

**STUDIES ON THE PROPERTIES OF  
PULTRUDED KENAF FIBER REINFORCED  
UNSATURATED POLYESTER COMPOSITES**

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**UNIVERSITI SAINS MALAYSIA**

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**STUDIES ON THE PROPERTIES OF PULTRUDED KENAF  
FIBER REINFORCED UNSATURATED POLYESTER  
COMPOSITES**

**by**

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**Thesis submitted in fulfillment of the requirements  
for the Degree of  
Master of Science**

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## DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitles “Studies on the properties of pultruded kenaf fiber reinforced unsaturated polyester composites”. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or university.

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## LIST OF ABBREVIATIONS

AS	Acidic solution
ASTM	American Society for Testing and Materials
CaCO <sub>3</sub>	Calcium Carbonate
CMC	Ceramic Matrix Composite
CTE	Coefficient of Thermal Expansion
D	Diffusion coefficient
DSC	Differential Scanning Calorimetry
DTG	Derivative Thermal Gravimetry
DW	Distilled water
EPTA	European Pultrusion Technology Association
FRIM	Forest Research Institute Malaysia
FRP	Fiber Reinforced Polymer
FTIR	Fourier-Transform Infrared Spectroscopy
HDPE	High-density polyethylene
KFRPC	Kenaf Fiber Reinforced Unsaturated Polyester Composite
LDPE	Low-density polyethylene
MMC	Metal Matrix Composite
NaCl	Sodium Chloride
NF	Natural Fiber
NFRP	Natural Fiber Reinforced Polymer
NFRPC	Natural Fiber Reinforced Polymer Composite
PAI	Polyamideimide
PA-6	Polyamide 6
PA-6,6	Polyamide 6,6



PBT	Polybutylene terephthalate
PC	Polycarbonate
PCL	Polycaprolacton
PEEK	Poly(etherether ketone)
PEI	Polyetheneimide
PET	Polyethylene terephthalate
PLA	Poly lactic acid
PMC	Polymer Matrix Composite
PP	Polypropylene
PPS	Polyphenylenesulphide
PVA	Polyvinylalcohol
RF	Radio Frequency
RTM	Resin Transfer Molding
SEM	Scanning Electron Microscopy
SW	Seawater
TGA	Thermo Gravimetric Analysis
VARTM	Vacuum Assisted Resin Transfer Molding
USP	Unsaturated Polyester

## LIST OF SYMBOLS

$\alpha$	degree of decomposition
$\text{CH}_2\text{OH}$	Class of carbohydrates
$(\text{C}_6\text{H}_{10}\text{O}_5)_n$	Cellulose
cm	centimeter
$\text{COO}^-$	Carboxyl group
$^\circ\text{C}$	Degree Celsius
$d$	Diameter
$\epsilon_0$	Expansion ratio
$f$	Fineness (in Tex)
$\text{g/cm}^3$	Density
g/km	Tex
h	thickness
$\text{H}^+$	Hydrogen
j	Radius
kg/ha	yield of fiber
kN	Load
$\Delta L$	Change in length
m	meter
$m_c$	Mass of composites
$m_f$	Mass of fiber
mg	milligram
mm	millimeter
Mm	Maximum weight gain
mm/min	Crosshead speed
ml/min	Flow rate

$M_t$	Weight of moisture content
$M_0$	mass of the dried material
$M_\infty$	Weight gain at equilibrium
$\mu\text{m}/\text{m}^\circ\text{C}$	Thermal expansion coefficient
N	Number of roving
NaCl	Sodium Chloride
-OH	Hydroxyl group
$\rho$	Density
$P_c$	Permeability coefficient
pH	Measure of the acidity or basicity
psi	Pressure (Pound per square inch)
$S_c$	Sorption coefficient
$\sqrt{t}$	$\sqrt{\text{Time}}$
UTS	Ultimate Tensile Strength
$V_c$	Volume of the composites
$w_0$	Initial weight
$w_t$	Weight after immersion
%	Percentage
$\approx$	Almost equal to

## **KAJIAN TERHADAP SIFAT-SIFAT KOMPOSIT POLIESTER TAK TEPU TERPULTRUSI DIPERKUKUHKAN GENTIAN KENAF**

### **ABSTRAK**

Komposit poliester tak tepu diperkukuh dengan gentian kenaf (KFRPC) terdiri daripada gentian kenaf (KF) dan poliester tak tepu telah dihasilkan menggunakan mesin pultrusi dan diacuan ke bentuk rod. KF dan poliester tak tepu dianalisa dan dicirikan sifat penguraian, fizikal dan mekanikal, seperti perendaman, kekuatan, rentangan, kebolehan serapan dan permukaan spesimen. Dalam kajian ini, KF dan poliester tak tepu telah direndam ke dalam air suling (DW), air laut (SW) dan larutan asid (AS) pada suhu bilik untuk mengkaji sifat spesimen dalam keadaan rendaman dalam jangka masa yang panjang. KF yang direndam dalam air laut menunjukkan penyerapan air yang tertinggi, sedangkan KF yang direndam dalam larutan asid menunjukkan penyerapan air yang terendah. Kekuatan tegangan KF yang direndam telah didapati menurun dengan peningkatannya ini menunjukkan masa perendaman, penguraian KF telah berlaku. Pemeriksaan lanjut ke atas KF yang direndam menggunakan SEM merungkai struktur mikro penguraian dalam KF dengan menunjukkan kehadiran retakan mikro dan permukaan kasar yang banyak. Seterusnya, kesan penyerapan air pada sifat pelenturan poliester tak tepu juga telah dijalankan. Taburan serapan air didapati banyak apabila poliester tak tepu direndam dalam air suling daripada air laut dan larutan asid mengikut turutan. Selepas rendaman, poliester tak tepu didapati mengalami penurunan kekuatan lenturan. Ini adalah disebabkan peranan resapan molekul air ke dalam poliester tak tepu. KFRPC telah berjaya dihasilkan menggunakan kaedah pultrusi dengan kandungan isipadu KF sebanyak 50%, 60% dan 70%. Kepelbagaian kandungan KF ini kemudiannya diuji untuk mengetahui sifat mekanikal pada setiap komposit. Dengan ujian yang

dijalankan, 70% kandungan KF memberi sifat kekuatan yang lebih baik berbanding yang lain. Selanjutnya kajian yang mendalam telah dijalankan terhadap 70% KF dengan menganalisa dan mengkaji sifat penguraian komposit dalam rendaman air suling, air laut dan larutan asid. Seterusnya kajian dijalankan bagi mengetahui sifat mekanikal, terma dan fizikal komposit ini. Dalam kajian penyerapan air, sifat peresapan komposit ini telah menuruti hukum Fick. Kandungan taburan resapan ( $D$ ) dan keseimbangan lembapan ( $M_m$ ) telah dihitung dengan mengukur setiap timbangan spesimen pada selang masa tertentu.  $D$  dan  $M_m$  yang tertinggi telah direkodkan bagi komposit yang direndam dalam air suling seterusnya rendaman dalam larutan asid dan air laut. Sifat lenturan dan mampatan bagi komposit yang direndam ini telah didapati menurun dengan peningkatan peratus resapan air. Dalam kajian terhadap sifat terma pula, komposit yang direndam dalam air laut didapati memberikan peratus berat mengurang lebih banyak berbanding larutan asid dan air suling.

