ENHANCEMENT OF BONDING STRENGTH OF PALMYRA PALM FIBER REINFORCED POLYMER MATRIX COMPOSITES

BY

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A thesis submitted to Materials and Metallurgical Engineering Department, Bangladesh University of Engineering and Technology (BUET), Dhaka, in partial fulfillment of the requirements for the degree of Bachelor of Science in Materials and Metallurgical Engineering.

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Riad Morshed Rezaul Md. Tashfiul Amin Chowdhury Dhaka, 2016

CERTIFICATE

This is to certify that the thesis entitled **"Enhancement Of Bonding Strength Of Palmyra Palm Fiber Reinforced Polymer Matrix Composites"**, submitted by **Riad Morshed Rezaul(1011028) and Md. Tashfiul Amin Chowdhury(1011040)** in partial fulfillment of the requirements for the award of Bachelor in Science in the Department of Materials and Metallurgical Engineering of Bangladesh University of Engineering and Technology (BUET), Dhaka in an authentic work carried out under my supervision and guidance.

To my best knowledge the matter embodied in the thesis has not been submitted to elsewhere for the award of any degree.

Date: 24 February, 2016 BUET, Dhaka Professor Dr Md. Aminul Islam Department of Materials and Metallurgical Engineering of Bangladesh University of Engineering and Technology (BUET), Dhaka

Dedicated to our Parents and well wishers

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Abstract

The use of Palmyra palm fiber for reinforcing polymer has already been considerably studied both from a scientific and commercial point of view over recent years. These materials are environmental friendly and have pleasant aesthetical properties. Palmyra palm tree fibers are attractive fillers for thermoset polymers, they are biodegradable, and the present research work has been undertaken with an objective to explore the "Enhancement of matrix fiber bonding strength of palmyra palm fiber reinforced polymer composites". The tensile, flexural, thermal properties of the pure polyester resin were studied. Furthermore the same properties are studied with the aligned reinforcing element (palmyra palm fiber). For more detail study, the fibers were sodium hydroxide (NaOH) treated by 2 and 4 weight percent. These properties including pure, untreated fiber composite,2% and 4% treated composite castings were also investigated under load of 20 kg.

Among these materials, the fibers showed better tensile and flexural properties and pure polyester casting showed comparatively inferior properties. The properties of the composites fall between these two. Chemically treated fibers showed degradation of properties. But when these treated fibers were use to make composites, then the composites shows superior tensile and flexural properties.

Composites reinforced with treated fibers showed better tensile propertied upto 2% NaOH treated but there is a decline in the tensile properties in case of composites reinforced with 4% NaOH treated fiber.

When the same casting was prepared using 20 kg load, there is an enhancement of properties comparing to unloaded condition.

The tensile and flexural modulus of the composite showed an increase in tensile and flexural modulus when the fibers were alkali treated up to a certain point. Further treated leads to deterioration of physical properties.

Improved tensile and flexural properties means enhanced bonding between the fiber and matrix.

Thermo gravimetric analysis (TGA) was used to know the decomposition of composites as a function of increasing temperature. TGA results reveals that degradation starts from temperature 163.47 degree and end up 409.38 degree Celsius

Chapter 1

Introduction

A fibre reinforced polymer (FRP) is a composite material consisting of a polymer matrix imbedded with high-strength fibres, such as glass, aramid and carbon, Palmyra palm [1]. Generally, polymer can be classified into two classes, thermoplastics and thermosettings. Thermoplastic materials currently dominate, as matrices for bio-fibres; the most commonly used thermoplastics for this purpose are polypropylene (PP), polyethylene, and poly vinyl chloride (PVC); while phenolic, epoxy and polyester resins are the most commonly used thermosetting matrices [2]. In the recent decades, natural fibres as an alternative reinforcement in polymer composites have attracted the attention of many researchers and scientists due to their advantages over conventional glass and carbon fibres [3]. These natural fibers include flax, hemp, jute, sisal, kenaf, coir, kapok, banana, henequen and many others [4]. The various advantages of natural fibres over man-made glass and carbon fibres are low cost, low density, comparable specific tensile properties, nonabrasive to the equipments, non-irritation to the skin, reduced energy consumption, less health risk, renewability, recyclability and biodegradability [5]. These composites materials are suitably applicable for aerospace, leisure, construction, sport, packaging and automotive industries, especially for the last mentioned application [6]. However, the certain drawback of natural fibres/polymers composites is the incompatibility between the hydrophilic natural fibres and the hydrophobic thermoplastic matrices. This leads to undesirable properties of the composites. It is therefore necessary to modify the fibre surface by employing chemical modifications to improve the adhesion between fibre and matrix [6].

There are many factors that can influence the performance of natural fiber reinforced composites. Apart from the hydrophilic nature of fibre, the properties of the natural fibre reinforced composites can also be influenced by fibre content / amount of filler.

In general, high fibre content is required to achieve high performance of the composites. Therefore, the effect of fibre content on the properties of natural fibre reinforced composites is particularly significance. It is often observed that the increase in fibre loading leads to an increase in tensile properties [7]. Another important factor that significantly influences the properties and interfacial characteristics of the composites is the processing parameters used. Therefore, suitable processing techniques and parameters must be carefully selected in order to yield the optimum composite products. There are tremendous research conducted to evaluate performance of natural fiber and it

reinforced composite [8-10].

These eco friendly materials have various interesting properties, which make itan attractive and comparable to traditional synthetic reinforced material. Some

Of natural fiber advantages are abundantly available, inexpensive, low density, high specific stiffness and strength, lightweight, desirable fiber aspect ratio, minimal healthhazards, non-abrasive, outstanding insulation properties, enhanced energy recovery, renewability and biodegradability [9-13]. However, although natural fiber reinforced composite seem to give a promising benefit as compare to synthetic fiber, there are still several critical issues need to be addressed before manufacturing industry gains full confidence to enable wide-scale acceptance of this material in a global market. The shape, size and strength of the natural plant fibers may vary widely depending on cultivation environment, geographical origin, plant maturity, retting technique, and composite manufacturing process [14-15].

The numerous highlight problems in dealing with natural fiber were its hydrophilic nature, which leads to an adhesion problem with hydrophobic nature of the polymer matrix.

Hydrophilic character of natural fibers are incompatible with hydrophobic polymer matrix and have a tendency to form aggregates. Furthermore, it exhibit poor resistant to moisture, which lead to high-water absorption, subsequently resulting in poor mechanical properties and dimensional stability of the natural fiber reinforced composites [15-16]. Therefore, chemical modifications either on natural fiber, polymer matrix or both materials are an alternative solution to overcome these challenges [17-21]. The chemical modification is attempted to improve natural fiber hydrophobic nature, interfacial bonding between matrix and fiber, surface roughness and wettability, and also decrease moisture absorption, leading to the enhancement of mechanical properties of the natural fiber Reinforced composites [22]. Mercerization is a common fiber treatment that extensively used by the number of researcher [23-26]. However, for various types of fiber, distinct mercerization treatment conditions were used, and very limited paper took a partial review on these condition interaction effects on fiber and its composite properties enhancement.

This article aims to review the reported works on the effects of fiber loading, chemical treatments, manufacturing techniques and process parameters on tensile properties and flexural properties of palmyra palm fiber reinforced polymer matrix composites to justify its bonding strength.

Chapter 2

Literature Review

2.1 Study Composite Materials

Many of our modern technologies require materials with unusual combinations of properties that cannot be met by the conventional metal alloys, ceramics, and polymeric materials. This is especially true for materials that are needed for aerospace, underwater, and transportation applications. For example, aircraft engineers are increasingly searching for structural materials that have low densities, are strong, stiff, and abrasion and impact resistant, and are not easily corroded. This is a rather formidable combination of characteristics. Frequently, strong materials are relatively dense; also, increasing the strength or stiffness generally results in a decrease in impact strength [27].

Material property combinations and ranges have been, and are yet being, extended by the development of composite materials. Generally speaking, a composite is considered to be any multiphase material that exhibits a significant proportion of the properties of both constituent phases such that a better combination of properties is realized. According to this **principle of combined action**, better property combinations are fashioned by the judicious combination of two or more distinct materials.

Therefore with knowledge of the various types of composites, as well as an understanding of the dependence of their behaviors on the characteristics, relative amounts, geometry/distribution, and properties of the constituent phases, it is possible to design materials having properties that are better than those found in metal alloys, ceramics and polymeric materials. The complex composite structure of a modern ski is shown in figure 2.1.

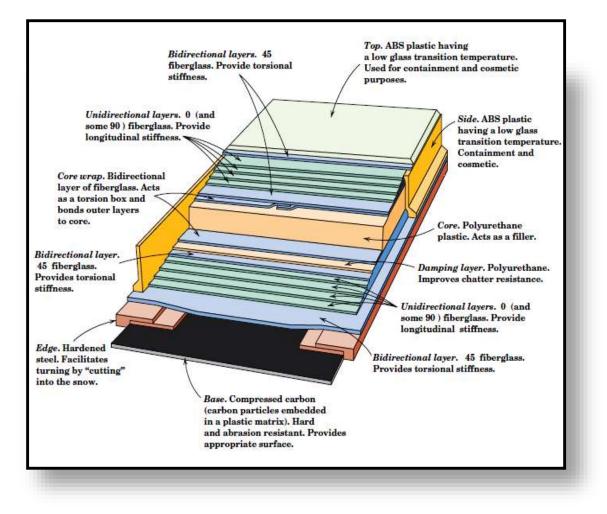


Figure 2. 1: Complex composite structure of a modern ski [27]

2.2 Composite Materials

From the previous section we can conclude a composite is a multiphase material that is *artificially made*, as opposed to one that occurs or forms naturally. They are engineered materials made from two or more constituent materials with significantly different physical or chemical properties which remain separate and distinct on a macroscopic level within the finished structure. The different materials work together to produced a new material, which combines all of the properties of the previously separate materials. Within the composite it is still possible to easily tell the different materials apart. They do not tend to blend or dissolve into each other. For example wood is a composite of cellulose fibers held together with a glue or matrix of soft lignin.[27]

Many composite materials are composed of just two phases; one is termed the **matrix**, which is continuous and surrounds the other phase, often called the **dispersed phase**. The properties of composites are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase. "Dispersed phase geometry" in this context means the shape of the particles and the particle size, distribution, and orientation; these characteristics are represented in Figure 2.2

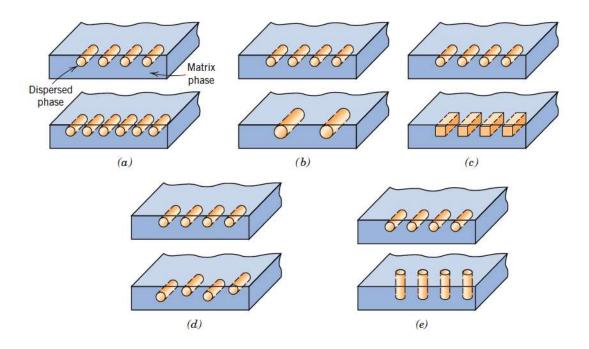


Figure 2. 2: Schematic representation of geometrical and spatial characteristics of dispersed phase that may influence the properties of composites: (a) concentration, (b) size, (c) shape, (d) distribution, and (e) orientation. [27]

2.3 Classification of Composites

Composites can be classified according to its two main components:

- ✤ Matrix
- Dispersed phase

2.3.1 Composite Classification on the Basis of the Dispersed Phase

One simple scheme for the classification of composite materials is shown in Figure 2.3, which consists of three main divisions:

- Particle-reinforced,
- Fiber-reinforced, and
- Structural composites.

At least two subdivisions exist for each. The dispersed phase for particle-reinforced composites is equiaxed (i.e., particle dimensions are approximately the same in all directions); for fiber-reinforced composites, the dispersed phase has the geometry of a fiber (i.e., a large length-to-diameter ratio). Structural composites are combinations of composites and homogeneous materials [27]

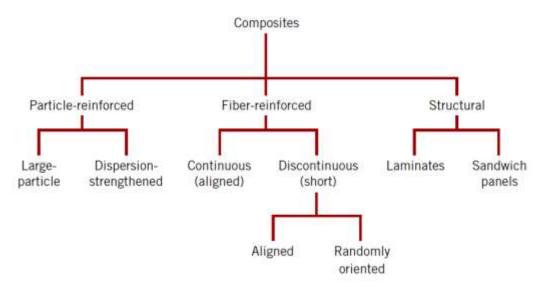


Figure 2. 3 : Classification scheme for composites on the base of the dispersed phase [27]

2.3.1.1 Particle-Reinforced Composites

As shown in figure 2.3, **large-particle** and **dispersion-strengthened composites** are the two sub-classifications of particle-reinforced composites. The distinction between these is based upon reinforcement or strengthening mechanism. The term "large" is used to indicate that particle–matrix interactions cannot be treated on the atomic or molecular level; rather, continuum mechanics is used. For most of these composites, the particulate phase is harder and stiffer than the matrix. These reinforcing particles tend to restrain movement of the matrix phase in the vicinity of each particle. In essence, the matrix transfers some of the applied stress to the particles, which bear a fraction of the load. The degree of reinforcement or improvement of mechanical behavior depends on strong bonding at the matrix–particle interface [27].

Some polymeric materials to which fillers have been added are really large-particle composites. Again, the fillers modify or improve the properties of the material and/or replace some of the polymer volume with a less expensive material; the filler.

Another familiar large-particle composite is concrete, which is composed of cement (the matrix), and sand and gravel (the particulates).

Particles can have quite a variety of geometries, but they should be of approximately the same dimension in all directions (equiaxed).For effective reinforcement, the particles should be small and evenly distributed throughout the matrix. Furthermore, the volume fraction of the two phases influences the behavior; mechanical properties are enhanced with increasing particulate content [27].

For dispersion-strengthened composites, particles are normally much smaller, with diameters between 0.01 and 0.1 m (10 and 100 nm). Particle–matrix interactions that lead to strengthening occur on the atomic or molecular level. The mechanism of strengthening is similar to that for precipitation hardening. Whereas the matrix bears the major portion of an applied load, the small dispersed particles hinder or impede the motion of dislocations. Thus, plastic deformation is restricted such that yield and tensile strengths, as well as hardness, improve.

Metals and metal alloys may be strengthened and hardened by the uniform dispersion of several volume percent of fine particles of a very hard and inert material. The dispersed phase may be metallic or nonmetallic; oxide materials are often used. Again, the strengthening mechanism involves interactions between the particles and dislocations within the matrix, as with precipitation hardening [27].

2.3.1.2 Fiber-Reinforced Composites

Technologically, the most important composites are those in which the dispersed phase is in the form of a fiber. Design goals of **fiber-reinforced composites** often include high strength and/or stiffness on a weight basis. These characteristics are expressed in terms of **specific strength** and **specific modulus** parameters, which correspond, respectively, to the ratios of tensile strength to specific gravity and modulus of elasticity to specific gravity. Fiber-reinforced composites with exceptionally high specific strengths and moduli have been produced that utilize low-density fiber and matrix materials. As noted in Figure 16.2, fiber-reinforced composites are too short to produce a significant improvement in strength [27]

2.3.1.2.1 Influence of Fiber Length

Some critical fiber length is necessary for effective strengthening and stiffening of the composite material. This critical length is dependent on the fiber diameter *d* and its ultimate (or tensile) strength σ_f^* , and on the fiber–matrix bond strength (or the shear yield strength of the matrix, whichever is smaller) τ_c according to

$$l_c = \frac{\sigma_f^* d}{2\tau_c}$$

For a number of glass and carbon fiber–matrix combinations, this critical length is on the order of 1 mm, these ranges between 20 and 150 times the fiber diameter [27].

When a stress equal to σ_f^* is applied to a fiber having just this critical length, the stress-position profile shown in Figure 2.4*a* results; that is, the maximum fiber load is achieved only at the axial center of the fiber. As fiber length *l* increases, the fiber reinforcement becomes more effective; this is demonstrated in Figure 2.4*b*, a stress-axial position profile for l > lc when the applied stress is equal to the fiber strength. Figure 2.4*c* shows the stress-position profile for l < lc.

