The effect of hybrid filler loading on mechanical and physical properties of Epoxy Bio-Composite

by

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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in partial fulfillment of the requirement for the
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(MECHANICAL ENGINEERING)

Approved by,

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(AP Dr.Puteri Sri Melor binti Megat Yusoff)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2012

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project. The original work is my own except as specified in the references and acknowledgment, and that the original work contained herein have not been undertaken or done by unspecified sources of persons.

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MUGILAN MALAI RAJAN

ABSTRACT

The aim of study was to investigate the mechanical and physical properties of natural hybrid fillers reinforced epoxy composite. Hybrids fillers used in this study were kenaf bast fibers and rice husk. Particulate rice husks were treated with 10% NaOH solution while kenaf bast fibers were treated with 3 wt% of vinyltriethoxysilane in order to improve the adhesion bonding between natural fillers and epoxy matrix. The fillers and matrix were prepared at 5 levels of fillers ratio between kenaf bast fibers and rice husks (10:0, 10:10, 5:15, 3:17 and 0:40). All composites were fabricated using hand lay-up techniques. Mechanical and physical performance of hybrid composites were evaluated and studied. The results revealed that tensile strength of hybrid composite increased from 9.54 MPa to 12.91 MPa while flexural strength of hybrid composites showed increment from 10.86 MPa to 13.11 MPa as kenaf bast fiber loading increased in the hybrid composition. However pure epoxy exhibited higher tensile and flexural strength, 33.44 MPa and 27.75 MPa, respectively compared to those of the hybrid composites. This was mainly due to weak adhesion bonding between hybrid fillers and matrix, agglomeration of kenaf bast fibers and irregularity of shapes and size of rice husks particulates. Water absorption results revealed that hybrid composites water uptake decreased as the rice husks particulates loading increased. This was mainly due to rice husks have rich content of silica which increased the moisture resistance. Kenaf bast fiber reinforced epoxy bio-composite has the highest water intake due to its hydrophilic characteristics.

Key words: Kenaf bast fiber, Rice husks, Epoxy, Mechanical properties, Water absorption.

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LIST OF ABBREVIATIONS

RH	Rice Husks
KBF	Kenaf bast fibers
PP	Polyproplyene
VTS	Vinlytriethoxysilane
NaOH	Sodium Hydroxide
ASTM	American Standard of Testing and Material
ISO	International Organization of Standardization

NOMENCLATURES

℃	Degree Celcius
σ_t	Tensile Strength
σ_f	Flexural Strength
L	Length
b	Width
D	Depth or Thickness
g	Gram
P	Load
μm	Micrometer
m	Meter
min	Minute
mm	Millimeter
mm^3	Cubic millimeter
cm^3	Cubic centimeter
MPa	Megapascal
GPa	Gigapascal
wt%	Weight Percent

CHAPTER 1

INTRODUCTION

1.1 Background

Natural fibers reinforced composite or known as bio-composite has been researched extensively for past few years [1-3]. Polymer bio-composite is made from single natural filler and a polymer based matrix. Natural fibers act as reinforcing agent where it functions is to strengthen the mechanical and physical properties of the polymer matrix. The function of polymer is to bind the fibers together and distribute the external loads of the composite among the fibers. There are two categories of polymer: thermoplastic and thermosets. Thermoplastic is type of polymer that softens when heated and hardens when cooled. It can undergo the same cycle repetitively without damaging its molecular structure. Thermoset is a type of polymer that softens when heated but hardens permanently when cooled.

Thermoset polymer is widely used as adhesives in many industries such as manufacturing, aerospace, automotive, construction and other industries. Synthetic fillers are vastly used in order to strengthen mechanical and physical properties of thermosets. However, synthetic fillers are hazardous to the environment and release carbon dioxide to atmosphere during burning and lead to greenhouse effect [4]. Moreover cost of synthetic filler is rising due to limitation of raw material. Types of synthetic filler used in industry are glass fibers, polyolefins, polyester and polyvinyl choloride.

Due to serious drawback of synthetic fillers with regards to environmental issues, natural fibers reinforced composite has attracted many researchers as alternative to replace synthetic composites. Natural fillers specifically lignocellulose fillers have significant advantages over synthetic fillers. The advantages include high specific strength, low density, biodegradable and large availability resulting in low cost of composite product. In addition, natural fillers are non-abrasive to the processing

equipment resulting in longer life span of equipment [5-10]. Due to these lucrative advantages offered by lignocellulose material, various natural fillers mainly from wood, cotton, flax, jute and many others have been researched and used in reinforcing thermoset.

Hybrid bio-composite is a combination of 2 or more different type of natural fillers in matrix composition [10-11]. These hybrid fillers offer range of properties that cannot be offered by single natural reinforcement. The combination of hybrid fillers such as palm oil leaf, kenaf, hemp and other lignocellulose fibers with polymer matrices to enhance bio-composite properties that are competitive with synthetic composite is gaining attention. In this research, kenaf bast fiber and rice husk have been selected based on the high content of cellulose and lignin in the fillers, respectively and both fibers are largely available in Malaysia.

Malaysia is a rice producer country. Rice husk shown in Figure 1-1 remains one of the largest agro wastes in the country. Every 1000 kg of rice grain harvested, there will be 200 kg by-product of rice husks [6]. Rice husks are composed of cellulose (35%), hemicellulose (25%), lignin (20%), and ash (17% which is 94% silica) and moisture (3%) [12]. Most of the rice husk is burnt in large quantities which increase air pollution and global warming. Therefore, the opportunity to utilize the agro waste can be advantageous to the environment, economy and technology.

Figure 1-1: Rice husks

Hisbiscus cannabinus or known as kenaf as shown in Figure 1-2, is commercially grown in Malaysia due to its vast advantages compared to other conventional crops such as jute and hemp. It is 100% biodegradable and recyclable fibers. In ancient

times, people have utilizes kenaf in many ways such as making ropes and canvas. Nowadays, kenaf is used as packaging material, insulation and paper production. Kenaf required less amount of time to mature with minimum of fertilizer and water compared to other conventional crops. Kenaf contains (60-80%) cellulose, (5-20%) lignin and up to (20%) moisture [13]. Kenaf bast fiber has the potential as a reinforcing agent for thermoset polymer due to its superior toughness, high tensile strength and high content of cellulose compared to other natural fillers [13].

Figure 1-2: Kenaf plants

1.2 Problem Statement

Bio-Composites have been researched throughout past years using different types of individual natural fillers as reinforcing agent such as jute, hemp, sisal, and coconut husk resulting in improved mechanical properties of the composites [1-3]. The strength of bio-composites differs from each other due to types of fibers used and their chemical composition of fibers which includes content of cellulose, lignin and hemicellulose. However, the most significant drawback in bio-composite is the weak adhesion bonding between fillers and polymer matrix [2-4, 8-10, 14]. The presence of hydrophilic lignocellulose and hydrophobic matrix in the composite have weakened the adhesion bonding between two elements resulting in poor mechanical and physical strength compared to synthetic composite.

Coupling agent [8, 10, 14] is introduced in order to overcome this problem. The role of coupling agents is to treat fibers individually before they are blended with polymer in order to further improve the interfacial bonding between fillers and matrix. Improved interfacial bonding will then result in enhanced mechanical and

physical properties of bio-composite. Even with chemical treatment of the natural fibers, bio-composites are still inferior to those of synthetic fiber composites.

In the attempt to further improve the properties of bio-composites, hybrid bio-composite is introduced recently. Hybrid bio-composite can be defined as a composite with two or more types of natural fillers as reinforcement. Hybrid bio-composite offers more advantages instead of using single type natural filler as reinforcing agent [10-11]. However, more research on hybrid bio-composite is required particularly its influence on mechanical and physical properties. In this research, hybrid bio-composite is investigated using kenaf bast fiber and rice husk flour. Kenaf and rice husks have high content of cellulose and lignin, respectively. Cellulose provides the basic structure of fiber and strength while lignin provides elongation to the fiber. Rice husk and kenaf bast fiber are chosen in this study due to high contents of cellulose and lignin.

1.3 Objective

This research is to investigate the effect of incorporating varying levels of hybrid filler on mechanical and physical properties of epoxy bio-composites. The fillers used were kenaf bast fibers and rice husk in the composition of the hybrid fillers will be varied accordingly.

