hemicellulose. The degree of polymerization for hemicellulose microfibrils was presumably by hydrogen bonding. Because of its open structure containing many hydroxyl and acetyl groups, hemicellulose is hygroscopic, partly soluble in water, soluble in alkali and easily hydrolyzed in acids.

Research by Bismarck et al. (2005) showed lignin is the only component in plant fiber that remains obscure. Lignin is a complex hydrocarbon polymer with both aliphatic and aromatic constituents. They are totally insoluble in most solvents and cannot be broken down to monomeric units. Lignin is generally amorphous and hydrophobic in nature. It is the compound that gives rigidity to the plants. It is thought to be a complex, three-dimensional copolymer of aliphatic and aromatic constituents with very high molecular weight. It is believed that the structural units of lignin molecules are derivatives of 4-hydroxy-3-methoxy phenylpropane with hydroxyl, methoxyl, and carbonyl groups per building unit.

According to Craig & Beyler (1993), thermal decomposition is a process of extensive chemical species change caused by heat". Thermal degradation is a process whereby the action of heat or elevated temperature on a material, product, or assembly causes a loss of physical, mechanical, or electrical properties. According to Anis et al (2012), initial degradation temperatures of kenaf are 73°C, while maximum degradation temperatures are 345 °C with char residues at 38.43%. According to Morsyleide et al. (2009) argue that degradation of kenaf fiber curve includes two main degradation stages. The first stage is attributing to the evaporation of water between room temperature to 150°C and 190°C is believe correspond to degradation of hemicelluloses while, the second stage corresponding to thermal degradation of cellulose. Kim (2010) agreed that

higher degradation temperature of fiber is related to crystallite structure of fiber itself. The increase of crystalline index and crystallite size provided higher thermal stability of the fiber. A greater crystallite structure required a higher degradation temperature (Sherif & Mohammad, 2011; Yang & Kokot, 1996).

Natural fibers usually contain cellulose, hemicellulose and lignin. This applies to kenaf fiber as well. The degradation of the cellulose begins around 250°C because the cellulose is a homopolymer of glucose. It is a semi crystalline polymer. It is nearly decomposed totally into volatile product at 400°C (Patrick, Ian, André, & Patrick, 2006); Golnaz, 2007); Patricia, Alon, Aitor, Jose, & In, 2009; Tantaley et al.). The lignin is a polyphenolic polymer formed with three elementary motifs, the coumarylic, sinapylic, and coniferylic alcohols. It has a tridimensional structure. The polymerisation of the lignin is randomly shared at the vegetable secondary wall and its structure varies from one species to one another (Panshin & Zeeuw, 1980). Even if the lignin is less stable than the cellulose, its degradation is very slow with the high rate of non-volatile products.

Several studies confirmed the extensive of the range of the degradation of the lignin in two or three stages (Jaroslav, 2005; Jakab, Faix, & Till, 1997; Sun, Lu, & Sun, 2000; Franceschi, Luciano, Carosi, Cornara & Montanari, 2004); Tantaley et al.). The breakdown of hemicellulose by heat begins around 140°C (284°F) and becomes exothermic - i.e., the process liberates heat at temperatures above 225°C (437°F). Toasting yields furfural, hydroxymethyl furfural, maltol, cyclotene6 and a host of other sugar condensation products en route to the highly condensed structures which give the brown color of caramel. Acetic acid and methyl alcohol are also formed. Thus the breakdown of hemicellulose yields wood sugars which add to the body of the matured

product, toasty flavors and color. With the exception of furfural these compounds have sweet-associated burnt sugar or caramelized aromas and flavors. In addition, Ramiah (1970) proved there are numerous other compounds released during toasting which have similar characteristics.

According to Espert et al. (2004), one of the most important parameter effects on the mechanical properties and dimensional stability of the composites is the poor resistance of the fibers to water absorption. Moisture absorption into the composite materials can be accounted by three major mechanisms. First is the diffusion of water molecules inside the microgaps between polymer chains; second is the capillary transport of water molecules into the gaps and flaws at the interface between fibers and the polymer due to incomplete wet ability and impregnation; finally, the third mechanism is the transport of water molecules by micro cracks in the matrix that are formed during the compounding process (Ahmad et al., 2010; Ghasemi & Kord, 2009).

When exposed to humid environments, glass fiber-epoxy composites absorb moisture and undergo volumetric expansion. Moisture and the stresses associated with moisture induced expansion can result in low damage tolerance, with an adverse effect on long-term structural durability. Differential volumetric expansion of the matrix and the fibres is observed due to moisture absorption, and this further leads to the generation of localized stress and strain fields in the composite. The fractured sample from tensile testing was used for swelling testing.

#### 2.2.1 Kenaf

Kenaf or its scientific name (*Hibiscus cannabinus*), originating from Africa, has traditionally been a source of bast fiber in India and China, which together account for more than 75 percent of worldwide kenaf production. Research on kenaf first began in the United States in 1957 and has continued sporadically since that time by White and Higgins (1964). Moreover, an attractive feature of kenaf found by Nimmo (2002) is that up to 40 percent of the stalk yields usable fiber, roughly twice that of jute, hemp or flax, which makes the fiber quite economical. Also, the plant can grow from seed to heights of 3.6 m to 4.3 m (12 ft to 14 ft) in five to six months. Kenaf fibers come from the bast of the plant, and have been widely used as a jute-like material in the past.

A diagram of the classification of various fibers is presented in Figure 2.1. Asian kenaf is cultivated predominantly for the fiber, and soaking of the stalks during the retting process, combined with manual removal of the fibers, is believed to result in superior reinforcement quality.



Figure 2.1: Classification of natural fibers (Harriette, 2004).

Comparatively, U. S. kenaf producers, including Kenaf Industries of South Texas (Raymondville, Texas) and Greene Natural Fibers (Snow Hill, N. C.), use field retting and mechanical separation. The kenaf core materials, which is can be used as an absorbents and animal bedding, have more ready markets. While both companies supply some fiber to the composites market, they also are pursuing other applications, such as extruded plastic fencing and decking, and furniture padding (Brosius, 2006; Suwinarti, et. al., 2005). Today kenaf fibers as reinforcement of composite material arouse researcher's interest.

### 2.2.1.1 General characterization of kenaf fibers

Raw kenaf fiber obtained from outer bark, is actually a bundle of lignocellulosic fibers. The fiber bundle size depends on the number of ultimate cells in each bundle. Most lignin is present between the ultimate cells. Kenaf contains approximately 0.7 % of cellulose, 21.6 % of lignin and pectin, and other components (Ramaswamy, et. al. 1995). Lignin must be extracted to separate the fibers (Tao et al., 1997; Calamari et al., 1997). Generally, kenaf fibers are shorter at the bottom of the stalk and longer at the top. The increase in length from the bottom to the top was not gradual, but S-shaped (Han, et. al. 1997). There is more variation of the fiber length at the top of the stalk. Also, the longest fibers are located at the top. On the other hand, different parts of a plant have different chemical and physical properties. That is, the chemical compositions and fiber properties of plant tissue taken from the roots, stem, trunk and leaves are different and are also different at different stages of the growing season (Rowell & Han, 1999). Fiber length increases in the early part of the growing cycle, and then decreases again for the plant

matured (Cho et. al. 2001). This may be an advantage in harvesting of the fiber before the plant matures.

# 2.2.1.2 General characterization of mechanical properties of kenaf fibers and kenaf fibers composites.

The tensile properties of kenaf single fibers have been reported by Bolton (1994). Kenaf single fibers exhibit tensile strength and tensile modulus of 11.91 GPa and 60 GPa respectively. Kenaf fibers have been used as reinforcement in thermoplastics such as polyethylene by Chen & Porter (1994), polypropylene (Rowell et al., 1994-1995; Chen et al., 1995; Karnani et al., 1997) and thermosetting resins like polyester (Aziz et al., 2005) and polybenzoxazine (Dansiri at al., 2002). Unidirectional (UD) composites of polyethylene and kenaf fibers have been studied. The fibers were untreated and coupling agent has been used. Tensile properties of the UD composites have been tensile tested and it was reported that kenaf fibers enhanced the tensile properties of polyethylene.