## **STUDIES ON THE PROPERTIES OF PULTRUDED KENAF FIBER REINFORCED UNSATURATED POLYESTER COMPOSITES**

### **ABSTRACT**

Kenaf fiber reinforced unsaturated polyester composites (KFRPC) consisting of kenaf fiber (KF) and unsaturated polyester were produced by pultrusion machine, molded into rod specimens. KF and unsaturated polyester was being analyzed and characterized in terms of degradation, physical and mechanical properties, such as immersion, strength, elongation, absorbency and surface characteristic. In this research, KF and unsaturated polyester were immersed in distilled water (DW), seawater (SW) and acidic solution (AS) at room temperature to study the behavior of the specimen after long term immersion. KF immersed in SW exhibited the highest level of water absorption, whereas KF immersed in AS showed the lowest water absorption values. The tensile strength of the immersed KF decreased with increasing immersion time implying the degradation of the fiber. Investigation on the microstructure of immersed kenaf fibers using SEM reveal the degradation of the KF with presence of microcracks and increased in the surface roughness. Effect of water immersion on the bending behavior of the unsaturated polyester was also experimentally investigated. Water diffusion was observed more in DW than SW and AS, respectively. The unsaturated polyester experienced significant reduction of the flexural strength. These are attributed to the function of the water molecules absorbed in the unsaturated polyester. KFRPC were successfully produced with 50%, 60% and 70% of KF by volume, using pultrusion method. The various percent of KF volume were tested to identify the influence of KF volume (%) on the mechanical properties. Then the KFRPC of 70% KF volume were being analyzed and characterized in terms of degradation in DW, SW and AS; mechanical, thermal and

physical characteristic. The kinetics of water absorption of the KFRPC conforms to Fickian's Law of diffusion. Diffusion coefficient ( $D$ ) of water absorption and maximum moisture content ( $M_m$ ) were calculated by measuring the water uptake of specimen at regular time interval.  $D$  and the  $M_m$  highest values were recorded for KFRPC immersed in DW followed by AS and SW. The mechanical properties were found to decrease with increasing of the water uptake. The decay in properties is attributed to the plasticization of the fiber-matrix interface and swelling of the kenaf fiber cause of hydrolysis. In thermal properties study, it was found that KFRPC immersed in SW reduced more mass percentage than AS and DW, respectively.

## **CHAPTER 1**

### **INTRODUCTION**

Fiber reinforced polymers (FRP) products are a class of composites and mostly used in engineering application. The uses have been steadily increased for the past several years (Muliana et al., 2006 and Katz & Neta, 2000). FRP is considered better than other conventional materials, such as steel and reinforced concrete due to its high stiffness-to-weight-ratio and good resistance to environmental agents and fatigue. For example, FRP's has successfully been used in the rehabilitation of existing bridge and have recently shown potential as construction materials in new infrastructure components.

Numerous applications of fiber-reinforced polymer (FRP) require high volume production and low cost of materials. Scientists and fiber producers are exploring the alternatives from agricultural natural fiber for utilization. Productions of nonwood pulp are mounted and the uses of kenaf fiber have drawn scientists' attention due to its high productivity and good fiber properties (Webber et al., 1999). In addition, kenaf fiber is biodegradable, low density, environmentally friendly and able to grow in a wide range of climatic conditions. The uses of kenaf fiber in automotive, marine, aerospace and furniture are not yet popular due to the domination of glass and metal uses. In order for natural fiber reinforced plastic to compete, it has to accommodate the existing FRP manufacturing technology.

Lots of manufacturing methods can be used to produce FRP products. These include hand lay-up, hot press and compression molding. In the case of construction and building applications the products are in the form of long and continuous



profiles. Several processing techniques are available to produce this kind of profile such as extrusion, filament winding and pultrusion. To date, a limited amount of work is reported on producing fiber-reinforced polymer (FRP) with natural fiber (NF) using pultrusion machine. For pultrusion, the fiber has to be in continuous form similar to its counterparts such as Aramid, glass, carbon, etc. as those synthetic fibers are readily available in roving, but it has difficulties to be produced in the roving form due to limitation in natural length. Pursuant to that, NF has to be made in yarn form in order to put them together such as twisting, plaiting etc. These arrangements have a huge implication processing aspects of FRP, especially when the pultrusion process is concerned because of losing fiber and poor resin infiltration as a result of geometry thickness of the resin and poor interfacial adhesion.

Pultrusion technique is more widely used commercially due to fast, easy to fix and continuous manufacture. The pultruded structure shapes of product include, I-beams, channels, round and square bar, and wide flange beams, round tubes, square tubes, flat sheets, and angle and concrete embedment angles. The structural shapes are used in a wide range of applications such as cellular communication, transportation, water and waste water, aeronautical defense, building construction and especially appliance and equipment.

The work has been done to focus on the natural fiber reinforced polymer composites manufacturing using the pultrusion technology process. Investigation of the processing parameters and the properties of the composites have been conducted. Current research on kenaf fiber is dealing with the fiber characterization and the acceptability of the fiber in pultruded composites. Other than that, the exposures to

various environmental conditions were carried out to evaluate the degradation and determine the properties of the composites.

### **1.1 Problem statements**

The first problem is most of the fiber reinforced polymers (FRP's) are made of synthetic fibers, which are usually expensive. Pursuant to the price of these synthetic fibers, the initial fabrication cost of FRP is relatively high. This factor has rendered FRP less competitive in the market. Alternatively, the use of natural fiber (NF) in producing fiber reinforced polymers (FRP) will directly reduce the cost of FRP and consequently, make FRP more competitive.

Secondly, so far, in manufacturing the natural fiber reinforced polymers (NFRP) there are a few research activities reported using the pultrusion method (Md Akil et al., 2010; Lackey et al., 2008(a); Angelov et al., 2007; Van de Velde & Kiekens, 2001; Tsang et al., 2000). The use of this method creates the potential of continuous production of profiles with a constant cross-section. Until now this method has been developed by the researchers to produce the natural fiber reinforced by using sisal, flax, hemp and jute but not on the kenaf fiber.