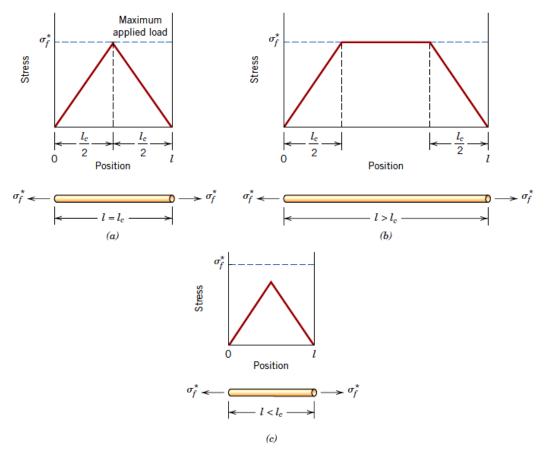


Figure 2. 4 : Stress-position profiles when fiber length l (a) is equal to critical fiber length(b)is greater than critical length, and (c)is less than critical fiber length [27]

Fibers for which l >> lc(normally l > 15lc) are termed *continuous; discontinuous* or *short fibers* have lengths shorter than this. For discontinuous fibers of lengths significantly less than lc, the matrix deforms around the fiber such that there is virtually no stress transference and little reinforcement by the fiber. These are essentially the particulate composites as described above. To affect a significant improvement in strength of the composite, the fibers must be continuous [27].

2.3.1.2.2 Influence of Fiber Orientation and Concentration

The arrangement or orientation of the fibers relative to one another, the fiber concentration, and the distribution all have a significant influence on the strength and other properties of fiber-reinforced composites. With respect to orientation, two extremes are possible: (1) a parallel alignment of the longitudinal axis of the fibers in a single direction, and (2) a totally random alignment. Continuous fibers are normally aligned (Figure 2.5*a*), whereas discontinuous fibers may be aligned (Figure 2.5*b*), randomly oriented (Figure 2.5*c*), or partially oriented. Better overall composite properties are realized when the fiber distribution is uniform.

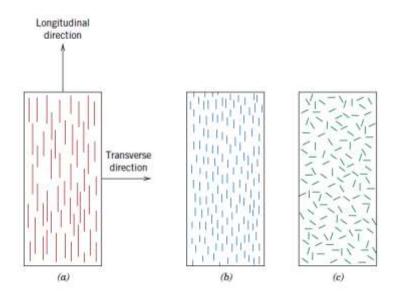


Figure 2. 5: Schematic representation of (a) continuous fibers (b) discontinuous and aligned, and (c) discontinuous and randomly orientated fiber-reinforced composites [27]

2.3.1.3 Structural Composite

A structural composite is normally composed of both homogeneous and composite materials, the properties of which depend not only on the properties of the constituent materials but also on the geometrical design of the various structural elements [27]. The two most common structural composites are:

- Laminar Composites
- Sandwich Panels

2.3.1.3.1 Laminar Composites

Laminar composites are composed of two-dimensional sheets or panels that have a preferred high strength direction such as found in wood or continuous and aligned fiber-reinforced plastics. The layers are stacked and cemented together such that the orientation of the high-strength direction varies with each successive layer as shown in figure 2.6 Example of a relatively complex structure is plywood [27].

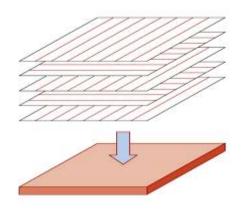


Figure 2. 6: Successive stacking in laminar composites [27]

2.3.1.3.2 Sandwich Panels

Sandwich panels, considered to be a class of structural composites, are designed to be light-weight beams or panels having relatively high stiffnesses and strengths. A sandwich panel consists of two outer sheets, or faces, that are separated by and adhesively bonded to a thicker core. The outer sheets are made of a relatively stiff and strong material, typically aluminum alloys, fiber-reinforced plastics, titanium, steel, or plywood; they impart high stiffness and strength to the structure, and must be thick

enough to withstand tensile and compressive stresses that result from loading. The core material is lightweight, and normally has a low modulus of elasticity. Core materials typically fall within three categories: rigid polymeric foams (i.e., phenolics, epoxy, polyurethanes), wood (i.e., balsa wood), and honeycombs (figure 2.7).

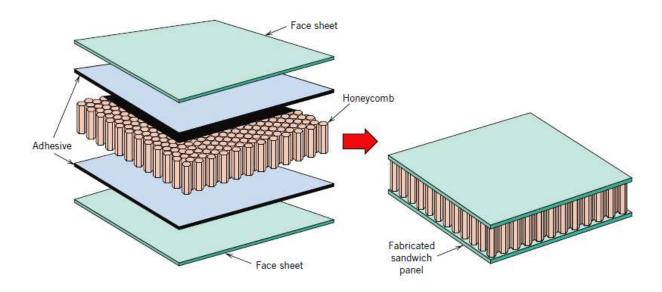


Figure 2. 7: Schematic diagram showing construction of a honeycomb core sandwich panel [27]

2.3.2 Composite classification on the basis of matrices

Three type's composites are available if classified on the basis of matrices:

- Metal Matrix Composites (MMC)
- Ceramic Matrix Composites (CMC)
- Polymer Matrix Composites (PMC) [27]

2.3.2.1 Metal Matrix Composites (MMC)

As the name implies, for **metal-matrix composites** (MMC's) the matrix is a ductile metal. These materials may be utilized at higher service temperatures than their base

metal counterparts; furthermore, the reinforcement may improve specific stiffness, specific strength, abrasion resistance, creep resistance, thermal conductivity, and dimensional stability [27]

In a MMC one component is a metal and other may be different metal or another material, such as a ceramic or organic compound. It can therefore be concluded that a metal matrix composite (MMC) is a material consisting of a metallic matrix combined with a ceramic (oxides, carbides) or metallic (lead, tungsten, molybdenum) dispersed phase.

The major advantages of MMC's can be listed as follows:

- High strength even at elevated temperatures
- High stiffness (modulus of elasticity)
- High thermal conductivity
- Excellent abrasion resistance
- Good creep resistance
- ✤ High wear resistance
- High thermal stability

2.3.2.2 Ceramic Matrix Composites (CMC)

Ceramic matrix composites (CMC's) are a subgroup of composite materials as well as a subgroup of technical ceramics. They consist of ceramic fibers embedded in a ceramic matrix, thus forming a ceramic fiber reinforced ceramic (CFRC) material. The matrix and fibers can consist of any ceramic material, whereby carbon and carbon fibers can also be considered a ceramic material.

The motivation to develop CMC's was to overcome the problems associated with the conventional technical ceramics like alumina, silicon carbide, aluminium nitride, silicon nitride or zirconia – they fracture easily under mechanical or thermomechanical loads because of cracks initiated by small defects or scratches. The crack resistance is – like in glass – very low. To increase the crack resistance or fracture toughness, particles (so-called monocrystalline whiskers or platelets) were embedded into the matrix [27].

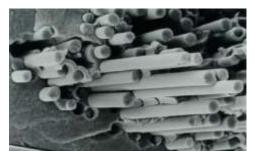


Figure 2. 8: Fracture surface of a fiber-reinforced ceramic composed of sic fibers and SiC matrix [27]

The fiber pull-out mechanism shown is the key to CMC properties

Some of the typical properties of Ceramic Matrix Composites are:

- High mechanical strength even at high temperatures
- High thermal shock resistance
- High stiffness
- High toughness
- Low density
- High corrosion resistance even at high temperatures

2.3.2.3 Polymer Matrix Composites (PMC)

Polymer-matrix composites (**PMC's**) consist of a polymer resin as the matrix, with fibers as the reinforcement medium. These materials are used in the greatest diversity of composite applications, as well as in the largest quantities, in light of their room-temperature properties, ease of fabrication, and cost.

Use of non-reinforced polymers as structure materials is limited by low level of their mechanical properties: tensile strength of one of the strongest polymers - epoxy resin is 20000 psi (140 MPa). In addition to relatively low strength, polymer materials possess low impact resistance.

Reinforcement of polymers by strong fibrous network permits fabrication of Polymer Matrix Composites (PMC) characterized by the following properties:

- High tensile strength;
- ✤ High stiffness;
- High Fracture Toughness;
- Good abrasion resistance;
- ✤ Good puncture resistance;
- ✤ Good corrosion resistance;
- ✤ Low cost.

2.4 Types of Polymer Matrices

The two main kinds of polymers matrices used are:

- Thermosets
- Thermoplastics

2.4.1 Thermosets

Thermosets have qualities such as a well-bonded three-dimensional molecular structure after curing. They become permanently hard when heat is applied and do not soften upon subsequent cooling. During the initial heat treatment, covalent crosslinks are formed between adjacent molecular chains; these bonds anchor the chains together to resist the vibrational and rotational chain motions at high temperatures. In thermosets crosslinking is usually extensive. Thermosets are usually harder, stronger, and more brittle than thermoplastics, and have better dimensional stability. Merely changing the basic composition of the resin is enough to alter the conditions suitably for curing and determine its other characteristics. They can be retained in a partially cured condition too over prolonged periods of time, rendering Thermosets very flexible. Thus, they are most suited as matrix bases for advanced conditions fiber reinforced composites. Figure 2.8 shows some kinds of thermosets.

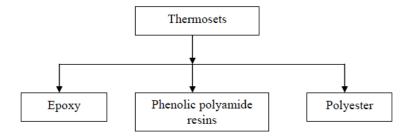


Figure 2. 9: Kinds of thermosets [27]

2.4.2 Advantages of Thermoset over Thermoplastics

Traditional Fiber Reinforced Polymer Composites, or FRP Composites for short, use a thermosetting resin as the matrix, which holds the structural fiber firmly in place. Common thermosetting resin includes:

- Polyester Resin
- Vinyl Ester Resin
- Epoxy

- Phenolic
- Urethane

The most common thermosetting resin used today is polyester resin, followed by vinyl ester and epoxy. Thermosetting resins are popular because uncured, at room temperature; they are in a liquid state.

This allows for convenient impregnation of reinforcing fibers such as fiberglass, carbon fiber, or Kevlar [27].

2.4.3 Properties and Benefits of Thermoset Resins

As mentioned, a room temperature liquid resin is easy to work with. Laminators can easily remove all air during manufacturing, and it also allows the ability to rapidly manufacture products using a vacuum or positive pressure pump.

(Closed Molds Manufacturing) Beyond ease of manufacturing, thermosetting resins can exhibit excellent properties at a low raw material cost.

- Properties of thermoset resins include:
- Excellent resistance to solvents and corrosives
- Resistance to heat and high temperature
- Fatigue strength
- Tailored elasticity
- Excellent adhesion
- Excellent finishing (polishing, painting, etc.)

In a thermoset resin, the raw uncured resin molecules are crossed linked through a catalytic chemical reaction. Through this chemical reaction, most often exothermic, the resin creates extremely strong bonds to one another, and the resin changes state from a liquid to a solid.

A thermosetting resin, once catalyzed, it cannot be reversed or reformed.

Meaning, once a thermoset composite is formed, it cannot be remolded or reshaped. Because of this, the recycling of thermoset composites is extremely difficult. The thermoset resin itself is not recyclable, however, there are a few new companies who have successfully removed the resin through pyrolization and are able to reclaim the reinforcing fiber [27].

2.5 Polyester

Polyester is a category of polymers that contain the ester functional group in their main chain. As a specific material, it most commonly refers to a type called polyethylene terephthalate (PET). Polyesters include naturally occurring

chemicals, such as in the cutin of plant cuticles, as well as synthetics through stepgrowth polymerization such as polybutyrate. Natural polyesters and a few synthetic ones are biodegradable, but most synthetic polyesters are not. This material is used vary widely in clothing.

Depending on the chemical structure, polyester can be a thermoplastic or thermoset. There are also polyester resins cured by hardeners; however, the most common polyesters are thermoplastics [28].

Fabrics woven or knitted from polyester thread or yarn are used extensively in apparel and home furnishings, from shirts and pants to jackets and hats, bed sheets, blankets, upholstered furniture and computer mouse mats. Industrial polyester fibers, yarns and ropes are used in tyre reinforcements, fabrics for conveyor belts, safety belts, coated fabrics and plastic reinforcements with high-energy absorption. Polyester fiber is used as cushioning and insulating material in pillows, comforters and upholstery padding. Polyester fabrics are highly stain-resistant— in fact, the only classes of dyes which *can* be used to alter the color of polyester fabric are what are known is dispersing dyes [29].

Polyester fibers are sometimes spun together with natural fibers to produce a cloth with blended properties. Cotton mixed with Polyester blends can be strong, wrinkle and tear-resistant, and reduce shrinking. Synthetic fibers in polyester also create materials with water, wind and environmental resistance compared to plant-derived fibers. Cons of cotton and polyester blends include being less breathable than cotton and trapping more moisture while sticking to the skin. They are also less fire resistant and can melt when ignited [30].

Polyester blends have been renamed so as to suggest their similarity or even superiority to natural fibers (for example, China silk, which is a term in the textiles industry for a 100% polyester fiber woven to resemble the sheet and durability of insect-derived silk).

Polyesters are also used to make bottles, films, tarpaulin, canoes, liquid crystal displays, holograms, filters, dielectric film for capacitors, film insulation for wire and insulating tapes. Polyesters are widely used as a finish on high-quality wood products such as guitars, pianos and vehicle/yacht interiors. Thixotropic properties of spray-applicable polyesters make them ideal for use on open-grain timbers, as they can quickly fill wood grain, with a high-build film thickness per coat. Cured polyesters can be sanded and polished to a high-gloss, durable finish.

Liquid crystalline polyesters are among the first industrially used liquid crystal polymers. They are used for their mechanical properties and heat-resistance. These traits are also important in their application as an abradable seal in jet engines.

2.5.1 Types

Polyesters as thermoplastics may change shape after the application of heat. While combustible at high temperatures, polyesters tend to shrink away from flames and self-extinguish upon ignition. Polyester fibers have high tenacity and E-modulus as well as low water absorption and minimal shrinkage in comparison with other industrial fibers.

Unsaturated polyesters (UPR) are thermosetting resins. They are used as casting materials, fiberglass laminating resins and non-metallic auto-body fillers. Fiberglass-reinforced unsaturated polyesters find wide application in bodies of yachts and as body parts of cars.

According to the composition of their main chain, polyesters can be:

Compos ition of the main chain	Homo- or copolym er	Examples of polyesters	Examples of manufacturing methods
Aliphati c	Homopol ymer	Polyglycolide or Polyglycolic acid (PGA)	Polycondensation of glycolic acid
		Polylactic acid (PLA)	Ring-opening polymerization of lactide
		Polycaprolactone (P CL)	Ring-opening polymerization of caprolactone
		Polyhydroxyalkano ate (PHA)	
		Polyhydroxybutyrat e (PHB)	
	Copolym	Polyethylene	

Table 2. 1: Composition of the main chain [30]

	er	adipate (PEA)	
		Polybutylene succinate (PBS)	Polycondensation of succinic acid with 1,4-butanediol
		Poly(3- hydroxybutyrate- co-3- hydroxyvalerate)(P HBV)	Copolymerization of 3-hydroxybutanoic acid and 3-hydroxypentanoic acid, butyrolactone and valerolactone (oligom eric aluminoxane as a catalyst)
Semi- aromatic	Copolym er	Polyethylene terephthalate (PET)	Polycondensation of terephthalic acid with ethylene glycol
		Polybutylene terephthalate (PBT)	Polycondensation of terephthalic acid with 1,4-butanediol
		Polytrimethylene terephthalate (PTT)	Polycondensation of terephthalic acid with 1,3-propanediol
		Polyethylene naphthalate (PEN)	Polycondensation of at least one naphthalene dicarboxylic acid with ethylene glycol
Aromati c	Copolym er	Vectran	Polycondensation of 4-hydroxybenzoic acid and 6-hydroxynaphthalene-2- carboxylic acid

Increasing the aromatic parts of polyesters increases their glass transition temperature, melting temperature, thermal stability, chemical stability...

Polyesters can also be telechelic oligomers like the polycaprolactone diol (PCL) and the polyethylene adipate diol (PEA). They are then used as prepolymers.