Indeed, the tensile modulus of the UD composite with 57 vol. % of fibers was 7 times as much as tensile modulus of polyethylene, and its tensile strength was 4 times higher. Many research projects have been carried out to determine the mechanical properties of kenaf fibers reinforced polypropylene (Rowell et al., 1994-1995; Chen et al., 1995); Karnani et al., 1997; Feng et al., 2001) and described polypropylene composites with high kenaf fiber content (Sanadi et al., 2005). The purpose was to find an alternative to wood particles, and low or medium density hardboards. The plasticization is the process used to reach this high fiber content. The study reports much higher flexural properties of kenaf/PP containing 85 % or 60 % of kenaf if compared with

wood fiber boards. The effect of the maleic anhydride (MAH) as a coupling agent has been intensively investigated (Sanadi et al., 1995; Chen et al., 1995; Karnani et al., 1997) and significant increase in flexural and tensile strengths was reported for maleated polypropylene (2 wt. % of MAH). However, no significant difference can be observed between kenaf fiber reinforced polypropylene and kenaf fiber reinforced maleated polypropylene (MAPP) regarding the tensile and flexural moduli. The tensile and flexural moduli increase with increasing fiber content. The positive effect of the maleic anhydride agent on the bonding of kenaf/PP has been reported. Besides, it was shown that the specific tensile and flexural moduli of kenaf/PP composite with 50 wt. % of fiber content (7.2 and 7.3 GPa, respectively) have comparable values as for the glass fiber/PP composite with 40 wt. % of fiber content (7.3 and 5 GPa, respectively). The papers (Sanadi et al., 1994) and (Feng et al., 2001) also reported better adhesion between kenaf fibers and polypropylene in the presence of maleic anhydride.

Karnani et. al. (1997) focused their research on the improvement of kenaf/PP interfacial adhesion. Maleic anhydride was used as a coupling agent and kenaf fibers were treated with silane (2 wt. % in water). SEM observations of kenaf/MAPP composite show better wettability of the fibers in comparison with kenaf/PP composites. At 20 wt. % of fiber content, the tensile strength and modulus increase from 26.9 MPa/2.7 GPa (PP without MAH) to 38.1 MPa/3.2 GPa (PP with 5 % of MAH) (Karnani et. al.1997). There was also reported by Aziz et al. (2005) that a significant mechanical improvement after surface modification of kenaf fibers with silane (42.5 MPa/3.3 GPa at 20 wt. % of fiber content). Selections of four different polyester resins have been used in composites containing kenaf fibers (Aziz et. al. 2005). One of them was a conventional unsaturated

polyester, the others have been modified to improve the adhesion to natural fibers (*e.g.* make them more polar).

An alkali surface treatment of the fiber was done by using of 6 % NaOH solution. Composites with 60 vol. % fibers content have been produced and tested in bending. Modified polyester exhibited better flexural properties. Nishino et al. (2003) investigated the mechanical properties of a composite made of kenaf fiber and poly-L-lactic acid (PLLA). Young's modulus (6.3 GPa) and tensile strength (62 MPa) of the kenaf/PLLA composite (fiber content 70 vol. %) were comparable to those of traditional composites. The effects of the molecular weight of PLLA, and orientation of the kenaf fibers in the sheet on the mechanical properties of the composite were also investigated.

This composite showed superior mechanical and thermal properties based on the strong interactions between the kenaf fibers and PLLA matrix. Both the anisotropic and quasi-isotropic composites could be obtained by lamination of kenaf sheets with preferential orientation of the fiber. Table 2.1 shows a comparison of literature data concerning mechanical properties of kenaf *versus E* glass (low alkali borosilicate glass), indicating that kenaf fiber can be a good reinforcement candidate for high performance biodegradable polymer composites (Nishino et. al. 2003).

Fibers	Properties						
	Density	Tensile	e-	Specific	Elongation	Cellulose/	Microfibril
	$(g/cm^3)$	strength	modulus	(e/density)	at failure	lignin	angle*,deg
		(MPa)	(GPa)		(%)		
e-	2.6	2000	76	29	2.6	-	-
glass							
kenaf	1.5	350-	40	27	0.33-0.88	75-90	9-15
		600					

Table 2.1: Kenaf fibers and e-glass fibers properties (Almn, 2006).

\*the term micro fibril angle(MFA) in wood science refers to angle between the direction of the helical windings of cellulose micro fibrils in the secondary cell wall of fibers and tracheid and the long axis of cell. Technologically, it is usually applied to the orientation of cellulose micro fibrils in the s2 layer that makes up the greatest proportion of the wall thickness, since it is this factor which most affects the physical properties of wood.

Research by Dansiri et al. (2002) shows composite with 60 vol. % fibers content have been produced and tested in bending. One of the composites, reinforced with 56 vol. % of fiber content, had respectively almost 2 and 3 times higher flexural modulus and strength in comparison to an unmodified composite with higher fiber content (63 vol. %). A moisture absorption test showed a weight increase divided by 3 if compared unmodified ( $\pm 60$  %) and modified polyester composites ( $\pm 20$  %). The biodegradable polymer poly-L-lactic (PLLA) was used to produce kenaf fiber reinforced composites with a 70 vol. % of fiber content.

Interesting tensile properties were reported and were attributed to the strong bonding among kenaf fibers and PLLA. UD composites with 20 wt. % of kenaf fiber using polybenzoxazine (PBZX) resin matrix produced and investigated by authors Dansiri et al. (2002) were tested in bending and it was shown that they had lower flexural strength but higher flexural modulus if compared to the composites made of unsaturated polyester resin.

#### 2.2.1.3 Use of kenaf

Today, one of the major values of kenaf is its use in some countries in a limited scale, in the production of pulp and paper as substitute of wood (Nimmo, 2002). Kenaf offers many significant advantages in this application, including a short harvest period and no chlorine bleaching. Kenaf paper is stronger, whiter, longer lasting, more resistant to yellowing, and it has better ink adherence than wood pulp paper (Kaldor, 1989). Kenaf leaves and stems have a potential as livestock feed. The breakthroughs and advances in environmental technology have resulted, among others, from intensive research in the kenaf industry.

Natural fiber/plastic compounds has widely used in construction and housing industry, food packaging, automotive industry, oil and chemical absorbents animal bedding and poultry litter and soil-free potting mix. With the increasing applications of composites with kenaf fibers more and more assessment is needed to get a better understanding of interfacial bonding of the materials. The environmental conditions, such as high moisture and high temperature, can limit the usefulness of polymer composites by deterioration of their mechanical properties during service.

Modiboo et al. (2007) investigated the moisture inhibition by alkalized kenaf fiber using moisture sorption method and he found that these fibers had excellent specific properties and had the potential of being outstanding reinforcing fillers in plastics industry. Aziz & Ansell (2004) stated alkalized fiber composites have higher elastic storage modulus, E` and lower damping capacity and the standard laminating polyester resins are suitable for manufacturing of natural fiber composites with useful engineering properties . Huda et al. (2010) evaluated the mechanical and thermal properties of alkalized or silane treated kenaf fibers reinforced polylactic acid. It was found that treated fiber composite offered superior mechanical properties. Kenaf fibers have been investigated and very good mechanical properties have been reported. The specific stiffness is comparable to the glass fiber ones, and the price is 3 to 2 times lower than of the glass fibers; the elongation at failure is also comparable to that one of glass fibers. Thermoplastic and thermosetting matrices have been used to produce kenaf fibers reinforced composites. A positive effect of MAH on the bonding and the wettability of kenaf fibers/polypropylene were reported. The pre-treatment of the fibers improves the mechanical properties of the composite. The standard laminating polyester resins are suitable for manufacturing of kenaf fiber reinforced laminated bio-composites with useful engineering properties.

Information concerning specific combinations of environmental effects (rice husk and temperature) on mechanical properties of kenaf fibers is very difficult to be found in literature.

#### 2.2.1.4 Kenaf fiber mat process

#### 2.2.1.4.1 Carding and needle punching process

Carding is a basic process in the production of yarns and fiber webs of nonwoven fabrics to disentangle fiber stock into individual fibers with minimum fiber breakage and parallelization distribution, and then convert the fibers into the form of a web. Hao et al. (2007) stated that kenaf fibers were prepared by using carding machine which enables uniform thickness of kenaf fiber mat. Carding action is the most important kind of mating surface interaction in the carding machine. To have a carding interaction, the teeth of both the mating surfaces must be in a hold state relative to the fiber load. Hwang et al. (2001) claimed that the carded webs are produced using conventional carding machines. A carding machine processes the fibers mechanically through a series of rollers covered with wires. The web is produced by condensing the fibers on a doffer. The carding stages involve the actions of opening, allowing wastes to fall out, and then carrying the fibers into an open condition so that they can be blended in a random way (Figure 2.2). If this could be carried out with complete opening, mixing together, and uniform orientation of the fibers, then the carding process would be ideal.