Thirdly, despite the substantial advantages of natural fiber reinforced polymers (NFRP's), some critical issues regarding their environmental durability, sustainability and long term performance still remain. It should be noted that previous research has demonstrated significant losses in their mechanical properties (Huang, 2009), integrity (Yu et al. 2006) when exposed to environments for prolonged period of time.

## **1.2 Statement of the Purpose**

The purpose of this research is to produce pultruded composite using kenaf fiber as fiber reinforcement and studies their performance properties.

## **1.3 Hypotheses**

- Increasing the kenaf fibers content will strengthen the kenaf fiber reinforced unsaturated polyester composites and make them a tougher natural fiber composite.
- Increasing immersion time of water absorption test in various environment conditions will weaken the kenaf fiber reinforced unsaturated polyester composites.

## **1.4 Research objectives**

The present study is aimed to develop a natural fiber reinforced polymer composite based on kenaf fiber and unsaturated polyester using pultrusion method. Degradation, physical, chemical, mechanical, and thermal properties of the natural fiber reinforced polymer was studied. The primary objectives of the study are listed below:

1. To analyze and characterize the degradation properties of kenaf fiber (KF) under various environment conditions such as distilled water, seawater and acidic solution in terms of chemical, mechanical and physical characteristic

2. To study and analyze the degradation properties of unsaturated polyester under various environment conditions such as distilled water, seawater and acidic solution in terms of mechanical properties.
3. To identify the effect of kenaf fiber content on the mechanical properties of kenaf fiber reinforced unsaturated polyester composite.
4. To determine the mechanical and thermal properties of pultruded kenaf fiber reinforced unsaturated polyester composites (70% of kenaf fiber volume) after immersion under different environment distilled water, seawater and acidic solution.

## **CHAPTER 2**

### **LITERATURE REVIEW**

Composites are one of the important groups of engineering materials. It had a long history of being manufactured and been utilized by human beings, but the use of composites materials started becoming a concern as early as 1908. Since then, composite materials were considered as an alternative material and the products from it were comparable and useable for various applications. Because of its credibility and accomplishment, the usage of composites has increased. The purpose of this project is to study one of the various types of composite namely kenaf fiber reinforced composites, so that they can be used in manufacturing industry. The literature review includes an introduction to composites; fiber reinforced composites, natural fiber reinforced composites, kenaf fiber and the analysis method of the pultrusion process.

#### **2.1 Introduction of composite**

Composite is a material consisting of two or more type of materials bonded into one product (Callister, 2003). It is initially being invested in United States for almost 102-years-old. However, in the 1940s the new market of composites was being utilized, and it started capturing the attention of the industries with the introduction of polymeric based composites. Composites were first applied on the fabrication of sheets, tubes and pipes for electronic purposes (Bledzki & Gassan, 1999). Then the composite materials had become more widely designed in various applications such as automotive components, aerospace structures, sporting equipment, consumer goods, marine and other manufacturing applications.

There are various types of composites. Composite has a type of structural, particle reinforced and fiber reinforced consisting of two sub-parts for each major type: sandwich panels and laminates, a large particle and a dispersion-strengthened, continuous and discontinuous fiber. The types of the composites are shown in Figure 2.1.

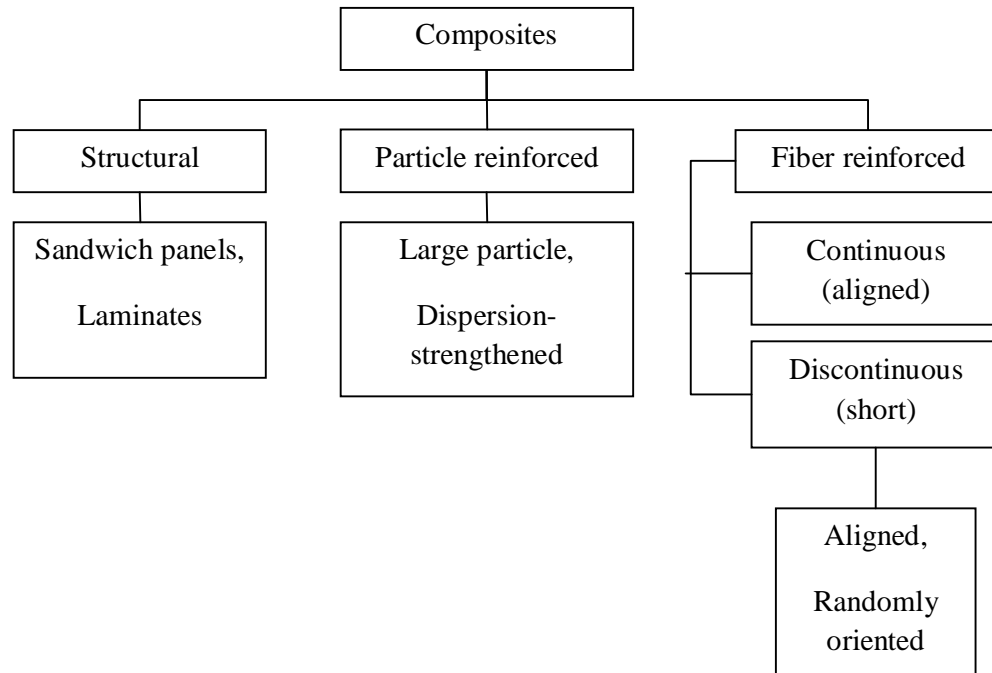


Figure 2.1 A classification scheme for the various composite types (Callister, 2003).

In each type, the properties are different depending on the structure, shape and the orientation of the materials in the composites. The structural types consist of laminates produced by stacking a number of plies in uniaxial-oriented fibers or woven fabric. The arrangement of plies shape can be in splay orientations according to the predetermined loading on the component structure. In sandwich structures, the less dense core of outer panels gave it high strength and stiffness. The shape of particle reinforced type is different from the structural type, where particles are in

spherical, ellipsoidal, polyhedral or irregular. The load will be shared by the particles and matrix where the matrix transfers some of the applied stress to the particles. According to Callister (2003), a large interaction of particle/matrix cannot be treated on the molecular level even in continuum mechanics uses. Finally, the fiber shapes of fiber reinforced are in the form of staple, whiskers with short and in continuous length. Variables that affect the properties are the length to diameter ratio of the fiber, strength interference between the fiber/matrix and the amount of the fibers. Among all the various types, further study is more focusing on the fiber reinforced composites.

#### 2.1.1 Fiber reinforced composites

Fiber reinforced composites consist of fiber material and embedded with the matrix. The fiber material that bonded in the matrix makes the composites high in modulus and strength (Mallick, 1993). The important thing to understand about fiber reinforced composites is that the fibers in composites are the load-carrying while the matrix surrounding the fibers is to protect it from environmental damages due to humidity and temperatures (Mallick, 1993). Moreover, the properties of the fiber reinforced composites strongly depend on the way the fibers are placed in the composites. Aligned fibers (long continuous fibers) in the direction of the load and the axis of the fiber result in greater strength than discontinuous fibers (short fiber). Other than that, the main concept of a fiber reinforced composite is the content of the matrix materials. The matrix materials can be polymers, metals and ceramics.

