



**WATER ABSORPTION BEHAVIOUR AND ITS
EFFECT ON THE MECHANICAL PROPERTIES OF
HYBRID INTERWOVEN CELLULOSIC FIBRE
COMPOSITES**

by

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
FESEM	Field Emission Scanning Electron Microscope
FRP	Fibre Reinforced Polymer
CO ₂	Carbon Dioxide
EFB	Empty Fruit Bunch
KK	Woven Kenaf Composite
JJ	Woven Jute Composite
HH	Woven Hemp Composite
KJ	Interwoven Kenaf/Jute Hybrid Composite
KH	Interwoven Kenaf/Hemp Hybrid Composite
NaOH	Sodium Hydroxide
PP	Polypropylene
PVC	Polyvinyl Chloride
UV	Ultraviolet
NFCs	Natural Fibre Composites

LIST OF SYMBOLS

W_t	Fibre loading (weight fraction)
$\Delta M_{(t)}$	Moisture content percentage
M_o	Mass of dry sample
M_t	Mass of immersed sample at specific time
D	Diffusion coefficient
Π	Pi
k	Initial slope of moisture content versus square root of time curve
h	Thickness
M_∞	Maximum moisture content

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Sifat serapan air dan kesannya terhadap sifat-sifat mekanikal komposit hibrid diperkuat anyaman fiber selulosa

ABSTRAK

Bagi memenuhi permintaan masyarakat terhadap ‘produk hijau’, fiber dari sumber semulajadi mula menjadi perhatian, mengantikan fiber sintetik sebagai bahan penguat di dalam komposit. Fiber semulajadi adalah murah, mudah diperolehi, tidak bertoksik, tidak kasar, kurang impak terhadap kesihatan dan mempunyai kekuatan dan modulus spesifik yang tinggi di mana ini menjadikan fiber semulajadi adalah pilihan yang sesuai bagi mengurangkan kebergantungan terhadap fiber yang berasaskan petroleum. Sebagai kesinambungan kepada kajian mengenai komposit berunsurkan bahan penguat semulajadi, kajian ilmiah ini mengkaji sifat serapan air dan kesannya terhadap sifat-sifat mekanikal komposit hibrid diperkuat anyam fiber selulosa. Komposit hibrid dari anyaman benang kenaf/jute dan kenaf/hemp dihasilkan melalui proses infusi vakum menggunakan epoksi sebagai matrik. Anyaman kenaf, jute dan hemp juga disediakan sebagai perbandingan. Ujian serapan air dijalankan pada suhu bilik (25°C) dengan merendam sampel komposit didalam air paip sehingga kandungan air menjadi tepu. Sampel anyaman kering dan juga basah kemudiannya diuji dengan ujian tegangan dan lenturan mengikut spesifikasi di dalam ASTM D638 dan ASTM D790. Bahagian sampel yang patah kemudiannya dianalisa menggunakan mikroskop imbasan (FESEM) untuk mengenal pasti impak serapan air terhadap ikatan fiber/matrik. Pada permulaan ujian, serapan air adalah tinggi dan kemudiannya semakin perlahan sehinggalah kandungan air di dalam komposit menjadi tepu selepas direndam selama 1400 jam. Komposit anyaman kenaf merekodkan kadar serapan air yang paling tinggi dan melalui hibridisasi dengan jute dan hemp, sifat rintangan air fiber kenaf meningkat sebanyak 46 dan 64%. Dalam keadaan kering mahupun basah, komposit hibrid didapati mempunyai sifat-sifat mekanikal yang lebih tinggi berbanding dengan komposit anyaman tunggal. Pada keadaan kering, kekuatan regangan dan lenturan komposit hibrid kenaf/jute adalah 11 dan 22% lebih tinggi berbanding komposit anyaman kenaf dan juga 16 dan 39% lebih baik berbanding komposit anyaman jute. Bagi sampel kering komposit hibrid kenaf/hemp pula, peningkatan kekuatan regangan dan lenturan sebanyak 4 dan 17% berbanding komposit anyaman kenaf dan 9 dan 33% berbanding komposit anyaman hemp direkodkan. Seperti sampel kering, peningkatan kekuatan regangan dan lenturan juga dialami oleh sampel basah komposit hibrid. Peningkatan sifat-sifat mekanikal ini adalah disebabkan oleh pemilihan fiber penguat yang betul, penyerapan matrik yang baik dan juga pembahagian beban yang berbeza diantara fiber di arah membujur dan melintang. Bagaimanapun, sifat-sifat mekanikal ini berkurangan mengikut masa rendaman. Kekuatan regangan komposit anyaman kenaf, jute, hemp, hibrid kenaf/jute dan kenaf/hemp pada masa tepu berkurangan sebanyak 75, 69, 67, 72 dan 69% berbanding kekuatan regangan mereka dalam keadaan kering. Mengikut susunan yang sama, kekuatan lenturan sampel basah juga berkurangan sebanyak 73, 64, 57, 69 dan 67% berbanding sampel kering. Seperti imej-imej yang ditunjukkan oleh FESEM, pengurangan sifat-sifat mekanikal ini adalah disebabkan oleh ikatan fiber/matrik yang lemah, keretakan pada matrik, void dan juga kehilangan resin yang dialami oleh sampel yang direndam.

