

ENGINEERED NATURAL FIBRE COMPOSITES FOR OIL AND GAS APPLICATION: A REVIEW

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ABSTRACT

This paper affords a review of natural fiber reinforced composites centering on types and sources of natural fiber, fibers modification, fiber treatments, matrices (renewable and petrochemical), including their mechanical characteristic. This study established reasons why many companies especially in the oil and gas industry are looking at natural fiber composite materials: such as low price of natural fibers, reduction of weight, easy to recycle and are green in nature; when compared to petroleum based fibers. The paper also spotlights on future innovation and invention on the applications of reinforced composite, and the concerns that need to be resolved to encourage research in this area of study.

Keywords: Natural fibre, fibre composites, oil and gas facilities, bio-composite

1. INTRODUCTION

Natural fibers are simply defined as fibers that are not synthetic or manmade but from plants and animals. Plant fibers are distinguished according to its natural characteristic. This can be clearly represented as shown in the figure 1:

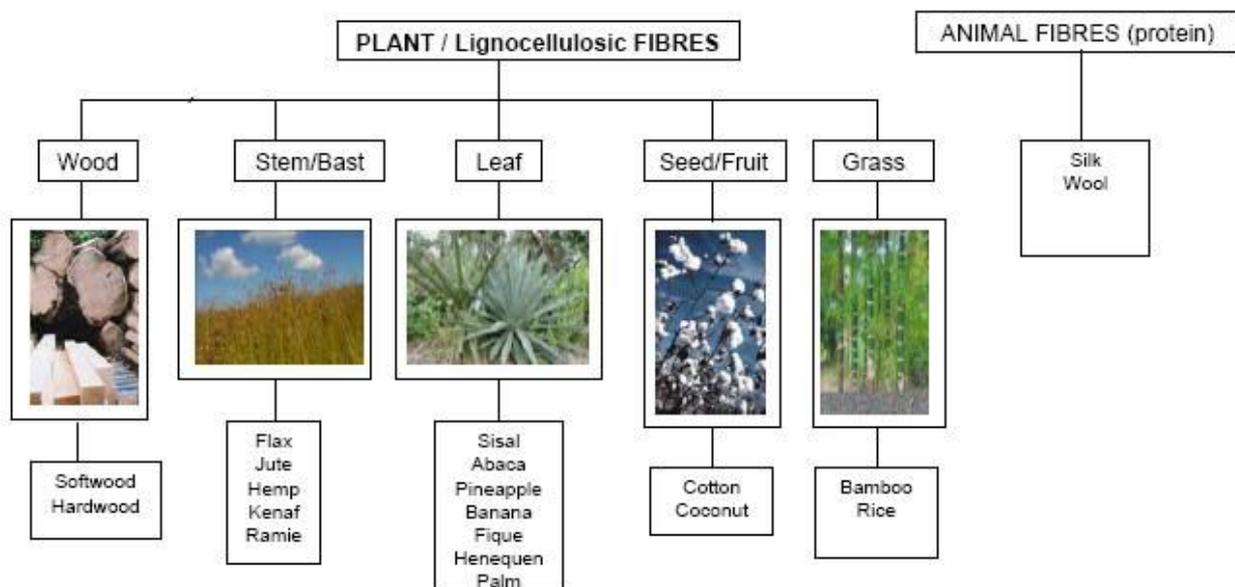


Figure: 1. Classification of natural fibers (Chandramohan and Marimuthu, (2011))

This paper evaluates the natural fibre composites application especially in the oil and gas industries operations, as the resistance of corrosion on pipe work, light weight driven application. Remarkable progress has been recorded in structural applications, which has shown significance of natural fibre composites as an advanced performance material in diverse sector such as oil and gas, marine, railways, automobiles, building and construction, medical etc. The “Advanced Natural Fibre Composites Programme” is being employed by Information Technology, Council for Assessment and Forecasting; they are independent institute below the Science and Technology Department, Govt. of India. The aim of the programme is to improved composites utilization and application and linkage between laboratory-industry for sustainability. Not long, attention was given to utilize natural fibre based composites in oil and gas

facilities. As much as, components cost of composite surpass that of their metallic ones but has lower cost in handling and installation. The cost is even lower when compared to costly metals that are corrosion-resistant such as copper-nickel alloys and their likes. Furthermore, the resistance of corrosion advanced reliability and lead to lower through-life costs. Gibson (2003) outlined number of obstacles in the usage of Natural fibre composites, which was noted recognized in the 1980's. These were:

1. Requirements by regulatory body, particularly on combustibility
2. Deficient in relevant information on performance, particularly in environments that is hostile (including fatigue, wear, erosion and impact abuse, even in environments that contain fluids).
3. Deficient in procedures for efficient design and standards of working, and unfamiliarity combination on the designers part
4. The composites industry fragmented structures, and
5. Complexity in leveling up fabrication processes to build very huge composite structures

In the last two decades most research undertaken on oil and gas is focused on eliminating the obstacle 1-3,

2. APPLICATIONS OF NATURAL FIBER COMPOSITES IN OIL AND GAS FACILITIES

Natural fiber composites are rapidly captivating as better substitute to conventional materials in terms of high pressure and violent environmental conditions. The innovations in technology in terms of processes and products developments, composites has become eye-catching contender for application in oil and gas such as applications in topside, piping system, sub-sea down-hole tubing etc. This is due to the different requirements for design especially in weight savings and show high strength-to-weight ratio compared to conventional materials, which can resist high pressure and hostile condition of environment (Razak et al., 2012)

In the industrial quarter composite materials has exhibited superior resistance to corrosion, excellent performance in fatigue, and resistance to good temperature extremes and wear. In the past two decades, composite materials have wide-ranging usage especially in the oil and gas industry in terms of low thermal conductivity and thermal expansion of low coefficient, stiffness and higher axial strength and such, when the configuration it's composite to suite specific application. Composite has made tremendous impact on reducing the manufacturing cost and oil rigs erecting by replacing heavy metal by composite pipes in pipeline project. It is has shown that, in platforms of offshore for diverse applications, the cost compensation of products in composite are much larger when they replace corrosion-resistant metals that are expensive such as duplex/super duplex stainless steel, copper-nickel alloys, titanium etc. used for various application in offshore platforms. Researchers have been broadly made in material made up of composite as well as prediction of life expectancy, technology in joining, methods of inspection, materials standardization, and development in database. This has made composite application more attractive in the sub-sea application in the areas of down-hole tubing and others. The determination of composite mechanical strength, durability when exposed to liquid depend resin selection made. In material selection, the key issues are smoke and fires toxicity while mechanical include impact resistance and unfavorable environments. The offshore application that is most successful for natural fibre composites is in the area of pipe work for aqueous liquids. The design performance-based guidelines for of natural fibre reinforced epoxy (NFRE) pipes have drastically hastened the application in the offshore sector.

The United Kingdom Offshore Operators Association (1994) initiated it and became a standard ISO draft (ISO 2000). It is also, observed that the most major structural materials, epoxy, polyester and vinyl ester, can be prohibited from use in areas where toxicity and smoke will be a difficulty (Gibson, 2001). Modar, the modified acrylic resins can be applied in definite toxicity-sensitive areas, which is not broadly used offshore while the natural fibre reinforced epoxy is broadly used in pipe and tubular works. Table 1a, b and c, list current application of natural fibre composites with their near future used. Natural fibre composites may find brilliant usage in fabrication of the following:

- outlines for oil pollution barricade
- Railings, ladders and gratings on oil-drilling platforms and ships
- Systems for walkway
- Rods for suckers

Table: 1a. Recent applications of natural fiber composites in oil and gas, offshore facilities (Gibson, 2003)

Fire protection	Walkways and flooring	Life boats
Blast protection	Handrails	Buoys and floats
Corrosion protection structures	Subsea anti-trawl	ESDV protection
Partition walls and shelters	Casings	Boxes, housings
Aqueous pipe systems	J-tubes	Loading gantry
Tanks and vessels	Caissons	Pipe refurbishment
Firewater systems	Cable trays and ladders	Riser protection
Pipe liners	Accumulator bottles	Bend restrictors
Separator internals housings	Well intervention	Subsea instrument

Table 1b: A few current applications of natural fiber composites for oil and gas, off-shore facilities

S/No	Application
1	Composite Grids/ Gratings
2	Hand rails & Ladder Components
3	Aqueous Piping System
4	Water & fuel storage tanks, Vessels
5	Low pressure composite valves
6	Spoolable type thermosetting tubes
7	Sump Caissons and pull tubes
8	Cable support systems
9	Modular paneling for partition walls
10	High pressure accumulator bottles
11	Flexible & Floating Risers, Drill pipe
12	Sub - sea structural components
13	Boxes, housings and shelters
14	Fire water pump casing & sea water lift pump casing
15	Tendons
16	Offshore bride connecting between platforms
17	Blast & Fire protection

Table 1c: Future applications in oil and gas, offshore facilities

Rigid Risers	Coilable Turbing	Flexible Risers
Tendons	Primary Structures	Separators

According to Faruk et al., (2013) the application of natural fiber composite has gained more grounds than glass and carbon fiber. Although, glass and carbon is higher in strength compared to natural fiber but mostly preferred by the oil and gas industries due its reduced weight, recycling, and market enticements.

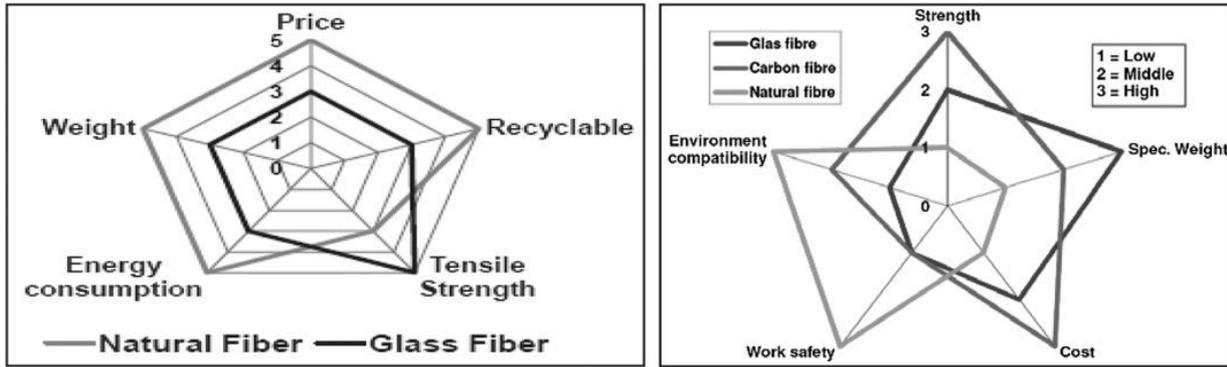


Figure: 2. Comparison between natural fiber, glass fiber, and carbon fiber

From available literatures, it has shown that the source of raw material play an integral part in the properties, volatility in prices and advantages in environment and government encouragement. The use of bio-based plastics as reinforced matrices for natural fiber composites is more popular in the technology market.

