Modification on natural fiber and its effect on the mechanical properties of polymer composites

by
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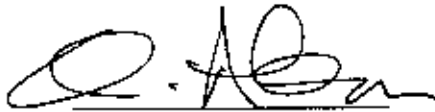
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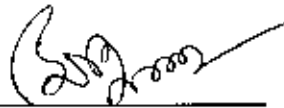   Dept. of Materials & Metallurgical Engineering
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It is hereby declared that this thesis or any part of it has not been submitted elsewhere for the award of any degree or diploma.

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DEDICATION

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ACKNOWLEDGEMENT

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ABSTRACT

Natural fiber composites combine good mechanical properties with a low specific mass and seem to be an alternative material to glass-fiber reinforced plastics in some technical application. The main purpose of adding cellulose-based filler to thermoplastics is to reduce cost/ unit volume and improve stiffness. This project deals with characterization of different types of treated and untreated jute fibers, to fabricate natural fiber-reinforced polymer composites and to investigate adhesion and interfacial bond characteristics with a view to optimizing key properties. Different types of jute were collected from Bangladesh Jute Research Institute. These jute types are raw jute, retted jute, scoured jute. These fibers are treated with maleic anhydrate and sodium hydroxide. Then the untreated and treated fibers of three types of fibers are characterized. Tensile strength, linear density, characteristics strength, weibull modulus are determined by following standard. Retted jute fiber possesses greater strength than that of raw jute fiber Because the fiber surface of retted jute are smoother than that of raw jute Any types of treatment natural (retting), chemical (Maleic Anhydride, Sodium Hydroxide) causes weight loss of fiber. Which leads to decrease in diameter of individual fiber and decrease in linear density of fiber. Both MAH and NaOH treatment increase the strength of jute fiber But in case of NaOH treatment the increase is larger.

Both types of treated and untreated fibers thermally characterized by Differential Scanning caloriemetry (DSC) and Thermo gravimetric analysis (TGA). Scanning electron microscopy techniques used to investigate the structure and surface morphology of untreated and treated jute fibers. Water absorption test are carried out and the weight loss associated with two types of treatment calculated. The presence of roughness in case of raw jute fiber surface structure facilated evaporation of moisture at lower temperature. The percentage degradation for the α-cellulose of retted fiber under treated and untreated condition was always higher than that of raw jute fiber. In case of NaOH treated retted fibers, the α-cellulose decomposition peak changed from endothermic to exothermic. The removal of hemicellulose during NaOH treatment leading to the destruction of the chemical linkage between the constituents might have some influence in inverting the α-cellulose decomposition peak from endothermic to exothermic. The untreated and treated fibers are chopped to three definite size (5mm, 4mm, 2 mm). Composites are fabricated by using polypropylene as matrix materials with these various types of chopped jute fibers by hot

pressing Four different volume percentages of fibers are incorporated Mechanical characterization of these fabricated composites was done using Universal Testing Machine. ASTM D3039 test method was followed for tensile test of composite specimen; ASTM D790 test method was followed for flexural or three point bend test of composites Composite with larger fiber size shows greater strength Overall composite tensile stresses are lower than that of reference polymer. MAH treated jute fiber composites showed lower stress than that of untreated jute polypropylene composites and NaOH treated jute polypropylene composites. Tensile moduli of all composites are greater than that of reference polymer. In case of MAH treatment the composite shows better tensile modulus. Failure surface and failure profile were observed under a scanning electron microscope (SEM) to reveal the post failure characteristics of the composite body. Predominant mode of failure for individual loading condition of these composites were analyzed comparing the failure profile. Scanning electron microscopy shows that there are fiber pull out in tensile failure surface and polymeric residue on fiber surface. Failure mode also interface dependent. During tensile test the fibers that lie perpendicular to the load directions acted as flaw and non-loadbearing component of composites. Flexure stress of three types of composites are lower that that of reference polymer. MAH treated jute fiber composites showed lower stress than that of untreated jute polypropylene composites and NaOH treated jute polypropylene composites. Flexure stiffness increase to a considerable amount than that of reference polymer. Composites are also thermally analyzed using DSC and TGA. TG/DTG, DSC curve of composites shows that final degradation temperatures of composites are higher than that of jute fibers. MAH treated jute polypropylene composites showed higher decomposition temperature for 1st peak whereas NaOH treatment lowers this peak temperature for retted fiber. All composites showed lower 2nd peak temperature than that of jute fibers.

CHAPTER – 1
INTRODUCTION
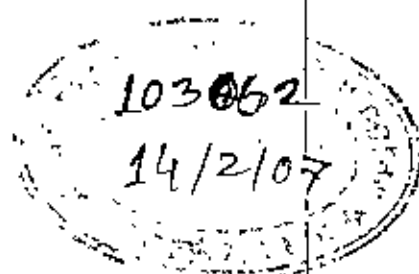
In recent years, a worldwide ecological awareness has increased and there is a renewed interest in the production of low-cost, biodegradable materials with improved properties. The use of cellulosic or lignocellulosic materials such as cellulose fibers, wood fibers, sisal or jute fibers, nut flour etc. as fillers or as reinforcement for polymeric matrices has increased. These materials are inexpensive and they are easily obtained from renewable natural resources. They also posses other advantages such as low density and flexibility, their use result in low equipment wear during their processing, and most important, they are biodegradable in comparison to manmade fibers such as glass fibers which are still the most commonly used reinforcement in composites. Cellulose fibers have been frequently used as the reinforcing phase in thermosetting matrice but the presence of hydroxyl groups on the surface of the cellulose fiber is, synonymous of both high energy and a hydrophilic character. On the contrary, most of the thermoplastic matrices used for composites such as polypropylene, polyethelene etc. have a modest cohesive energy and a strong hydrophobic character (non-polar materials). Because of natural fiber's high level of moisture absorption, poor wetability by non-polar plastics, and insufficient adhesion between untreated fibers and the polymer matrix, lead to debonding with age. These necessitates further research on these topics.

In general, the use of fiber-reinforced plastic composites is continually increasing; nevertheless, certain aspects of their behavior are still poorly understood. Due to these aspects, the situation for natural fiber-reinforced plastics is much more unsatisfactory as for glass-fiber-reinforced plastics because of the lack of systematic and detailed information. Due to this fact this project deals characterization of different types of treated and untreated jute fibers, to fabricate natural fiber-reinforced polymer composites and to investigate adhesion and interfacial bond characteristics with a view to optimizing key properties. The specific aims related to this orientation are:

- Treatment of different kind of fibers to develop the interfacial bonding characteristics.
- Development of fiber plastic blending techniques for good fiber/matrix compatibility in semi finished, finished or pellet products.
- Evaluation of composite performances by determining its mechanical properties

This thesis consists of several chapters. Chapter 2 contains brief literature review to help to get some idea about the continuous progress of natural fiber reinforced polymer composites and current interest of the project. The third chapter describes the experimental procedure that are used throughout this project such as treatment of different types of fibers, mechanical and thermal characterization of both treated and untreated fibers, composites fabrication, mechanical characterization (tensile and flexure tests) of fabricates composites and thermal characterization of composites. Chapter four deals with the results obtained from the experimental works and the discussion about the effect of treatment on fibers as well as in composites. Scanning electron miscopy helps the analyses to be in detail In fifth chapters the finding from the works are listed

CHAPTER 2
THEORETICAL BACKGROUND

2.1 INTRODUCTION

Lignocellulosic fibers have become a focus of intense interest in recent years. They have attracted the attention of scientist worldwide because of their tremendous advantages and they are solid proof that it is possible to obtain materials that perform well using environmentally friendly reinforcements. Natural fibers are classified according to their source – plants, animal or minerals. In general, it is the plant fibers that are used to reinforce plastics in the composite industry. Many varieties of plant fibers exist, such as hairs (cotton, kapok), fiber-sheafs of dicotylic plants (e.g. flax, hemp, jute and ramie), and hard fibers (sisal, henequen and coir), not to mention the large number of fibers obtained from trees. The most abundant are wood fibers from trees, however, other fiber types are beginning to emerge in use The abundance of the raw material is also a concern to the manufacturing industry, and the pressures on it to use ever 'greener' technologies have made this area of research of worldwide interest

The growing interest in lignocellulosic fibers is mainly due to their economical production with few requirements for equipment and also due to the reduction of the total mass of the composite as a result of the low density of natural fiber.

They also present safer handling and working conditions compared to synthetic reinforcements. The most interesting aspect about natural fibers is their positive environmental impact. As they come from a natural resource they are completely biodegradable and nonabrasive material and can be easily eliminated after the degradation of the polymer. Also the hollow cellular structure provides excellent insulation against heat and noise. The high sound attenuation of lignocellulosic composites is another advantage [1]. Vegetable fibers also provide a relatively reactive surface, which can be used for grafting specific groups and introducing new functional entities. In addition, the recycling by combustion of polysaccharide filled composites is easier in comparison with inorganic fillers.

The inherent polar and hydrophilic nature of lignocellulosic fibers and the non-polar characteristics of most thermoplastics results in compounding difficulties leading to non-uniform dispersion of fibers within the matrix, which impairs the efficiency of the

composite. This is a major disadvantage of natural fiber-reinforced composites. Another problem is that the processing temperature of composites is restricted to 200°C, as vegetable fibers undergo degradation at higher temperature. Another setback is the high moisture absorption of natural fibers leading to swelling and presence of voids at the interface, which results in poor mechanical properties. It is apparent that the advantages of natural fibers far out weight the limitation and major drawbacks can be minimized by means of chemical modifications.

Composite materials that have both economic and environmental benefits are being considered for applications in the automotive, building, furniture, and packaging industries. Mineral fillers and fibers are used frequently in the plastics industry to achieve desired properties or to reduce the cost of the finished article. Over the last few years a number of researchers have been involved in investigating the exploration of cellulosic fiber as load bearing constituents in composite materials. Polymer based composites having natural fibers (jute, flax, hemp etc) as reinforcement have the potential to be attractive alternative to synthetic fiber composites, and are currently being explored for novel uses in automotive parts, geo-textiles, and building composites [2].

Glass, carbon, boron and kevlar fibers are being used as reinforcing materials (fiber reinforced polymer, FRP), which have been widely accepted as materials for structural and non-structural application The main reason is the high specific modulus, high specific stiffness and high specific strength compared with conventional materials. These materials are in general expensive and the main field of application is automotive and aviation purpose and in some cases larger civil structures. Therefore, natural fibers like, jute, banana, cotton, coir, sisal have attracted the attention of scientists and technologist for application in consumer goods, low cost housing and civil structures. These materials posses better electrical resistance, good thermal and acoustic insulating properties and higher resistance to fracture. As, jute fiber is inexpensive and commercially available in the required form, so it appears to be a promising material as reinforcement The specific strength and modulus of jute is higher than plastic and is a good substitute for conventional fibers in many cases [2].

The jute composites may be used in everyday applications such as lampshades, suitcases, paperweights, helmets, shower and bath units They are also used for covers of electrical appliances, pipes, postboxes, roof tiles, grain storage silos, panels for partition and false ceilings, bio-gas containers, and in the construction of low cost, mobile or pre- fabricated

buildings which can be used in times of natural calamities. Good quality jute fibers have three principal chemical constituents, namely, α-cellulose, hemicellulose and lignin. In addition, they contain minor constituents such as fats and waxes, inorganic (mineral) matter, nitrogenous matter and traces of pigments like β-carotene and xanthophylls [3]

As in synthetic fiber composites, the mechanical properties of the final product depend on the individual properties of the matrix, fiber and the nature of the interface between the two. Where the fiber is an agricultural one, it is possible to tailor the end properties of the composite by selection of fibers with a given chemical or morphological composition. Several studies of fiber composition and morphology have found that cellulose content and micro fibril angle tend to control the mechanical properties of cellulosic fibers [4]. Higher cellulose content and lower micro fibril angle result in higher work of fracture in impact testing. Sisal and banana fibers show better reinforcing efficiency than coir and the specific strength properties of the composites are comparable to those of glass fiber reinforced plastics (GFRP). On the other hand, coir fiber, despite having low strength and modulus, improves the impact resistance of polyester due to its large strain energy absorption [2]

2.2 VARIETIES OF JUTE

Jute is the common name given to the fiber extracted from the stems of plants belonging to the genus *Corchorus*, family Tiliaceae. Kenaf is the name given to the rather similar fiber obtained from the stems of plants belonging to the genus *Hibiscus*, family Malvaceae, especially the species H *cannabinus* L Only two species of *Corchorus*, namely, C *capsularis* L. and C. *olitorius* L., are grown commercially, although around 40 wild species are known, but other species of *Hibiscus*, particularly *H. sabdariffa* L are sometimes also marketed as kenaf [5].

These plants are examples of a number of woody-stemmed herbaceous dicotyledons grown in the tropics and subtropics Most of the plants cultivated for fiber, including jute and kenaf, are grown from seed annually, but a few are grown as perennials. Jute is the most important fiber of this type, and it is probable that, in the industrial and engineering uses of textiles more jute fiber is used than any other single fiber. Kenaf is used in many countries where it is grown, but its international market is much smaller than that for jute. In many marketing statistics, figures given for the production or utilization of "jute and allied fibers" include all the fibers in this group. "Allied fibers" are suitable for processing on jute spinning systems

[5]

2.3 THE CULTIVATION AND PROCESSING OF JUTE

Jute has a long history of service to man. The production of jute has occasioned enormous industrial development. It provides a livelihood for millions of people. Today, stabilized economically, established in its traditional markets, advance technology, the jute industry looks to broader utilization of its products.

Jute is an annual plant of the genus Corchorus, grown entirely for its fiber Jute is a rainy season crop, sown from March to May according to rainfall and type of land, and harvested from June to September depending on whether the sowings are early or late. It thrives best in damp heat, and the climatic conditions. Mesta, or Kenaf, botanically known as Hibiscus Cannabinus, is also grown in these climatic conditions. Mesta is a coarser, more brittle fiber, and is used by the jute mills in admixture with jute to obtain certain desired properties. Jute plants are ordinarily ready for harvesting about four months after sowing The jute plant is an herbaceous annual that grows to an average of 3 to 3 6 m in height, with a cylindrical stalk about as thick as a man's finger. The two species (mesta & kenaf) grown for jute fiber are similar and differ only in the shape of their seedpods, growth habit, and fiber characteristics. Most varieties grow best in well-drained, sandy loam and require warm, humid climates with an average monthly rainfall of at least 75 to 100 mm during the growing season. The plant's light green leaves are 10 to 15 cm long, about 5 cm wide, have serrated edges, and taper to a point [5].