Water absorption behaviour and its effect on the mechanical properties of hybrid interwoven cellulosic fibre composites

ABSTRACT

With aim to fulfill the public need that eager for 'green product', natural based fibres started to receive attention to substitute synthetic fibres as reinforcement in composites. Natural fibres is relatively cheap, easy to get, non-toxic, non-abrasive, less harmful to human health and possessed high specific strength and modulus which make them the perfect choice to reduce the dependence towards the petroleum based fibres. As the extension to the research involving woven and natural fibre hybrid composites, this study investigated the water absorption behaviour and its effect on the mechanical properties of hybrid cellulosic fibre composites. Hybrid composites consists of interwoven kenaf/jute and kenaf/hemp yarns were prepared by infusion process using epoxy as the matrix. Woven kenaf, jute and hemp composites were also prepared for comparison. Water absorption test were conducted according to ASTM D590 standard by immersing the composite samples in tap water at room temperature until the water content reach saturation. Dry and water immersed samples of both woven and interwoven hybrid composites then were subjected to tensile and flexural tests according to ASTM D638 and ASTM D790, respectively. Fractured portion of the samples were examined using field emission scanning electron microscope (FESEM) to observe the effect of water absorption towards the fiber/matrix interface. Water uptake was rapidly increased at the beginning and getting slower until reach saturation at 1400 hours of immersion time. Among individual woven composites, woven kenaf composite had the highest water uptake and through hybridization with jute and hemp fibres, water resistance properties of kenaf fibre were improved by 46 and 64%. At both, dry and wet condition, mechanical properties of interwoven hybrid composites was found to be greater than their individual woven composites. In dry condition, tensile and flexural strength of interwoven kenaf/jute hybrid composite were 11 and 22% higher than woven kenaf composite and 16 and 39% greater than woven jute composite. For dry samples of interwoven kenaf/hemp hybrid composite, increment of 4 and 17% over woven kenaf composite and 9 and 33% over woven hemp composite were recorded for their tensile and flexural strength. Similar as the dry samples, increase of tensile and flexural strength was also observed for the water immersed samples of the interwoven hybrid composites. Amount of water uptake increased as the immersion time increased and reduced the strength and modulus (tensile and flexural) of the water immersed samples. Tensile strength of woven kenaf, jute, hemp, interwoven kenaf/jute and kenaf/hemp hybrid composites at their saturation state were reduced by 75, 69, 67, 72 and 69% over their strength at dry condition. Following the similar sequence, flexural strength of the saturated samples also reduced by 73, 64, 57, 69 and 67% than the dry condition samples. Those reductions attributed by debonding, matrix cracking, and delamination as shown through the FESEM images.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Exploration on the natural resources fibres as substitution to the petroleum based fibres were rapidly growth in the last few decades and nowadays, natural fibre reinforced polymer composites are widely used in the automotive industry, sport equipment and marine structure (Pickering, Efendy, & Le, 2016). In comparison with the synthetic fibre, natural fibre possessed high specific strength, readily available at low cost, renewable and less abrasive to processing equipment and these features correspond well with the consumer demands and environmental sustainability. Cellulose based natural fibres that are widely studied and applied in composites engineering applications are flax, hemp, jute, ramie, kenaf and sisal (Faruk, Bledzki, Fink, & Sain, 2012).

Hybrid composite consists of at least two types of fibre in a single matrix system. Interest in hybridization started when there was increasing demand for a lighter material with improved toughness, especially for aircraft application. Early focus of hybridization is reducing the cost by combining carbon fibre with cheaper glass fibre. Through hybridization, the mechanical performance of the composites were improved which attributed by the changes in applied load distribution and failure development. Now, hybridization has been

widely explored such as pseudo-ductility, ductile fibre and natural fibre hybrids composites (Swolfs, Gorbatikh, & Verpoest, 2014).

