EFFECT OF CALCIUM CARBONATE ON MECHANICAL AND THERMAL PROPERTIES OF KENAF RECYCLE POLYPROPYLENE/ POLYETHYLENE WOOD PLASTIC COMPOSITES

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EFFECT OF CALCIUM CARBONATE ON MECHANICAL AND THERMAL PROPERTIES OF KENAF RECYCLE POLYPROPYLENE/ POLYETHYLENE WOOD PLASTIC PLASTIC COMPOSITE

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I dedicate this thesis to my family for nursing me with affection and love. My supervisor, Dr. Ros Azlinawati for her generous guidance, knowledge and time. Lastly, for all my friends that always understand and encourage me throughout many crisis.

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ABSTRACT

Wood recycle plastic composite (WrPC) was prepared using kenaf wood flour, recycle polypropylene (rPP), recycle polyethylene (rPE) and maleic anhydried polypropylene (MAPP) in the presence of calcium carbonate (CaCO₃) filler. Kenaf wood flour, rPP, rPE, MAPP 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 thermalgravimertric analysis (TGA) were used to study the thermal properties. Scanning electron microscope (SEM) was used to investigate the morphology. The effect of CaCO₃ on mechanical properties, thermal properties and morphology were studied. Tensile strength of WrPC was increase from 7.72 MPa to 8.26 MPa due to transfer of stress from polymer matrix to CaCO₃ particles. Impact strength was decrease from 7.14 KJ/m² to 6.14 KJ/m² due to the improved stiffness of the composites which was attributed to interaction between the polymer and CaCO₃ fillers. Prepared WrPC exhibit two Tg as a result from immiscible blend of Kenaf, rPP, rPE and CaCO₃. T_{g2} of WrPC with CaCO₃ loading slightly increased from 162 °C to 163 °C due to strong interfacial interaction between CaCO3 and kenaf/rPP/rPE, which reduced the free volume and restricted the motion of rPP and rPE chains. Besides, decomposition of WrPC was affected by the addition of CaCO₃. CaCO₃ loading prohibits complete combustion of the sample since the degradation temperature of CaCO₃ is high. The internal structures of WrPC have better dispersion and fewer voids.

ABSTRAK

Komposit kayu dan plastik yang dikitar semula (WrPC) telah disediakan dengan menggunakan serbuk kayu kenaf, polipropilena yang dikitar semula (rPP), polietilena yang dikitar semula (rPE) dan polipropelina maleik anhidrida (MAPP) dalam kehadiran kalsium karbonat (CaCO3) pengisi. Serbuk kayu kenaf, rPP, rPE, dan MAPP dicampur secara manual dan dimasukkan ke dalam pengekstrud satu skru. Mesin Universal Tegangan (UTM) dan penguji hentaman izod telah digunakan untuk mengkaji sifat-sifat mekanikal. Kalorimeter pengimbasan pembezaan (DSC) dan analisis thermalgravimertric (TGA) telah digunakan untuk mengkaji sifat haba. Mikroskop imbasan elektron (SEM) telah digunakan untuk menyiasat morfologi. Kesan CaCO₃ pada sifat mekanik, sifat haba dan morfologi telah dikaji. kekuatan tegangan WrPC meningkat daripada 7.72 MPa kepada 8.26 MPa disebabkan oleh pemindahan tekanan daripada polimer matriks kepada zarah CaCO₃. Kekuatan hentaman turun daripada 7.14 KJ/m² kepada 6.14 KJ/m² kerana peningkatan kekerasan disebabkan oleh interaksi antara polimer dan pengisi CaCO₃. WrPC yang disediakan mempunyai dua suhu peralihan kaca, Tg kerana campuran kenaf, rPP, rPE dan CaCO₃ tidak sepenuhnya bercampur. Tg2 WrPC dengan tambahan CaCO3 meningkat sedikit daripada 162 °C kepada 163°C kerana interaksi kuat antara CaCO3 dan kenaf/rPP/rPE yang menyebabkan kekosongan dalam komposit berkurang dan menghadkan pergerakan rantaian rPP dan rPE. Selain itu, penambahan CaCO₃ turut memberi impak terhadap penguraian WrPC. CaCO₃ telah mencegah pembakaran lengkap sampel kerana suhu degradasi CaCO₃ adalah tinggi. Struktur dalaman WrPC dengan kehadiran CaCO₃ mempunyai penyebaran yang lebih baik dan sedikit lompang.

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LIST OF SYMBOLS

%	Percentage
Wt.%	Weight percentage
μ	Micron (10^{-6})
°C	Degree Celsius
σ_t	Tensile strength
Et	Tensile modulus
Et(%)	Elongation at break
H _m	Heat of fusion
T _c	Crystallization temperature
Tg	Glass transition temperature
T _m	Melting temperature

LIST OF ABBREVIATIONS

DSC	Differential Scanning Calorimeter
HDPE	High density polyethylene
LDPE	Low density polyethylene
MAPP	Maleic anhydride polypropylene
MA	Maleic anhydride
PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl chloride
PCC	Precipitate calcium carbonate
rPE	Recycle polyethylene
rPP	Recycle polypropylene
SEBS-g-MA	Maleated styrene- ethylene/butylenes-styrene
SEM	Scanning Electron Microscope
SMA	Styrene maleic anhydried
TGA	Thermogravimetric analysis
UTM	Universal Testing Machine
WPC	Wood Plastic Composite
WrPC	Wood recycle Plastic Composite