The growth rate annually from 2003 to 2007 was 38%. In same vein, Europe annual growth rate was high as 48%. Its worldwide capacity from 2007 to 2013 increased from 0.36 million to 2.33 million metric tons and by 2020 it is expected to increase to 3.45 million metric tons. In the recent years, the increased in reviews, patents, and books on natural fiber composites publication shows its importance. Satyanarayana et al., (2009) in their work stated that “the number of publications and patents on biodegradable lignocelluloses’ fiber based composites (1995-2007) found in the ISI database by entering the words “biodegradable, polymer, fiber” and it is seen that after year 2000, the number of publications and patents rose appreciably” as shown in figure 3.

Faruk et al., (2012) studied 525 papers on natural fibers in polymer composites starting from 2000 to 2010 and added very current results from 2010 to 2012. But their review excluded natural fibers from animals (e.g., wool or silk) or cotton or man-made cellulosic fibers and also wood fiber is excluded.

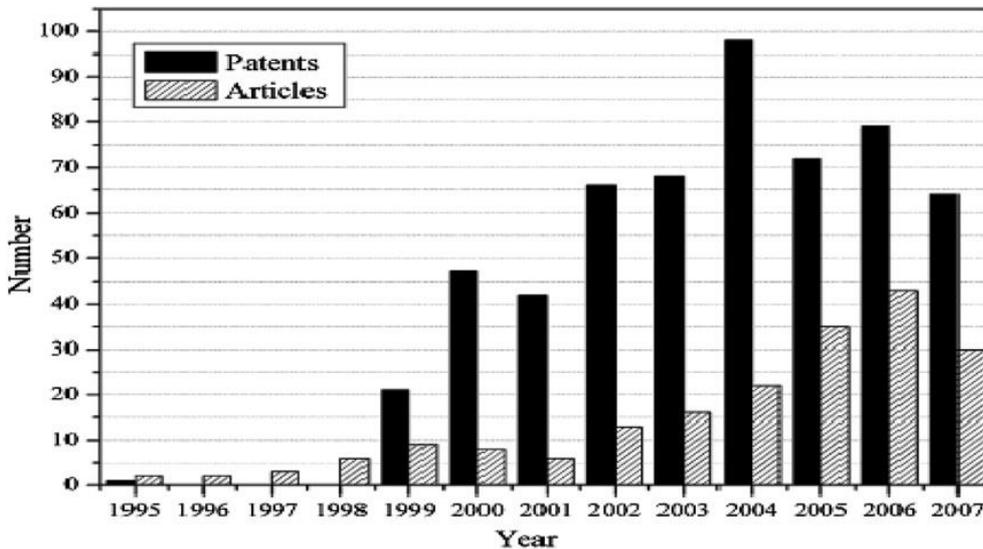


Figure: 3. Overview of publications and patents on biodegradable lignocelluloses’ fiber based composites from 1995-2007.

3. MANUFACTURING PROCESS OF NATURAL FIBER COMPOSITES

In principle, processing techniques for “natural fiber composites” are similar to those of the synthetic fibers. According to the length, orientation and type of the reinforcing fiber, short randomly oriented, unidirectional fibre (raw and carded) and woven fabric are used to incorporate into the reinforcement for the thermoset and thermoplastic resin. The most basic fabrication method is hand layup techniques which are used for thermoset composites; it is the manual mixing process between the fiber and resin. In this procedure, there is composite uniformity in terms of thickness, fiber to resin ratio and the content of void throughout the sample depends on the manual skill used (Pickering et al., 2007). Alternatively, vacuum assisted resin transfer moulding have been widely used for composite fabrication. In this process thermoset resin is used to pull inwards under the vacuum pressure and mix with the fiber. Due to this, better chance of mixing among the fibre and resin can be achieved and more precise spacing in the fiber with thickness uniformity of the final composite obtained. It eases the use of utmost content of the fiber with considerably lesser percentage of constituents in the void than the hand lay-up process (Pickering et al., 2007). An additional technique, is the pultrusion

process, it involves the use of thermoplastic and thermoset matrix. The reinforcement is pulled through heated die and mixed with resin to produce the composite profile. For compression molding using thermoplastic matrix (the reinforcement is mixed with resin and heated plate are pressed) and molding by injection (the resin fibre are added to the machine and melted into mass of fluid, after that they are injected under high pressure into the form) are widely used for composite fabrication (Adekunle et al., 2010). This process involves high temperature (over 200°C) and pressure (5MPa) for complete reaction between fibre and matrix. The principal manufacturing techniques used to produce composites is outlined below: (source: www.sme.org)

- i. Manual Lay-Up
- ii. Automated Lay-Up
- iii. Spray-Up
- iv. Filament Winding
- v. Pultrusion
- vi. Resin Transfer Molding

The process of cutting reinforcement materials to size with the used of hand range and devices which are power-operated, After that, the pieces cut are impregnated with material made up of wet matrix, and after that they are laid in excess to the surface of mold that are covered with a release agent with a coated resin gel called Manual lay-up process. In order to remove the air that is trapped and guarantee distribution of uniform impregnated reinforcement material is called hand-rolled, while more of the material being reinforced is added to build-up the thickness of the required part. Prepreg is another technique of Manual lay-up, used to pre-impregnated reinforcement material, which eradicates handling separate part the reinforcement and resin that provides more control of consistent reinforcement and contents of resin. Prepreg is preserved in the refrigerator to avoid premature curing (www.sme.org).

In spray-up methods, it is designed with spray gun that sprayed resin on the surface of prepared mold, which continuously reinforcement into appropriate lengths as the spraying of resin is done. The composite parts are cured after the lay-up. The heated air assisted the curing processes and the oven is used to heat platen presses with the used of autoclaves. The time range for curing is from one hour to half a day or longer. Non-adhering film made up of plastic, it is usually polyester and that is sealed all over the mold plate and lay-up material. A space is gradually created below the bag forcing it against the lay-up process. The entrapped air and excess resin is drawn out using this process. In producing large, complex shaped parts, the effective method is the Vacuum bag molding. Another method is the filament winding process; it involves wrapping the tow of narrow fiber or tows of band impregnated resin of fiber around a mandrel of the required shape which is required which is being produced. After removing the mandrel, the shape produced is hollow. Filament winding process can be used to produce the following - tubing, pipe, pressure vessels, tanks and similar shape items. There are two types of filament winding-hoop or helical winding. The hoop winding process, the tow is approximately perpendicular to the rotating mandrel axis. The rotation of the mandrel moves to the delivery supporting carriage of the material with band width of one and thereby butting one edge of band that is after the earlier band. The helical winding process, deposition of material in a path that is helical in one direction only, it involves turning around on the end and also returns back in a path that is helical in the direction of opposite. The mandrels may be designed using a metallic or non-metallic materials that is collapsible and for easy removal of part or may be dissolved after the curing process.

The pultrusion method is a continuous process primarily used in producing long, shapes in straight form with a constant cross-section. Pultrusion is like extrusion the only difference is just that the material composite is being pulled out, instead of pushing it, through a die. Manufacturing by pultrusions are done using continuous reinforced fibers known as 'roving' which gives reinforcement in transverse and longitudinal in form of materials made up of mat or cloth. The reinforcement's pattern are impregnated in a resin by drawing it throughout wet-out station of the resin; and usually shaped inside a performing or guiding system. The next is to shape and cure them in a set of dies or preheated die. After curing, the pultrusion length is saw-cut. Pultrusions shapes produced are always in solid or hollow form, and typical applications is in rod and bar, tubing, pipe, rungs and ladder rails, also for different types of supports.

Resin molding transfer method are used for the production of big, items that are complex in nature such as shower and bath enclosures, parts of aircraft, cabinets, and components parts for automotive. It is a process of using, a set of halves of the loaded mold with material being reinforced clamped together. Resin is fed by gravity or pumped into the infusing mold material being reinforced. After filling the resin inside the mold, then it is plugged and allowed at a particular temperature to cure. After it has been cured, the next is to separate the halves of the mold and removing the produced part for trimming into the final and concluding process (www.sme.org).

Studies shows that the manufacturing technique has strong effect on the natural fibre reinforced thermoplastic composites mechanical properties. It affects the content, distribution of fibre, aspect ratio of the fibre and orientation of the fibre (Aziz and Ansell, 2004; Liu et al., 2007). There are several manufacturing techniques (e.g. injection moulding, compression and extrusion) that are used in the composite industry. However, the compression moulding is preferred when non-woven kenaf mats are used as reinforcing material for the fabrication of composite plates. Compared to other moulding techniques, the compression moulding has the advantages of producing composite plates with uniform material distribution and high fibre aspect ratio due to reduced fibre breakage. Thwe and Liao (2002) investigated on the mechanical and moisture absorption properties of short bamboo fibre reinforced polypropylene composites; and short bamboo-glass fibre reinforced polypropylene hybrid composites. The laminates were fabricated by the compression moulding method. They reported that the inclusion of glass fibre as additive (20 wt%) and the use of coupling agent (maleic anhydride polypropylene) noticeable improved the resistance to moisture absorption since the results indicated substantial drop in the moisture content at saturation.

4. MODIFICATION OF SURFACE FOR NATURAL FIBER COMPOSITES

Natural fibers surface are modified via different methods. The development of new natural fibre composites that is beneficial with thorough and fundamental understanding of the surface and fiber. The limitations and inadequacy of NFCs, if they are used as composites reinforcement, are connected to the lesser strength properties, resistance to poor absorption of moisture, adhesion with low interfacial, limited temperature of maximum processing (about 200°C), and dimensional stability and lower durability (shrinkage and swelling). The treatments of NFCs are carried by four types of methods, chemical, physical, biological and nanotechnology (NT). The methods of modification are of diverse efficiencies for the improvement of fibers mechanical properties, the bonding existing in the matrix and fibre will lead to the development of various properties of final products. If the hydrophilic nature of natural fibres is decreased, then the adhesion improvement on the interface can be achieved by modification of fibre, they can be performed using physical or chemical methods. The treatment by physical methods affects only the fibres surface structure while the composition of chemical for the fibres is not changed. Examples of physical treatment, is corona method of treatment (Ke et al., 2008; Sakata et al., 1993), cold plasma treatment (Wang et al., 1993) and thermal treatment (Li et al., 2008) is in the literature. Alternatively, modifying the surface natural fibres by applying chemical compounds changes the surface chemical composition and so improve the adhesion between the fibres and matrix of the polymer. Chemical coupling is an important method chemical modification (Bledzki and Gassian, 2008) they are carried out *via* treatment of fibre with maleic anhydride-polypropylene copolymers in order to improve the interface between the fibres based cellulose and polypropylene (Bledzki and Faruk, 2003).