In addition, Lee et al. (2009) agreed that carding provides a uniform blend of the two fibers; this is followed by needle punching, then pre-pressing and finally hot prepressing to form composite materials. Needle punching is the processed involve the fibers are forced to entangle in the z direction and interlock with other neighboring fibers (Figure 2.3).



Figure 2.3: Carding process for manufacturing bio-composites (Lee et al., 2009).

#### 2.3 Glass fibers

According to Sheppard & Laurel (2013) the basic raw materials for glass fiber products are a variety of natural minerals and manufactured chemicals. The major ingredients are silica sand, limestone, and soda ash. Mainly it is from silicon-oxide, which gives 55-65 % of glass. It contains other metal-oxide sand together with the silicone they conjugate a macromolecule with high cohesive energy primer atomic or ionic bonds. Glass can be made from silica alone. However, silica has a very high melting point (1700°C), so soda ash is added to lower it melting point due to research by John F. (2007). Adding a lot of soda ash makes glass too soluble in water, so limestone is added to reduce its solubility. Other ingredients may include calcined alumina, borax, feldspar, nepheline syenite, magnesite, and kaolin clay, among others. 6 % lime and 4 % magnesia (magnesium oxide) are added to the basic mix to make sheet of glass. Unlike most solids, glass is not made of crystals and does not have the same rigid structure. It is an amorphous solid.

Waste glass, also called cullet, is also used as a raw material. The raw materials must be carefully weighed in exact quantities and thoroughly mixed together (called batching) before being melted into glass. John F. (2007) stated that when glass is very hot it flows slowly like a thick liquid. Studies by Bryan (1999) shows glass fibers are manufactured by drawing molten glass into very fine threads and then immediately protecting them from contact with the atmosphere or with hard surfaces in order to preserve the defect free structure that is created by the drawing process. The characteristic diameter of the single fibers is  $8-17 \mu m$ .

Instead of being formed into yarn, the continuous or long-staple strand may be chopped into short lengths. The strand is mounted on a set of bobbins, called a creel, and pulled through a machine which chops it into short pieces. The chopped fiber is formed into mats to which a binder is added. After curing in an oven, the mat is rolled up.

Glass fibers are as strong as any of the newer inorganic fibers but they lack rigidity on account of their molecular structure. The properties of glasses can be modified to a limited extent by changing the chemical composition of the glass, but the only glass used to any great extent in composite materials is ordinary borosilicate glass, known as E-glass. The largest volume usage of composite materials involves E-glass as the reinforcement. S-glass (called R glass in France) has somewhat better properties than Eglass, including higher thermal stability, but its higher cost has limited the extent of its use. Wallen & Brown (1994) have recently described the properties of experimental calcium aluminate glass fibres with stiffness's as high as 180GPa.

Czvikovszky &Nagy, (2000) stated that the advantages of glass fiber are it was cheap, available in big amounts, UV resistant, chemically inert, and electrical insulator. However, it also has the disadvantages such as highly abrasive (at specific manufacturing technologies where friction is presented on the tool surface), relatively high density, and brittle and low Young's modulus. The glass fiber is the most widely used reinforcing fiber, it's physical and mechanical properties are listed in Table. 2.2.

Fiber type	Density ,ρ [g/cm <sup>3</sup> ]	Tensile strength [GPa]	Young's modulus, E [GPa]	Strain at break, ε [%]
Glass	2,5-2,8	3,2-4,	6 70-85	1,8-5,7

Table 2.2: Mechanical properties of glass fibers (Czvikovszky &Nagy, 2000).

#### 2.4 Polymer matrix composite (PMC)

Strong (2000) stated that polymer matrix composite (PMC) is a material consisting of polymer (resin) matrix combined with a fibrous reinforcing dispersed phase. PMC represents 90% of all composites. Two types of polymers that are often used as a matrix material for the fabrication of PMC are thermoset such as epoxies, phenolics and thermoplastics such as low density polyethylene (LDPE), nylon and acrylics. Properties of PMC are determined by properties of the fibers, orientation of the fibers, concentration of the fibers and properties of the matrix. Reinforcement of polymers by strong fibrous network permits the fabrication of PMC that is characterized by the following properties such as high tensile strength, stiffness, and fracture toughness, good abrasion, puncture and corrosion resistance as well as low cost and simple fabrication method. The only drawback of PMC is that it has low thermal resistance and high coefficient thermal expansion (CTE). PMC are mainly used for manufacturing secondary load bearing aerospace structures, boat bodies, canoes, kayaks, automotive parts, radio controlled vehicles, sport goods, bullet proof vests and other armors parts, brakes and clutch linings.

#### 2.4.1 Thermosetting resin

According to Mohammad (2007), thermoset resin was defined as a plastic material which was initially a liquid monomer or oligomers or a pre-polymer, which was cured by either application of heat or catalyst to become an infusible and insoluble material. Thermoset polymers have covalent bonds linking the polymer chains in three dimensions.

These links prevent the chain from sliding past one another resulting in higher modulus and improved creep resistance. Usually thermosets were more brittle than thermoplastics. The polymer chains in thermosets were below their glass transition at room temperature, making them glassy. Prior to cure, thermosets were liquid or made to flow under pressure to any desired from. Once cured, a tightly bound three dimensional network structure was formed in the resin and hence the resin cannot be melted, reshaped and reprocess by heating. Therefore, during composite manufacturing, the impregnation process followed by the shaping and solidification were done before the resin begin to cure. However, thermosetting products were removed from the mold at the fabrication temperature, typically 200-300°C.

Thermosetting resins were brittle at room temperature and have low fracture toughness. On the other hand, due to its three dimensional cross-linked structure, thermoset resins have high thermal stability, chemical resistance, high dimensional stability and also high creep properties. However, thermoset polymers have some drawback that are not being recyclable and having less variable processing technique.

# 2.4.2 Polyester

Polyester resin is the most widespread used thermoset matrix material; it can be built up from different acids, glycol and monomer (Figure 2.4) according to Czvikovszky & Nagy (2000).



Figure 2.4: Condensation polymerization of a polyester (Hoa, 2009).

In general it is a viscous fluid, which contains polyester solved in a monomer (styrene). The styrene helps to lower the viscosity and promotes the crosslinking of the linear molecules without a byproduct. The polyester resins can be stored only to a limited period because they tend to gel with time.

As this time is too long to wait for during manufacturing the crosslinking process can catalyzed (catalyst) and accelerated (accelerator). After adding the initiator the crosslinking reaction starts. The initiator itself is not taking part in the chemical reaction, it is only catalyzing that. After a few minutes the crosslinking has started the system reaches the gel time, meanwhile an exoterm reaction occurs because of the chain polymerization. To the final hardening the temperature rises radically (Fig. 2.5)



Figure 2.5: The exotherm heat effect of unsaturated polyester; G represents gel time; H

hardening time

Unsaturated polyester acts as a matrix. The matrix is homogenous, linearly elastic, and isotropic. The material properties of a homogeneous body are independent of position in the body.

Thus physical properties from a representative material sample may be obtained from any location in a homogeneous body, provided the orientation of the sample is fixed relative to a set of axes attached to the body. In the present text, the term material properties include mechanical properties, as well as material properties such as density. The measured material properties of the isotropic body are independent of the orientation of the representative material sample taken to characterize its behavior (i.e. independent of the axis of testing).

Unsaturated polyester combines an unsaturated dibasic acid and a glycol dissolved in a monomer, generally styrene, including an inhibitor to stabilize the resin. Organic peroxides, such as methyl ethyl ketone peroxide (MEKP) and a promoter are combined with the resin to initiate a room temperature (R.T.) cure. In this liquid state, polyester may be processed by numerous methods; including Hand Layup (HLU), vacuum bag molding, spray-up and compression molded Sheet Molding Compound (SMC).

Research by Kresonm (2012) shows suitable ambient conditions for working with unsaturated polyesters. Unsaturated polyester resins are often designed for use at ambient conditions. There are however ambient conditions that limit the use of unsaturated polyester resins. Many believe that the safest ambient conditions for unsaturated polyester resins is a temperature of 25°C and therefore most unsaturated polyesters are manufactured and standardized to work at this temperature. In practice though, it is difficult to achieve controlled temperature of 25°C without the use of costly temperature control systems and insulation.

