler at room temperature then the mixture extruded into single screw extruder at processing temperature.

2. Characterization of the WrPC using :
    a) Universal Testing Machine (UTM)
    b) Izod Impact
    c) Thermal Gravimetric Analysis (TGA)
    d) Differential Scanning Calorimeter (DSC)
    e) Scanning Electron Microscope (SEM)

CHAPTER 2

LITERATURE REVIEW

2.1 Wood Plastic Composites

Wood plastic composite (WPC) can be referred to any composite that consists of plant fiber thermoset or thermoplastic. According to Sumit et al. (2015), thermosets are plastics that cannot melted after it cured whereas thermoplastics are plastics that can melted repeatedly. Therefore, the property of plastics allowed other material such as wood fibers to be mixed together in order to produce wood plastic composite products. Nowadays, wood plastic composite market has increased as it available in various application such as doorframes, furniture, docks and wall studs. Thus, an environmentally friendly fiber source for plastic is highly needed due to high cost of woods and competition of woods resources from traditional wood sector (Han *et al.,* 2008). The basic understanding of polymer and wood is essential in the study of wood polymer composite. Polymer has high molecular weight that consists of molecules multiple of simple unit and it can be natural or synthetic. The examples of natural polymer are cellulose, collagen and keratin while synthetic polymer are polypropylene and polyethylene. Moreover, a polymer called as plastic when it has other materials such as stabilizer, plasticizers or other additives in it and the molecular structure is developed through the process of polymerization (Kristiina *et al.,* 2008). Thermoset and thermoplastic are polymers that crosslink extensively and been used with wood as well as other natural fiber. Furthermore, wood is stiffer and stronger than synthetic polymer which make it suitable for filling or reinforcing. Composite made from both properties of polymer and wood.

2.2 Wood Recycle Plastic Composites

Most of polymer materials are available in the form of plastic. According to Scott (1999), the products present excellent properties of impermeability to water and organism, high mechanical strength, low density and low cost. Nevertheless, it will accumulate in the environment if it is not recycled and can cause pollution. Plastic can be divide into virgin plastic and recycle plastic where virgin plastic has higher quality compare to recycle plastic. Most of the industry had changed the production of wood plastic composite (WPC) to wood recycle plastic composite (WrPC). This was due to the increasing cost of virgin plastic and its availability was limited. Utilization of recycle plastic in WrPC will reduce the amount of waste disposal and can improve the recycle plastic market (Yihua *et al.*, 2008).

Moreover, it claimed that the costs of recycle plastics in manufacturing of WrPC are cheaper compare to the virgin wood fibers. The combinations of recycle plastic and wood fiber have become major attention because it offers good mechanical properties. According to Sellers et al. (2000) reported that the products of recycled polyethylene (rPE) or polystyrene (rPS) with pine wood fibers at ratio of 50:50 through high-pressure pressing molding had showed good mechanical properties that suitable for construction materials. A research found that the mechanical properties of WrPC having recycled plastic of HDPE and PP showed a similar behavior to the composite that made from virgin plastic (Yihua *et al.*, 2008). Thus, WrPC provide better solution in environmental impact; give high profit to the market as well as beneficial to consumer.

2.3 Materials

Wood recycle plastic composite (WrPC) is an environmentally way of combining recycle plastic such as recycle polyethylene and polypropylene and wood material where the properties can be improve with addition of mineral filler (Behzad *et al.*, 2011). The composite typically consists of four major elements that are wood flour, thermoplastic plastic, coupling agent and lubricant (Shao *et al.*, 2012). Otherwise, wood plastic composites also defined as composite materials containing wood in the form of fiber and thermoplastic. Composite is the mixture of more than one elements and the properties can be different. Thus, in manufacturing of wood plastic composite, wood powder, recycle plastic, mineral filler and coupling agent will be mixed together in which the ratio can be 50:50 of wood powder and recycle plastic or 40:60 of wood powder and recycle plastic. The physical and mechanical properties of WrPC are highly depend on the material formulation. The behavior of each parameters must be understand so that better performance and high quality products can be produce.

2.3.1 Wood Flour

In wood plastic composite, wood flour is referring to wood reduced into finely particles, which easy to put into plastic processing instrument as compare to fiber gained from wood. Wood can be divided into softwood (gymnosperms) and hardwood (angiosperms) that been classified by botanical and anatomical features rather than its hardness. The example of softwood species are pines, firs, cedars and spruces whereas hardwood species are oak, maples and ashes. Wood is use as reinforcement in polymer composite where the method results in the form of fiber or particulate. Thus, wood fibers is referred to the spindle-shape cells of wood and it is the main structural element of wood on the macroscopic scale which offer good reinforcing potential because of their high strength allowed efficient transfer of stress to the fiber (Kristiina *et al.,* 2008). Therefore, natural fibers as kenaf fiber is reinforce with polymer matrix to form fiber reinforced polymer composites that comparable to existing materials. In the past, kenaf was used as a cordage crop to make twine, sackcloth and rope but nowadays there are various new applications that have been work out including building materials and also paper products by using kenaf. The word kenaf came from Persian origin which refer to the plant having short day, warm season and herbaceous plant with average diameter of fiber 67.6 µm (Mahjoub *et al.*, 2014). Kenaf fiber was classified in the species of *Hibiscus cannabinus* where genus is *Hibiscus* and family *Malvaceae* acquired from stems of plants that also comprises cotton *(Gossypium spp.)* and okra *(Abelmoschus esculentus L. Moench)* (Saba *et al.*, 2015). Physically, kenaf plant has a single, straight and brancless stalk. It made up of an inner woody core and an outer fibrous bark surrounding the core. Furthermore, the common length of filaments is about 1 mm, which consists of discrete individual fibres (2-6 mm) long. They are composites of largely cellulose, lignin and hemicelluloses. Fiber properties can be depend on several factors such as age, source, separating technique and even the history of the fiber itself. Kenaf been chosen in this study because it is fiber crop grown that easily to get. It was reported that kenaf grow faster where the growing season was about 4-5 month with 4-5 m of height and 25-35 mm of diameter. Three types of fiber consists in kenaf plant are bast, core and pith. Kenaf bast and core hold an excellent mechanical property that can replace glass fiber in polymer composites as reinforcing elements. Figure 2.1 shows the kenaf core in the form of flour that been used in this research.

Figure 2.1. Kenaf core.

2.3.2 Recycle Plastic

Thermoplastic is one of the composition present in the WPC products and it was reported that 60 million pounds of thermoplastics have been used in manufacturing of WPC in which the volume of polyethylene is about 90%, 10% polypropylene while PVC and resin is about 35-40% whereas virgin plastic is 60-65% (Anatole, 2007). The classification of plastics into virgin and recycle type gives a lot of opportunity for a new development of product due to several factors such as cost and environmental impact. Even though virgin plastic has high quality but the price is also expensive. Thus, with growing used of plastic, most of industries provide an alternative in producing recycle plastic that is cheaper and eco-friendly. Thus, thermoplastic such as polyethylene is highly suitable for mechanical recycle as the properties well preserved throughout the cycles of processing and ageing. Polyethylene is one of the plastic that been produced highly with low melting temperature between 130 °C to 160 °C that allowed the use of cellulose fiber as filler with low risk of significant thermal degradation. It can be produce in broad range of viscosity and mix well with fillers. Otherwise, polyethylene is soft which making PE-based composite deck boards easier to nail, screw, cut and saw (Anatole, 2007). The other properties of PE is it has near-zero moisture absorption and very high resistance to chemicals such as strong acids. Polyethylene is special as it available in many form including low-density polyethylene (LDPE), high-density polyethylene (HDPE) and high-molecular weight HDPE (HMW-HDPE). Therefore, by filling polyethylene with wood fiber increases flexural strength to about 1600-2200 psi of resulting composites, as for HDPE based composite materials, the flexural strength up to 3000 psi and by adding coupling agent can cause the strength to increase about 3800 psi and higher (Anatole, 2007). Figure 2.2 shows recycle polyethylene that preferred in the blend of composite.

Figure 2.2. Recycle polyethylene.

2.3.3 Coupling Agent

In every composites must have different of coupling agents that fit to the type of composite. Coupling agents is also known as compatibilizers which improving the blend homogeneity of variant or incompatible materials (Kristiina *et al.,* 2008). In addition, by adding coupling agent into the composite can reduce water absorption as well as minimizing swelling of fibers. Compatibilizing is a surface-active phenomenon where it creates compatibility between the surfaces of materials. In wood plastic composite, wood surfaces are electrostatic whereas the polyolefin resins are non-polar. Therefore, coupling agents will ensure the different parts of the molecule have different dipole moments and become compatible (Kristiina *et al.,* 2008). Maleated polyolefin is manufacture by grafting maleic anhydride onto a backbone of polyethylene or polypropylene that is use as coupling agent in WPC. The function of maleic is it provides compatibility with the wood fiber surfaces whereas the polymer backbone is compatible with the matrix resin and the product can be differentiate based on the degree of maleation and molecular weight of the polymer (Kristiina *et al.,* 2008). The most common copolymers containing maleic anhydride are maleate polypropylene (MAPP) and maleate polyethylene (MAPE). Both been used widely in the WPC in which the anhydride groups of copolymer react with the surface hydroxyl groups of woods polymer and forming ester bonds while the other end entangles with the polymer matrix because of the similar polarity (Han *et al.,* 2008). An expectation was made where MAPE provide an efficient compatiblizer for both PE/clay and PE/wood systems in fabrication of composites. Figure 2.3 shows the coupling agent of PE-MAH.

