UNIVERSITI TEKNIKAL MALAYSIA MELAKA

DURABILITY OF RUBBER WOOD FLOUR-POLYMER COMPOSITE EXPOSED TO ACCELERATED FREEZE THAW CYCLING

This report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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DECLARATION

I hereby, declared this report entitled "Durability of Rubber Wood Flour-Polymer Composite Exposed to Accelerated Freeze Thaw Cycling" is the result of my own research except as cited in references.

Signature : *Arni Nabila*

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Date : 26 JANUARY 2022

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

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ABSTRACT

Environmentally, composite reinforced with natural fibers due to great potential to substitute the traditional composite reinforced with glass fiber. Wood Polymer Composite (WPC) have more consistent mechanical properties. Despite such advantages, WPCs are prone to deterioration from causes such as moisture and freeze-thaw cycling, fire exposure, and biological attack. Deterioration of WPCs negatively impacts performance, limits expected service life, and inhibits consumer confidence, which prevents the widespread use of WPCs as a preferred material for suitable construction applications. In this study, the objective is to study the effect of freeze thaw cycle onto the mechanical properties of the rubber wood reinforced recycled polypropylene. Then, to correlate the effect of freeze thaw cycle onto the fracture surface of rubber wood reinforced recycled polypropylene by using Scanning Electron Microscopy (SEM) to evaluate the rubber wood flour-plastic composite in terms of durability performance on mechanical properties using Universal Testing Machine (UTM). This experimental were carried out by the preparation of the rubber wood flour-polymer composite pallet then optimize of the morphological structure using SEM and crystalline structure using XRD to be analyzing the crystal structure to identify crystalline phase in a material. The result showed the durability performance on mechanical properties using UTM. An addition, to identify the testing, the WPC pallets must freeze thaw cycling for 2, 3, 4, 5 cycles respectively to control behavior in considering the performance. Moreover, the result indicated that study of the potential of WPCs application for the purpose of better durability performance on mechanical properties and the data collected after completing the sample fabrication and testing experiment. All the hypotheses and discussions will be supported by the previous research statement based on the effect of water absorption, flexural behaviour and properties, fracture surface analysis, mechanical and physical properties.

ABSTRAK

Dari segi alam sekitar, komposit diperkukuh dengan gentian semula jadi kerana potensi besar untuk menggantikan komposit tradisional yang diperkukuh dengan gentian kaca. Komposit Polimer Kayu (WPC) mempunyai sifat mekanikal yang lebih konsisten. Walaupun terdapat kelebihan sedemikian, WPC terdedah kepada kemerosotan daripada punca seperti kitaran kelembapan dan beku-cair, pendedahan kebakaran dan serangan biologi. Kemerosotan WPC memberi kesan negatif terhadap prestasi, mengehadkan jangka hayat perkhidmatan dan menghalang keyakinan pengguna, yang menghalang penggunaan WPC secara meluas sebagai bahan pilihan untuk aplikasi pembinaan yang sesuai. Dalam kajian ini, objektifnya adalah untuk mengkaji kesan kitaran pencairan beku ke atas sifat mekanikal polipropilena kitar semula bertetulang kayu getah. Kemudian, untuk menghubungkaitkan kesan kitaran pencairan beku ke atas permukaan patah polipropilena kitar semula bertetulang kayu getah dengan menggunakan Scanning Electron Microscopy (SEM) untuk menilai komposit tepung-plastik kayu getah dari segi prestasi ketahanan pada sifat mekanikal menggunakan Mesin Pengujian Sejagat (UTM). Eksperimen ini dijalankan dengan menyediakan palet komposit tepung-polimer kayu getah kemudian mengoptimumkan struktur morfologi menggunakan SEM dan struktur hablur menggunakan XRD untuk menganalisis struktur hablur bagi mengenal pasti fasa kristal dalam sesuatu bahan. Keputusan menunjukkan prestasi ketahanan pada sifat mekanikal menggunakan UTM. Selain itu, untuk mengenal pasti ujian, palet WPC mesti membekukan kitaran pencairan untuk 2, 3, 4, 5 kitaran masing-masing untuk mengawal tingkah laku dalam mempertimbangkan prestasi. Selain itu, keputusan menunjukkan bahawa kajian tentang potensi aplikasi WPC untuk tujuan prestasi ketahanan yang lebih baik pada sifat mekanikal. dan data yang dikumpul selepas melengkapkan fabrikasi sampel dan eksperimen ujian. Semua hipotesis dan perbincangan akan disokong oleh pernyataan kajian terdahulu berdasarkan kesan penyerapan air, kelakuan dan sifat lentur, analisis permukaan patah, sifat mekanikal dan fizikal.

ABSTRAK

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DEDICATION

Only

my beloved father, Mohd Zainuddin

my appreciated mother, Saupisah Hussin

my adored sisters, Arisya, Amrina and Annur

for giving me moral support, money, cooperation, encouragement, and understandings.

Thank You So Much & Love You All Forever.

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LIST OF SYMBOLS

cm	- Centimeter
m	- Meter
%	- Percent
g/cm^3	- Grams per centimeter cube
wt. %	- Weight percent
mm	- Millimeter
MPa	- Mega Pascal
°C	- Degree Celsius
nm	- Nanometer
kg.cm3	- Kilogram centimeter cube
kg	- Kilograms
mm/min.	- Millimeter per minute
kN	- Kilo newton
W	- Sample width
S	- Span length
a	- Notch length
B	- Sample thickness
K_{IC}	- Fracture toughness
W_m	- Matrix mass
W_f	- Fibre mass
T_i	- Thickness before immersion
T_f	- Thickness after immersion
m	- Mass
v	- Volume
°C/min	- Degree Celsius per minute

LIST OF ABBREVIATIONS

ASTM	-	American society for testing and materials
DSC	-	Differential scanning calorimetric
SEM	-	Scanning electron microscope
XRD	-	X-ray diffraction
TA	-	Thermal analysis
FT	-	Freeze thaw
WF	-	Wood fibre
WPC	-	Wood plastic composite

CHAPTER 1

INTRODUCTION

1.1 Research Background

A composite material is a macroscopic blending of two or more distinctive materials which have a conspicuous interface between them. According to Friedrich, (2018) described that the WPCs contain up to 70% of wood fibres embedded in a thermoplastics matrix such as polypropylene (PP), polyethylene (PE) or polyvinyl chloride (PVC). As a composite, the wood fibre reinforcement adds strength and stiffness, while the polymeric matrix transfers the applied load throughout the material. In addition, WPCs have more consistent mechanical properties, which enables reduced member cross-sections and a corresponding decrease in the amount of material needed for design. Moreover, WPCs are lightweight and can be formed into a wide variety of custom shapes using different processing techniques.

The demand for WPCs primarily stems from the automotive and construction sectors for applications such as interior or exterior car parts and decking, fencing or siding materials. Based on Luible (2016) by consolidating essential material, composites can be intended to provide structural properties and as additional fundamental materials that have exceptional properties for electrical, thermal, tribological, environmental, and biomedical application. For construction, WPCs have some advantages over competing products, but they are also susceptible to environmental deterioration mechanisms. Moreover, Román (2019) indicated

WPCs is a material containing pigments. In fact, inorganic pigments have an excellent UV absorption, good IR-reflective properties, and heat stability and a good choice as additives used for making composites for outdoor applications.

Further research work needs to be done to achieve study to estimate the durability of the composites manufactured from plastic waste of different sources. Researchers like Turku, (2018) and Marossy, (2019) are more focused on using another method in which composite samples were weathered under accelerated freeze-thaw cycling and xenon-arc light standard conditions. Then the results showed that the composites had significant changes in their flexural properties and behaviour. In the same weathering conditions, the property changes in the reference, processed from virgin polymer, were insignificant according to the ANOVA test. Thus, the focus is on the performance of WPCs especially particularly with respect to the effect of moisture and freeze thaw cycling onto the mechanical properties of the WPCs.

1.2 Problem Statement

WPCs are prone to deterioration from causes such as moisture and freeze-thaw cycling, fire exposure, and biological attack. Deterioration of WPC negatively impacts performance, limits expected service life, and inhibits consumer confidence, which prevents the widespread use of WPCs as a preferred material for suitable construction applications.

Currently, the literature lacks consensus on whether freezing temperatures in the presence of moisture cause freeze-thaw-induced damage in addition to moisture-induced damage in WPCs. Furthermore, the wood fibre reinforcement swells due to the absorption of moisture, and the absorbed moisture further expands upon freezing. Therefore, according to a study reported (Wang et al, 2005) that the outdoor applications of these materials have raised concerns about their durability, including fungal resistance, ultraviolet resistance, moisture resistance and dimensional stability. A few weathering test methods have been developed to stimulate natural weathering at an accelerated rate so that long term weathering effects can be rapidly estimated.

In addition, since the WPCs have become increasingly used for outdoor applications, a need to understand their physical and mechanical behaviour under weathering conditions has risen. Based on the research above, the previous studies have generally concentrated on the performance of the effect of water immersion freeze-thaw cyclic treatment on the mechanical properties. Thus, this research is focusing on WPCs effect of influence of moisture and freeze thaw cycling onto the mechanical properties to optimize the performance of WPCs.

1.3 Objectives

The objectives are as follows:

(a) To study the effect of freeze thaw cycle onto the mechanical properties of the rubber wood reinforced recycled polypropylene.

(b) To correlate the effect of freeze thaw cycle onto the fracture surface of rubber wood reinforced recycled polypropylene.