### 2.1.1.1 Classifications of fiber reinforced composites

The various combinations of fiber reinforced composites are usually known as polymer matrix composites (PMC), metal matrix composites (MMC) and ceramic matrix composites (CMC). There are different properties in each of the matrices composite materials. These materials are used by the industry to deal with the different kind of performance and characteristics. Fiber reinforced composites made of polymer matrix have become commonly used in various industries. The reasons why polymers were widely used as matrix material in composites are because the material is easy to handle and cheaper due to low pressure and temperature were needed. Unfortunately, the strength and stiffness of polymers are not enough for structure but by adding the reinforcement, it can improve the polymers properties. However, metal matrix composites (MMC) are good in conductivity, have metallic bond, crystalline in structure, operating in higher temperatures, resistance to degradation (Kainer, 2006) and fire-resistance compared to polymers. The combination with ductile materials such as metals makes them stiffer. The examples of metals are aluminium, magnesium, copper, and titanium. In ceramic matrix composites (CMC), fibers are added to brittle matrix material to increase toughness. The ceramics have high strength and high resistance to oxidation and deterioration of mechanical properties at high temperatures but are prone to brittleness. Other fibers reinforced composites are carbon-carbon composites and hybrid composites. Carbon-carbon composites have a high strength, fracture toughness, resistance to creep, and resistance to thermal shock. The carbon-carbon composites are commonly used in rocket engine components, thermal moulds, and biomedical implants. A new fiber-reinforced composite is known as the hybrid, consists of two or more different kinds of fibers in a single matrix. Applications of hybrid composites are for transport



structural components, sporting goods and lightweight orthopedic components (Callister, 2003; Cheon et al., 1999; Fink, et al., 2010).

#### 2.1.1.2 Processing of fiber reinforced composites

Manufacturing techniques can produce different kinds of fiber reinforced composites. It is depending on the type of application, whether to use it in structural or nonstructural. Commercially manufacturing techniques can be pultrusion process, preprag lay-up and filament winding. The pultrusion is a low cost continuous production method producing minimum waste with a high volume manufacturing process (Mazumdar, 2002). The process is done by pulling reinforced fiber through a resin to coat the reinforcement. Then, pre-form plates are use to performed the shape and lastly it is pulled through the heated die to cure the resin. Other techniques are by using preprag lay-up where the material is in tape form which calendaring rollers press the carrier paper, the heated resin sheet and the continuous fibers together to form a tape. This process required number of plies layer unidirectional before curing at required temperature and pressure. Another process of manufacturing the fiber reinforced composites is by filament winding technique. In this process the fibers are wound into cylindrical shape and fed through resin bath then wound on a mandrel to a required shape and thickness before curing in an oven. The products that are made using this technique are pressure vessels, pipes, and helicopter rotor blades. All the processes are directionally to bring the reinforcement and matrix together in the form of fiber reinforced composites to give an improved interfacial bond.

## **2.3 Fiber reinforced polymer (FRP) composites**

Fiber reinforced polymer (FRP's) has been successfully used in various applications, especially in construction and building industries. These composites offer alternative materials to steel and aluminum for similar applications. There are various aspects that had encouraged the use of these composites such as high adaptability, ease of handling, damage tolerance and low cost of fabrication (Rowell, 2007). While Katz and Neta (2000) reported fiber reinforced polymer (FRP) exhibit corrosion resistance and is electromagnetically transparent. There were two types of fiber reinforced polymer composites namely synthetic and natural fiber.

### **2.2.1 Synthetic fiber reinforced polymer composites**

To date, most fiber reinforced polymer composites are produced using synthetic fibers typically glass fiber, carbon fiber and, etc. Glass fiber is the common fiber reinforcement for polymers and widely used because it increases the strength and stiffness properties of the polymers. It is the cost-effective and the most proven way of reinforcing polymers to increase tensile and flexural modulus and strength due to the carefulness produced in controlled, uniform and symmetrical shapes with high aspect ratios (Tolinski, 2009). Other than glass fiber, carbon fibers were used as reinforcement in polymers. Carbon fiber is stronger, lighter than glass fiber but prohibitive expensive for most reinforced applications (Tolinski, 2009). Although carbon fiber is expensive, it was already being used in thermoset composites in aerospace and high-end applications. Fiber reinforced polymer composites using synthetic fibers are not very competitive in terms of price as in comparison to steel and galvanized steel.

### 2.2.2 Natural fiber reinforced polymer composites

Based on the research, the main focus is totally on natural fiber reinforced polymer composites. In order for the fiber reinforced polymer composites to advance as one of the competitive materials in construction, the price has to be more competitive. One way of making it more competitive is by reducing the cost of fiber. The use of natural fiber (NF) in place of synthetic fiber will tremendously reduce the cost of fiber reinforced polymer due to the low prices of natural fiber (NF) as well as to lighten the products, reduce brittleness and improve wear qualities. Tolinski (2009) reported that, the natural fibers exhibit similar properties as glass fiber, but with lighter in weight and consume less energy in production. While Herrera-Franco and Valadez-González (2004) reported that, the natural fiber is able to impart the composite in certain benefits such as low density, fewer machines wear, no health hazards and a high degree of flexibility in comparison to the glass and carbon the traditional engineering fibers. Other than that, Lawton and Fanta (1994) and Joshi et al. (2004) sighted in Singha and Thakur (2008) reported that natural fiber reinforcing materials better than glass and carbon fibers by their specific strength properties, easy availability, ease of separation, enhanced energy recovery, high toughness, non-corrosive nature, good thermal properties, reduced dermal and respiratory, renewability, biodegradability and non-toxicity (Tserki, et al., 2005). In this report, the details of natural fiber characteristic, gained by using kenaf fibers to produce natural fiber reinforced polymer composites will be reviewed.

## **2.3 Natural fiber**

Nowadays, natural fibers are becoming very widely known and extremely valuable due to their characters. Most of the researcher and engineering interest had attempted to use natural fibers in product applications by shifting from monolithic materials to fiber-reinforced polymeric material composites (Wambua et al., 2003; Joshi et al., 2004; Reis, 2006; Alves et al., 2009). Numerous research works were generated concerning natural fibers based on plant fibers (Herrera-Franco & Valadez-González, 2004; Silva-Andrade et al., 2008; Liu et al., 2010; Murali Mohan Rao, 2010). Natural fiber (NF) has good specific strengths and modulus, economical viability, low density and no risk to human health when the particle is inhaled and biodegradability (Chawla, 1998; Joseph et al., 2002; Mohd Edeerozey et al., 2007). There are varieties of natural plant fibers used as reinforcement fiber for fiber-reinforced polymer (FRP's) such as hemp, sisal, and jute and recently kenaf fiber. In the past 200 years, India has produced and used the kenaf as crop applications but still northern Africa was the first to domesticate the uses of kenaf (Webber & Bledsoe, 2002c).