Figure 2.3. Maleic anhydride polyethylene.

2.3.4 Mineral Filler

In contrast, recycled plastic only itself cannot produce best quality of wood plastic composite. Thus, mineral filler such as nanoclay can enhance the mechanical and physical properties of WPC. Nanocomposites like nanoclay have become a major growth segment for the plastic industry and one of it is using layered silicate nanoclays as in situ reinforcement (Behzad *et al.,* 2011). The properties of this material such as high surface area, large aspect ratio and high cationic exchange capacity will provide good quality products. Recently, it been verified that there were improvements of physical and mechanical properties of tensile modulus and strength, flexural modulus and strength thermal stability, fire resistance as well as barrier resistance for various thermoplastic and thermoset nanocomposites at low silicate content (Behzad *et al.,* 2011). Nanoclay is categorize in mineral group in which it is naturally occurring layered materials with special interest of its ability to cation exchange in the interlayer space that showing unique mechanical properties. Furthermore, nanoclay possessed of aluminum silicate layers that organized in parallel to form stacks with a regular Van der Waals gap in between them called internal spacing or gallery (Meysam *et al.,* 2013). Otherwise, by having large surface area (~750 m^2/g) and higher aspect ratio between 100-1000, nanoclay filler is suitable to overcome the drawback of WPC and it is reported that with the aid of exfoliating and completely dispersing clay platelets have improved the mechanical properties, thermal and dimensional stability of PE based WPC (Gong *et al.,* 2013). Figure 2.4 shows nanoclay that added in the composition of material.

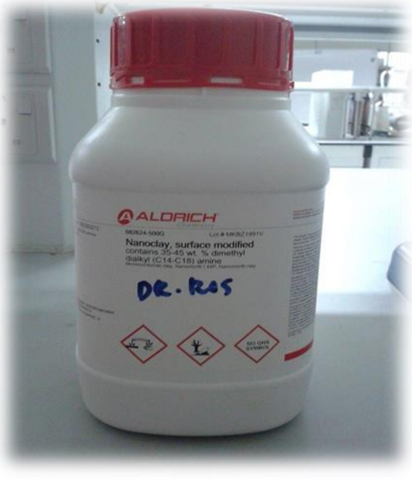

Figure 2.4. Nanoclay.

2.4 Preparations of Wood Plastic Composites

According to Sumit et al. (2015), WPC was prepared using wood powder as fillers and polypropylene granules resin as matrix in the presence of coupling agent and nanoclay as reinforcing fillers. The wood core dried in the oven at 105 °C and sieved down to particle size of 40-60 mesh. The plastic granules and different contents of nanoclay were performed in a single-screw extruder with temperature was set at 180 °C in fed zone, 210 °C for melting while 200 °C for extruder die. Before fed into the first zone of extruder, the polypropylene and nanoclay must be premixed first. The coupling agent used was about 2% and amount of nanoclay was different from 0% to 5%. For overall view, the wood cores, maleic anhydride polypropylene (MAPP) together with polypropylene/nanoclay granules were performed in a single-screw extruder. Mechanical and physical properties such as flexural strength, impact strength, as well as tensile strength of the wood plastic composite were observed. Besides, WrPC can be prepared with different ratio of wood powder and recycle plastic so that it can easily been extruded. It reported that by increasing wood powder loading, the tensile strength could be increase. The presence of compatibilizing agent by mixing it together with wood powder and recycle plastic can enhance the interfacial bonding strength of the material.

In other research, the composite prepared by mixing recycle HDPE with sawdust particles and maleic anhydride at high speed mixer for about 5 minutes. After that, the mixture was molded in a compression molding machine and heated with different temperature of each sample formulation such as 130 °C, 150 °C or 170 °C. Then, with pressure of 2500 psi, the sample transferred to hot-pressed for 50 minutes. Several tested were conducted including mechanical test, dynamic mechanical thermal analysis (DMTA), thermal gravimetric analysis (TGA) and scanning electron microscopy (SEM). Therefore, the result showed that by increasing the temperature, the flexural and compressive strength increased. However, with higher amount of oil palm sawdust caused poor interfacial adhesion and depression between sawdust and polymer matrix (Thanate *et al.*, 2012)

2.5 Applications

Wood plastic composites considered in indoor and outdoor applications including decking, wood flooring, fencing and many more. In the industry of music, drumstick is use to play the drum. The production of the material is simple and no specification needed. The polymer technology is widely applied and the drumstick had to feel like wood while the sound produces just like wood. Typically, drumstick made from wood such as oak and male but in contrast, polypropylene drumsticks can have same characteristic as wood drumstick (Kristiina, 2008). Thus, wood fiber was added to the polypropylene which the characteristic very much like wood drumstick. This is one of an oriented wood fiber-plastic composites material.

Furthermore, different shapes of wood fiber/plastic composite and different concentrations of wood fibers are possible to produce hardwood flooring (Kristiina, 2008). The quality of this product is depend on its mechanical and physical properties. Therefore, several test need to be conducted in order to compare the properties and to provide high quality products. Wood plastic composite decking boards is usually low in cost but the quality is high. The composition of this product consists of hard wood fibers, recycled plastic like polyethylene, bonding agent, tint and additives. This product is eco-friendly, weather resistance and long lasting which make it special and very convenient.

For instance, park furniture was one of the product that be seen at parks or gardens where the material is completely made by wood plastic composites. The quality of wood and the ease of workability of plastic had improved the properties of material. Some advantages of this product are resistance to moisture, non-toxic and guarantee to not crack or rust. In producing more product for its purpose, wood plastic composite material can minimize the further cutting of trees and plastic used as they can be replaced by waste wood and recycle plastic.

CHAPTER 3

METHODOLOGY

3.1 Materials

Five kilogram of kenaf core fiber in the form of powder and 5 kg of recycle polyethylene pellets were supplied by Innovative Pultrusion Sdn Bhd. 1 kg of maleic anhydride polyethylene (MAPE) and nanoclay surface modified contains 35-45 wt% of dimethyl dialkyl (C14-C18) amine with size ~200nm were obtained from Sigma Aldrich. Table 3.1 shows the list of materials that used and its functions.

Table 3.1
List of materials and functions

Materials	**Function**	**Specific Name/ Chemical Formulation**
Kenaf flour	Filler	Hibiscus cannabinus
Recycle Polyethylene	Matrix	$(C_2H_4)_n$
Maleic anhydride polyethylene (MAPE)	Coupling agent	$(C_4H_2O_3.C_2H_4)_n$
Nanoclay	Reinforcing filler	Montmorillonite (MMT)

3.2 Composites Preparation

In this research, kenaf core fiber and recycle polyethylene had used to prepare WrPC. All components weighed according to the formulation stated in the Table 3.2. Then, manually premixed kenaf core fiber, recycle polyethylene (rPE), maleic anhydride polyethylene (MAPE) and nanoclay in plastic container. The mass ratio of kenaf and recycle polyethylene parted to 40:60 as shown in the Table 3.2. Next, the mixture was compounded through single-screw extruder (LBE20-30/C, Labtech Engineering) that divided into three section called solids conveying, melt and melt pumping. The temperature for each section was set differently such as solids conveying (210 °C), melt (220 °C) and melt pumping (210 °C) with screw rotation speed at 60 rpm. Figure 3.2(a) shows the single-screw extruder used in the process of composites. The mixture weighed and formed into a mat on aluminum caul plate using a 380mm×380mm forming frame. Teflon sheet was used in order to avoid direct contact of the plastic with the metal plates of the hot-press. After that, WrPC mats was subjected to the hot pressing at 195 °C with pressure of 750 psi for 20 minutes to prepare sample for mechanical testing. The sample then placed in desiccator for at least 24 hour prior to testing in order to keep it dry and maintain the low humidity. Two types of WrPC had produced where one of the WrPC made with the addition of nanoclay whereas the other made without the addition of nanoclay to compare their mechanical properties. Figure 3.2(b) and (c) shows two type of composites that produced throughout this research.