1.4 Scopes of the Research

The scopes of study are as follows:

a) From objective 1: Within the first objective, the effect of aging onto the composite will be characterized by flexural testing using Universal Testing Machine.
b) From objective 2: Within the second objective, the fracture surface of the aged and non-aged composite will be evaluated using SEM and their correlation with mechanical will be investigated. Within this objective also can determine the crystallographic structure of material using XRD analysis.

1.5 Report Organization

The organization of this report is as follows. Chapter 1 begins with research background, problem statement, objectives, and scope of the research. The rationale of research is delineated to better define this thesis. Chapter 2 literature review comprises previous study or research about the durability of rubber wood floor-polymer composite that is exposed to accelerated freeze–thaw cycling. Chapter 3 methodology describes all the raw materials, testing method used will be stated in the research about the effect of moisture and freeze thaw cycling onto the mechanical properties of the WPCs, testing method, theory of interface and interphase. Chapter 4 is analysing the information collected after running testing through tensile and fracture testing machines, then discussing the effect of moisture and freeze thaw cycling onto the mechanical properties of the WPCs. In Chapter 5, conclusions and recommendations about this research are examined.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Wood polymer composites (WPC) consist of natural plant fibres which are combined in a polymer matrix, such as polypropylene (PP), polyethylene (PE), or polyvinyl chloride (PVC). (Machado et al., 2016) described that WPCs can be produced from environmentally friendly materials, such as wood fibres, unused natural resources, and recycled thermoplastic resins. WPCs commonly used in many building applications which consist of several applications of WPCs such as outdoor exposure or ground contact. Although WPCs technology continues to grow together as the manufacturing processes start to polish up, WPCs can also be used in other industries, such as the automobile and consumer electronics sectors according to Kim & Pal (2011). WPCs are identically a solid wood with high moulding performance and typically as an action such as combining the best properties of wood and plastic which good performance resulting which makes a product brittle and may become more brittle by cold exposure but further can increase the mechanical properties such as high durability, specific strength, specific stiffness, and long-term resistance to wear.

2.2 Polymer Composite

A composite is a combination of two or more combinations of two or more materials which can be categorized into two categories which are plant-based and animal-based fibres that are made to become a single material according to Butylina (2011). To be used as reinforcement, pure fibres as shown in Figure need to be extracted and separate from all next connections that were existence in the natural plant or animal raw material (hemicelluloses, lignin, wax, proteins).

Zini & Scandola (2011) explained that wood is a fibrous composite because it is commonly used in the form of wood pulp. Figure 2.1 shows the specific mechanical properties of natural fibres closer to the synthetic fibres and specific tensile strength of the flax fibres compared with the glass fibres. Additional advantages of using the natural fibres in composites are their renewability, biodegradability, nontoxicity, good insulation properties and low machine wear.

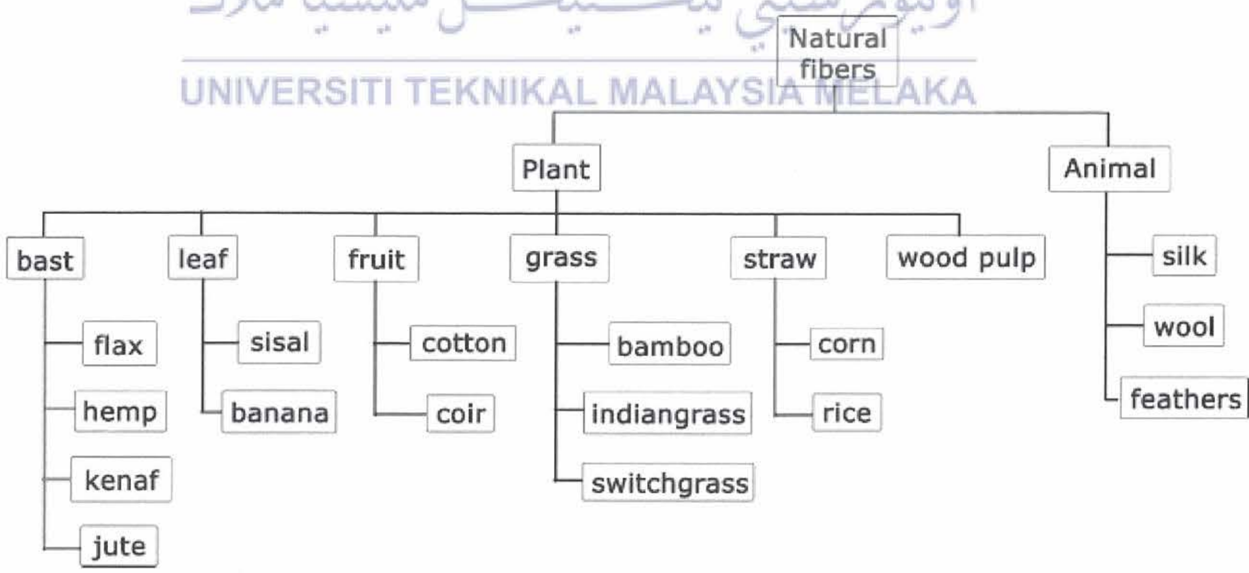


Figure 2.1: Classification of Natural Fibres (Zini & Scandola, 2011)

2.3 Wood Fibre

Wood fibre are elements of cellulosic that are extracted from trees and used to make materials. Wood Fibre can be categorized into two classes which are softwood and hardwood. However, Valente (2016) described that cellulose, hemicellulose, and lignin are the structural polymers of hardwoods. Hardwoods contain a smaller number of extractives of non-structural constituents compared to the softwoods as shown in the Figure 2.2.

In North America, virgin (non-recycled) wood fibre was extracted from hardwood (deciduous) trees and softwood (coniferous) trees. Besides, wood fibre can be a primary product. Thus, wood fibres can also combine with thermoplastics to produce strong, waterproof products for outdoor usage such as deck boards or outdoor furniture. Ashori (2010) investigated that by using wood fibres as reinforcement to thermoplastic resins, it can yield composite materials with increasing strength and stiffness.

(a) (b)

Figure 2.2: Structure of (a) Hardwood (Beech) Structure and (b) Softwood (Scots Pine) Structure (Valente et al., 2016)

2.4 Thermoplastic

Thermoplastics are the most important elements of plastic materials that are available in their consumption which is approximately 80% or more of plastic consumption. Thermoplastics are softened and sometimes melted when heated to a flow-able state and under high pressure so that they can repeat the cycles of heating and cooling without causing severe damage. In addition, thermoplastic is often added as an additive or filler to improve specific properties such as both mechanical properties and thermal properties.

The thermoplastic has many advantages including recyclability, short processing cycles, the melting and softening by heating which allows thermoforming. In contrast, the disadvantage is decreasing in strength and stiffness and giving high relaxation behaviours which increase temperature. However, thermoplastic is popular in the application of WPCs including PE, PP and PVC.

2.4.1 Polypropylene (PP)

Polypropylene has a similar chemical structure to polyethylene, but PP has better strength, stiffness, and heat resistance but at low temperature, the impact strength is quite poor. The properties of the PP such as chemical resistance, dimensional stability, heat resistance, rigidity, toughness, surface gloss and low cost. However, the pure PP are not suitable for use in load-bearing applications due the specific heat for PP is lower than PE. Therefore, due to excellent quality and versatility, PP were manufactured using injection, extrusion, and compression moulding. PP has also been used in the textile industry and PP also in thin-film packaging according to John Wiley & Sons (2003).

2.5 Manufacturing Process

Manufacturing processes are methods which convert raw materials into products. The process used to manufacture the WPCs have several methods including injection moulding and extrusion but the process that is common and important in the application is compression moulding. For example, WPCs being produced by mixed wood floors with plastic and adhesive, the next process was moulded and then formed by using a compression moulding machine.

Compression moulding of thermoplastic consists of four stages which is firstly, material is in a mould and is open heated. Secondly, material is compressed at high pressure to fill the hot cavity space between the two parts of the heated mould and softened just shown in Figure 2.3. Then mould is shaped and then is cooled to solidify. Finally, the upper mould is opened to remove the product. In addition, the compression moulding has an advantage, which is no sprues, but it also has a disadvantage which includes small outputs, high labour costs and low output rates because it takes a long time to heat and then cool the material parts before demoulding. This method process is according to M. Biron (2007).

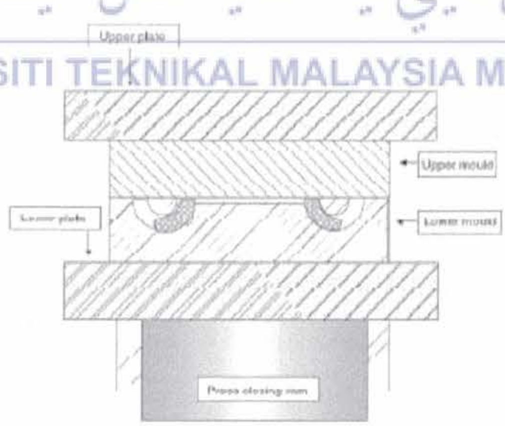


Figure 2.3: Illustration of Compression Moulding Machine (M. Biron, 2007)

According to Salim Hiziroglu (2017), PP granules and rubber wood fibre granules were extruded into WPC granules and then the compression moulding machine is used to make the specimens. The specimen is made with 7wt% calcium carbonate content which is the optimum level of filler in overall mechanical and physical properties of WPCs specimen which resulted in the highest strength of composite according to Thanate Ratanawilai (2017). K.B. Adhikary (2008) stated that wood fibre, PP and additive filler were compounded in a high-speed mixer and then mixed to manufacture the WPCs pellets. Then the mixed samples were pressed in a compression moulding according to S. Pang (2008). S.M.L. Rosa (2007) investigated that the samples were pressed to cool off while the samples were still under compression before removal.