### **2.3.1 Kenaf fiber**

Kenaf has a long history of being planted by people. It is a 4000-year-old crop that is being used by the native in ancient Africa. The usage of kenaf products is increasing due to low cost and easy to produce. Other than that kenaf is biodegradable and environmental compatibility.

Kenaf is the common name given to the fiber extracted from the bast of stems of plants belonging to genus *Hibiscus*, family *Malvaceae*, especially the species *H.cannabinus L.* This plant has a single, straight stem consist of an outer and inner bark. Normally, these kenaf plants grown from seeds but some are grown as perennials at the tropics and subtropics area. Kenaf can grow up to 2.5 and 3.5m height in optimum temperature between 22 and 30°C with a minimum annual moisture requirement of 150 mm in a soil pH between 6.0 and 6.8, shown in Table 2.1 (Rowell & Stout, 2007). Kenaf is an annual plant due to its rapid growth and considered as the renewable source with good fiber quality (Kaldor, 1992) and inexpensive (Nishimura et al., 2002). Abdul Khalil et al. (2010) have studied the characteristics and physical properties of kenaf fibers that are cultivated in the region of Malaysia. As results of intensive research, kenaf plantation in Malaysia can be harvested three times a year instead of once a year as reported by many locations other than Malaysia.

Table 2.1 Climatic requirements for growing kenaf (Rowell & Stout, 2007)

Plant	Optimum temperature (°C)	Minimum moisture (mm)	Optimum soil pH	Growing cycle (days)	Fiber yield (kg/ha)
Kenaf	22 - 30	120	6.0 – 6.8	150 - 180	1700

Kenaf fibers develop in the layer of bark in the stem of the plants. The kenaf plants are usually height up to 3.5 m and the diameter of basal is about 25 mm when it reaches a mature time. Then it is important to harvest the kenaf plants in precise time when about ten flowers are bloom and after older flowers have set their seed. Each kenaf plant harvested components had its purpose, which are; seeds gave oil and protein composition; leaves and shoot are forage; the core stem attributes for

forest-product substitutes, absorbents and structural materials (Webber & Bledsoe, 2002b). During harvest, the plants are cut close to the ground and the leaves were removed from stem. Then the stems are tied into bundles to submerge in water for retting. Retting is a process to remove the unwanted bark material (Webber et al., 2002a) and separate the outer layers of the stem from the core. The process takes 5 to 30 days for completion using moisture with bacteria or chemical/ enzymes. The retting must be in specific condition degree where the fibers can easily be stripped and not be weakened by microorganisms in order to produce good quality fiber (Rowell & Stout, 2007).

### 2.3.2 Structure of kenaf fiber

Kenaf is a straight quickly grow plants. It grows up to 4-5 month growing season with 25 to 35 mm in diameter. Parts of kenaf stem were shown in Figure 2.2 by Nishimura et al. (2002) for their sampling research. Each sample was prepared

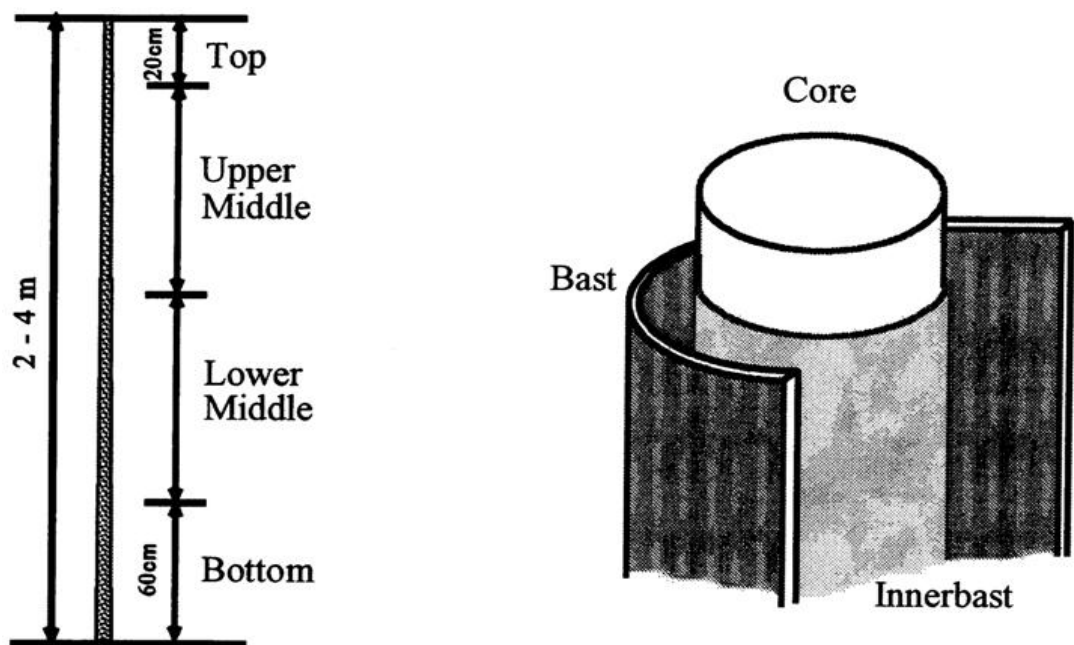


Figure 2.2 Parts of kenaf stem (Nishimura et al., 2002).

from four different position and three parts; bast, inner bast and core. The core of the kenaf plant is in a spongy tissue under the bast of the fiber. They studied the structural characterization of kenaf lignin among three different of kenaf: Everglades (*Hibiscus cannabinus L.*), Aokawa-3 (*H. cannabinus L.*), and Mesta (*H. sabdariffa L.*). The report show that the bast contained lignin in lower amounts than the core, while inner bast showed similar results to the core from the same positions.