Table 3.2
Chemical composition of materials

Components	**With mineral filler**	**Without mineral filler**
Kenaf (wt %)	38.8	38.8
Recycle Polyethylene (wt %)	58.2	58.2
Maleic anhydride polyethylene (wt %)	3	3
Nanoclay (phr)	3	-

Figure 3.1. The (a) single-screw extruder, (b) kenaf/rPE/MAPE (c) kenaf/rPE/MAPE/nanoclay.

3.3 Material Synthesis Methods

In order to demonstrate the overview of the overall process in this research, a flow chart in Figure 3.2 be drawn as below.

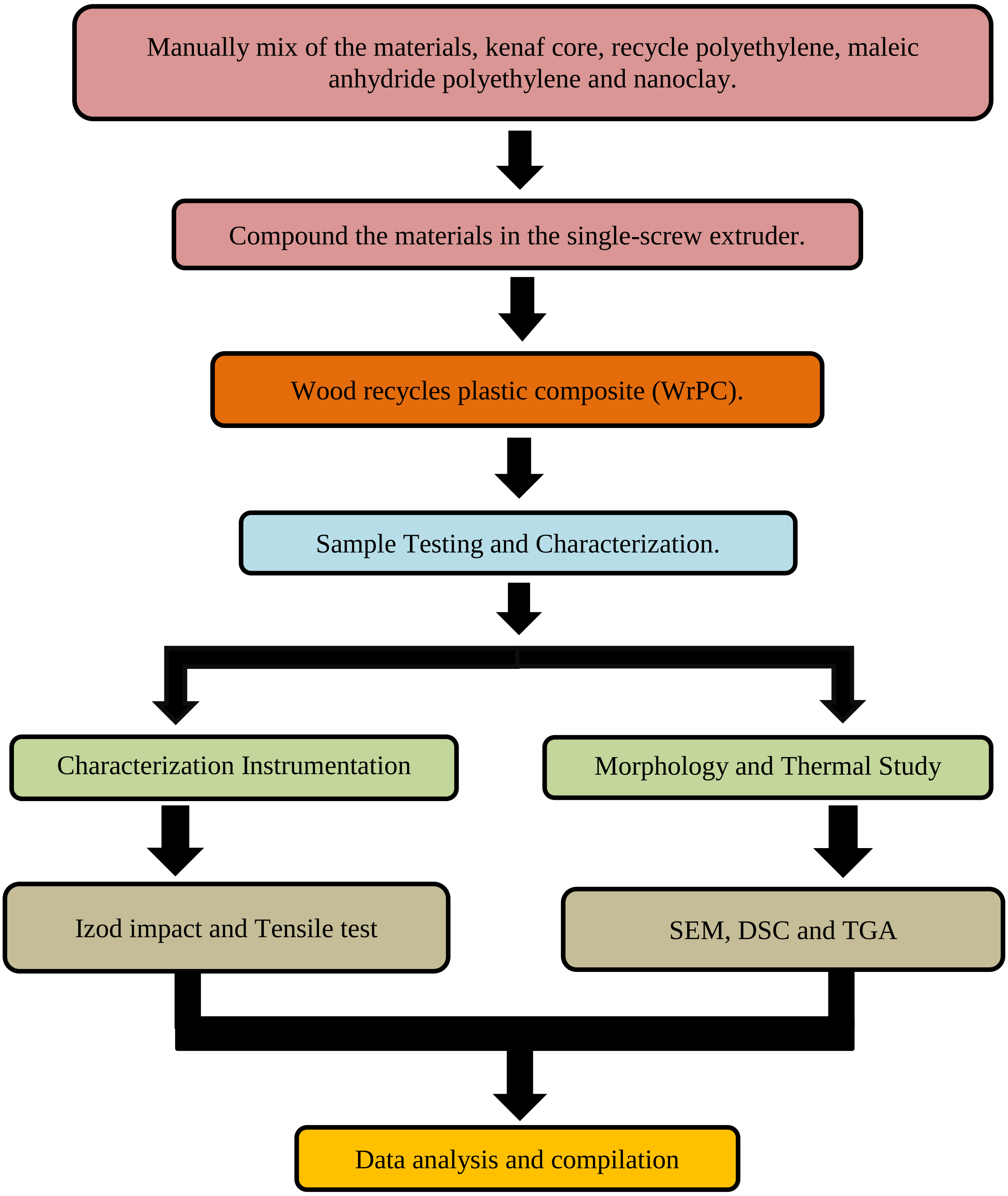

Figure 3.2. Steps for making wood recycle plastic composites.

3.4 Material Characterizations

In principle, characterization of wood recycle plastic composite prepared in extruder achieved by using approaches and techniques similar to those available for WrPC in general. The mechanical, thermal and morphology properties of the sample were studied by means of various methodology or techniques such as Tensile Test, Izod Impact Test, Differential Scanning Calorimeter (DSC), Thermal Gravimetric Analysis (TGA), and Scanning Electron Microscope (SEM). Therefore, in this research, WrPC characterization considered in relation to the components used in the production of wood recycle plastic composite. For each characterization of mechanical testing, five samples been tested while powdered form was used for thermal and morphology testing.

3.4.1 Universal Testing Machine (UTM)

Universal Tensile Machine (UTM) been used to determine tensile strength, modulus strength and elongation at break. Thus, UTM assessments been carried out according to ASTM D3039 with rectangular cross-section (25mm×250mm). The test speed of 2 mm/min was implemented for reinforced composite testing. The sample been positioned vertically in the grips of the testing machine and the grip need to be tightened evenly and firmly to avoid any slippage. The specimens elongated as the tensile test start and the load value (F) was recorded until a rupture of the specimen occur. The tensile properties such as tensile strength, tensile modulus and elongation at break were calculated by Bluehill 2 software provided with the instrument. The testing was repeated for five times in order to get the mean value. Figure 3.3 shows the dimension needed for tensile testing.

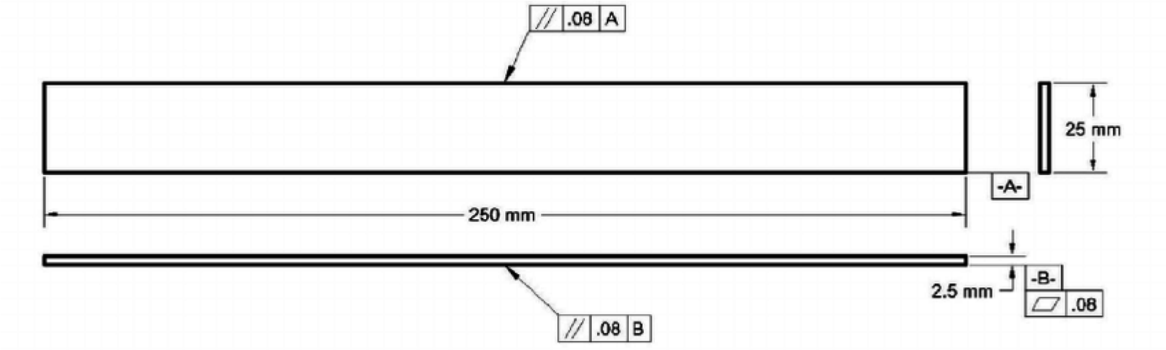

Figure 3.3. Dimension for tensile testing.

3.4.2 Izod Impact

In this study, AE-1CITFC, Advance Equipment been used to measure the impact strength of notched impact specimens under ambient conditions, in the compliance with ASTM D256 as shown in figure 3.5. The sample dimension measured at 13 mm × 60 mm. A notching machine been used to create a precision notch in specimens with notch depth of 2.84 mm. Then, a standardized pendulum type hammer with an antifriction bearing breaks the specimen and the impact energy absorbed by the sample was calculated. The testing been repeated for five times in order to get the mean value.

3.4.3 Thermal Gravimetric Analysis (TGA)

Thermal gravimetric analysis (TGA) been used to determine the thermal degradation of the organic component in the blend materials and composite. TGA assessment was carried out according to ASTM E 1131 using TGA/DSCTHT/1600, Mettler Toledo. The wood recycle plastic composite was scanned from 24-1000 ^{o}C, at a heating rate 10 ^{o}C/min. The composite was placed in an open platinum pan where nitrogen was used together as the purge gas at a flow rate of 20 mL/min. Onset temperature of a 10% and 75% weight loss deviation from the baseline of the thermal gravimetric (TG) curve was applied as indicator for resulted products thermal stability.

3.4.4 Differential Scanning Calorimeter (DSC)

Differential Scanning Calorimeter (DSC) was used to determine glass transition temperature (T_g) of the synthesized product. DSC assessment was carried out according to ASTM D 3418 using Netzsh, Polyma214. This testing was conducted at a heating rate 10°C/min in order to ensure high resolution of the DSC curve and in the temperature range of 30 to 500 °C. Meanwhile the liquid nitrogen rate was maintained at 20 ml/min and the calorimeter was calibrated with an indium reference. The DSC curve was plotted as heat flow versus temperature and the glass transition temperature of WrPC was determined using the tangent method. The mid-point of the first endothermic baseline shift in the DSC heating curve was taken as the T_g.