2.3.1 Retting of Jute

Retting is the process used to ensure the long bast fibers undamaged. It involves decomposition by bacteria and moulds (fungus) by which the pectin (binds the fibrous and non-fibrous portion of the stalk) is broken down. Once the pectin is broken down the fiber and the stalk can be easily separated. There is a period of drying following the decomposition period

The plants are harvested by hand with a sickle and cut close to the ground. The cut stems are then tied into bundles, the leaves removed as much as possible, and the bundles submerged in water for retting. This is the process by which the bundles of cells in the outer layers of

the stem are separated from the woody core and from non-fibrous matter by the removal of pectin and other gummy substances. The action involves water, microorganisms, and enzymes, and takes between 5 and 30 days to completion, depending on the temperature of the water. Usually hot humid weather condition accelerates retting. The suitable temperature range was in between 25-30°C [5].

Constant supervision is required, and the time of removal is critical, because if the degree of retting is insufficient, the fiber cannot easily be stripped from the woody core and may be contaminated with cortical cells; and if retting proceeds too far, the fiber cells themselves may be attacked and weakened by microorganisms. A difficulty in the retting procedure is that the thicker parts of the stem take longer to ret than the thinner parts do; consequently, if the butt ends of the stem are fully retted, the top ends are over retted and damaged. This can be avoided by stacking the bundles of stems upright with the butt ends in water for a few days before immersing the whole stem; but with fiber intended for export, it is usual to cut off the partly retted butt ends and sell these separately as "cuttings." Correct retting is an essential first step in the production of good quality fiber. Controlling the quality of water along with improving microorganisms used in the process are the keys to improved fiber quality. Stripping the fiber from the stem is done by hand, after which the fibers are washed and dried under sunlight.

In Bangladesh under water retting or deep water retting or simply water retting is most familiar among the jute growers. So to extract jute fiber from the stem deep water retting is mainly selected [6]. There are several different retting process; Water Retting, Dew Retting, Warm Water Retting, Green Retting and Chemical Retting.

The jute plant's fibers lie beneath the bark and surround the woody central part of the stem. The fiber strands nearest the bark generally run the full length of the stem. A jute crop is usually harvested when the flowers have been shed but before the plants' seedpods are fully mature. If jute is cut before then, the fiber is weak; if left until the seed is ripe, the fiber is strong but is coarser and lacks the characteristic luster [5].

Gummy materials hold the fibers together, these must be softened, dissolved, and washed away to allow extraction of the fibers from the stem, a process accomplished by steeping the stems in water, or retting. Soft tissues in the outer layer of the stem, between the wood on the inside, and the cortex on the outside, surround the jute fiber. During retting, these tissues are

softened with the results that the fiber can be separated readily in the subsequent process of stripping. After harvesting, the bundles of stems are placed in the water of pools or streams and are weighted down with stones or earth. They are kept submerged for 10-30 days, during which time bacterial action breaks down the gummy tissues surrounding the fibers. After retting is complete, the fibers are separated from the stalk by beating the root ends with a paddle to loosen them; the stems are then broken off near the root, and the fiber strands are jerked off the stem. The fibers are then washed, dried, sorted, graded, and baled in preparation for shipment to jute mills. In the latter, the fibers are softened by the addition of oil, water, and emulsifiers, after which they are converted into yarn. The latter process involves carding, drawing, roving, and spinning to separate the individual fiber filaments; arrange them in parallel order; blend them for uniformity of color, strength, and quality; and twist them into strong yarns The quality of the fiber depends greatly on the care exercised in retting [5].

Warm water retting is similar to water retting, but the stalk is soaked for 24 hours when new water is added and brought to an elevated temperature for about 2 or 3 days. Dew retting is another traditional method. It relies on frequent rain and dew. Once the stalks are cut, they are spread evenly over the ground, allowing the rain and dew to induce the breakdown of the pectin. This method, without proper equipment, is labor intensive. There is a fine line between retted and rotted, so the stalks must be lifted at the right time otherwise it will be useless. This process may take one to two weeks with favorable weather (humid and warm), but usually four weeks is necessary After retting, there is a period of drying and the fibers are then stored for further processing. Dew retting produces a coarse fiber, light brown in color [6].

Green stalks are mechanically processed, separating the fiber from the stalk The high quality fiber can be refined for the textile industry The remaining stalk can be used in the paper and fiberboard industries [7] Chemical Retting consists of placing stalks in a processing tank where chemical agents are used to dissolve the pectin. By maintaining a constant processing temperature the retting time can be reduced to 48 hours while producing a very high quality fiber. After the retting process the fiber and the stalk are loosely held together and must be decorticated, scutched, hackled and combed to remove the remaining pieces of stalk, broken fibers and extraneous material. Mechanical decortications equipment can be used in conjunction with turbine scutchers to separate the fiber and the non-fibrous portion [8].

2.3.2 Pulping

Pulping is the process whereby wood or non-wood fibers are treated either chemically, mechanically, chemico-mechanically, thermo-mechanically or using suitable solvent. Pulping process turned the fibers into slurry, which is frequently used in papermaking There are many grades of paper, each requiring particular slurry characteristics. Chemical pulping is most often used with the wood chips. Chemicals are added to the wood and/or fiber product and it is cooked in a pressure cooker. Once the lignin is dissolved the liquid that is left is called black liquor. Chemical recovery procedures are used and the resulting liquid from this process is often burned away It is expensive for non-wood fibers such as jute, which contains high lignin content because sometimes a significant portion of the non-wood fiber is lost [9].

2.3.3 Bleaching of Jute Fiber

The bleaching process of jute and allied fiber in modem composite preparation technology is not only concerned with brightening fiber yam or fabric by the destruction of natural coloring matter, but it frequently makes a significant contribution towards subsequent advantage in fabrication of composite and improvements in properties and appearance of natural fiber reinforced polymer composite [5]

Jute is bleached in jute to bleaching liquor ratio of 1: 20. It is important to achieve the right degree of stability. If the pH is too low, bleaching does not take place; if the pH is too high the oxygen in hydrogen peroxide decomposes rapidly even before it has time to bleach the stalk. It is virtually impossible to adjust to the optimum pH with alkali alone, and it's necessary to add a stabilizer, keeping the pH within the limits of 8-10. The most commonly used stabilizer for bleaching jute & allied fiber is sodium silicate, which has the advantage of giving protection against those contaminants, which can cause catalytic decomposition. Typical bleaching liquor can be made up as follows:

1 Sodium silicate, 2 to 3 percent

2. Caustic soda, 0.6 to 1 4 percent

3. Hydrogen peroxide (35 percent) 3 to 5 percent

4. Wetting agent or detergent as required

All the percentages are calculated on the weight of the fibers. The container is filled with water to- about one-third capacity and brought to a boil, then the fibers are fed in, and the

remainder of water added. The water temperature drops. The temperature of the solution is then raised gradually to the desired temperature, making sure residual pockets of air escape. In an open vessel, bleaching may take five to six hours [10].

Jute bleaching is being carried out at Bangladesh Jute Research Institute following the above procedure. Required amount of jute fiber is treated with a solution containing 15g/l hydrogen peroxide, 10g/l sodium silicate, 2g/l sodium carbonate and 0.5g/l wetting agent (Hostapol) to maintain jute to liquor ratio of 1: 20 at temperature of 90-95°C for 1hr. It is then washed several times with water and dried. Sodium silicate and metasilicate are stabilizers, which are added to control the decomposition of hydrogen peroxide (H_2O_2) to water (H_2O). Whenever possible, a simple solution of hydrogen peroxide (and possibly a stabilizer) is recommended for stain removal because as the hydrogen peroxide loses oxygen, it bleaches and converts to water. Therefore, there is no danger of continuing chemical action [10].

Hydrogen peroxide can be used on all vegetable fibers and its bleaching effect is permanent. Bleaching before exposure generally accentuates the discoloration of the fiber compared with the color of unbleached jute. Although part of this is due to the heightened contrast between the nearly white bleached fiber and the exposed fiber. The onset of yellowing varies considerably with the use of different bleaches. Alkaline or neutral hypochlorite, a cheap bleaching medium, gives a product with a rather rapid yellowing tendency, whereas alkaline hydrogen peroxide gives a good white color and a less marked yellowing. Sodium chloride applied under acid conditions, results in the least yellowing tendency, but care must be taken that, in obtaining the best conditions to prevent yellowing, no drastic loss of strength takes place [5, 11]

The trend is to avoid the use of any kind of chlorine chemicals, so called total chlorine free (TCF) bleaching. The use of elemental chlorine for bleaching is not recommended. Only elemental chlorine free (ECF) process are acceptable but total chlorine free (TCF) processes are preferable (from an environmental perspective). The reason of discarding elemental or chlorine compound bleaching for jute fiber is because some of these chlorinated organic substances are toxic, and include dioxins, chlorinated phenols, and many other chemicals. It is generally not practical to recover chlorinated organics in effluents since the chloride content causes excessive corrosion while adopted industrially. There are many advantages to bleaching with hydrogen peroxide. Before bleaching with chlorine bleach, it was necessary

to boil cotton with caustic soda solution to remove any traces of proteins. Since hydrogen peroxide does not react with proteins, a permanent white is obtained without the preliminary alkali boil. Hydrogen peroxide bleaching can take place with scouring. The presence of impurities has a desirable effect of stabilizing the hydrogen peroxide. Finally, there is a less risk of tendering due to over bleaching [6].

The objective of bleaching is to remove the small fraction of the lignin remaining after retting. Oxygen, hydrogen peroxide, ozone, per acetic acid, sodium hypochlorite, chlorine dioxide, chlorine and other chemicals are used to transform lignin into an alkali soluble form. An alkali, such as sodium hydroxide, is necessary in the bleaching process to extract the alkali soluble form of lignin. Sodium carbonate can be used for the same purpose as that of the sodium hydroxide, because it behaves most like sodium hydroxide alkali in aqueous solution. More over softening of fibers is facilitated along with fair bleaching treatment and to maintain the pH of the solution. Surfactants or wetting agents are a type of chemicals that lower the water's tension, permitting it to expand and infiltrate more easily into the fiber body. These are mixed with grease and water to reduce the adhesion of soils. Surface-active substances are frequently used as auxiliaries in bleaching. If these entail the enhancement of fiber or fabric wet ability, these could be expected to also improve the efficiency of the bleaching process, although strong capillary action occurs in between the two mediums. Additionally the wetting agent acted as a stabilizer of the colloidal suspension formed during bleaching [6].

2.3.4 Woolenization of Jute Fiber

Jute fibers are treated with 20% caustic soda solution for 10 minutes at room temperature. The fibers were hydro - extracted to recover caustic solution followed by water wash and neutralized with 2g/l sulfuric acid The stalks are then again hydro - extracted and then dried. When jute fiber is treated with strong alkali, profound changes occur in its physical structure. Lateral swelling occurs, together with considerable shrinkage in length, as a result of which the fiber is softened and develops a high degree of crimp or waviness. The crimp gives wool like appearance to the fiber. On stretching the fibers to break, the crimp is straightened and thereby the extensibility of the fiber is increased. The effect is small at alkali concentrations of up to about 10%, but the extensibility increases rapidly at concentrations of 15% and upward and may reach 8 or 9% [7].

Water wash is done several times to remove the excess NaOH solution and to collect it followed by 2g/1 sulfuric acid solution treatment. This treatment provides neutralized fiber, which is again followed by water extraction to remove sulfuric acid form the fiber Strong mineral acids or acid salts known to cause hydrolysis of the cellulose resulting in the damage of the fiber structure, so the acid concentration is kept low enough, just to ensure the neutralization process [8].

2.4 PROCESSING OF JUTE

The first step in preparing the fiber is "batching," consisting of blending the various fibers to obtain uniformity in strength and color to give the precise quality of yarn for spinning [12].

In the first mechanical operation in the mill, the jute is fed into a softener in which the jute, treated with an emulsion oil and water, passes between sets of heavy spiral fluted rollers. This process renders the fiber thoroughly pliant and removes any barky portions adhering to the fiber [5, 12].

The fibers are then carded in machines, known as breaker cards and finisher cards, which reduce the average length of the fibers by teasing and combing, and deliver them in the form of a long continuous ribbon, 5" or 6" in width, called sliver. The carded jute is next fed into drawing machines which draw out and attenuate the sliver, parallelize the fibers, and by means of a doubling process, produce a smoother, more even sliver. The last operation in the preparing department is roving, a process which imparts a slight twist to the sliver and delivers the material on to bobbins in the form of rove, a loose yarn ready for spinning. Other spinning machinery known as sliver spinning, an extra drawing operation is substituted for the roving step. This machine delivers a crimped sliver [12].

Spinning frames convert the rove to finished yarn. After spinning, the yarns are wound into the form required -spools for warp yarn and cops for weft yarn – for subsequent processing. Jute yarn is processed much like other textile fibers, the yarn it being dressed (i.e. sized or starched), before being passed on to the warp beam ready for weaving. Jute fabrics are of simple construction and are woven on a variety of looms. Woven fabrics are inspected, damped and calendared to produce the desired smoothness of finish [12].

The chemical treatment procedures of jute fiber and to that of woven fabric are the same.

2.5 EXTRACTION, CHEMICAL STRUCTURE & COMPOSITIONAL CHANGES OF JUTE FIBER

Cellulose is the main building block of natural fiber. It was observed that when plant tissue cotton linters, root tips, pit and ovules from the flowers of trees were treated with an acid-ammonia followed by water extraction a constant fibrous material is formed. Since then it has been accepted that cellulose is a linear polymer consisting of D - anhydro-glucose unit joined together by β-1, 4 glycoside linkages [10].

The anhydro-glucose units do not lie exactly in plane but assume a chair confirmation, with successive glucose residues rotated through an angle of 180° about the molecular axis. The chemical structure of cellulose is available in many books of organic chemistry and books on natural polymer. The mechanical & properties of cellulose containing natural fiber is well established and reviewed by many researchers during the past few years. A lot of work has been done on the chemical composition of jute. Jute, like most vegetable fibers, contain a portion of acetyl groups that are readily hydrolyzed by dilute alkali to acetic acid, and quantification of acetic acid per unit weight of fiber provides an index of acetyl content. The acetyl content of any particular type of fiber shows some variation according to their growing condition. The higher acetyl content in Capsularies over them in olitorius has science been confirmed The changes in chemical composition at different stages shows that there is little difference in cellulose, homocellulose, and lignin contents, but that xylan, ash, and iron contents decrease as the plant matures. The aggregate fiber length increases as the growing season progresses. Studies of the chemical composition differences among leaves and stems shows that the pentasans, lignin, and cellulose content increase with age, while the protein and hot water-extractives content decrease with age. The top part of the plant shows similar trends, but the top part has less cellulose, pentosans, and lignin but higher hot-water extractives and protein than the bottom part of the plant [5].