As can be deduced from the above table when 2 parts of catalyst are added to 100parts resin at 25°C a gel time of 11minutes is obtained. This gel time lengthens to 33 minutes at 15°C and shortens to 7minutes at 30°C –this illustrates the effect temperature has on the curing of an unsaturated polyester resin.

The unsaturated polyester resin contains many reactive ingredients that allow it to harden when a peroxide catalyst is added to it. The key ingredient for ambient cure is a chemical compound termed "accelerator" or "promoter." The way it works is that most peroxide catalysts are stable at ambient conditions when heat energies are low. At elevated temperatures the heat energy of the system increases and the peroxide becomes active. In order to make the peroxide active at ambient conditions without using heat, accelerator is added to the resin which excites the peroxide causing it to become active and initiate the hardening of the resin.

At temperatures above 35°C the activity of the peroxide is exponentially increased due to the increased heat energy. These results in a much shorter gel time and the product will generate too much heat during its cure. Excessive heat buildup could damage the mold. The product may also shrink and stress cracking in the part may occur. It is important to take note of the above phenomena when working with unsaturated polyesters at ambient conditions. When polyester resins have been cross-linked they are rigid, infusible and insoluble. There are so many varieties of polyester resins now commercially available that is difficult to give typical values for physical properties of cured materials. Furthermore, polyester resins are mostly used in conjunction with glass fibre and the physical properties of the final products greatly depend on the type and quantity of glass fibre incorporated. This point is illustrated by Table 2.3 given below. In which are given comparative values of some polyesters have good heat stability showing little weight loss up to about 200 °C. The electric insulating properties of cured polyesters are satisfactory for many purposes but the polar nature of the ester group results in a relatively high power factor and dielectric constant and so the use of resins in high frequency applications is limited.

Table 2.3: Typical values for various properties of cured polyester, unfilled and reinforced

D.	Unfilled	Glass chopped	Glass woven	Glass roving
·Sx.	casting	strand laminate	cloth laminate	rod
. 6		(hand lay-up)	(hand lay-up)	(extruded)
Specific gravity	12	16	17	19
Tensile strength	9 000	20 000	50 000	120 000
$(lb/in^2)$				
Flexural strength	18 000	30 000	60 000	150 000
$(lb/in^2)$				
Compressive strength	20 000	20 000	35 000	70 000
$(lb/in^2)$				
Impact	2	20	25	70
strength(unnotched)				
(ft lb/in <sup>2</sup> )				
Glass content	0	30	55	70
(% weight)				

(Kresonm, 2012).

Cross-linked polyesters are resistant to a wide range of organic solvents but they are attacked by chlorinated hydrocarbons (e.g., chloroform, ethylenedichroride), esters (e.g. ethyl acetate) and ketones (e.g., acetone and methyl ethyl ketone). The ester groups in the polymer provide sites for hydrolytic attack and strong alkalis cause appreciable degradation. The polymer is, however resistant to most inorganic and organic acids, with the exception of strong oxidizing acids. alcopyright

#### 2.4.3 Curing Agent

Hardener or curing agent can be defined as a material that chemically combines with a synthetic resin to produce a hardened product (ASTM, 1983).

# 2.4.3.1 Methyl ethyl ketone peroxide (MEKP)

According to Li et al. (2004) MEKP is a commonly used curing agent for thermosetting polyester resins, a cross-linking agent and catalyst used in the production of other polymers and polyester resins.in chemistry and biology, catalysis is the acceleration (increase in rate) of a chemical reaction by means of a substance, called a catalyst, which is not consumed by the overall reaction. A catalyst provides an alternative route of reaction where the activation energy is lower than the original chemical reaction. Catalysts participate in reactions but are neither reactants nor products of the reaction they catalyze. The process of autocatalysis is where the product of a reaction helps to accelerate the same reaction.

They work by providing an alternative pathway for the reaction to occur, thus reducing the activation energy and increasing the reaction rate. MEKP is normally produced in the phlegmatizer (dimethyl phthalate, DMP) with acid as a catalyst. Moreover, the product with a concentration up to 10% active oxygen is neutralized, and then is brought to the required concentration by further dilution with phthalate. MEKP is ordinarily a mixture of several isomers, all isomers contain the bivalent –O-O- linkage, and the molecules and their anions are powerful nucleophiles.

Addition by Metivier and Hatrick (1981) claimed Methyl ethyl ketone peroxide (MEKP) is organic peroxide, a high explosive similar to acetone peroxide, and is very dangerous to prepare. MEKP has density of 1.170 g/cm3 (20°C) and molecular weight 210.22. MEKP is a colorless, oily liquid at room temperature and pressure. It is prepared from butanone and hydrogen peroxide (figure 2.6). Dilute solutions of MEKP are used in industry and by hobbyists as the catalyst which initiates the polymerization of polyester resins used in glass-reinforced plastic, and casting. MEKP does this through the production of free radical.



Figure 2.6: Chemical structure of Methyl ethyl ketone peroxide (MEKP).

#### 2.5 Manufacturing methods of polymer composites

The layered structure provides the main technical advantage of the composite construction. The strength and stiffness of the whole product or part can be optimized with a low weight in a way that the best properties are in the preliminarily defined load directions. The design steps are defining the main loading directions, analyzing each layer according to failure criterions and to design the whole lay-up of the composite part. During designing the producibleness should be taken into account (costs, technology, length of serial, etc.), which determines the manufacturing process according to Czvikovszky and Nagy (2000).

One of the main questions is the handling of the crosslinking process: to convert the basic oligomer and monomer containing material to a fully cross-linked structure. Practically it is important that fully conversion occurs (which means that 100 percent of the reactive groups are converted) because physical, mechanical and chemical properties are greatly influenced by the conversion. The residual non-reacted monomer can significantly alter the properties of the composite product. Several processing method is available for manufacturing composite parts. During the exercise the hand lay-up technology will be used to produce a sample. The schematic of hand lay-up is shown in Figure 2.7.



Figure 2.7: Schematic diagram of hand lay-up technique.

#### 2.5.1 Hand lay up

Some ten years ago the first modern polymer composites were serial manufactured by hand lay-up, by laminating layers on top of each other. This technology is widely used because its low cost and ease of manufacturing. The hand lay-up is the most cost effective in short series and prototype building polymer composite processing technique. The surface of the positive or negative mold must be treated with a release agent, so the part can be easily demolded.

The outer layer of the product is the gel coat, a resin rich layer containing fillers to protect the part against the environmental and mechanical effects (weather, UV, wear and scratch, etc.). After the gel coat the layers giving the sufficient mechanical strength and thickness are applied, the impregnation is done by with the help of brushes and rollers. The most important is to produce a well impregnated, air and void free wall thickness during the hand lay-up process. The final product can be built up from dozens of layers, but care must be taken when the matrix material is chosen (gel-time, exothermic heat). The wall thickness of the laminate can vary; it can contain local reinforcements, stringers, ribs, metal inserts or can be a sandwich construction. Curing (crosslinking) should take place according to the chosen resin system, usually at room temperature, or in some cases elevated temperatures can be used. It is advisable to use a post curing (at elevated temperature) step, this helps the fully conversion of reactive groups.

Post curing of cold cured laminates is recommended (typically 2 hours @ 80°C or 16 hours @ 40°C depending on resin type). Prior to application of the fiber and resin, the mold cavity is coated with either polyvinyl alcohol or a non-silicon wax to aid component release. Release is simply achieved by tapping wedges between mold and component or by the use of compressed air to gently force the pieces apart. Chopped Strand Mat is most commonly although woven roving is used when stronger and stiffer laminates are required.

A gel coat is applied to the mold surface to produce a resin rich smooth surface for aesthetic and environmental protection purposes. For improved surface finish and corrosion resistance a surface veil is used which is applied with an embedded fabric for reinforcement or mixed with resin for smooth surface. The advantage of wet lay-up is that it is a simple technique requiring low capital investment and no requirement for highly skilled labor.

The disadvantages are that it is not suited for strength or weight critical primary structure as the fiber orientation and local resin content cannot be well controlled. The nature of the wet lay-up also has health and safety implications since the vapors from low

molecular weight low monomers can be harmful. Vacuum assisted wet lay can help prevent the presence of voids, caused by air trapping, in the laminate. By applying a vacuum to the part after lay-up voids are reduced and consolidation is improved. The part is sealed within a plastic film (bagging material) and the air extracted using a vacuum pump. Finally, cutting, grinding and other post processing steps can be done.