3.4.5 Scanning Electron Microscope

The morphology of the fracture surfaces after tensile testing was observed using Carl Zeiss Scanning electron microscopy (SEM) which obtains micro structural images using a scanning electron beam. This technique revealed the fiber orientation in fiber reinforced polyethylene/nanoclay toughened composites together with the information of the bond between the fibers and matrix. The samples was cut into small pieces and placed onto a SEM stage. An electron beam with accelerating voltage of 10kV was used on each sample. Magnification of 500x and 1000x was set to observe the micrographs of the fracture. Therefore, the micrographs produced refer as surface topography of the samples.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Mechanical Properties

For this part, two different test of samples were performed including tensile testing and Izod impact. The objective of this research is to compare the mechanical properties of WrPC without the addition of nanoclay and with the presence of nanoclay and articulate it based on the results obtained from the testing.

4.1.1 Tensile Properties

The tensile test was measured in order to show the ability of material to withstand forces that tend to pull apart and determine to what extent the material stretches before breaking. Figure 4.1 shows that the tensile strength of WrPC with the addition of nanoclay slightly decreased from 14.67 MPa to 14.07 MPa. On the testing of mechanical strength, the result of tensile test by incorporating nanoclay should improve the mechanical properties of sample. According to Wang et al. (2006), a higher content of nanoclay (>3 phr) caused the decrease in tensile strength due to the agglomeration of nanoclay layer. Furthermore, the reduction also due to lower degree of exfoliation and a lower of polymer nanoclay surface interactions. By referring Figure 4.2, the performance of tensile modulus increased from 1470.84 MPa to 2001.11 MPa with the presence of nanoclay in WrPC. Usually, clay has structure that is more rigid and the modulus of a composite material depends on the ratio of filler modulus to the matrix (Mohd Rosnan & Arsad, 2013). Thus, the stress transferred from the polymer matrix to the nanoclay filler caused the tensile modulus to increase which make the composites become stiffness.

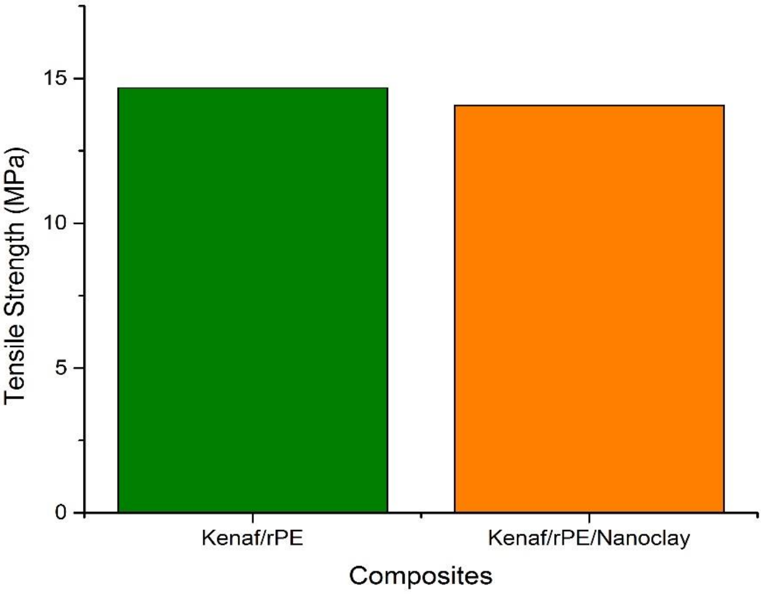

Figure 4.1. The effect of WrPC with and without nanoclay on tensile strength.

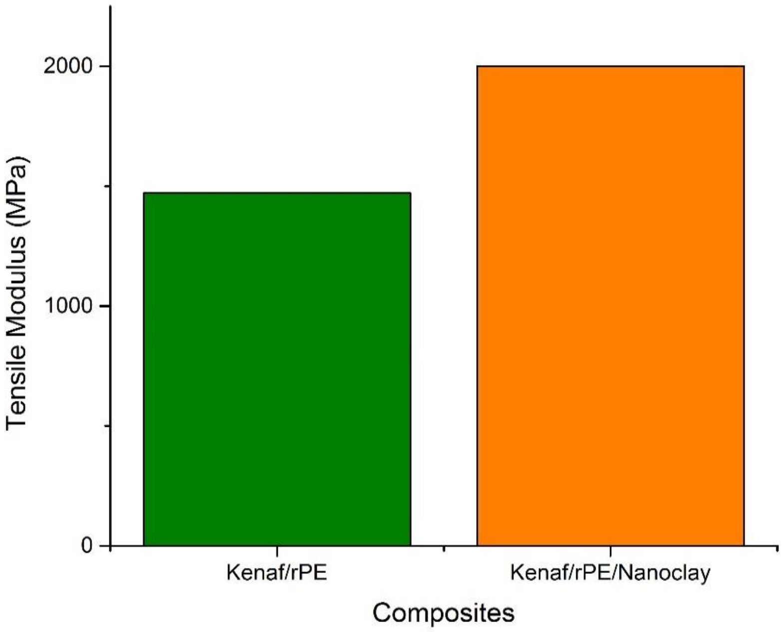

Figure 4.2. The effect of WrPC with and without nanoclay on tensile modulus.

Figure 4.3 represents the elongation at break of WrPC. From the figure, the elongation at break of the WrPC reduced about 0.011% by addition of nanoclay and increased to 0.013% without the presence of nanoclay. The properties can slightly increase when nanoclay was loaded due to its good dispersion and can provide better adhesion in the system. Unfortunately, it was not applicable for this study. Mineral filler can be assume as structural elements that embedded in the polymer matrix (Onuegbu *et al.*, 2011). Thus, unsuitable concentration of mineral filler contents do not allowed it to restrain the polyethylene molecules. Utilization of 3 phr nanoclay might create localized strain which decreased the ductility of matrix.

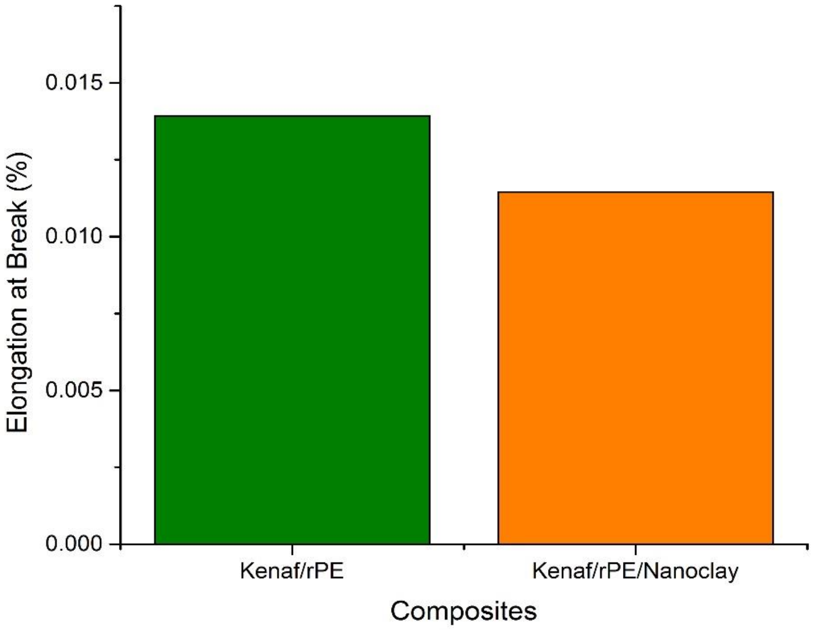

Figure 4.3. The effect of WrPC with and without nanoclay on elongation at break.

4.1.2 Izod Impact

A material should has an ability to withstand a sudden impact so that it applicable for any practical application. Figure 4.4 shows that the impact strength of kenaf recycle polyethylene with nanoclay was increased. The result showed the highest value of impact strength was 8.22 kJ/m^2. The increasing in impact strength of prepared composites indicates that exfoliation of nanoclay has improved the performance of fiber/nanoclay-matrix interfacial. In addition, the existing of nanoclay caused the distribution of applied stress over a large volume at the base of the notch become more effective. Thus, it helps to overcome the propagation of crack and absorb the additional energy that prevents brittle fracture to occur (Onuegbu *et al.*, 2011). Nevertheless, it found that the impact strength decreased to 6.07 kJ/m^2 for WrPC without nanoclay. The interaction of fiber and matrix become poor without the addition of nanoclay. The result does not consistent with the tensile strength as it found that the concentration of nanoclay might cause a major influence in the testing of mechanical properties. Table 4.1 shows the summary of mechanical properties of WrPC.