CHAPTER 1

INTRODUCTION

1.1 Background of The Study

Wood plastic composite (WPC) can be defined as composite material lumber made up from wood and plastic. The most distinct properties of WPC are their ability to resist against degradation in outdoor environment which make WPC as a suitable material for outdoor application than untreated lumber product. Examples products from WPC material are deck, kitchen cabinets, indoor furniture, benches, railings and fences. Previously, only virgin thermoplastic polymers such as polyethylene (PE), polypropylene (PP) and poly vinyl chloride (PVC) are used in WPC manufacturing. The composition between wood and plastic in WPC affect the properties of finished product. For instance, the resultant product of WPC that contain too low or too high ratio of plastic to wood might produce product with undesirable visual appearance and characteristics. As the result, the cost to manufacture WPC is quite high due to expensive virgin plastic materials used. Hence, recycled plastic has started to be used in the production of WPC instead of virgin plastic since 1990 and the usage has increase significantly in recent years. Principia Partners (Ashori, 2008) state that the largest supplier of wood-plastic composite lumber, Trex has purchases an average of over 227,000 kg of plastic scrap each day and Power (as cited in Winandy et al., 2004) found CorrectDeck have uses about half virgin plastic and the other half (20 percent) is obtained from recycled grocery bags and used pallet wrap. Moreover, Winandy et al. (2004) also have listed few products of recycle WPC produced commercially in the USA. The main components in WPC are plastic and wood. There are many types of wood that can be used to manufacture WPC such as Kenaf and Radiata pinus. Kenaf is an annual herbaceous plant that can grow in wide range of weather condition. Kenaf usually will grow from 1.5 to 3.5 m with a woody base. Recently, Kenaf have attracted a lot of attention due to their high rate of production. They are able to grow in shorter time which is 4 to 6 months when compare to trees that need 10 to 25 years before being used for industry purpose. Kenaf have several components such as stalks, leaves and seeds. The stalk is made up of woody core and fibrous bark. The fibrous of kenaf has excellent flexural strength and superior tensile strength. WPC is produced by mixing wood particle, molten plastic and other additives together. The mixture is then processed by compression, injection molding or extrusion to form composite material.

Worldwide plastic production has continued to rise since the first mass production of plastic in 1950s. Stastically, in 2014 worldwide plastic production was reported approximately 311 million metric tons, which emphasize increment of plastic production around 4 percent over 2013. This phenomena show more resources are being used in order to meet the increased demand of plastic and more plastic waste is being produced. Besides, the property of plastic itself as not biodegradable material worsen the situation which result plastics waste becoming a major stream in solid waste. Hence, it would be rewarding if plastic waste can be reused in everyday application in order to improve material properties such as the impact strength and the hardness, besides able to use into new products (Jayaraman and Halliwell, 2009).

Previous researcher has realized the important to recycle these nonbiodegradable wastes and have taken initiative to study the properties of recycle plastic and compare it to virgin plastic. The uses of recycle plastic in application have generated controversial debate. Research by Pattanakul et al. (1991) and Achilias et al. (2007) have support that properties of recycle plastic not too different with virgin. Pattanakul et al. (1991) which investigate the properties of recycled high density polyethylene (HDPE) from milk bottles have reported the properties recycle HDPE recover from milk bottles was not too different from those of virgin resin. Studies by Achilias et al. (2007) also found the mechanical properties of recycled product is almost identical to the virgin polymer . However, many research and studied have argued this statement. According to Aurrekoetxea et al. (2001), although recycled polypropylene have greater crystallization rate, higher crystallinity and equilibrium melting temperature than those measured for virgin polypropylene, however elongation at break and fracture toughness of recycle polypropylene decrease. Despite conflict and different opinion in research, recycle plastic have been used in many applications due to lower cost production especially in China. Stastically, from 2006 to 2012, plastic waste imports to China rise from 5.9 metric tons to 8.9 metric tons for industrial purpose such as in manufacturing of desk, flower vases, and tables. Besides, recycle plastic are also popular in wood plastic composite manufactured (Kazemi Najafi, 2013).

WPC made from recycle plastic waste however suffers from some drawbacks. Reinforce material such as coupling agent and mineral filler needs to be added in wood recycle plastic composite (WrPC) manufacturing in order to improve the mechanical properties. Mineral filler are synonym in plastic molding compound. Mineral filler such as calcium carbonate, clay, talc, silica and wollastonite are blended with plastic resin in order to reduce the costs and thermal expansion. It also enhances the plastic's moldability and stability. Other additive in WPC is coupling agent. Coupling agent is chemical or substances that are used in order to treat a surface so that chemical bridge could happen between materials. They link between two surfaces by covalent, polymer entanglement or strong secondary mechanism such as hydrogen bond. Coupling agent can be categorized into two types which are bonding agent and surfactants. By adding these additives, the mechanical strength of WrPC could be improved (Cui, 2008).

1.2 Problem Statement

Generally, the cost of WPC should be cheaper than wood material since it is lighter. However, according Consumer Reports magazine (as cited in Klyosov, 2007), the cost of WPC in market is relatively high as a result from expensive cost of plastics, wood, fillers, and additives (Chanda and Roy, 2006). Hence, development of WPC material by using recycle plastic is very appealing since it will lower the cost production and able to utilize the recycle plastics which are largely generated globally. Accordingly, recycle 95% PP/ 5% PE (rPP/rPE) has been used in this study to prepare WrPC on the basis of kenaf. Combination of rPP and rPE were used to imitate real life plastic wastes which are usually from mixture of different types polymer. Additional cost is needed in order to separate these polymers to their own types. Besides, mixtures of polyethylene and polypropylene have higher mechanical properties than homopolymer.