2.5.2 Lamination According to Sharifah (2012), lamina (layer, ply) is the arrangement of unidirectional or multidirectional fibers in a matrix to form a thin layer is called a layer, ply, or lamina of composite material. In general, several such layers are bonded together, with layer orientations chosen to form single multilayered sheet that has optimum material performance characteristics tailored for specific loading and environmental conditions. Laminate define as a stack of laminate bonded together to form a single multilayered sheet of composite material is called laminate. The stacking sequence is the order in which plies of varying orientations are staked one on top of the other. In this text, the stacking sequence is specified starting from the top of the laminate.

Code designed to specify laminate composites as concisely as possible as related to (a)the orientation of each ply relative to reference axis (x) (b) the number of laminate at each orientation and (c) the stacking sequence of the laminate in the laminate. A lamina that has straight parallel fibers is called a unidirectional ply. Two commonly encountered stacking sequences are shown in Figure 2.8. A cross-ply laminate is made by stacking alternating 90° and 0° plies. An angle-ply laminate is obtained by alternate stacking of  $\pm \theta$  plies.



Figure 2.8: Cross-ply and angle ply laminate configuration (Nettles A.T., 1994).

Rules of design heuristics (Sharifah, 2012).

The rules of design heuristics to improve strength are as follows:

1. Homogeneous lay-up is recommended for strength controlled design. Heterogeneous laminates should be avoided for strength-critical designs. In the case where heterogeneous laminates cannot be avoided, it is generally best to stack primary load-carrying plies toward the laminate core.

2. Minimize groupings of plies with same orientations to create a more homogeneous laminate and to minimize interlaminar stress and matrix cracking during the service period. If plies must be grouped, avoid grouping more than four plies of the same orientation.

3. Avoid grouping of 90° plies and separate 90° plies by a  $0^{\circ}$  or  $45^{\circ}$  ply to minimize interlaminar shear and normal stress.

4. Separate  $\pm \theta$  plies to reduce interlaminar shear stress.

5. Shield primary load carrying plies by positioning inside of laminate to increase tensile strength and buckling resistance.

6. To avoid large-scale matrix cracking and delamination, the ply angle difference between the adjacent plies must not exceed 45°.

7. Avoid positioning tape plies with fibers oriented perpendicular to edge at the laminate mid-plane to lessen high interlaminar at free edges.

8. If tape plies with fibers oriented perpendicular to a free edge should be stacked at midplane, stack no more than approximately three plies.

9. When there exists a hole, avoid locating tape plies with fibers oriented perpendicular to loading direction at the laminate midplane to lessen interlaminar stress around a hole. The above rules of design heuristics should be carefully applied according to loading and conditions.

#### **CHAPTER 3**

#### METHODOLOGY

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#### 3.1 Materials

In this study, the kenaf fibers were obtained from Malaysian Agricultural Research and Development Institute (MARDI), Malaysia. All the kenaf mats in this study were used without any surface treatment. A standard unsaturated polyester resin was supplied by Castmesch Technologies Sdn. Bhd. and glass fibers were provided by local company. MEKP (solution in dimethyl pthalate) used was from Kaum Jung Akzo Nobel Peroxides Ltd by trade name Butanox M60. Methyl alcohol (methanol) and ethyl alcohol (ethanol) was from QREC (ASIA) Sdn. Bhd. used for swelling and dimensional stability test. Figure 3.1 shows the kenaf and glass fiber in mat form with 170mm length x 130mm width.

The typical properties of unsaturated polyester (UP) and methyl ethyl ketone peroxide (MEKP) properties were stated in table 3.1 and 3.2. UP has hazy, pinkish color in appearance with gel time 18 to 23 minutes at 25°C with 1% MEKP. Density of UP was 1.4 with specific gravity  $1.12 \text{ g/cm}^3$  and volumetric shrinkage at 8%. MEKP has colorless in appearance. Density of MEKP was 1.15 with melting point at  $-8^{\circ}$ C and boiling point at  $109^{\circ}$ C.



Figure 3.1: Kenaf and glass fiber in mat form.

Table 3.1: Typical properties of unsaturated polyester resin.

Characteristics	Description
Appearance	Hazy; Pinkish
Non-volatile	56-59
Viscosity @25°c,cps	
-brookfield,#3/60	450-600
-brookfield,#3/6	900-1350
Thixothropic index	1.5-2.8
Geltime@25°c,minutes	
-1%mekp	18-23
Specific gravity,g/cm <sup>3</sup>	1.12
Volumetric shrinkage,%	8
Density,g/cm <sup>3</sup>	1.4
$\bigcirc$	

Table 3.2: Typical properties of MEKP

Characteristics	Description
Molecular formulation	$C_4H_{10}O_4$
Molar mass,g/mol	122.12
Appearance	Colourless, high viscosity liquid
Density,g/cm <sup>3</sup>	1.15
Melting point, °C	-8
Boiling point,°C	109

#### **3.2** Preparation of kenaf fiber.

Kenaf which is in mat form were cutted into 170mm length and 130mm width. After water has been spray lightly for better bonding and uniform dispersion between fibers in kenaf mat. The kenaf mat was pressed using hot press at temperature 100°C for 8 minutes to get uniform thickness.

#### **3.2** Preparations composites.

The hand lay-up technique was used in the fabrication of kenaf composites and kenaf/glass fiber hybrid laminates. To fabricate laminate, the matrix material was prepared from a general purpose unsaturated polyester resin catalyst in a weight ratio of 100 UP:2 MEKP A steel frame mold of size 170mm x 130mm x 50mm was used in making the laminates. The mold was covered by OHP plastic film for easy removal and to create a smooth surface finish of the laminate.

After laminating process, the impregnated kenaf and glass fiber at different layers; 1, 2, 3, and 4 layers were pressed at 100°C for 5 minute and postcured at 70°C for 5 minute. The annealing step was applied to avoid sudden room temperature that can cause crack or bending composite. In the end, the specimen would be ready and taken out from the mold. Samples were made from the laminates with parallel edges of dimensions 170mm x 130mm. The formulation was shown in Table 3.3. The hand lay-up process was simplified in Figure 3.2.

First sample was used one layer of kenaf mat impregnated in unsaturated polyester (UP) by filled up 20mm (thickness) of the mold by UP, then impregnated kenaf mat into it before add on the rest of UP to fill up the mold.

While putting the OHP plastic by palm of hand, it was handed with extra care to avoid the bubble formation during hand lay-up process. The mold with the plate and OHP plastic in between then was flipped over to make sure the other side of the kenaf mat also fully impregnated with UP and no bubble formation. Again, palm was used vertically and horizontally while removing the bubble formation as maximum as possible. The same step was used for sample with one layer of glass fiber. The volume of UP was reduce significantly with the increasing of layer number.

The sample that only contains one kenaf mat was named as K represents one kenaf. The sample with one glass fiber mat was named as F represents one glass fiber. The sample with one kenaf mat and one glass fiber mat was named FK. The sample with one kenaf mat in between two glass fiber mat was named FFK. While the sample with one glass fiber mat in between two kenaf mat was named KKF. Lastly, the four layer number of lamination containing two kenaf mat and two glass fiber mat arrange alternately was named FFKK. The total six samples was produce in this study for further investigation based the effect of layer number on mechanical properties and dimensional stability of hybrid kenafglass fiber unsaturated polyester composite.



Table 3.3: Formulation of kenaf-glass	s fiber hybrid unsaturated polyester
composites	8.

Filler	UP	МЕКР	
	(%)	(%)	
1 Kenaf	100	2	
1 Glass Fiber	100	2	
1 Kenaf/1 Glass Fiber	100	2	
2 Kenaf/1 Glass Fiber	100	2	
1 Kenaf/2 Glass Fiber	100	2	
2 Kenaf/2 Glass Fiber	100	2	

Average weight of kenaf mat = 25.4 Average weight of glass fiber = 20.0

Total volume of the composite=1105g/cm<sup>3</sup>

#### **3.3** Testing and Characterization

#### 3.3.1 Tensile test

Tensile properties are determined using specimens of dimension 100mm x 15mm x 5mm, following the ASTM 3039 as shown in Figure 3.3.