Common types of reinforcement form in fibre reinforced composites are fibre, yarn and fabric. Generally, how the reinforcement was arranged or layered affect the final performance of the composites and fabric form yield outstanding properties rather than the others. Fabric can be classified as woven, knitted or non-woven. Woven fabric is interlacing between two sets of yarns along the longitudinal (warp) and transverse (weft) direction. Interloping between yarns at weft or warp direction defined the knitted fabric and for non-woven fabric, it is termed as the bonding of raw material (fibre) using mechanical, chemical or solvent (Mison, Islam, Epaarachchi, & Lau, 2014).

In particular, all polymer composites absorb moisture and the water molecules can act as a plasticizer by influencing simultaneously the fibres, the matrix and the interface, thus creating regions of poor transfer efficiency, which results in a reduction of mechanical properties. It is now well established that composite materials are sensitive to humidity through absorption of water leading to differential swelling between the fibres and the matrix. This situation is even more complicated in the case of natural fibres, which are known to exhibit a poor resistance to water absorption, due to their hydrophilic nature that is related. (Sethi & Ray, 2015).

As extension to the research involving hybrid and woven structure, this research main focus is investigating the water absorption behavior of hybrid composite made from interwoven kenaf/jute and kenaf/hemp yarns to observe their responses when subjected to moisture and at the same time predict their capability for outdoor applications. In addition, the mechanical properties of the composites as the effects of hybridization and water absorption was also investigated.

1.2 Problem Statement

Fibre Reinforced Polymer (FRP) composites are widely used in various applications. They have been applied in the automotive industry, marine structure, interior of an aircraft and many more. The 'famous' fibres used as reinforcement in composites are glass, carbon and kevlar/aramid fibres. The issue here is that those 'famous' fibres are synthetic, derived from the petroleum resources; which are limited, expensive and require high processing energy. Furthermore, burning those synthetic fibres for disposal released high amount of carbon dioxide to the environment, depleting the ozone layer, exposing us to harmful UV radiation; which lead to several health issues.

In order to reduce the usage of the synthetic fibres, there is a method called hybridization. Through hybridization, composites with a balance in mechanical, chemical and physical properties can be achieved by maintaining the advantages and alleviate some disadvantages of both reinforcement fibres. Hybridization of the synthetic fibres with natural based fibre not only reduce the cost, but also ensure the sustainability of supplies, since the natural fibres are largely harvested around the world. However, there are less reported work about hybrid composites that solely constituted of natural fibre. The common hybrid combination and configuration seen in the literature is the natural fibre been layered in between the synthetic fibre.

During processing, reinforcement in woven fabric form is easier to handle since the fibre are well arranged compared to non-woven fabric. Furthermore, mechanical strengths of composites made of woven fabric were higher than non-woven fabric due to the good fibre orientation and load distribution. By taking the advantages of natural fibre and woven

structure, the mechanical properties of hybrid composites made from the interwoven of kenaf/jute and kenaf/hemp fibre at their dry and wet condition were investigated in this study.

Mechanical performance of fibre reinforced polymer composites were affected with the presence of moisture, which tailored the interfacial adhesion and reduce the reliability of the material either for short or long-term applications. Among the effect of moisture are matrix cracking, delamination and fibre fracture and those damages ease the water penetration into the composite system. Due to the hygroscopic nature, water absorption of natural fibre was higher than the synthetic fibre. Therefore, further research is still required to improve their moisture resistance property and widen their applications. In this study, the water absorption behaviour of interwoven cellulosic fibre been characterized based on their water uptake, diffusion, sorption, permeability coefficient and mode of the diffusion process to predict their suitability as outdoor applications. The outcome from this study will be beneficial to the research community in order to fully utilized the natural resources fibres in the composites industry.

Due to the availability of kenaf, jute and hemp fibre in Malaysia, these fibre were selected as reinforcement in the present study. Furthermore, high strength kenaf fibre is believed to improve the tensile and flexural properties of jute and hemp fibre through hybridization. In another way around, jute and hemp fibre had the capability to enhance the water resistance properties of kenaf fibre.

1.3 Research Objectives

In this present study, hybrid composite from interwoven kenaf/jute and kenaf/hemp yarns reinforced epoxy composites were fabricate using vacuum infusion technique. In order

to expand the utilization of natural based fibres in composite industry, the following objectives are emphasized in this study:

- To investigate the water absorption behaviour of the interwoven hybrid composites and compared with the individual woven composites.
- To determine the effects of hybridization and water absorption on the tensile and flexural properties of the woven composites.
- To analyse the morphological structure of dry and water immersed samples after tensile and flexural tests.