The mixture of rPP/rPE has different melting temperature and not compatible with each other. According to Kulshreshtha and Vasile (2003), copolymers have lower stiffness and have higher impact strength than homopolymers. Besides, low compatibility of hydrophilic Kenaf wood and hydrophobic of plastic has result in poor mechanical properties of WPC. These behaviours have contributed to degradation quality of WrPC than WPC products.

The uses recycle plastic in composite material has been studied by several researchers before. It has been proven that properties of WrPC especially impact strength was reduce when compare with virgin plastic (Kamdem et al., 2004, Najafi et al., 2007, Chaharmahali et al., 2010, Ghahri et al., 2012). On the other hand, different results have been reported for flexural and tensile properties of WrPC. Some researchers have pinpointed that WrPC have same properties with composite from virgin plastic (Adhikary et al., 2008, Selke and Wichman, 2004, Najafi et al., 2006), some indicated lower properties, (Kamdem et al. 2004) while some reported superior properties (Najafi et al., 2009).

To address the issues of WrPC such as poor impact strength, and poor tensile and flexural properties, calcium carbonate filler were employed in this research. This filler is expected to improve mechanical properties such as tensile properties and impact properties of resulting WrPC.

1.3 Scope of The Study

This work is proposed in order to improve mechanical properties of wood recycle plastic composite by using calcium carbonate filler. To achieve the goals of this works the scopes were listed below:

- Manually pre-mix kenaf wood flour, recycle polyethylene/polypropylene, MAPP coupling agent and calcium carbonate filler at room temperature. Then, the mixture will be extruded into single screw extruder at processing temperature.
- ii. Characterizations of the prepared wood recycle plastic composite in the presence of calcium carbonate filler by using :
 - a) Universal testing machine (UTM)
 - b) Izod impact testing
 - c) Thermal Gravimetric Analysis (TGA)
 - d) Differential Scanning Calorimetry (DSC)
 - e) Scanning Electron Microscope (SEM)

CHAPTER 2

LITERATURE REVIEW

2.1 Wood Plastic Composite

Wood plastic composites (WPC) are commonly known as composite materials made from wood and polymer. Besides, WPC can also define as plastic filled with cellulose fiber and other ingredients. The pioneer of the first generation WPC was achieved by Japanese wood engineer, Sadao Nishibori in 1970. The purpose of this idea is to get cheaper material (Pickering, 2008). WPC material is still considered as a new material in regard to the usage of natural lumber as a building material. WPC materials are basically made from thermoplastic and a lignocellulosic component (eg: wood). Variety of wood can be used in WPC manufacturing such as pinus, kenaf, and oak. Besides, WPC can be made from any type of plastic which have lower processing temperature than 250°C since higher temperature will cause rapid thermal degradation for untreated lignocellulosic materials. Majority of today's WPC are from PE, PP, and PVC. WPC are commonly processed by injection molding or extrusion. Polymer need to have high molecular weight to be process in extruder to produce composite with good melt strength which in contrast with injection mold. In injection mold, the polymer needs to have low molecular weight to maintain low viscosity during processing (Faruk et al., 2007). The manufactured of WPC usually takes place above melting temperature of thermoplastics material and undergo further processed to fabricate different type of WPC products with any shape, colour and sized.

WPC have been used in many applications especially as furniture and decoration. For instance, WPC used in door frame, railing, fence, and interior panel.

WPC are getting a lot of interest in today market due to their good characteristic which considerably ideal for many applications. Markets research found that WPC are expected to reach 3.8 billion by the end year 2016 with a 10% growth per annum.

2.2 Material

In this research WPC are made from by mixing wood flour, recycle plastic, coupling agent and mineral filler together and is processes by extrusion to form composite material. The recycle plastic and wood flour are blend together in ratio of 60:40. Additive such as coupling agent (3 wt.%) and mineral filler (16.5 phr) also added in the mixture before undergo extrusion process. These materials are presented below:

2.1.1 Wood flour

According to Reineke (1966), wood flour indicates finely divided particles of wood which comparable with size, appearance, and texture of cereal flours (as cited in Niska and Sain, 2008). Wood flour is composed by fiber bundles with ratio 5 to 1 than individual wood fiber. It has gain popularity as filler in plastic because it is less expensive, high availability, has biodegradable feature, low density and possesses high stiffness. Despite all these advantages, cellulose, lignin, pectin and hemicelluloses in wood flour have result in water absorption which leads to degradation of fiber-matrix interface. Furthermore, Raj et al., 1989 also state that the hydroxyl groups between wood fibers will form hydrogen bonds that cause the fibers to lump together and lead to uneven distribution of wood flour used as filler usually range from 180 to 840 μm. The basic steps to produce wood flour are: (i) size reduction by using mill, and (ii) size classification by air screening (Reineke et al., 1966). There are variety types of wood flour for composite such as wood flour from pines, oak and kenaf.

Kenaf (Hibiscus cannabinus, L.) is an annual herbaceous plant that can grow in wide range of weather condition. Kenaf usually will grow from 1.5 to 3.5 m with a

woody base. It is famous with it cellulosic source that exhibit economic and ecological advantages. It has low density, non-abrasiveness during processing, high specific mechanical properties, and biodegradability (Nishino et al., 2003). Previously, kenaf have been actively plant and grow because of two reasons. Firstly, because it able absorbs nitrogen and phosphorus in soil while the other reason is because it has the ability to accumulate carbon dioxide at high rates. Nowadays, kenaf have been used in many applications such as in pulp and paper industries, textiles, absorbent and animal feed. Kenaf currently used as material in paper industry to replace other type of woods to avoid deforestation. Kenaf plant consists of several components such as stalks, leaves and seeds. The stalk is made up of woody core and fibrous bark surrounding the core. 30 to 40 percent of the stem dry weight of kenaf is from the bark while remaining 60 to 70 percent is from the core. The fibrous of kenaf which has excellent flexural strength and superior tensile strength has influence the uses of kenaf in variety of products (Akil et al., 2011).