The major drawbacks of natural fibers in particular composites are the unfortunate compatibility among fiber matrix and their higher absorption of moisture. So, modifications of natural fiber are allowed because it leads to modification in surface properties of the fiber in order to get better bond with dissimilar matrices. A very good stiffness and strength might be attained if there interface is strong that is naturally very brittle with propagation of easy crack throughout the fiber and matrix. From the matrix to the fiber, the stress transfer efficiency can be reduced by an interface that is weaker. Extensive study was done and it was stated in literature review, it was explained that the interface significance and effect of various types of modification on surface for the physical and mechanical properties of natural fiber reinforced composites. There is observation in the trend which show a chemical modification preference (alkaline, acetylation, silane, benzoilation, acrylation and acrylonitrile grafting, maleated coupling, permanganate, peroxide, and isocyanate treatment) compared to physical modification (corona and plasma treatment). It has been made known that silane and maleated method of treatment is becoming a suitable choice method because of its beneficial results (Lie et al., 2007; John and Anandjiwala, 2008; Xie et al., 2010). The suppliers of additive has improved their additives with an amount of higher anhydride functional groups than the earlier grades existing that was used in 1980 and 1990 century, that form additional chemical links for the sites, causing a significant performance development at lower contents of additive. The composites absorption of water can be reduced using coupling agents which has not leads to reduction in performance of long-term. For natural fibers, the processing methods of using the technology of enzyme are substantially increasing. Recently, the enzymes utilization in textile field and modification of natural fiber is rapidly increasing (Bledzki et al., 2010). The technology is accepted because the enzymes application is friendly in the environment. The catalyzed reactions are very precise with a performance of focused. Furthermore, the technology of enzyme is cost effective, leading to better quality of product for mostly use modification by maleated and silane.

5. Chemical modification of natural fiber composites

The chemical modification of fiber surface, use the entire fibers or chemical agents throughout. A fiber that has strongly polarized cellulose is incompatibly inherent with polymers that are hydrophobic. Presently, numerous research projects are carried out on the utilization of cellulose-based plastics as fibers reinforcement. Though, these fibers contain cellulose, hemicellulose, and lignin (Rowell, 1997). Ghali et al., (2011) studied the effect of chemical modification of luffa cylindrica fibers on the mechanical and hygrothermal behaviours of polyester-luffa composites.

Seena et al., (2005) investigated the acetylation banana fibre effect in reinforced composites of phenol formaldehyde and it was revealed that the tensile modulus and tensile strength and impact strength is improved compared to untreated banana fibre composites. Liu et al., (1994) also investigated the acetylation effect in composites of natural fibre (rayon, cotton, polystyrene with wood as matrix) and it was seen, by the use micro-debonding test and it was revealed that acetylated had improved shear interfacial strength of the fibres. In addition, it was revealed that increase in acetylation fibres free surface energy. Zafeiropoulos et al., (2002) studied the acetylation of hemp, flax and wood of fibre and succeeded in taking away non-crystalline fibres constituents, changing the free energy surface of the fibre, also changing the surface topography characteristics of the fiber, and improving the stress transfer interface efficiency of the fiber (Tserki et al., 2005)

Mohd Yuhazri et al., (2011) investigated the NaOH effect on kenaf fiber reinforced polyester composite. It was seen that the composite mechanical properties increases with increase in alkali concentration. Mohamed Edeerozey et al., (2007); Sharifah et al., (2004) reported that the fiber from kenaf can be reinforced by using thermoplastic and thermoset plastic. Though, we can achieve the increase in property by surface modification of the fiber.

Aziz et al., (2005) investigated the modified polyester effect of resins in alkali-treated kenaf fiber composites. Four different polyester resins were used in this investigation. Hemp has been used traditionally, for rope making but these days the fiber from hemp is used to make items such as shoes, clothing and toys. The fiber is non-toxic, fully biodegradable, and recycled. Rajeev et al., (1997) investigated the tensile properties of treated kenaf fiber by using silane with fiber from sisal. They establish that the based composites on the modified matrix generally have better mechanical properties to those containing the matrix that is unmodified.

Maya Jacob et al., (2010) conducted experimental investigation using coupling agent called zein. The characterization of fiber is done using FTIR. It was reported that chemically modified fibers from kenaf were seen to have better mechanical and viscoelastic material properties. In order to decrease sensitivity of the moisture and decay through biological and to optimize the fiber matrix interface properties, the natural fibers used in polymer composite can be treated by physical and chemical methods (Feng et al., 2001). If the fibers is treated with appropriate chemicals, the fibers reinforcing efficiency in the composite and adhesion interface between fibers and most matrices of polymers was evaluated Mattoso et al., (1997); Martins and Joeke (2003). The surface of the fiber is cleaned using chemical treatment; the surfaces are chemically modified, delayed the absorption of moisture process and also the surface roughness is increased. It was reported that the treatment by alkalization enhance the mechanical properties of the fiber from kenaf as to untreated kenaf fiber Mohd Edeerozey et al., 2007. The following process were used to a get better modification of sisal fiber surface/interaction of matrix: alkali treatment, H₂SO₄ treatment, conjoint H₂SO₄ and dewax alcohol/benzol treatment, treatment by alkali, treatment by thermal, treatment using acetylated, thermal-alkali treatment and alkali-thermal treatment Li et al., 2000.

Ray and Sarkar (2001) studied the jute fibers occurring changes after treating it with 5% NaOH solution for various time 0, 2, 4, 6, and 8 hrs. A weight loss of 9.63% was measured during treatment in 2 hr with a hemicelluloses drop content ranging from 22 to 12.90%. It was reported that the modulus and tenacity of treated fibers increase by 45% and 79%, and the strain breaking were reduced by 23% after treatment for 8 hr. After treating for 6 hr, the fibers crystallinity only increases.

Ray et al., (2001) studied the treatment of jute fiber with 5% NaOH period of 2, 4, 6, and 8 hrs. Thermal analysis report shows that desorption of moisture were seen at a temperature that is lower in the fibers that are treated. The fiber fineness which gives additional surface area may cause moisture evaporation. The loss of moisture for treated alkali jute fiber for a period of 6 and 8 hrs decreased because of the fibers crystallinity increase. In all the fibers that are treated, it was observed that the hemicelluloses percent degradation is decreased.

Mwaikambo and Ansell (2002) investigated crystallinity index, thermal resistance and morphology on the surface of alkali untreated and treated natural fibers. They reveal that the solution of alkali (NaOH) concentration influence the fibers thermal resistance. They also reported that a cellulose fast degradation was between 0.8 and 8% of NaOH, and the degradation was observed to be insignificant outside this range. The crystallinity index of hemp fiber insignificantly dropped while sisal, kapok, and jute fibers showed a small raise in crystallinity index at the concentration of NaOH ranging from 0.8-30%. SEM micrograph of all the fibers that are untreated revealed the surface was relatively smooth whereas, all treated alkali fibers confirmed that surfaces are uneven because of molecular weight loss of hemicellulose and species.

Sydenstricker et al., (2003) conducted research on the alkali (NaOH) treated sisal fibers for thermal properties. They revealed that fibers density and lignin content was reduced with NaOH treatment. Furthermore, alkali treatment caused a significant reduction in absorption of moisture in sisal fiber. They reported that TGA thermograms of treated NaOH fiber were observed to be resistant thermally than the fibers that are untreated.

Razera and Frollini (2004) investigated the alkali (NaOH) treatment effect on jute-phenolic resin composites physical properties. The treatment of fiber was at 5% NaOH solution. It was reported the impact strength, tensile strength, and

break at elongation of treated NaOH fiber composites was found to be the highest while the lowest was the uptake of water. SEM micrograph analysis of the surface of fracture impact showed that treated alkali fibers surrounded with the matrix to a high level than the fibers untreated. In addition, the mechanism of pull-out could be seen in fiber of untreated jute. The adhesion improvement in phenolic resin and jute fibers resulted from the treatment of NaOH that contributed to the hydroxyl and hydroxymethyl groups' reaction of jute fibers and phenolic resin respectively.

Mwaikambo and Ansell (1999 and 2002) found that alkalization of plant fibers (sisal, hemp, jute and kapok) effectively changes the topologies surface of the fibers and their crystallographic structures. However, the sodium hydroxide (NaOH) concentration for alkalization must be taken into consideration. Moreover, they have reported that surface removal of impurities on fibers from plant is a benefit for adhesion of fiber matrix. This might assist both bonding reaction and mechanical interlocking due to the exposure of fibrils for chemical like the dyes and resins.

Bisanda (2000) also examined the alkali treatment effect on the ability of wetting and consistency of sisal-epoxy composites. Treatment of sisal fiber in NaOH solution resulted in more rigid composites with lower porosity and hence higher density. The treatment has been shown to improve the adhesion characteristics because of an increase in surface tension and surface roughness. From the analysis, the resulting composites showed improvements in the water resistance and compressive strength. It was reported that the removal of lignin in intercrystalline and intracrystalline and extra waxy substances on the surface by increasing the possibility of alkalization for chemical bonds and mechanical interlocking substantially.

Ray and Sarkar (2001) conducted a research on the characterization of alkali-treated jute fibers for mechanical and physical properties. The treatment with alkali jute fibers using 5% solution showed that majority of the changes occurred within 2-4 hours of treatment. The weight loss due to losing their cementing capacity in the fiber structure, separating the fibers from the strands, and dissolution of hemicellulose was maximized at these treatment hours. Thus, a sharp drop in the linear density was measured from 33.0-14.5 denier after 6 hours of treatment. The loss of hemicellulose led to closer packing of its cellulose chains. On further treatment with alkali solution from 4-8 hours, the fibers crystallinity was seen to have improved. The fibers modulus and tenacity were seen to be better by 45 and 79%, in that order and the breaking percentage strain were decreased by 23% after 8 hours of treatment. This indicated that fibers become stiffer and more brittle with increase in fibers crystallinity. The rate of change of tenacity, modulus, and breaking strain showed the possibility of two different mechanisms before and after 4 hours of treatment. Furthermore, Jahn et al., (2002) explained that when flax fibers were subjected to NaOH treatments, so called mercerization, the extent of polymorphic change of cellulose I to cellulose II was taken place within the crystalline domains of the fiber depends on the alkali concentration.