Figure 3.3: Diagram of sample dimension for ASTM 3039.

The tensile testing of all specimens was performed on the Instron mechanical testing machine 5569. Preparing samples testing according to Ghani et al (2012) in a tensile experiment, the specimen is gripped firmly by mechanical jaws at the wide portion on either side and extended by means of a tensile testing machine. The pulling is carried out at a crosshead speed of 2 mm/min and a 50 mm extensometer. Modulus values were determined from the secant slope of the stress–strain curve in the strain range 0.05–0.25% in accordance to ISO 527:1997. The kenaf/fiberglass unsaturated polyester hybrid
composite is a rigid material, so the low speed was used. The higher speeds used to test flexible materials (Ghani et al., 2012). Typical stress strain curves for a brittle material and ductile material was shown in Figure 3.4.



Figure 3.4: Typical stress strain curves for a brittle material and ductile material

Calculated entities:

Tensile stress  $\rightarrow$  measured the force at any time divided by the original cross sectional area of the waist portion.

Tensile strain  $\rightarrow$  the ratio of the difference in length between the length marked by the gauge marks and the original length,

Yield strength  $s_Y \rightarrow$  ultimate tensile strength (strength value prior to fracture),  $s_t$ 

Elastic modulus,  $E \rightarrow$  ultimate elongation (strain value at fracture),  $e_t$ 

## 3.3.2 Flexural test

Flexural properties are important in assessing the resistance of materials to bending. The determination of the flexural properties is in accordance with ASTM D 790 for the standard test methods for the flexural properties of un-reinforced and reinforced plastics and electrical insulating materials using instron machine 5569. A typical experimental set-up is as the one shown in Figure 3.5 (Ghani et al., 2012).



Figure 3.5: Flexural test experimental set-up (Czvikovszky & Nagy, 2000).

©The 3 point bending test of the composite laminate was conducted on the universal testing machine. The displacement can be measured by the moving head and the extensometer. In this research the span used was 50mm. The flexural properties are determined using specimens of dimension 80mm x 12mm x 5mm, following the ASTM D 790 as shown in Figure 3.6 and speed 2 mm/min.



Figure 3.6: Diagram of sample dimension for ASTM D 790.

Basic equation in bending stresses in beams



Figure 3.7: Cross sectional view of beam

Calculated entities:

The maximum stress caused by bending is calculated by the following formula:

$$S = \frac{3FL}{2bd^2}$$
(3.2)

Where S refer to stress (N/m<sup>2</sup>), *F* is load or force at break or at yield (N), *L* is span of specimen between supports (m), *b* is width (m) and *d* is thickness (m).

If the load recorded corresponds to the value at failure occurs (S) corresponds to the flexural strength. The maximum strain due to bending (compression and tensile is estimated by Czvikovszky & Nagy (2000).

$$\varepsilon = \frac{6Dd}{L}$$

Where e refer to strain (dimensionless i.e., no units), D refer to deflection at the centre of the beam (m) ,d is thickness (m) and L is specimen's length of span between supports (m).

The flexural modulus from the recorded load (F) and deflection (D) is:

$$E_B = \frac{3}{4} \frac{FL^3}{bd^3D}$$

(3.4)

(3.3)

# 3.3.3 Water, methanol and ethanol absorption test

The water, methanol and ethanol absorption test was done by using rectangular specimens. The sample is oven dried at 50 °C for 24 hour to a constant weight ( $M_0$ ). The sample are periodically taken out from beaker; surface dry with absorbance paper and weight using an analytical balance with 0.1 mg precision ( $M_1$ ). The samples are immersed until saturated by average from 3 specimens for each composite. The result of water, methanol and ethanol carry absorption is determined by using equation:

$$E_B = \frac{M_1 - M_0}{M_0} \times 100 \tag{3.5}$$

Where  $M_0$  is the initial dry weight,  $M_1$  is the weight after immersion in water/solution.

#### Thermogravimetric Analyzer Test (TGA) 3.3.4

Studies on thermal properties of unsaturated polyester/kenaf/fiberglass hybrid composites are using Pyris diamond TG/DTA, Perkin Elmer Instruments. The heating

composues are using Pyris diamond TG/DTA, Perkin Elmer Instruments. The heat rate of TGA condition is 20°C/min and temperature range is 30°C to 800°C in the air.

#### **CHAPTER 4**

#### **RESULTS AND DISCUSSION**

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#### 4.1 Tensile properties

The tensile properties of kenaf single fibers have been reported by Bolton (1994). Tensile strength is the maximum stress that the material can support. Tensile stress occurs when an object is subjected to forces that make the object expands. The important factor to obtain a good fiber reinforce composites is the adhesion between matrix polymers or the fiber and interphase properties. Abbreviation of F represent one layer of glass fiber mat impregnated in unsaturated polyester composite, K represent one layer of kenaf mat impregnated in unsaturated polyester composite. FK represent two layer hybrid kenaf/glass fiber unsaturated polyester composite with arrangement glass fiber-kenaf-glass fiber. KKF represent three layer of hybrid kenaf/glass fiber unsaturated polyester composite with arrangement glass fiber-kenaf-glass fiber. KKF represent three layer of hybrid kenaf/glass fiber unsaturated polyester composite with arrangement glass fiber-kenaf-glass fiber-kenaf. Abbreviation of F, K, FK, FK, KKF and FFKK was applied in Chapter 4 both in figure and discussion.

Figure 4.1 shows the effect of number of layer kenaf mat and glass fiber on tensile strength of hybrid kenaf-glass fiber unsaturated polyester composites. Based on the results, four layer of lamination (FFKK) shows the highest tensile strength 52.665MPa. Nettles A.T. (1994) agreed that increasing of layer number increase the tensile strength of lamination based on direction of arrangement of kenaf/glass fiber mat.

Observation on overall tensile result shows a similar pattern of brittle failure. It is assumed that the brittle failure is due to the matrixes system and ductile behavior is produced mainly by the higher percentage of kenaf fiber weight percentage as reported by Ya`acob A.M. (2011). The sudden failure at kenaf section indicated that the majority of the tensile strength was provided by glass fiber, not the kenaf fiber.

Kenaf single fibers exhibit tensile strength and tensile modulus of 11.91 GPa and 60 GPa, respectively. The tensile properties of glass fibers have been reported by Kresonm (2012). According to Otzurk (2001), good tensile strength depends more on effective and uniform stress distribution. It can be seen in Figure 4.1 that higher glass fiber content resulted in higher tensile strength of lamination.

The tensile strength of three layer sample contains two layer of kenaf mat and one layer of glass fiber (KKF) is the lowest 35.67MPa. This result was caused by the uneven distribution of kenaf mat loading. The KKF sample were de-bonding in between the interphase layers of kenaf/glass fiber composites samples due to un-treated surfaces that resulted in the interphase failure of specimen as in research by Ya`acob A.M. (2011).



Figure 4.1: Effect of number of layer kenaf mat and fiberglass on tensile strength of hybrid kenaf-glass fiber unsaturated polyester composites.

Resilience is the capacity of a material to absorb energy when it is deformed elastically. The modulus of elasticity is a measure of the stiffness of the material, but it only applies in the linear region of the curve. If a specimen is loaded within this linear region, the material will return to its exact same condition if the load is removed. At the point that the curve is no longer linear and deviates from the straight-line relationship, Hooke's Law no longer applies and some permanent deformation occurs in the specimen. This point is called the "elastic, or proportional, limit".

Figure 4.2 shows the effect of number of layer kenaf mat and glass fibers on tensile modulus of hybrid kenaf-glass fiber unsaturated polyester composites. Two layer of laminate (FK) have the highest tensile modulus 4549.265MPa. This is due to surface interaction between the resin and glass fiber and kenaf mat increase. Superior mechanical properties increase proportionally with surface roughness. Glass fiber has lowest modulus

1599.675MPa .The tensile modulus increase with increasing of layer content up to two layers but decreasing with three layer of laminate. The decreased value of tensile modulus may due to damaged matrix, deteriorated interfacial adhesion and bond strength, between matrix and fiber. The increasing of filler loading contributes to the overall stiffness of the composite.



hybrid kenaf-glass fiber unsaturated polyester composites.