2.1.2 Recycle Plastic

Plastics production has continued to increase since the first mass production of plastic in 1950s. Stastically, in 2014 worldwide plastic production was reported approximately 311 million metric tons, which emphasize increment of plastic production around 4 percent over 2013. This phenomena show more resources are being used in order to meet the increased demand of plastic which result more plastic waste is being produced. Several attempts have been taken to reduce plastic waste globally. One of these attempts is by plastic recycle. Plastic can be recycled to produce new polymeric product. However, they might have different characteristic than virgin plastic as they are collected from different sources and may have undergo different processing condition. Different colour, grade and contaminant in post-consumer plastic also result varying outcome of product characteristics. One example of problem of post-consumer plastic is plastic can be categorized into thermal, biological, mechanical and chemical. Usually, more than one degradation type happens at one time. For instance, thermo-oxidative degradation and thermo-mechanical degradation happen in

recycle polypropylene which results in loss of mechanical properties, and crucial components in plastic. Although, there are some reports which show recycle plastics have some characteristics that are identical to virgin plastic (Pattanakul et al., 1991, Achilias et al., 2007), it have been proved that majority of recycle plastic have distinct characteristic when compare with virgin plastic. Hence, it is really beneficial to study and discover method that capable to improve recycle plastic characteristic as it will affect the properties of resultant product.

Polypropylene and polyethylene are example of polymers that are commonly used in daily life. Yogurt container, chair, and ketchup bottles are some example of PP applications. PP has good chemical resistance and has minimal water absorption due to non-polar structure. Chemical formula for PP is $(C_3H_6)n$. Besides, the average molecular weight of PP varies between 200,000-600,000 g/mol. PP typically has density of 0.90±0.92 g/cm³, tensile strength 21±37 MPa, thermal expansion 150 K⁻¹× 10^6 and thermal conductivity 0.17±0.22 W/mK. Polyethylene (PE) can be divided into two types which is high density polyethylene (HDPE) and low density polyethylene (LDPE). Both types of polymers are chemically inert. However, when LDPE are exposed to light and oxygen, they will loss strength and tear resistance. LDPE have lower density and lower tensile strength than HDPE. LDPE has density 0.91±0.93 g/cm and tensile strength of 8±23 MPa while HDPE has density of 0.94±0.96 g/cm and tensile strength of 18±35 MPa. In contrast with density and tensile strength, LDPE has higher thermal expansion and thermal conductivity than HDPE. (Niska and Sain, 2008)

2.1.3 Coupling Agent

Coupling agents can be defined as the chemical or substances that are used in order to treat a surface so that chemical bridge could happen between materials (Hyer M.W., 2009). Generally, coupling agent work as bonding agent where it link between two surfaces by covalent, polymer entanglement or strong secondary mechanism (eg: hydrogen bond). The week interfacial bonding between the wood flour and polymer matrix can lead to complication when processing take place and results in poor mechanical properties of finished WrPC products. Moreover, (Raj et al., 1989) research

found that the hydroxyl groups between wood fibers will form hydrogen bonds that cause the fibers to lump together and lead to uneven distribution of wood fiber throughout the hydrophobic polymer matrix when WrPC is compounded. By introducing coupling agent, they can help to form chemical bridge between wood fiber and polymer plastic by chemically linked with the hydrophilic cellulosic fiber on one side, while promote the wetting of the hydrophobic polymer chain on the other side. Hence, coupling agent is vital in WrPC manufacturing to overcome issues regarding incompatibility between wood flour and polymer matrix.

Furthermore, there are two type of coupling agent which is bonding agent and surfactants. Derivatives of maleic anhydride and siloxanes are recognized as chemical which are widely used as coupling agent. Maleated styrene–ethylene/butylenes–styrene (SEBS-g-MA) or styrene-maleic anhydride (SMA) behave as coupling agent in WrPC by forming of ester bonds between MA and –OH group in wood (Niska and Sain, 2008). Besides, Silanes can also act as coupling agents since they can be hydrolyzed to expose the active silanol groups toward the hydroxyl groups which result in bond formation (Xie et al., 2010).

The idea on using coupling agent in WrPC was first discovered by Meyer in 1968 (as cited in Kim and Pal, 2010). Later, Gaylord (1972) have been awarded with pattern of Maleic anhydride as a coupling agent in cellulose–polyethylene and cellulose–PVC in the presence of free radical initiator. The uses of coupling agent in WrPC start to get researcher attention in 1980s where a series of patterns have been granted to several researchers (Coran et al., 1978, Woodhams et al., 1984).

There are three basic coupling treatments of WrPC which are: (i) one-step process where coupling agents are directly coated on wood fiber and polymer during mixing, (ii) two-step process, which engaging coating or grafting of coupling agents on wood fibers prior to mixing, and (iii) third process, where a part of polymer and wood fibers are treated with a coupling agent and made as a master batch which then can be eventually used in WrPC manufacturing by proper dilution (Kim and Pal, 2010).