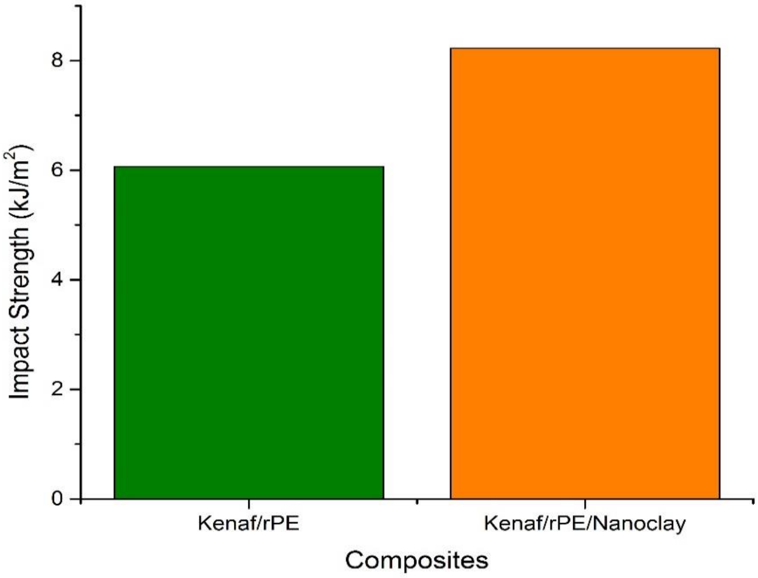

Figure 4.4. The effect of WrPC with and without nanoclay and on impact strength.

Table 4.1

Summary of the mechanical properties of kenaf recycle polyethylene with and without nanoclay

Samples	**Tensile Strength, σ_t (MPa)**	**Tensile Modulus, E_t (MPa)**	**Elongation at Break, ε_t (%)**	**Impact Strength (kJ/m^2)**
Kenaf/rPE	14.66	1470.84	0.0139	6.07
Kenaf/rPE/nanoclay	14.07	2001.11	0.0115	8.22

4.2 Thermal Analysis

For this part, two different test of samples were performed including Thermal Gravimetric Analysis (TGA) and Differential Scanning Calorimeter (DSC).

4.1.2 Thermal Gravimetric Analysis (TGA)

The thermal degradation of material can be determined by thermo gravimetric analysis. The crucial part of this analysis was the parameter measured in which weight of the sample is lost at first point, the mid-point of the degradation and the last measurement of degradation known as char. Figure 4.5 shows the TG curves for kenaf/rPE without the addition of nanoclay and kenaf/rPE with the addition of nanoclay. From the analysis, the initial decomposition temperature, $T_{10\%}$ for WrPC with nanoclay decreased slightly from 289.50 °C to 279.34 °C. This is because under nitrogen flow, the catalysis effect towards the degradation of the polymer matrix was not observed (Olewnik *et al.,* 2010). Furthermore, the decomposition temperature, $T_{50\%}$ also decreased for WrPC with nanoclay from 427.16 °C to 419.16 °C. This is because the amount of exfoliated nanoclay is not enough to enhance the thermal stability at this point. The temperature at 600 °C indicates the formation of residue. From the graph, it shows that the residue formed for WrPC with nanoclay is higher that is 0.95% while WrPC without nanoclay only 0.71%. Therefore, the result showed lower thermal stability as the thermal degradation occurred faster for WrPC with nanoclay. According to Olewnik et al. (2010), organoclay can be a barrier effect in improving the thermal stability and can be a catalyst that leading to the decrease of thermal stability. Table 4.2 represents the TG data of WrPC prepared.

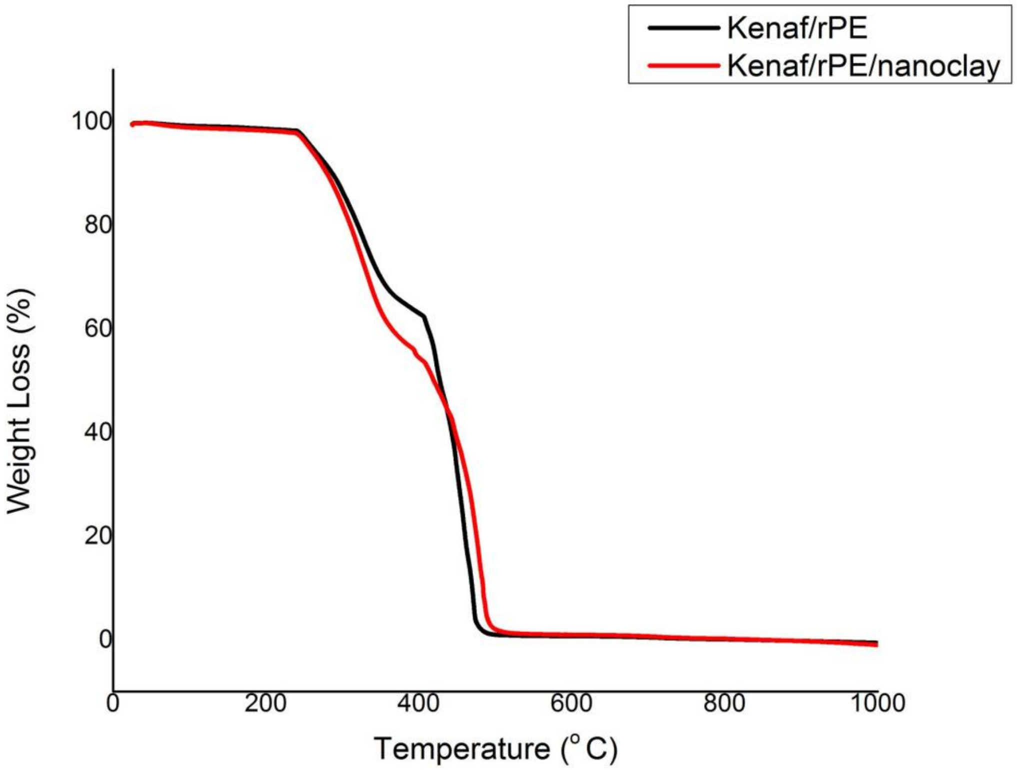

Figure 4.5. TG analysis of WrPC with and without nanoclay.

Table 4.2
TG data for kenaf/rPE and kenaf/rPE/nanoclay

Samples	**Temperature at weight loss (°C)**		**Residue at 600 °C (%)**
	$T_{10\%}$	**$T_{50\%}$**	
Kenaf/rPE	289.50	427.16	0.71
Kenaf/rPE/nanoclay	279.34	419.16	0.95

4.2.2 Differential Scanning Calorimeter (DSC)

Most of the time, DSC analysis is one of the convenient method for analysing melting and crystallization temperature of samples. Figure 4.6 represents DSC analysis of WrPC. From the figure, it shows that the WrPC with addition of nanoclay caused a slightly different in the temperature of glass transition, T_g for about 133.5 °C compare to the WrPC without nanoclay that is 133.8 °C. The decreased in T_g might due to the morphology of nanocomposite which it is partially exfoliated and caused the blockage of crystalline growth in the composites (Olewnik *et al.*, 2010). Furthermore, there was no melting temperature peak because of miscible blending. The crystallization temperature also shows slightly difference between samples in the presence of nanoclay that is 270 °C while without nanoclay is 280 °C. According to Olewnik et al. (2010), clay platelets can be as nucleating centers and favour crystallization by providing level of nucleation density Thus, this applied to the temperature of crystallization, as it was believe that organoclay consist of nucleating effect that affect the degree of crystallinity (Olewnik *et al.*, 2010). Table 4.3 shows the DSC data that were studied including glass transition temperature, T_g and crystallization temperature, T_c.