Zefeiroopoulos et al., (2002(a)) studied two different surface modification treatments (acetylation and stearamation) on two grades of flax fibers (green and dew retted flax). Acetylation has been found to increase the percent weight gain of fibers at high levels (around 15-17%), which implies that the treatment is affecting the bulk and not just the surface of flax fibers. An investigation of the acetylation condition indicated that the presence of moisture in the fibers is beneficial for the reaction, as it hydrolyses acetic anhydride to acetic acid, a compound that swells the fiber and facilitates the reaction. For stearamation, the percent weight gain of the fiber after treatment indicated that only the surface is affected and not the bulk, as in the case for acetylation. It has been found that both treatments result in a fibers non-crystalline constituent removal and alter the surface characteristics topology. Also, the results showed that both treatments change the fiber surface free energy. Acetylation increased fiber surface energy whereas stearamation decreasing it (Zefeiroopoulos et al., 2002(b)). They reported that acetylation improved the stress transfer efficiency at the interface for both green and dew retted flax fibers. The optimum treatment time was 2 hours for acetylation of dew retted flax and 4 hours for acetylation of green flax. The stearic acid treatment enhanced the efficiency stress transfer at the interface but only for lower treatment times. The optimum treatment time was 36 hours for both green and dew retted flax fibers. It has also found that stearamation for 90 hours deteriorated the interface in both green and dew retted flax fibers. The reasons were because the longer time of treatment deteriorated the fiber strength, and an excess of stearic acid present on the fiber surface may have acted as a lubricant more than as a compatibilizer. Similarly, Joseph et al., (1996(a)) investigated the chemical treatment effect on the tensile properties of sisal fiber-reinforced using low density polyethylene (LDPE) composites. Treatment using chemicals such as NaOH, isocyanate, permanganate, and peroxide were carried out to get a better bonding at the interface of fiber-polymer. Alkali treated fiber composites showed better tensile properties than untreated composites. This was because their rough surface topography and aspect ratio were increased. They found out that cardinal derivative of toluene diisocyanate (CTDIC) treated composites exhibit better mechanical properties. It is because the structure of long chain of CTDIC linked to fibers cellulosic makes it more compatible and hydrophobic and extremely dispersible in the LDPE matrix. The SEM micrographs also showed the strong fiber matrix adhesion in sisal-LDPE composites.

Fung et al., (2002) investigated the matrix maleation effect and content of fiber on process ability, morphology, and mechanical properties of sisal fiber-reinforced PP (PP) composites. In order to improve the interfacial bonding between sisal fiber and PP, the PP matrix was maleated (MAPP) by blending PP and maleic-anhydride-grafted-PP in the weight ratio of 9:1. It was reported that the sisal fiber/MAPP composites has a lower viscosity of melt (as shown by

measurements of torque rheometer) than the sisal fiber/PP composites at 20 wt% sisal content of fiber. It was observed that PP maleation showed an improvement in tensile strength in term of mechanical properties. The improved sisal fiber/matrix interfacial bonding prevented fracture mechanisms such as fiber/matrix debonding and fiber pull-out.

It was reported that treatment with alkaline have two different effects on the fiber: (1) it increases the surface roughness that results in a better mechanical interlocking; and (2) Increases the exposed cellulose amount on the surface of the fiber, therefore causing an increase in the possible number of reactive sites. Aziz and Ansell (2004) studied the alkalization effect and alignment on the mechanical and thermal properties of hemp and kenaf fiber composites. In their study, long hemp and kenaf fibers were used in as-received condition and alikalized in a 6% NaOH solution. They were combined with cashew nut shell liquid (CNSL) resin and hot-pressed to form natural fiber composites. It was apparent from the composites mechanical properties that treated long kenaf-CNSL and treated long hemp-CNSL were fairly comparable to glass fiber polymer reinforced composites. However, maximum toughness and maximum strength can never be simultaneously achieved. It was suggested that the composites must be considerably designed for suitable applications and desired mechanical properties. Moreover, wide endothermic DSC peaks were presented in all the thermograms of the composites indicating the presence of moisture. The presence of water in composites tends to develop plasticity in the material, thus mechanical properties. Sreekala and Thomas (2003) reported that oil palm fibers discovered to be phenolic materials reinforcement is excellent, but its main disadvantage is hydrophilic nature. Thus they have investigated the effect of various surface fiber modifications such as latex coating, mercerization, and gamma irradiation, treatment with silane, treatment with isocyanate, peroxide and acetylation treatment on water-absorption characteristic of fibers from oil palm.

Absorption behavior of water distilled at 30, 50, 70 and 90°C was investigated. They reveal that treatment reduces the temperatures of water uptake. The decrease in water uptake was because of its changes in chemical and physical that occurred in the modifications of fibers. Treatment reduced the fibers mechanical strength. Breaking strain of the fibers was significantly increased the treatments apart from treatment with silane. There was enhancement in young's modulus showing mercerized and silane treatment. During the stage of swollen the stiffness of the fiber was considerably reduced.

Mohanty et al., (2000) conducted a research on surface modifications of two types of jute fabrics, i.e. hessian cloth (HC) and carpet backing cloth (CBC), involving dewaxing, cyanoethylation, alkali treatment and grafting on performance of biodegradable jute-fabric/Biopol (poly(3-hydro-xybutyrate-co-8%-3-hydroxyvalerate) composites. From the results, it was revealed that mechanical properties like impact strength, tensile strength and bending strength increase in comparison to pure Biopol due to reinforcement with jute fabrics. More than 5% enhancement in tensile strength properties, bending strength is 30% and impact strength is 90% of the composites relative to pure Biopol sheets were observed. Furthermore, degradation investigation reveals that after 15 days of burial compost more than 50% weight loss of jute/Biopol composites was occurred.

Singh et al., (2000(a)) used FT-IR microscope in reflectance mode (ATR) to study the effect of various coupling agents such as organosilane, zirconate, titanate and substituted methacrylamide on treated natural fibers (sisal). The results showed an irregular physisorption/chemisorption of coupling agents, their penetration beyond the surface, and a decrease in hydrophilicity of fibers. The difference in the properties of chemically treated and untreated and fibers was also verified in the polymer composites.

Pickering et al., (2003) investigated the two silane-based coupling agent's effect, gamma-aminopropyltriethoxysilane (GS) and dichlorodiethylsilane (DCS), on radiata pine or pinus radiata wood fiber without and with pre-treatment using NaOH. The results showed that concentrations of silicon up to 3.2 wt % was find on the surface of the fiber because of silane coupling, however, It was found that pre-treatment value was reduced dramatically. Nuclear Magnetic resonance (NMR) shows that between the fibers coupling had occurred and reaction of DCS producing linkages between the ether hydroxyl groups on fibern of wood and silane. Treatment and pre-treatment have been found to have insignificant effect on fiber strength. Sheets of composite were manufactured by fiber blending of (5, 10, and 20 wt %) with polyethylene (PE) follow by extrusion. Increase in fiber strength contents was observed at 5 wt% for all composites treatments contrast to fiber that is untreated. This was mainly due to an increasing compatibility of the fiber surface to PE. Though, at higher fiber contents there was no such improvement obtained. Evidence suggested that the void production was limiting the strength of composite. Graft copolymerization of methyl methacrylate (MMA) onto wood-fiber, in dispersed media, has been studied as function of temperature, concentration of redox initiator, and surfactant (Roman-Aguirre et al., 2004). They discovered that PMMA grafting onto wood-fiber was toughly depending on variations in concentration and temperature of initiator and surfactant. Scanning Electron Micrograph (SEM) showed the existence of small agglomerates regularly distributed on the modified fiber surface. These agglomerates remained on the fiber surface after 24 hours of chloroform extraction and were presumably grafted PMMA. Infrared analysis of the acid hydrolysis products of the modified fiber showed a clear characteristic signal of carbonyl groups at 1734cm^{-1} . Such signal indicated the presence of fiber-PMMA chemical links, since simple physical interactions would not remain after the cellulose hydrolysis. Mishra et al., (2001) also conducted the research on graft copolymerization of acrylonitrile (AN) on modified chemically treated sisal fibers using combination of NaIO_4 and CuSO_4 as initiator in medium of

aqueous temperature range of 50-70°C. PAN grafting onto chemically modified sisal fiber was done to improve the surface and bulk mechanical properties for its potential use as reinforcing fiber for polymer composites. The optimum grafting condition was to use NaIO₄ and CuSO₄ concentrations of 0.008 mol/L and 0.002 mol/L, respectively with 0.1g chemically modified sisal fiber, and 1 ml AN, at 60°C. The best tensile strength and modulus was obtained at 5% grafting of the fiber. The AN-grafted sisal fibers are expected to act as compatible reinforcing fibers with several hydrophobic thermoplastics and thermosettings for preparation high performance composites.

Jana and Prieto (2002(a)) investigated on the three coupling agents effect (Silquest® A-1100, Silquest® A-2120, and hydroxymethylated resorcinol) on the morphology of epoxy coating on wood flour particles and the mechanical and physical properties of the composite. They have found that all three coupling agents slowed the epoxy curing reactions and completely promoted the coverage of wood flour particles by crosslinked epoxy. Although a hydroxymethylated resorcinol coupling agent provided better morphology around wood flour particles, much smaller-size epoxy particles of a more uniform size were formed. Nevertheless, both tensile and impact strengths were reduced due to the use of coupling agents.

Another interesting research concerning to the effectiveness of (graft copolymer of PP and maleic anhydride copolymers (MAH-PP) as coupling agents in jute-PP composites was done by Gassan and Bledzki (1997). They reported that the concentration of MAH-PP solution, the treatment time, and the processing parameters influence the composites mechanical properties. Composites flexural strength of MAH-PP treated fibers were find to be high than that of unmodified fibers, and increased with fiber loading. The cyclic dynamic values of load increase indicate that the coupling agent reduced the progress of damage. Therefore, dynamic strength (dynamic failure stress at load increasing test) of the MAH-PP modified composites was raised by about 40%. SEM investigations confirmed that the increase in properties was caused by improved fiber matrix adhesion. Also, there were fewer tendencies for fibers to pull out the matrix.

5.1 Mercerization

Alkalinization (or mercerization) is a common method for treatment of natural fiber surfaces. Mercerization can be definition by standard using the ASTM D1965: as the method of interacting with a fairly concentrated aqueous solution of strong base by subjecting it to vegetable fiber, in order to achieve huge swelling with fine structure in resultant changes, mechanical properties, morphology and dimension (Bledzki and Gassian, 1999). In this method, lignocellulosic fibers are immersed in aqueous NaOH solution for a period of time. NaOH solution dissolves lignin, wax and oils from the fiber surface and leaves a clean and porous cellulosic surface. This treatment leads to higher specific mechanical properties since cellulose has much higher mechanical properties compared to lignin and also increases specific surface area leading to better interaction with the matrix.

Sgriccia et al., (2008) studied the alkali treatment effect on morphological and absorption of water properties of numerous natural fibers. The fibers were submerged in 5% sodium hydroxide solution for 1hr at temperature of room. They concluded, via SEM microscopy, that alkali treatment of hemp fiber led to hemicellulose and lignin removal from the fiber surface. As presented in Figure 4, surface of hemp fibers is much clearer after alkalinization. The authors reported however that fiber alkalinization increased their water uptake. After 700 hours of immersion in distilled water, epoxy composites based on untreated hemp absorbed around 16% water; while alkali treated hemp had a water uptake of around 22%.