1.4 Scope of the Research

Material that been used in this study are kenaf, jute and hemp fibre in yarn form. The yarns were manually weaved using locally constructed wooden frame and type of weaving pattern is plain structure. In the construction of interwoven kenaf/jute and kenaf/hemp fabric, the warp direction is the kenaf yarns and the weft direction is jute and hemp yarns. Infusion manufacturing technique using epoxy as polymer matrix was selected for the fabrication of composites. Water absorption test were conducted at room temperature using tap water; following the ASTM D570 standard to determine the percentage of water uptake and others absorption properties such as diffusion, sorption, and permeability coefficient. For mechanical test, tensile and flexural properties were determined as elucidated in ASTM D638 and ASTM D790 standards, respectively. Average results from five specimens were recorded for each type of composites. Finally, fiber/ matrix bonding at dry and saturated condition were examined using FESEM.

1.5 Organisation of Thesis

In this thesis, the background theory, purpose and limitation of the research are presented in the first chapter. Literature survey involving natural fibre, hybridization, woven structure, and water absorption behaviour of the FRP are summarized in chapter two. This is followed by chapter three, where the selected materials and methods used in experiments been explained in details. All the testing result were presented and discussed in chapter four. Last but not least, chapter five presents the conclusions of this thesis and some suggestions for future works are provided.

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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

FRP composites have been widely used in varieties of application and several limitation of petroleum based fibres; mainly cost and environmental concern, had increased the demand towards the natural based composites. Hence, the development of natural fibre as reinforcement in composites from the previous reported works are summarized in this chapter. Starting with the classification of natural fibre, this chapter explained their advantages and mechanical properties that suit them as substitution to the synthetic fibres. As a method where the optimum performance of composites could be achieved, combination and configuration of fibres in hybrid composites were then discussed. Next, application of textile processing method in the FRP composites was reviewed, as these kinds of fibres arrangement resulted in greater mechanical properties over the others orientation. Natural fibres absorption behaviour was also discussed in the final sub-chapter as it is the main criteria to be consider when the natural fibre composites are meant for outdoor applications.

2.2 Classification of Fibres

With the increasing price of concrete, steel and timber, FRP composites started to receive attention as an alternative to those conventional materials in the infrastructure industry (Dittenber & GangaRao, 2012). Reinforcements (fibre), in a desired orientation impregnated with a polymer (matrix) are the main two components of a composite. The polymer could be thermoplastic (polypropylene, polyethylene and poly vinyl chloride) or thermoset (phenolic, epoxy and polyester) (Ku, Wang, Pattarachaiyakooop, & Trada, 2011). As for the reinforcement, fibres can be classified into two classes; natural fibres and synthetic fibres. Based on the sources, natural fibres can be divided to plants, animals and minerals groups; while synthetic fibres can be subcategorized to organic and inorganic groups as given in Figure 2.1.

Derived from petroleum or petrochemical sources, glass fibre is the common synthetic fibre use as reinforcement. In comparison with the other synthetic fibre such as aramid and carbon, glass fibre are cheaper and possessed higher strength; which suitable for low to medium load applications (Wambua, Ivens, & Verpoest, 2003).

Main component of the plant based fibre is cellulose, whereas animal based fibre consists of protein. As for mineral based natural fibres, they exist in the asbestos group and due to high risk during inhalation, it was banned in most country (Wambua, et al., 2003). With higher strength and stiffness compared with the animal and mineral based fibres, plant based fibre found to be the perfect replacement to those synthetic fibres (Pickering et al., 2016). Compared to the synthetic fibres, plant based natural fibres possessed high specific strength and stiffness, renewable and biodegradable, require only small processing energy and served well as insulator (Jawaid, & Khalil, 2011).