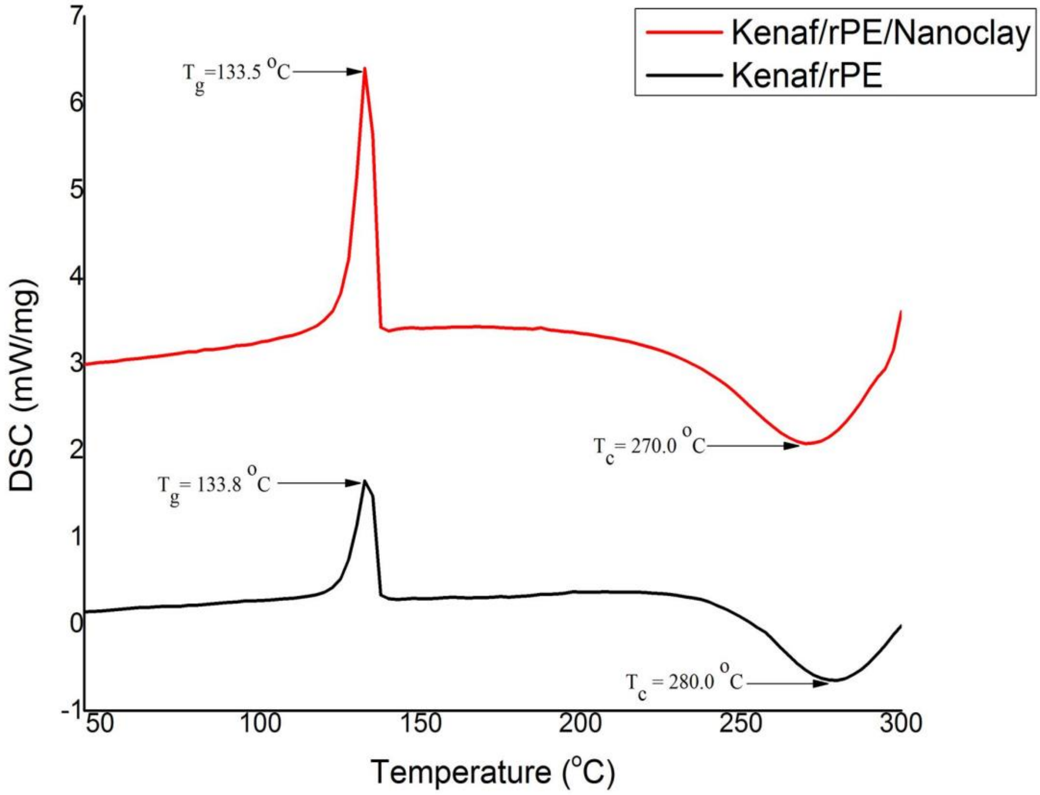

Figure 4.6. DSC analysis of WrPC with and without the addition of nanoclay.

Table 4.3
DSC data for kenaf/rPE and kenaf/rPE/nanoclay

Samples	T_g/°C	T_c/°C
Kenaf/Rpe	133.8	280.0
Kenaf/rPE/nanoclay	133.5	270.0

4.3 Morphological Analysis

To investigate the homogeneity, the part of samples after tensile test was observed through SEM. The micrographs of tensile fracture were obtained at the magnification of 1000× as shown in the figure 4.7. The dispersion condition of nanoclay inside the composite was compared.

Figure 4.7(a) shows the morphology of sample without nanoclay. It clearly seen that there were void formed in the boundary. Thus, the combination of kenaf/rPE with 40:60 ratio caused a weak interaction and weak mechanical adherence between them. As discussed before, the addition of nanoclay depending to its concentration can leads to reduction in tensile strength. However, nanoclay can act as a compatibilizer in order for it to attach or penetrate into the blends and eventually enhance the properties of material (Mohd Rosnan *et al.*, 2013). Figure 4.7(b) represent SEM micrographs of composite in the addition of nanoclay. It was observed that the tiny white spots of nanoclay have filled the voids between the matrix and fiber. The surface of fracture becomes smoother which indicate that there was an enhancement of interfacial adhesion. Besides, the uniformly dispersed of nanoclay was due to the size of nanoclay (~200nm). Likewise, it been discussed in the literature.

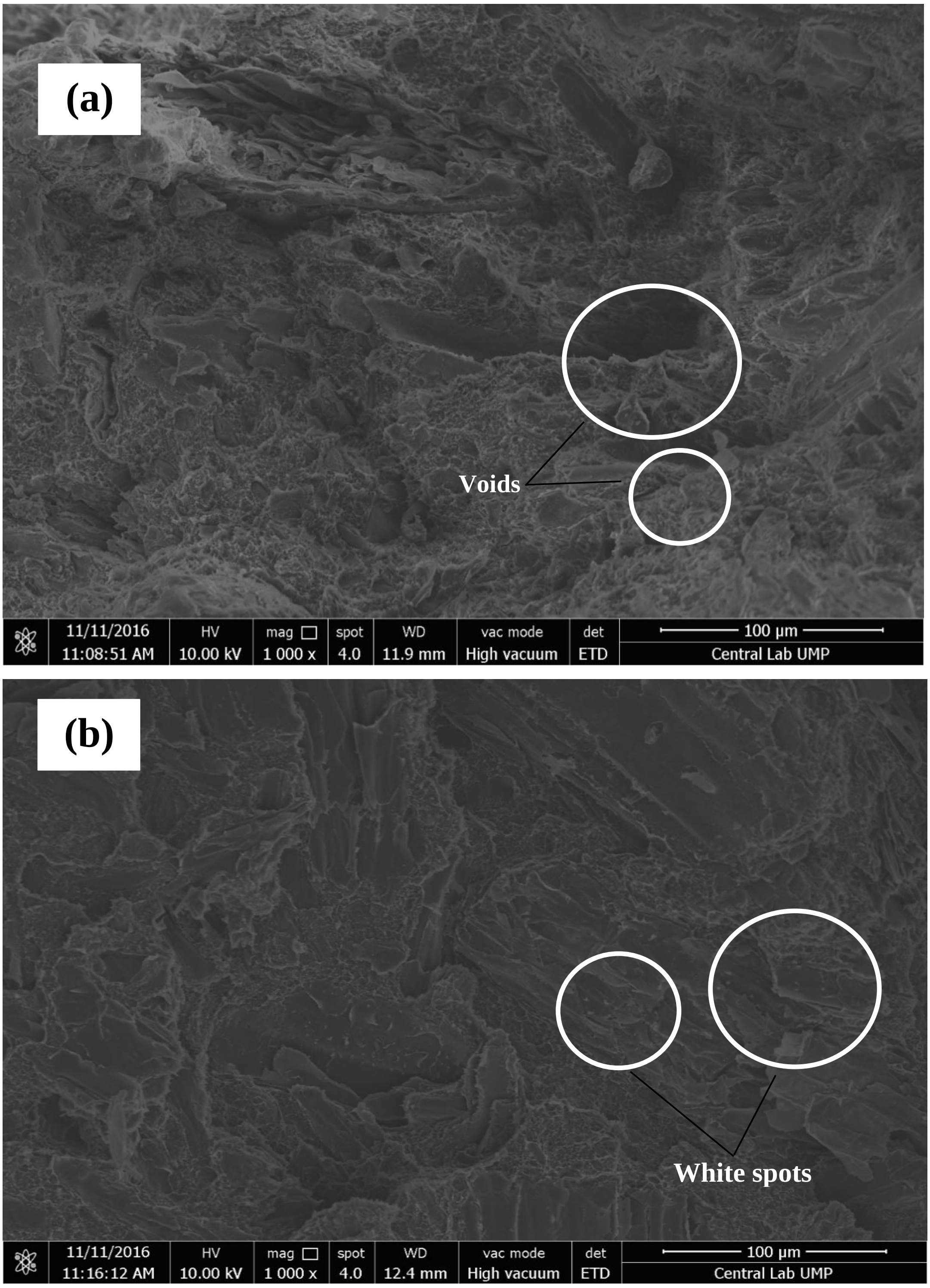

Figure 4.7. The (a) SEM micrograph of kenaf/rPE, 1000× magnification, (b) SEM micrograph of kenaf/rPE/nanoclay, 1000× magnification.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

The main objectives of this research is to compare the mechanical and thermal properties of kenaf recycle polyethylene wood plastic composite in the addition of nanoclay and without the addition of nanoclay. This chapter concluded the overall results and gives recommendations on possible further work in this area of impact modifier toughened polymer composites.

5.1 Conclusion

The following conclusions were made according to the mechanical, thermal and morphology results and discussions for kenaf recycle polyethylene wood plastic composites.

i. The incorporation of nanoclay in the kenaf recycle polyethylene wood plastic composite reduced the tensile strength and elongation at break due to the concentration of nanoclay that been added in the blend.

ii. However, the toughness of the composites increased with the existing of nanoclay. The increased on impact strength shows that the composite can withstand high impact and provide better quality of product.

iii. TGA analysis showed that in the existing of nanoclay has lower the thermal stability of the nanocomposite.

iv. DSC test showed that the addition of nanoclay caused the performance of glass transition temperature, melting temperature and crystallization temperature decreased.

v. The morphology analysis showed that the void that presence in the structure can recovered by the white spots particle of nanoclay that make the sample become less brittle due the fractured.

5.2 Recommendations

There are some aspects in this research that can be considered in future. From the finding, a few recommendations for future study as shown below:

i. Study the mechanical properties which varying contents of mineral fillers.
ii. Extruder must be connected with suitable die to ensure the sample obtain is easy to use for hot-pressed.
iii. Make sure during extruded, the sample is loading continuously so that well blend composites can be obtained.
iv. Ensure that the temperature of extruder and hot-pressed is a bit higher than the melting point of the composition so that the composites form is nice and in a good appearance.