From the literature review, the production of WPCs used a compression system which is appropriate for manufacturing sample composite. Therefore, the mixing of wood fibre and plastic matrix with additives was a suitable manufacturing process for the WPCs since it is very important due to the demand of their application.

2.6 The Freeze Thaw Mechanism

Generally, Ossi Martikka (2011) research was only focused to study the effect of moisture and freeze-thaw cycling on wood-plastic composites containing pigments. In Malaysia, there were climatic reasons such as moist and freeze-thaw actions are commonly important, which is water immersion-freeze-thaw durability in determining the service life of WPCs. The water absorption process in WPCs showed that the process is followed by the mechanisms which are described by Fick's law. In this study, the mechanical properties of the WPC were measured before and after freeze-thaw cycling. To study the surface qualities, scanning electron microscopy (SEM) was applied and to study fracture surfaces after a mechanical testing, Differential scanning calorimetry (DSC). The thermal testing of WPCs was also studied.

According to Klyosov (2007), WPC is considered a porous material because the polymer itself can be porous when it is filled with lignocellulosic fibre and other additives at high temperature. The resistance of the WPC to moisture and freeze-thaw was tested under cyclic conditions specified by the standard EN 321. The test samples were exposed for a few cycles, each comprising immersion in water at 23 degrees (approximately), freezing at the temperature of -20 degrees (approximately).

Laurent M. Matuana (2005), investigated that freeze–thaw cycling shown in Figure 2.5 was done in accordance with a modified ASTM D6662–01 which refers to the standard for polyolefin-based plastic lumber decking boards. One complete freeze–thaw cycle consists of three parts which are soaking in the water at 21°C with an accuracy of ±3°C until equilibrium moisture content (EMC) (refer Figure 2.4). During the water submersion, each sample was weighed every 24 h until the weight gain was less than 1.0% in the 24 hours' period. After the water submersion, the samples were frozen for 24 h at the controlled temperature of - 27°C with an accuracy of ±3°C. Finally, the samples thawed for 24 hours in a controlled environment (21±3°C and 50± 5% RH) using a humidity chamber according to Jeanette M. Pilarski (2005).

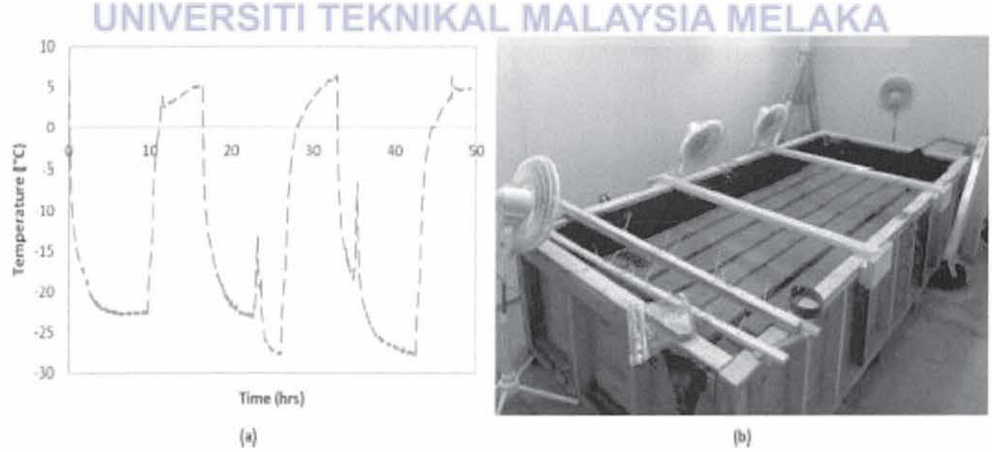


Figure 2.4: Freeze-thaw conditioning: (a) ambient temperature of room and (b) setup (Jeanette M. Pilarski, 2005)

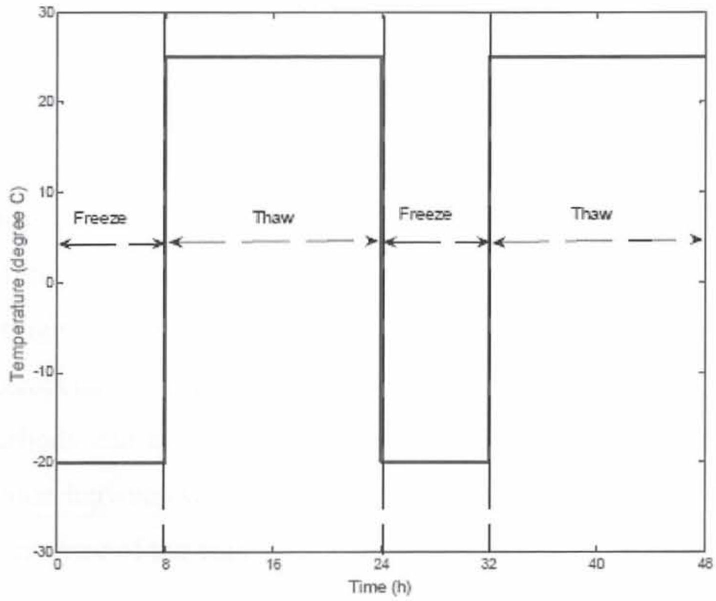


Figure 2.5: Two full cycles of the cyclic freeze-thaw weathering (Laurent M. Matuana, 2005)

2.6.1 Influence of Freeze Thaw on the Properties of Wood Polymer Composite

In weathering exposure, material often degrades by the loss of mechanical properties. Material mechanical properties were usually measured through mechanical testing to characterize degradation of WPCs. The most often measured properties include tensile strength, flexural modulus, and hardness according to Pilarski and Matuana (2006). Thus, they also depend on the test procedure, testing method, reporting of the property values, and manufacturing process of the materials that was investigated by J. Prachayawarakorn, J. Khamsri, K. Chaochanchaikul and N. Sombatsompop (2006). In addition, the mechanical properties are considered in the design of goods and the process of materials selection. The design and selection of material for various applications are often considered based on mechanical properties such as tensile and flexural strength and hardness.

2.7 Tensile Properties

In a study of the durability of WPC with high wood content, tensile strength was measured to quantify the effects of weathering on WPC. Usually, WPCs are selected to obtain desirable mechanical properties at low cost. Such properties could be considered the most important quality of polymer composites for most of the application. For homogenous material, the mechanical testing methods can effectively describe the mechanical properties of a material. However, for heterogeneous materials with complex structures like wood flour polymer composite, the methods can only provide indirect and limited insight into the nature of the mechanical interaction between wood particles and polymer matrix is a failure mechanism and the long-term performance of the composites according to Oberdorfer and Golser (2005) (Refer Figure 2.6).

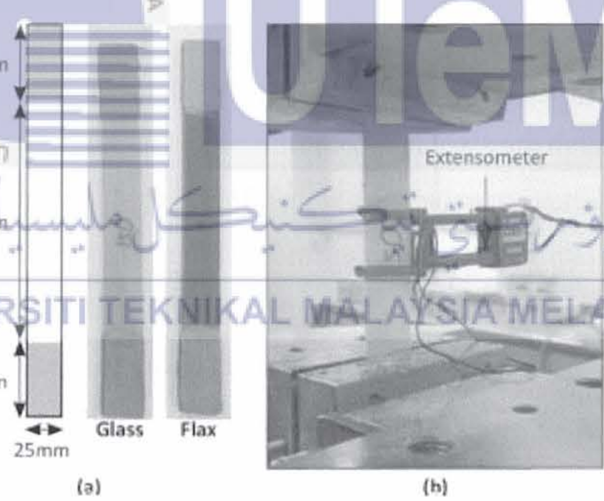


Figure 2.6: Tensile testing (a) specimens and (b) setup (Oberdorfer and Golser, 2005)

Pilarski and Matuana, (2006) reported that the effect of moisture during freeze-thaw cycling is the main reason for loss in the mechanical properties of the composites The results showed that the reference composite had higher changed in mechanical properties to the composites, and this became even more pronounced after the freeze-thaw cycling test.

The effects of freeze–thaw cycling on physical and mechanical properties of WPCs were studied in the present research. The freeze–thaw cycling did not have significant effects on thickness swelling of specimens while it caused decrease in MOR and MOE values at the first cycle. Repetition of freeze–thaw cycling proved not to bring about significant changes in these properties. Residual MOR and MOE values were higher in layered specimens and un-layered specimens were more sensitive in freeze–thaw cycling. MOE values were affected more by freeze–thaw cycling in comparison with MOR values. PVC content had positive effects on both physical and mechanical properties. Around 60–70% of the mechanical properties were retained even after three freeze–thaw cycles. However, the 30–40% mechanical properties loss is considerable indicating that the produced composite materials should be protected against freeze–thaw cycles in service.

2.8 Hardness Properties

The strength and toughness of fibre-reinforced materials are determined by the interface between the fibres and matrix. which is defined as an imaginary surface forming a boundary between the phases (a wood particle and the surrounding polymer matrix). A strong interface creates a material that displays a good composite between the matrix and the fibre. A weaker interface reduces the efficiency of stress transfer from the matrix to the fibre which acts as a filler and as a result the strength and stiffness are lower than in the composites according to Jacob et al. (2005).

2.9 Flexural Properties

Flexural test is a measurement of the ability of a material to withstand bending forces applied perpendicular to the longitudinal axis and is a combination of tensile and compressive stress shown in Figure 2.7. When the specimen is subjected under load, the side of material that opposes the loading undergoes the tensile stress, but the side of the specimen being loaded undergoes compressive stress. These stresses reduce to the centre of the specimen then the flexural specimen is not in a state of uniform stress on the specimen. Theoretically, the centre is a plane that is called the neutral axis, which is not where stress and strain occur.