Studies of the changes in kenaf fiber properties during the growing season shows that the bast single fibers are longer than the pith fibers and that both decrease in length with age. Pith single fibers are twice as wide and have twice the cell wall thickness as bast single fibers; both dimensions decrease with age. Finally, the lumen width is greater in pith fibers as compared to that of bast single fibers, and both decrease with age. In a recent study,

changes in kenaf as a function of the growing season showed that the average length of a bast and core (stick) fiber increased as the plant aged. The surface characteristics of jute fiber at different stages of growth shows that at the early stages of growth, there was an incomplete fonuation of the middle lamella in the cell wall and that the parallel bundles of fibrils were oriented as an angle with respect to the fiber axis that gradually decreased with growth [5].

2.6 FINE STRUCTURE AND PHYSICAL PROPERTIES OF JUTE FIBER

The locations of the three main chemical components of the fibers are reasonably well established α-Cellulose forms the bulk of the ultimate cell walls, with the molecular chains lying broadly parallel to the direction of the fiber axis. The hemi cellulose and lignin, however, are located mainly in the area between neighboring cells, where they form the cementing material of the middle lamella, providing strong lateral adhesion between the ultimates. The precise nature of the linkages that exist between the three components and the role played by the middle lamella in determining the fiber properties are not completely understood and a thorough understanding of the inter cell structure is still awaited [5].

Cellulose is found not to be uniformly crystalline. However, the ordered regions are extensively distributed throughout the material, and these regions are called crystallites. The threadlike entity, which arises from the linear association of these components. is called the micro fibril, it forms the basic structural unit of the plant cell wall. These micro fibrils are found to be 10-30 nm wide and indefinitely long containing 2-30,000 cellulose molecules in cross-section Their structure consists of a predominantly crystalline cellulose core. This is covered with a sheath of paracrystalline polyglucosan material surrounded by hemicelluloses In most natural fibers these micro fibrils orient themselves at an angle to the fiber axis called the "micro fibril angle" [2, 5].

The measurement of the micro fibril angle via small angle and wide angle X-ray scattering of the cellulose in cell walls of softwood is an essential step in the process of understanding the elastic, creep and strength properties of a particular fibrous specimen An alternative method by which the micro fibril angle is measured is that the specimen is irradiated in a direction at 45° to both sets of cell walls. It is not easy to extract a value for the micro fibril angle from the diffraction pattern because the diffractions from the individual fibers overlap. X-ray diffraction patterns show the basic cellulose crystal structure. But in jute and kenaf, although

the crystallite orientation is high, the degree of lateral order is relatively low in comparison with, for example, that of flax. There is also considerable background x-ray scattering arising from the non-cellulosic content of the fiber. The cellulose molecular chains in the secondary walls of ultimate cells lie in a spiral around the fiber axis. The effect of this is to produce double spots in the x-ray diffraction patterns, the centers of the spots being separated by an angular distance of twice the Bragg angle [2].

There is a particular relationship between the fiber orientation and extension to that of the spiral angle. X - Ray measurements shows the angle to decrease with extension, explained by spring structure. To carry out similar measurements of the extension/spiral angle relationship for low-angle fibers such as jute and kenaf is difficult. Because of the changes in angle are small, the overlapping of the spots in the x-ray diffraction pattern could introduce significant errors. It appears that the helical spring theory could be used to calculate the order of magnitude of the extensibility of the fiber and to rank fibers accordingly. This concept could be used in explaining the reinforcing mechanism of these fibers in polymer matrix [Roger M. Rowell, 1998]. Considerable works on measuring the physical properties are the main area of concern of the textile engineers and composite research scientist [2]

Jute and kenaf are strong fibers but exhibiting brittle fracture and have only a small extension at break. They have a high initial modulus, but show very little recoverable elasticity. Tenacity measurements recorded in literature vary widely, and although some of this variation is due to differences in the methods of measurement, a major part is due to variations in linear density of the fibers themselves. The breaking load of the fibers, however, had the much higher coefficient of variation of 40% [2, 13].

The initial Young's modulus of the fibers, calculated from the slope of the load elongation curve. The value for any particular group of fibers, of course, will be dependent on the linear density, to some extent owing to the dependence of tenacity values on this factor. Examination of the effect of delignification on the bending modulus of jute, using the fringe technique has shown that successive extractions of lignin from the same fibers resulted in an increasing flexibility and a decreasing Young's modulus [5]. The physical properties of natural fiber with the comparing data of the artificial fiber are presented below in table 2.1.

Table 2.1: Mechanical properties of natural fibers compared to conventional composite reinforcing fibers [5].

Fiber	Density (g cm^{-3})	Elongation at break (%)	Tensile strength (MPa)	Young's Modulus (GPa)
Cotton	1.5-1.6	7 0-8 0	287-597	5.5-12.6
Jute	1.3	1.5-1.8	393-773	26.5
Flax	1.5	2 7-3.2	345-1035	27.6
Hemp		1 6	690	
Ramie		3 6-3.8	400-938	61.4-128
Sisal	1.5	2.0-2.5	511-635	9 4-22 0
Coir	1.2	30 0	175	4.0-6.0
Viscose (cord)		11 4	593	11.0
Soft wood Kraft	1.5		1000	40 0
E-glass	2.5	2 5	2000-3500	70 0
S-glass	2 5	2 8	4570	86 0
Aramid (normal)	1 4	3.3-3.7	3000-3150	63 0-67.0
Carbon (standard)	1 4	1.4-1 8	4000	230.0-240 0

2.7 FIBER MORPHOLOGY

All plant species are built up of cells. When a cell is very long in relation to its width it is called a fiber The fiber is like a microscopic tube (i.e., a wall surrounding a central void referred to as the lumen). Moreover, when the cell wall is made up mainly (85% or more) of cellulose, hemi cellulose and lignin, is known as lignocellulosic fibers [5].

The cell wall of a fiber is made up of a number of layers: the so-called primary wall, the first layer deposited during cell development and the secondary wall (S), which again is made up of three layers (S$_1$, S$_2$ and S$_3$). In all lignocellulosic fibers these layers contain cellulose, hemi cellulose and lignin in varying amounts. The individual fibers are bonded together by a lignin-rich region known as the middle lamella. Cellulose attains its highest concentration in the S$_2$ layer (about 50%) and lignin is most concentrated in the middle lamella (about 90%), which, in principle, is free of cellulose. The S$_2$ layer is usually by far the thickest layer and

dominates the properties of the fibers In the S_2 layer the micro fibrils run almost parallel to the fiber axis. With S_2 representing about 50% of the cell wall, this gives the fibers a very high tensile strength. Hemicelluloses are also found in all plant fibers Hemicelluloses are polysaccharides bonded together in relatively short, branching chains They are intimately associated with the cellulose micro fibrils, embedding the cellulose in a matrix Hemicelluloses are very hydrophilic (i e., containing many sites to which water can readily bond) [5].

Lignin is a Latin word for wood. Lignin is the compound that gives rigidity to the plant. Without lignin, plants could not attain great heights (e g , trees) or the rigidity found in some annual crops (e.g., straw). Lignin is a three-dimensional polymer with an amorphous structure and a high molecular weight Of the three main constituents in fibers, it is expected that lignin would be the one with least affinity for water. Another important feature of lignin is that it is thermoplastic (i.e., at temperatures around 90°C it starts to soften and at temperatures around 170°C it starts to flow) [5].

The combined effect of the three main constituents results in properties that are unique for plant fibers. The most important are:

1. Very good strength properties, especially tensile strength In relation to its weight the best bast fibers attain strength similar to that of Kevlar.

2 Very good heat, sound and electrical insulating properties.

3 From a waste point of view, combustibility is an advantage. Products can be disposed of through burning at the end of their useful service lives and energy can simultaneously be generated

4. Because of their tendency to absorb water, natural fibers is biodegradable under certain circumstances through the actions of fungi and/or bacteria.

5. Because of the hygroscopicity of the fibers, products and materials based on plant fibers are not dimensionally stable under changing moisture conditions. This is the greatest disadvantage in relation to industrial use of plant fibers However, if necessary, this may be controlled at an extra cost by a number of known treatments (e.g., heat treatments or chemical modification procedures such as acetylation):

6 The hydroxyl groups present in the cell wall constituents not only provide sites for water absorption but also are available for chemical modification (e.g., to introduce dimensional stability, durability, or improved oil/heavy metal absorption properties).

In each plant, the rings of fiber cell bundles form a tubular mesh that encases the entire stem

from top to bottom. Two layers can usually be distinguished, connected together by lateral fiber bundles, so that the whole sheath is really a lattice in three dimensions cell bundles form the links of the mesh, but each link extends only for a few centimeters before it divides or joins up with another link. After extraction from the plant, the fiber sheath forms a flat ribbon in three dimensions [5].
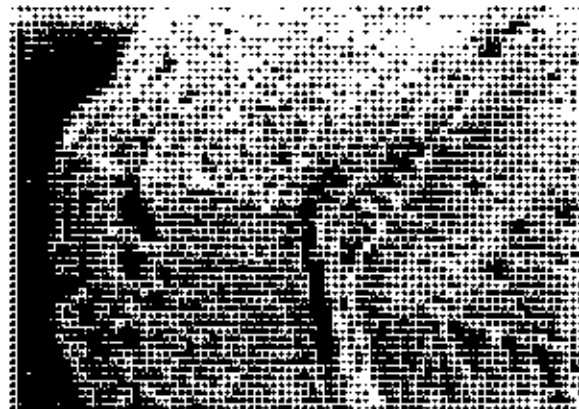
Fig 2.1: Jute Stem (Combined transverse section and longitudinal section).
Magnification x 70. [5].

Figure 2.1 shows the transverse and longitudinal view of jute stem. It is clearly understood that when a transverse section of a jute stem is examined under the microscope, the cell structure is seen clearly. Each cell is roughly polygonal in shape, with a central hole, or lumen, comprising about 10% of the cell area of cross section. The microscopic examination of the longitudinal view of the fiber shows the overlapping of the cells along the length of the fiber. The cells are firmly attached to one another laterally, and the regions at the interface of two cells are termed the middle lamella. Separation of cells can be effected by chemical means, and they are then seen to be threadlike bodies ranging from 0.75 to 5 mm in length, and referred to as ultimate cells. A single fiber thus comprises a bundle of ultimate cells. In the figure 2.1 transverse sections of jute stem show that the number of ultimate cells in a bundle ranges from a minimum of 8 or 9 to a maximum of 20-25. Bundles containing up to 50 ultimate cells are sometimes reported, but in such cases it is questionable whether the fiber is truly single in the botanical sense or is comprised of two fibers adhering together. A minimum number of cells in the cross section are evidently necessary to provide a coherent and continuous overlapping structure. The cells are some 200 times longer than their width. The ultimate cell dimensions of kenaf and many other fiber-bearing dicotyledons are similar to those of jute. A distinction has been made between jute like fibers and flax [4]

2.8 CHEMICAL MODIFICATION OF CELLULOSE FIBERS

When it comes to using natural fibers as reinforcement in composite materials many problems occur at the interface due to imperfect bonding. Therefore modification of the fibers by chemical treatment is the one large area of research. The modification of the fibers can both increase or decrease the strength of the fibers, and thus an understanding of what occurs structurally is of paramount importance. Chemical modification of cellulose fibers is usually applied to correct for deficiencies of the fibers. Modification may result in improved performance of the composites produced. This can be done through several approaches, including acetylation, cyanoethylation, woolenization, bleaching and plasma activation graft polymerization with vinyl monomers, which are very well described in the literature. These, however, will increase the fiber cost [2, 13].

The major drawback of using cellulose fibers as reinforcing agent is the high moisture absorption of the fibers due to hydrogen bonding of water molecules to the hydroxyl groups within the fiber cell wall. This leads to a moisture build-up in the fiber cell wall (fiber swelling) and in the fiber-matrix interface. This is responsible for changes in the dimensions of cellulose-based composites, particularly in the thickness and the linear expansion due to reversible and irreversible swelling of the composites. Consequently, the fiber-matrix adhesion is weak and the dimensional stability of cellulose-based composites particularly for outdoor applications will be greatly affected. Researcher investigated the effect of weathering on jute-polyester reinforced composites & found decreases in composite properties because of weathering [2, 5]. Adsorption and desorption of moisture by the composites during the study produced high shrinkage stresses which caused surface crazing and debonding of resin and jute fibers. This led to a reduction in mechanical properties of the composites studied [2].

Figure 2.2 shows the hydrogen bonding system in cellulose i.e. the main constituent of the natural fiber. The thinner lines means the in-plan, the thicker line denotes above plan and the dashed line signifies the position of atoms of cellulose molecule. Two types of hydrogen bonding have been identified in cellulose namely intermolecular and intramolecular type hydrogen bonding. These differences in hydrogen bonding mean that surface chains have some freedom to move. The lack of intramolecular hydrogen bonding in the surface chains also means that they can form more hydrogen bonds to water or adjacent polysaccharides. It is an anomaly that cellulose α and β, with more intramolecular hydrogen bonding, cannot

form as many hydrogen bonds from chain to chain as the surface form of cellulose, yet the chains within a crystalline unit are held together with spectacular tenacity [5, 14].
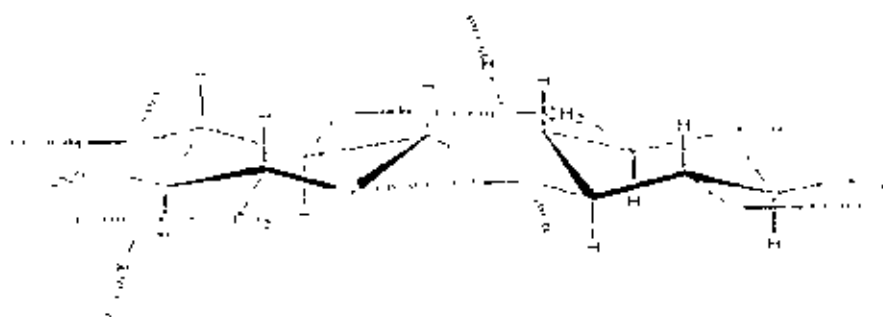

Fig 2.2 : Hydrogen bonding of cellulose [5].