Studies by Kim et al., 2012 have used Maleic anhydride grafted polypropylene (MAPP) coupling agent in bamboo fiber (rPP/rPE) composite. MAPP has density of 0.91 g/cm³. MAPP treat the surface of polymer by esterification process. The hydroxyl groups in wood undergo esterification into an ester group. Chemical alteration of wood fibers with acetic anhydride changes the cell wall polymer hydroxyl groups with acetyl groups, altering the properties of polymers to hydrophobic. As in wood, surface treatment is done by nucleophilic addition of an organic acid anhydride. Acid catalyst will accelerate the reaction by making the carbon of the carboxylic group more positive to increase the nucleophile attraction (Kim and Pal, 2010).

2.1.4 Mineral Filler

Mineral filler are widely use in WrPC industry. Filler is divided into two categories which are organic and inorganic. Filler such as wood flour and shell flour are organic filler while calcium carbonate, aluminium trihydrate, wollastonite, and kaolin fall under inorganic filler category. Filler usually added to WrPC during processing in order to reduce the cost of WrPC, improve the stiffness, flexural strength and increase bending and strength and improve flame resistant. Besides, filler also can work as lubricating agent which improves WrPC processing. Manufacturer's ICC-ES reports (as cited in Klyosov, 2007), Geodesk by LDI Composites has been produced with addition about 20% mineral filler from CaCO₃ and aluminosilicates.

Among variety of inorganic filler, CaCO₃ are commonly used in WrPC. CaCO₃ mineral filler is one of major and abundant mineral, that can be derive into two types which is natural calcium carbonate and precipitated calcium carbonate. Natural CaCO₃ are acquired from mining while precipitated CaCO₃ prepared in laboratory by chemical reaction. CaCO₃ has density around 2.7–2.9 g/cm³ and Mohs hardness approximately 3 to 4. It has from 2 to 6×10^{-6} 1/F linear coefficient of thermal expansion which 10 to 20 % lower than HDPE. The big different in thermal coefficient of HDPE and CaCO₃ cause HDPE filled with CaCO₃ has lower coefficient of expansion. Particle of CaCO₃ have irregular shape with particle size range from 0.02 to 30 µm. Besides, there are three forms of CaCO₃ that can be used as filler which is milled, coated and precipitated.

Majority of CaCO₃ filler in industry used are in milled form (Klyosov, 2007). Filler is really important in WrPC. The size, shape, and content of CaCO₃ can affect mechanical properties of composites by altering micro morphology of base plastics.

2.3 Preparation of Wood Plastic Composite

Kim et al. (2012) prepared WrPC using bamboo fiber as fillers and recycle polypropylene/ polyethylene (rPP/rPE) resin as matrix in the presence of silane coupling agent and precipitated CaCO₃ (PCC) as reinforcing fillers. rPP/ rPE and PCC were blended first in ratio 50:50. Then, the pallets were diluted in different composition of PCC which is 6, 12 and 18 wt.% and were compounded with bamboo filler with 60 wt.% and were extruded. The compounded composite were then pelletized and dried in oven in 24 hours before go through injection molding at temperature of 1785 °C.

Huuhilo et al. (2010) study the impact of mineral filler to moister resistance of WrPC. In this study, WrPC was prepared by using conifer sawdust, polypropylene, variety types mineral fillers including CaCO₃, coupling agent and lubricant in the ratio of 44:30:20:3:3 respectively. The composite was compounded in counter-rotating twinscrew extruder where the temperature in mixing chamber was kept at 190 °C while die temperature at 180 °C. The screw speed also kept at 3 rpm throughout the process.

Kord (2011) prepared composite using wood flour, high density polyethylene (HDPE) and CaCO₃ in the presence of anhydride coupling agent. The wood flour was first dried in oven at (65±2) °C for 24 hours. Wood flour, HDPE, coupling agent, and CaCO₃ mineral filler were then mixed together in mixing chamber of Hake Internal Mixer for about 10 minutes. The resultant composite was then grounded and was dried in oven at 105 °C for 4 hours.

2.4 Application

WrPC have been used as material for indoor or outdoor applications due to their properties which considerably ideal for many applications. Todays, a lot of companies have invested their money to manufacture and sell WrPC products since number people who interested to buy WrPC product increase significantly around the world. Example companies involved in wood-plastic composite market are Trex, Geodesk, EverGreen and Lakeshore companies. The markets for WrPC are expected to reach 3.8 billion by the end year 2016 with a 10 percent growth per annum

Products from WrPC can be found around us. WrPC have been used in wide range of application such as material for furniture (eg: deck, kitchen cabinets, indoor furniture), house decoration (eg: wall decoration and floor), and garden decorations (benches, railing, and fence). Besides, WrPC material also recently gains a lot of interest as green material in automotive industry. Car panel, floor console and window frame are some parts of car that are made from WrPC. Ashori (2008) also has stated that good strength and stiffness in WrPC has result increasing used of WPC in automotive industries.