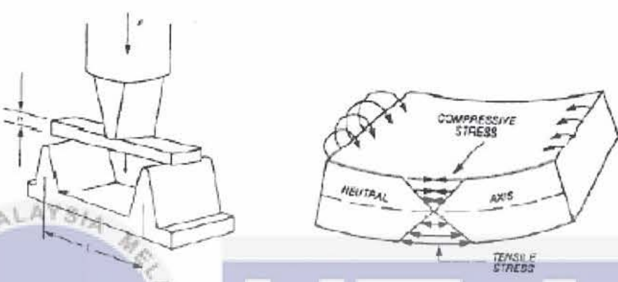


Figure 2.7: Illustrated Flexural Strength Testing (Pilarski & Matuana, 2006)

The Freeze-Thaw specimens were useful for beam size for flexural strength determination. But the retained flexural strength was not useful for prediction of Durability Factor (DF). Based on findings from this study, it may be concluded that freeze-thaw cycling of 40-100 cycles caused 24-30% change in the flexural strength. It should be noted that all beams' fractures took place in the middle third of the span length during flexural test according to Pilarski & Matuana (2006).

2.10 Thermal Properties

Theoretically, the enthalpy and crystallization value of the composites increased with increasing different contents of filler of WPCs. The results indicated that these seven formulations exhibited nearly identical peak melting and crystallization temperatures at about 161°C and 123°C (respectively) according to Y. Wang (2020). From the figure below, the WPCs filler with different chemical contents exhibited slightly higher values of melting temperature, enthalpy and crystallization compared to those of control samples. In contrast, the cooling temperature of the samples presented slightly lower values as compared with WPCs. Therefore, the WPCs filler with 7% CC is optimum crystallization. In addition, it can resist such high temperatures in thermal testing.

2.11 Applications

Wood polymer composite (WPC) has emerged as a dynamic growth material in residential and industrial applications according to Adhikary (2008). However, these applications are mostly in interior non-structural purposes like automotive, furniture and building industry although exterior wood market is the main target for WPCs. The mechanical properties of WPCs may not be sufficient for heavy loading structure applications, but there has been an increasing interest in using WPCs material for applications where the stability and durability are important.

By today there is no applicable concept according to which bio-based plastics can be developed for the purpose of high durability in façade applications as demanded by building construction. The outdoor applications of these materials have raised concerns about their durability, according to W. Wang (2005) and Yeh & Gupta (2008) explained that WPCs mostly manufactured through extrusion, injection moulding processes and also compression moulding can be used in the automotive which is in manufactured a dashboards or screen-doors of vehicles and construction industries such as interior floor coverings, profiled parts for doors and windows, ornamental panels, external shutters, pavements, garage or entrance doors as shown in Figure 2.8.

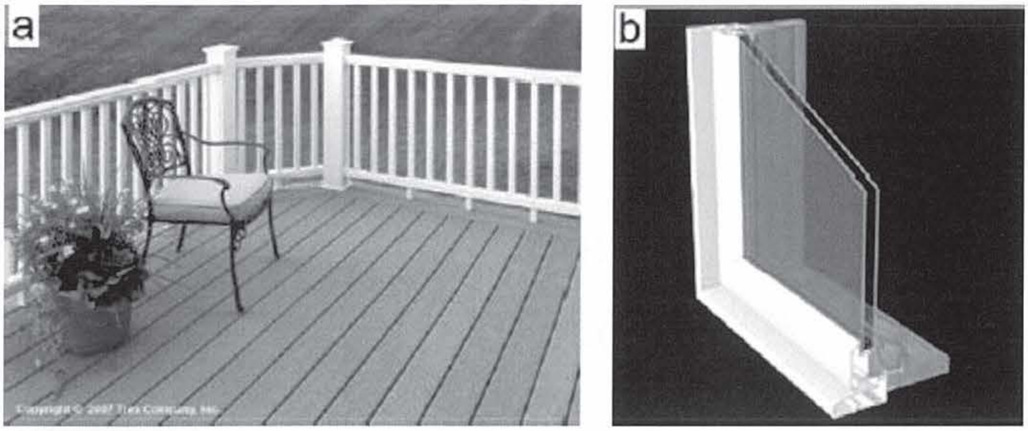


Figure 2.8: Application of wood plastic composites: (a) Decking (b) Window frame (Yeh & Gupta, 2008)

CHAPTER 3

METHODOLOGY

This chapter explained the proposed methodology of this research which consists of the principles of methods that will be performed to complete this research. The material selection, processing and testing will be presented as well after referring respectively to the specification and particular of previous research. The main point that will be highlighted in this methodology is suggesting the suitable methods, recommendation tools and the techniques to complete this research.

3.1 Introduction

The purpose of the methodology is to study the effect of freeze thaw cycle on the mechanical properties of rubber wood floor-plastic composite (WPCs). The procedures were set up according to the scopes of the research to achieve the objective set up. In addition, the ASTM D 790 was used as the benchmark of all procedures that involved standard tools, techniques and testing of the specimen. Besides, the setup of procedures was also based on previous researchers. The flow chart shown in Figure 3.1 describes the overall process that obtains in the result which includes the starting from material preparation, freeze thaw cycle process, testing method and analysis of data.

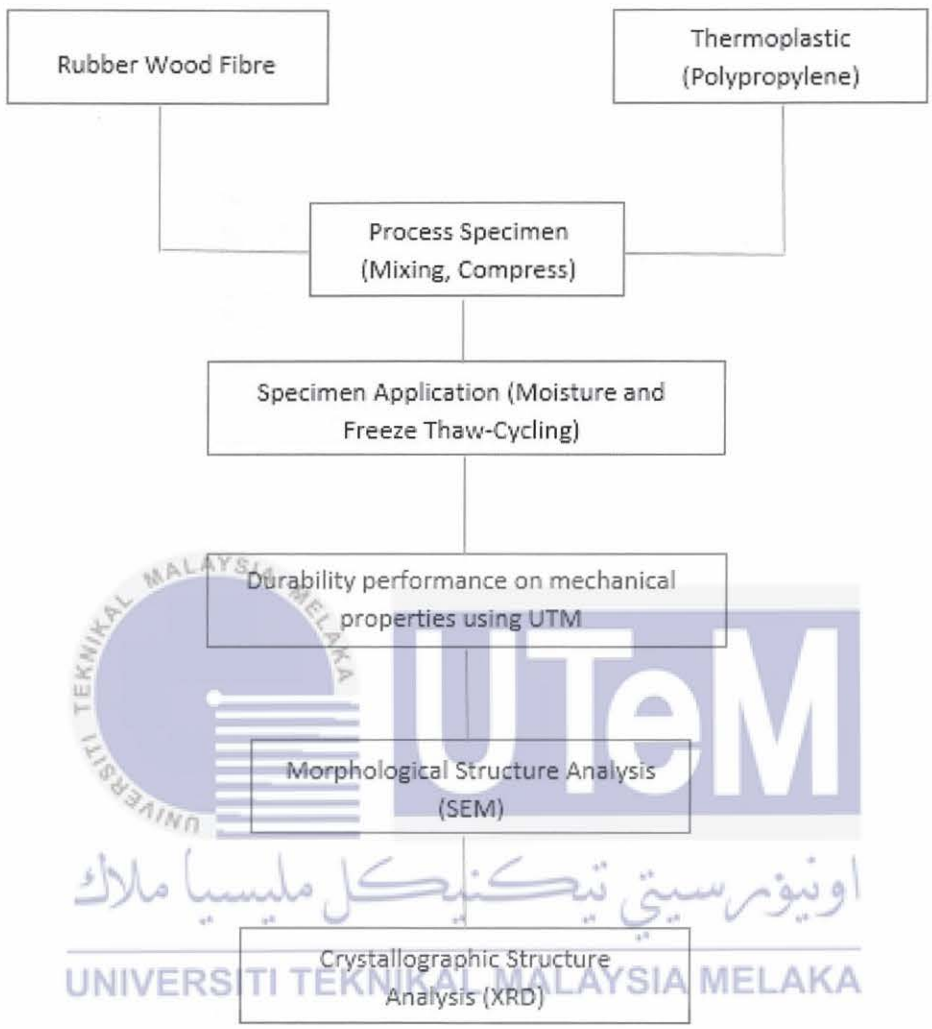


Figure 3.1: The process flow chart

3.2 Materials

The materials used in this stud is wood polymer composite consist of 50.3 wt.% of rPP, 37.5 wt.% of rubber wood flour, 7 wt.% of calcium carbonate 3.9 wt.% of MAPP, 0.2 wt.% of UV and 1 wt. % of lubricant. WPC granules were shown in Figure 3.2. The materials used were in a form of granules where their composition was optimized and extruded by Prince of Songkhla University, Thailand.

Figure 3.2: WPC granule

3.3 Methods

3.3.1 Preparation of Sample Preparation

Approximately 125g WPC granules were dried in drying oven for 24 hours and then compressed using hot press (Carver. USA) at 190 °C, pressure 870-1000 psi, for 30 min in the 200mm x 200mm x 3mm window frame mould and then water quench to room temperature afterward. Then, the plate of WPC was machined into test specimens according to the standards of ASTM D 790 for flexural as shown in Figure 3.3.