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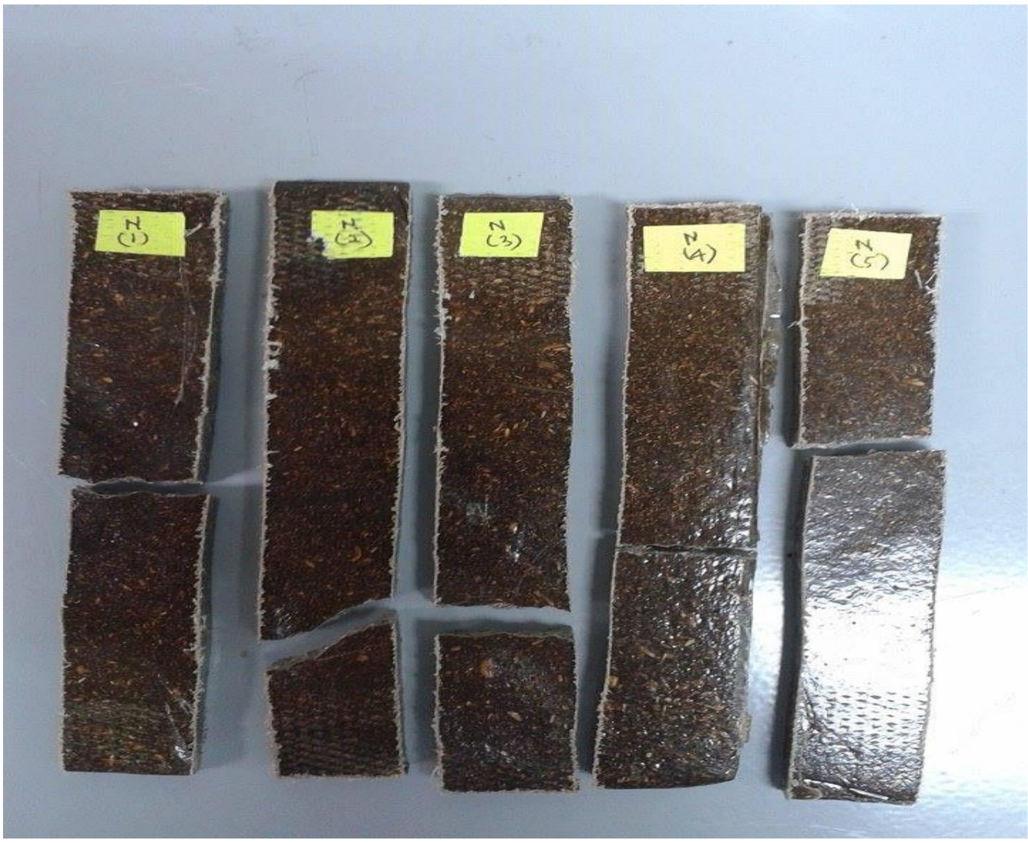
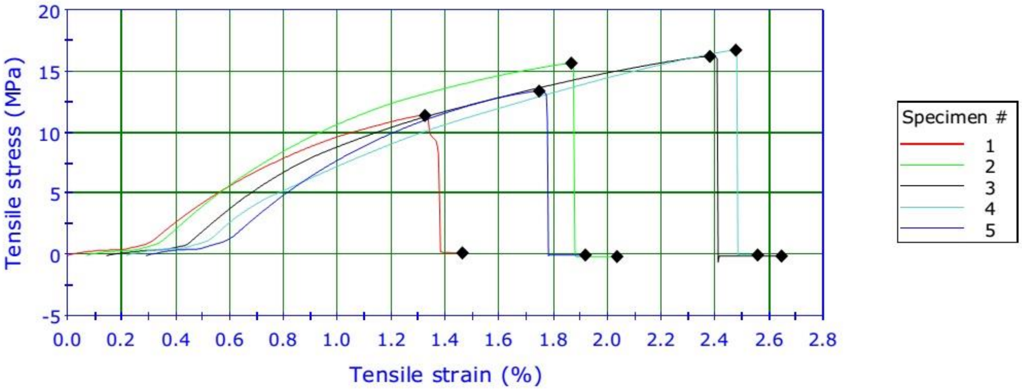
APPENDIX A

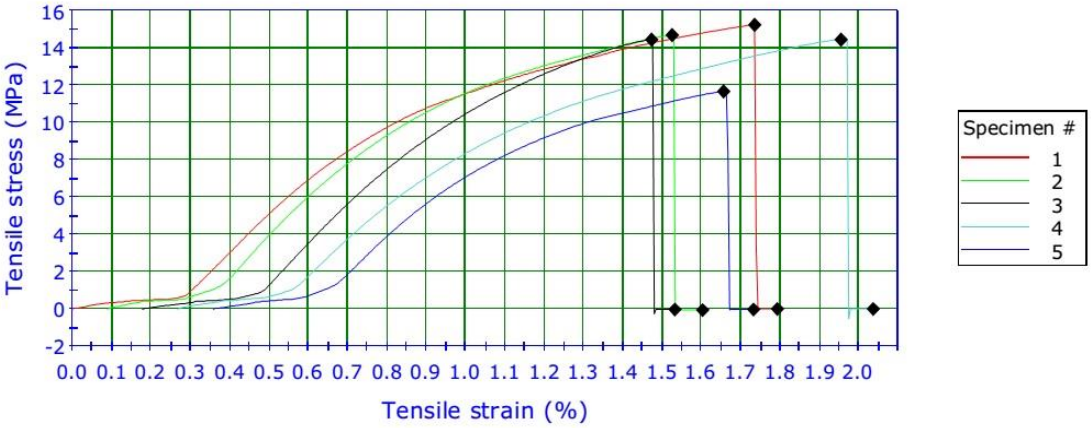
SAMPLE OF TESILE TESTING



Appendix A1



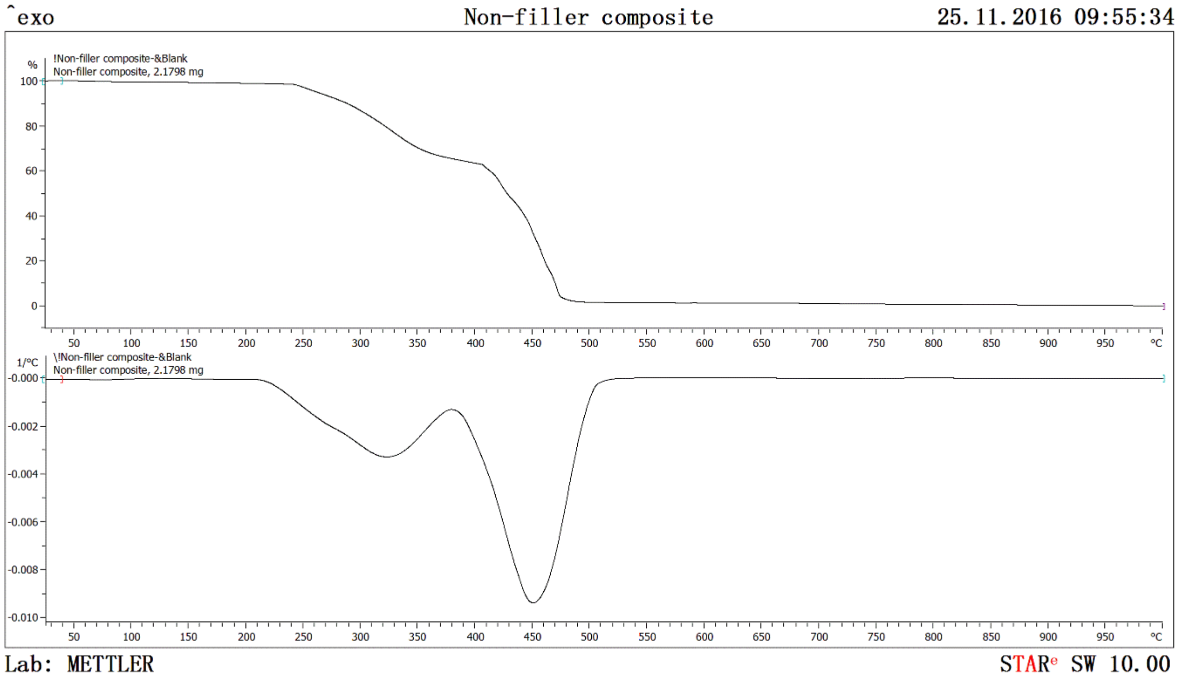
Appendix A2


Tensile stress (MPa)
16
14
12
10
8
6
4
2
0
-2
0.0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 2.0
Tensile strain (%)
Specimen #
1
2
3
4
5