CHAPTER 3

METHODOLOGY

3.1 Material

Kenaf powder size $\pm 300 \ \mu m$ was supply by Innovative Pultrusion Sdn. Bhd. The fresh powder was dry at $102 \pm 3 \ ^{\circ}$ C for 24 hours in oven to moisturize content of 2-3 % (dry-base). Recycle polypropylene (rPP), recycle polyethylene (rPE) and maleic anhydride polypropylene (MAPP) was supply by a Chemtura Cooperation. Calcium Carbonate, CaCO₃ size 2.7 μ m was supply by Schaefer Kalk (Malaysian) Sdn. Bhd to study the effect of mineral filler on mechanical properties of prepared WrPC. Table 3.1 shows the material and function of each material used to prepare WrPC in this

Material	Function	Scientific name/
		Chemical formulation
Kenaf flour	Filler	Hibiscus cannabinus, L
Recycle polyethylene	Matrix	$(C_2H_4)n$
Recycle polypropylene	Matrix	$(C_3H_6)n$
Maleic anhydride grafted polypropylene (MAPP)	Coupling agent	$(C_4H_2O_3 \cdot C_3H_6)n$
Calcium carbonate	Mineral filler	CaCO ₃

Table 3.1		
List of materials	and func	tions

3.2 Composite Preparation

In this study, rPP, rPE and were used as a matrix while kenaf flour act as suspended particle of prepared WrPC. Kenaf wood flour was first dried in 40 ± 3 °C for 24 hours in oven to moisturize content of 2-3 % (dry-base). Then, all components were weighted according to their formulations as in Table 3.2 where kenaf and rPP are mixed at optimized ratio of 60:40 wt.%. The process is continued by manually premixed kenaf wood flour, rPP, rPE, MAPP coupling agent, CaCO₃ mineral filler in plastic container for 15 minutes. After that, the mixture was compounded through single screw extruder (Labtech Engineering) model LBE20-30/C as in Figure 3.2a. The temperature during compounding in feeding section is 230 °C, 240 °C in middle section while at front section is 230 °C. During this process, the screw rotation speed is maintained at 60 rpm. Prepared composite were then subjected to hot-pressing using $250 \text{mm} \times 350 \text{mm}$ forming frame at 5.17 MPa for about 20 minutes at temperature 215 °C. Figure 3.2b and 3.2c show WrPC obtain after hot press. The samples then were store in desiccator before undergo mechanical and thermal testing. There are two types of WrPC that have been produced, where one type of WrPC is made with addition of CaCO₃ filler while the other one is for reference made of WrPC without addition of CaCO₃ filler.

Composition of composite.					
Composite	rPP (wt. %)	rPE (wt. %)	Kenaf flour (wt. %)	MAPP (wt. %)	CaCo ₃ (phr)
No addition of filler	55.29	2.91	38.80	3.00	-
With addition of filler	55.29	2.91	38.80	3.00	16.50

Table 3.2Composition of composite.



a)



(b)

(*c*)

Figure 3.2. a)Extruder used to compounded prepared composite b)Kenaf rPP/rPE c) Kenaf rPP/rPE/CaCO₃ mineral filler

3.3 Flowchart

In order to demonstrate the overview of the overall process in this research, a flow chart in Figure 3.2 below has been drawn. The upper part of the flowchart shows the processes used to produce wood recycle plastic composite. While the bottom part of the flowchart shows two types of testing to be done after composite is produced.



Figure 3.2. Overall processes to carry out this research

3.4 Characterization Instrumentation

Characterization of composite prepared was achieved by using approaches and techniques similar to those available for composite in general. Prepared composite were characterized by using Universal Testing Machine (UTM), Izod Impact Test, Differential Scanning Calorimetry (DSC) and Scanning Electron Microscopy (SEM). For each characterization, 5 samples were tested and the average from these result were calculated.

3.4.1 Universal Testing Machine (UTM)

Tensile test was carried out according to ASTM D3039 by using Universal Testing Machine (Instron, model 8112) in room temperature at speed of 3mm/minutes with initial gauge length of 90mm on dumbbell-shape specimens. The standard dimension of specimen was prepared as in Figure 3.3. Bluehill 2 software was used to calculate the tensile strength, Young's modulus and elongation at break from the load-extension curve.



Figure 3.3. Standard dimension of specimen for tensile test.

Source: Masirek et al.,(2007)

3.4.2 Izod Impact Test

The test was conducted according to the ASTM D256 standard test method to determine impact resistance of notched specimens in room temperature using (Advance Equipment) impact tester machine. The dimension samples prepared for this test were $13\text{mm} \times 60\text{mm}$. The specimens are notched at 2.84mm using notch cutter (Advance Equipment).

3.4.3 Thermogravimetric Analysis (TGA)

TGA measures the weight changes amount in composite as a function of temperature. This test was carried out according to ASTM E 1331 and was scan from 30-700 °C by thermal analysis system at heating rate 10 °C/min. The sample was placed in open platinum under constant nitrogen flow of 20 ml/min flow rate.

3.4.4 Differential Scanning Calorimeter (DSC)

Prepared composite were characterized using (Mettler Toledo) to determine their melting and crystallization behavior. 7-10 mg sample was sealed with aluminium pans and heated from 30 °C to 300 °C and cooled back to 30 °C after the sample at maximum temperature. The scanning rate for this test is at 10 °C/min.

3.4.5 Scanning Electron Microscopy (SEM)

Scanning electron microscope (Carl Zeiss) was used to examine the structure of WrPC sample. The sample was cut to small pieces and placed onto a SEM stage. An electron beam with accelerating voltage at 10 kV was used on each sample.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Mechanical Properties

4.1.1 Tensile properties

The tensile test measure the ability of a material to withstand forces that tend to pull it apart and determine to what extent the material stretches before breaking (Huuhilo et al., 2010). The tensile strength, tensile modulus and percentage elongation at break was recorded and illustrated in Figure 4.1, 4.2, and 4.3 respectively.