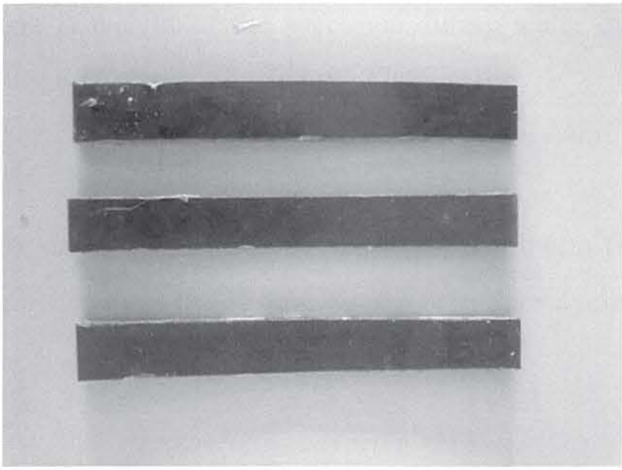


Figure 3.3: Specimen parameter for flexural testing (ASTM D790)

3.3.2 Freeze Thaw testing

In the study, by the standard EN 321, the resistance of the composites which used to moisture and freeze-thaw actions was tested under cyclic conditions specifically. The sample of WPC were exposed into three cycles which is each to comprising immerse in the water temperature at 23°C, freeze at the temperature at -20°C and dry at the temperature at 70°C. The flexural properties of the unaged and aged specimens were determine using flexural test.

Water absorption freeze-thaw (WFT) cycling of the composites was performed according to the ASTM D6662-01 (Figure 3.4). The cycling of WFT consists of three steps:

1. Soaking in a water bath at 27°C until the samples reach equilibrium moisture content. The sample weight is measure at certain interval and the percentage of water absorption is calculated using Equation 3.1

$$\% \text{ Water absorption} = \frac{Wt - Wo}{Wo} \, x \, 100\% \qquad (3.1)$$

2. Where, Wt is weight of the specimen during water immersion, Wo is initial weight (g)Freezing for 24hr in a freezer at -27°C

3. Thawing at 27° C and approximately 50% RH for 24hr.

All composites were exposed into four WFT cycles. Minimum five samples are used at each stage and will be mechanically tested after three freeze-thaw cycles.

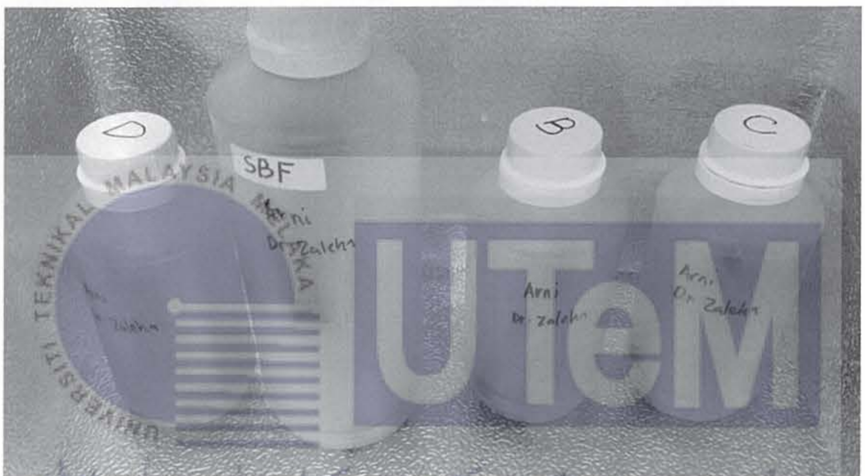


Figure 3.4: Freeze-thaw process

3.3.3 Mechanical Testing

In this study, the flexural test is used to characterize strength and stiffness of the non-aged and aging WPC by applying a load perpendicular to the specimen's longitudinal axis at a specified rate in the centre of the specimen (Figure 3.5). The flexural test was carried out according to the ASTM D790 using a Universal Tensile Machine (Shimadzhu AGS-X, Japan) which at a crosshead speed of testing of 2mm/min and fitted with a 20kN load cell. Five specimens were tested by each batch. However, the dimension of the sample was 74 mm x 12 mm x 3 mm (long x width x thickness). The specimens were placed on two supported stages and loads which were applied with a support span of 48 mm.

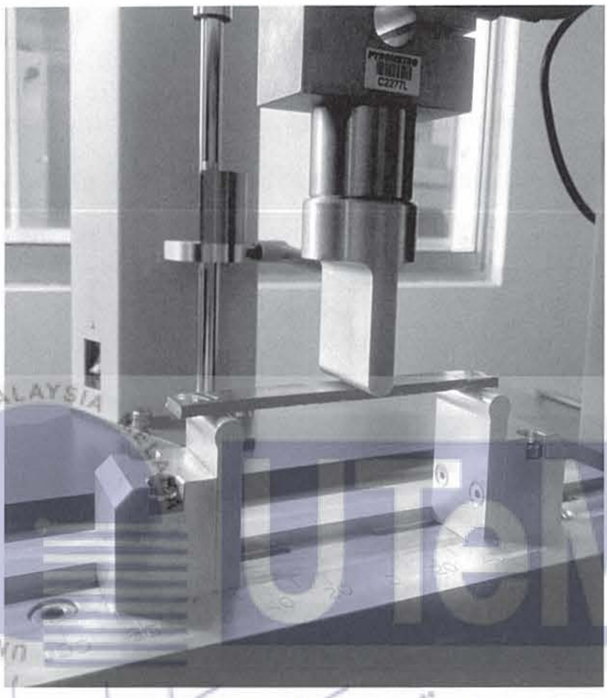


Figure 3.5: Flexural testing (ASTM D790)

The flexural strength is when now of break, the maximum stress in the outer region of the specimen occurred and then determined by calculate using Equation 3.2:

$$\text{Flexural Strength, } \sigma_F = \frac{3FL}{2bd^2} \tag{3.2}$$

Where σ_F is maximum flexural stress in unit MPa, where F is the load at the fracture point in N unit, L is a support span in unit mm, b and d is a width and depth of beam in unit mm.

The tangent modulus of elasticity was calculated by using the slope of a stress-strain graph using Equation 3.3:

$$\text{Flexural modulus, } E_F = \frac{L^3 m}{4bd^3} \qquad (3.3)$$

Where E_F is the modulus of elasticity in bending condition where the unit is in MPa, L is a support span in mm then m is a slope of the tangent to the initial straight-line portion of the load-deflection curve where the unit is N/mm.

3.4 Phase Analysis

X-ray diffraction (XRD) analysis of compounded samples was carried out with a Siemens D500 X-ray diffractometer with Cu Kα radiation. The diffractometer was equipped with a diffracted beam graphite monochromator, tuned to Cu Kα radiation, and a scintillation detector. Diffraction patterns were collected in reflection-mode geometry from 10° to 60° 2θ at a rate of 2° 2θ/min. The XRD samples were cut from the film samples with similar thickness of 10 mm. X-ray diffraction (XRD) tests were made with D/max2200 (Rigaku Corporation, Japan). The test parameters selected for the XRD included Cu butt, 0 kV of voltage, 30 mA of current, 4°/min rotating speed, and a 0.02° step distance.

3.5 Morphology Analysis

Microscopic were observed and carried out with an accelerating voltage of 10kV using a Carl Zeiss Sigma scanning electron microscope (SEM) (EVO 50, Carl Zeiss, Germany) as shown in Figure 3.6. For fracture surfaces, by using carbon cement, the specimens were mounted to the trunk. All samples were coated with carbon to avoid a static discharge effect before SEM examination. The fractured surfaces of composites were observed at 10-500x magnification.

Figure 3.6: Scanning Electron Microscope (SEM) machine

CHAPTER 4

RESULTS AND DISCUSSIONS

This chapter is mainly explaining the data collected after completing the sample fabrication and testing experiment. All the hypotheses and discussions will be supported by the previous research statement based on the effect of water absorption, flexural behaviour and properties, fracture surface analysis, mechanical and physical properties.

4.1 Overview

Moisture absorption increased with increased soaking time for all composites until saturation at about 7 days until no more weight gain. As no significant weight gain was found for WPCs during this period, it seems likely that moisture only penetrated the composites through the fibre matrix interface. The equilibrium moisture content decreased with an increased number of times the materials were reprocessed. The decrease in moisture content and diffusion coefficient with increased number of times the materials were reprocessed can be explained by several effects.

Flexural test was performed on sample before and after water immersion in distilled water at a room temperature. Figure shows the result of flexural behaviour respectively. For pure polypropylene, it is hydrophobic and absorbs very limited water and for wood flour reinforced polypropylene composite, the ability of water absorption is much better than pure polypropylene and the result exhibit their mechanical properties, the result show change in modulus and strength of flexural behaviour can be neglected. Generally, water absorption can degrade the mechanical properties of the composites. However, water molecules will lead to the change of structure for natural fibre, matrix, and interface between them, thus further degrading the mechanical properties of the composite. In this paper, the effect of the accelerated freeze thaw cycle towards mechanical and physical properties of WPCs was investigated.

4.2 Effect of Water Absorption on the Mechanical Properties

The moisture uptake of WPCs is an important consideration for the applications of WPCs including outdoor versus indoor use, environmental degradation, and load bearing properties. The water absorption of the composites panels was monitored by full water immersion over a period of 7 days (Figure 4.1). The result show that the composite absorbs the water rapidly in the first 96 hours before reach equilibrium at the end of 144 hours (7 days) of the immersion period. The composite is made of hydrophilic rubberwood fibre, in which very susceptible to moisture according to Butylina (2011).