Cellulose micro fibrils contain two crystalline forms, cellulose α and β, in which the chains are packed slightly different. The chain conformation in both forms is similar, a flat ribbon with an 180° twist between successive glucosyl residues This chain conformation is established by two hydrogen bonds parallel to the glycosidic linkage α and β, forms the core of each crystalline unit, in the micro fibrils from higher plants,but at the surface there are chains that do not conform to either of these crystalline allmorphs [5, 14].

The inherent polar and hydrophilic nature of the jute and kenaf fibers and the nonpolar characteristics of the polyolefin's lead to difficulties in compounding and result in inefficient composites. Proper selection of additives is necessary to improve the interaction and adhesion between the fiber and matrix phases The natural fiber imparts lower durability and lower strength compared to artificial fibers. However, low specific gravity results in a higher specific strength and stiffness than glass. This is a benefit especially in parts designed for bending stiffness. In addition, the natural fibers offer good thermal and acoustic insulation properties along with ease in processing technique without wearing of tool. The primary drawback of using cellulose fibers is their limited thermal stability with noticeable degradation occurring as the melt processing temperature approaches 200°C. This excludes some manufacturing processes and limits the type of thermoplastic that can be used to such low-temperature polymers as polypropylene, polystyrene and polyethylene. Higher processing temperatures that reduce melt viscosity and facilitate good mixing, however, are possible, but only for short periods. If degradation occurs, cellulose fibers can be responsible for the formation of tar-like products and pyrolysis acids that may have various damaging effects both on the processing equipment and the composite properties [5]

The equilibrium moisture held by jute when exposed to atmospheres of different relative humidity (RH) shows appreciable hysteresis according to whether absorption from low humidity or resorption from high humidity is concerned. Thus, at 65% RH and 14.6% for resorption of wet fiber, whereas exposure to 100% RH gives an equilibrium regain of 34-35%. These are average values, and different samples of fiber may show minor differences. It will be noted that, at 65% RH, the equilibrium regain of jute is about 6% higher than that of cotton. Jute swells in water to the extent of about 22%, a value similar to that of cotton, despite the greater proportion of noncrystalline material in jute. Delignification has a pronounced effect, and it is reported that when the lignin content has been reduced to 0.78% the swelling may reach almost 40%. Apart from swelling, delignification also affects the equilibrium regain of jute fiber. Other than dimensional stability, the hygroscopic and hydrophilic nature of cellulose fibers also influences the composites process ability. The tendency of cellulose fibers to absorb moisture causes off gassing (void formation) during compounding. This results in a molded article with a microstructure having variable porosity and resembling that of high-density foam. The pores formed will act as stress concentration points which then lead to an early failure of the composite during loading. Agro-based fibers change dimensions with changing moisture content because the cell wall polymers contain hydroxyl and other oxygen-containing groups that attract moisture through hydrogen bonding. The hemicell uloses are mainly responsible for moisture absorption, but the accessible cellulose, noncrystalline cellulose, lignin, and surface of crystalline cellulose also play major roles. Moisture swells the cell wall, and the fiber expands until the cell wall is saturated with water. Beyond this saturation point, moisture exists as free water in the void structure and does not contribute to further expansion. This process is reversible, and the fiber shrinks as it loses moisture [5].

2.8.1 Acetylation

One simple method that can be used to overcome the water absorption problem of jute fiber involves acetylation. The effect of acetylation on jute fibers at different reaction times and reaction temperatures imparts different surface characteristics. The extent of moisture regain and thermal stability of fibers can be improved by this treatment. It was observed that the thermal stability of acetylated jute is higher than that of untreated jute. Other techniques that have been investigated have been alkalization and acetylation. There is a considerable amount of debate in the literature on the benefits of treatment of plant fibers by alkalization and acetylation [5].

By far, the most research has been done on the reaction of acetic anhydride with cell wall polymer hydroxyl groups to give an acetylated fiber Both jute and kenaf have been reacted with acetic anhydride. Without a strong catalyst, acetylation using acetic anhydride alone levels off at approximately 20-weight percent gain (WPG). It was observed that the rate and extent of moisture swelling are greatly reduced because of acetylation [5]. The MAHgPP (maleic anhydride grafted polypropylene) coupling agent is able to act as a compatibiliser for polar natural fiber and nonpolar polymer matrix systems. A hypothetical model of the interface between MAHgPP with hydroxyl groups of jute fiber is shown in Fig 2.3 [15].
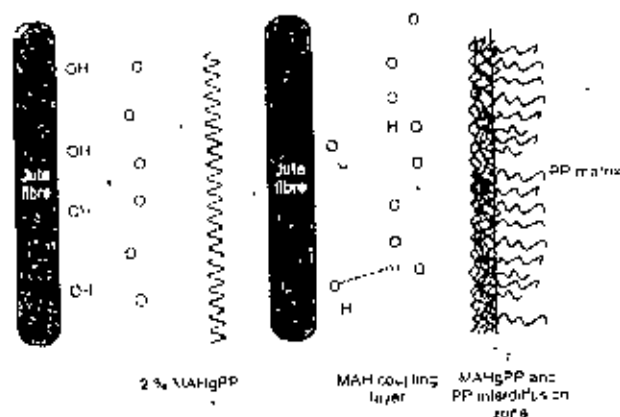

Fig 2.3 Hypothetical Structure of MAH g PP coupling agent and jute fiber at the interface [15]

The strong interfacial adhesion between the fiber and MAHgPP treated PP matrix can be understood from this model, in which likely both chemical (ester bond) and physical interactions (hydrogen bond) should be formed between the cellulose fiber and coupling agent The PP chain of MAHgPP diffuses into the PP matrix involved interchain entanglements. On the other hand, the maleic anhydrate group forms both covelent and hydrogen bonds with the hydroxyl group of the fiber. These cause better adhesion between the fiber and the matrix Therefore the transfer of stress from the matrix to the fibers is improved and lead to higher tensile strengths [15].

Chemically modified jute and kenaf cloth have been tested against brown-, white, and soft-rot fungi and tunneling bacteria in a fungal cellar. It was observed that the cloth was destroyed in less than 6 months, while cloth made from acetylated fiber above 16 WPG showed no attack after 1 year [5]

Acetylation results in a small decrease in values in modulus of elasticity, and tensile strength. The small decrease in some strength properties resulting from acetylation may be attributed to the hydrophobic nature of the acetylated fiber, which may not allow the water-soluble phenolic or isocyanate resins to penetrate into the fiber [5]

2.8.2 Grafting

Jute fiber/fabrics can be modified chemically through graft co-polymerization and through incorporation of different resin systems by different approaches. Polymeric coatings of jute fiber with phenol-formaldehyde or resorcinol formaldehyde resins by different approaches are highly effective in enhancing the reinforcing character of jute fiber, giving as high as 20-40% improvements in flexural strength and 40-60% improvements in flexural modulus. These modifications improve the matrix-matrix resin wettability and lead to improve bonding. Jute can be graft copolymerised with vinyl monomers such as methyl methacrylate, ethyl acrylate, styrene, vinyl acetate, acrylonitrile and acrylamide in the presence of different redox initiator systems such as vanadium - cyclohexanol, vanadium cyclohexanone etc. Grafting of polyacrylonitrile (10-25%) imparts 10-30% improvements in flexural strength and flexural modulus of the composites Grafting of poly methyl methacrylate is also effective in this respect, though to a lower degree [13].

Pedro Herera-Franco and Gonzalo Escamilla, CICY, Mexico has found that the compatibility can be improved by grafting a matrix-compatible polymer onto the fiber surface [Pedro Herera-Franco, 2002]. Initiation by free radicals is one of the most common methods used for the grafting of vinylic monomers onto cellulose. These free radicals are produced as a result of a reaction of the cellulosic chain in a redox system. In this reaction, oxidation of the anhydroglucose units occurs along the cellulosic chains and macro cellulosic radicals are generated on the surface of the fiber. These reactions modify the properties of the fiber, and since one of the roles of the cellulose fibers in composites is to give stiffness and strength to the polymeric matrix, the mechanical properties of henequen *(Agave fourcoydes)* cellulose fibers grafted with methyl methacrylate (MMA) has been examined by this group. They also used poly methyl methacrylate PMMA for grafting. The effect of reactant concentration along with the change in surface properties and morphology of fiber was observed by the treatment [13]

2.8.3 Alkalization

When jute fiber is treated with strong alkali, profound changes occur in its physical structure. Lateral swelling occurs, together with considerable shrinkage in length, as a result of which the fiber is softened to the touch and develops a high degree of crimp or waviness. The crimp gives wool like appearance to the fiber, and much attention has been given to assessing the commercial possibilities for this chemical modification. On stretching the fibers to break, the crimp is straightened and thereby the extensibility of the fiber is increased. The effect is small at alkali concentrations of up to about 10%, but the extensibility increases rapidly at concentrations of 16% and upward and may reach 8 or 9%. At the same time, however, the tensile strength of the fiber decreases with an increased alkali concentration, but it is interesting to note that the product of extensibility and tensile strength, the breaking energy appears to pass through a maximum at 16-20% concentration [2, 5, 16].

Note that mercerization process is familiar to the textile scientist where the fiber is chemically treated to increase its luster and to increase the affinity towards dyeing [G Canch'E-Escamilla, 2002] P. Bel-Berger has discussed the nature of the chemical changes occurring in jute on mercerization [17].

The crimp statistics of fiber is essential to understand the fiber mechanical property Two parameters are measured to define the crimp, namely the e-root mean square or eRMS value of the width (D) and the number of crimps per unit length of the stretched fiber (n). As the crimp is three-dimensional, the fiber is rotated during the measurements. Typical values for jute fibers immersed in variable NaOH concentration, time variation and temperature effects are reported to influence the values of (D) and (n). Examination of the tensile properties of jute fibers before and after alkali treatment and has shown that the tenacity decreases with increasing the alkali concentration, an initial value of 3 6 g/den falling to 2.5 g/den at 9% alkali and to 1.5 g/den at 24% alkali (at 2°C and 1 hr immersion). The extension at break, referring to the fiber length before immersion, increases from 1.2% without alkali treatment to 3.6% at 9% alkali, and then falls slightly to 2.4% at 24% alkali [5]

The tenacity changes are no doubt linked with the losses in weight that occur with alkali treatment, but there may be more profound changes taking place internally within the ultimate cells. Such changes are at present imperfectly understood, but if useful commercial developments are to be made, further investigation of structural changes appears essential.

Moreover, the crimp is a "once-only" effect; to be really useful; some small degree of elasticity must be introduced into the fiber. That is to say, the woolenizing treatment does not confer elasticity on the fiber [11].

Alkalization of plant fibers changes the surface topography of the fibers and their crystallographic structure. However, care must be exercised in selecting the concentration of caustic soda for alkalization, as results show that some fibers at high NaOH concentrations have reduced thermal resistance. It is believed that the increase in the crystallinity index measured by Wide Angle X-ray Scattering (W AXS) is in fact an increase in the order of the crystallite packing rather than an increase in the intrinsic crystallinity. A high crystallinity index is likely to result in stiff, strong fibers of interest in the formation of plant fiber composites. Fourier Transformation Infrared (FTIR) spectroscopy provided additional information on the reactivity fibers following treatment by alkalization and acetylation. However, Scanning Electron Microscopy (SEM) results indicate that after chemical treatment, at low concentration, all the fibers except kapok possess rougher surfaces [2, 5].

These alkalization effects on natural fibers are of particular importance for fiber matrix adhesion and the creation of high fiber surface area required for the optimization of fiber - resin reinforcement The modification of cellulose fibers, therefore, develops into changes morphology and increase in hydroxyl groups These changes will effectively result in improved surface tension, wetting ability, swelling, adhesion and compatibility with polymeric materials [2, 5].

2.8.4 Miscellaneous Chemical Treatment

Jute can be made to react with acrylonitrile in the presence of alkali under conditions that do not reduce the tensile strength of the fibers to any important extent. The properties of cyanoethylated cotton have been known for some time, and this particular chemical modification is claimed to provide increased stability against degradation by acids and heat [5, 13].

Experiments with jute yarn at the British Jute Trade Research Association, have shown that, untreated yarn subjected to hydrolysis with 0.2 N H_2SO_4 acid at 100° C for 60 min retained 20% of its initial strength, a yarn cyanoethylated to 4.6% nitrogen content retained 80% of its strength under similar conditions [5].

Jute yarns cyanoethylated to different extents showed increasing resistance to degradation by heating; whereas untreated yarn heated at 160°C for 24 hr retained only 55% of its initial strength, similar yarn with a nitrogen content of 4.9% retained 90%. Resistance to rotting was also examined by incubation of yarns under degrading conditions, which caused complete breakdown of strength after 2 weeks. Cyanoethylation up to 1.5% nitrogen resulted in little improvement, but for 2.8% nitrogen and over, even 16 weeks of incubation reduced strength by only 10%. By comparison, yarns undergoing copper naphthenate treatment with 1.2% copper retained only 30% of strength under similar conditions of exposure. If the nitrogen content approaches about 3%, cyanoethylation gives most effective protection against rotting, just as is said to be the case for cotton [5, 13].

Jute is chemically treated with isopropyl triisostearoyl titanate (abbreviated as titanate), g - aminopropyl trimethoxy silane (abbreviated as silane), sebacoyl chloride (SC), and toluene diisocynate (TDI). All these reagents are expected to block the hydroxy groups of jute thus making the fibers more hydrophobic. These surface modifiers penetrate and deposit into lumens of cell wall of fiber minimizing the possible extent of moisture ingress [5, 13].

N. E. Zafeiropoulos explained the effect of acetylation and stearic acid treatments upon the fiber strength [13]. Stearic acid treatment does not affect significantly the fiber strength for either green and dew retted flax at low reaction times. However, at higher reaction times there was a significant deterioration of the fiber strength for both types of flax. The effect of these treatments on the interface has been assessed using the single fiber fragmentation test [5].

2.9 POLYPROPYLENE

2.9.1 Introduction

Since its introduction in the mid-1950's, polypropylene has established itself in many engineering applications through its combination of strength, rigidity, temperature resistance, chemical inertness, low price and ease of fabrication.