2.5.2 Polyester resin

Polyester resins are unsaturated synthetic resins formed by the reaction of dibasic organic acids and polyhydric alcohols. Maleic Anhydride is a commonly used raw material with diacid functionality. Polyester resins are used in sheet moulding compound, bulk moulding compound and the toner of laser printers. Wall panels fabricated from polyester resins reinforced with fiberglass — so-called fiberglass reinforced plastic (FRP) — are typically used in restaurants, kitchens, restrooms and other areas that require washable low-maintenance walls [30].

Unsaturated polyesters are condensation polymers formed by the reaction of <u>polyols</u> (also known as polyhydric alcohols), organic compounds with multiple alcohol or hydroxy functional groups, with saturated or unsaturated dibasic acids. Typical polyols used are glycols such as ethylene glycol; acids used are phthalic acid and maleic acid. Water, a by-product of esterification reactions, is continuously removed, driving the reaction to completion. The use of unsaturated polyesters and additives such as styrene lowers the viscosity of the resin. The initially liquid resin is converted to a solid by cross-linking chains. This is done by creating free radicals at unsaturated bonds, which propagate in a chain reaction to other unsaturated bonds in adjacent molecules, linking them in the process. The initial free radicals are induced by adding a compound that easily decomposes into free radicals. This compound is usually and incorrectly known as the catalyst. Substances used are generally organic peroxides such as benzoyl peroxide or methyl ethyl ketone peroxide [30].

Polyester resins are thermosetting and, as with other resins, cure exothermically. The use of excessive catalyst can, therefore, cause charring or even ignition during the curing process. Excessive catalyst may also cause the product to fracture or form a rubbery material.

2.5.3 Polyester processing

After the first stage of polymer production in the melt phase, the product stream divides into two different application areas which are mainly textile applications and packaging applications. In table 2, the main applications of textile and packaging of polyester is listed.

Table 2. 2: Textile and packaging polyester application list [30]

Textile	Packaging
Staple fiber (PSF)	Bottles for CSD, Water, Beer, Juice, Detergents
Filaments POY, DTY, FDY	A-PET Film
Technical yarn and tire cord	Thermoforming
Non-woven and spunbond	BO-PET Biaxial oriented Film
Mono-filament	Strapping

Polyester-based polymer (melt or pellet)

Abbreviations: PSF = Polyester Staple Fiber; POY = Partially Oriented Yarn; DTY = Draw Textured Yarn; FDY = Fully Drawn Yarn; CSD = Carbonated Soft Drink; A-PET = Amorphous Polyester Film; BO-PET = Biaxial Oriented Polyester Film;

A comparable small market segment (much less than 1 million tonnes/year) of polyester is used to produce engineering plastics and masterbatch.

In order to produce the polyester melt with a high efficiency, high-output processing steps like staple fiber (50–300 tonnes/day per spinning line) or POY /FDY (up to 600 tonnes/day split into about 10 spinning machines) are meanwhile more and more vertically integrated direct processes. This means the polymer melt is directly converted into the textile fibers or filaments without the common step of pelletizing. We are talking about full vertical integration when polyester is produced at one site starting from crude oil or distillation products in the chain oil \rightarrow benzene \rightarrow PX \rightarrow PTA \rightarrow PET melt \rightarrow fiber/filament or bottle-grade resin. Such integrated processes are meanwhile established in more or less interrupted processes at one production site. Eastman Chemicals were the first to introduce the idea of closing the chain from PX to PET resin with their so-called INTEGREX process. The capacity of such vertically integrated production sites is >1000 tons/day and can easily reach 2500 tons/day.

Besides the above-mentioned large processing units to produce staple fiber or yarns, there are ten thousands of small and very small processing plants, so that one can estimate that polyester is processed and recycled in more than 10 000 plants around the globe. This is without counting all the companies involved in the supply industry, beginning with engineering and processing machines and ending with special additives, stabilizers and colors. This is a gigantic industry complex and it is still growing by 4–8% per year, depending on the world region.

2.5.4 Synthesis

Synthesis of polyesters is generally achieved by a polycondensation reaction. See "condensation reactions in polymer chemistry". The general equation for the reaction of a diol with a diacid is :

```
(n+1) R(OH)_2 + n R'(COOH)_2 \rightarrow HO[ROOCR'COO]_n roh + 2n H_2O [30]
```

2.5.4.1 Azeotrope esterification

In this classical method, an alcohol and a carboxylic acid react to form a carboxylic ester. To assemble a polymer, the water formed by the reaction must be continually removed by azeotrope distillation [30].

2.5.4.2 Alcoholic transesterification



2.5.4.3 Transesterification:

An alcohol-terminated oligomer and an ester-terminated oligomer condense to form an ester linkage, with loss of an alcohol. R and R' are the two oligomer chains, R" is a sacrificial unit such as a <u>methyl group</u> (<u>methanol</u> is the byproduct of the esterification reaction) [30].

2.5.4.4 Acylation (HCl method)

The acid begins as an acid chloride, and thus the polycondensation proceeds with emission of hydrochloric acid (HCl) instead of water. This method can be carried out in solution or as an enamel [31].

2.5.4.5 Silyl method

In this variant of the hcl method, the carboxylic acid chloride is converted with the trimethyl silyl ether of the alcohol component and production of trimethyl silyl chloride is obtained [31]

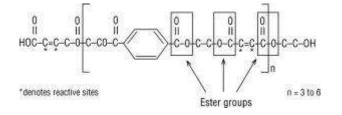
2.5.5 Acetate method (esterification)

- a) Silyl acetate method
- **b**) Ring-opening polymerization

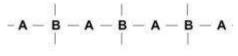
Aliphatic polyesters can be assembled from lactones under very mild conditions, catalyzed anionically, cationically or metallorganically. A number of catalytic methods for the copolymerization of epoxides with cyclic anhydrides have also recently been shown to provide a wide array of functionalized polyesters, both saturated and unsaturated.

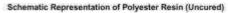
2.5.6 Structural Isomers of Polypropylene

The figure below shows the idealised chemical structure of a typical polyester. Note the positions of the ester groups (CO - O - C) and the reactive sites ($C^* = C^*$) within the molecular chain.

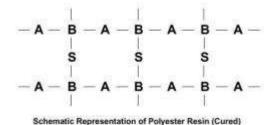


The molecular chains of the polyester can be represented as follows, where 'B' indicates the reactive sites in the molecule.





With the addition of styrene 'S ', and in the presence of a catalyst, the styrene crosslinks the polymer chains at each of the reactive sites to form a highly complex threedimensional network as follows:



The polyester resin is then said to be 'cured'. It is now a chemically resistant (and usually) hard solid. The cross-linking or curing process is called 'polymerisation'. It is a non-reversible chemical reaction. The 'side-by-side' nature of this cross-linking of the molecular chains tends to means that polyester laminates suffer from brittleness when shock loadings are applied [31].

2.5.7 Chemical and Physical Properties of Polyester

2.5.7.1 Physical properties of polyester

- a) **Tenacity:** Polyester filaments and staple fibre are strong due to their crystalline nature. The crystalline nature permits for the formation of highly effective Vander wall's forces as well as since hydrogen bonds which provided the fibre its good tenacity. The tenacity remains unchanged when wet since the fibre resists the entry of water molecules to a significant extent.
- b) **Elastic plastic nature:** The very crystallinity of the fiber prevents wrinkling and creasing. Repeated stretching and straining causes, distortion of the polymer system as the Vander wall's forces cannot withstand much stretching.
- c) **Hygroscopic nature:** Filaments and staple fibers are hydrophobic. The lack of polarity and the very crystalline structure resists the entry of water molecules into the polymer system. The hydrophobic nature of the polymer system attracts fats, greases, oils, acid or any other greasy soils. It is believed to be oleophilic. The oleophilic nature makes it not easy to remove grease by soap but by dry-cleaning with organic solvents.
- d) Thermal properties: It is a poor heat conductor and it has low resistance to heat. It melts on heating. Polyester textile materials can be permanently heat-set. It is a thermoplastic fibre meaning that it is capable of being shaped or turned when heated. Thermoplastic fibres heated under strictly controlled temperatures soften and can then be made to similar to a flat, creased or pleated configuration. When cooled thermoplastic fibres retain the new configuration.

2.5.7.2 Chemical properties of polyester

- a) Effect of acids: These polymers are resistant to acids.
- b) **Effect of alkalis:** Alkaline conditions as seen in laundering hydrolyse the ester groups in polyester polymers. The crystalline nature prohibits hydrolysis to a greater extent and it is the surface of filament which gets hydrolysed. Continued laundering results in hydrolysis and materials get fewer as the surface film of the fibre gets lost.
- c) **Effect of bleaches:** It does nor requie bleaching. It retains its whiteness and requires only chlorine bleaches to be used when essential.

- d) **Sunlight:** It withstands the sun's ultra-violate radiations and is resistant to acidic pollutants in atmosphere.
- e) **Color Fastness:** It is not easy for dye molecule to penetrate the fibre when dyed, it retains its color after regular wash.
- f) Micro-Organisms: It is resistant to bacteria and other mcro-organisms.

Some of the important properties of polypropylene have been listed below:

- Light in weight.PP has the lowest density (i.e 0.90-0.92 g/cm3) of the resins used in packaging.
- Excellent resistance to stress and high resistant to cracking (i.e. It has high tensile and compressive strength)
- ✤ High operational temperatures with a melting point of 160°C
- Excellent dielectric properties
- It is highly resistant to most alkalis and acid, organic solvents, degreasing agents and electrolytic attack. On the contrary is less resistance to aromatic, aliphatic and chlorinated solvents and UV.
- Non-toxic
- Non-staining
- ✤ Easy to produce, assembly and an economic material

The table below shows the physical properties and mechanical properties of polyester.

Table 2. 3: Physical and Mechanical properties of Polyester [31]

Property	Epoxy	Polyester
Viscosity at 250C µ (cP)	12000-13000	250-350
Density ρ (g.cm ⁻³)	1.16	1.09
Heat distortion temperature HDT (°C)	100	85
Modulus of elasticity E (GPa)	5.0	3.3
Flexural strength (MPa)	60	45
Tensile strength (MPa)	73	40
Maximum elongation (%)	4	1

Table 1. Properties of epoxy and polyester resins.

2.6 Palmyra palm fiber

Environmental awareness, new rules, and legislation are forcing to seek new materials that are ecofriendly and do not have any harmful effect on environment. For the past several years, public attention has gone on natural fibers as a resource due to the fast growth. Now-a-days, natural fibers are widely used as reinforcements both in partially and totally biodegradable natural fiber Composites. Natural fibers like, cotton, coir, sisal jute etc. have attracted the attention of scientists and technologists.

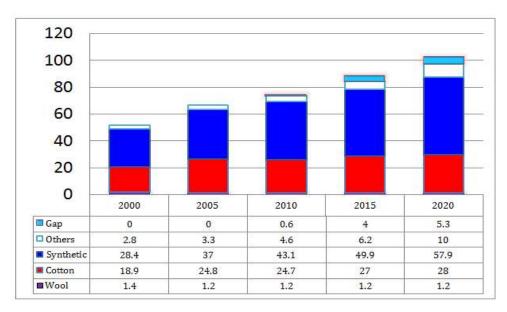


Figure 2. 10: Chart showing demand for fibers [31]

From chart, it is seen that in the year 2010 world fiber demand was 73.8 million metric tons this will be 102.4 million metric tons by 2020. But experts show that world total fiber production will be 97.1 million metric tons including wool 1.2 million metric tons, cotton 28 million metric tons, synthetic 57.9 million metric tons and others 10 million metric tons. So there will be a gap between demand and supply, which is about 5.3 million metric tons due to the reduction of arable land and limitation of water availability [31].

We know that first basic two needs of human beings are food and cloth. The meeting process of these two is contradictory to each other. If we grow more raw materials for clothing then we will have less land to grow food crops. If we can able to use the wastage of food crops as alternative source for clothing, then it will be a good solution to meet up the fiber demand. Such type possible new sources are banana, pine apple fiber, betel nut husk fiber etc.

The promotion of the use of natural fibers as CO2 neutral resource is believed to contribute to a greener planet. The transition towards a bio-based economy and sustainable developments as a consequence of the Kyoto protocols on greenhouse gas reduction and CO2 neutral production offers high perspectives for natural fiber

markets. Plant fibers from agricultural crops are renewable materials which have potential for creating green products and replacing synthetic materials which are currently being used such as glass fiber, carbon fiber and plastic fibers. Among others, natural fibers (e.g., flax, jute or sisal) reinforced materials have important significance for reduction of density in automobile construction components due to its higher specific stiffness and specific tensile strength. Many attempts were made by the scientists and technologists to utilize natural fibers in the fabrication of composites. The low cost, less weight and density makes the natural fibers an attractive alternative. In recent years, due to increased awareness, extensive studies have been done on various new natural fibers such as sisal, pineapple, banana, oil palm, areca nut etc.

2.6.1 A brief view of Palmyra Palm (Borassus spp)

Borassus (Palmyra Palm) is a genus of five species of palms native to tropical regions of Africa, Asia and New Guinea. They are tall palms, capable of growing up to 30 meter high. The leaves are long, fan-shaped, 2 to 3 m in length. The flowers are small, in densely clustered spikes, followed by large, brown, roundish fruits.

The 5 species of Borassus are Borassus aethiopium - African Palmyra Palm (tropical Africa) Borassus flabellifer - Asian Palmyra Palm (southern Asia) Borassus heineanus - New Guinea Palmyra Palm (New Guinea) Borassus madagascariensis - Madagascar Palmyra Palm (Madagascar) Borassus sambiranensis - Sambirano Palmyra Palm (Madagascar) [31]

2.6.2 Scientific classification

Kingdom	Plantae
Division	Magnoliophyta
Class	Liliopsida
Order	Palmaceae
Family	Palmae
Genus	Borassus L.
Species	Borassus flabellifer

2.6.3 Botanical description

The Palmyra palm is a member of the family Palmae which includes evergreen trees. It is a long-lived plant that may live as long as over 100 years. It is one of the most important homestead and plantation crops of Bangladesh with multipurpose uses. The botanical description of this plant is cited under the following heads according to Morton [17].



Figure 2. 11: Palm tree [31]

2.6.4 Stem

The palmyra palm is a large tree up to 30m high and the trunk may have a circumference of 1.7m at the base. It erect, woody and unbranched stem with no secondary thickenings. In most of the varieties it takes 3 to 4 years to develop a stem above the ground [31].

2.6.5 Leaves

There may be 25-40 fresh leaves. They are leathery, gray green, fan-shaped, 1-3 m wide, folded along the midrib; are divided to the center into 60-80 linear- lanceolate, 0.6-1.2 m long, marginally spiny segments. Their strong, grooved petioles, 1-1.2 m long, black at the base and black-margined when young, are edged with hard spines [31].

2.6.6 Fruits

Each palm may bear 6-12 bunches of about 50 fruits per year. An average crop of B. Flabellifer in Ceylon is 350 fruits. The coconut-like fruits are three-sided when young, becoming rounded or more or less oval, 12-15 cm wide, and capped at the base with overlapping sepals. The outer covering is smooth, thin, leathery, and brown, turning nearly black after harvest. Inside is a juicy mass of long, tough, coarse, white fibers coated with yellow or orange pulp. Within the mature seed is a solid white kernel which resembles coconut meat but is much harder. When the fruit is very young, this kernel is hollow, soft as jelly, and translucent like ice, and is accompanied by a watery liquid, sweetish and potable [17].



Figure 2. 12: Palm fruit [31]

2.6.7 Inflorescence

Palms generally start to form inflorescences at the beginning of the dry season (November to January). The male and female inflorescences are carried on separate trees: the male tree begins to develop the inflorescence in November or December while the female tree commences one to two months later. Each palm may bear from eight to fifteen inflorescences per year. The male inflorescence lasts approximately 45 to 60 days and the female 60 to 70 days. Both male and female inflorescences are "tapped" for juice collection. Some palms, especially the female, also have inflorescences during the rainy season [31].