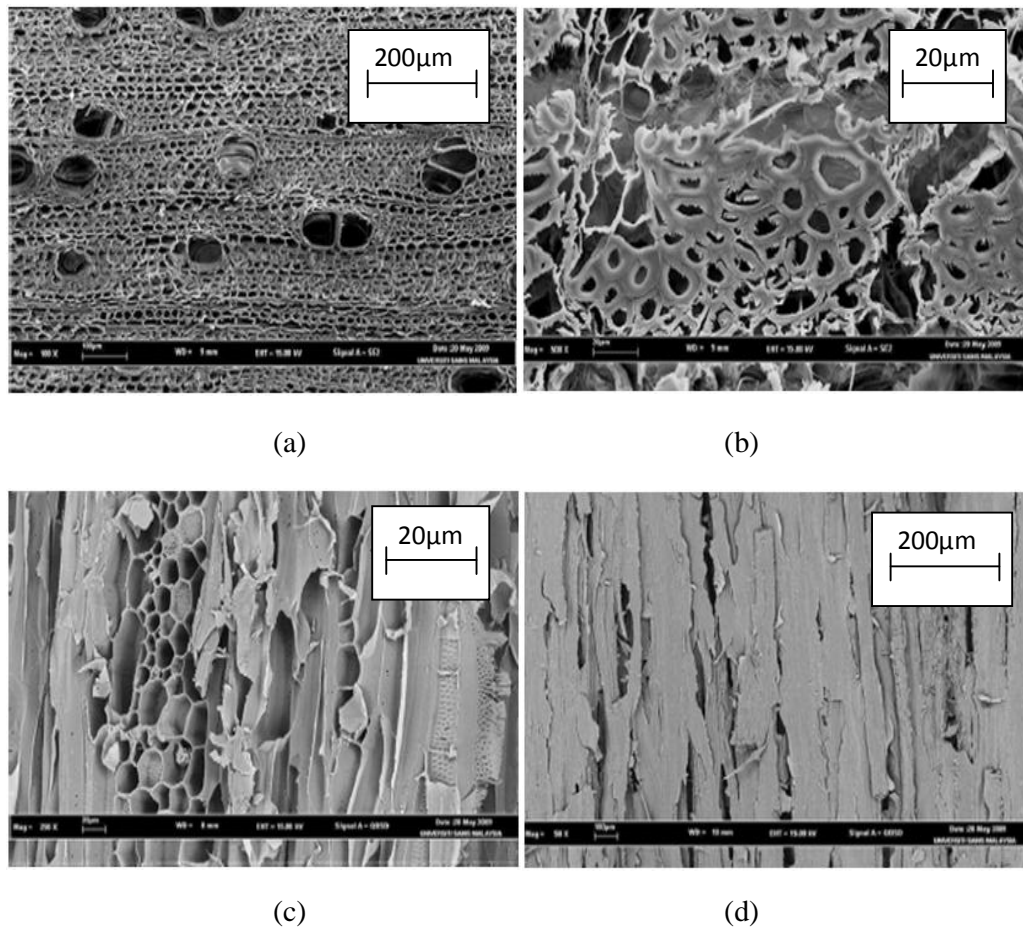


Figure 2.3 Scanning electron micrograph of kenaf fiber (a) transverse section of core fibers; (b) transverse section of bast fibers; (c) longitudinal section of core fibers; (d) longitudinal section of bast fibers (Abdul Khalil et al., 2010)

Bast fibers are the bundles of specialized cells called sclerenchyma (Brian et al., 2009) which the cell walls are a very thick cause of the cellulose microfibrils oriented along the fibers. Generally, sclerenchyma combines with xylem and phloem (tissue) to transport water and sugar away along the plant. In the vessel member walls and surface of core present small pits and can be viewed in Figure 2.3. Some basic characteristic properties of kenaf stem were shown in Table 2.2 by Abdul Khalil et al. (2010). Abdul Khalil et al. (2010) studied the kenaf cell wall ultra structure, lignin distribution and chemical composition of Malaysian cultivated kenaf fiber. It shows the data of dimension ranged, density, the pH and the proportion of bast, core and stem of the kenaf stems. This fiber characteristic is depending on the maturity of the plant, cultivars, location and climate.

Table 2.2 Characteristic and properties of kenaf stems (Abdul Khalil et al., 2010).

Characteristic/ Properties	Bast	Core	Stem
<b>Dimension (cm)</b>			
Height (range)	-	-	145 – 250
Diameter	-	152 (0.095)	1.74 (0.212)
Perimeter	-	5.73 (0.131)	6.60 (0.101)
<b>Proportion (%)</b>			
Cross-section area	21.96 (2.03)	78.04 (2.51)	-
Weight proportion	32.2	68.5	-
<b>Density (g/cm<sup>3</sup>)</b>			
		0.21 (0.038)	0.29 (0.044)
<b>Acidity (pH)</b>			
	7.13	5.21	5.87

*Value in the ( ) shows standard deviation value*



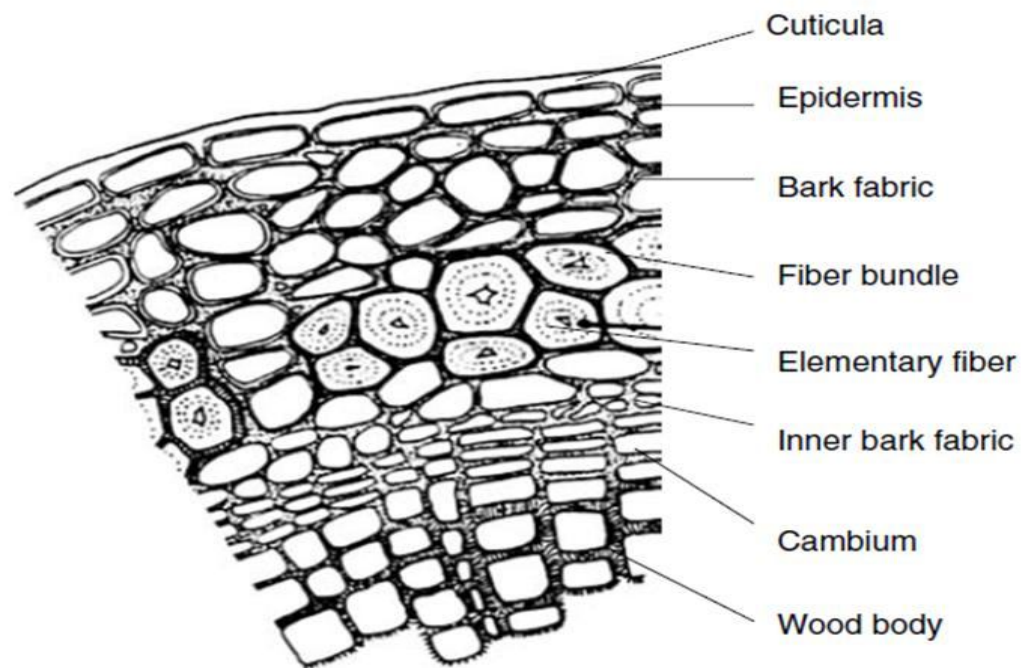


Figure 2.4 Section through a typical stalk (Munder et al., 2005 source from Ruschmann, 1923).