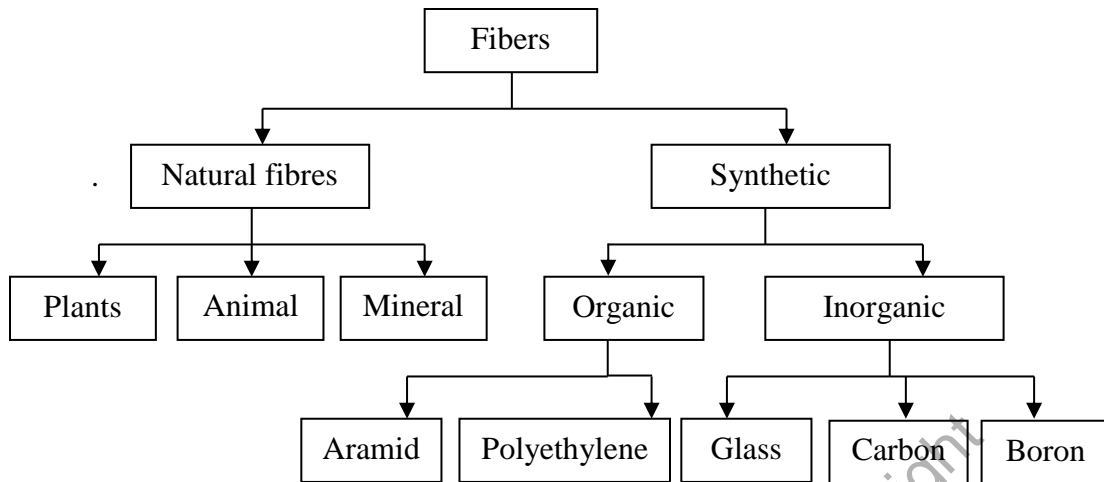


Figure 2.1: Classification of fibres (Jawaid & Khalil, 2011)

2.3 Natural Plant Based Fibre

Structure of a natural plant cell wall, as shown in Figure 2.2, is called the microfibril; consists of one primary cell wall and three secondary cell walls, with a lumen; an open channel in the center of the microfibril. Each layers of cell wall contains cellulose in a lignin-hemicellulose matrix. Structure and contents of the cell walls are different according to species and parts of the plants (Dittenber & GangRao, 2012). As the three main chemical components of natural fibres, cellulose, hemicellulose and lignin played an important role in determining the properties of the fibres. Strength and stiffness were measured based on the cellulose content, whereas hemicellulose and lignin protect the fibres from degradation (Akil, Omar, Mazuki, Safiee, Ishak, & Bakar, 2011).

Natural fibres can be characterizes according to their shapes, sizes, orientations, and thickness of the wall. However, in determining the fibres strength, the dimensions of the individual cells is very important. Length, width and diameter of the fibres are varies

depending on the species, maturity, location of the plant and the process involved during the fibres extraction (Jawaid & Khalil, 2011).

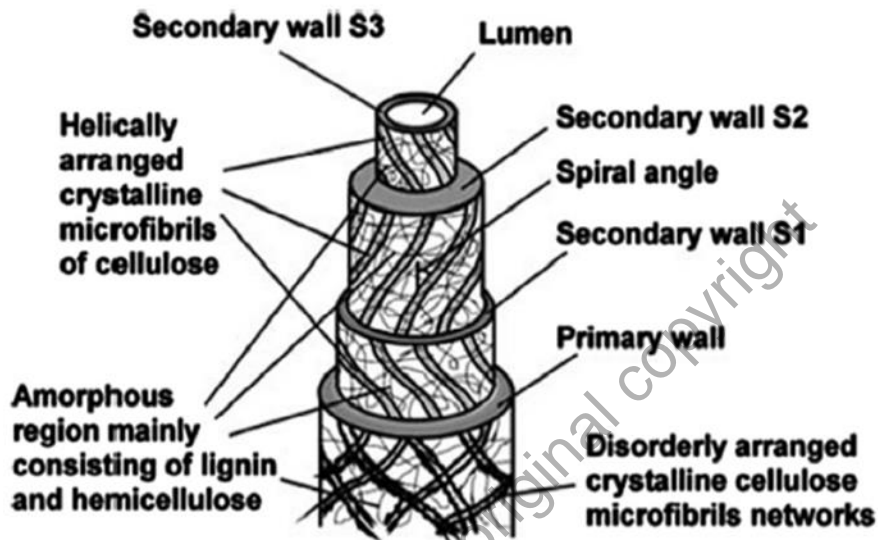


Figure 2.2: Structure of single fibre cell (Rong, Zhang, Liu, Yang, & Zeng, 2001; John & Thomas, 2008)

According to Faruk et al. (2012), plant based natural fibres can be divided to several types as presented in Figure 2.3. Bast based fibres are the common fibres seen in the literature due to their higher cellulose content and the microfibrils were oriented in the fibre direction. Even though synthetic fibres such as nylon, rayon, aramid, glass, and carbon are extensively used, these materials are expensive and non-renewable resources. As for natural fibres, they are renewable and recyclable; with fewer residues when they were burned for disposal, returning less carbon dioxide (CO₂) to the environment. Table 2.1 listed the advantages and disadvantages of natural plant based fibres, in comparison with the synthetic fibres.