2.6.8 Geographical distribution and habitat

It grows wild from the Persian Gulf to the Cambodian-Vietnamese border; is commonly cultivated in Bangladesh, Cambodia, China South-Central, India, Jawa, Laos, Lesser Sunda Is., Malaya, Myanmar, Socotra, Sri Lanka, Sulawesi, Thailand, and Vietnam, South and Southeast Asia and occasionally in other warm regions including Hawaii and southern Florida. In India, it is planted as a windbreak on the plains The Palmyra palm has long been one of the most important trees of India, where it is used over 800 different ways. In India, Borassus is a very important palm, and it has been estimated that as many as 40 million palms may be growing in Tamil Nadu State alone, making it one of the most common trees in India, ranking second only to the coconut palm. The leaves are used for thatching, mats, baskets, fans, hats, umbrellas, and writing paper. The stalks are used to make fences and also produce a strong, wiry fiber suitable for cordage and brushes. The black timber is hard, heavy, and durable and is highly valued for construction. The tree grows well in poor acidic sandy soils and produces juice during the dry season in regions where plant growth is negligible in the absence of irrigation. The palm tree receives no agronomic inputs other than what is applied to companion crops [31]

2.6.9 Palmyra production in Bangladesh

Palmyra palm is produced over the country in Bangladesh as homestead crop; however, the major Palmyra palm producing areas of Bangladesh are- Roadside in the village area, Allis of agricultural field and homestead areas [31]

2.6.10 About Palm Fiber

The fiber is extracted from the palm fruit. The chemical compositions of the fiber are α -Cellulose 53.4%, Hemi-cellulose 29.6% and Lignin 17% [32]. The amount of Hemicellulose can be decreased by treating with strong alkali [32]. The strength of fiber is 70.8 MPa, Modulus 10.8 GPa and Elongation 34.8% [32]. The strength, modulus and elongation can be increased by alkali treatment [32]. The fiber can be dyed with both reactive and basic dye. It can be used instead of synthetic fiber where more strength is necessary, e.g. Blending with cotton fiber it can be used to produce fabric which reduce cost and increase strength.

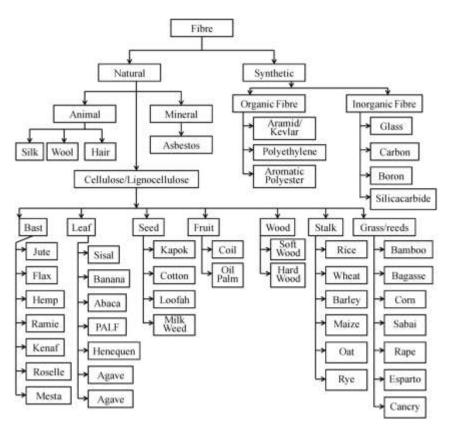


Figure 2. 13: Classification of fibers [32]

However it must be mentioned that the processing temperature of lignocellolusic fiber reinforced composites is restricted to 200^oC-220^oC, as these fibers undergo degradation at higher temperatures above this range.

2.7 Components of fiber

The fiber comprises of:

- ✤ Cellulose and the
- Hemicelluloses.

Cellulose content ranges from 40 to 50% of the dry wood weight, and hemicelluloses range from 25 to 35%.

2.7.1 Cellulose

Cellulose, the major chemical component of fiber wall and contributing 40-45% of the wood's dry weight, is composed of linear chains of D-glucose linked by β -1,4-glycosidicbonds (Figure 2.15) with the degree of polymerization from 10,000 in native wood to 1,000 in bleached kraft pulps. Each D-anhydroglucopyranose unit possesses hydroxyl groups at C2, C3, and C6 positions, capable of undergoing the typical reactions known for primary and secondary alcohols. The molecular structure imparts cellulose with its characteristic properties: hydrophylicity, chirality, degradability, and broad chemical variability initiated by the high donor reactivity of hydroxyl groups. The empirical formula for cellulose is (C₆H₁₀O₅) n where 'n' is degree of polymerization (DP).

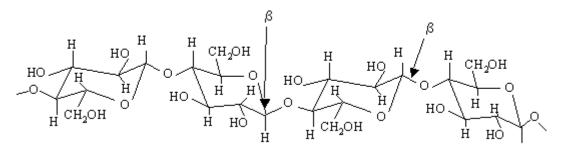


Figure 2. 14: Molecular structure of cellulose [33]

Cellulose has a strong tendency to form intra- and inter-molecular hydrogen bonds by the hydroxyl groups on these linear cellulose chains, which stiffen the straight chain and promote aggregation into a crystalline structure and give cellulose a multitude of partially crystalline fiber structures and morphologies.

2.7.2 Hemicellulose

Unlike cellulose, hemicelluloses have lower DP (only 50-300) with side groups on the chain molecule and are essentially amorphous. Hemicelluloses are mixtures of polysaccharides synthesized in wood almost entirely from glucose, mannose, galactose, xylose, arabinose, 4-*O* methylglucuronic acid, and galacturonicacid residues. Some hardwoods contain trace amounts of rhamnose. Generally, hemicelluloses are of much lower molecularweight than cellulose and some are branched. They are intimately associated with cellulose and appear to contribute as a structuralcomponent in the plant. Some hemicelluloses are present in abnormally large amounts when the plant is under stress; e.g., compression wood has a higher than normal galactose content as well as a higher lignin content. Hemicelluloses are soluble in alkali and easilyhydrolyzed by acids [33].

The arrangement of cellulose and hemicellulose in plant cell walls is shown in figure 2.15

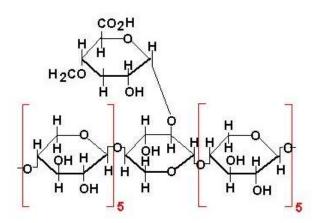


Figure 2. 15: Structure of hemicellulose [33]

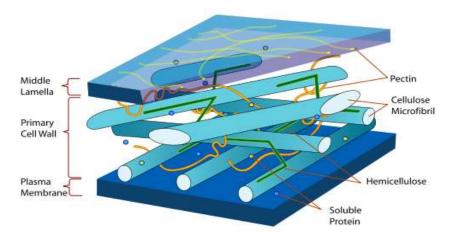


Figure 2. 16: Plant cell wall [33]

2.7.3 Lignin

Lignin is the third component at around 27% in coniferous wood opposed to 23% in deciduous trees. Lignin confers the hydrophobic properties reflecting the fact that it is based on aromatic rings. It is a complex brown constituent of the wood that cements the cellulose fibers together. It fills the spaces in the cell wall between cellulose, hemicellulose, and pectin components, especially in tracheid, sclereid and xylem cells (figure 2.20). It is covalently linked to hemicellulose and, therefore, crosslinks different plant polysaccharides, conferring mechanical strength to the cell wall and by extension the plant as a whole [33] Lignification occurs in the S1, S2 and S3 layers of the cell wall (Figure 2.18)

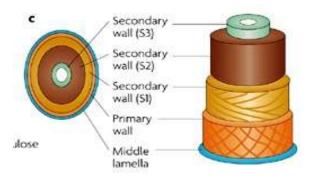


Figure 2. 17: Lignification occurs in the S1, S2 and S3 layers of the cell wall [33]

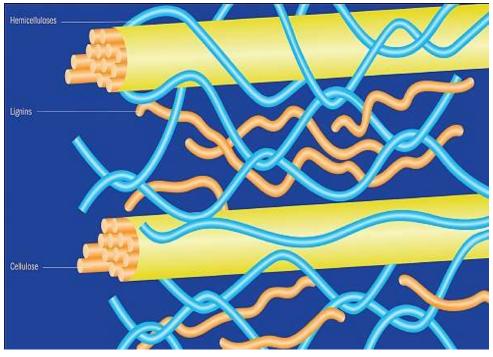


Figure 2. 18: A schematic of a plant cell wall showing cellulose fibrils laminated with hemicellulose and lignin polymers.

Several researchers have put their effort in manufacturing composites using variety of natural fibres (with and without chemical treatment), matrices (thermoplastic, thermosetting plastic etc.) and characterized them for their physical, chemical, mechanical, thermal and electrical properties. Out of that a part of authors have focused on making and characterization of the composites reinforced with short palymyra palm fibrous waste, stem, stem long, palm/jute, palm/banana, borassus fruit fibre in various matrices and the highlights of the same are described below in the order of borassus fruit, short, short hybrid, long, long hybrid palmyra palm fibre reinforced polymer composites.

So based on the above key points, it is concluded that till today i.e. January, 2014 no one has utilized the petiole fibre in manufacturing of the composites. An attempt is made by the Srinivasababu in the year 2009 itself to introduce a new fibre i.e. Palmyra palm petiole and its composites. The objectives of the present work are described under:

- Good extraction of palmyra palm petiole fibre.
- Chemical treatment of fibre.
- Removal of moisture from fibre. Fibre characterization and morphological study.
- Manufacturing and characterization of the composites at various loading conditions.

2.8 Mercerization process

Mercerization is an alkali treatment process. It is widely used in textile industry [34]. The standard definition of mercerization as proposed by ASTM D1965 is: the process of subjecting a vegetable fiber to an interaction with a fairly concentrated aqueous solution of strong base, to produce great swelling with resultant changes in the fine structure, dimension, morphology and mechanical properties. Therefore, mercerization is a chemical modification process that changed the chemical constituent behavior in natural fiber. The effect of alkali on cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. The schematic illustration of the swelling process in cellulose is shown in Fig. 1 [35]. Native cellulose (i.e. cellulose as it occurs in nature) shows a monoclinic crystalline lattice of cellulose-I, which can be changed into different polymorphous forms through chemical or thermal treatments. The important forms of alkali-cellulose and cellulose-II are shown in Fig. 2. Fig. 1 schematic illustration of the swelling process in cellulose [35] Fig. 2 Lattice structure of cellulose I and cellulose II The type of alkali and its concentration will influence the degree of swelling, and hence the degree of lattice transformation into cellulose-II. It has been reported that Na⁺has got a favorable diameter, able to widen the smallest pores in between the lattice planes and penetrate into them. Consequently, sodium hydroxide (NaOH) treatment results in a higher amount of swelling. This leads to the formation of new Nacellulose-I lattice, a lattice with relatively large distances between the cellulose molecules, and these spaces are filled with H2O molecules. In this structure, the OHgroups of the cellulose are converted into O - Na-groups, expanding the dimensions of molecules. Subsequent rinsing with water will remove the linked Na-ions and convert the cellulose to a new crystalline structure, i.e. cellulose-II, which is thermodynamically more stable than cellulose-I. NaOH can cause a complete lattice transformation from cellulose-I to cellulose-II. Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide. The following reaction takes place as a result of alkali treatment [36]:

$Fiber - OH + NaOH = Fiber - O - Na + H_2O$

As reported in much literature, natural fiber chemical constituent consists of cellulose and other non cellulose constituent like hemicellulose, lignin, pectin and impurities such as wax, ash and natural oil [39-40]. This non cellulose material could be removed by appropriate alkali treatments, which affect the tensile characteristic of the fiber [36-39].

Mercerization was found to change fiber surface topography, and the fiber diameter was reported to be decreased with increased concentration of sodium hydroxide concentration

[40]. Mercerization treatment also results in surface modifications leading to increase wettability of coir fiber polyester resin as reported by Prasad et.al [41]. It is reported in that alkaline treatment has two effects on the henequen fiber: (1) it increases

surface roughness, resulting in better mechanical interlocking; and (2) it increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites [42]. Consequently, alkaline treatment has a lasting effect on the mechanical behavior of natural fibers, especially on their strength and stiffness.

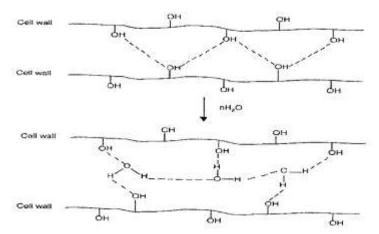


Figure 2. 19: Schematic illustration of the swelling process in cellulose [35]

		NaOH Treatment Parameters				
Natural Fiber	Matrix	rix Concentration % Temp. (⁰ C) Duration Effect/Comments	Effect/Comments	Ref.		
Pineapple, sisal	Polyester	5 & 10 %	30	1 h	At 10% NaOH, excess delignification occurred. Thus, fiber become weaker	[46]
Sisal	Polyester	0.25,0.5,1.0, 2.0, 5.0 & 10% w/w	Room temperature	1 h	NaOH treatment decrease fiber density. 10% treatment results a rougher surface than untreated	[47]
Hemp & kenaf	Polyester	6 %	19 ± 2	48 h	Cell wall densification identification from small positive change in fiber density observation	[48]
Curaua	Biodegradable resin	5, 10 & 15wt %	Room temperature	1 h & 2 h	Decrease of fiber diameter, fiber weight, fiber density and tensile strength with increasing NaOH content	[49]
Henequen	HDPE	2% w/v	25	1 h	Surface modification increase the area of contact and further expose the cellulose microfibril. Thus, improve fiber wetting and impregnation	[50]
Kenaf	12	3, 6 & 9%	Room temperature and 95 ⁰ C for 6% NaOH	3 h	3% NaOH was ineffective to remove impurities on fiber surface, 9% NaOH show cleanest fiber surface	[51]
Pineapple		2% w/v	95	2 h	Decrease in tensile strength and Young modulus were probably due to decrease in the degree of crystallinity and crystallite orientation	[52]
Kenaf, Flax & Hemp	Epoxy	5%	Room temperature	1 h	NaOH treated kenaf and hemp composite absorb more water then silane only or alkali and silane treated samples	[23]
Sisal & Oil palm	Natural rubber	0.5, 1, 2 & 4%	Ambient temperature	1 h	Alkali treated composite exhibited better tensile properties than silane treated composite	[24]
Coir	Polypropylene	2, 4, 6, 8 & 10%	Room temperature	4 week	Denser 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.	[53]

Table 2. 4: Properties of various natural fibers

2.9 Tensile Testing

A tensile test, also known as tension test, is probably the most fundamental type of mechanical test you can perform on material. Tensile tests are simple, relatively inexpensive, and fully standardized. By pulling on something, you will very quickly determine how the material will react to forces being applied in tension. As the material is being pulled, you will find its strength along with how much it will elongate [43].

You can learn a lot about a substance from tensile testing. As you continue to pull on the material until it breaks, you will obtain a good, complete tensile profile. A curve will result showing how it reacted to the forces being applied. The point of failure is of much interest and is typically called its "Ultimate Strength" or UTS on the chart [43].

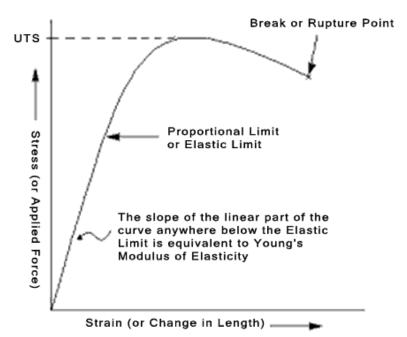
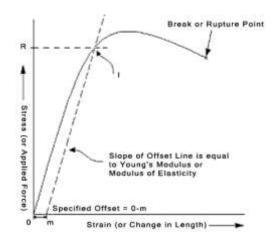


Figure 2. 20: Schematic of a stress-strain curve [43]

2.9.1 Hooke's Law



For most tensile testing of materials, you will notice that in the initial portion of the test, the relationship between the applied force or load and the elongation the specimen exhibits is linear. In this linear region, the line obeys the relationship defined as "Hooke's Law" where the ratio of stress to strain is a constant, or $\frac{\sigma}{\epsilon} = E$. E is the slope of the line in this region where stress (σ) is proportional to strain (ϵ) and is called the "Modulus of Elasticity" or "Young's Modulus".

Figure 2. 21: Schematic of stress-strain curve [43]

2.9.2 Modulus of Elasticity

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". From this point on in the tensile test, the material reacts plastically to any further increase in load or stress. It will not return to its original, unstressed condition if the load were removed [44].

2.9.3 Yield Strength

A value called "yield strength" of a material is defined as the stress applied to the material at which plastic deformation starts to occur while the material is loaded [44].