In each growing plant, the main part of the fibers contains in stalks of the outer bast fiber plants. The stalks consist of rings fiber from a tubular in the stem from top to bottom as shown in Figure 2.4. The fiber bast has thicker walls where it consists ~65% of cellulose and ~21% of lignin and pectin, and other compositions. The stalks of fiber content increases with the length growth and age of the plants (Munder et al., 2005). Generally, at the bottom of the stalk the fibers' lengths are shorter and longer at the top. The fiber length will be increased at the early part of the growing cycle and then decreased again when the plant matured. Each part of a plant have different physical and chemical properties.

### 2.3.3 Chemical composition of kenaf fiber

Generally, every part of plant has different chemical and physical properties as well as different in state, place and growing season. The changes occur along the stalks in the plant raw materials. There are three chemical constituents in kenaf fibers namely  $\alpha$ -cellulose, hemicelluloses and lignin. Table 2.3 shows the general data on the chemical compositions of kenaf while in Table 2.4 shows the data according to different researchers. The  $\alpha$ -cellulose is tracing sugar residues and glucose with composition of 55 to 59% of glucosan, 1.8 to 3.0% of xylan, 0.8 to 1.2% of glucuronic acid, together with galactan, araban, mannan, and rhamnosan. Hemicellulose are isolated from  $\alpha$ -cellulose by treatment with dilute alkali consist of a chain of D-xylose residue and lignin gives 8 to 12.5% of xylan, 2 to 4% of galactan, 3 to 4% of glucuronic acid, together with araban and rhamnosan (Rowell & Stout, 2007). Other than that, kenaf fibers contain minor constituents such as 0.4 to 0.8% of fats and waxes, 0.3 to 5% of inorganic matter, 0.8 to 1.5% of nitrogenous matter and pigments (Rowell & Stout, 2007).

In a recent study, Webber & Bledsoe (2002c) and Rowell & Han (1999) reported that in early 1960's; Clark & Wolff (1962, 1965, 1969) and Clark et al. (1971) were carried out a lot of research about kenaf. At that time area, kenaf was a new fiber crop, and they determine that the source was a good in properties due to less energy and chemical for processing the paper products. The researchers also carried out the first study on the changes in chemical composition of kenaf including the stem and between leaves and stem as a function of the growing season (Rowell & Han, 1999). Ohtani et al. (2001) also studied the influence of the chemical composition of kenaf, but it is between bast and core on the alkaline pulping

response. They found that bast and core were different in respect to chemical components and alkaline pulping response of whole kenaf.

Table 2.3 Chemical composition of kenaf fibers (Rowell, 2008).

Type of fiber	Cellulose	Lignin	Pentosan	Ash
<b>Core fiber</b>				
Kenaf	37 - 49	15 - 21	18 - 24	2 - 4

Table 2.4 Chemical compositions of kenaf fibers on various places grown. (Rowell, 2008).

Scientific name	Common name	Place grown	cellulose	Lignin
Hibiscus cannabinus	Kenaf, hurds	Illiniois, US	34.7	15 - 18
Hibiscus cannabinus	Kenaf	Florida,US	34.0	10.5
Hibiscus cannabinus	Kenaf, stem	Maryland, US	36.5	13.2
Hibiscus cannabinus	Kenaf, stem	Gorgia, US	40.2	7.7
Hibiscus cannabinus	Kenaf, whole	-	37.4	8.0
Hibiscus cannabinus	Kenaf, bast	-	42.2	17.4
Hibiscus cannabinus	Kenaf, core	-	33.7	13.4
Hibiscus cannabinus	Kenaf, bottom	-	35.3	-
Hibiscus cannabinus	Kenaf, top	-	29.8	-
Hibiscus cisplantinus	Kenaf	North Carolina,US	30.5	-
Hibiscus eelveldeanus	Kenaf	Florida, US	36.3	-

Table 2.4 Continued.

Pentosans	Alcohol benzene	Hot water	1% NaOH	Ash
-	3.7	-	30.9	-
21 – 23	3.4	-	29.4	-
22.7	4.3	11.2	33.0	-
19.7	3.3	7.4	28.4	-
16.1	-	-	-	4.1
16.0	-	-	-	5.5
19.0	-	-	-	2.9
20.1	3.4	9.7	32.4	-
-	5.5	12.8	39.6	-
-	2.5	-	33.2	-
-	7.5	-	31.0	-

#### 2.3.4 Chemistry of natural fibers

The major components of natural fibers substances are cellulose, hemicelluloses, lignin and pectin regard to the physical properties of the fibers. The chemical compositions that occur are changes along the stalks of the plant raw materials. Generally, the content of  $\alpha$ -cellulose, lignin and ash decrease from the base of the stalks to the top of the natural plant because mature tissues (at the base) accumulate higher amounts of metabolic products than the younger parts at the top (Abdul Khalil et al., 2009). The differences of components in chemical characterization of along natural fiber also been studied by Morrison et al. (1999), Nishimura et al. (2002) and Komiyama et al. (2008).

### 2.3.4.1 Cellulose

Cellulose is the main component of all plant fibers; the name was suggested by Anselme Payen in 1838 due to the largest numbers of cell walls of the plants (Bledzki & Gassan, 1999). The structure of cellulose is  $(C_6H_{10}O_5)_n$  consists of  $-CH_2OH$  and  $-OH$  groups. Cellulose is a polydisperse linear homopolymer consisting of region- and enantioselective  $\beta$ -1,4-glycosidic linked D-glucose units (Heinze & Liebert, 2001). The chemical structures descriptive of the principal cellulosic fibers are shown in Figure 2.5. Each repeating unit contains hydroxyl groups. The hydroxyl groups form intermolecular bonds and one of it forms intermolecular hydrogen bonds.

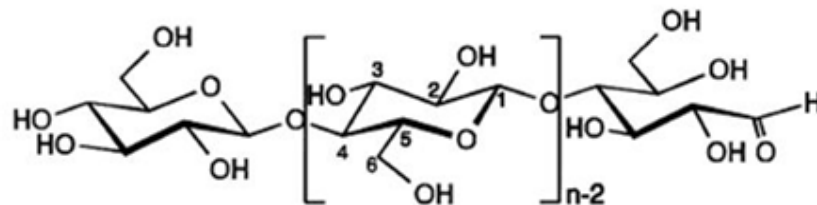


Figure 2.5 Chemical structures of cellulose (Heinze & Liebert, 2001).

In general, the cross-sectional shapes of cellulose differ from one plant to another. For example, cellulose of kenaf is larger in terms of cross-sectional shape in comparison with cotton fiber. Summary of cross-sectional shape for several plant fibers is summarized in Figure 2.6. There are different cross-sectional shapes of cellulose. It is because each type has its own cell geometry and the conditions determine the mechanical properties. Cellulose represents a vast potential feedstock for a number of industries and has created a great deal of research interest such as the production of liquid fuels (alcohol), pharmaceuticals, food and chemical feedstock.