1.4 Scope of study

The objective of the study was to investigate the mechanical and physical properties of hybrid filler reinforced epoxy composite in terms of tensile strength, flexural strength and water absorption. In the sample preparation, 5 levels of hybrid filler ratio of kenaf bast fiber and rice husks (10:0, 10:10, 5:15, 3:17, and 0:40 wt%) were performed. 3 wt% of silane as coupling agent was applied to kenaf bast fiber while 10% NaOH solution was applied to rice husks particulate in order to improve the adhesion bonding between kenaf bast fibre and matrix. Hand lay-up technique was applied to compound the thermoset and fillers. Mechanical and physical properties of hybrid bio composite in terms of tensile strength, flexural strength and water absorption were studied.

CHAPTER 2

THEORY AND LITERATURE REVIEW

2.1 Theory

2.1.1 Composite

A composite material is artificially made from two or more materials which have different chemical and physical properties [15-16]. Composite composes of two phases; binder or known as matrix and reinforcement. Reinforcement provides strength and rigidity to enhance the mechanical and physical properties of matrix meanwhile, the function of matrix is to maintain and hold the orientation of reinforcement fibers and helps to transfer loads among the reinforcement fibers [15].

Reinforcing fillers consist of two main categories; synthetic and natural fillers. Figure 2-1 shows the classification of reinforcing fibers. Common synthetic fillers used in composites are carbon, glass, and aramid. Synthetic composites are used in various industries such as aerospace, automobile and sporting goods due to their high tensile strength, high temperature and chemical resistance.

Reinforcement fillers can also be classified into two types; fiber (discontinuous and continuous) and particle (variety of geometries and sizes). The discontinuous and continuous fibers are differentiated by the critical length of fiber. Critical length affects the strength and stiffness of composite material. For particulate fillers, they are required to be small and evenly distributed throughout matrix in order to increase its composite strength [15].

Polymer matrix composites are greatly used in composite applications compared to other types of matrixes such as carbon, metallic and ceramic [16]. The polymer matrix is widely applied due to ease of fabrication, cost effective and able to be produced large quantities. Polymer-matrix composite is made from a polymer resin with fibers as reinforcement. The matrix also provides protection to the fibers from

chemical reaction with environment and mechanical abrasion. Failure in composite occurs when large amount of fibers failed to absorb the load. Therefore it is important that the adhesive bonding between fiber and matrix is strong.

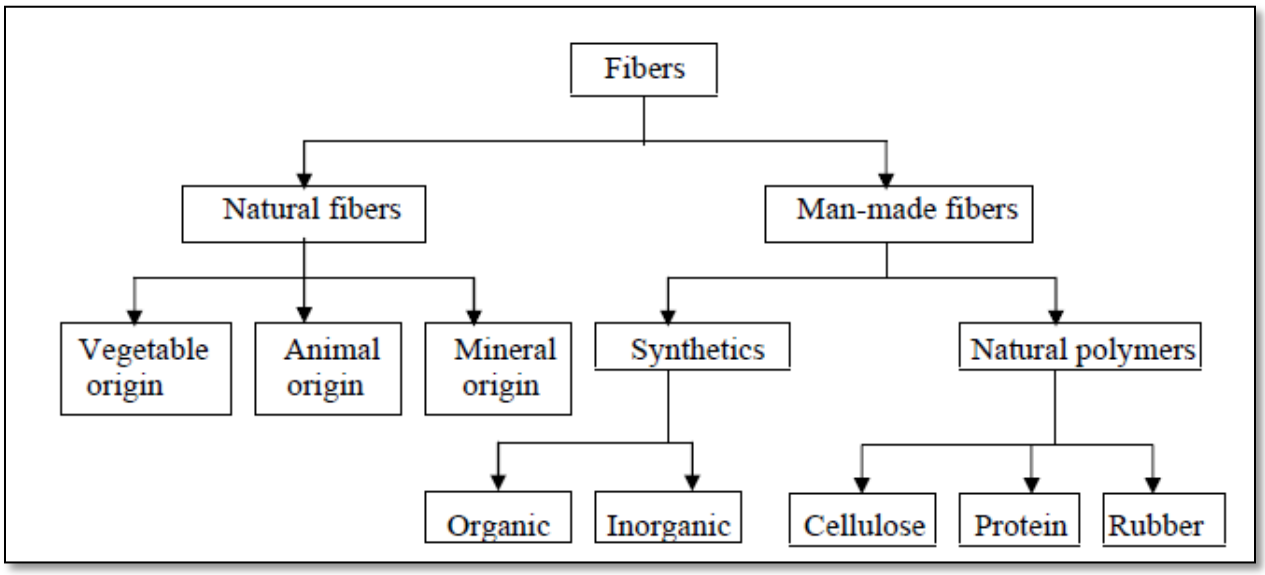


Figure 2-1 Classification of fibers [15]

2.1.2 Polymer

Polymer can be defined as an organic material with repeating molecular units [16]. Polymer is categorized into two types based on its behavior towards elevated temperature: thermoplastic and thermosetting.

Thermoplastic is a type of a polymer that is able to re-melt and re-mould for many cycles without damaging its molecules structures [17]. Common thermoplastics materials are polyethylene, polypropylene, polystyrene and polyvinyl chloride. Thermoplastics consist of long and flexible chain with some degree of braches structures. During moulding process, thermoplastic is heated, moulded and cooled to shapes. The molecules tend to be aligned to each other and create a secondary bonding force between them. During melting process, the heat energized the molecules and breaks the bonding forces between the molecular chains. The thermoplastic eventually softens. Figure 2-2 shows the molecular structure of a thermoplastic.

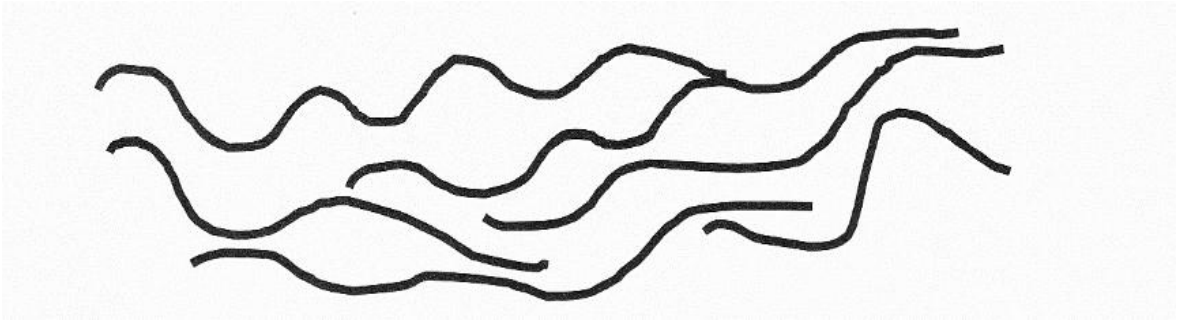


Figure 2-2: Schematic structure of thermoplastics [17]

The advantages of thermoplastics are thermoplastics can be used in welding due to its softening characteristics when heat is applied. Besides that, the process cycle of thermoplastic is short due to the absence of crosslinking structure. The wastes of thermoplastics can be recyclable due to its reversible property upon heating. In addition, the toxicity level of thermoplastics are low due absence of reactive chemical in its molecules structures [17].

The disadvantages of thermoplastics are its modulus retention is decreases as temperature increases compared to thermoset due to absence of crosslinks between molecules. The tooling systems of moulding thermoplastics are costly due to it has to withstand high temperature and pressure [17].

Thermoset is a type of polymer that irreversibly cures and once the thermoset is hardened it cannot be reheated and melted to its initial form [17]. In terms of molecular structure, before thermoset hardens, the molecules are independent macromolecules similar to thermoplastics. However during the final state of thermoset forms a three-dimensional network structure due to cross-linking process. During cross-linking process, the molecules combined with each other and form larger molecules resulting in high melting temperature of the thermoset. Even if the thermoset is reheated after its final state, it will reach the decomposition temperature before melting point temperature is reached. Figure 2-3 shows the thermoset before crosslinking and after crosslinking. Common thermosets used in industries are polyester, urea, and epoxy.