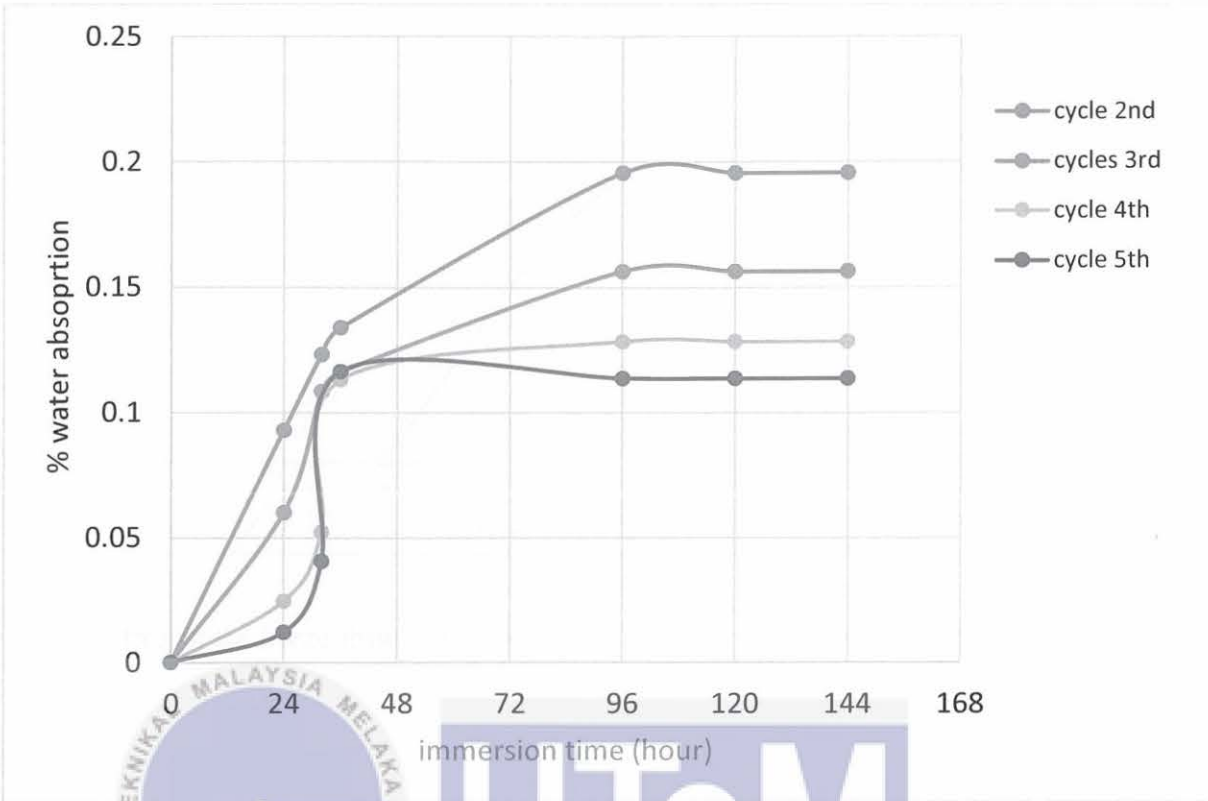


Figure 4.1: The percentage of water absorption with immersion time

In Figure 4.2 showed the percentage of the weight loss of the aged specimen after freeze thaw cycles. The composite has in significant weight loss in the first two cycles of the freeze thaw process. However, as the period of the freeze thaw increased, a significant weight loss was observed , in which 16% for the 5th freeze-thaw cycle. This is possible due the breakdown of the rubberwood phase during the water immersion according to Mak & Fam (2019), water loss of WPCs is plotted versus weight loss from day 1 until day 7 (24 h per day).

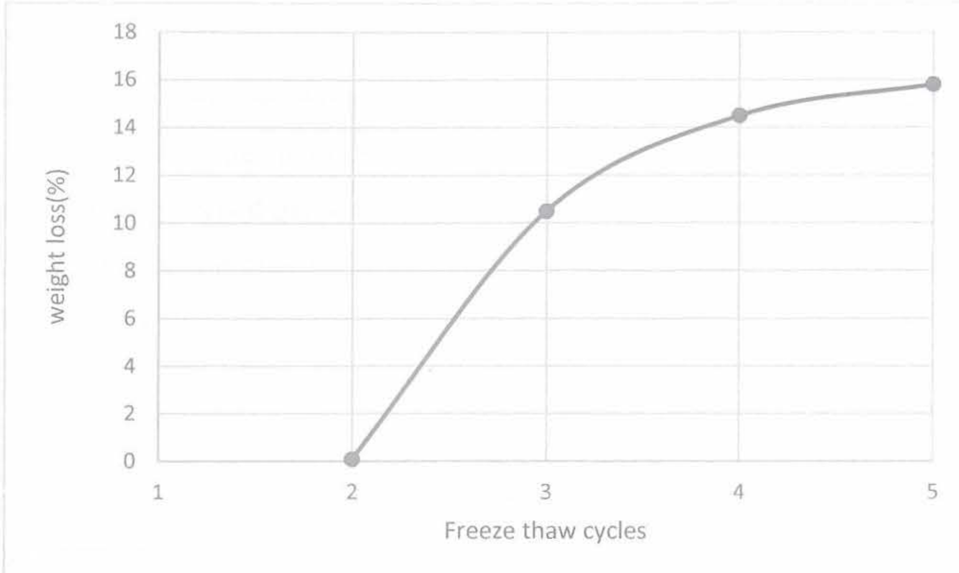


Figure 4.2 : Effect of the freeze-thaw on the weight loss of rubberwood/recycled PP composite

4.3 Effect of Freeze Thaw Cycle on the Mechanical Properties

The effect of water immersion-freeze-thaw actions on the flexural properties is shown is shown in Figure 4.3 till 4.5. When the effects of freeze–thaw cycles on the flexural strength of the composite polymer were investigated it was seen in this figure, as the number of freezes–thaw cycles increased, the flexural strengths of the sample decreased. However, the fibre reinforced polypropylene has a better comparative performance than non-fibrous one. In addition, it was observed that the fibre reinforced samples, which had less flexural strength than the control sample prior to freeze–thaw cycles, showed higher flexural strength than the control sample after freeze–thaw cycles (Çavdar, 2014).

Moisture absorption has shown to have a primary impact on the properties of weathered composites. Absorbed moisture degrades interfacial interaction in the composite, reducing stress transfer and hence, lowering the mechanical properties. Also, presence of water causes wood particles swelling that leads to stress in matrix and micro cracking formation, thus reducing their mechanical properties. Similar finding has been reported by Nafchi (2015).

Micro cracks then contribute to further water ingress into the composite at the later exposure. The effect of water is often irreversible, and the properties of the materials are not recovered after drying. Wood particles encapsulated into the matrix are less accessible to water which makes the material properties less dependent on the ambient humid conditions according to Golmakani (2021).

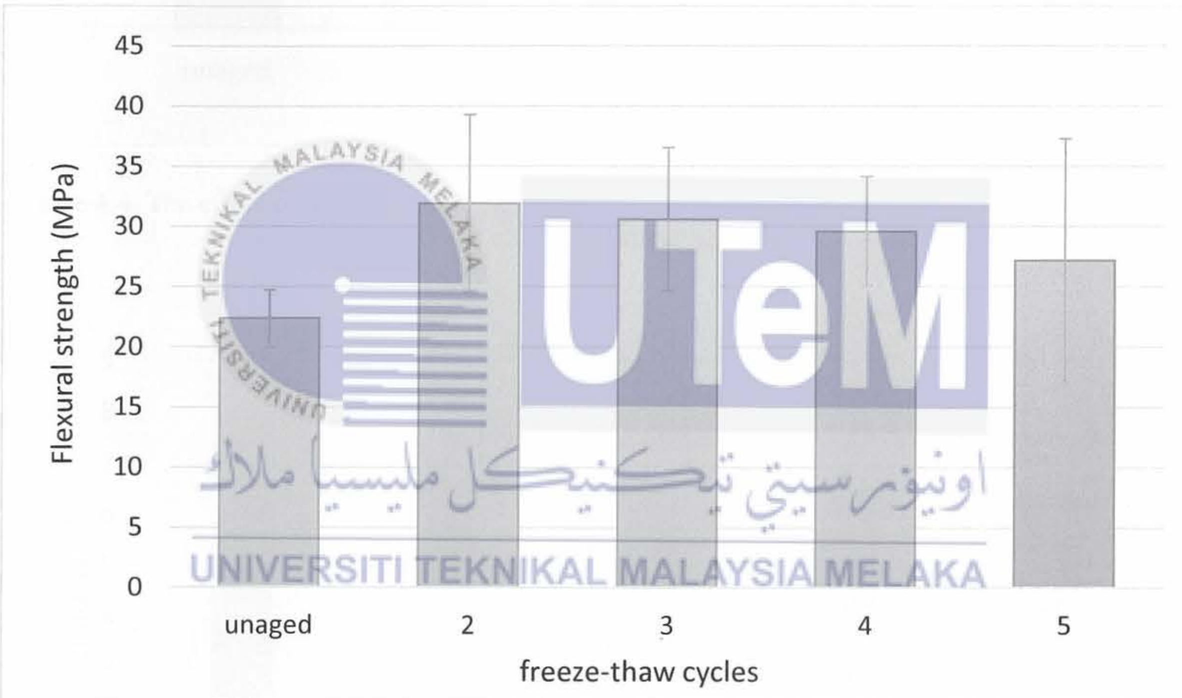


Figure 4.3 The effect of feeze-thaw cycles on the flexural strenght of the rubberwood/recycled PP composite