Polypropylene is a member of the polyolefine group of polymers. It is related structurally to polythene and is made by polymerizing propylene, the next member in the olefine series after ethylene. Until the early 1950's all attempts to polymerize propylene into solid, useful

thermoplastics had failed, only syrupy liquids being obtained, but in 1953 Professor K. Ziegler in Mülheim discovered that certain combinations of metal alkyl complexes and transition metal halides catalyse the polymerization of ethylene so that the reaction can be carried out at temperatures and pressures a little above atmospheric. Previously, polymers of ethylene had been prepared only by using high temperatures and very high pressures. Ziegler's work provided the first example of a new type of polymerization reaction. The following year Professor G. Natta of Milan, working in collaboration with the Montecatini Chemical Company, prepared solid, high-molecular weight polymers of propylene using catalysts of the Ziegler type. He further developed these catalysts so that they became stereospecific. These discoveries have led to the industrial production of stereoregular polypropylene, a rigid, easily moulded material with the excellent chemical resistance and electrical insulation properties normally expected of polyolefines. In contrast, polypropylene in a randomly polymerized form is of little commercial interest

2.9.2 Nature of polypropylene

Polypropylene molecules can assume any of the three structures that is atactic, isotactic or syndiotactic. It is doubtful if molecule consisting wholly of any one structure ever exist and molecules of commercial polypropylene are predominantly isotactic, but with short lengths of atactic and syndiotactic material incorporated in them. Two main types of polypropylene are available, homopolymers of propylene, and ethylene/ propylene copolymers in which propylene is the major constituent; the latter may contain various amounts of ethylene and a subdivision into low ethylene (7% by weight) and high ethylene (15 % by weight) copolymers is appropriate. However, this characterization of copolymers must not be regarded as unique because much depends on the manner of incorporation of the ethylene. It should be added that random polymers are not considered here.

2.9.3 Grades and forms

Both homopolymers and copolymers can be obtained in various grades, all of which have virtually the same density, but which differ in melt viscosity and in the degree to which they are stabilized against oxidation; they thus meet the requirements of specific end-uses and processing techniques. Many special grades have also been developed, these include glass-filled and flame-retardant materials and grades to produce articles with reduced dust pick-up and improved weathering performance.

Polypropylene is usually sold as regularly cut granules, but some grades are also offered in the form of free-flowing powders. In its natural form polypropylene is translucent and white, but compounds in a wide range of colours are available, and the natural material itself can easily be coloured by the fabricator if so desired. Semifabricated forms such as film, foil, sheet, block, rod and tube are all produced commercially and are readily available. These are convenient starting materials in the making of prototypes

2.9.4 Processing

Polypropylene is easily processed by conventional techniques. It can be moulded on all modern injection moulding machines with adequate heating capacity and good temperature control, and readily extruded into film, pipe, wire and cable covering, sheet, monofilament and fiber, and tubes for subsequent blowing into bottles and other hollow articles. Compression moulding, although not economic on a commercial scale, can be used to make blocks and sheets for prototype work.

Polypropylene sheet is readily bent and shaped into simple curves when heated, and it can also be vacuum formed. The usual machining techniques for thermoplastics are applicable to polypropylene Jointing techniques such as welding and cementing can also be used, although the strength of cemented joints is not adequate for every application because of the chemical inertness or the material, and whenever possible it is desirable to design articles so that adhesives are not required.

2.9.5 Properties

Some physical properties of polypropylene are given in Table 2.2. As with all crystalline polymers, they depend on the amount of crystallinity; in polypropylene, the proportion of stereoregular polymer represents the amount of potentially crystallizable material, and it thus determines the softening temperature, stiffness, hardness and tensile strength; increasing the amount of amorphous atactic material reduces the total crystallinity and hence leads to a reduction in value for all these properties. However, the actual crystallinity of a finished article depends also on the rate at which the molten material is cooled and solidified. On crystallization the crystallites tend to arrange themselves in ordered aggregates known as spherulites. On slow cooling from the melt few crystal nuclei are formed, and this results in a small number of large spherulites. On fast cooling (quenching) the rate of nuclei formation is

very much higher and a large number of small spherulites are formed. Slow cooling produces a higher overall crystallinity. This dependence of crystallinity and texture on cooling rate must be *borne* in mind during processing, in general, slowly cooled articles are rather more rigid and temperature resistant than those, which are rapidly quenched, but these properties are obtained at the expense of a slight decrease in transparency and toughness.

Table 2.2: Properties of polepropylene

Property	Units	Typical Value
Density	g/cm^3	0.90-0 91
Crystalline melting point	°C	165-175
Coefficient of linear thermal expansion		
-20 to 20°C	°C^{-1}	9x 10^{-5}
	°F^{-1}	5x 10^{-5}
20 to 80°C	°C^{-1}	1.35x 10^{-4}
	°F^{-1}	0.75x 10^{-4}
Thermal conductivity, 20 to 150°C	cal /cm s °C	3 5 to 5x 10^{-4}
	B ThU in/ft^2 h °F	1.0 to 1.45
Specific heat, 20 to 80°C	cal /g °C	0.46
	B ThU/lb°F	0.46
Flammability		burns slowly with drips

A further variable which affects the physical properties of polypropylene and its flow properties at high temperatures is the molecular weight. As the molecular weight increases, the impact strength increases. Molecular weight also has a marginal effect upon stiffness and hardness because crystallinity, which determines stiffness, is slightly dependent upon molecular weight. Low molecular weight tends to be associated with higher crystallinity, other things being equal. The average molecular weight of polypropylene is not normally measured and in practice polymers are classified by the melt flow index (MFI) at 230°C and 2.16 kg

For ethylene/propylene copolymers the properties are determined by the proportion of ethylene present and by the way in which it is introduced into the chain structure, as well as by the factors already mentioned above.

2.10 POSSIBLE ORIENTATION OF FIBERS IN COMPOSITE

In case of the fiber reinforced polymer the fibers are more or less arranged in a pattern shown in the figure 2.4 below. The figure shows a two-dimensional lay out of the fiber that can be seen from the top, or the figure is some schematic view of fiber-reinforced polymer composite [18].
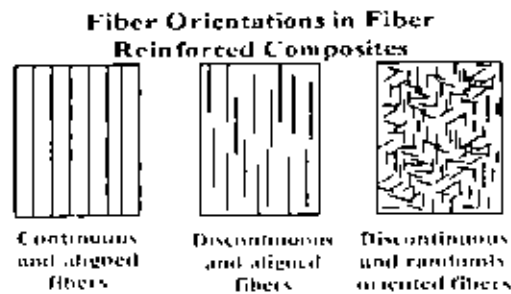

Fig 2 4 Possible fiber orientation in polymer matrix composite

There are other types of polymer composite called laminates, which are more or less made from the chopped strand mat or woven fabric reinforced composite. The chopped strand mat reinforced polymer composite is a complex system of laminates and discontinuous fiber reinforced polymer composite that has anisotropic property of the part There are some possible orientations of laminates is possible. Those are termed as $0°$ laminate, $0 - 90°$ laminate. $0 - 0°$ laminate and cross ply. In between these the $0 - 90°$ laminate and cross ply are fabricated for laminated composite part. Now a day the balk molding compound (BMC) technology and sheet molding composite (SMC) technology has got wide application . In case of BMC the whole part is fabricated from chopped discontinuous fiber reinforced composite. The SMC is random discontinuous fiber reinforced flat panels are fabricated with thixotropic chemicals to resist polymerization for the time being and the entire body is embedded in lamination of woven fabric and then follow any suitable fabrication technique Resin systems such as epoxies and polyesters have limited use for the manufacture of structures on their own, since their mechanical properties are not very high when compared to, for example, most metals However, they have desirable properties, most notably their ability to be easily formed into complex shapes Materials such as glass, aramid and boron have extremely high tensile and compressive strength but in 'solid form' these properties are not readily apparent. This is due to the fact that when stressed, random surface flaws will cause each material to crack and fail well below its theoretical 'breaking point'. To overcome this problem, the material is produced in fiber form, so that, although the same number of

random flaws will occur, they will be restricted to a small number of fibers with the remainder exhibiting the material's theoretical strength. Therefore a bundle of fibers will reflect more accurately the optimum performance of the material. However, fibers alone can only exhibit tensile properties along the fiber's length [18].

When the resin systems are combined with reinforcing fibers such as glass, carbon and aramid exceptional properties can be obtained. The resin matrix spreads the load applied to the composite between each of the individual fibers and protects the fibers from damage caused by abrasion and impact High strengths and stiffness, ease of molding complex shapes, high environmental resistance all coupled with low densities, make the resultant composite superior to metals for many applications [18]

Since polymer matrix composites combine a resin system and reinforcing fibers, the properties of the resulting composite material will combine something of the properties of the resin on its own with that of the fibers on their own, as shown in figure 2.5
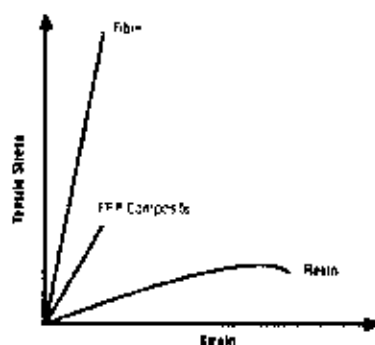
Fig 2.5: Schematic representation of the mechanical properties of fiber, resin matrix and composite [18].

The mechanical properties of most reinforcing fibers are considerably higher than those of un-reinforced resin systems. The mechanical properties of the fiber/resin composite are therefore dominated by the contribution of the fiber to the composite The surface interaction of fiber and resin is controlled by the degree of bonding that exists between the two. This is heavily influenced by the treatment given to the fiber surface, and a description of the different surface treatments and 'finishes' is given here. The manufacturing process used largely governs the amount of fiber in the composite. However, reinforcing fabrics with closely packed fibers will give higher fiber volume fractions in a laminate than will those of

fabrics which are made with coarser fibers, or which have large gaps between the fiber bundles. Fiber diameter is an important factor here with the more expensive smaller diameter fibers providing higher fiber surface areas, spreading the fiber/matrix interfacial loads. Generally, the stiffness and strength of a laminate will increase in proportion to the amount of fiber present. However, above about 60-70% (depending on the way in which the fibers pack together) although tensile stiffness may continue to increase, the laminate's strength will reach a peak and then begin to decrease due to the lack of sufficient resin [18]

Finally, since reinforcing fibers are designed to be loaded along their length, and not across their width, the orientation of the fibers creates highly 'direction-specific' properties in the composite. This 'anisotropic' feature of composites can be used to good advantage in designs, with the majority of fibers being placed along the orientation of the main load paths. This minimizes the amount of parasitic material that is put in orientations where there is little or no load. Overall, the properties of the composite are determined by:

i) The properties of the fiber.
ii) The properties of the resin.
iii) The ratio of fiber to resin in the composite (Fiber Volume Fraction).
iii) The ratio of fiber to resin in the composite (Fiber Volume Fraction).
iv) The geometry and orientation of the fibers in the composite

The first two will be dealt with in more detail later. The ratio of the fiber to resin derives largely from the manufacturing process used to combine resin with fiber, as will be described in the section on manufacturing processes. In general, since the mechanical properties of fibers are much higher than those of resins, the higher the fiber volume fraction the higher will be the mechanical properties of the resultant composite [18]

In practice there are limits to this, since the fibers need to be fully coated in resin to be effective, and there will be optimum packing of the generally circular cross section fibers. In addition, the manufacturing process used to combine fiber with resin leads to varying amounts of imperfections and air inclusions. Typically, with a common hand lay-up process as widely used in the boat-building industry, a limit for fiber volume fraction is approximately 30-40%. With the higher quality, more sophisticated and precise processes used in the aerospace industry, fiber volume fractions approaching 70% can be successfully obtained. The geometry of the fibers in a composite is also important since fibers have their highest mechanical properties along their lengths, rather than across their widths. This leads

to the highly anisotropic properties of composites, where, unlike metals, the mechanical properties of the composite are likely to be very different when tested in different directions. This means that it is very important when considering the use of composites to understand at the design stage, both the magnitude and the direction of the applied loads. These anisotropic properties can be very advantageous since it is only necessary to put material where loads will be applied, and thus redundant material is avoided [18]

It is also important to note that with metals the material supplier largely determines the properties of the materials, and the person who fabricates the materials into a finished structure can do little to change those 'in-built' properties. However, a composite material is formed at the same time, as the structure is itself being fabricated. This means that the person who is making the structure is creating the properties of the resultant composite material, and so the manufacturing processes they use have an unusually critical part to play in determining the performance of the composite [18].

Tensile controlled performance of a fiber reinforced composite material system is primarily dependent on three factors: (1) strength and modulus of the fibers, (2) strength and chemical stability of the matrix, and (3) effectiveness of the bond between matrix and fibers in transferring stress across the interface. Anything in the total system can be a weak link that is the limiting factor in composite mechanical properties. From a mechanics point of view, the discontinuities of mechanical properties between fibers and matrix at the interface are a source of sharp gradients (or possibly discontinuities) in material behavior, which may act as a nucleation mechanism for damage development and as an obstruction to damage growth [19].

Optimum bonding is responsible for maximum static and dynamic mechanical properties and environmental resistance. For example, a stronger interface bond is believed to increase the static strength in general. However, a strong interface bond also yields a brittle composite, whereas a weaker interface results in higher fracture toughness composite. Because of the lack of understanding of bonding mechanisms occurring at the fiber-matrix interface, optimal material combinations for existing fibers, adherents, or polyester matrix materials are difficult to achieve. The lack of ability to a priori select surface and interfacial conditions is due to a lack of understanding of the structure, composition, and mechanical behavior of interfaces in a composite. The situation is also complicated by the variability of the fiber strength. Therefore, all fibers will not be stressed to their ultimate values at the same loading

level. Those fibers that break earlier will cause localized interfacial shear stress and stress concentrations in adjacent fibers. Thus, progressive damage may result [19].

It is clear that, with so many factors to consider and conflicting evidence in the literature, there is no simple means of specifying the optimum nature of the interface to improve the mechanical performance of composites. The stress state at the interface is greatly influenced by external loading conditions, local geometry, the nature of the interface, and the stiffness difference between fiber and matrix. As a result, the effect of the interface can be completely different for different loading conditions and different material systems [16].