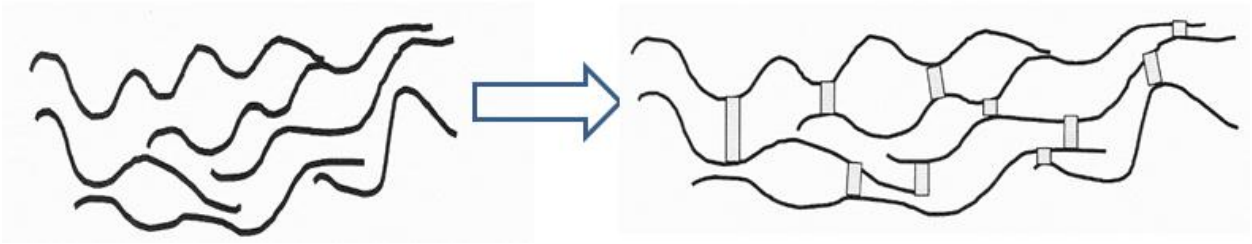


Figure 2-3: Thermosets before crosslinking and after crosslinking [17]

The advantages of thermoset are thermosets have higher modulus retention due to the presence of the three dimensional structures. Thermosets have exhibit better creep behavior than thermoplastics due to restricted movement of macromolecules by the links between chains. In addition, thermosets fabrications are simple and low-cost tooling system are required [17].

The disadvantages of thermosets are thermosets have longer natural curing time which results in low production compared to thermoplastics. Furthermore, the waste of thermosets cannot be recycled due to the irreversibility of thermosets.

2.1.3 Natural Filler

Natural filler, specifically, lignocellulose materials are greatly emphasized as reinforcing filler in polymers. Natural fillers have been used in many industrial applications due to their advantages over synthetic fillers. Natural fillers are environmental friendly, biodegradable, largely available, low in density and high in modulus strength [5-10]. Furthermore, the biodegradability of plant fibers is able to improve the ecosystem of the environment. Table 2-1 shows the annual production of natural plant fibers in the world.

Lignocellulose materials are made from cellulose, lignin and hemicelluloses. Cellulose provides the basic structure of the cell wall and strength of the fiber. The role of hemicellulose is to form as supporting matrix for cellulose. Lignin acts as a binder to cellulose and hemicellulose. It also provides elongation to the fiber. The lignocellulose materials are hydrophilic due to the presence of large amount of OH group. Each type of natural fillers has different mechanical properties due to

difference in chemical and structural composition, fiber types and growth condition. Table 2-2 and 2-3 show the mechanical properties and chemical composition of natural fibers and conventional reinforced fibers.

Table 2-1: Annual production of natural plant fibers in the world [10]

Fiber Source	World Production 10^3 Tons	Origins	Fiber Source	World Production 10^3 Tons	Origin
Abaca	70	Leaf	Nettles	Abundant	Stem
Bamboo	10,000	Stem	Oil Palm Fruit	Abundant	Fruit
Banana	200	Stem	Palm rah	Abundant	Stem
Broom	Abundant	Stem	Ramie	100	Stem
Coir	100	Fruit	Rosselle	250	Stem
Cotton Lint	18,500	Stem	Rice Husk	Abundant	Fruit/Grain
Elephant Grass	Abundant	Stem	Rice Straw	Abundant	Stem
Flax	810	Stem	Sisal	380	Stem
Hemp	215	Stem	Sun Hemp	70	Stem
Jute	2500	Stem	Wheat Straw	Abundant	Stem
Kenaf	770	Stem	Wood	1,750,000	Stem
Linseed	Abundant	Fruit			

Table 2-2: Chemical composition of natural plant fibers [10]

Fiber	Cellulose (Wt%)	Hemicelluloses (Wt%)	Lignin (Wt%)	Pectin (Wt%)	Moisture Content (Wt%)	Waxes	Microfibrillar Angle (Deg)
Flax	71	18.6-20.6	2.2	2.3	8-12	1.7	5-10
Hemp	70-74	17.9-22.4	3.7-5.7	0.9	6.2-12	0.8	-
Jute	61.1-71.5	13.6-20.4	12-13	0.2	12.5-13.7	0.5	8
Kenaf	45-57	21.5	8-13	3-5	-	-	-
Rice Husks	35	25	20	-	3	-	-
Ramie	68.6-76.2	13.1-16.7	0.6-0.7	1.9	7.5-17	0.3	7.5
Nettle	86	-	-	-	11-17	-	-
Sisal	66-78	10-14	10-14	10	10-22	2	10-22
Henequen	77.6	4-8	13.1	-		-	-
PALF	70-82	-	5-12.7	-	11.8	-	14
Banana	63-64	10	5	-	10-12	-	-
Abaca	56-63	-	12-13	1	5-10	-	-
Oil palm EFB	65	-	19	-	-	-	42
Cotton	85-90	5.7	-	0-1	7.85-8.5	0.6	-
Coir	32-43	0.15-0.25	40-45	3-4	8	-	30-49
Cereal Straw	38-45	15-31	12-20	8	-	-	-

Table 2-3: Mechanical properties of natural plant fibers [10]

Fiber	Density (g/cm^3)	Elongation (%)	Tensile Strength (MPa)	Young Modulus (GPa)
Cotton	1.5-1.6	7.0-8.0	287-597	5.5-12.6
Jute	1.3	1.5-1.8	393-773	26.5
Flax	1.5	2.7-3.2	345-1035	27.6
Hemp	-	1.6	690	-
Ramie	-	3.6-3.8	400-938	61.4-128
Sisal	1.5	2.0-2.5	511-635	9.4-22.0
Coir	1.2	30.0	175	4.0-6.0
Viscose (cord)	-	11.4	593	11.0
Soft Wood Kraft	1.5	-	1000	40.0
E-glass	2.5	2.5	2000-3500	70.0
S-glass	2.5	2.8	4570	86.0
Aramid (normal)	1.4	3.3-3.7	3000-3150	63.0-67.0
Carbon (standard)	1.4	1.4-1.8	4000	230-240

2.1.4 Silane Treatment

The presence of large amount of hydroxyl group (OH) in natural fibers provides poor compatibility with the non-polar polymer such as polypropylene and HDPE [2-4, 8-10, 14]. Hydrogen bonds may form between hydrophilic fibers and result in agglomeration of natural fibers. In addition, lack of wetting between fibers by matrix causes weak interfacial adhesion. This results in reduction of the composite strength. Therefore, fiber treatment is important in order to increase the interfacial bonding between natural fibers and polymer matrix which will increases the mechanical and physical properties of composite. Silane coupling agent is one of the technologies used to treat fibers.

The chemical structure of silane coupling agent is $R_{(4-n)}$-Si-(R'X_n) where R is an alkoxy, X is an organofunctionality that reacts with natural fiber surface and R' is an alkyl bridge that connects the silicon atom and the organofunctionality [14,18]. Silane coupling molecules are bifunctional whereby it forms a bridge between natural fibers and polymer while its organofunctional group bonds with polymer resin. Figure 2-4 shows hydrolysis process of the silane coupling. During hydrolysis process, the silane monomer is hydrolyzed and produces a reactive silanol group with the presence of water and catalyst (acid or amino base) [18].

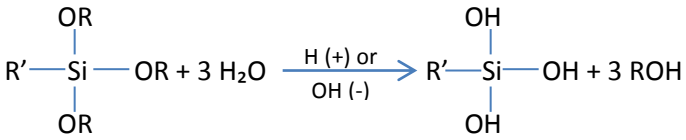


Figure 2-4: Silane coupling undergoing hydrolysis process

The silanol group will react with natural fibers and forming polysiloxane layers on the fiber surface. Water sorption reduces significantly due to polysiloxane layer which reduces the size of the cell wall [18]. With the modification by silane coupling agent, natural fibers became hydrophobic in nature and formed better interfacial bonding with polymer resulting in improved mechanical and physical properties of the composite.

2.2 Literature Review

Most researches in bio composite field are mainly focused on the types of natural fibers, chemical treatments of fibers, and size of fibers which are reinforced with thermoset matrix such as polyester and epoxy. Natural fibers have been researched extensively for past two decades [1-3] due to the attractive advantages offered by natural fibers. Many works have indicated that bio-composite have achieved higher value in terms of mechanical and physical properties compared to pure polymers [2-4, 8-10, 14]. However, the strength of synthetic composites is still far greater than that of bio-composite. The main drawback of bio-composite is the weak adhesion bonding between fibers and polymer matrix [2-4, 8-10, 14].