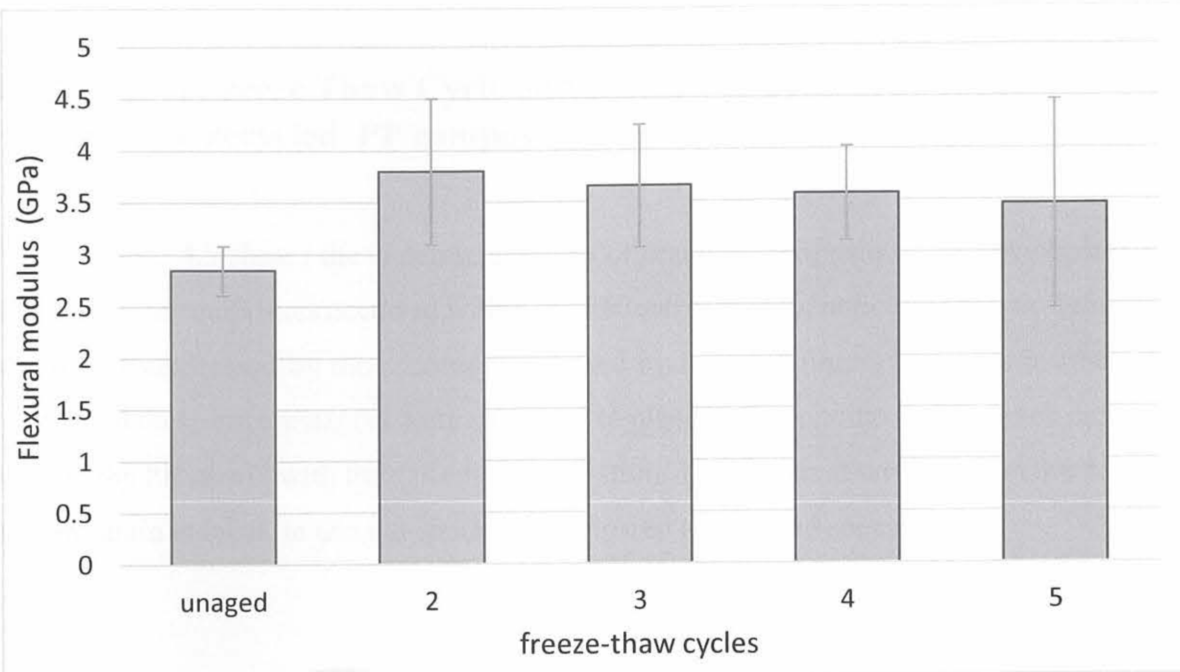


Figure 4.4 The effect of feeze-thaw cycles on the flexural modulus of the rubberwood/recycled PP composite

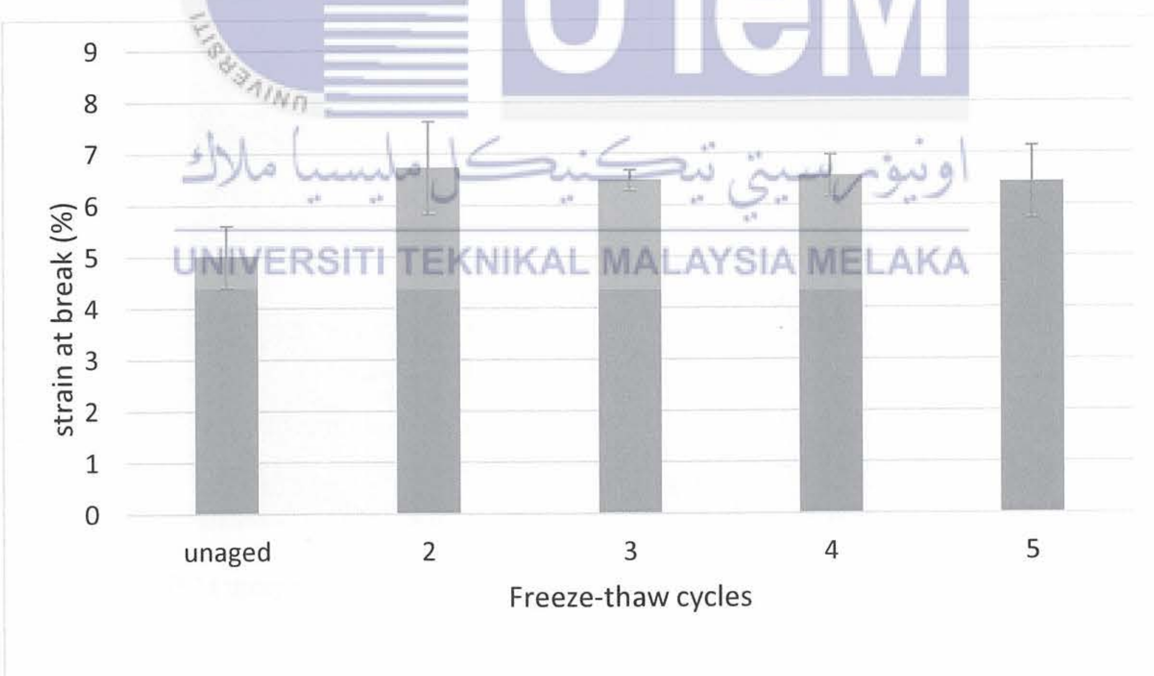


Figure 4.4 The effect of feeze-thaw cycles on the ductility of the rubberwood/recycled PP composite

4.4 Effect of Freeze Thaw Cycle on the fracture behaviour of the rubberwood/ recycled PP composite

Figure 4.5 shows the fracture surfaces of unaged composite. It can be observed that fracture of the composites occurred within the PP matrix without noticeable plastic deformation which was suppressed by the constraint imposed by the rigid fibres. Materials in front of the crack tip in these circumstances were subjected to plane strain conditions. The crack propagated through the PP matrix with little plastic deformation. This observation supported the finding of the low strain at break in unaged specimen compared to the aged specimens.

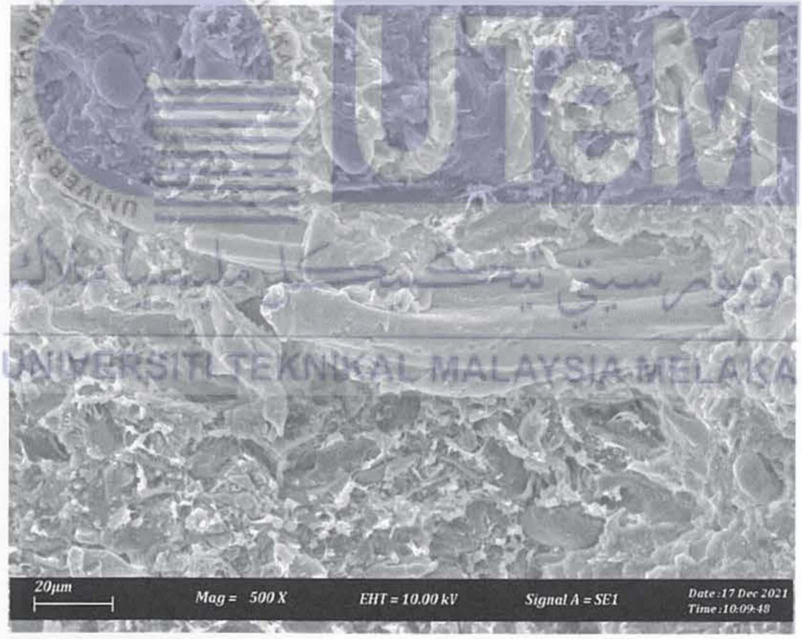
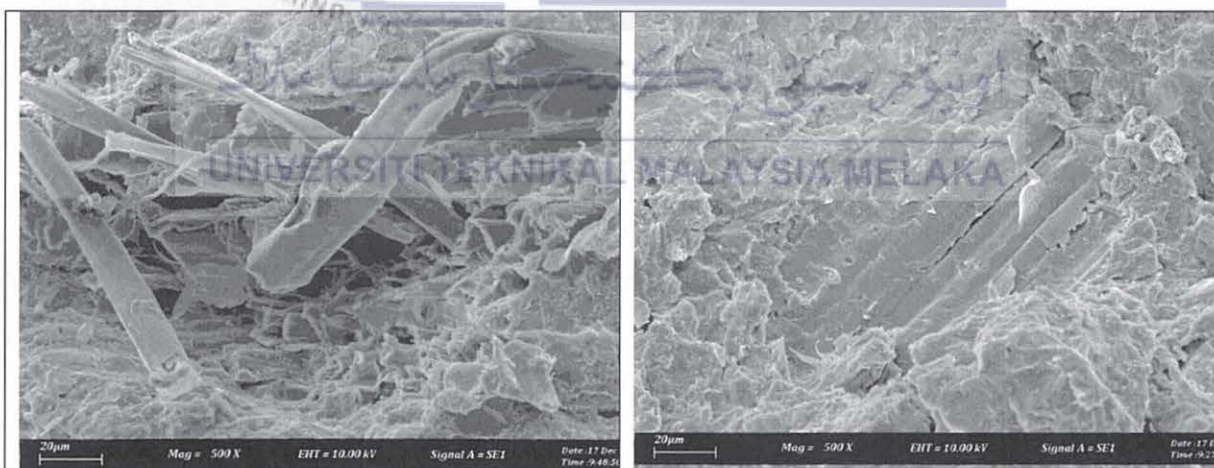


Figure 4.5 SEM morphology of flexural fracture surface of unaged WPC, magnification 500x