For most polymer matrix composites, fibers are much stiffer than the matrix Therefore, fibers carry most of the tensile load, and final fracture of the composite is determined by fiber fracture events. The key element to the tensile failure evaluation is to study the stress concentration, caused by fiber fractures. The non-uniformity of fiber strength will result in a distribution of fiber breaks at different stress levels After each fiber breakage, the local load has to be redistributed, which results in stress concentrations in fibers adjacent to the broken fibers. The stress concentrations will influence the growth of the cracked region to include additional fibers. As the stress level increases, the composite will have groups of broken fibers. Each group has an ineffective length and stress concentrations varying with group size. The stress concentration and ineffective length only depend on the size of the group (number of broken fibers), which are functions of applied stress. High shear stresses cause interfacial failure, which serves to arrest the propagation of the crack by redistributing the local stress [19].

During the surface treatment, the physical and chemical characteristics of the fiber surface changed through oxidative attack at the fiber surface, which fixes oxygen at the edges of the basal planes and removes carbon layers. The result is an increase in the active oxygen content by reaction of polar functional groups such as carboxyl, hydroxyl, and lactone groups. Note that surface treatment of .a fiber can affect the interfacial bond as well as the fiber's tensile strength Extensive surface treatment may attack the fibers and reduce the fiber strength and fiber volume fraction of the composite. This suggests that the decrease of tensile strength for a higher surface treatment level may be partially a result of the decrease of fiber strength from surface treatment [19].

Normally the composite property is mathematically expressed as the following equation

$$X_c = X_f V_f + X_m V_m \dots \dots \dots \dots \dots \dots \dots \dots \dots (2.1)$$

$$V_f + V_m = 1 \quad \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots (2.2)$$

Where $'X'$ is the property of concern, $'V'$ is the volume fraction and subscript "c", "f" and "m" holds for the composite, reinforcing fiber and the matrix.

In general the fiber failure strain is lower than the matrix failure strain. Assuming in general that all fibers have same strength, the tensile rupture of composite will precipitate a tensile rupture in the composite Although the composites were fabricated with random and discontinuous chopped jute fiber but an assumption may be made that the composite behavior is isotropic Thus equation can be written for the tensile strength prediction of the composite specimen.

$$\sigma_c = \sigma_f V_f + \sigma_m V_m \quad \dots \dots \dots \dots \dots \dots \dots \dots (2.3)$$

Where the σ holds for the stress or simply the strength of composite fiber and the matrix and subscript "c", "f" and "m" holds for the reinforcing fiber and the matrix.

Thus using 3 equations the longitudinal tensile strength of the composite body can be estimated as

$$\sigma_{Ltu} = \sigma_{fu} V_f + \sigma_{m'} (1 - V_f) \dots \dots \dots \dots \dots \dots \dots \dots (2.4)$$

Where σ_{Ltu} is the longitudinal tensile strength of composite specimen, $\sigma_{f\,u}$ is the fiber ultimate tensile strength and $\sigma_{m'}$ is the matrix stress at fiber failure strain.

For effective reinforcement of the matrix, the fiber volume fraction in the composite must be greater than a critical value, i.e.

$$V_{fc} = (\sigma_{Ltu} - \sigma_{m'})/(\sigma_{fu} . \sigma_{m'}) \quad \dots \dots \dots \dots \dots \dots \dots (2.5)$$

2.12 TREATMENT, CHARACTERIZATION AND MECHANICAL PROPERTIES OF COMPOSITE

N. E. Zafeiropoulos 2002 focused on the flax /polyolefin composite properties on acetylation and stearation on flax fiber (green and dew retted) and the results were discussed in terms of process variables, such as temperature, time of reaction, reactant concentration and recycling. He showed that surface treatments, although has negative impact on economics, are potentially able to overcome the problem of incompatibility between the hydrophilic natural fiber to that of the hydrophobic polymer matrix. He applied three characterization techniques viz X - ray diffraction, scanning electron microscopy and inverse gas chromatography for the treated and untreated fiber. It was found that both treatment results in removal of non - crystalline constituents of the fiber, and alter the characteristic of the surface topography. Change in free energy of the fiber surface was also noticed; with acetylation increasing it and stearation decreasing it. He also reported various other treatments; corona treatment, plasma treatment, maceration, heat treatment, graft polymerization, silane treatment and treatment with many other chemicals and showed that these past used methods posses many disadvantages e g expensive equipments and chemicals.

N E Zafeiropoulos [20] in his work also showed the effect of these treatments upon the interface of flax fiber/polypropylene composite and is assessed by means of single fiber fragmentation test He found that both the treatment led to improvement of stress transfer efficiency at the interface and both treatments were optimized. Keeping in mind that the key issue to develop a mathematical model all of his approaches was to formulate the key parameter the so-called 'interfacial shear strength', which has been linked directly with the interfacial bond.

Seena Joseph [21] fabricated composite with banana fiber and glass fiber with varying fiber length and fiber loading, using the phenol formaldehyde resole matrix Prepreg route was followed for the preparation of composite Hand lay-up method followed by compression molding was adopted for composite fabrication. Analysis of tensile, flexural and impact properties of these two FRP composite showed that the optimum length of fiber required for banana fiber and glass fiber are different in the matrix. Both banana fiber and glass fiber showed an increase in properties with fiber loading. Interfacial shear strength obtained from single fiber pull out test and revealed that mechanical interlocking is a more predominant

phenomenon in case of banana fiber rather than glass fiber. SEM studies evaluated the fiber matrix interaction. Comparison with the experimental and theoretical data explains the strength and stiffness facts of composite.

G. Canche - Escamilla [22] investigated the mechanical behavior of rigid thermoplastic matrix - cellulose fiber composite Investigation concluded that these materials exhibit a brittle behavior, i.e. they posses a high modulus and deforms very little before fracture. Such behavior makes the characterization of interfacial properties difficult to evaluate. The composite materials were prepared by mixing the polymeric matrix and the cellulose at 180°C in a blender torque rheometer and then molded in a carver laboratory press using a force of 44.49 N. The flexural, impact and compressive properties were investigated utilizing the polymethyl methacrylate (PMMA) and poly (styrene-co-acrylonitrile) (SAN) and the effect of the fiber surface treatment on the composite were studied. The flexural strength of the composite remains constant when the fiber is grafted with (PMMA) and a brittle interface around the fiber formed regardless of the fiber content. Study of poly (butyl-acrylate) (PBA) grafted fiber reinforced composite did not resemble the property of the former, but showed low flexural strength at higher fiber content. In case of impact loading the presence of elastomeric type material PBAA grafted cellulose fiber seem to provide an alternative method of energy dissipation in the composite, thus, showing a better impact behavior then the composite with other surface treatment. The impact behavior seems to be improved by the mechanical properties of the cellulose fiber. The low elastic modulus of cellulose fiber results from the attack of grafting process, which affected the rigidity of the composite. The research group tried the compression testing of these composite materials using two geometries; first one is rectangular shaped and other is hourglass shaped. They found that the glassy PMMA and SAN matrix able to yield even at room temperature. The fiber matrix interfacial properties were assessed by SEM studies.

Dipa Ray et al [16] studied treatment of jute fiber with 5% NaOH for 0, 2, 4, 6 and 8 hour at 30°C and showed that the crystallinity in the jute fibers was improved along with the higher modulus. The tenacity was also increased. Mechanical testing of these composite showed that the fiber pull out mechanism is predominant mode of failure for 0 - 4 hour treated one and transverse fracture with minimum pull out is seen in 6 - 8 hour treated composite.

Dipa Ray et al [23] worked on the Vinyl ester - resin - matrix with untreated and 5% NaOH treated jute fibers for 4 and 8 hours with different fiber loading from 23 - 30 volume% and

were subjected to dynamic mechanical and thermal analysis to, determine their dynamic properties as a function of temperature and reported that the dynamic mechanical and thermal properties are dependent on fiber content, presence of additives like filler, compatibiliser, and impact modifier, fiber orientation and the mode of testing. Jute/vinyl ester composites containing raw jute and alkali treated fibers were fabricated in the form of cylindrical rod of 6mm diameter using hollow glass tube and jute were preheated to 100°C for 4 hours prior to use.

Jochen Gassan [24] studied the tension and fatigue behavior of different natural fiber reinforced plastic. The composite were made of flax and jute yarns and woven as reinforcements for epoxy resin, polyester resin and polypropylene. Study inferred that fiber type, textile architecture, interphase properties, fiber properties and content were found to affect the fatigue behavior strongly; the results were illustrated with the damping vs. applied maximum load curves. It was found that natural fiber reinforced plastic with higher fiber strength and modulus, stronger fiber - matrix adhesion or higher fiber fraction posses higher critical loads for damage initiation and higher failure loads. In addition damage propagation rate were reduced. A finding about unidirectional composite shows less sensitivity to fatigue induced damage than woven reinforced ones.

J. Gassan [25] also investigated the influence of fiber matrix adhesion in jute fiber reinforced polypropylene composite behavior under fatigue and impact loading. It was shown that a strong interface is connected with a higher dynamic modulus and reduction in stiffness degradation with increasing load cycles and applied maximum stress. The specific damping capacity resulted in higher values for the composite with poor bonded fiber. Stronger fiber - matrix adhesion reduced the loss energy by non - penetration impact tested composite. Test performed at different temperature showed bigger loss of energy for cold and warm conditions compared to room temperature. Post - impact dynamic modulus was observed to have dependency on the poor and good fiber matrix adhesion.

Dash et al [26] studied the weathering and thermal behavior of jute - polyester composite that were fabricated with untreated and bleached slivers with 60% loading of fiber by weight. Both types of composites were subjected to water absorption & outdoor weathering tests to asses their relative performance under environmental conditions. Bleached slivers reinforced composite showed less water absorption than that of the untreated fiber reinforced composite. The mechanical properties like

tensile and flexural were measured for both the weathered and unweathered specimen A comparison between the weathered and unweathered specimen showed that the decrease in tensile strength along with increase in tensile modulus. Flexural and internal laminar shear strength was decreased after weathering. The study tried to establish relation between the fibers - matrix adhesion. Thermal analysis (TG/DTG and DSC) was also carried out to explain the thermal stability of these composite and bleached slivers reinforced composite showed less good thermal stability than that of the untreated fiber reinforced composite.

F. R Cichocki Jr. [27] focused his work on the structure property relationship of the composite system. Research indicates that natural fibers exhibit complicated anisotropy structure. In this study, dynamic mechanical and thermal mechanical techniques have been employed to measure the elastic and thermal expansion characteristics of a model composite system containing jute fiber over a broad temperature range. The results of these measurements were in turn incorporated into a number of pertinent micro mechanical models to estimate the thermoplastic properties of natural fiber. The jute fibers investigated in this study exhibited considerable elastic and thermal expansion anisotropy. The longitudinal Young's modulus has been determined to exceed the transverse fiber modulus by much as an order of magnitude in certain temperature regimes. The fiber exhibit negative thermal expansion coefficients along there length; however, large positive thermal expansion coefficients, similar in magnitude to many polymeric materials, have been explained in this research.

D Kugler [28] developed a compression test fixture and analysis method for composite ring, since; low compressive strength in composite cylinders has been an ongoing problem in several arenas. High-density polyethylene (HDPE) matrix was used to fabricate composite rings. The fixture loads the ring in hydrostatic compression by taking advantage of the Poisson's effect and an axially loaded complaint ring high density polyethylene on the outside of the complaint ring.

Analysis is presented to calculate the stress and strain in the composite. O.A. Khondker [29] reported about tensile and flexural properties of jute fiber-based thermosetting composites using woven and flat-braided jute fabrics. Composites were manufactured by hand lamination method. Tensile and flexural properties were investigated to study their dependence upon the surface modifications due to bleaching and coating treatments on the

fiber materials. Test samples were polished and pre- and post-failure examinations were carried out using optical and scanning electron microscopy in order to analyze the test results and their relationships to the state of resin impregnation into the fiber bundles, fiber/matrix interfacial interactions and the fracture and failure mechanisms. Full bleaching treatment on the woven jute fabrics clearly improved mechanical performance of the resultant composites by removing surface impurities and enhancing hydrophobicity of the cellulosic fiber surface, as compared to natural and half bleached specimens. While tensile properties of flat-braided jute composites slightly degraded due to coating treatments, flexural properties showed clear improvements. The changes in the mechanical properties were broadly related to the accompanying modifications and to the state of micro structural imperfections, viz. fiber/matrix interfacial adhesion, severity of resin matrix shrinkage during curing process, fiber/matrix debonding and distribution of disbonds within the matrix region, and also to the relative fiber filament density along the loading axis, in the cured composite structure.

Mubarak A Khan [30] reported the interfacial bond strength can be optimized only when the relationship between the level of fiber-matrix adhesion and the mechanical and fracture behavior of composite clearly understood. This study establishes the relationship between jute fiber-vinyl ester interfacial bond strength in both 0° (longitudinal) and 90° (transverse) directions of the composites. Thermodynamic properties such storage modulus, loss modulus and tanδ of the composite were performed in 0° (longitudinal) direction. To improve these properties the surface of jute yarns were modified with 2-hydroxyethyl methacrylate (HEMA). Treated jute yarns were characterized by FTIR, ESEM and XPS. FTIR and XPS spectra showed that the deposition of HEMA in the jute surface. Rough surfaces of treated yarns were observed by ESEM. Improved mechanical properties such as shear strength (12 MPa), tensile strength (130 MPa) bending strength (225 MPa) were observed as a result of surface treatment. The enhanced storage modulus 10.5 GPa of HEMA treated jute composites was obtained which is 300% and 200% higher than that of vinyl ester and composite of untreated yarn. The tanδ values of composites were found minimum with compared to pure resin. The tensile fracture surfaces were investigated by ESEM. The results of ESEM showed that better interfacial adhesion with treated jute composite compare to untreated one.

K. L Pickering et al [31] investigated the effect of a maleated polypropylene- (MAPP) coupling agent on the mechanical properties of composites based pine *(Pinus' Radiata-* a softwood and fast growing plantation timber) fiber prepared with a polypropylene matrix by

using a twin-screw extruder followed by injection moulding. An increase of more than 100% for strength and 302% for Young's modulus were obtained by using 60 (wt) % fiber and 2 (wt) % MAPP, compared to the unreinforced matrix. Fiber treatment with NaOH reduced the strength but improved Young's modulus. No significant improvement in strength was observed by using benzoyl peroxide (BP). Young's modulus of the composite was increased up to a point with the addition to BP, after which a decrease was observed

K. L. Pickering et al [31] in another paper stated single fiber tests on four different types of natural fiber. These included three different types of wood, as well as hemp. Tensile Strengths (not taking the lumen into account) ranged from 970 MPa for hemp down to 196 MPa for the thinning wood fibre. Strengths were then recalculated based on true cross-sectional areas obtained using scanning electron micrographs (SEM) The strength value for hemp was unaffected as it was found to have a solid section, but the apparent values for the wood fibers were approximately doubled The effect of malefic anhydride (MA)-grafted polypropylene (MAPP) coupling agent on the strength and Young's Modulus of wood and hemp fiber reinforced composites were also assessed The increases in tensile strength and Young's modulus for hemp compared to pure polypropylene were 76% and 171% respectively and were 11 % stronger than wood fiber composites. Slab wood composites had the highest tensile strength and Young's modulus value among the wood fiber composites.