Studies were done on influence of natural fibers loading on polymer matrix in terms of mechanical properties. Studies have showed that by increasing the fiber content in polymer matrix, the mechanical properties of composite also increased. In an experiment using coconut shell particle as fillers in epoxy polymer, the coconut shell particles were varied between 0% and 20%. The result has showed that the increment of filler particle in epoxy improves the load bearing capacity of composite [19]. Results of other experiments using different fiber loadings have also showed similar results [20-21].

Natural fiber size plays an important role in influencing the strength of polymer composites. Studies were conducted to identify the optimum size of fibers to yield the sufficient strength of the bio-composite. Studies showed that the longer the fiber length, the higher the strength compared to particulate fiber reinforced polymer composite. In a research of mechanical characterization of epoxy composite reinforced with phormium tenax leaf fiber [2], two types of fibers were used, short fibers and longs fibers. Provided with same amount of weight of fibers condition, the results showed that long fiber of phormium tenax leaf fiber increased the mechanical properties of composite by 29% compared to using short fiber. In another experiment using rice-husk flour (RHF), wood flour (WF), and thermo-mechanical pulp (TMP) in polypropylene (PP), TMP-PP composite showed higher strength than WF-PP and

RHF-PP [22]. Similar results of other experiments [23-24] also indicated that using longer fiber provide higher strength to the composite than using particular fiber.

Type of fibers used in reinforced polymer also plays an important role in determining the mechanical and physical properties of the bio-composite. The strength of fibers depends on chemical composition of fibers such as cellulose, hemicellulose and lignin [25-26]. Cellulose provides strength to the fiber, hemicellulose provides support to the cellulose while lignin provides the elongation. Rich content of cellulose in the fiber provides high tensile and flexural strength. In an experiment of using sisal, kenaf, hemp, jute and coir in reinforced plastics, hemp bio-composite showed highest tensile and flexural strength while coir bio-composite showed the lowest tensile and flexural strength. This was due to coir having the least amount of cellulose content [26].

The major drawback of bio-composite is the weak adhesion bonding between natural fibers and polymer matrix. Therefore, coupling agent is applied to treat the fibers before reinforcing them into the polymer matrix. Coupling agent is a type of chemical treatment whereby it will change the structure of fibers permanently. Coupling agent improved the strength and stiffness of bio-composite [8, 10, 14, 27-28] due to the good interfacial bonding between fibers and polymer matrix. A study was conducted to compare the effect of treated jute fiber and untreated jute fiber reinforced polymer matrix [14] in term of mechanical properties of bio-composite. The result showed that tensile modulus of bio-composite with silane treated jute increased slightly. The increment of tensile modulus was due to improved adhesion bonding between fibers and polymer matrix.

Silane coupling agent is commonly used in fabrication of composite in term of fiber treatment to improve the adhesion bonding between natural fibers with polymer matrix. Silane is commercially available in large scale. Silane also consist of multiple and large functional groups that are able to react with any polymer to be used. Silane is able to react with OH due to the presence of alkoxysilane in its molecular structure [18]. A study was conducted to compare the effect of a new polymeric coupling agent between silane and maleic anhydride for polypropylene-wood flour (WF)

composite on mechanical properties. The results showed that silane was more efficient than maleic anhydride in influencing the mechanical properties of the bio-composite as silane was less polar than maleic anhydride which consequently improved the adhesion bonding between wood flour and polypropylene [28].

Even with chemical treatment of rich content of cellulose fibers, the mechanical and physical properties of natural filler reinforced composites are still inferior compared to synthetic fiber-filled composite. Therefore, researchers are moving towards to the hybrid fillers reinforced bio-composite in order to improve the strength of bio-composite further [10-11]. In an experiment of incorporating carbon fiber and kenaf fibers in polymer composite, the results showed that tensile and flexural strength increased of the composites compared to that using only carbon fiber due to strong bonds between the hybrid fibers [29].

More works and studies need to be conducted and carried out to study the influence of hybrid natural fillers on mechanical and physical properties of bio-composite. Therefore, current study attempt to incorporate both kenaf bast fiber and rice husk particulate filler in an epoxy based bio- composite to further strengthen the composites.

CHAPTER 3

METHODOLOGY

3.1 Materials

3.1.1 Matrix

Diglycidyl ether of Bisphenol A (DGEBA) with density, 1.1-1.5 g/cm³ in the form of clear epoxy resin and reactive polyamide were used as curing agent with density, 1.0-1.5 g/cm³. The weight ratio of epoxy resin and hardener was 2:1.

3.1.2 Reinforcement

Kenaf bast fibers and rice husks were used as natural fillers. Kenaf bast fibers were supplied by Forest Research Institute Malaysia (FRIM) and rice husks were supplied by Padiberas Nasional Berhad (BERNAS).

3.2 Processing of Composite Material

3.2.1 Fiber Preparation

Rice husks were grinded and sieved into particulate range of 225-425 μm ELE International Lab Test. Kenaf bast fibers were chopped using scissors in order to obtain fiber length in the range of 5 cm-7 cm. Both fillers were place in oven for 24 hours at 80°C in order to remove moisture contents.

3.2.2 Fiber Treatment

Kenaf bast fibers were treated with 3 wt% of vinyltriethoxysilane (VTS) in an ethanol solution for 1 hour. The pH of solution was controlled between 3.5- 4 with the presence of acetic acid. Treated kenaf bast fibers were dried in the oven at 80°C for

24 hours. Meanwhile, rice husks were treated with 10% of sodium hydroxide (NaOH) solution at room temperature for 3 hours. This process was known as mercerization. The rice husks particulates were rinsed in tap water many times to remove the excess of NaOH. The mercerized rice husks were dried in the oven at 80℃ for 24 hours. Both samples were stored in bottles with desiccant sachets.

3.3 Composites Fabrication

The mould used for the hybrid composite fabrication was a plastic rectangular mould which was 250 mm in length and 250 mm in width. A thin layer of releasing agent was applied on the mould before the epoxy was laid up. Epoxy mixture was mixed with rice husks particulates in a specific formulation shown in Table 3-1. The epoxy mixture was laid up in the mould uniformly by using a small roller. Kenaf bast fiber was added into the mould and arranged randomly like a mat. Another layer of epoxy was applied uniformly on the kenaf bast fibers. The composite material was left to cure for 24 hours at room temperature followed by 3 hours of post curing in oven at 80℃. After the composite was fully cured, then it was removed from the mould and cut to required specimen sizes for testing.

Table 3-1: Formulation of natural fillers epoxy bio composites

Formulation No.	**Rice husks (wt%)**	**Kenaf bast fiber (wt%)**	**Epoxy (wt%)**
1	-	-	100
2	40	-	60
3	-	10	90
4	10	10	80
5	5	15	80
6	3	17	80

3.4 Composites Testing

3.4.1 Mechanical Testing

The mechanical properties of specimens were measured using LLOYD Instrument LR5K Universal Testing Machine. Five specimens of each formulation were tested to obtain its tensile strength and flexural strength. Tensile and flexural test were performed on the specimens according to ISO 527-2 and ISO 178 respectively. The specimens with crosshead speed of 5 mm/min and 2 mm/min were used for tensile and flexural tests respectively. Tensile and flexural tests were calculated using the following formula.

Tensile strength,

$$\sigma_t = \frac{P}{bd} \quad (1)$$

Where: σ_t = tensile strength (MPa)

P = load (N)

b = width of specimen (mm)

d = thickness of specimen (mm)

Flexural strength,

$$\sigma_f = \frac{3PL}{2bd^2} \quad (2)$$

Where: σ_f = flexural strength (MPa)

L = distance between support span (mm)

P = load (N)

b = width of specimen (mm)

d = thickness of specimen (mm)

3.4.2 Water Absorption

The specimens were weighed before immersing in the water at room temperature. The specimens were taken out periodically for 6 days. The excess water on the surface of specimens was removed using soft cloth. The final weights of specimens were recorded. Water absorption was calculated, using equation (3).

Water Absorption:

$$W(\%) = \frac{M_w - M_d}{M_d}\ x\ 100\ \% \qquad (3)$$

Where:

M_w = weight of specimen after immersion

M_d = weight of specimen before immersion

3.4.3 Morphology

The fillers were scanned using Oxford Leo 1430 Scanning Electron Microscope to examine their surface morphology. The fibers were placed on aluminum stubs and coated with thin layer of gold in order to avoid electrostatic charging.