2.9.4 Offset Method

For some materials (e.g., metals and plastics), the departure from the linear elastic region cannot be easily identified. Therefore, an offset method to determine the yield strength of the material tested is allowed. These methods are discussed in ASTM E8 (metals) and D638 (plastics). An offset is specified as a % of strain (for metals, usually 0.2% from E8 and sometimes for plastics a value of 2% is used). The stress (R) that is determined from the intersection point "r" when the line of the linear elastic region (with slope equal to Modulus of Elasticity) is drawn from the offset "m" becomes the Yield Strength by the offset method [44].

2.9.5 Alternate Moduli

The tensile curves of some materials do not have a very well-defined linear region. In these cases, ASTM Standard E111 provides for alternative methods for determining the modulus of a material, as well as Young's Modulus. These alternate moduli are the secant modulus and tangent modulus [44].

2.9.6 Strain

You will also be able to find the amount of stretch or elongation the specimen undergoes during tensile testing. This can be expressed as an absolute measurement in the change in length or as a relative measurement called "strain". Strain itself can be expressed in two different ways, as "engineering strain" and "true strain". Engineering strain is probably the easiest and the most common expression of strain used. It is the

 $e = \frac{L - L_0}{L_0} = \frac{\Delta L}{L_0}$. Whereas, ratio of the change in length to the original length, the true strain is similar but based on the instantaneous length of the specimen as the

 $\epsilon = \ln(\frac{L_i}{L_0})$, where L_i is the instantaneous length and L_0 the initial test progresses, length [44]

2.9.7 Ultimate Tensile Strength

One of the properties you can determine about a material is its ultimate tensile strength (UTS). This is the maximum load the specimen sustains during the test. The UTS may or may not equate to the strength at break. This all depends on what type of material you are testing . . . Brittle, ductile or a substance that even exhibits both properties. And sometimes a material may be ductile when tested in a lab, but, when placed in service and exposed to extreme cold temperatures; it may transition to brittle behavior [44]

2.9.8 Flexible materials

- ASTM D638 Standard Test Method for Tensile Properties of Plastics •
- ASTM D828 Standard test method for tensile properties of paper and paperboard using constant-rate-of-elongation apparatus
- ASTM D882 Standard test method for tensile properties of thin plastic sheeting
- ISO 37 rubber, vulcanized or thermoplastic-determination of tensile stress-• strain properties

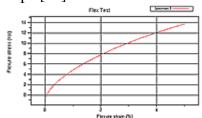
Tensile tests measure the force required to break a plastic sample specimen and the extent to which the specimen stretches or elongates to that breaking point. Tensile tests produce stress-strain diagrams used to determine tensile modulus. The resulting tensile test data can help specify optimal materials, design parts to withstand application forces, and provide key quality control checks for materials [44]. Tensile tests for plastics provide:

- Tensile Strength (at yield and at break) •
- **Tensile Modulus** •
- **Tensile Strain**
- Elongation and Percent Elongation at yield

2.10 Flexural testing

The flexure test method measures behavior of materials subjected to simple beam loading. It is also called a transverse beam test with some materials. Maximum fiber stress and maximum strain are calculated for increments of load. Results are plotted in a stress-strain diagram. Flexural strength is defined as the maximum stress in the outermost fiber. This is calculated at the surface of the specimen on the convex or

tension side. Flexural modulus is calculated from the slope of the stress vs. Deflection curve. If the curve has no linear region, a secant line is fitted to the curve to determine slope [45]



A flexure test produces tensile stress in the convex side of the specimen and compression stress in the concave side. This creates an area of shear stress along the midline. To ensure the primary failure comes from tensile or compression stress the shear stress must be minimized. This is done by controlling the span to depth ratio; the length of the outer span divided by the height (depth) of the specimen. For most materials S/d=16 is acceptable. Some materials require S/d=32 to 64 to keep the shear stress low enough [46].

2.10.1 Types of flexure tests

Flexure testing is often done on relatively flexible materials such as polymers, wood and composites. There are two test types; 3-point flex and 4-point flex. In a 3-point test the area of uniform stress is quite small and concentrated under the center loading point. In a 4-point test, the area of uniform stress exists between the inner span loading points (typically half the outer span length).



Flexural test with three-point loading Figure 2. 22: Schematic of a flexural test with three-point loading [46].

2.10.2 Typical materials

a) Polymers

The 3-point flexure test is the most common for polymers. Specimen deflection is usually measured by the crosshead position. Test results include flexural strength and_flexural modulus [47]

b) Flexural Strength

Maximum fiber stress developed in a specimen just before it cracks or breaks in a flexure test. Flexural yield strength is reported instead of flexural strength for materials that do not crack in the flexure test. An alternate term is modulus of rupture [47].

c) Flexural Modulus of Elasticity

Alternate term for modulus in bending [47].

d) Wood and Composites

The 4-point flexure test is common for wood and composites. The 4-point test requires a deflectometer to accurately measure specimen deflection at the center of the support span. Test results include flexural strength and flexural modulus [47].

e) Brittle Materials

When a 3-point flexure test is done on a brittle material like ceramic or concrete it is often called modulus of rupture (MOR). This test provides flex strength data only, not stiffness (modulus). The 4-point test can also be used on brittle materials. Alignment of the support and loading anvils is critical with brittle materials. The test fixture for these materials usually has self-aligning anvils [47].

2.11 Hardness measurement

Shore Hardness is nothing but a measure of the hardness of a given material or how resistant it will be to permanent indentation. It is measured by the depth of indentation that is created on the material with a specified force. The measuring instrument is actually a durometer, but Shore Hardness is named after its inventor Albert Shore [48].

Durometer is the international standard for measuring the hardness of rubber, plastic, and most nonmetallic materials. The hardness of a material is its resistance to surface penetration. Harder materials have more wear resistance, but they are also less flexible. Note that an object may fall within more than one scale—for example, a typical shoe heel is 95 Shore OO, 70 Shore A, and 22 Shore D [49].

Accordingly, there are different Shore Hardness scales for measuring the solidity of different materials with varying properties, like rubbers, polymers and elastomers. In fact, there are as many as 12 different scales depending on the intended use, and each scale results in a value between 0 and 100. You can safely assume that higher values indicate higher hardness and vice versa [50].

However, you will find that only two of these scales are most commonly used for measuring the hardness of rubber compounds - A scale for softer ones and D scale for harder ones [51].

DUROMETER HARDNESS SCALES

SHORE A	SHORE D	MATERIAL COMPARISON EXAMPLES	
0		Very soft, flesh like (baby's bottom)	_
10		Very soft, rubbery	_
20		Soft (rubber bands)	_
30		Medium soft (art gum eraser, bathtub caulk)	_
40		Firm (red rubber pencil eraser)	_
50	5	Bungee strap, auto radiator hose	_
60	15	Vinyl feet for chairs, sneaker sole	_
70	25	Vinyl garden hose, rubber ducky	_
80	35	Extension cord plug, Tupperware	_
90	45	Vinyl watch strap	_
100	55	Rubber printing press rollers	_
and the second second	65	Skate wheels, polyethylene	_
	75	Computer housings, ABS	_
	85	Very hard	_
	95	Kitchen counter laminate	_

Shore hardness chart



Figure 2. 23: Shore hardness chart [51]

To elaborate on these universal scales:

- Shore A Hardness Scale is used for measuring the hardness of flexible mold rubbers. These can range in hardness from very soft and flexible, to medium and somewhat flexible, to hard with almost no flexibility at all. It follows that Shore A0 obviously denotes extremely soft and gel-like rubbers. While semi-rigid plastics will be measured at the highest end of the Shore A Scale [51].
- Shore D Hardness Scale is reserved for measuring the hardness of hard rubbers, semi-rigid plastics and hard plastics.

While not needed for plaster casting, shore hardness proves helpful when you have to choose your materials such as which silicone rubber should be used for making a mold or cast.

If your mold or cast requires flexibility, or your model has severe undercuts, opt for a lower Shore A Hardness number that will stretch easily and can be sealed properly as well. You will be able to easily extract the original model from the rubber mold or the subsequent casts from the silicone rubber mold, after they have cured properly.

On the other hand, a rubber with a higher Shore Hardness number will be quite stiff and not flexible at all. Delicate molds and casts will tend to break during the demolding process. But such rubbers are suitable for concrete castings as they offer more resistance to abrasions and extrusions. This feature will be sorely lacking in the softer rubber compounds.

Therefore, while shore hardness is not needed for plaster casting, it plays a crucial level in making rubber molds and casts, especially with silicone rubber.

Now that you know all about how the durometer of rubbers are calculated, you may still be uncertain as to what durometer to select for your mold making needs. Never fear as environ molds has made the selection process easy. Standard molds are most often made with a Shore A 25-30 durometer, so environmolds has formulated its moldrite 25 as a Shore A 25 rubber – a firmness which is perfect for everyday mold making. For prosthetics. Models with extreme undercut and doll making a Shore A 10 is the preferred rubber of choice. Therefore, you would select artmlods' skinrite 10 translucentsilicone for those applications. For a firmer rubber and a addition cure silicone which is also food safe for baking you should select bakesil which is a Shore A 37. We also offer a number of selections to choose from for casting materials. For strong, rigid and all purpose casting material, a Shore D 70-75 is generally preferred. Artmolds, kastez Resin is formulated for cured durometer of Shore D 75 [51].

2.12Thermo Gravimetric Analysis (TGA)

and **Definition**: Thermo gravimetric Analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere.

An Alternate Definition: TGA is a technique in which, upon heating a material, its weight increases or decreases [52].

A Simple TGA Concept to remember: TGA measures a sample's weight as it is heated or cooled in a furnace.

A TGA consists of a sample pan that is supported by a precision balance. That pan resides in a furnace is heated or cooled during the experiment. The mass of the sample is monitored during the experiment. A sample purge gas controls the sample environment. This gas may be inert or a reactive gas that flows over the sample and exits through an exhaust.

Perkinelmer offers two types of tags, a top-loading TGA 4000 and a bottomloading or hangdown, Pyris 1 TGA. The TGA 4000 supports the sample pan above the balance via a "stem" support rod. The Pyris 1 TGA supports the sample pan via a "hangdown" below the balance. Both styles take advantage of gravity to obtain very accurate and reproducible measurements.

2.12.1The TGA Technique

- TGA measures the amount of weight change of a material, either as a function of increasing temperature, or isothermally as a function of time, in an atmosphere of nitrogen, helium, air, other gas, or in vacuum.
- Thermal gravimetric analysis can be interfaced with a mass spectrometer RGA to identify and measure the vapors generated, though there is greater sensitivity in two separate measurements.

- Inorganic materials, metals, polymers and plastics, ceramics, glasses, and composite materials can be analyzed.
- Temperature range from 25°C to 900°C routinely. The maximum temperature is 1000°C.
- Sample weight can range from 1 mg to 150 mg. Sample weights of more than 25 mg are preferred, but excellent results are sometimes obtainable on 1 mg of material.
- Weight change sensitivity of 0.01 mg.
- Samples can be analyzed in the form of powder or small pieces so the interior sample temperature remains close to the measured gas temperature [53].

2.12.2 ASTM Test Methods Using Thermogravimetric Analysis [53]

- ASTM D2584 Standard Test Method for Ignition Loss of Cured Reinforced Resins
- ASTM E1131 Standard Test Method for Compositional Analysis by Thermogravimetry
- ASTM E1641 Standard Test Method for Decomposition Kinetics by Thermogravimetry Using the Ozawa/Flynn/Wall Method
- ASTM E2008 Standard Test Method for Volatility Rate by Thermogravimetry

Chapter 3

Experimental procedure

3.1 Fiber collection

Fiber is collected from the stalk of the palm tree. The stalk of the palm leaf is collected, then these are kept in water for 5 days for the easy separation of the fiber. The palm stalks are taken out from the water and dried in the sun. By this time the stalk gets a bit soft. So, using hand and knife the fibers are extracted with utmost caution. Young palm trees are preferred because the fibers of the young palm stalk have better overall properties.

These stalks of palm leaf were crushed by sugarcane crushing machine with an aim for easy separation of the fiber since separation of natural fiber without machine is somewhat cumbersome.

The lignin and cellulose components of palmyra palm fiber are separated from the fiber by using hand as neat and clean as possible. Still hundred percent separations is not possible. This was subsequently done by alkali treatment i.e. NaOH treated.

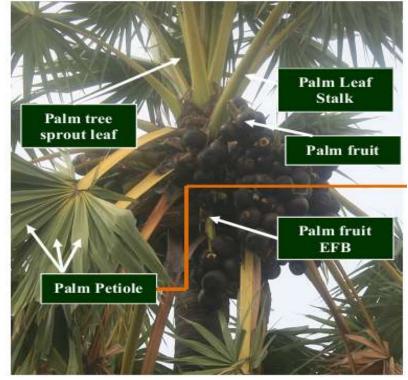


Figure 3. 1: Palmyra Palm tree



Figure 3. 2: Palmyra palm fiber

The fiber diameter was measured by using stereoscope.



Figure 3. 3: Stereoscope(Fiber diameter measurement)

3.2 Polyester resin casting

Steps

- 1. Casting resin and catalyst
- 2. Disposable graduated paper mixing cups
- 3. Wooden stir sticks
- 4. A mold

3.2.1 Casting resin

A polyester resin noted for its clarity and ability to be cast in mass. In its liquid form, casting resin has a consistency of corn syrup.

The styrene odor of casting resin is less objectionable if you have good ventilation when pouring. Once cured, your cast pieces will have little if any odor. Casting resin should be stored at room temperature (72° F.), out of direct sunlight, and out of reach of children. If stored properly, shelf life is approximately nine to twelve months.

For the preparation of the composite dried Palmyra Palm fibres are used as reinforcement and polyester resin is used as the matrix material.

It is mixed along with the corresponding hardener in a 10:1 ratio by weight.

We intend to make the fibers aligned.

Two aluminium frames are taken. In this Al frame holes are made by drilling. 30 holes are prepared in each frame. Then the frames are fixed tight in the mold using superglue. Then individual fibers are entered in one hole and taken away from the other hole of another frame. 3-4 fibers are taken at a time this way. After that the fibers are made aligned handheld as much as possible and then are glued to maintain that alignment. After the fiber alignment, the polyester resin is mixed with hardener in a beaker and stirred for 2 minutes to ensure adequate mixing.. It is then treated with vacuum for 1.5-2 minutes to remove gas bubbles. This treated polyester resin is gently poured over the fiber arrangement. A few minutes have to be weighted for the curing to take place. This is the onset of the composite formation. The composites are cured under a load of 2kg for about 24 hours so that then they don't spread randomly and create gaps and regions of uneven thickness before it is removed from the mould. It is then post cured in the air for another 24 hours after removing from the mould. Specimens of suitable dimension have been cut for mechanical testing and utmost care has been taken to maintain uniformity in the samples.

The same procedure is to be carried out using 20 kg load on the composite.

3.2.2Casting of composite

(1) The die used for pure polyester casting was also used for the casting of composite including the polyester matrix reinforced with fibers untreated, 2% naoh treated and 4% naoh treated.

- (2) Millot paper was spread on the die surface and was attached with the superglue.
- (3) This time also a a pair of parallel aluminium frame has been used but unlike the pure casting the frames are drilled into hole by drilling machine.
- (4) Diameter of the holes is maintained maximum 1mm with distance between the holes being 4mm. All composite castings were prepared by the same frame pairs.
- (5) The distance between the frames is maintained 130mm.
- (6) Now the fibers were introduced through one hole and taken out from another hole of the opposite frame. The alignment of the fibers is maintained throughout the length of the frame.
- (7) 3-4 fibers are aligned at a time and the fiber strings were kept in tension using the hand and then the fibers are glued with the edges of the dies. All fibers are aligned in sequence.
- (8) When all fibers are set tension then other two sides which were opened were sealed by other two frames. Some tissue papers and lightweight slabs are used to support the frame on the die.
- (9) Polyester resin was weighted and taken in a cup.
- (10) 2 weight percent of methyl ethyl ketone peroxide (MEKP) which is used as hardener was mixed with the resin.
- (11) The mixture was then stirred for 1-1.5 minutes then vacuum treated for 1.5 minutes for the removal of gases and bubbles.
- (12) Immediately after vacuum treatment the resin mixture was poured onto the aligned fiber arrangement. Pouring should be done carefully so that all corners of the frames are filled with the resin.
- (13) The arrangement is left for 5 minutes for curing.
- (14) After curing the millot paper is spread over the surface of the casting with due care as to avoid accumulation of bubbles between the casting and millot paper.
- (15) To avoid warping lightweight slabs were put on the casting surface and it is kept in this way 8-10 hours.
- (16) After that desired the composite is obtained.