K. L. Pickering et al [31] also showed that hemp fiber reinforced thermoplastic composite materials are strong, stiff, lightweight and recyclable, but possess mechanical properties well below their potential values. The purpose of this research is to produce an improved hemp-fiber reinforced polypropylene composite by optimizing the fiber strength, fiber processing, composite processing and fiber-matrix interfacial bonding. An investigation was performed on a crop of New Zealand grown hemp to determine the effect of plant growth duration on fibre strength. By conducting single fibre tensile tests on the hemp fibres, it was discovered that the optimum growing period was 114 days, producing fibres with an average tensile strength of 857 MPa. An investigation was then conducted to determine a suitable fiber treatment method to separate the fibers from their fiber bundles and to remove lignin and pectin, whilst retaining the fiber strength. An alkali treatment consisting of digesting the fibers in a 10% NaOH solution at a maximum temperature of 160°C and a hold time of 45 minutes was found to produce strong fibers with low lignin content and good fiber separation. Finally, the alkali treated fibers, polypropylene and a maleated polypropylene (MAPP) coupling agent were compounded in a twin-screw extruder, and injection molded

into composite tensile test specimens. The strongest composite produced consisted of polypropylene with 40% wt fiber and 2% wt MAPP, and had a tensile strength of 38 MPa, and a stiffness of 4 GPa.

Mubarak A. Khan [32] in another work explained that due to biodegradable and ecofriendly behaviors, natural fibers like jute, sisal etc. have taken the central stage in the present day research in the field of polymeric science and technology. Jute yarns were grafted with acrylic monomers like acrylamide (AM) and [3-(trimethoxysilyl) propylmethacrylate] (silane) by using UV radiation to improve the mechanical properties. The surfaces of the treated and untreated jute were characterized by X-ray photo-spectrometer (XPS), and FTIR. In order to further improvement, jute yarns were pretreated under UV radiation at different radiation intensities and were grafted with 30% silane and 30% AM under *in situ* UV radiation at optimized condition. Though the different intensities of UV pretreated jute yarns were grafted with both monomers but silane treated (silanized) jute yarn showed better enhancement of mechanical properties at 25 UV passes. Water uptake and degradation properties of untreated and treated jute yarns were studied in various conditions such as simulated weathering and in soil containing 25% moisture. The UV pretreated silanized jute yarn showed lesser water uptake as well as less weight loss and mechanical properties as compared to other treated samples.

M. Jacob et al. [1] deal with the aspects of interfacial and surface characterization of natural fibers and their composites. The different chemical modifications of natural fibers and characterization aspects have been discussed. The adhesion between fiber and matrix is a major factor in determining the response of the interface and its integrity under stress Both fiber surface and polymer matrix surface can be modified to obtain a strong interface. The interfaces are characterized by XPS, nuclear magnetic resonance (NMR), FTIR, UV/VIS and micro-mechanical techniques.

M. N. Belgacem studied several approaches of suface modification (a) physical treatments such as corona, plasma, laser, vacuum ultraviolet and γ-radiation treatments; (b) chemical grafting by direct condensation, including surface compatibilisation with hydrophobic moieties and copolymerisation with the matrix. The copolymerization approach called upon different strategies: (1) the use of bi-functional molecules capable of reacting with OH groups of the cellulose surface and leaving the second functions available for further exploitation; (2) the direct activation of the surface and the subsequent grafting from

polymerization and (3) the condensation of organomettalic compounds, followed by their coupling with suitable reactive molecules or macromolecules. The characterization of the modified surfaces involved a variety of techniques, including elemental analysis, contact angle measurements, inverse gas chromatography, X-ray photoelectron and FTIR spectroscopy, water uptake, etc. they concluded that the most promising approaches of surface modifications are those capable of giving rise to continuous covalent bonds between the cellulose surface and macromoleculer matrix, because they optimize the mechanical properties of the ensuing composites and protect them from water absorption.

E. Mader [15] investigates the effect of maleic anhydride grafted polypropylene (MAH gPP) coupling agents on the properties of jute fiber/ polypropylene (PP) composite with two kinds of matrix (PP1 and PP2). Both mechanical behavior of random short fiber composites and micro-mechanical properties of single fiber model were examined. Taking into account interfacial properties, a modified rule of mixture (ROM) theory is formulated which fit well to the experimental results. The addition of 2 wt% MAHgPP to polypropylene matrix can significantly improve the adhesion strength with jute fibers and in turn the mechanical properties of composites. They found the intrinsic tensile properties of jute fiber are proportional to the fiber's crosssectional area, which is associated with its perfect circle shape, suggesting the jute fiber's special statistical tensile properties The theoretical results are found to coincide fairly well with the experimental data and the major reason of composite tensile strength increase in humidity aging condition can be attributed to both improved polymer matrix and interfacial adhesion strength.

CHAPTER – 3
EXPERIMENTAL

3.1 COLLECTION OF JUTE AND POLYMER

Jute fibers that are used in the present studies were collected from Bangladesh Jute Research Institute (BJRI). Jute fibers are of prepared from BT tossa, grade of jute and depending on the treatment condition the types are a) raw jute, b) retted jute, c) retted and scoured jute.

Retting means simply the water rating that is widely done in many places of Bangladesh. The ponds, lakes and the rivers are the main areas that are used for this purpose. Different treatment of jute fibers was carried out at BJRI. BJRI provided detail information about retting and other treatments. These are available in literature review chapter. Table 3.1 provides brief information about the jute fiber types, treatment procedure and place of allocation.

Polymer supplied by a supplier It was polypropylene of commercial grade named Polypropylene Net wt. 25 kg manufactured by the Polyolefin company (Singapore) Pte.Ltd. To study the influence of the interface between the fiber and the polypropylene on the composite properties, all the fibers viz raw jute fiber, retted jute fibers and scoured jute fibers are used in different condition, such as,

      i.   As received condition

      ii.   NaOH treated condition

      iii.   Maleic Anhydride treated condition

Table 3.1 Types of jute fiber

Name of Jute fiber	Treatment	Identification	Treatment place	Pictorial view
Raw jute	None	Raw jute	n/a	

Name of Jute fiber	Treatment	Identification	Treatment place	Pictorial view
Retted jute	Simple water rating	JR' implies Jute Retted	BJRI	
MAH Raw jute	Reported below	"MAH Raw" implies maleic anhydride treated	MME Department	
NaOH Raw jute	Reported below	NaOH Raw implies NaOH treated	MME Department	
MAH retted	Reported below	"MAH R" implies maleic anhydride treated Retted jute	MME Department	

Name of Jute fiber	Treatment	Identification	Treatment place	Pictorial view
NaOH Retted	Reported below	"NaOH R" implies NaOH treated retted jute	MME Department	
Retted and Scoured jute	Treated with 3% NaCO₃ Lisaphol (wetting agent). Temp. 90-100 °C Time. 1 hr Washing Drying	"JS" implies Jute Retted and Scoured	BJRI	
MAH Retted and Scoured jute	Reported below	MAH JS implies MAH treated scoured jute	MME Department	
NaOH Retted and Scoured jute	Reported below	NaOH JS implies NaOH treated scoured jute	MME Department	

3.2 TREATMENT CARRIED OUT IN THESIS WORK

These different types of jute fibers are chemically treated to enhance its compatibility, interfacial adhesion. The treatments are:

1. Maleic anhydride treatment
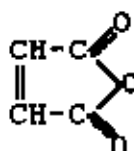
2. Alkali treatment NaOH

3.2.1 Maleic Anhydride Treatment:

The maleic anhydride (MAH) has been applied in a 8 wt.% with respect to the fiber weight The fiber was esterified for 10.5 hours with maleic anhydride dissolved in boiling acetone (T=50±5°C) with a fiber:solvent ratio of 1:25 (weight/volume). Thereafter, the fiber was washed several times in cold acetone and distilled water. Finally, the fiber was dried in an oven at 105°C for 24 h at least before any testing.

3.2.2 Alkaline Treatment:

The alkaline treatment was done by treating the fiber samples with a NaOH –solution with a NaOH-concentration of 30% for 4hr at a temperature of 28°C, followed by washing the fibers in distilled water. Finally, the fiber was dried in an oven at 105°C for 24 h at least before any testing.

3.3 DETERMINATION OF WEIGHT LOSS AFTER TREATMENT

The untreated and treated fibers were tested for their weight change. The loss in weight was calculated by weighing a fixed amount of dry and cleaned fiber (W_1). The amount of fiber was weighed again after treatment (W_2). The %weight loss was calculated as

$$\% \text{Weight loss} = (W_1 - W_2)/W_1 * 100 \dots\dots\dots\dots\dots\dots\dots (3.1)$$

It was observed that the fibers were somewhat leached and became finer. Fiber fineness was determined in terms of linear density by gravimetric method.

3.4 SEM IMAGING OF JUTE FIBERS

The scanning electron microscope (SEM) was utilized to find out the 3- dimensional images of the 9 types of untreated and treated jute fibers. The SEM shows very detailed surface topography at much higher magnifications than that of light microscope shows. The images created by light waves are rendered black and white. The project work utilizes 10 KeV source for imaging. A basic understanding of the conditions and techniques of sample preparation and microscopy is important if meaningful and reproducible structural information are to be recorded and related to functional properties of the specimens.

In order to assess the effect of the treatment upon jute fiber surface, characterization of nine types jute fibers was done in SEM (Model no XL-30, SL no – D1276, Philips, The Netherlands). A vacuum unit sputter coater (Model no XL-B7341, SL no – A1934, Belgium) was implemented to deposit a thin metallic layer of gold of 70nm thickness on the fibers. It has to be noted that when natural materials are examined with SEM, damage from electron beam may occur. In order to avoid this operating voltage was kept constant and a thicker layer of gold was deposited instead of usual 40 nm thickness [20, 34].

3.4.1 Sample Preparation for Imaging

Most specimens are supported on a thin film of plastic or evaporated carbon Micrographs, therefore, contain an image of the specimen superimposed with that of the support film. The added substructure of the supported film constitutes a major source of noise that obscures fine specimen details. The support film also reduces specimen contrast. Although not generally required, with certain specimens, ultra thin films (<5 nm) or no film at all may be useful for the conductive specimen but not for the non-conductive stuff. Carbon adhesive tape was used for the imaging, which was adhered on the one-centimeter dia-disc of aluminum-stub sample holder. The carbon adhesive tape is usually known as the SEM conductive tape.

The SEM used to produce the images in this project is operated under high-vacuum conditions and a battery of pumps serve to keep the inside of the instrument (including the specimen chamber) at low pressure. While this fact is not a consideration when one

examines materials (mineral, semiconductors, metals, etc.), it is a major concern when one wishes to examine biological samples such as jute. Biological samples contain large amount of water located in very fragile structures (cells). The presence of even small amounts of water in the SEM system can seriously degrade the vacuum, reducing the quality of the images. The problem then, is removing the water from cells without disrupting their fragile structure.

To start, the structural components of the cells are chemically stabilized. An alternate method, which was used in the present study, involves replacing the water with organic solvents such as acetone and liquid carbon dioxide followed by oven drying. The sample is then placed in a chamber and heated under pressure. At this point, the fluid is transformed spontaneously to gas, which can be vented from the system. The result is a dry sample whose structure is unaltered

Once the fixation and drying procedures were complete, the specimens were affixed to specimen mounts or "stubs". Conductive adhesive were used to hold the specimen on the mount.

Jute samples were prepared carefully to withstand the vacuum inside the microscope. Since jute is a biological specimen along with hydrophilic and hydrogroscopic nature, so to ensure proper vacuum and for the machine safety the jute fibers were dried in a special way that prevents them from shriveling. Because the SEM illuminates them with electrons, they also have to be made to conduct electricity. Electrical conduction was made by the following procedures:

- Sample container were washed with acetone, followed by the individual types of treated jute fibers were washed with acetone for five minutes and placed in a petridish allowed to dry-out of acetone.

- The fibrous samples in petridish were cut into small pieces of about 5±1 mm in length and allowed to dry at 100°C in a resistive heating type oven, for 12 hours [4, 13, 34]. Temperature attainment lag is 20 minutes so drying was continued excluding this time of temperature attainment.

- Moisture contamination after acetone-wash of fibrous sample was avoided using

airtight sample container. Use of polyethylene hand glove during process ensures no moisture contamination from hand to jute fiber.

- Sample holder made of aluminum disk first washed with acetone followed by drying. The small length (0.5 - 0.8cm) of jute sample was stuck to sample holder.

- In such cases a very thin layer of conductive material can be laid down on the surface of the sample. These coatings are usually composed of fine-grained, highly conductive metals such as gold, palladium, and platinum. A coating of gold/palladium alloy about 100 nanometers thick is usually sufficient to ensure adequate viewing of nonconductive samples in the SEM and during the project work 70nm coating of gold was applied on the jute fiber [13].

- It was sputtered for 30 seconds under vacuum to ensure a conductive gold coating on the non-conductive fibrous material.

- The sample is now ready for the SEM imaging and sample holding stab is put inside it. Then the machine is started sequentially, ensuring vacuum, ensuring inert atmosphere inside the machine and imaging.

The images produced by the SEM, as such, can be output to a monitor for viewing or to a film-recording device for photographic output.

3.5 DETERMINATION OF LINEAR DENSITY OF DIFFERENT JUTE FIBERS

Linear density can be defines as mass per unit length. The preferred units of measurement are grams and meters, or multiples or submultiples of these. The tex unit, grams per kilometer, is recommended for yearns, fibers and other textile strand. Linear density of different types fibers determined following ASTM D 1577 standard. Average linear density, in tex, of single fibers in a bundle is calculated from mass and length measurements on the bundle and the number of single fibers in the bundle by following procedure:

- The specimens tested in the standard atmosphere for testing textiles, which is $21 \pm 1°$ C and $65 \pm 2\%$ relative humidity.
- The bundle of fibers is placed on a cutting device. Make certain that the fibers are in parallel alignment.