3.5 Activities/ Gantt Chart & Milestones

The Gantt chart of the project activities is in the Appendix.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Scanning Electron Microscopy

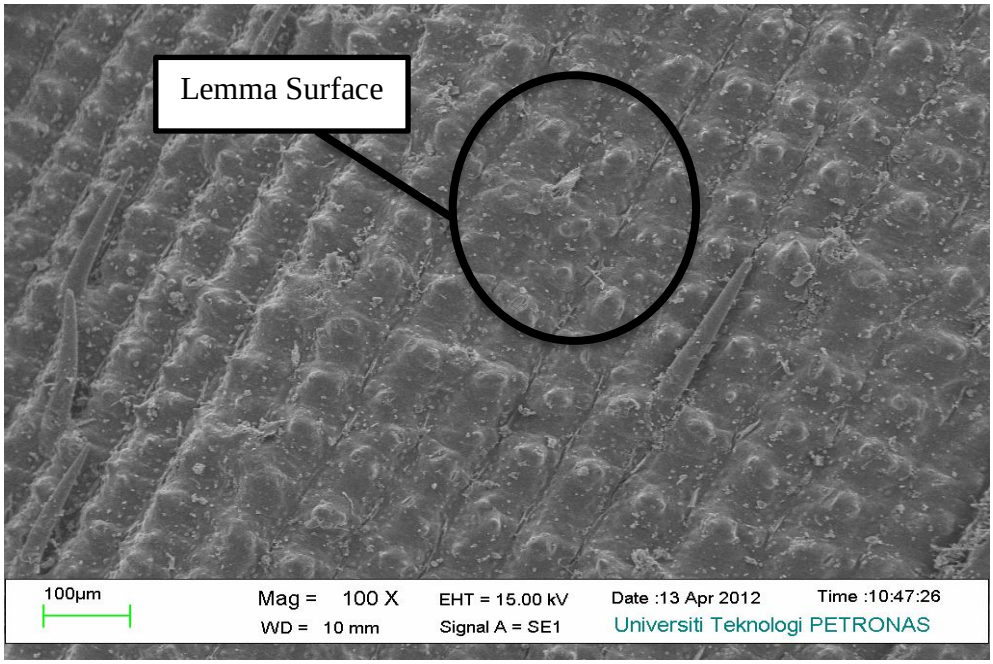
Rice husks and kenaf bast fiber surface morphology before silane treated were examined using SEM.

4.1.1 Rice husks Morphology

Rice husks consist of lemma and palea. Figure 4-1 (a) showed the rice husk at initial condition before silane treatment. The lemma surface was ridged with presence of conical protrusions on the rice husks surface as illustrated in the Figure 4-1(a) [30]. At higher magnification, Figure 4-1(b) showed the presence of silica at the outer layer of the epidermis. Higher intensity of brightness at particular area indicated greater silica concentration. Silica layer formed the outer layer epidermis which provides protection against microorganism attacks.

4.1.2 Kenaf bast fiber Morphology

Figure 4-2(a) showed the surface of kenaf bast fibers covered with a layer of lignin, hemicellulose, waxes and other impurities as well [31]. Besides that, cavities were present on the fibers surface providing a good site for bonding with composite matrix.



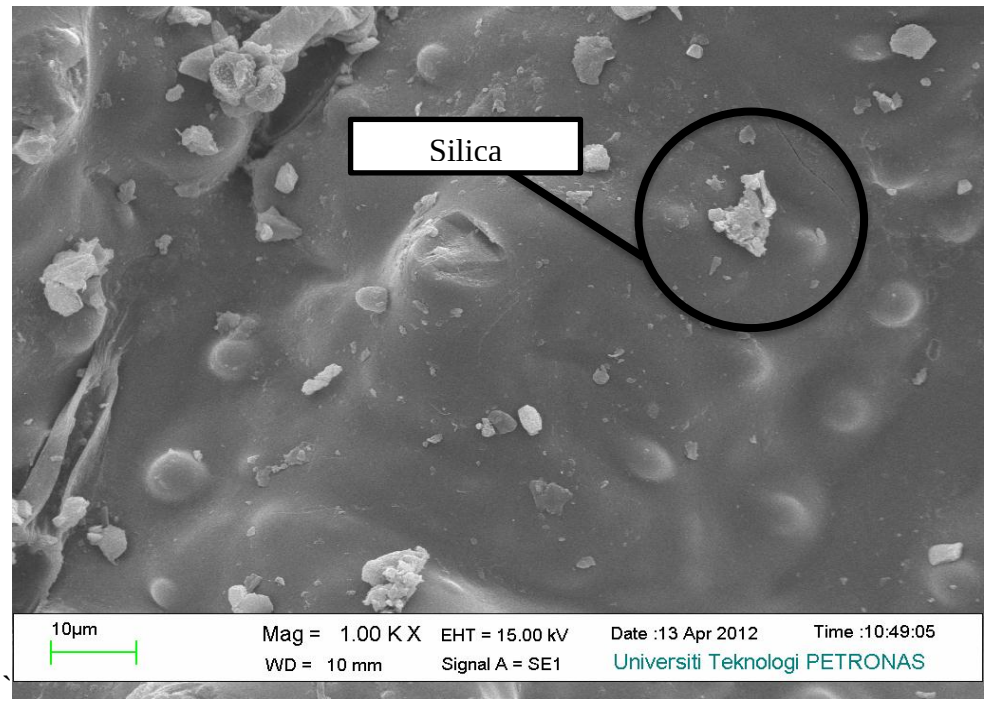


Figure 4-1: Rice husks fiber morphology: (a) 100 magnification, (b) 1000 magnification