Generally, the higher the value of tensile strength of a material, the better the performance of the material will be. From Figure 4.1, it is clear to understand the effect of CaCO₃ content in the prepared composites. The maximum tensile strength of 8.26 MPa is achieved by WrPC when loaded with CaCO₃ filler. It was found that the presence of CaCO₃ mineral filler in WrPC increases the tensile strength from 7.72 MPa to 8.26 MPa. This is because, as the mineral filler loading, the stress was transferred from the polymer matrix to the CaCO₃ filler and this situation increased the tensile strength of the WrPC (Fairuz et al., 2016). Besides, the improvement of tensile strength by addition of CaCO₃ filler is a result from a good binding between the particles and the matrix.

Based on Figure 4.2, the statistical analysis indicated that the mineral filler loading had significant effect on the mechanical properties of WrPC. The influence of CaCO₃ content on the tensile modulus of prepared WrPC composite was shown in Figure 4.2. As can be seen, the tensile modulus of composite was affected by mineral filler. The modulus tensile of composite increased from 1719.27 MPa to 2160.97 MPa when load with $CaCO_3$. The filler acts as a barrier to the propagation of micro-cracks and imparts a higher flexural modulus. Moreover, $CaCO_3$ filler is stiffer than the matrix and it deforms less, causing an overall reduction in matrix strain, especially in the vicinity of the particles as a result of the particle matrix interface (Borkar et al., 2007).

From Figure 4.3, the elongation at break of WrPC when load with $CaCO_3$ mineral filler is lower which is 0.85% than WrPC without addition of $CaCO_3$ filler where the elongation at break is 0.89%. This is expected due to the improved stiffness of the composites which was attributed to interaction between the polymer and $CaCO_3$ fillers (as cited in Khalaf, 2015). Besides, large amount of agglomeration was more apparent as a result of the high surface energy of $CaCO_3$ could contributed to lower elongation at break of WrPC with $CaCO_3$ load (Yang et al., 2006).



Figure 4.1. The effect of filler CaCO₃ on tensile strength of WrPC



Figure 4.2. The effect of CaCO₃ on tensile modulus of WrPC



Figure 4.3. The effect of CaCO₃ filler on elongation at break of WrPC

4.1.2 Izod Impact Properties

The ability of component to withstand a sudden impact is obviously of great importance for any practical application of the material. Impact resistance of a composite is the measure of total energy dissipated in the material before final failure occurs (Khalaf, 2015). Figure 4.4 shows the impact strength of WrPC with addition of CaCO₃ filler and without CaCO₃ filler.

Figure 4.4 shows the effect of CaCO₃ on the impact strength of prepared WrPC. As can be seen, the impact strength was influence by CaCO₃ content. The impact strength of prepared WrPC in the presence of CaCO3 filler decreased from 7.14 KJ/m² to 6.14 KJ/m². The lower impact strength might due to effect of brittleness of CaCO₃. Besides, according to Sudheer et al. (2014), the effect of fillers on the impact performance is due to the volume they take up. Fillers unlike the matrix are incapable to dissipate stress through shear yielding prior to fracture. Therefore, the total ability of the material to absorb energy is decreased. Moreover, incorporation of fillers may also hinder the local chain motions of polymer molecules that enable them to shear yield, thereby lowering the composites to absorb energy during crack propagation. The above results show that the presence of CaCO₃ mineral filler will lower the toughness of WrPC.



Figure 4.4. The effect of CaCO₃ filler on impact strength of WrPC

	Tensile	Tensile	Elongation at	Impact
Composite	strength,	modulus,	break,	Strength
	σ_t (MPa)	E _t (MPa)	E _t (%)	(kJ/m^2)
Kenaf rPP/rPE	7.72	1719.27	0.89	7.14
Kenaf rPP/rPE/CaCO ₃	8.26	2160.97	0.85	6.14

Table 4.1Summary of mechanical properties of WrPC

4.2 Thermal Properties

4.2.1 Thermogravimetric Analysis (TGA)

TGA was carried in order to study the thermal decomposition of prepared WrPC (Pai Verneker et al., 1977). From TGA analysis, the relationship between the percentage of weight loss against temperature for prepared WrPC with and without CaCO₃ filler loading was recorded. The obtained result is shown in Figure 4.5.

Based on Figure 4.5, it can be observed that the curve of both prepared WrPC only has one decomposition step. However at 249 °C, both prepared WrPC with and without addition of CaCO₃ mineral filler losses about 2.91% of its initial mass. This behavior may indicate the loss of moister in prepared WrPC. It also can be detected that at temperature 470 °C, prepared WrPC without addition of CaCO₃ filler have almost decomposed completely, where it undergo almost 100% degradation. WrPC with CaCO₃ loading as predicted prohibits complete combustion of the composite because 16.23 % of the sample still remained at 700 °C.

Hence, it can be assume that $CaCO_3$ responsible for retarding thermal decay, since they have high thermal resistance. According to (Galwey and Brown (1999), the temperature for degradation of $CaCO_3$ is high which is around 900 °C. Moreover, macro sized of $CaCO_3$ particles could fill available spaces between kenaf and recycle plastic in the composite and prevent releasing volatile products made by decomposing of components in the composite (Qin et al., 2004). CaCO₃ does not decomposed under experimental condition used, but remain behind as residue while prepared WrPC undergo complete degradation (Yang et al., 2006, Lei et al., 2007). Besides, according to Allen and Edge (1992), the strong interaction between CaCO₃ particles, rPP and rPE chains may also reduce chain movements and thus retard conveying free radicals produced during fragmentation process of polypropylene as a result of thermal degradation. Hence, diffusion of volatile oligomers is hindered by CaCO₃ and the degradation of WrPC shifts towards higher temperature regions (Luyt et al., 2009).