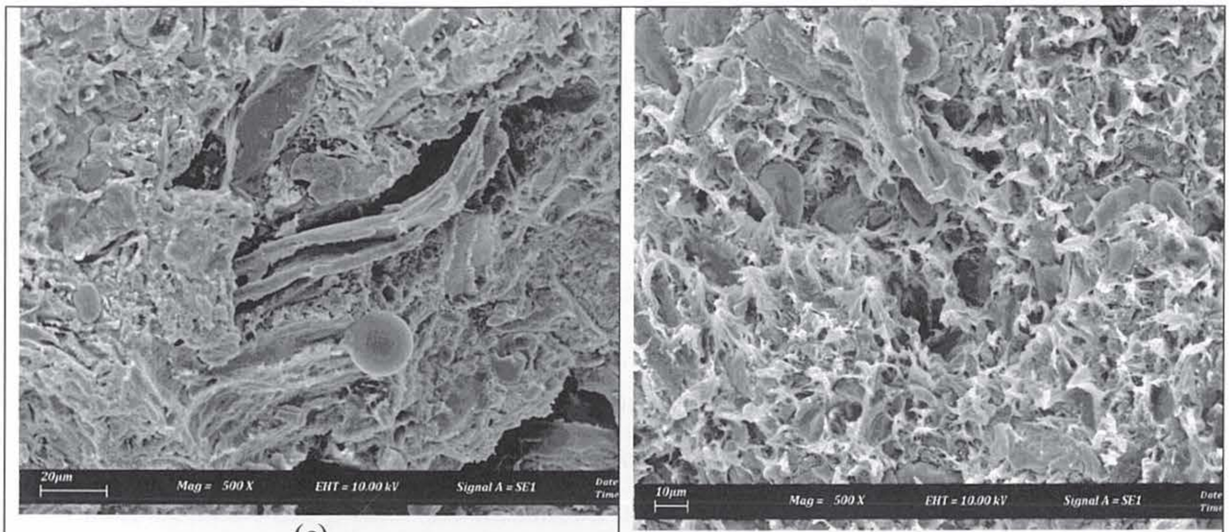
Figure 4. 6 showed the fractured surface of the aged specimens at different freeze-thaw cycles. In general, the composite fractured with more ductile manner, indicated by the rough

surface of the polypropylene. This supported the finding of increase if strain at break with the present of water immersion in Figure 4.4. It also can be observed that the wood flour fibres were much more distinguishable and fracture in with drawn out from the matrix, suggesting better interfacial adhesion. As a result of improved adhesion, fibres on the crack path were split into two pieces rather than being deboned and pulled out from the matrix. This finding is supported by the previous study according to Lee (2021).

When exposed to moisture, the composite with a high wood flour content has strong mechanical characteristics and increases water absorption. Furthermore, the inclusion of filler components in a finely and consistently distributed manner in the composite with a stable morphology. Consequently, the interface of wood flour, polymeric matrix, and chemical content occurred, resulting in a very fine homogeneous morphology and enhanced mechanical and physical characteristics of the sample according Alavi (2014).



(a) (b)



(c) (d)

Figure 4.6 SEM morphology of: WPC (b) after 2 cycle of freeze thaw, WPC (c) after 3 cycle of freeze thaw, WPC (d) after 4 cycle of freeze thaw, WPC (e) after 5 cycle of freeze thaw with magnification 500x.

The results reveal that the border between the wood and polymer may be the strongest section of the WPC structure, and that freeze–thaw damage, such as aggregate fallout, softening, and cracking, mostly does not occur in this region. In this work, microstructural examinations of the surface of WPCs specimens were made after 28 cycles of freeze–thaw testing, which found microspores, freeze–thaw fractures, and hydrates of WPCs (Figure). The results of freeze–thaw cycles revealed highly dense microstructures with many microspores and micro fractures, as well as quite broad (2m) cracks. Thus, SEM data show that substantial damages occurred because of freeze–thaw cycles.

4.5 X-ray diffraction (XRD) Analysis

Analysis of the X-ray diffraction patterns indicated that the characteristics of the WPCs with treatments were similar. However, the XRD peak of the WPCs were shown in Figure 4.7.

As shown in the XRD pattern, the maximum peak values of WPCs for side in general, the 2θ diffraction patterns for the five samples were similar, although there was little difference in the intensity which represents the difference in the relative crystallinity value.

Glass Transition Temperature (Tg) is the point at which a material alters state which is from a glass-like rigid solid to a more flexible. Tg for polymer is 170K-500K (-103.1°C until 226.85°C). From the figure, the peak of polymer in figure were crystallize. It is because the value of Tg for polymer was also depends on the tacticity of the polymer. The greater the order in a macromolecule the greater the likelihood of the molecule to undergo crystallization. For example, isotactic polypropylene is usually more crystalline than syndiotactic polypropylene, and atactic polypropylene is considered uncrystallisable since the structure of the polymer chain lacks any regularity. In fact, most atactic polymers do not crystallize. The high crystallinity and strong intermolecular interactions also greatly increase the mechanical strength.

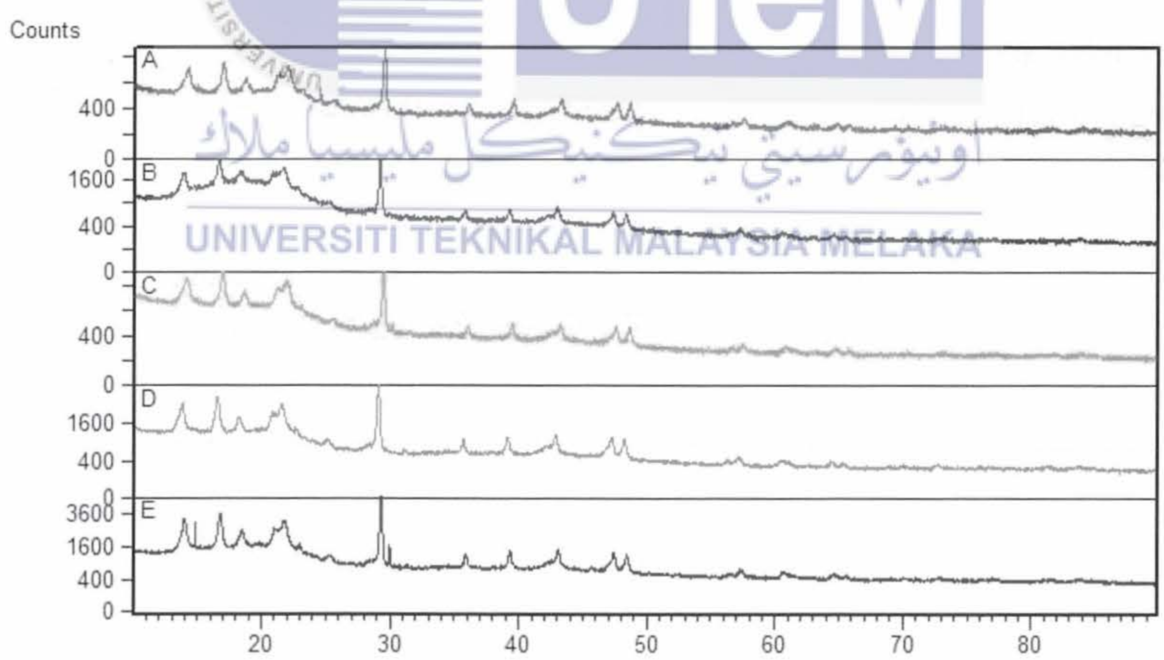


Figure 4.7: XRD pattern that indicated the characteristics of the WPCs. (A) = unaged, B = 2nd cycles, C = 3rd cycle, D = 4th cycle, E = 5th cycle

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The first objective of this research is to study the effect of freeze thaw cycle onto the mechanical properties of the rubber wood reinforced recycled polypropylene. The findings showed that:

(a) Flexural strength and modulus decrease with the increase of the freeze thaw cycles. The optimum flexural strength and modulus are 33 MPa and 3.8 GPa , given by 2 cycles.

(b) The strain at break increase with the present of the moisture from 5% (Unaged) to 6.5% (5 cycle freeze thaw), suggesting that the present of the water molecule induced the plasticizing effect in the composite .

(c) The phase analysis showed that the increasing number of freeze thaw cycles lead to the change of flexural strength of the sample, thus the modulus of the sample. However, ductility is increased.

Within the second objective of this research is to correlate the effect of freeze thaw cycle onto the fracture surface of rubber wood reinforced recycled polypropylene to freeze thaw cycles. The conclusions as follows:

(a) The unaged specimen fractured with brittle fracture of the WPCs, induced in the tension section of the composite.

(b) The only poor interface bonding was caused by the void appearing at the interface zone. The crack propagation has propagated through the void which will lower the mechanical strength.

(c) The present of the water during the freeze-thaw process cause the composite to fracture in more ductile manner with rough matrix surface and drawn of the rubberwood fibre.

5.2 Recommendations

After going through this research, there are few recommendations suggested so be able to call attention to the important aspect that needs to be focused on for the coming research topic about rubber wood floor reinforced recycled polypropylene in the future. The significant recommendations for this objective as follow:

a) The impact test is suggested for the coming research topic on WPCs to verify the mechanical properties of WPCs in facing the impact force like a boat's body will bump with rock when it reaches the shore that will crush on the hard surface of the ground.

b) The interfacial bonding strength or adhesion strength can be determined more accurately through the Z-direction test which is able to clarify the interfacial bonding of composite and polymer by reverse force applied at both surfaces.

c) For instance, studies an investigation into the effect of freeze-thaw cycling under sustained load is recommended. Also, test on different type of composite mixture with other material.

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