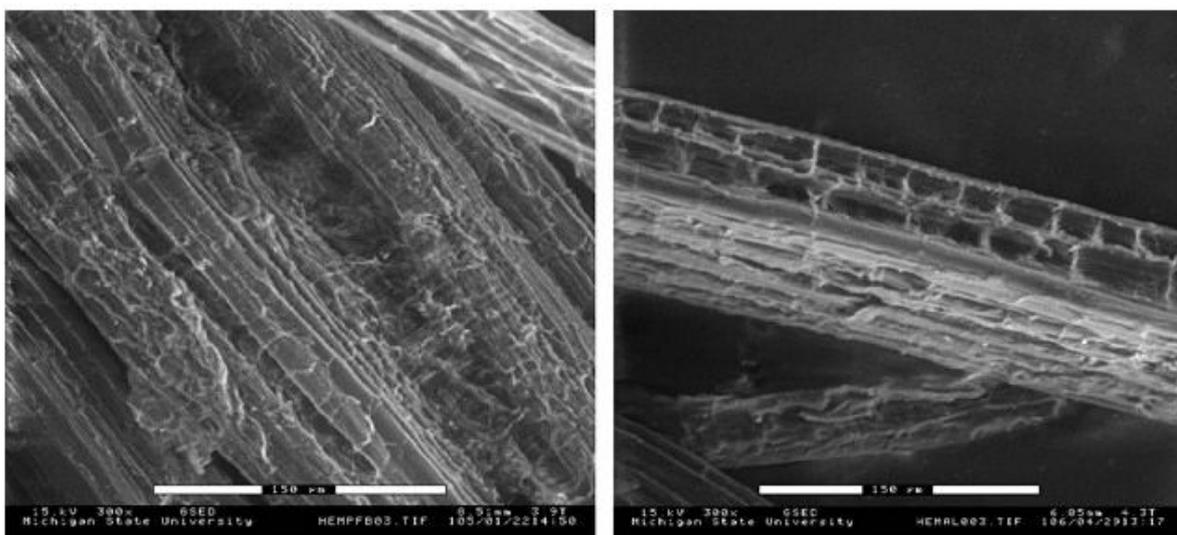


Figure 4: SEM micrographs of (a) untreated and (b) alkali treated hemp fibers (Sgriccia et al., 2008).

Mercerization was discovered that it can change surface topography of the fiber, it was reported that the diameter of fiber decrease with increase in sodium hydroxide concentration (Mwaikambo and Ansell, 2006). The treatment by mercerization results in surface modifications leads to wettability increase of coir fiber polyester resin as stated by Prasad et al., (1983). It was stated that treatment using alkaline have two effect on the henequen fiber: (1) Surface roughness is increased, leading to improved mechanical interlocking; and (2) The quantity of exposed cellulose increases the surface fiber, hence increase the possible number of reaction sites (Valadez-Gonzalez et al., 1999). Thus, treatment by alkaline has a long-lasting consequence on the natural fibers mechanical behavior, particularly in stiffness and strength. An outstanding discussion on mercerization effect and also the behavior observed were revealed by Symington et al., (2009). They emphasize on two important treatments by mercerization, which are NaOH concentration and time of processing might rise by 30% composite interfacial strength using an appropriate procedure in pre treatment. Generally the ranges in concentration of NaOH appear to be 1-25% and time of processing from 1-60 minute.

Generally, Sodium hydroxide (NaOH) is the most used chemical for bleaching and/or cleaning the surface of plant fiber (Mwaikamb and Ansell, 1999). Sodium hydroxide also structure changes of the fine native cellulose I to cellulose II by a procedure known as **mercerisation**.

Therefore, no matter the strength of condition of mercerization used there cannot be total conversion. The delignification of the fiber was as a result of the resultant increased in the tensile strength of the alkali treated fiber. The alkali treatment increased the surface roughness resulting in better mechanical interlocking and the amount of cellulose exposed on surface of the fiber (Wang, 2004). Therefore increases the number of possible reaction sites and allows better wetting of the fiber. When the alkali treatment was higher than the optimum conditions, excess delignification of the fibre took place and resulted in the weakness or fibre damage which leads to reduction on the tensile strength of the treated fiber.

Table 2 shows a literature summary mercerization stress treatment parameters focused on concentration of NaOH, processing temperature and duration of soaking. From Table 2, it shows a number of researches were conducted to determine the optimum concentration NaOH solution during the mercerization process at specific soaking duration and/or temperature of soaking.

Table 2: Gathered common mercerization treatment parameters from literature

Natural Fiber	Matrix	NaOH Treatment Parameters			Effect/Comments	Ref.
		Concentration %	Temp. (°C)	Duration		
Pineapple, sisal	Polyester	5 & 10%	30	1h	At 10% NaOH, excess delignification occurred. Thus, fiber become weak	(Mishra et al., 2003)
Sisal	Polyester	0.25,0.5,1.0,2.0 5.0 & 10% w/w	Room Temperature	1h	NaOH treatment decrease fiber density. 10% treatment results a rougher surface than untreated	(Sydenstricker et al., 2003)
Hemp & kenaf	Polyester	6%	19±2	48h	Cell wall densification identification from small positive change in fiber density observation	(Aziz and Ansell, 2004)
Curaua	Biodegradable resin	5,10 & 15wt%	Room Temperature	1h & 2h	Decrease of fiber diameter, fiber weight, fiber density and tensile strength with increasing NaOH content	(Gomes et al., 2004)
Henequen	HDPE	25w/v	25	1h	Surface modification increase the area of contact and further expose the cellulose microfibril. Thus, improve fiber wetting and impregnation	(Herrera and Valadez, 2005)
Kenaf	-	3,6 & 9%	Room Temperature and 95°C for 6% NaOH	3h	3% NaOH was ineffective to remove impurities on fiber surface, 9% NaOH show cleanest fiber surface	(Edeerozey et al., 2007)
Pineapple	-	2% w/v	95	2h	Decrease in tensile strength and Young modulus were probably due to decrease in the orientation	(Munawar et al., 2008)
Kenaf, Flax &	Epoxy	5%	Room Temperature	1h	NaOH treated kenaf and hemp composite absorb more water	(Syriccia et al., 2008)

Hemp					than silane only or alkali and silane treated samples	
Sisal & Oil palm	Natural rubber	0.5, 1, 2 & 4%	Ambient temperature	1h	Alkali treated composite exhibited better tensile properties than silane treated composite	(John et al., 2008)
Coir	Polypropylene	2, 4, 6, 8, & 10%	Room Temperature	4week	Dener NaOH solution provided more Na ⁺ and OH ⁻ ions to react with the substances on the fiber, causing greater amount of lignin, pectin, fatty acid and the cellulose to leach out, this would be detrimental to the fiber strength.	(Gu, 2009)
Ramie	PLA	5% w/v	Room Temperature	3h	Alkali treat composite exhibited better tensile properties than silane treated composite	(Yu et al., 2010)
Hemp	PLA	5%	Ambient temperature	30min	Show higher tensile strength (75.5MPa) and Young modulus (8.2GPa)	(Sawpan et al., 2011)

Usually there are two stages of performance evaluation when dealing with natural fiber reinforce composite. First, is the characteristic evaluation of natural fiber (Defoirdt et al., 2010; Aslan et al., 2011) and secondly is the composite fabricated evaluation (Asasutjarit et al., 2009). The majority of the review in literature was centered on two stages by considering some of the fiber modification (Bettini et al., 2010) or polymer matrix (Rassmann et al., 2011) and evaluates the final performance of the composite which is usually produced with variation matrix mixing ratio of the fiber. Numerous study have been conducted to assess natural fiber performance and the composite reinforcement (Rodriguez et al., 2011; John and Anandjiwala, 2008; Sgriceia et al., 2008; Alwar et al., 2009; Shinoj et al., 2010; Shinji, 2008). This evaluation of performance depend mostly characterization determination on the composite and can be obtained in terms of mechanical, thermal and physical properties. To determine ability of the material these characteristics are significant, mainly under severe and conditions that is critical, that are connected directly with performance of engineering.

In order to determine material ability these characteristics are important, particularly under critical and extreme conditions that are connected directly with performance of engineering. This paper was zoomed in effect of parameters for mercerization on natural fiber physical properties and the reinforced composite mechanical properties. Table 3 shows the natural fiber properties and fiber composite. In the stage of fiber, the properties of stress were shear interfacial strength of fiber-matrix and the fiber tensile properties. For the stage of composite, the following mechanical properties were reviewed and they are flexural strength, tensile strength and impact strength.

Table 3: Mercerization treatment effect on polymer composite mechanical properties

Natural Fiber	Matrix	Fiber Properties		Polymer Composite Properties			Effect/Comments	Ref.
		Shear Strength (MPa)	Tensile Strength (MPa)	Tensile Strength (MPa)	Flexural Strength (MPa)	Impact Strength (KJ/m ²)		
Sisal	Polyester	6.9 at 2% NaOH	375.4 at 2% NaOH	-	-	-	Sisal surface smoother at NaOH less than 2%, rougher when more than 5%	(Sydenstricker et al., 2003)
Hemp & kenaf	Polyester	-	-	-	Treated long fiber> Treated short fiber> untreated fiber	Untreated hemp polyester composite show greater work of fracture value	Flexural strength depends on fiber alignment and reason rich areas location	(Aziz and Ansell, 2004)
Curaua	Bio-degradable resin	-	Decreased for treated fiber compared	Treated = 137 Untreated = 124 (slightly)	-	-	2 factors contributed to composite tensile strength: 1: Interfacial bonding improvement,	(Gomes et al., 2004; 2007)

			to untreated fiber	higher) [49] Untreated PF = 275, PS = 327 10% NaOH PF = 276 PS = 334 [166]			2: Decrease in the coefficient of variation of alkali treated fiber strength	
Kenaf	-	-	6% NaOH at 95°C is 243.7	-	-	-	9% NaOH was too strong and might damage the fibers, thus resulting lower tensile strength	(Edeerozey et al., 2007)

In many textile industries, the process of mercerization was investigated on the temperature condition between 15-18°C, sodium hydroxide concentration 31-35%, treatment time of 55s (Mansikkamak et al., 2007). Mwaikambo et al., (2003) studied the mercerization effect on the hemp fibres with their mechanical properties and reveals that hemp fibres tensile strength have reached the maximum (1050MPa) when the sodium hydroxide concentration was 6%, and the Young modulus of hemp fibres reached maximum (65GPa) when the sodium hydroxide concentration was 4%, in contrast with the composite of hemp fibres without any pretreatment, Based on this modification tensile strength can increase, strain and modulus of composite 47.75%, 22.54% and 66.67% respectively. Research conducted by Gulati et al., (2006) reveals that treatment with mercerization of hemp fibres can acquire a bigger surface energy and the lowest absorption free energy and enthalpy of absorption.