The amount of stretch or elongation the specimen undergoes during tensile testing can be expressed as an absolute measurement in the change in length or as a relative measurement called "strain". It is the ratio of the change in length to the original length,

$$e = \frac{L - L_0}{L_0} = \frac{\Delta L}{L_0}.$$

The effect of number of layer kenaf mat and glass fiber on elongation at break of hybrid kenaf-glass fiber unsaturated polyester composites have been shown in Figure 4.3. The result shows one, three and four layer of laminate have elongation at break at 0.02 percent. Two layer of laminate have 0.01 percent. Lower elongation at break may be due to lower resilience of the fiber. According to Rozman et al. (2005), decrease deformability of a rigid interface between filler and the matrix component, thus increase the stiffness as shown in tensile modulus result.



Figure 4.3: Effect of number of layer kenaf mat and fiberglass on elongation at break of hybrid kenaf-glass fiber unsaturated polyester composites.

## 4.2 Flexural properties

Flexural strength is the limit of a material to withstand flexural stress without failing. If an object is subjected to flexural stress, it will undergo both tension and compression behavior because of bending moment. In theory, an object's flexural and tensile strengths would be in similar ranges if there is homogeneity in the materials used, meaning that the substances used are mixed in equally. If the substances are not uniformly mixed, then the flexure and tensile strength might drastically vary in different areas of the object. Moreover, another factor that can change an object's flexural and tensile strengths are defects. In this study, a composite with torn fibers might increase its tensile strength, as the fibers can stretch longer, but it will decrease its flexural strength, especially when load is applied on the area where the fibers are weakest.

Figure 4.4 shows the effect of number of layer hybrid kenaf- glass fiber unsaturated polyester composites on flexural strength. Flexural strength of glass fiber (F) and two layer (FK) hybrid kenaf- glass fiber unsaturated polyester composite was 95.51MPa and 92.77MPa respectively. Flexural strength were found to decrease significantly as the filler loading increased due to poor matrix adhesion which lead to debonding by the matrix (Kresonm, 2012). According to Ekhlas et al. (2012), the longer fiber with poor adhesion to the unsaturated polyester matrix created stress concentration zones and this is suggested a reason for the decrease of the flexural strength properties of the composites made with kenaf- glass fiber hybrid composites of various number of laminate. Moori et.al. (2012) stated that the first fracture in glass fiber reinforced plastics is caused by the de-bonding between fiber and matrix, and the bonding strength was closely related to the interfacial strength.



Figure 4.4: Effect of number of layer kenaf mat and fiberglass on flexural strength of hybrid kenaf-glass fiber unsaturated polyester composites.

Flexural modulus is a measure of a material deformation and strain when weight or force is applied. It describes the ability of a material, with a specific cross-section, to resist bending when placed under stress. This term is used to define the relationship between a bending stress and the resulting strain. Strain is a measure of the amount that a material will deform when a stress is applied.

Elastic strain is reversible and will disappear after the stress is removed, meaning the material will return to its original state. At high levels of stress, a material will permanently deform and will not return to its original dimensions. Figure 4.5 show the highest flexural modulus is two layer unsaturated polyester kenaf- glass fiber hybrid composites which are 3078.31 MPa followed by four layer unsaturated polyester kenafglass fiber hybrid composites at 2742.663MPa. Higher fiber content allows better distribution of fiber where stress can be transferred more effectively. Fibers work as carriers of load in the matrix. At higher volume fractions, the matrix is sufficiently restrained and the stress is more evenly distributed. This results in reinforcing effects outweighing the effects of the stress concentrations and results in increase in flexural strength and modulus (Bibo & Hogg, 1996; Sreekala, Jayamol, Kumaran, &Sabu, 2002).



Figure 4.5: Effect of number of layer kenaf mat and fiberglass on modulus of elasticity of hybrid kenaf-glass fiber unsaturated polyester composites.

## 4.3 Thermal properties

Thermal degradation is a process whereby the action of heat or elevated temperature on a material, product, or assembly causes a loss of physical, mechanical, or electrical properties. Natural fibers usually contain cellulose, hemicellulose and lignin. This applies to kenaf fiber as well. The degradation of the cellulose begins around 250°C because the cellulose is a homopolymer of glucose. It is a semi crystalline polymer. It is nearly decomposed totally into volatile product at 400°C (Patrick et. al., 2006; Golnaz, 2007); & Patricia et. al. 2009). The lignin is a polyphenolic polymer that was less stable than the cellulose; its degradation is very slow with the high rate of non-volatile products.

Figure 4.6 shows the TGA curve of glass fiber composite (F) and kenat mat composite (K). From the result obtain, it shows that the degradation temperature for cellulose is at 250 °C, hemicellulose at 225 °C and lignin at 140 °C. Kenaf composites (K) begin to degrade at temperature around 294 °C and end at 370 °C lower than studies by Anis et al (2012). Glass fiber composites (F) begin to degrade at temperature around 315 °C and end at 430 °C. Char residues for sample K and sample F was 11.924% and 8.2885% respectively. Figure 4.6 shows that sample with glass fiber as reinforcement give higher thermal stability compared to kenaf fiber as reinforcement. The reason is inheritance of glass fiber as synthetics material which can withstand higher temperature.

The mean coefficient of thermal expansion over the temperature range from -30° to 250°C, the expansion measurements were made on annealed bars using ASTM D 696. A lower coefficient of thermal expansion in the high strength glasses allows higher dimensional stability at elevated temperature.



Figure 4.6: Degradation temperature of pure fiber glass and pure kenaf fiber in unsaturated polyester resin.

Figure 4.7 shows the curve of sample with glass fiber as reinforcement (F), kenaf fiber as reinforcement (K) and kenaf- glass fiber hybrid as reinforcement (FK). As discuss earlier, sample F with glass fiber as reinforcement has higher thermal stability because a lower coefficient of thermal expansion in the high strength glasses allows higher dimensional stability at elevated temperature. When glass fiber is added in kenaf composites, the thermal behavior show some increment in thermal stability compared to sample with kenaf alone as reinforcement. The degradation temperature sample F with glass fiber as reinforcement increase from 370°C to 400 °C.



Figure 4.7: Degradation temperature of two layer of lamination with pure fiber glass and pure kenaf fiber as control sample.

Figure 4.8 shows three layer sample (KKF) which used two layer kenaf with one layer glass fiber as reinforcement and sample FFK which used two layer of glass fiber with one layer of kenaf as reinforcement. Degradation temperature of KKF and FFK composites increases from 256°C to 406 and 452.34°C respectively. Increasing glass fiber content has increase the degradation temperature as well with char residues which is 17.85% and 19.33% for KKF and KKF respectively.



Figure 4.8: Degradation temperature of three layer of lamination with pure fiber glass and pure kenaf fiber as control sample.

Figure 4.9 shows four layer laminate (FFKK) have degradation temperature at 400°C with char residues at 29.346%. All sample show increasing degradation temperature with increasing layer number of glass fiber. Char residues also increase with increasing layer number of hybrid kenaf- glass fiber unsaturated polyester composites. Summary of initial degradation temperature, maximum degradation temperature and char residues have been simplified in Table 4.3.



Figure 4.9: Degradation temperature of four layer of lamination with pure fiber glass and pure kenaf fiber as control sample.

 Table 4.1: Thermal properties of unsaturated polyester kenaf- glass fiber hybrid composites with different reinforcement.

	F	K	FK	FFK	KKF	FFKK
Initial	315	294	294	294	294	294
degradation						
temperature						
Maximum	430	370	400	452.34	406	400
degradation						
temperature						
Char residues	8.28	11.92	13.15	17.85	19.33	29.346

#### 4.4 Swelling behavior

Polymer swelling is a thermodynamic phenomenon, and it gives a measure of the amount of liquid absorbed by the polymer under equilibrium conditions. For ideal systems, the sorption isotherm is assumed to be linear (Henry's law), i.e. the liquid concentration inside the polymer is proportional to that outside. However, for the systems studied, the swelling curve is curved rather than linear. Thus, the assumption of ideal sorption behavior cannot be used in the present study due to Chuang et al. (2000). Moisture absorption into the composite material can be accounted by three major mechanisms. First was the diffusion of water molecule inside the microgaps between polymer chains; second was the capillary transport into the gaps and flaws at the interface between fibers and the polymer due to incomplete wettability and impregnation; finally, the third mechanism was the transport of water molecules by micro cracks in the matrix that are formed during the fabrication process. (Ahmad et al. (2010); Ghasemi & Kord (2009)).