Figure 3. 4: Arrangement of aluminium frame and die



Figure 3. 5 : Aligned fiber



Figure 3. 6: Completion of fiber Arrangement



Figure 3. 7: Fiber alignment operation



Figure 3. 8: Various stages of fiber alignment

3.2.3 Alkali treatment

The Palmyra Palm (Borassus spp) fibres are then prepared for treatment with sodium hydroxide (NaOH) solution to improve its surface properties and provide better adhesion with the matrix after the removal of lignin and pectin from the surface of the fibers.

NaOH solution is prepared for two separate concentrations of 2% and 4%. Palmyra Palm samples are first dipped in 2% NaOH solution and 4% NaOH solution.

The time for which each stack is dipped in NaOH solutions is 5 minutes.

The stacks are then taken out and washed with distilled water for 2/3times.

Then they are dried in a heat oven at 70°C for 1 hour each.

Drying is done to remove moisture content and to prevent the sticking together and clogging of the fibres with each other.

For the preparation of the composite dried Palmyra Palm fibres are used as reinforcement and polyester resin is used as the matrix material.

It is mixed along with the corresponding hardener in a 10:1 ratio by weight.

We intend to make the fibers aligned.

Two aluminium frames are taken. In this Al frame holes are made by drilling. 30 holes are prepared in each frame. Then the frames are fixed tight in the mold using superglue. Then individual fibers are entered in one hole and taken away from the other hole of another frame. 3-4 fibers are taken at a time this way. After that the fibers are made aligned handheld as much as possible and then are glued to maintain that alignment. After the fiber alignment, the polyester resin is mixed with hardener in a beaker and stirred for 2 minutes to ensure adequate mixing.. It is then treated with vacuum for 1.5-2 minutes to remove gas bubbles. This treated polyester resin is gently poured over the fiber arrangement. A few minutes have to be weighted for the curing to take place. This is the onset of the composite formation. The composites are cured under a load of 2kg for about 24 hours so that then they don't spread randomly and create gaps and regions of uneven

thickness before it is removed from the mould. It is then post cured in the air for another 24 hours after removing from the mould. Specimens of suitable dimension have been cut for mechanical testing and utmost care has been taken to maintain uniformity in the samples.

The same procedure is to be carried out using 20 kg load on the composite.

The different surface chemical modifications, such as chemical treatments, coupling agents and graft co-polymerization, of natural fibers aimed at improving the tensile properties of the composites were performed by a number of researchers. Alkali treatment, also called mercerization, is one of the most popular chemical treatments of natural fibres. Sodium hydroxide (NaOH) is used in this method to remove the hydrogen bonding in the network structure of the fibres cellulose, thereby increasing fibres surface roughness [54]. This treatment also removes certain amount of lignin, wax and oils covering the external surface of the fibres cell wall, depolymerises the native cellulose structure and exposes the short length crystallites [55]. Acrylic acid treatment was also reported to be effective in modifying the natural fibres surface. A study on flax fibres-reinforced polyethylene biocomposites by Li et al. found that the efficiency of such a treatment was higher than alkali and silane treatment [55].

The chemical coupling method is also one of the important chemical methods, which improve the interfacial adhesion. In this method the fiber surface is treated with a compound that forms a bridge of chemical bonds between fiber and matrix. The chemical composition of coupling agents allows them to react with the fiber surface forming a bridge of chemical bonds between the fiber and matrix. Most researchers found these treatments were effective and showed better interfacial bonding [55].

3.2.4 Specimen Preparation for tensile test

- (1) Measure the thickness, width and gage length of polymer samples in mm. These dimensions should be approximately the same for each sample.
- (2) Also make note of any sample defects (e.g. Impurities, air bubbles, etc.).
- (3) Samples of pure polyester resin and also the composites reinforced with three aligned fibers per sample are taken from the casting by hacksaw and the final finishing of the sample was done by grinding.

3.2.4.1 Test procedure

- (1) Enter geometry of the sample before starting.
- (2) Click on the Start button. Both the upper and bottom grips will start moving in opposite directions according to the specified pulling rate. Observe the experiment at a safe distance (about 1.5 meters away) at an angle and take note of the failure mode when the specimen fails.(NOTE: Be sure to wear safety glasses. Do not come close to equipment when the tensile test is running).
- (3) A plot of tensile stress (mpa) versus tensile strain (mm/mm) will be generated in real-time during the experiment

3.2.4.2 End of Test

- (1) The machine will stop automatically when the sample is broken.
- (2) Press the "Return" button on the digital controller. Both the upper and lower grips will be returned to their original positions automatically.
- (3) Turn the two handles in the open directions to remove the sample



Figure 3. 9: Tensile test of fiber



Figure 3. 10: Specimen for tensile test

3.3 Flexural Test Procedure:

Most commonly the specimen lies on a support span and the load is applied to the center by the loading nose producing three point bending at a specified rate. The parameters for this test are the support span, the speed of the loading, and the maximum deflection for the test. These parameters are based on the test specimen thickness and are defined differently by ASTM and ISO. For ASTM D790, the test is stopped when the specimen reaches 5% deflection or the specimen breaks before 5%. For ISO 178, the test is stopped when the specimen breaks. If the specimen does not break, the test is continued as far a possible and the stress at 3.5% (conventional deflection) is reported.

3.3.1 Specimen size:

A variety of specimen shapes can be used for this test, but the most commonly used specimen size for ASTM is 3.2mm x 12.7mm x 125mm (0.125" x 0.5" x 5.0") and for ISO is 10mm x 4mm x 80mm.

Standard specimen thickness is 4 mm (0.16 in), standard specimen width is 13 mm (0.5 in) and standard specimen length is 20% longer than the support span. If the standard specimen is not available, alternative specimen sizes may be used.

3.3.2 Data:

Flexural stress at yield, flexural strain at yield, flexural stress at break, flexural strain at break, flexural stress at 3.5% (ISO) or 5.0% (ASTM) deflection, flexural modulus. Stress/Strain curves and raw data can be provided.

3.3.3 Equipment Used:

Universal Testing Machine Four Point Flexural Fixture Three Point Flexural Fixture

3.3.4 Testing method

The test method for conducting the test usually involves a specified test fixture on a universal testing machine. Details of the test preparation, conditioning, and conduct affect the test results. The sample is placed on two supporting pins a set distance apart and a third loading pin is lowered from above at a constant rate until sample failure.

Calculation of the flexural stress σ_f

 $\sigma_f = \frac{3FL}{2bd^2}$ for a rectangular cross section $\sigma_f = \frac{FL}{\pi R^3}$ for a circular cross section

Calculation of the flexural strain ϵ_f

$$\epsilon_f = \frac{6Dd}{L^2}$$

Calculation of flexural modulus E_f

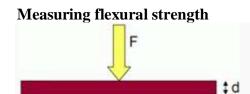
$$E_f = \frac{L^3 m}{4bd^3}$$

In these formulas the following parameters are used:

- σ_f = Stress in outer fibers at midpoint, (<u>mpa</u>)
- $\epsilon f = \text{Strain in the outer surface, (mm/mm)}$
- $E_{f} =$ flexural Modulus of elasticity,(mpa)
- F = load at a given point on the load deflection curve, (N)
- L = Support span, (mm)
- b = Width of test beam, (mm)
- d = Depth of tested beam, (mm)
- D = maximum deflection of the center of the beam, (mm)
- *m* = The gradient (i.e., slope) of the initial straight-line portion of the load deflection

Curve,(P/D), (N/mm)

• R = The radius of the beam, (mm)



L

Figure 3. 11: Beam under 3 point bending

For a rectangular sample under a load in a three-point bending setup

$$\sigma = \frac{3FL}{2bd^2}$$

- *F* is the load (force) at the fracture point (N)
- *L* is the length of the support span
- *B* is width*D* is thickness

For a rectangular sample under a load in a four-point bending setup where the loading span is one-third of the support span:

$\mathbf{\sigma} = FL/bd^2$

- *F* is the load (force) at the fracture point
- *L* is the length of the support (outer) span
- *B* is width
- *D* is thickness

For the 4 pt bend setup, if the loading span is 1/2 of the support span (i.e. $L_i = 1/2 L$ in Fig. 4):

$$\sigma = \frac{3FL}{4bd^2}$$

If the loading span is neither 1/3 nor 1/2 the support span for the 4 pt bend setup (Fig. 4):

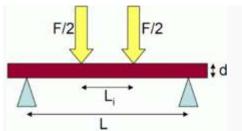


Figure 3. 12: Beam under 4 point bending

$$\sigma = \frac{3F(L-L_i)}{2bd^2}$$

• L_i is the length of the loading (inner) span

In our testing, the following dimension for the flexural specimen was used:

Gauge length: 96mm

Width: 12.5mm

Thickness: 6mm



Figure 3. 13: Specimen for flexure test

3.4 Measurement of hardness

The following steps were followed:

- 1. Three parallel lines were drawn on the casting surface having arbitrarily equidistant.
- 2. Each line was divided into 10 equal parts.
- 3. Shore hardness tester was put in the intersection of lines hold it for 11 seconds.
- 4. Average hardness of all three lines was taken.
- 5. The same procedure was repeated for each cast product.

3.5 TGA analysis

Steps for TGA analysis are

- 1. The sample is cut from the cast product.
- 2. Grinding of the sample should be done very carefully so that the weight of the sample exceeds no more than 30 mg.
- 3. Then this sample is ready for TGA analysis.

Chapter 4

Results and discussion

4.1Tensile properties

Generally, the tensile properties of composites are markedly improved by adding fibers to a polymer matrix since fibers have much higher strength and stiffness values than those of the matrices [3, 8]. The tensile test was carried out for fibers, pure polyester resin, composite of fiber reinforced polyester and composites composed of fibers treated with 2% NaOH and 4% NaOH respectively. In each case, a fiber length of 110mm was maintained. Each sample was tested and Young's modulus, tensile strength and strain to failure were measured.

The stress-strain curve for the raw fibers having 110mm length is shown in figure 4.1 to 4.3 respectively.

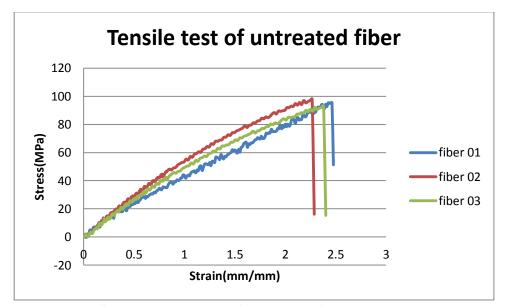


Figure 4. 1: Stress-strain curve of untreated fiber

From figure 4.1 it is evident that, we have tensile strength close to 100 MPa for raw fiber with strain upto about 2.5mm. The slope of curve for first fiber was steeper than the other two, so is the strain.

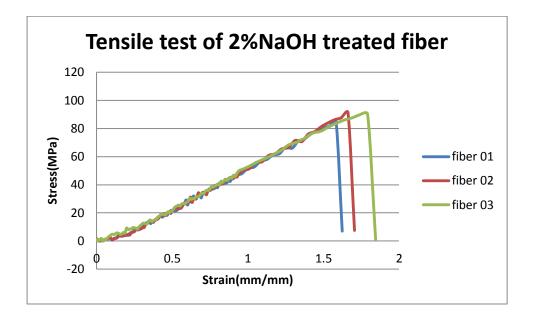


Figure 4. 2: Stress-strain curve of 2% NaOH treated fiber

From figure 4.2 it is evident that, we have tensile strength reduces somewhat to 90 MPa for fiber treated with 2% NaOH, with strain goes down below 2mm. So, fiber treatment with alkali solution starts reducing the tensile strength and also strain.

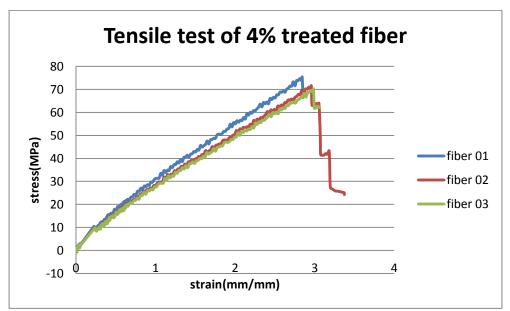


Figure 4. 3: Stress-strain curve for 4% NaOH treated fiber

Figure 4.3 is the stress- strain curve for fibers treated with 4% NaOH. This time the tensile strength further reduces to below 80MPa. But there is an increase in strain. So, it is seen that a raw fiber has the maximum tensile strength compared to the treated one. Further treatment would decrease the tensile properties.

4.1.1 Tensile test of composites

The tensile test of the composite was carried out, which is prepared by reinforcing the polyester resin with the fibers both treated and untreated. The stress-strain graphs are showed from figure 4.4 to figure 4.11. But first of all the tensile test of the pure polyester matrix is to be carried out.

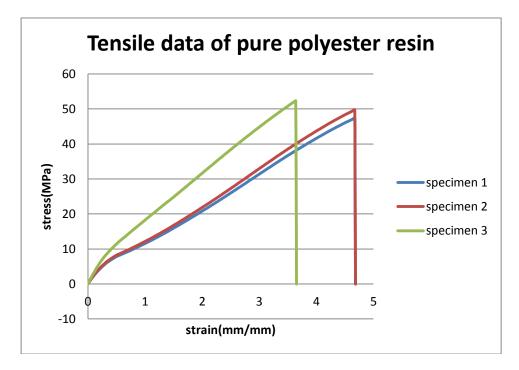


Figure 4. 4: Tensile stress-strain curve for pure reinforced polyester

In figure 4.4, it is obvious that the polyester resin will have a low tensile strength than the raw fiber with increased strain. The tensile strength just crosses 50 MPa with elongation close to 5mm. So, the composites should have the tensile strength in between the raw coir fiber and the pure polyester resin.

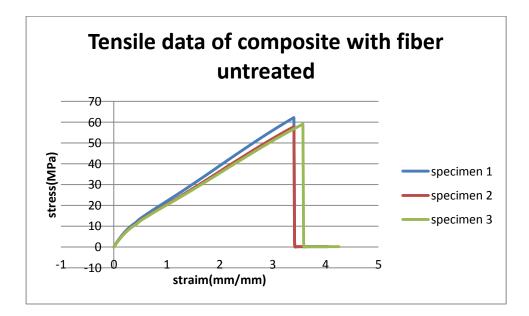


Figure 4. 5: Tensile stress-strain curve for untreated fiber reinforced polyester

Form the figure 4.5 it is seen that, the tensile strength of the composite shows higher values than the pure casting just exceeding 60 MPa. But there is a decrease in the strain. Therefore a characteristic composite has been prepared. The increase in tensile strength of the composite is attributed to the creation of the fiber-matrix bonding. When the tensile load is applied the relatively weak matrix transfers its load to the fiber unlike the pure matrix where the applied load is solely borne by the matrix alone. The composite will not tear apart until the fiber reaches the threshold to carry the applied load. Chemical reaction takes place between the fiber and the matrix that help create the bonding.

The fiber was treated with alkali like in our experiment which was done with NaOH by varying the weight percent of 2 and 4 respectively. Alkali treatment removes the lignin and pectin from the surface of coir fiber therefore porosities are explored. Matrix flows in the pore of the fibers and therefore a good interlocking is created. Therefore, there should be an increase in the tensile properties. The tensile properties of the composite with coir fiber of 2% and 4% NaOH treated are shown in figure 4.6 and figure 4.7 respectively.