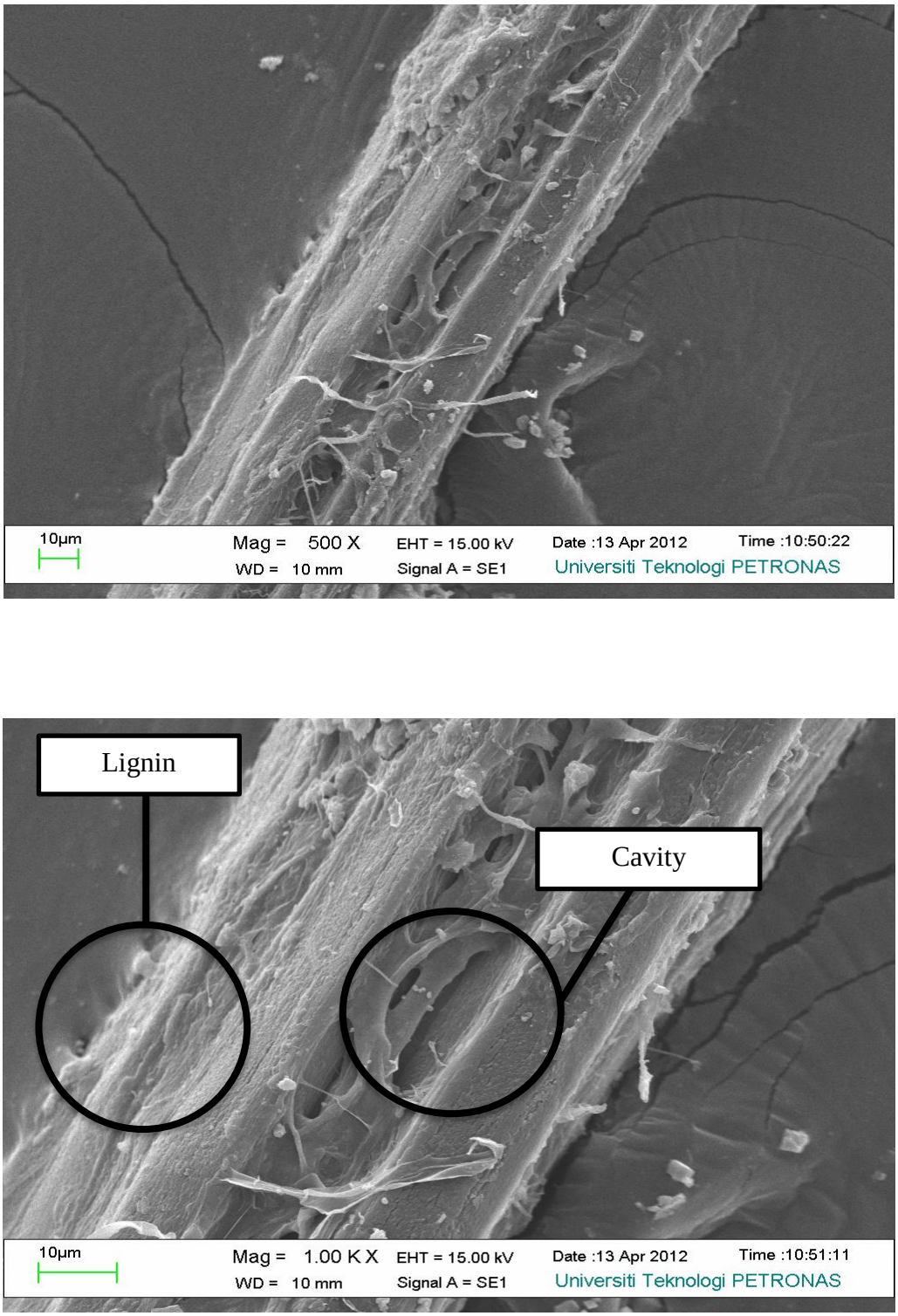


Figure 4-2: Kenaf bast fiber morphology: (a) 500 magnification, (b) 1000 magnification

4.2 Effect of hybrid filler loading on the Mechanical Properties

4.2.1 Tensile Strength:

The effect of hybrid filler loading on tensile strength of kenaf bast fibers, rice husks and hybrid fillers reinforced epoxy composite is shown in Figure 4-3. Incorporating rice husks particulates at 40 wt% with epoxy resulted in a 50.8% decrease in tensile strength, from 33.44 MPa to 16.45 MPa compared to pure epoxy while kenaf bast fibers at 10 wt% with epoxy resulted in a 67% reduction in tensile strength from 33.44 MPa to 16.45 MPa. Meanwhile, kenaf bast fibers at 40% loading were unable to reproduce due to difficulty in incorporating them into epoxy composition as their bulk density were very high.

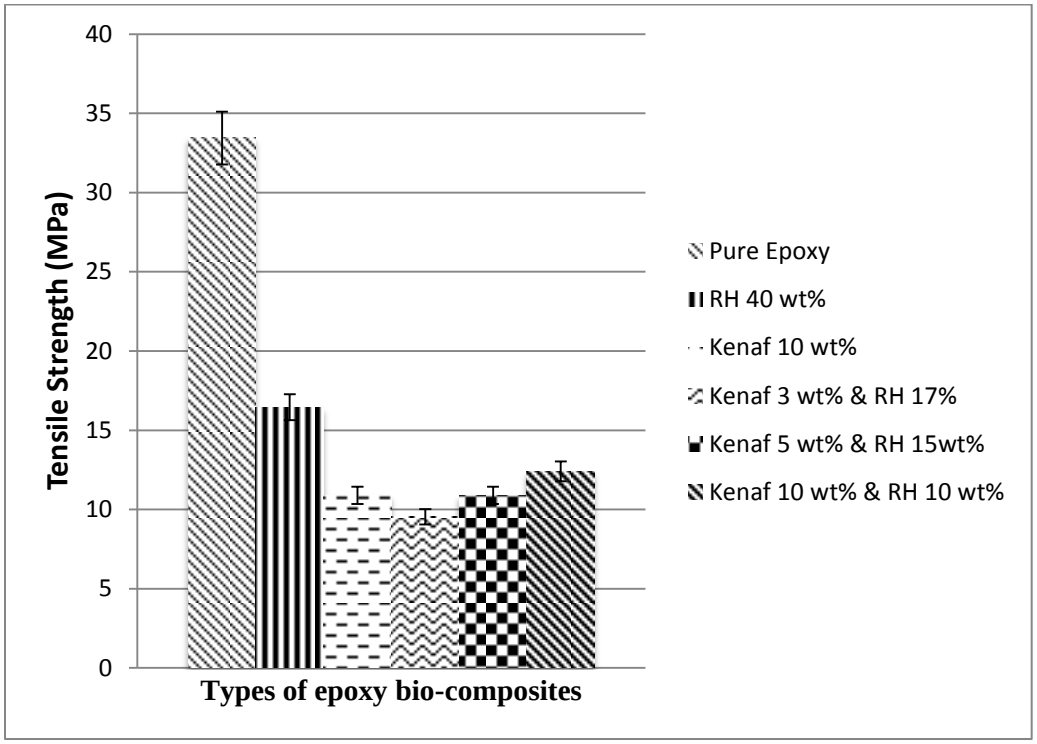


Figure 4-3: The effect of hybrid filler loading on tensile strength.

In the hybrid filler studies combination of 10 wt% rice husks particulates and 10 wt% kenaf bast fibers resulted in increased of tensile strength of the composite by 13.8% compared to 10 wt% kenaf bast fiber containing composite. The rice husks have

demonstrated its reinforcing effect to the composite which contributed to improved tensile strength of the composite.

However, as the kenaf bast fiber content was reduced from 10 wt% to 5 wt% while increasing the rice husks content to 15 wt%, a drop in tensile strength value of the composite was noted. In fact, the tensile strength value matched that of 10 wt% kenaf bast fibers reinforced epoxy composite. In other words, a 15 wt% rice husks give comparable reinforcing effect to a 5 wt% kenaf, a ratio of 3 rice husks: 1 kenaf bast fibers. This observation was further ascertained by the reduction in tensile strength value measured for 3 wt% kenaf bast fiber – 17 wt% rice husks reinforced epoxy composite. The coupling effect between rice husks and kenaf bast fiber fillers are seen for tensile strength and further corroborated by the composites flexural strengths measurements which are discussed next.

4.2.2 Flexural Strength:

Figure 4-4 shows the flexural strength of the kenaf bast fibers, rice husks and hybrid fillers reinforced epoxy composites at various filler loadings. Rice husks particulates at 40 wt% loading were introduced into the epoxy which resulted in a 24.5% drop in flexural strength compared to that of the pure epoxy. 10 wt% kenaf bast fibers incorporated into the epoxy resulted in a 57.7% decrease in flexural strength. As seen before in Figure 4-3, filler loading is observed to have an impact on the composites flexural strength too. Kenaf bast fibers at 40 wt% loading were not fabricated successfully due to high bulk density which causes processing limitation.

Comparing the results between tensile strength and flexural strength, the pure epoxy demonstrated a higher tensile strength of 33.44 MPa compared to its flexural strength of 27.75 MPa. Addition of fillers affected the tensile strength value of the composites to a greater extent compared to their flexural strength values. The composites flexural strength values are consistently higher than their tensile strength within the range of filler loading studied.

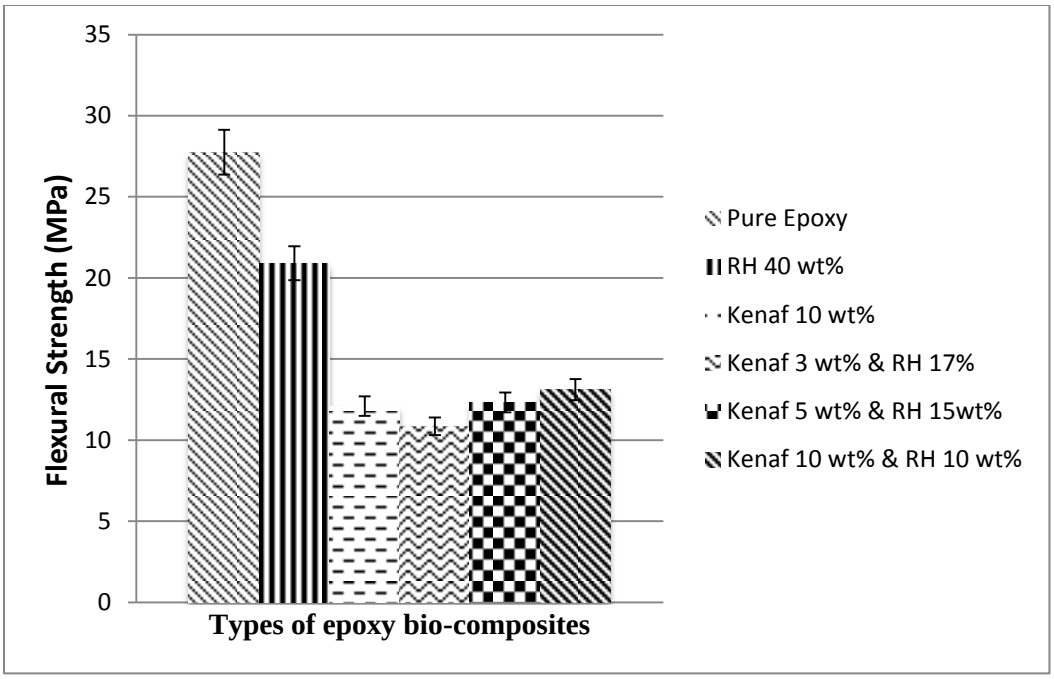


Figure 4-4: The effect of hybrid filler loading on flexural strength.