5.2 Acetylation

The natural fibre treatment by acetylation is generally referred as method of esterification for cellulose plasticizing fibres. Acetylation of fibres with and without an acid catalyst to graft acetyl groups onto the cellulose structure. It reacts with groups of hydrophilic hydroxyl and swells the fibre cell wall. There by decreasing the nature of hydrophilic fiber leading to improvement in composites dimensional stability (Sreekala et al., 2000). In general, acetic acid and acetic anhydride individually do not react sufficiently with cellulosic fibres. The chemical compound that is responsible for the characteristic sour and odor taste of vinegar is called **acetic acid**.

Vinegar is usually containing about 4 to 8% acetic acid. Acetic acid is a part of vinegar; acetic acid were used and produced by humans earlier before the dawn of history was recorded. Actually, it's comes from a Latin name called vinegar, *acetum*. Vinegar is produced from alcohol dilute solutions, like by the action of certain bacteria in the presence of oxygen and wine.

It was called vinegar, because it comes from French, *vin aigre*, meaning "sour wine." However, the production of vinegar may also be from other beverages that are fermented like cider or malt. Since it is acidic, Vinegar has many properties that are helpful around our surroundings. When evaporation takes place in hard water, the mineral deposits left, and also those seen on tea kettles and plumbing fixtures, acids dissolved, all these can be removed by vinegar. Since vinegar is acidic, it inhibits bacteria growth; they are used for foods preservation, for preserving things like pickled vegetables, and as a mild cleaning disinfectant. Certainly, the taste of vinegar sour, due its acidity, has made it popular for cooking due to the flavor and for salad dressings. For numerous years, bulk commercial acetic acid was manufactured by the ethanol oxidation. Nowadays, Monsanto process, is the most industrial production technique of acetic acid it involves the reaction with monoxide of carbon with methanol under the rhodium complex catalyst influence at pressures of 30-40 atm and temperature of 180°C.

The dissociation constant for acidic acid (K_a value) is 1.8×10^{-5} at 25°C. In the industrial sector, acetic acid is an important chemical. About 3.2×10^9 kilograms were manufactured since 1999 in the United States. The chemical is primarily used to manufacture different various acetate esters. These substances are formed by reaction of acetic acid with substance containing hydroxyl (-OH) group. Cellulose that is seen in wood and cotton is a material that is polymeric and contains multiple hydroxyl groups. When they react with acetic acid to yield cellulose acetate it is used in textiles and films making. Few of the photographic films are produced by cellulose acetate, and rayon is also produced from fibers of cellulose acetate. Acetic acid is another ester of vinyl acetate, they form poly (vinyl acetate) when they polymerizes, they are used in glues for wood and paper, in water-based latex paints.

An acid anhydride is a compound that is connected to an acid by water loss. The preparation of acetic anhydride is by the acetic acid dehydration at a temperature of 800C. Other uses of acetic anhydride include the production of

chemicals for industry, perfumes, pharmaceuticals, synthetic fibers, plastics, weed killers and explosives. They always react with water and a dehydrating agent in mixtures reaction where it is necessary for water removal.

For this, to accelerate the reaction, fibres are initially soaked in acetic acid and consequently treated with acetic anhydride between the time periods of 1 to 3 hours with high temperature. Moreover, this treatment provides rough surface topography with a smaller amount of void contents that give better mechanical interlocking with the matrix (Tserki et al., 2005; Xue et al., 2007). Rowell et al., (2000) investigated acetic anhydride treatment on several types of cellulosic fibres to analyse the effects of equilibrium moisture content and reported that, this treatment improved moisture resistance properties. It was because of the taking away of hemicellulose and constituents of lignin from the treated fibre. Mishra et al., (2003) used acetic anhydride treatment on alkali pre-treated (5% and 10% solution of NaOH for 1hr at 300°C) dewaxed sisal fibre with glacial acetic acid and sulphuric acid was shown to have improved matrix of fibre adhesion composites characteristics. Bledzki et al., (2008) investigated the concentration of acetylation treated flax fibres and found that 50% higher thermal properties were observed. Moreover, 18% acetylated flax fibre polypropylene composites showed around 25% increase in flexural and tensile properties compare to the fibre composites untreated.

Acetylation is one of the most commonly evaluated methods for making natural fibrous materials more durable. Many ways to achieve acetylation of natural fibres have been evaluated (Rowell, 1983). One of the more simple routes comprises soaking of the fibre material in acetic anhydride, heating to a reaction temperature of 100-120°C for 1-4h and extraction of unreacted acetic anhydride by applying, for instance, vacuum (Rowell *et al.*, 1986; Rana *et al.*, 1997; Hill *et al.*, 1998; Rana *et al.*, 1999; Zafeiropoulos *et al.*, 2002). Enhancement in surface interaction between lignocellulosic fibers and polyolefins can also be achieved via introduction of an acetyl functional group (CH₃COO-) to the fibers surface. Acetylation of natural fibers with acetic anhydride (CH₃-C(=O)-O-C(=O)-CH₃), for instance, substitutes hydroxyl groups on cellulose molecules with acetyl groups which leads to hydrophobic behavior of fibers (Stamboulis et al., 2000). The reaction is as follows:



Acetic acid (CH₃COOH) is produced as a by-product of the reaction which must be removed from the fiber before introduction into polyolefins.

Rong et al., (2001) studied the effect of sisal fibers acetylation to reinforce epoxy resins. Acetylation was performed by a 50% acetic acid aqueous solution for 5 minutes (fiber/solution ratio: 1/25). They reveal that improved interfacial bonding is due to creation of hydrogen bonds between acetyl groups (on fiber surface) and hydroxyl or amine groups in the epoxy resin.

Chemical reaction with acetic anhydride modifies the fibre cell wall hydroxyl groups (functional group -OH) with acetyl groups (functional group -COCH₃) so that they become less accessible to enzymes and become hydrophobic. Acetylation is supposed to occur throughout the fibre (Hill *et al.*, 1998). Typical WPG values are 5-20% swelling trials show that acetylation reduces the swelling of particle board from 45% to less than 10% after 5 days of soaking in water (Rowell *et al.*, 1986). Biodegradation studies in horticultural soil confirm the significant protective effect of acetylation; in the longer term, however, biodegradation is not avoided (Hill *et al.*, 1998; Westin *et al.*, 2008).

5.3 Coupling

Is a fibre modification method in which substances are used in small quantities for surface treatment so that bonding will take place between it and other surfaces between the fiber and matrix? They are subdivided into two wide categories: (1) bonding agents and (2) surfactants (also called surface active agents). Currently, more than forty coupling agents are used in research and production for composites of natural fibre (Lu et al., 2000). Usually, the mostly used treatments are: silanes and isocyanates. Though, the effect of reinforcement in this coupling agent is weaker than that of mercerization (Kushwaha and Kumar, 2010).

Maleated coupling agents can also be used for surface covering of natural fibers in order to compatibilize them with polymers. In this approach, natural fibers are soaked in a solution of maleic anhydride or maleated polymers. Maleic anhydride groups react with hydroxyl groups on the surface of cellulosic fibers which results in decreased hydrophilic behavior.

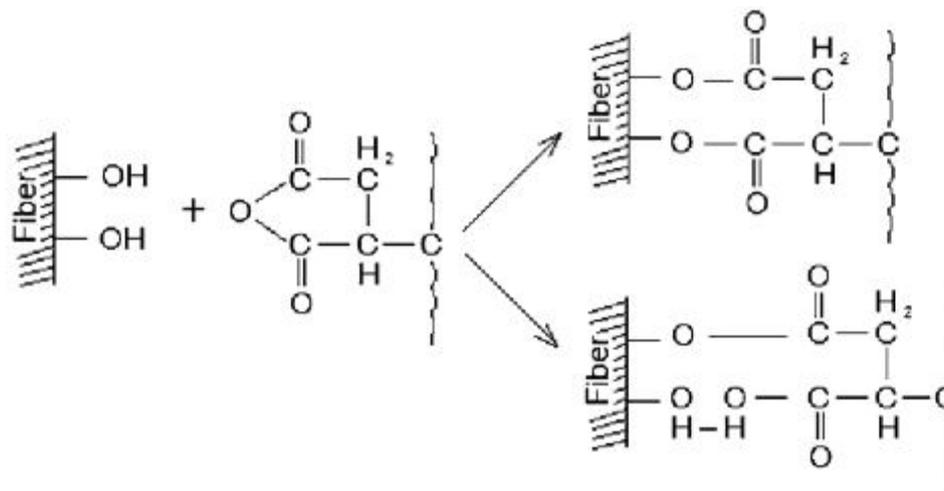


Figure 5: Surface treatment of natural fibers with maleated polypropylene (Ragoubi et al., 2010).

It involves the use of chemical to modify the fiber which enhances the interfacial adhesion. The fibre surface is treated with a compound forming chemical bonds with a bridge between fibre and matrix. There is numerous efficient processes of modifications by chemical methods, it includes;

- 1) Graft copolymerization.
- 2) Isocyanates treatment
- 3) Treatment with compounds that has methanol groups.
- 4) Organosilanes as coupling agents
- 5) Coupling agents of triazine.

- Chemical modification using graft copolymerization is an efficient process of natural fiber treatment. The methods involves using different monomers onto the cellulosic fibres surface (Gupta et al., 2002; Nayak et al., 1979).The reaction starts with free radicals molecule of cellulose. High-energy ionizing radiation of the cellulose is exposed. After they have been treated by transition metal ions, oxidative reagents, selected ions, initiate free radicals on cellulose, as initiating agents (Mishra et al., 2001).

-**Treatment with isocyanates:** The treatment by isocyanates can improve the composites mechanical properties of the reinforced fibres and PS or PVC as those cellulose fibres resin or polymer matrix. Polymethylene-polyphenyl-isocyanate (PMPPIC) in pure solution as plasticizer can also be used. If we compare the treatment of silanes and with that of isocyanates, it was seen to be clear, that isocyanatic treatment is more efficient than silane treatment. The same observation were seen, when PMPPIC is used to modify fibres or polymer matrix (Maldas and Kokta, 1989).

5.4 Furfurylation

Furfurylation of natural fibres is a recent development to protect natural fibre material. Fibres are impregnated with furfuryl alcohol or prepolymers of the furfuryl alcohol and cured at temperatures around 180°C. The resin encapsulates the fibres and at the same time occasionally may change the chemical structure of the fibre by reacting with hydroxyl groups of the lignin in the fibre. The mass percentage gain of resin by the fibres (WPG) is ca. 20% up to as high as 70% (Hoydonekx et al., 2007; Westin et al., 2008; Van der Zee et al., 2007). Soil burial trials have shown that furfurylation of fibres decreases the speed of decay significantly (Westin et al., 2008).