Figure 4.5. Relationship between weight losses at different temperature of WrPC.

4.2.2 Differential Scanning Calorimeter (DSC)

The DSC technique was used to determine the thermal decomposition of the WrPC. The DSC thermograms of WrPC without CaCO₃ filler and with CaCO₃ filler are as illustrated in Figure 4.6. The thermal parameters such as glass transition, T_g taken at glass transition peak, and heat of fusion, H_m were determined from the thermograms and summarized in Table 4.2.

Based on Figure 4.6, there are two glass transitions endotherms were detected at low temperature (T_{g1}) and higher temperature (T_{g2}) for both prepared WrPC. For WrPC without addition of CaCO3 filler, T_{g1} is at 127 °C and T_{g2} is at 162 °C. Meanwhile, for prepared WrPC with addition CaCO3, Tg_1 is at 127 °C and Tg_2 is at 163 °C. The appearance of two glass transition for both samples indicates that prepared composite is immiscible where kenaf, rPP, and rPE do not form homogenous mixture. Besides, the additions of CaCO₃ also cause slight increment of T_{g2} of prepared composite. This is cause by the strong interfacial interaction between $CaCO_3$ and kenaf/rPP/rPE, which reduced the free volume and restricted the motion of rPP and rPE chains, thus raising the T_g of WrPC (Yu et al., 2012, Chafidz et al., 2012).

Besides, Figure 4.6 clearly shows that there is no crystalline temperature, Tc for both WrPC without addition of CaCO₃ and WrPC with CaCO₃ loading. This show that the addition of small quantity of rPE (5%) in composite where majority of polymer used is rPP (95%) do not affect the crystalline behavior of amorphous rPP in prepared WrPC. Moreover, heat of fusion, H_m of WrPC in the presence of CaCO₃ is higher which is increased from 207.89 J/g to 235.11 J/g. This shows that CaCO₃ mineral filler support crystallization process and can be categorized as active filler in the sense of nucleation effect (Saujanya et al., 2002, Ning et al., 2007).



Figure 4.6. DSC thermograms of WrPC

Therman properties of prepared composite				
Samples	$T_{gl}/^{\circ}\mathrm{C}$	$T_{g2}/^{\circ}\mathrm{C}$	H_m /J g ⁻¹	
Kenaf/rPP/rPE	127	162	207.89	
Kenaf/rPP/rPE/CaCO ₃	127	163	235.11	

Table 4.2Thermal properties of prepared composite

4.3 Morphology Study

The micrographs of tensile fracture of WrPC with and without addition of $CaCO_3$ filler were obtained at the magnification of 1000x from Scanning Electron Microscope (SEM) and shown in Figure 4.7 and 4.8. The samples were sputter coated by Titanium using a sputter-coater to avoid charging under the electron beam.

From Figure 4.7, it can be observed that the dispersion of kenaf rPP/rPE is poor. Besides, it also can be seen that prepared composite have very inhomogeneous fracture surface. Moreover, it can be observed that, in white circle, there are many voids concentrated at one place which result to lower mechanical strength of the composite.

Figure 4.8 show SEM image of kenaf/rPP/rPE with addition of CaCO₃ filler. It can be seen, in yellow circle, the presence of CaCO₃ filler in the composite. Besides, it can be observed that the composite has better dispersion, interfacial bonding and compatibility than WrPC without addition of CaCO₃. Kenaf powder embedded in the matrix as a result of good compatibility and strong adhesion between rPP/ rPE and kenaf fiber. The fracture did not happen at interface due to the strong bonding between the matrix and kenaf powder. Moreover compatibilizing agent, MAPP also help to improve the interfacial bonding which result in better tensile properties of the composite.



Figure 4.7. SEM of composite kenaf/rPP/rPE at 1000x magnification.



Figure 4.8. SEM of composite kenaf/rPP/rPE/CaCO₃ at 1000x magnification.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In conclusion, this project has demonstrated the effect of $CaCO_3$ loading on mechanical, thermal and morphological properties of WrPC. From the experimental results, the following conclusions can be made.

- i. Addition of $CaCO_3$ mineral filler help to improve the tensile properties of WrPC. However, strain to break and impact strength was found lower with loading of $CaCO_3$.
- ii. Both prepared WrPC exhibit two glass transitions which is T_{g1} and T_{g2} . This shows that the prepared composite is immiscible blend. Besides, the addition of CaCO₃ has result in higher Tg.
- iii. TGA result show both prepared WrPC with and without addition of $CaCO_3$ only have one decomposition step. WrPC without $CaCO_3$ loading decomposed completely at 470 °C. Meanwhile, WrPC with $CaCO_3$ loading prohibits complete combustion of the matrix since 16.23 % of the sample still remained at 700 °C.
- iv. Morphology test show that WrPC with addition of CaCO₃ filler have better dispersion and fewer voids than WrPC without addition of CaCO₃ filler.

5.2 **Recommendation**

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

- i. Study the mechanical properties which varying content of mineral fillers.
- ii. Extruder must be connected with suitable die to ensure the sample obtain is easy to use for hot press.
- iii. Make sure during extrusion process, the sample is loading continuously.
- iv. The sample must be kept in desiccator before testing in order to ensure no moister content which will reduce result accuracy.

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APPENDIX A

Tensile Test



A.1. Tensile test on kenaf/rPP/rPE



A.2. Tensile test on kenaf/rPP/rPE/CaCO₃

APPENDIX B

Izod Impact Test







APPENDIX C

Thermogravimetric Analysis



APPENDIX D

Differential Scanning Calorimeter