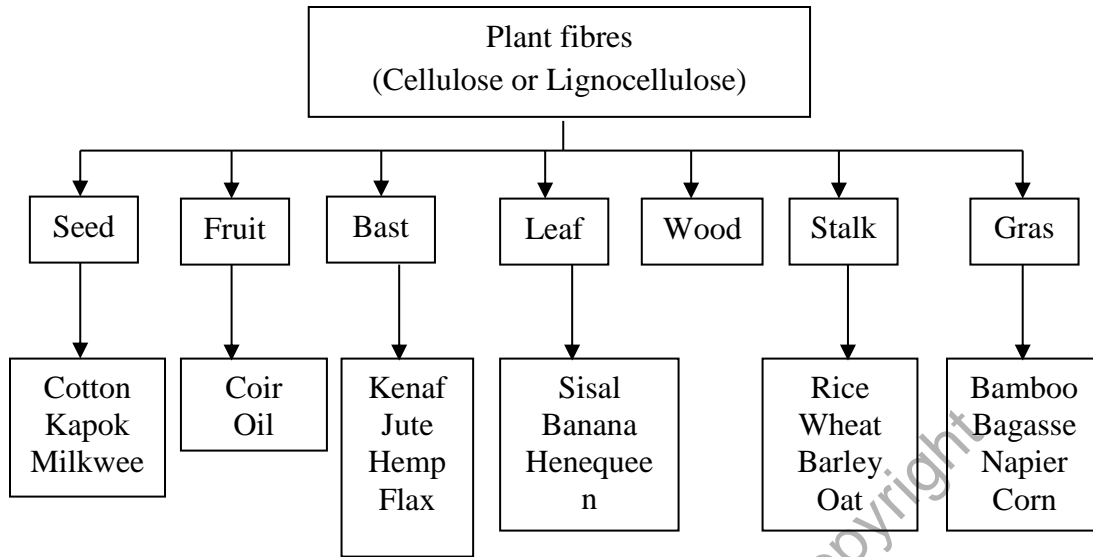


Figure 2.3: Classification of plant based natural fibres (Faruk et al., 2012)

Table 2.1: Advantages and disadvantages of natural fibres, in comparison with the synthetic fibres (Jawaid & Khalil, 2011)

Advantages	Disadvantages
High specific strength and stiffness	Low strength
Low processing energy	Varies in quality, depend on fibres sources
Less tool wear during processing	Low melting temperature
Renewable and biodegradable	Poor interphase bonding
Good insulator behaviour	Low fire retardant
Good thermal and acoustic insulating properties	Low durability

2.4 Kenaf Fibre

Kenaf fibre (*Hibiscus cannabinus*) can be obtained from bast and core of its plant. Among other plants, kenaf plant absorbs the highest carbon dioxide. Kenaf single fibres are about 2-6 m long, coarse and quite brittle with pale colour (Bledzki, Franciszczak, Osman, & Elbadawi, 2015). Kenaf is commercially available and economically cheap amongst other natural fibre reinforcing material. Kenaf plants are hard, strong and tough with a fibrous stalk, resistant to insect damage and require only few amounts of pesticides (Elsaid, Dawood, Seracino, & Bobko, 2011).

Fibre content influences the mechanical properties of composites. Higher fibre loading (up to 50-60%) increased the tensile and flexural strength of kenaf fibre composites (Ochi, 2008; Shibata, Cao, & Fukumoto, 2008; Ghani, Hyie, Berhan, Taib, & Bakri, 2012). Addition of kenaf fibre with fibre volume fraction of 15% in glass/polyester composite improved the tensile, flexural and impact strength of the composite. However, the impact strength found to be reduced when higher kenaf fibre content were added (Atiqah, Maleque, Jawaid, & Iqbal, 2014).

Water absorption behaviour of long kenaf/woven glass hybrid composite fabricated using cold press technique under three different environmental condition, i.e. distilled water, rain water, and sea water was investigated (Salleh, Taib, Mihat, Berhan, & Ghani, 2012). As the result, the moisture content is found to exhibit non-Fickian behaviour regardless of three different condition. Furthermore, liquid exposure of the composite deteriorates the fracture toughness due to the weakening of interface between fibre and matrix.