5.5 Oxidation

Modification by **oxidation** is carried out under mild condition. It involves the introduction of groups of carboxyl, group of aldehyde and ketone group into the chains of cellulose by the secondary group of hydroxyl into the chain of cellulose. After that, different primary (Nabar and Padmanabhan 1950-76) and secondary oxidative system have been investigated. Presently, because of the outstanding selective oxidation, TEMPO-NaBr-NaClO and TEMPO-NaClO-NaClO₂ oxidative systems have attracted more attention all over the world. Matsui et al., (1997; 1998) studied the effect of pretreatment in ozone oxidation on the graft copolymerization of methyl methacrylate on the hemp surface of the fibres. They reveal that as the oxidation time increases, the hydroperoxide (HPO) increases from 0 mol/cell molecule to 160 mol/cell molecule. Fibres crystallinity index decreases from 69.7% to 68.3%, but there is significant increase in grafting degree from 14% to 129%.

5.6 Crosslink

Crosslink modification involves the use of compounds that are multi functional having than two groups that are functional always used as crosslink agent to crosslink the cellulose interchain by reacting it with the groups of hydroxyl. Modification of crosslink cellulosic fibres is carried out by the method of etherification (Jones and Rayburn, 1961) and esterification (Clingman and Schwenker, 1965).The important commercial application for crosslinking of cellulose in textile finishing of cellulose-based fabrics with end-use properties are, e.g. resistance to wrinkle, permanent press and properties of easy care.

5.7 Silane Coupling Agents

Silane treatment is one the chemical modifications method that is frequently used to increase surface adhesion between lignocellulosic fibers and polymers. Silanes are chemical compounds (with Si_nH_{2n+2}) and are also commonly used to compatibilize glass fiber with polymers. In the presence of moisture, silanols are produced through hydrolyzation of alkoxy groups. Silanol groups then react with hydroxyl groups on the natural fiber surface and create stable covalent bonds with the cell walls. Therefore, hydrophilic behavior of the fiber decreases which leads to enhanced fiber/matrix interaction. An example of silane treatment of natural fibers is as follows (Stamboulis et al., 2000):



Abdelmouleh et al., (2002) studied the reinforcement of low density polyethylene (LDPE) and natural rubber using different types of cellulosic fibers. Cellulose fibers were added to the matrices before and after chemical treatments using three silane coupling agents namely: γ - ethacryloxypropyltrimethoxy (MPS), γ -mercaptoproyltrimethoxy (MRPS) and hexadecyltrimethoxy-silane (HDS). They proposed the following schematic illustration as shown in figure 6 to explain the interaction between LDPE and cellulosic fibers treated by MPS.

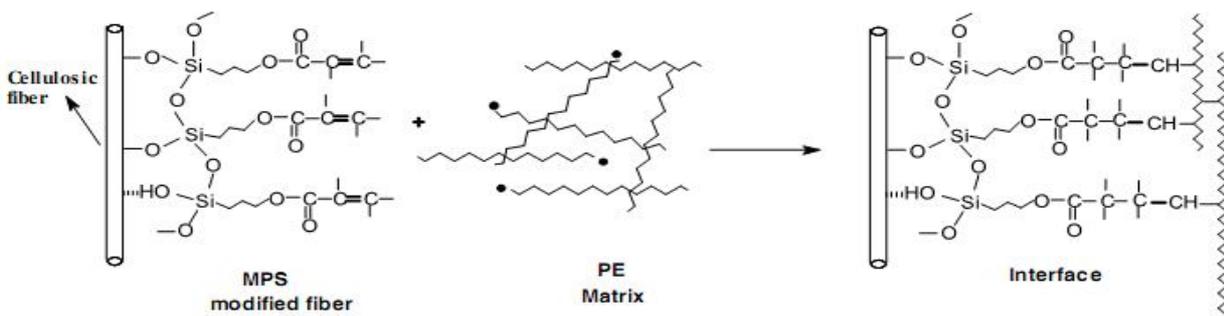


Figure 6: Schematic representation of interaction between LDPE and MPS-modified fiber (Abdelmouleh et al., 2002)

Increase interaction between matrix and cellulosic fiber was confirmed by morphological and mechanical characterizations. Morphological observations revealed that surface treatment with silanes increase the compatibility between LDPE and cellulosic fibers as shown in figure 7.

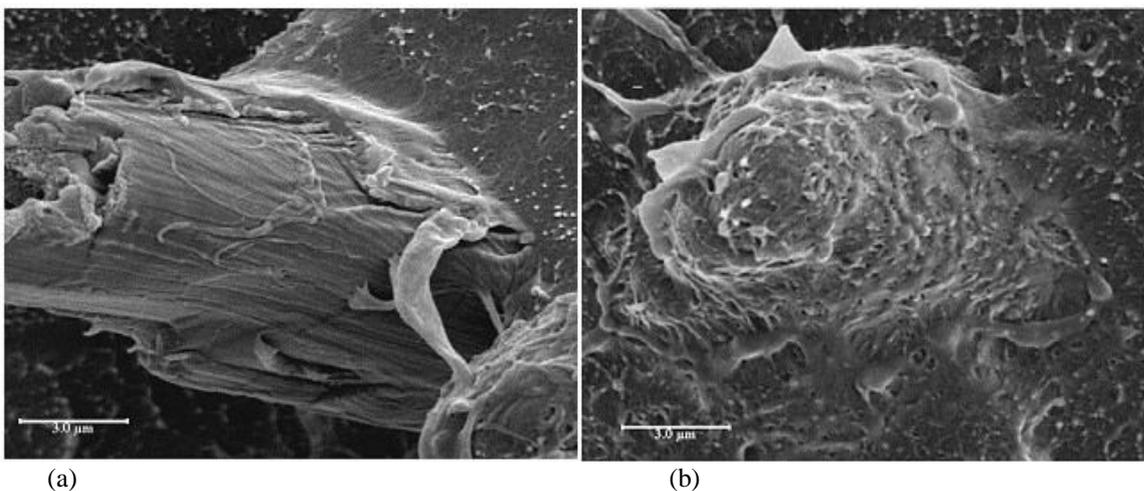


Figure 7: Effect of cellulosic fibers surface treatment with MPS: a) untreated and b) treated fiber (Abdelmouleh et al., 2002).

5.8 Biological

Biological modification methods involve treating with natural occurring microorganisms, namely fungi and bacteria. These treatments take place in aqueous environments and performed cheaply, but they are consuming time and pollute the water. The most commonly used fibre biological treatment is retting treatment. Retting is a method of controlled degradation of plant stems in order to free the fibres from the bundles of fibre, and to separate the fibre from the woody core and epidermis. During the process of retting, bacteria (predominantly Clostridia species) and fungi, give out enzymes to degrade pectic and hemicellulosic compounds in the middle lamella between the individual cells of the fibre (Prasad and Sain, 2003). In general, the retting process high quality fibre is produced, but it is dependent on the conditions of weather (Hepworth et al., 2000) and the acquired skill and farmer judgement. Some reports (Oujai and Shanks, 2004; Bhattacharya and Shah, 2004) showed the treatment with enzymatic was more favorable compared to treatment with conventional sodium hydroxide for removing noncellulosics from natural fibres. The removal of noncellulosics of fibre can be done without destroying the structure of cellulose crystalline by enzymatic treatment.

5.9 Benzoylation

Benzoylation treatment uses benzoyl chloride to decrease the hydrophilicity of the fibre and improve fibre matrix adhesion, thereby increasing the composite strength. It also enhances thermal stability of the fibre (Manikandan et al., 2001; Xue et al., 2007). During benzoylation treatment alkali pretreatment is used to activate the fibre hydroxyl groups. Then the fibre is soaked in benzoyl chloride solution for 15mins. Afterwards ethanol solution is used for 1h to remove benzoyl chloride that adhered to the fibre surface followed by washing with water and oven dried. Joseph et al., (1996) implied benzoyl chloride treatment on alkali pre-treated sisal fibre and reported higher thermal stability compared fibre composites that is not treated. Similar treatment was carried out on flax fibre reinforced low density polyethylene composites by Wang et al., (2007) and reported 6% and 33% improvement on tensile strength and moisture resistance properties.

5.10 Peroxide Treatment

Peroxide treatment can improve the interface properties of fibre and matrix. The peroxide-induced grafting of polyethylene adheres to the surface of fibre and the peroxide initiated free radicals react with the hydroxyl group of the fibre and matrix. As a result, good fibre matrix adhesion occurs. This treatment also reduced moisture absorption tendency by the fibre and improves thermal stability (Wang et al., 2007; Kalaprasad et al., 2004). Sapiha et al., (1990) reported that, treatment with benzoyl peroxide or dicumyl peroxide of the cellulosic fibre led to higher mechanical properties of composites. The mechanism of peroxide treatment involves alkali pre-treatment on the fibres then coated with benzoyl peroxide or dicumyl peroxide (around 6% concentration) in the acetone solution for 30 mins. Complete decomposition of peroxide can be achieved by heating the solution at higher temperature (Xue et al., 2007, Kalaprasad et al., 2004). Joseph et al., (1996) investigated optimum concentration of benzoyl peroxide (6%) and dicumyl peroxide (4%) treatment on short sisal fibre-reinforced polyethylene composites and better tensile strength was observed.

5.11 Nanotechnology Modification

Nanotechnology (NT) is a modification method introduced by the National Nanotechnology Initiative of USA and NT can be defined as the manipulation, understanding and control of matter at the dimensions within 1 to 100 nm. Presently, governments in the entire world are investing heavily in NT and many are seeing it as the next industrial revolution. There are huge quantity of research which the government are sponsoring presently, the applications of NT is across all sectors of the economy and enable the growth of new critical enabling science with wide commercial potential, such as nanoscale-based manufacturing processes, nanoelectronics and structured-nano materials. It was established in current years that the use of NT is used for the natural fiber modification in order to bring new ideas into the fibres surface and improve the final performance of products of fibres-based. This modification is applied in textiles (Sawhney et al., 2008; Xue et al., 2009), paper industry (Ngo et al., 2011) successfully. It is believed that the applications of NT to natural fibres modification have offered higher level of economic potential for the natural fibre-based industrial growth.

Different procedures were used to increase the usage of nanoparticles on the natural fibre surface, deposition through layer-by-layer (Dubas et al., 2006; Ugur et al., 2011) and sol-gel process (Wang and Chen, 2006; Li et al., 2007; Alongi et al., 2011; Brancatelli et al., 2011) are the major process which are usually used by various researchers. Various types of nanoparticles (e.g. AgNPs (Dubas et al., 2006; Chen and Chiang 2008; Zhang et al., 2009; Ravindra et al., 2010; Onar et al., 2007), TiO₂ (Onar et al., 2007; Zheng et al., 2011), SiO₂ (Schmidt et al., 2000; Yeh et al., 2007; Yeh et al.,

2007), ZnO (Yadav et al., 2006; Becheri et al., 2008) were developed to impart multifunctional properties (e.g. anti-bacteria, UV resistant, antiwrinkle finishing, water repellent) to natural fibres.