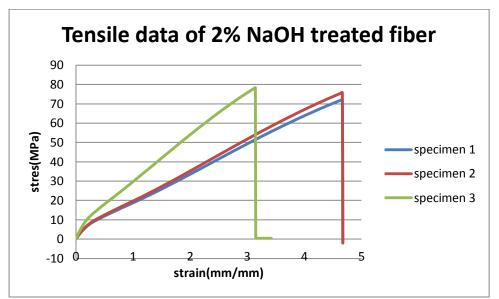


Figure 4. 6: Tensile stress-strain curve for the 2% NaOH treated fiber reinforced polyester

It is evident from the figure 4.6 that there is a slight increase in the tensile properties with tensile strength ranging between 75-80 mpa compared to about 60 mpa of the composite with untreated fiber, which indicates that fibers with alkali treatment and used it for the composite production will increase the tensile properties of the composite. Elongation is also found to increase a little bit except the tensile specimen 3 shows reduced elongation.

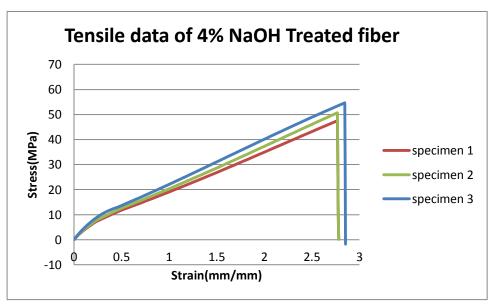


Figure 4. 7: Tensile stress-strain curve for the 4% NaOH treated fiber reinforced polyester

Moving to figure 4.7, it is seen that there is a reduction in the tensile properties, even showing the degradation compared to the composite with the untreated coir fiber. This is because the fibers treated with 4% NaOH have the cell wall increase in thickness so

the adhesion between the fiber and the matrix decreases. Thus there is a decrease in the tensile properties.

4.1.2 Tensile test with 20 kg load

Now the same sequence of testing is conducted with 20 kg load on the casting product. Using a load of 20 kg on the pure casting yields no appreciable change in the tensile properties of the matrix, perhaps a very slight increase in the tensile strength. But there is a decrease in the elongation. Figure 4.8 shows the fact. The very slight increase in the tensile strength is due to the increased pressure which accelerates the curing action during setting. Simultaneous application of pressure and curing leads to this increment.

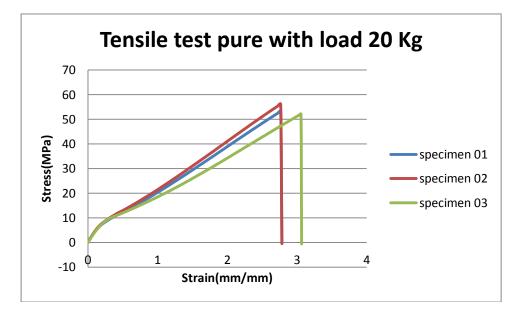


Figure 4. 8: Tensile stress-strain curve for pure polyester with 20 kg load

Figure 4.9 depicts the stress-strain curve for the fiber reinforced composite with fiber being untreated. It is evident that using the load increases the tensile property compared to the unloaded casting. The tensile strength increases to above 70 MPa compared to 60 MPa of the unloaded counterpart. But the elongation has decreased. Incorporation of fiber in the matrix leads to reinforcement and further addition of pressure helps improve the property. So the combination of treated fiber and added pressure on the composite leads to the superior tensile property.

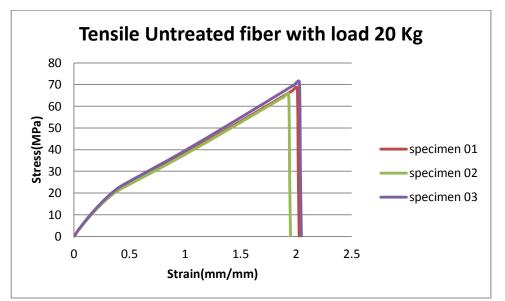


Figure 4. 9: Tensile stress-strain curve for untreated fiber reinforced polyester with 20 kg load

Same is observed in the case of the composite reinforced with 2% NaOH treated fiber. This composite with added load will show superior property compared to the unloaded one.

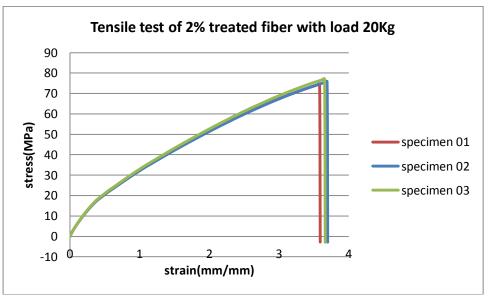


Figure 4. 10: Tensile stress-strain curve for the 2% NaOH treated fiber reinforced polyester with 20 kg load

The composite reinforced with 4% NaOH treated fiber shows no appreciable change in the tensile properties especially a decrease in strain.

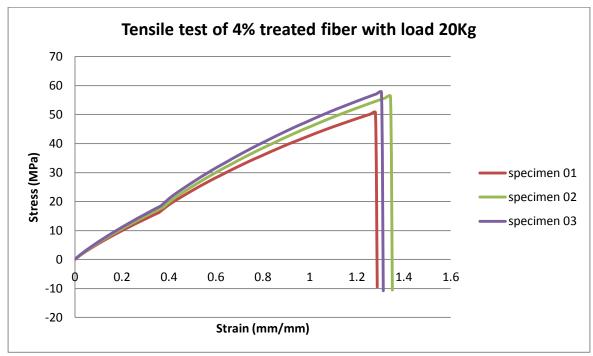


Figure 4. 11: Tensile stress-strain curve for the 4% NaOH treated fiber reinforced polyester with 20 kg load

So it is evident that, the tensile property of the composite having 2% NaOH treated fiber, prepared by the application of a load of 20 kg is superior compared to the other combination.

Alkali treatment, also called mercerization, is one of the most popular chemical treatments of natural fibres. Sodium hydroxide (NaOH) is used in this method to remove the hydrogen bonding in the network structure of the fibres cellulose, thereby increasing fibres surface roughness [54]. This treatment also removes certain amount of lignin, wax and oils covering the external surface of the fibres cell wall, depolymerises the native cellulose structure and exposes the short length crystallites [55]. Acrylic acid treatment was also reported to be effective in modifying the natural fibres surface. A study on flax fibres-reinforced polyethylene biocomposites by Li et al. found that the efficiency of such a treatment was higher than alkali and silane treatment [55].

4.2 Flexural test of composites

Flexural test was conducted. A flexural stress-strain curve were obtained and analyzed. Firstly the stress-strain curve for the pure polyester resin is observed. It shows a maximum flexural strength of about 65 MPa with a large strain of 13 mm/mm. Specimen 01 and specimen 02 touched the 50MPa and 60 MPa respectively with a strain below 10 mm/mm. Specimen 3 did not break and shows higher strength value. This is shown in figure 4.12.

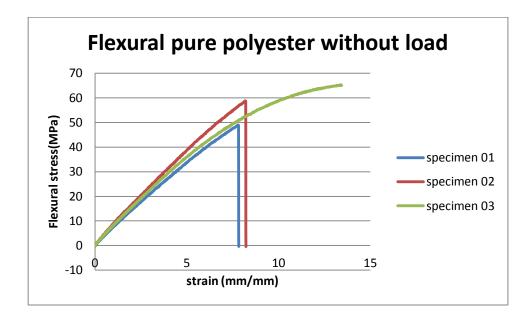


Figure 4. 12: Flexural stress-strain curve for pure polyester

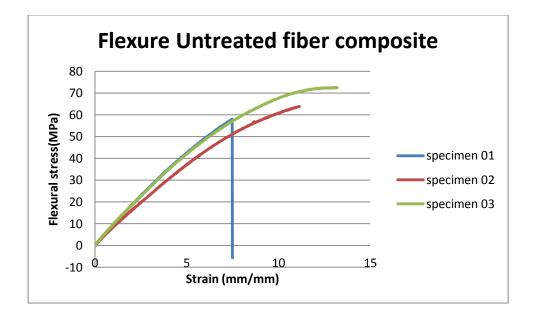


Figure 4. 13: Flexural stress-strain curve for untreated fiber reinforced polyester

Figure 4.13 shows the flexural property of the composite. The composite shows superior flexural property compared to the pure polyester. There is improvement on the bending properties.

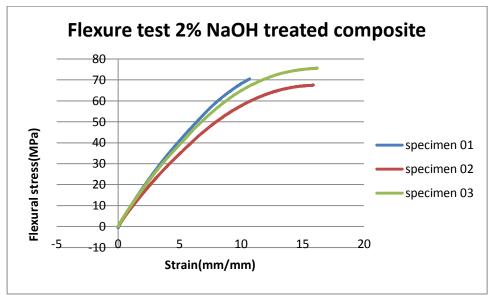


Figure 4. 14: Flexural stress-strain curve for the 2% NaOH treated fiber reinforced polyester

The flexural property of the composite having 2% NaOH treated fiber shows superior property. Both the bending properties and the flexure strength is found to be improved gradually compared to the untreated one.

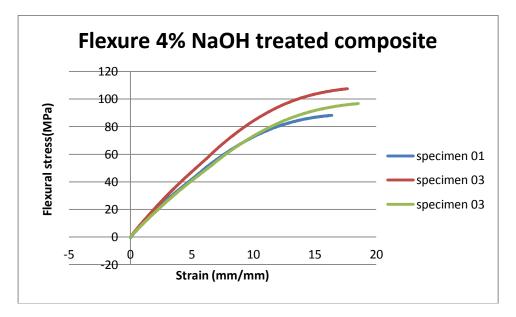


Figure 4. 15: Flexural stress-strain curve for the 4% NaOH treated fiber reinforced polyester

The flexural property of the composite having 4% NaOH treated fiber shows a very high value of the flexural strength. Here both the bending properties and the flexure strength is found to be improved significantly compared to the 2% NaOH treated counterpart.

The treated surface of fiber becomes rather smoother as compared to that of untreated fiber. Following the alkalizations, the pores became visibly clear and it this may be due to leaching out of waxy substances and impurities from the surface of fiber. The

alkaline treatment used on the fibers produce fibrillation and collapse of the cellular structure due to the remotion of the cementing material, which leads to a better packing of cellulose chains, which increases the effective surface area available for contact with the wet matrix

4.2.1 Flexure test with 20 kg load

Same sequence of operation was followed also in the case of flexure test using a load of 20 kg for finding variation in the property.

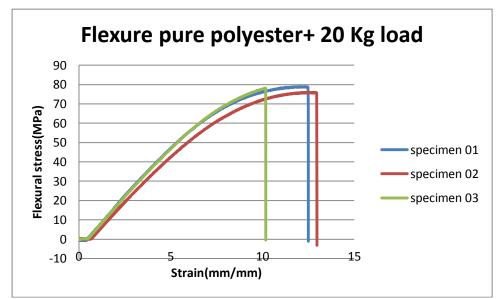


Figure 4. 16: Flexural stress-strain curve for pure polyester with 20 kg load

It is evident from figure 4.16 that the flexural strength has increased significantly compared to the same casting without load. There is also a considerable bending but all three specimens failed ultimately.

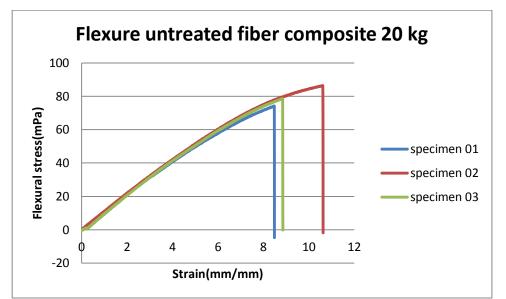


Figure 4. 17: Flexural stress-strain curve for untreated fiber reinforced polyester with 20 kg load

There is no appreciable change in the flexural property of the composite of untreated fiber with 20 kg load. There is very slight increase in the strength. Strain property has also improved (figure 4.17).

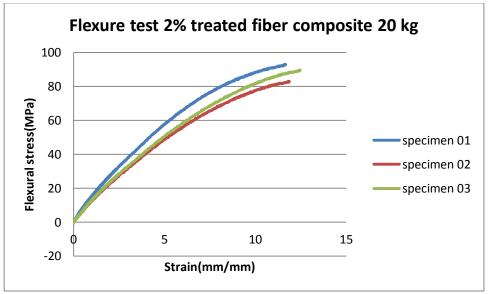


Figure 4. 18: Flexural stress-strain curve for the 2% NaOH treated fiber reinforced polyester with 20 kg load

From figure 4.18 it is evident that, flexural strength increases compared to the composite reinforced with 2% NaOH treated fiber without load. This composite also shows better properties than the composite reinforced with untreated fiber with 20 kg load. These fibers possess very good tensile strain values without failure.

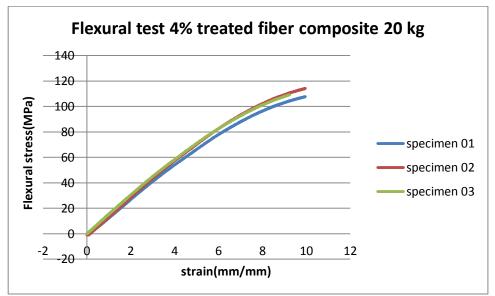


Figure 4. 19: Flexural stress-strain curve for the 4% NaOH treated fiber reinforced polyester with 20 kg load

From figure 4.19 it is seen that, flexural strength also increases compared to the composite reinforced with 4% NaOH treated fiber without load. This composite also shows superior properties than the composite reinforced with untreated fiber with 20 kg load. These fibers possess somewhat less tensile strain values without failure compared to the previous one.

4.3 Comparison of tensile modulus data

It is often observed that the presence of fiber or other reinforcement in the polymeric matrix raises the composite strength and modulus [56]. Here the tensile modulus is presented in a tabular form. From figure 4.20 we can see that, composite reinforced with 2% NaOH treated fiber shows superior properties. Alkali treatment improves the tensile property but upto 2 weight percent. When composite composed of 4% NaOH is concerned a decrease in tensile properties is observed.

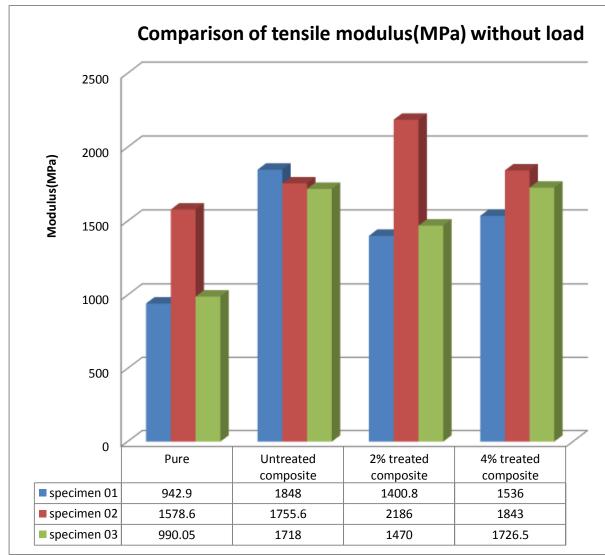


Figure 4. 20: Comparison of tensile modulus without load

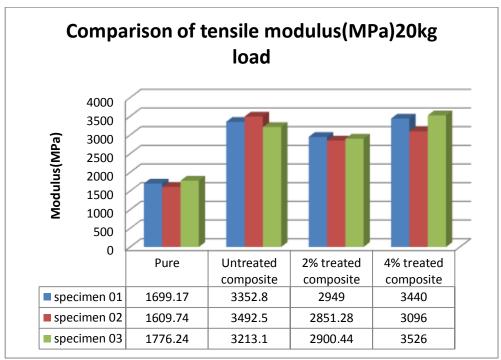


Figure 4. 21: Comparison of tensile modulus with 20 kg load

It is evident from the above figure that, tensile modulus of the castings with a load of 20 kg shows superior tensile modulus properties compared to the unloaded counterpart. But in this case the tensile modulus of the composite reinforced with untreated fiber show superior property. Alkali treatment of 2% to the fiber leads to the improved modus values.