Figure 4.10 shows the comparison of absorption percentage of sample with one layer of kenaf fiber (K) and sample with one layer of glass fiber (F) in different solvent which were water, methanol and ethanol. All composites show similar pattern of absorption, which is sharp at the beginning followed by gradual increase. The phenomenon can be explained by the analysis of water diffusion mechanism in composites based on Fick's Theory by Kushwaha & Kumar (2010). Case 1- Fickian diffusion in which the rate of diffusion is much less than that of the segment mobility. The equilibrium inside the polymer is rapidly reached and it is maintained with independence of time. Case 2 was relaxation control in which penetration mobility is much greater than the other relaxation processes as reported by Thwe et al. (2002). This diffusion is characterized by the

development of a boundary between the swollen outer part and the inner glassy core of the polymer. The boundary advances at a constant velocity and the core diminishes in size until an equilibrium penetration concentration is reached in the whole polymer. Case 3 was non Fickian diffusion; when anomalous diffusion is occurs where the penetration mobility and the polymer segment relaxation were comparable. In this case, an intermediate behavior between Case 1 and Case 2 diffusion will be seen.

Based on the Figure 4.10, sample F shows the lowest absorption in water which is 0.2 %. This is followed sample F in ethanol which shows absorption percentage at 0.5%. On the other hand, sample K shows the highest absorption percentage in methanol which is up to 5 %. Overall, sample F and sample K shows the highest absorption percentage in methanol 5 % and 3% respectively.



Figure 4.10: Effect of kenaf fiber and glass fiber in unsaturated polyester composite of absorption percentage in different solvent.

Figure 4.11 shows one layer kenaf fiber and one layer glass fiber (FK) in unsaturated polyester composite of absorption percentage in different solvent. Two layer kenaf-glass fiber hybrids composite in methanol shows the highest percentage of absorption up to 5 %. This pattern followed the result of one layer glass fiber and kenaf fiber in unsaturated polyester composite as in figure 4.10 before. Two layer kenaf-glass fiber hybrids composite (FK) in ethanol shows the lowest absorption percentage followed by water at 1% and 1.5 % respectively. Basically, the component with smaller volume will be absorbed to a greater degree. The molar volume of water is smaller compared to ethanol but water molecules will group together to organize into clusters due to the formation of hydrogen bonds among the water molecules according to Chuang et al. (2000). Ethanol can participate in hydrogen bonds.



Figure 4.11: Effect of one layer kenaf fiber and one layer glass fiber (FK) in unsaturated polyester composite of absorption percentage in different solvent.

Figure 4.12 shows two layer kenaf fiber and one layer glass fiber (FKK) and one layer kenaf fiber and two layer glass fibers (FFK) in unsaturated polyester composite of absorption percentage in different solvent. FFK in unsaturated polyester composites in methanol still shows the highest absorption percentage at 5%. Methanol is the simplest alcohol, containing one carbon atom. It is a colorless, tasteless liquid with a very faint odor and is commonly known as "wood alcohol."



Figure 4.12: Effect of two layer kenaf fiber and one layer glass fiber (FKK) and one layer kenaf fiber and two layer glass fiber (FFK) in unsaturated polyester composite of absorption percentage in different solvent.

Figure 4.13 shows two layer kenaf fiber and two layer glass fiber (FFKK) in unsaturated polyester composite of absorption percentage in different solvent. Four layer unsaturated polyester kenaf-glass fiber hybrid composites in methanol still shows the highest absorption percentage at 3.4%. However, the absorption percentage reduces with increasing fiber content. Hybrid kenaf-glass fiber unsaturated polyester composites that been immersed in ethanol have lower absorption percentage in every sample due to ethanol structure that avoid it molecule entrapped in composite structure as agreed by Chang et al (2000).



Figure 4.13: Effect of two layer kenaf fiber and two layer glass fiber (FFKK) in unsaturated polyester composite of absorption percentage in different solvent.

## 4.5 Dimensional stability

Dimensional stability is the ability of a substance or part to retain its shape when subjected to varying degrees of temperature, moisture, pressure, or other stress. Cellulosic fibers are hydrophilic in nature and will absorb water. Water absorption will result in swelling of the fibers. This will affect the dimensional stability of the composite lignocellulosic fibers / thermoset if exposed to an environment with high humidity. This composite will lose the mechanical properties due to the nature of the fibers tend to absorb moisture and cause low dimensional stability and degradation of the interaction between the fiber and the matrix will occur.

Dimensional stability has close relation with swelling behavior that has been discussed in subtopic 4.4 swelling behavior. Figure 4.14 show lowest dimensional stability percentage of kenaf as reinforcement in water which is only 1 %. Highest dimensional stability percentage of kenaf as reinforcement is in methanol which is 16 %. Dimensional stability percentage of glass fiber as reinforcement (F) is lowest in methanol and highest in water.



Figure 4.14: Effect of kenaf fiber and glass fiber in unsaturated polyester composite of dimensional stability in different solvent.

Figure 4.15 show lowest dimensional stability percentage of kenaf fiber and glass fibers as reinforcement in unsaturated polyester kenaf- glass fiber hybrid composites that immersed in ethanol which is only 4 %. Highest dimensional stability percentage of kenaf fiber and glass fibers as reinforcement in unsaturated polyester kenaf- glass fiber hybrid composites is in water which is 18 %. The dimension of sample very unstable in water due to water can be entrapped in fractured sample while ethanol and methanol will evaporate over time.



Figure 4.15: Effect of one layer kenaf fiber and one layer glass fiber (FK) in unsaturated polyester composite of dimensional stability in different solvent.

Figure 4.16 show lowest dimensional stability percentage both two kenaf fiber with one glass fibers as reinforcement in unsaturated polyester kenaf- glass fiber hybrid composites (KKF) and one kenaf fiber with two glass fibers as reinforcement in unsaturated polyester kenaf- glass fiber hybrid composites (FFK) that immersed in ethanol which is only 3 %. Highest dimensional stability percentage is FFK composite in water which is 11 %. The dimension of sample very unstable in water due to water can be entrapped in fractured sample while ethanol and methanol will evaporate over time. However, this percentage is lower than one layer and two layer composites laminate. Dimensional stability of composite laminate was increase with increasing of fiber content.



Figure 4.16: Effect of two layer kenaf fiber and one layer glass fiber (FKK) and one layer kenaf fiber and two layer glass fiber (FFK) in unsaturated polyester composite of dimensional stability in different solvent.

Figure 4.17 show two layer kenaf fiber and two layer glass fiber (FFKK) in unsaturated polyester composite immersed in water have the lowest percentage of dimensional stability at 3 % followed by immersion in methanol and ethanol 5 % and 7 % respectively. The reason for this phenomenon was due to hydrophilic group in kenaf fiber and hydrophobic group in glass fiber.



Figure 4.17: Effect of two layer kenaf fiber and two layer glass fiber (FFKK) in unsaturated polyester composite of dimensional stability in different solvent.

this temperature

### **CHAPTER 5**

## CONCLUSION

#### 5.1 Summary

According to the result obtain, it can be concluded that:

- 5.1.1 The effect of layer number on tensile properties of hybrid kenaf-glass fiber unsaturated polyester composite shows that highest tensile strength in sample with 4 layer number containing 2 kenaf mat fiber and two glass fiber as reinforcement (FFKK). This was proven that increase of layer number had increase the tensile strength.
- 5.1.2 The effect of layer number on flexural properties of hybrid kenaf-glass fiber unsaturated polyester composite shows that the highest flexural strength and modulus was observe in two layer of lamination (FK).
- 5.1.3 The thermal properties of hybrid kenaf-glass fiber unsaturated polyester composite better with increasing layer number of lamination. The highest degradation temperature was observed in sample with three layers of laminate (FFK) at 406°C.
- 5.1.4 The dimensional stability of hybrid kenaf-glass fiber unsaturated polyester composite was highest in ethanol.

# 5.2 Recommendation

This study serves to investigate the effect of filler content of hybrid kenaf-glass fiber polyester composite on mechanical properties and dimensional stability. It was observed that there still a room for improvement for kenaf fibers behavior used in this study. Therefore several recommendations can be made:

- Add more testing methods to investigate more mechanical properties, surface morphology, and porosity using impact test, scanning electron microscopy test and density test respectively.
- ii. Perform surface treatment to improve bonding between matrix-composite and matrix-matrix interfacial adhesion.
- iii. Increasing the number of laminate to obtain optimum fiber content.

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