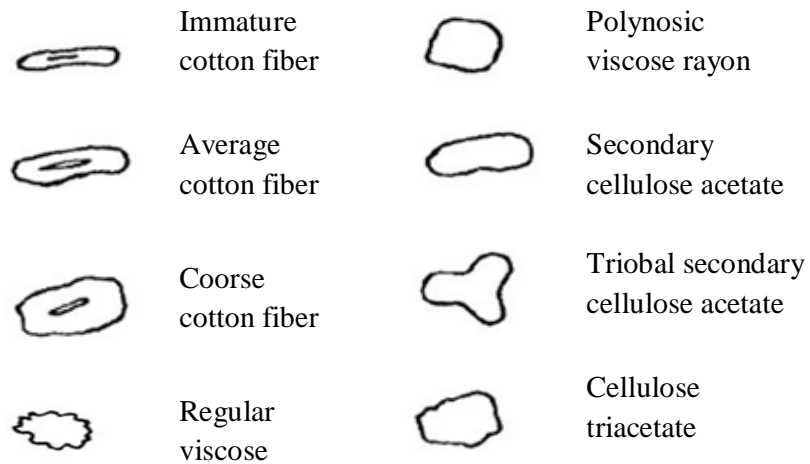


Figure 2.6 Cross-sectional shapes of some cellulosic fibers (Rebenfeld, 2002).

#### 2.3.4.2 Hemicelluloses

Hemicellulose is not a form of cellulose and it is different from cellulose. It is known as misnomer, which comprises a group of polysaccharides. The group is composed by a combination 5- and 6-carbon ring sugars. The differences of hemicelluloses than cellulose can be categorized in three matters. Firstly, it is due to the content of sugar monomers units such as xylose, mannose galactose rhamnose and arabinose, rather than cellulose that only consist 1,4- $\beta$ -D-glucopyranose units. Other than that, hemicelluloses exhibit considerable degree of chain branching whereas cellulose is in linear polymer. Finally the different in degree of polymerization of native cellulose is 10 to 100 times higher than hemicellulose (Bledzki & Gassan, 1999). The degree of polymerization of hemicelluloses is around 50 to 300. Figure 2.7 represents the example structures of main hemicelluloses (Curling et al., 2001).

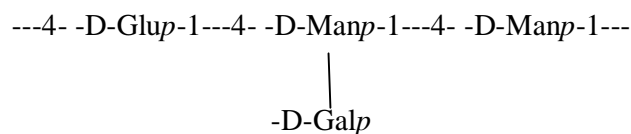


Figure 2.7 Representative structures of the hemi-cellulose components. (*Glu* = glucose, *Man* = mannose, *Gal* = galactose) (Curling et al., 2001).

### 2.3.4.3 Lignin

As the third most abundant natural polymer present on earth, lignin comes after cellulose and hemicelluloses. Lignin present in all natural fibers. Physically, lignin in cell walls protects most of the cellulose and hemicelluloses from enzymatic hydrolysis (Fioretto et al., 2005). Lignin is also chemically known as phenylpropane which is an amorphous polymer. The precursors of phenylpropane units are aromatic alcohols (monolignols) namely  $\rho$ -coumaryl(1), coniferyl(2) and sinapyl alcohols(3), as shown in Figure 2.8 (Buranov & Mazza, 2008). In most solvents, lignin cannot be soluble and it is generally hydrophobic in nature. Elementary analysis of lignin indicates different composition for different types of plant and making up 15% to 36% of its dry weight. Humphreys & Chapple (2002) reported that lignin has far-reaching on agriculture industry and the environment, making phenylpropanoid metabolism a globally important part of plant biochemistry (Humphreys & Chapple, 2002). Other than that, lignin is the major by-product in bioethanol production and as the separator of cellulose from wood for pulp and paper (Guozhan et al., 2009).

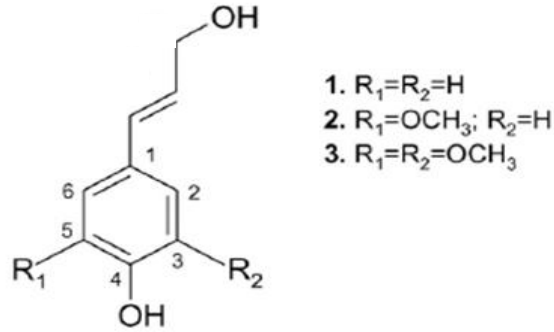


Figure 2.8 Probable structure of lignin (Buranov & Mazza, 2008)

#### 2.3.4.4 Pectin

Pectin is the component of the cell walls of all plants (Willats et al., 2006). It is a family of oligosaccharides and polysaccharides that contain 1,4-linked  $\alpha$ -D-galactosyluronic acid (GalpA) residues (Ridley et al., 2001). Figure 2.9 illustrates some of the major domains found in most pectins rather than definitive structures (Willats et al., 2006). It is mostly used as functional food ingredient in food industry.

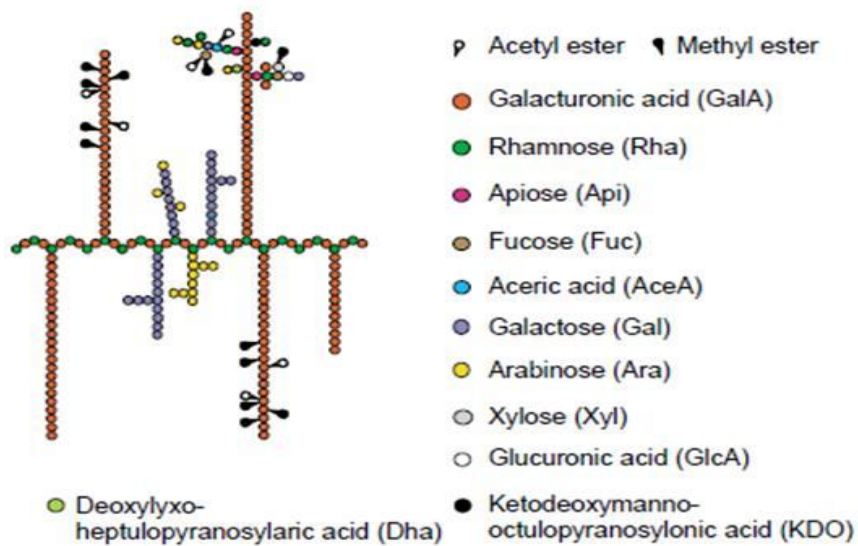


Figure 2.9 Schematic representations of the basic structure of pectin (Willats et al., 2006).