As mentioned earlier, the influence of the hybrid filler loadings on the composites flexural strength was analogous to that for tensile strength. The effectiveness of kenaf bast fibers in influencing the composites tensile strength compared to rice husks in a ratio of 1:3 respectively is repeatedly observed for the composites flexural strength as demonstrated in the Figure 4. For an instance, a 10 wt% kenaf bast fiber epoxy has a similar flexural strength with a 5 wt% kenaf bast fiber and 15 wt% rice husks epoxy composites.

As the kenaf bast fibers used increased, the tensile and flexural strength of the composites also increased. This was due to kenaf bast fibers being able to provide the strength and rigidity to epoxy and increased the ability of the composite to support the loads that being transferred from the epoxy matrix. Kenaf bast fibers and rice husks are known as lignocellulose materials which mainly consist of cellulose, lignin and hemicellulose. Cellulose provides the basic structure of cell wall and strength of the fibers [7-10]. Kenaf bast fibers have higher cellulose content which is more than 50% compared to rice husks which consist of 35 wt% [12-13]. In addition, kenaf bast fibers are long fibers which provide higher strength compared to particulate fillers

such as rice husks. This is due to long fibers being able to withstand more load than particulate fillers.

There are several factors contributing to the reduction of tensile strength and flexural strength of natural fillers reinforced epoxy bio-composite. Poor bonding between fibers and matrix and poor wetting due to hydrophilic of natural fibers and hydrophobic of matrix characteristics caused the significant decrease of tensile strength and flexural strength [2, 8-11,15]. Due to poor bonding between fibers and matrix, these fibers were unable to support the loads being transferred from the epoxy matrix [8-10, 16]. Although kenaf bast fibers and rice husks have undergone chemical treatment, however it seems that it did not improve the adhesion bonding between matrix and fibers considerably. Furthermore, agglomeration of kenaf bast fibers may have caused poor fiber dispersion of fibers in the epoxy composite which resulting in decrease tensile and flexural strength values. Bubbles trapped in the natural fillers reinforced epoxy bio-composite during composite fabrication may also contribute to the poor mechanical performance of the composite.

4.3 Effect of hybrid filler loading on the physical properties

4.3.1 Water Absorption

Figure 4-5 shows the relationships between water absorption and filler content of the kenaf bast fibers, rice husks and hybrid fillers reinforced epoxy composites at various filler loadings. All composites were immersed in water for 6 days. All samples showed similar pattern of water uptake where a sharp water absorption uptake was observed followed by a gradual increase until day 6 is observed. Pure epoxy showed the least water absorption followed by rice husks 40 wt%, 17 wt% rice husks - 3 wt% kenaf bast fiber and,15 wt% rice husks - 3 wt% kenaf bast fiber, 10 wt% rice husks - 10 wt% kenaf bast fiber, and 10 wt% kenaf bast fiber reinforced epoxy composites. This shows that reducing the kenaf bast fiber content and substituting it with rice husks has successfully reduced the water uptake. Kenaf bast fibers are hydrophilic material with high contact of hydroxyl group (OH). This hydrophilic behavior cause kenaf bast fibers to absorb more water uptake due to the hydrogen bonding between

filler and water molecules [17-18]. On the contrary, rice husks have high contact of silica in the composition which provided higher moisture resistance [12].

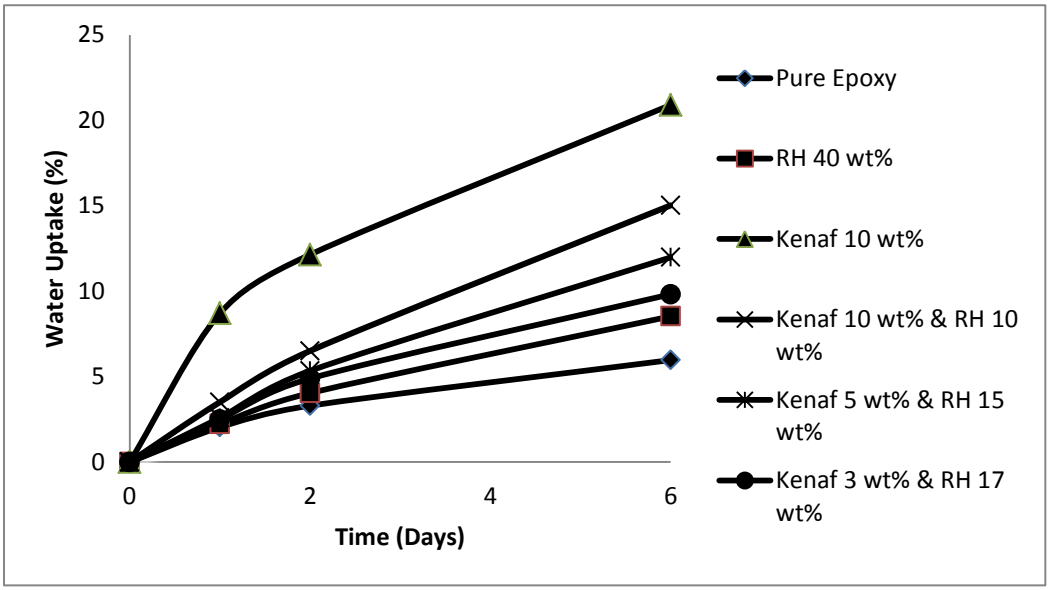


Figure 4-5: The effect of hybrid filler loading on water absorption.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The aim of the study was to investigate the potential of utilizing of the kenaf bast fibers and rice husks as the alternative hybrid fillers in epoxy composites material. Based on the results obtained, the tensile strength of hybrid composite increased from 9.54 MPa to 12.91 MPa while flexural strength of hybrid composites showed increment from 10.86 MPa to 13.11 MPa as kenaf bast fiber loading increased in the hybrid composition. However pure epoxy exhibited higher tensile and flexural strength, 33.44 MPa and 27.75 MPa respectively compared to those of the hybrid composites. The tensile and flexural strength of hybrid composite consisted of 5 wt% of kenaf bast fiber and 15 wt% rice husks showed similar values with the 10 wt% kenaf bast fiber reinforced epoxy composite. In other words, the effectiveness of kenaf bast fibers in influencing the composites tensile strength compared to rice husks was in a ratio of 1:3 respectively. The significant drop of the tensile and flexural strength of hybrid bio-composites compared to pure epoxy was due to weak adhesion bonding between hybrid fillers and epoxy matrix, agglomeration of kenaf bast fibers which caused poor fiber dispersion in the epoxy composites, irregularity of rice husks sizes and poor fabrication method.

Water absorption results indicated that hybrid composites water uptakes decreased as the rice husks particulates loading increased while kenaf bast fiber reinforced epoxy composite has the highest water uptake. This was mainly due to rice husks having rich content of silica which increased the moisture resistance and kenaf bast fibers are hydrophilic. Overall the mechanical and physical performance shows that rice husk and kenaf bast fibers could be used as alternative hybrid filler. However in order to obtain epoxy hybrid bio-composites with improved mechanical properties, further enhancement and optimization in terms of fabrication techniques and chemical treatment is required.

5.2 Recommendations

Chemical treatment using silane coupling agent and NaOH solutions were found that less effective on improving interfacial bonding between kenaf bast fiber and rice husks with epoxy matrix, respectively which results in a significant reduction in tensile and flexural strength of the hybrid composites. Therefore, proper chemical treatment with certain specific conditions is to be conducted according to different types of natural fillers.