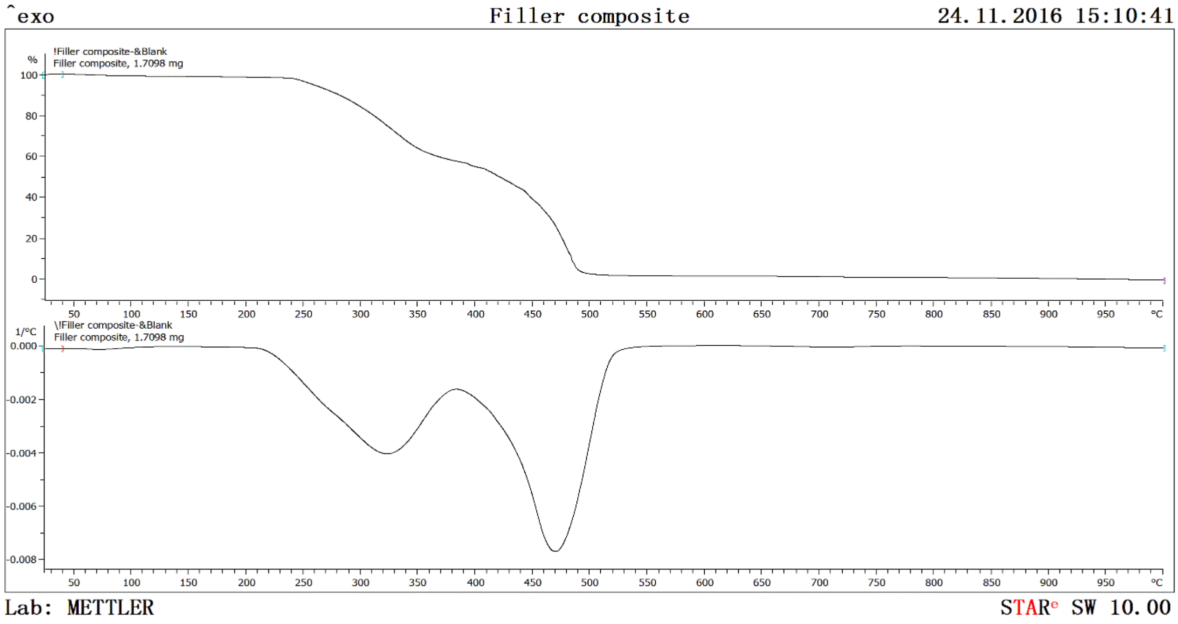
Appendix A3

APPENDIX B

SAMPLE OF TGA



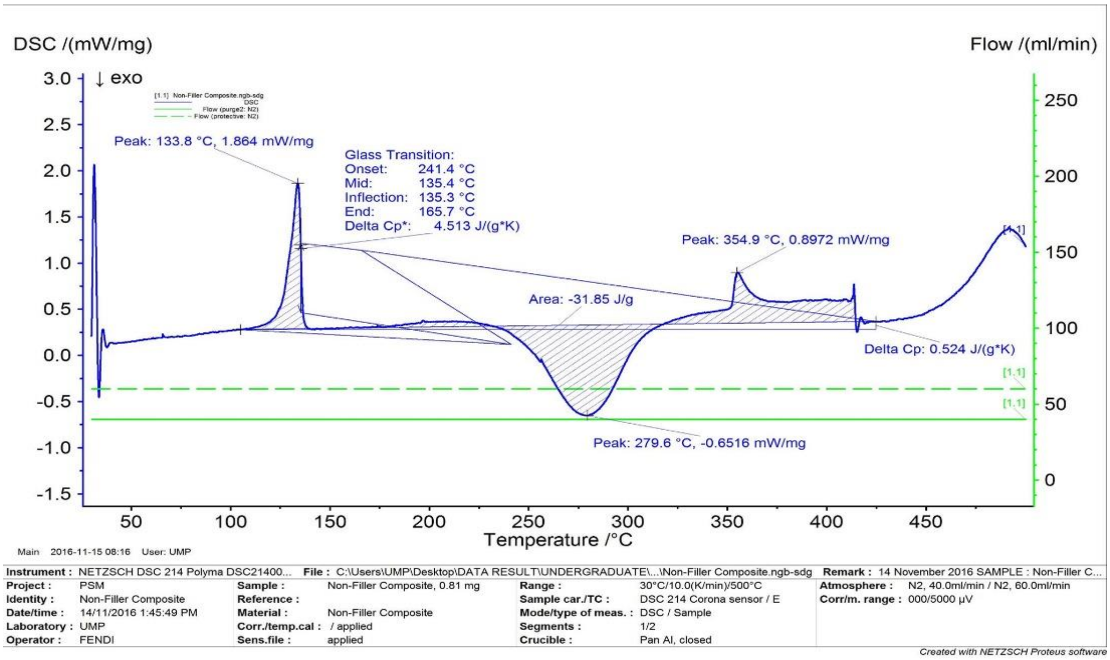
Appendix B1



Appendix B2

APPENDIX C

SAMPLE OF DSC

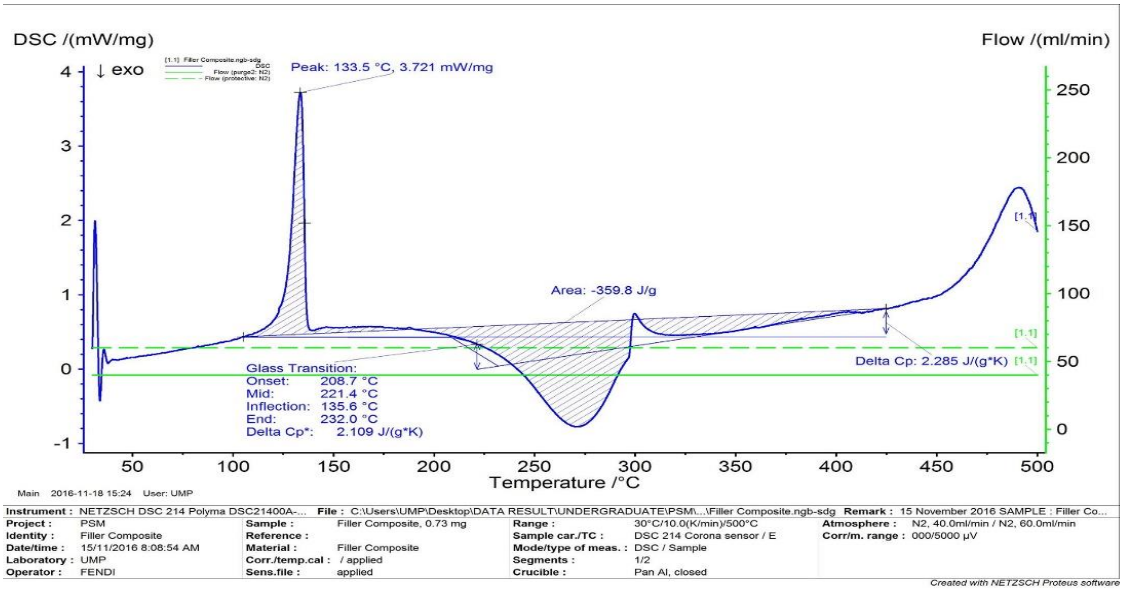


Instrument : NETZSCH DSC 214 Polyma DSC21400... **File :** C:\Users\UMP\Desktop\DATA RESULT\UNDERGRADUATE\...\Non-Filler Composite.ngb-sdg **Remark :** 14 November 2016 SAMPLE : Non-Filler C...

Project :	PSM	**Sample :**	Non-Filler Composite, 0.81 mg	**Range :**	30°C/10.0(K/min)/500°C	**Atmosphere :**	N2, 40.0ml/min / N2, 60.0ml/min
Identity :	Non-Filler Composite	**Reference :**		**Sample car./TC :**	DSC 214 Corona sensor / E	**Corr/m. range :**	000/5000 µV
Date/time :	14/11/2016 1:45:49 PM	**Material :**	Non-Filler Composite	**Mode/type of meas. :**	DSC / Sample		
Laboratory :	UMP	**Corr./temp.cal :**	/ applied	**Segments :**	1/2		
Operator :	FENDI	**Sens.file :**	applied	**Crucible :**	Pan Al, closed		

Created with NETZSCH Proteus software

Appendix C1



Instrument : NETZSCH DSC 214 Polyma DSC21400A-... **File :** C:\Users\UMP\Desktop\DATA RESULT\UNDERGRADUATE\PSM\...\Filler Composite.ngb-sdg **Remark :** 15 November 2016 SAMPLE : Filler Co...

Project :	PSM	**Sample :**	Filler Composite, 0.73 mg	**Range :**	30°C/10.0(K/min)/500°C	**Atmosphere :**	N2, 40.0ml/min / N2, 60.0ml/min
Identity :	Filler Composite	**Reference :**		**Sample car./TC :**	DSC 214 Corona sensor / E	**Corr/m. range :**	000/5000 µV
Date/time :	15/11/2016 8:08:54 AM	**Material :**	Filler Composite	**Mode/type of meas. :**	DSC / Sample		
Laboratory :	UMP	**Corr./temp.cal :**	/ applied	**Segments :**	1/2		
Operator :	FENDI	**Sens.file :**	applied	**Crucible :**	Pan Al, closed		

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Appendix C2