6. PHYSICAL MODIFICATION OF NATURAL FIBRE COMPOSITES

Physical modification is carried out by making use of a number of instruments to modify the fibre surface and structural properties for the purpose of raising the fibres strength. The fibres hydrophobicity thereby influences the matrix with mechanical bonding. The traditional process involves thermotreatment (Ray et al., 1976; Da Cunha et al., 1993), calendaring (Semsarzadeh, 1986; Bisanda and Ansell, 1991), and stretching (Haig et al., 1990). Thermotreatment is a methods used in natural fibres modification using traditional procedure. It involves subjecting the fibres into treatment by heat beyond the glass temperature of transition lignin; it was proposed that lignin will migrate and softened to the fibre surface. Based on the report by Da Cunha et al., (1993), lignin kraft is having a glass transition temperature at 142°C. Degradation of lignin begins around temperature of 214°C; therefore fibres are heat to 200°C could likely result in softening (Brett and Waldron, 1996). During the process of flax fibres heating above 150°C for about 2hr, the lignin and hemicellulose are depolymerised into lower phenolic functionalities and molecular aldehyde (Stamboulis, 2000), which are combined by further curing reaction forming into water resistant resins. The resin keeps together all the microfibrils. (Prasad and Sain, 2003) treated thermally hemp fibres are reserved in an air enclosed vessel as well an inert environment, and reveals there was fibres openings by heating, even at the length also along the direction of width and diameter.

(a) Physical modification involving **calendaring, stretching, treatment by thermo** and hybrid yarns production does not modify the fibres chemical composition but they change the structural properties and the surface the fibre and thereby influencing the polymer mechanical bonding (Adekunle et al., 2010). Other method of surface fibre treatment using method of physical can be electrical discharge like (cold plasma, corona). **Corona treatment** procedure is a process very interesting for activation of surface oxidation. The techniques change the energy of surface for the fibres cellulose and activation increases of the fibre and increase the amount of aldehyde groups (Sakata et al., 1993).

(b) **Heat Treatment:** The process of heat treatment to make natural fibre material more durable is the oldest of the methods discussed. The process usually consists of the following steps: starting material may be dried or fresh fibre material; high temperature kiln drying at 100-130°C; intensive heat treatment at 180-250°C for several hours; cooling down and remoisturing (StoraEnso, 2009). To prevent the fibres from burning, some processes use water vapour, others use nitrogen, others use natural oils like rapeseed, linseed or sunflower oil (Rapp, 2001).

7. DURABILITY OF NATURAL FIBER COMPOSITES

Durability of natural fiber composites in a dry environment does not seem to be an issue. The first generation of natural fiber composites using a petrochemical-based thermoplastic like PP or PE has been marketed as highly durable and free of maintenance (Morris and Cooper, 1998). The basic idea was that the natural fibres were encapsulated in a polymer that was resistant to environmental attack and therefore immune to weathering, etc. However, even improved compositions, including coupling agents, preservatives and UV absorbers, have shown that the natural fibres remain sensitive to decay (Westin *et al.*, 2008). Proper encapsulation of the fibres makes them less accessible for biodegradation, but not to such an extent that they become fully durable. For that purpose the fibre itself has to be modified to become more durable.

The biodegradation of natural fibres is related to the enzymatic activity of microorganisms. These micro organisms feel at home and become active at elevated moisture contents, while natural fibres easily take up water. The mere fact of water absorption already reduces the strength and stiffness performance of natural fibres. Also, uptake of water causes dimensional changes, thus causing cracks in the composite material and making the fibres more accessible for influences from outside. Biodegradation of the fibres causes further reduction in properties. Protection against biodecay is generally obtained (1) by making the fibres less accessible to enzymes, (2) by changing the substrate specific configuration such that enzymes do not recognise the polysaccharide polymers in the fibre anymore, (3) by removing the components most sensitive to biodegradation and (4) by making fibres less hygroscopic.

8. Performance of natural fiber composites

The performance of natural fiber depends on the following investigated mechanical properties. They includes - Tensile strength, flexural strength, and impact strength properties are the most frequently studied mechanical properties of natural fiber reinforced plastic composites. Impact strength is the weakest of all these materials in mechanical performance behaviour. Apart from these - tensile, compressive and flexural properties, dynamic mechanical behavior, the long-term performance (creep behavior), impact properties are studied for natural fiber composites.

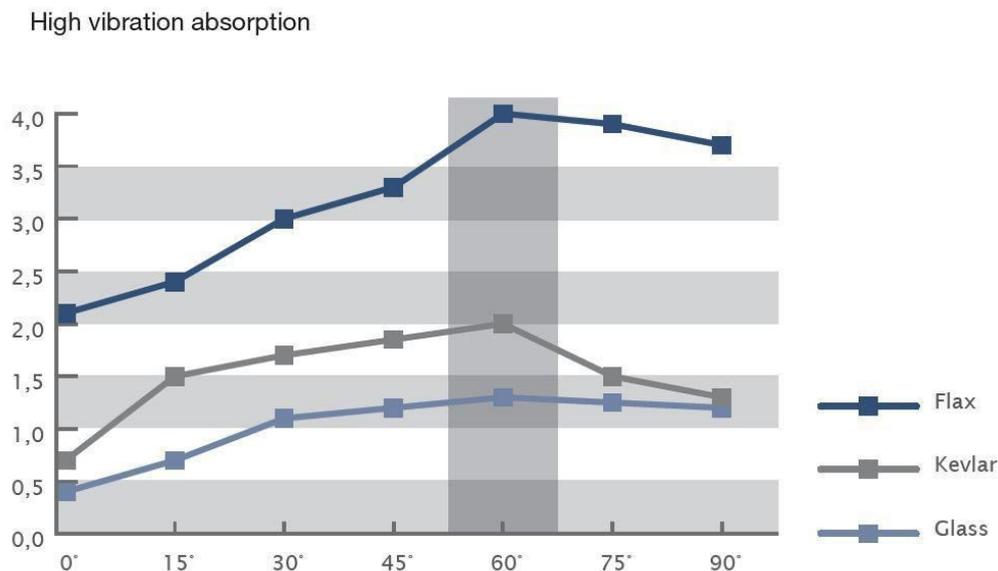


Figure 14: Properties of some fibers (Egidija, 2011)

9. DEVELOPMENTS OF NATURAL FIBER COMPOSITES

Natural fibers have many main areas of attention for the growth of natural fiber composites, which give the potential impacts to the economy, impacts to the environmental, and the natural fiber composite ability to meet economic, social and material needs in the globe. The natural fiber reinforced polymer composite has contributed to the enhancement in the growth of biocomposites in view of sustainability and performance. Many researches are going everywhere in the globe, it focuses natural fibre composites performance. Nowadays natural fiber reinforced bio-composites have taken over extensively in oil and gas sectors in many applications. Very soon, if natural fiber composite materials and design associated methods are adequately grown-up to let their application widespread eg, as construction materials. The development of methods, systems, and standards could see natural fiber composite materials at a distinct benefit over the traditional materials. Many researches are going on to expand natural fiber composites material and discover their use as construction materials in different oil and gas industries, especially for load bearing applications.

10. CONCLUSION

This review paper has discussed the top areas where natural fiber composites are penetrating the oil and gas industry. The important of natural fiber as agent of reinforcing in polymer composites was assessed and reviewed from status point, there performance and applications. It was reported that natural fiber reinforced composite materials have gained increase value in oil and gas, onshore, offshore, and various applications in the industry because of their weight is lighter, , carbon dioxide sequestration, competitive specific strength and stiffness, improvement in energy recovery, flexibility and ease to manufacture, and friendliness to the environment as well as their renewable nature.

It is stated that their scenario in the market for various load bearing applications for composites has changed because of the introduction of most recent biodegradable and biobased polymers. Nanotechnology shows much opportunity for the improvement of products for biocomposite in giving nanotechnology-based coatings to enhance uptake of water, volatile organic compounds and reduction in biodegradation and flame resistance. The usage of cellulose for nanocrystalline is beginning to explore for different uses because they are tougher than steel material and also stiffer than aluminum. Composite of nanocrystalline cellulose reinforced will soon offer higher value, performance, durability, service-life, and utility at the same time be a complete sustainable technology.

The natural fibre composite materials might be this century material revolution focusing on sustainability, “cradle-to-grave” design, eco-efficiency, green chemistry and, industrial ecology that might direct the growth of a latest invention of natural fiber materials. Natural fiber reinforced polymeric composites will succeed depending on suitable processing methods, fibers modification to develop the bond or adhesion among the biopolymer and the fiber, modification of matrix and after they are treated to get better a performance and long-standing toughness and retardancy in fire. Very soon, the application of natural fiber composites will increase in oil and gas sector. There different usage will be based on their more development. There are many problems to be solved before the use of these composites grows to be completely competitive with fiber synthetic composites. Natural fiber composites are sustainable and could be fully recyclable, but could be more expensive if fully biobased and biodegradable and they are extremely sensitive to temperature and moisture. If an appropriate matrix is applied, the natural fiber can be 100 percent biodegradable, but

this biodegradation may have difficulty in controlling it. Natural fiber composites demonstrate excellent specific properties, except their variability is high in terms of properties. Natural fiber weaknesses could be defeated through the growth of more highly developed natural fibers processing and the composites. Though, a complete superiority in environment for natural fiber composites contrast to fiber made up of synthetic composites is still having problem due to their relatively too much processing time, which means more energy is consumed.

Consequently, more study can be done on underlisted problems:

- (i) Extraction of fiber should give additional elemental and technical fibers for efficient embodiment into matrix of the composite to keep away the variability of fiber
- (ii) The interfacial properties that is improved among the natural fiber and matrix must be achieved
- (iii) To overcome the absorption of moisture relating toward long term strength (humidity, temperature and ultra violet radiation), fiber and matrix modification, resistance to fire, Properties and hybridization, durability, and optimization of processing and manufacturing focusing on the specific final products
- (iv) Biodegradability and assessment of life cycle must be carefully researched
- (v) The matrix, composites (thermoplastic and thermosets), coupling agents and additives prepared from resources that are renewable must be improved. Searching for latest and better biopolymers in order to substitute petroleum-based polymeric materials must be continued to completely meet up with the environmental goals in future
- (vi) Multi disciplinary research, biotechnology, involving agricultural, polymeric material, and manufacturing aspects of the composite should be investigated.
- (vii) Manufacturing technologies of the composite must be improved and accepted by the new bio-based polymer
- (viii) Recent development and growth of nano-composites, wide research should focus on cellulosic nano-fibers as well as inorganic nano-fillers (e.g., nano-clays) should be incorporated into the composite materials of natural fiber reinforced.

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