In addition, poor dispersion of kenaf bast fibers has greatly affects the mechanical properties of hybrid composites. Therefore, the fibers should be well mixes with the epoxy and achieve good distribution among each other in order to obtain better mechanical properties. Other than that, irregularity of rice husks sizes has also contributed to the poor mechanical performance of hybrid composites. The rice husks should be well grinded into powder form in order to achieve better adhesion bonding between kenaf bast fiber with epoxy matrix.

During in the composites fabrication, hand lay-up techniques were applied. Although the composites were uniformly laid up in the mould using a small roller, however there are still bubbles were appeared in the composites which affect its mechanical properties. Therefore, a vacuum condition is recommended to be applied during in the composites fabrication.

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APPENDIX I-GANTT CHART

Title: The effect of hybrid filler loading on mechanical and physical properties of epoxy bio-composite

GANTT CHART FYP 1

No	Activity	W1	W2	W3	W4	W5	W6	W7		W8	W9	W10	W11	W12	W13	W14
1	Project Topic Selection															
2	Research Work and Data Gathering															
3	Submission of Preliminiary Report				★											
4	Samples collection raw materials : Kenaf bast fibers and Rice Husks															
5	Samples preparations- Kenaf bast fibers and Rice husks particulates															
6	Samples chemical treatment: Kenaf bast fibers undergone silane coupling and Rice husks undergone NaOH solutions															
7	Submission of Interim Report															★

GANTT CHART FYP 2

No	Activity	W1	W2	W3	W4	W5	W6	W7		W8	W9	W10	W11	W12	W13	W14
1	Composite Preparations: Hand Lay-Up Techniques Applied-Hybrid Composites AND LAY UP TECHNIQUES APPLIED- HYBRID COMPOSITES															
2	Mechanical Testing: Tensile and Flexural Strength															
3	Physical Testing Water Absorption															
4	Submission of Progress Report									★						
5	Submission of Final Report															★
6	Oral Presentation															

APPENDIX II- MECHANICAL AND PHYSICAL TESTING RESULTS

Tensile Test Readings:

Sample	Tensile Strength (MPa)					
	Pure Epoxy	RH 40 wt%	Kenaf 10 wt%	Kenaf 10 wt% + RH 10 wt%	Kenaf 5 wt% + RH 15 wt%	Kenaf 3 wt% + RH 17 wt%
1	32.14	15.82	10.47	12.41	10.65	10.05
2	33.72	16.85	11.54	11.94	10.27	9.13
3	33.83	16.41	10.69	12.43	11.3	9.73
4	32.65	16.72	11.16	12.51	10.52	9.51
5	34.86	10.45	10.65	12.75	11.73	9.27
Average	33.44	16.45	10.90	12.41	10.89	9.54

Flexural Test Readings:

Sample	Flexural Strength (MPa)					
	Pure Epoxy	RH 40 wt%	Kenaf 10 wt%	Kenaf 10 wt% + RH 10 wt%	Kenaf 5 wt% + RH 15 wt%	Kenaf 3 wt% + RH 17 wt%
1	28.73	20.65	12.27	13.18	12.16	10.84
2	27.12	19.74	12.31	12.91	12.38	10.56
3	27.56	21.04	11.93	13.16	11.88	11.08
4	28.43	21.32	11.88	13.23	12.71	11.13
5	26.91	21.78	12.05	13.07	12.47	10.71
Average	27.75	20.91	12.09	13.11	12.32	10.86

Water Absorption readings:

1. Pure epoxy:

Sample	Water Absorption			
	Pure Epoxy			
	DAY 1		DAY 2	DAY 6
	Weight before (g)	Weight After (g)	Weight After (g)	Weight After (g)
1	3.154	3.198	3.248	3.342
2	3.015	3.076	3.115	3.194
3	3.124	3.206	3.234	3.308
Average	3.097	3.160	3.199	3.282
Water Intake (%)	0.00	2.04	3.19	5.97

2. Rice husks 40 wt% :

Sample	Water Absorption			
	Rice Husks 40 wt%			
	DAY 1		DAY 2	DAY 6
	Weight before (g)	Weight After (g)	Weight After (g)	Weight After (g)
1	3.082	3.150	3.207	3.334
2	3.152	3.217	3.276	3.416
3	3.021	3.092	3.144	3.294
Average	3.085	3.153	3.209	3.348
Water Intake (%)	0.00	2.22	4.04	8.53

3. Kenaf bast fiber 10 wt%:

Sample	Water Absorption			
	Kenaf bast fiber 10 wt%			
	DAY 1		DAY 2	DAY 6
	Weight before (g)	Weight After (g)	Weight After (g)	Weight After (g)
1	3.152	3.426	3.534	3.811
2	3.088	3.354	3.463	3.733
3	3.172	3.447	3.557	3.834
Average	3.137	3.410	3.518	3.792
Water Intake (%)	0.00	8.70	12.14	20.9

4. Kenaf 10 wt% + Rice Husks 10 wt% :

Sample	Water Absorption			
	Kenaf 10 wt% + Rice Husks 10 wt%			
	DAY 1		DAY 2	DAY 6
	Weight before (g)	Weight After (g)	Weight After (g)	Weight After (g)
1	3.124	3.233	3.327	3.593
2	3.074	3.182	3.274	3.532
3	3.113	3.221	3.272	3.579
Average	3.104	3.212	3.306	3.568
Water Intake (%)	0.00	3.50	6.50	15.02

5. Kenaf 5 wt% + Rice Husks 15 wt%:

Sample	Water Absorption			
	Kenaf 5 wt% + Rice Husks 15 wt%			
	DAY 1		DAY 2	DAY 6
	Weight before (g)	Weight After (g)	Weight After (g)	Weight After (g)
1	3.051	3.130	3.213	3.417
2	3.087	3.167	3.336	3.457
3	3.107	3.186	3.189	3.479
Average	3.082	3.161	3.246	3.451
Water Intake (%)	0.00	2.59	5.34	11.98

6. Kenaf 3 wt% + Rice Husks 17 wt%:

Sample	Water Absorption			
	Kenaf 3 wt% + Rice Husks 17 wt%			
	DAY 1		DAY 2	DAY 6
	Weight before (g)	Weight After (g)	Weight After (g)	Weight After (g)
1	3.118	3.196	3.270	3.424
2	3.134	3.212	3.282	3.441
3	3.121	3.194	3.285	3.464
Average	3.124	3.201	3.279	3.433
Water Intake (%)	0.00	2.49	4.89	9.82

APPENDIX III- ISO 527 & ISO 178

Tensile Properties:		
ASTM D638-94b vs ISO 527-93E		
	ASTM	ISO
Preferred Specimen Type:	Type 1	Type 1A (ISO 3167)
Specimen Dimensions (mm):		
Overall Length:	165 (min)	150 (min)
Length of Narrow Section	57±0.5	80±2
Radius (tab to gage):	76±1	20-25
Width @ ends:	19±6.4	20±0.2
Width of narrow portion:	13±0.5	10±0.2
Preferred thickness:	3.2±0.4	4±0.2
Gauge Length:	50±0.25	50±0.5
Initial grip distance:	115±5	115±1
Test Speed (mm/min)	5, 50, 500 mm/min as specified by the material spec. or based on time to rupture	50 mm/min for ductile materials. 5 mm/min for brittle materials (Per ISO 10350)

Flexural Properties:		
ASTM D790-92 vs ISO 178-93E		
	ASTM	ISO
Preferred Specimen Type:	Length: 127 mm	Length: 80±2 mm
	Width: 12.7 mm	Width: 10±0.2mm
	Thickness: 3.2 mm	Thickness: 4±0.2mm
Support Span:	Span to depth ratio of 16	Span to depth ratio of 16
Support Radius:	5±0.1 mm or 3.2 mm minimum up to 1.5 times the depth for 3.2 mm or greater specimen thickness	5±0.1mm
Loading Nose Radius:	5±0.1 mm or 3.2 mm minimum up to 4 times the specimen depth	5±0.1 mm
Test Speed:	1.3 mm/min ± 50 % for the preferred specimen	2 mm/min ± 20 % for the preferred specimen
Maximum Allowable strain:	5%	3.5% (at conventional deflection of 1.5 x height)