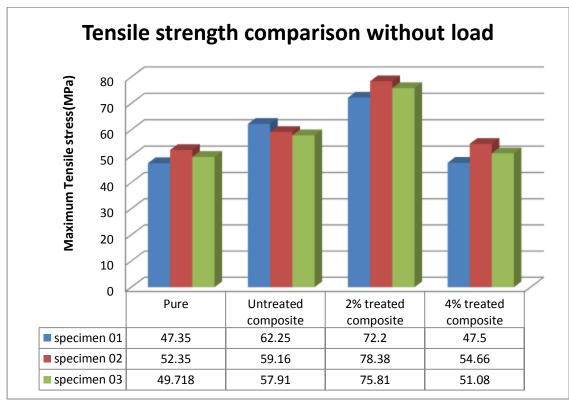


Figure 4. 22: Comparison of tensile strength without load

From the above figure we can see that when the fiber is treated with 2% NaOH, composite reinforced with this fiber shows superior properties.

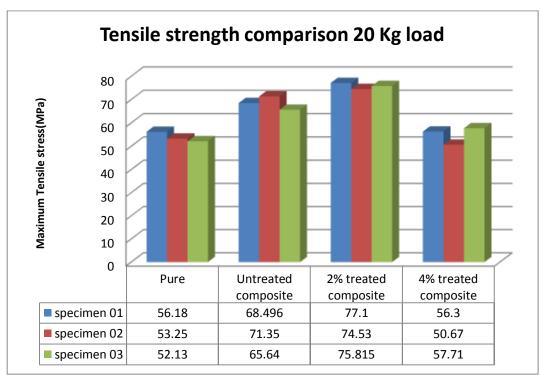


Figure 4. 23: Comparison of tensile strength with 20 kg load

Figure 4.23 indicates that the tensile properties of the composite reinforced with the fiber treated with 2% NaOH shows better results. In the case of 4% we see a decrease in the tensile properties.

The effect of fiber content on the tensile properties of fiber reinforced composites is of particular interest and significance for many researchers [57].

4.4 Flexural Comparison

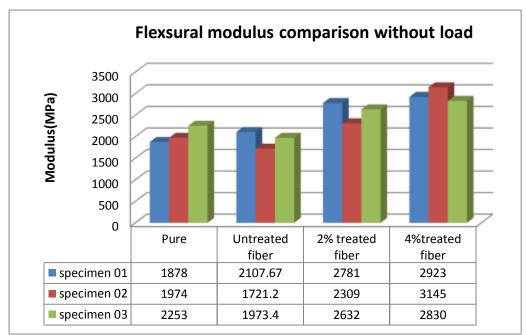


Figure 4. 24: Comparison of Flexural modulus with 20 kg load

From figure 4.24 we see that flexural modulus is optimum for the composite having 4% NaOH treated fiber. We see a gradual increase in the modulus values ranging from the pure casting to the alkali treated ones.

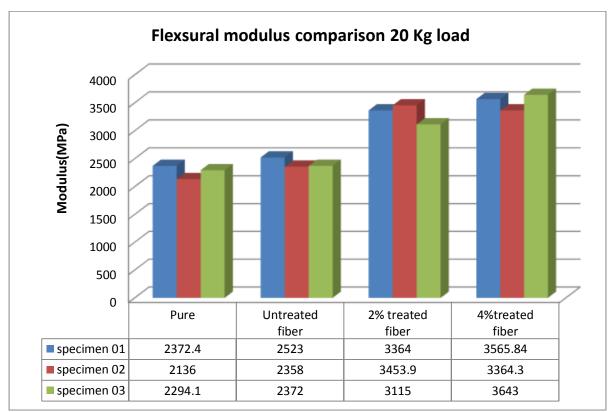


Figure 4. 25: Comparison of flexural modulus with 20 k load

From figure 4.25 we see that the flexural modulus of the composite increases gradually from the pure to the 4% NaOH treated composite. And it is this 4% NaOH treated composite that shows the best result.

Again from both figure 4.24 and figure 4.25 we see that we load is applied we see the increase in the property.

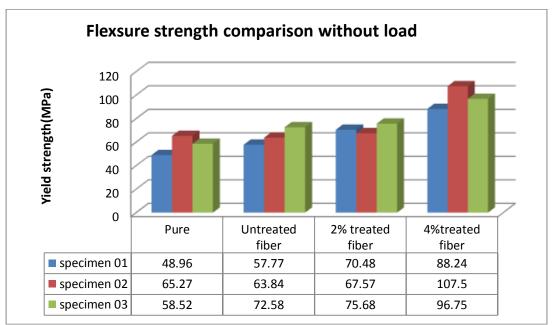


Figure 4. 26: Comparison of flexural strength without load

From the figure 4.26 we see that there is a gradual increase in the value of the yield strength ranging from pure to 4% NaOH fiber treated castings. Composite with 4% fiber treated shows the best property.

Due to alkaline treatment, hemicellulose and lignin are removed, the interfibrillar region is likely to be less dense and less rigid, and that makes the fibrils more able to rearrange themselves along the direction of tensile loading. When fibers are stretched, such arrangements among the fibrils would result in better load sharing and hence in higher stress development in the fiber [58].

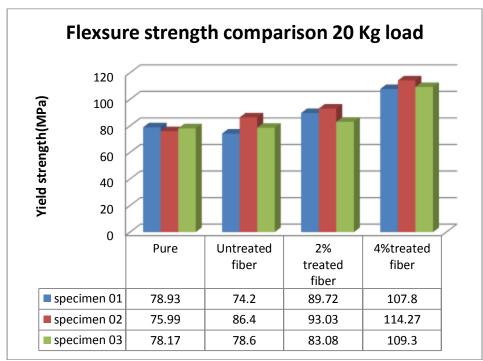


Figure 4. 27: Comparison of flexural strength of composites with 20 kg load

When load of 20 kg is applied the overall yield strength is improved. Here again we can see that composite having 4% treated fiber shows the maximum values of the yield strength. In both figure it is seen that the 4% fiber treated composite are having close values of yield strength.

4.5 Comparison of Fiber

When the fibers are treated, surface roughness is removed and surface of the fiber becomes smooth. That means the lignin and pectin components are removed and thus more refined fibers can be procured. If we take a look at the comparison of the tensile modulus of the fiber (figure 4.28), then it is clearly evident that, there is a gradual increase in the modulus values ranging from the untreated fiber to the 4% treated fiber.

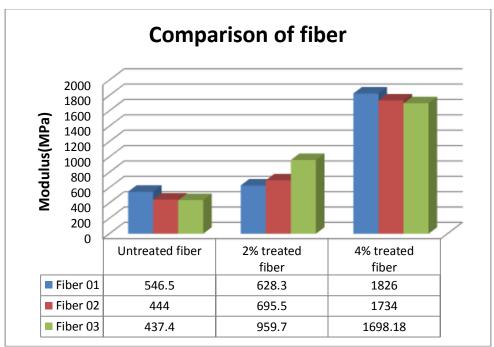


Figure 4. 28: Comparison of modulus of the fiber

When it comes to the comparison of the tensile strength we see the opposite scenario. The untreated or raw fiber shows superior property. Treated fibers lead to fiber smoothening which reduces the load bearing area of the fiber. Therefore the values of the tensile strength decrease in case the fibers are treated with NaOH. Figure 4.29 reveals the fact.

The excess of chemical solution did not only remove part of the boundary layers of the natural fibers but moreover caused some deterioration to the fiber particles themselves that reduced the overall tensile strength.

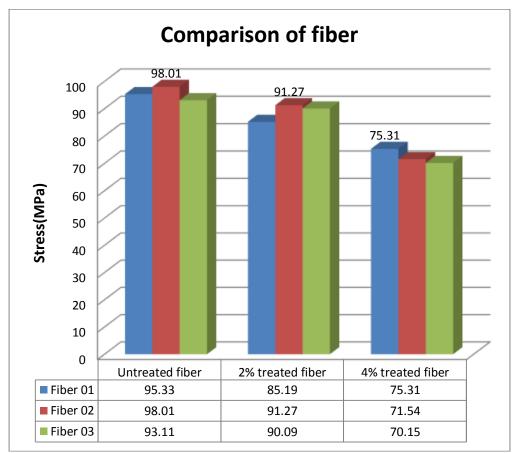


Figure 4. 29: Comparison of tensile strength of the fiber

4.6 Comparison of Hardness

Surface hardness of the composites is considered as one of the most important factors that govern the wear resistance of the composites. From figure 4.30 and figure 4.31 it is seen that the hardness values remains more or less close to each other no matter whether it is a pure polymer or a composite or whether it is loaded or unloaded.

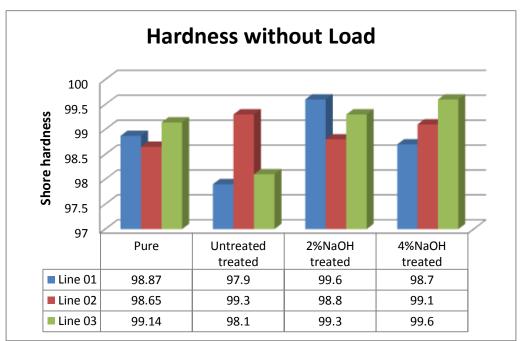


Figure 4. 30: Comparison of hardness with 20 kg load

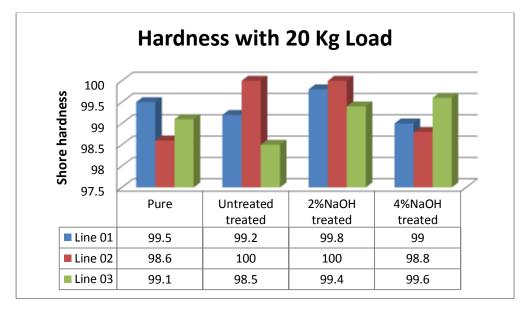


Figure 4. 31: Comparison of hardness values when the load is 20 kg

4.7 Comparison of All

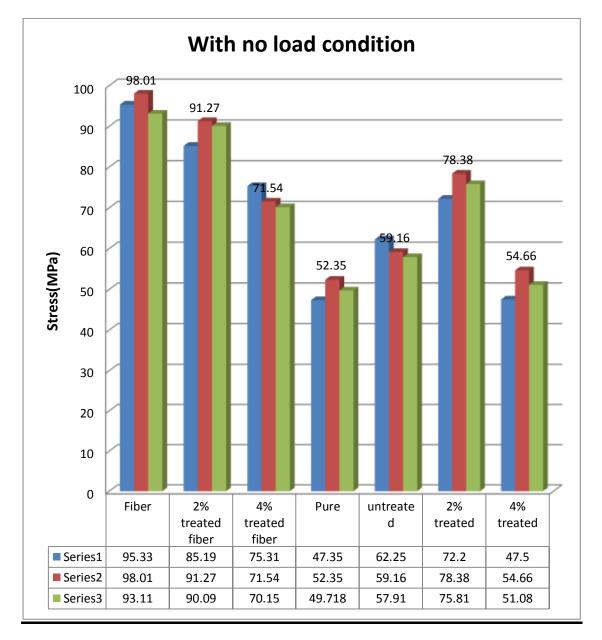


Figure 4. 32: Comparison of tensile strength with unloaded condition.

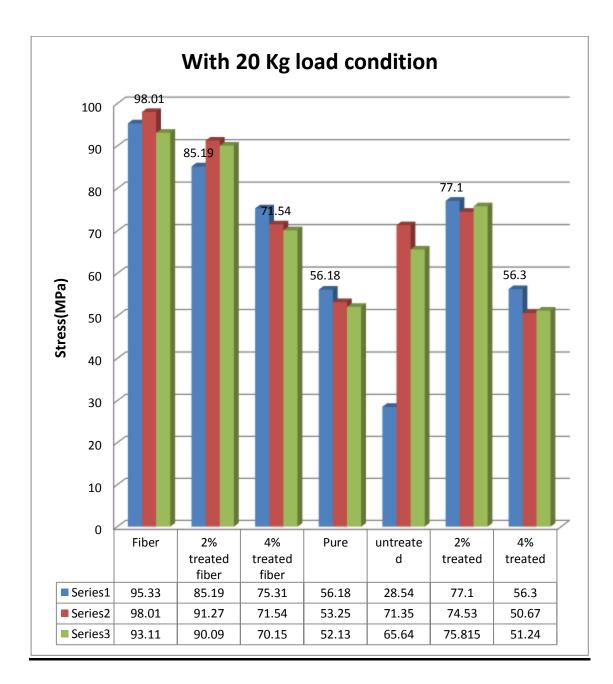


Figure 4. 33: Comparison tensile stress with 20 kg load

4.8 Fracture behavior

Fracture behavior of pure casting and composite shows dissimilarities. In case of pure casting polyester resin casting there a sharp fracture was observed across the cross sectional area.



Figure 4. 34: Fracture surface of pure polyester casting

Whereas in case of composite the cross sectional area of the fractured surface shows wavy or ripple like pattern which means the fracture surface is irregular and uneven across the cross section.



Figure 4. 35 : Fracture surface of the composite

In case of pure polyester resin there is no other load bearing element other than the matrix itself. So when the matrix reaches its threshold strength due to the absence of additional load bearing element there is a sharp fracture across the interface.

In case of composite the palmyra palm fiber the composite now has an additional load bearing element. Even if the matrix element exceeds its threshold the reinforcing fiber holds the composite from being broken. This is why the fractured surface of the composite shows wavy or ripple like pattern.

4.9 Thermo gravimetric analysis

TGA analysis was performed on the pure composite. The sample shows the thermal stability up to about 290⁰ celcius.

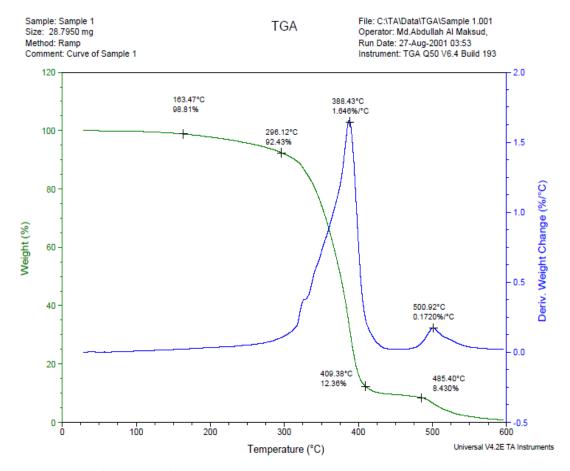


Figure 4. 36: TGA curve for pure polyester resin

Chapter 5

Conclusion and recommendation of future work

5.1 Conclusion

The following conclusions can be revealed-

- The raw fibers possess superior tensile properties compared to its pure polyester counterpart and further treatment with NaOH leads to the deterioration of the tensile property.
- Treatment of the raw fiber with 2 weight% NaOH and composite reinforced with this fiber shows an improvement in the tensile properties. But the polyester reinforced with 4 weight% NaOH treated fiber shows a decline in the tensile property. Therefore it is evident that fiber treatment up to a certain level should be maintained.
- Flexural yield strength gradually increases and it shows highest values for the composite of 4% NaOH treated fiber.
- Bending property of the composites of alkali treated fiber also increases.
- Application of a load of 20 kg increases both the tensile and flexural values compared to the composites prepared without load.
- Enhancement of fiber matrix bonding is increased by the application of load/pressure.
- No appreciable change in the hardness values was observed. Hardness values remained same over various composite.

Alkali treatment of the raw fiber leads the composite of improved property. Alkali treatment up to a certain point will give optimum properties. Alkali treatment yields better wet ability of fiber surface by the matrix which means an improved bonding will occur. Excessive fiber treatment damages the surface property of the fiber and the fiber matrix bonding becomes weak. Application of additional load further helps in strong bong between fiber and matrix and will improve the tensile and flexural properties of the composite.

Therefore, optimum alkali treatment and controlled and uniform loading of the casting product will improve the bonding strength of fiber and matrix which is in conformity with the increased numerical values of the properties.

5.2 Recommendation of future work

- 1. Alkali treatment with varying concentration should be carried out.
- 2. The fiber should be subjected to some other treatments.
- 3. The fiber should be used for preparing composites.

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