- Crimp removed by pretensioning the specimen under a tension.
- The specimen was cut to the selected length using cutting devices.
- The specimen weighed to the nearest 0.005mg.
- The number of fibers in bundle was count.
- Calculation of the average fiber linear density for each specimen using equ 3.2

$$T = 100W/(L*N) \dots\dots\dots\dots\dots\dots\dots(3.2)$$

where; T = average fiber linear density, dtex

    W = mass of bundle specimen, mg

    L = length of the bundle specimen, mm, and

    N = number of fibers in the bundle specimen.

3.6 DETERMINATION OF TENSILE PROPERTIES OF JUTE FIBERS

Mechanical characterization of these untreated and treated fiber was done using Instron-3369 tensile testing machine fitted with a 5N loadcell. ASTM D3379 test method was followed for tensile test of jute fibers.

3.6.1 Test specimens

Special care taken to assure obtaining representative strand or single filament segments. a statistically significant number of single-filament test specimens are gently separated from the strand bundle. Selection was random. The critical considerations are that the filament not be visibly damaged or attached to another filament, and have sufficient length. Segments used for area determinations shall represent the same population as those used for tensile testing

3.6.2 Procedure

a) Test specimen mounting

- A strand bundle from the material to be tested is loosely placed on a suitable work surface.

- A suitable single-filament specimen from the strand bundle was randomly choosed and carefully separated .

- The specimen gage length be the same for a given test group. For these test methods, the specimen gage length shall be between 20 and 30 mm.

- A tab was used for specimen mounting center the specimen over the tab slot with one end tabbed to the tab.

- The filament and tape was lightly stretched its opposite end to the tab.

- A small amount of suitable adhesive was carefully placed on the filament at each edge of the slot and bond the filament to the mounting tab.

b) Filament specimen testing

- The tensile test machine stabilized in accordance with the manufacturer's instructions.

- The test machine was calibrated before testing begins.

- The crosshead and chart recorders speeds was set to provide a test time to specimen fracture of about 1 min.

- A mounted test specimen was grasped in one tab area by the faces of the stationary jaws.

- The crosshead positioned so that the other tab grip area may be grasped.

- With the mounting tab unstrained, both sides of the tab was cut or burned it away very carefully at mid-gage When specimen damage occurs, the specimen discarded.

- Tensioned the specimen with the chart continuously recording the test load to failure and the indicated elongation

- Fiber diameter was determined from optical micrograph of stretched fiber on mounting tab at 100X.

3.7. WEIBULL PROBABILITY PLOTS

The Weibull probability distribution is a mathematical description of data, which can be used to evaluate many types of "weakest links" experimental data and has been very instrumental in describing tensile failure of fibers

The Weibull probability distribution is given by

$$P_f = 1 - \exp\left(-(\sigma - \sigma_c)/\sigma_0\right)^m V/V_0)..(3.3)$$

Where P_f is the probability of failure; σ is the failure stress; σ_c is a critical stress, below which fracture does not occur; σ_0 is a normalizing stress; m is a constant (Weibull modulus); V_0 is the volume of identical samples that survive loading to a tensile stress σ; V is given by $n V_0$ where n is a batch of samples that survive the stress.

Taking logs twice on both sides reduces the equation to

$$\ln(-\ln(1 - P_f)) = \ln(V/V_0) + m \ln (\sigma - \sigma_c)/\sigma_0..............................(3.4)$$

The gradient m can then be computed from the plot of $\ln(-\ln(1 - P_f))$ against $\ln \sigma_0$). In practice, since we do not assume any critical stress below which it is impossible for failure to occur, the value of σ_c can be set equal to zero and the x-axis variable taken as $\ln(\sigma)$.

Ranking the failure stresses in order of increasing strength and assigning a probability of failure of $P_f = n/N+1$ to the nth ranked specimen in a total sample size of N, is then used as a method of finding the failure probability of any given stress.

Weibull plots were produced to see how the variation in fiber strength reflects the distribution of flaws on fiber surfaces

3.8. THERMAL ANALYSIS:

Thermal properties (TGA/DTG and DSC) of different types of fiber were analyzed and compared. The composite specimens (fabricated by hot press) were cut to small particle form. Then TGA/DTG and DSC analyses were carried out using a Mettler TG 50 and DSC 25 module attached to a Mettler TC 11 4000 thermal analyzer. All the measurements were made under a nitrogen flow (Balance purge flow 40 mL per min, sample purge flow 60 mL per min), keeping a constant heating rate of 10°C per min and using an alumina crucible in TGA and platinum crucible in DSC.

DSC is a thermoanalytical technique in which heat flow is measured as a function of temperature or time. DSC is useful in characterizing thermal properties of raw materials, mixture of materials or finished products and also provides information quickly and easily on a minimum amount of sample. One treated fiber sample of 4-6 mg was placed in an aluminum sample pan and sealed with crucible sealing press; this pan was placed in the sample holder. The sample was heated at a controlled rate 10°C per min from room temperature to 600°C and a plot of heat flow versus temperature was produced. The resulting thermogram was then analyzed.

TGA also is a thermoanalytical technique in which weight loss is measured as a function of temperature or time. TGA is useful in characterizing thermal stability of raw materials, mixture of materials or finished products and also provides information quickly and easily on a minimum amount of sample. One treated fiber sample of 4-6 mg was placed in a platinum sample pan and this pan was placed in to the TGA furnace. The sample was heated at a controlled rate 10°C per min from room temperature to 600°C and a plot of weight change, and derivative weight change versus temperature was produced

3.9. FABRICATION OF COMPOSITE

Composites were fabricated by hot pressing using a Fortune Presses Manufactured by Fontijne Grotnes at 180°C using 1.2 bar pressure. Fiber size used 2mm, 4mm and 5 mm. each individual types were compounded to 10, 15,20,25 volume percent. A 27 by 27 cm metallic die used The fabrication process consists of several steps:

Cleaning of dies and spraying of mold releasing agent.

A thin layer of polypropylene spread on the die.

Fibers are distributed uniformly on the polypropylene layer. Disperser is used for uniform distribution of fibers.

Then another layer of polypropylene used.

The die is placed on the heated platen of the press.

Then applications of required pressure after attaining die temperature 180°C

After sufficient time of removal of pressure and cooling of die by water circulation.

Finally release of composites out of die

3.10. MECHANICAL CHARACTERIZATION OF COMPOSITES

3.10.1 Standardization and Practices

Mechanical characterization of these fabricated composite was done using Universal Testing Machine INSTRON 3369 ASTM D3039 test method was followed for tensile test of composite specimen; ASTM D 790 test method was followed for flexural or three point bend test of composite.

3.10.2. Preparation

The test sample is a precise part. A test specimen is the most important component of tensile testing, for it determines the actual physical properties of the materials being tested. The physical test specimen must conform to exacting dimensions and must be free of induced cold working or heat distortion.

Table 3.2: Standard dimensions for tensile specimen for fiber-reinforced polymer composite (ASTM D 3039)

Fiber Orientation	Width, mm	Overall Length mm	Thickness, mm	Tab thickness mm	Tab Bevel Angle,°	No of Test specimen for individual composite
Random-Discontinuous	22	220	2.2	Specimen thickness	None	5
Reference	22	220	2.2	Specimen thickness	None	5

It is clearly seen from the table 3.2 that the dimensions for the tensile characterization of composite systems are specified. Since composites were fabricated from chopped discontinuous jute fiber so the composites falls in the category random and discontinuous According to ASTM standard the tensile specimen were made. It is clearly specified that the specimen dimension must fulfill the recommended dimension or the test undertaken as non-standard practice. This standard is a must to follow when the specimen has commercial value and the data represented must follow the standard.
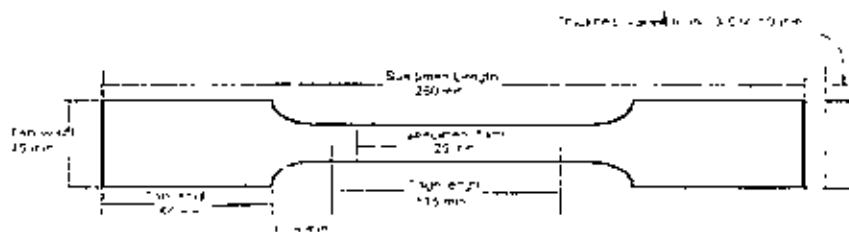
Fig 3.1. Schematic drawing for tensile specimen

The dimensions of the tensile specimen are specified in the table 3.2. and fig 3.1 shows the schematic drawing for tensile specimen. A standard cross head displacement rate of 2mm/min was used for test and the strength and elongation are measured using the following equations:

Ultimate Tensile Strength (UTS) σ_{uts} = P_{load}/A...(3.5)

Where P_{load} is the load needed to fail the specimen and A is the cross sectional area of the composite specimen,

% Elongation = {(final length – initial length)/initial length}*100 ....(3.6)

Tensile specimens were cut from the composite plate by an ebonite cutter machine some thing like jig saw. Here a compromise may be made such that, when a tensile specimen was cut from a composite plate the movement of saw puts a directional mark on the specimen that may act as stress concentration during tensile loading and grinding of these rough side also induces intentional stress concentration. Considering these facts it was realized that the specimen must be made in a separate mold to avoid these stress concentration marks in a tensile specimen.

3.10.3 Flexure Test Specimen Preparation

Flexure strength or Modulus of Rupture is the maximum bending stress in tension applied to a beam at failure. This testing allows the worker to receive information on the actual flex of a material before it has changed in either strength or make-up. A flexure test is relevant for design and specification purposes of an object that may do supporting or will be subject to bending. The material is subjected to a transverse (bending) force perpendicular to its longitudinal axis producing shear and tensile stresses in the beam.

Composites are tailored to meet application requirements for impact resistance, strength under load, durability, and other attributes. 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 was calculated at the surface of the specimen on the convex or tension side. Flexural modulus was calculated from the slope of the stress vs deflection curve. If the curve has no linear region, a secant line was fitted to the curve to determine slope.

Figure 3.2 shows the typical flexure specimen and test procedures for composite specimens. A bar of rectangular cross section rests on two supports and is loaded by means of a loading nose midway between the supports. To ensure the primary failure comes from tensile or compression stress the shear stress must be minimized. This was 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 L/d=16 is acceptable. Some materials require L/d=32 to 64 to keep the shear stress low enough. During the project the ratio of L to d was kept at a value of 16. The specimen is deflected until rupture occurs in the outer surface of the test specimen or until a maximum strain of 5% is reached, whichever occurs first.

Rate of crosshead motion is determined by using the following formula

Rate of crosshead motion (mm/mm) $R = ZL^2/6d$ ....(3.7)

Where Z is the rate of straining of the outer fiber, mm/mm/min; L is the length of the support span and d is the specimen thickness.

Flexural stress of the test specimen is calculated using the following formula, which has the value of

Stress in the outer fibers at mid points, (MPa) $\sigma_f = 3PL/2bd^2$ ....(3.8)

Where P is the load at a given point on the load deflection curve, L and d are the length of the support span, width and depth of the specimen and are expressed in mm. For composite specimens made of brittle polymer σ_f is some times stated as σ_{fB} and is known as the flexure stress at break.
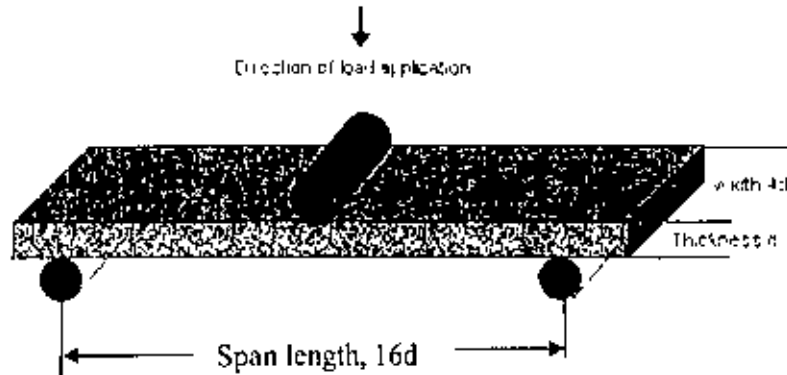
Fig 3.2. Typical flexure test setup of polymer composite.

Flexural strain is calculated using the following formula, which has the value of

Strain in the outer surface (mm/mm) $\epsilon_f = 6Dd/L^2$ ....(3 9)

Where D is the maximum deflection of the center of the beam, mm; L is the length of the support span and d is the specimen thickness.

In a 3-point test the area of uniform stress is quite small and concentrated under the center loading point. When a 3-point flexure test was done on a brittle material like thermoset polymers and thermoset polymer matrix based polymer composites, ceramic or concrete it is often called modulus of rupture (MOR). This test provides flexure strength data only, not stiffness (modulus).

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 carry out the flexure test for the chopped jute fiber reinforced unsaturated composite only four specimens were prepared from the fabricated composite plate. For laminated specimen four specimens were made and each test were carried out using span to depth ratio of 1: 16 and the width of each specimen was kept within the 4 to that of the span The support span end the compression end diameter was 10mm

3.11 SCANNING ELECTRON MICROSCOPY OF THE FAILED TENSILE COMPOSITE SPECIMEN

SEM was utilized to characterize the fracture surface, crack profile of chopped random discontinuous jute FIBER composites In order to assess the fracture behavior of the composite specimens, characterization of small portion of fracture face of specimen was placed in a Scanning Electron Microscope (SEM) (Model no XL - 30, SL no - 01276, Philips, The Netherlands).

A vacuum unit sputter coater (Model no - B7341, SL no - A1934, Belgium) was implemented to deposit a thin metallic layer of gold of 70nm thickness on the fracture surface. The reason of depositing such amount of gold on a nonconductive surface was stated in the section 3.2 of this chapter [20]. The specimens were kept in airtight bag and stored in desiccator Similar method of specimen preparation was followed, as indicated in the section 3.2.2 of this chapter.

CHAPTER – 4
RESULTS AND DISCUSSION

4.1 SEM EXAMINATION OF THE UNTREATED AND TREATED RAW JUTE

4.1.1 Raw jute fiber

In each plant, the rings of fiber cell bundles form a tubular mesh that encases the entire stem from top to bottom. Two layers can usually be distinguished, connected together by lateral fiber bundles, so that the whole sheath is really a lattice in three dimensions. The cell bundles form the links of the mesh, but each links extends only for a few centimeters before it divides or joins up with another link. After extraction from the plant, the fiber sheath forms a flat ribbon in three dimensions.

The jute refers to the sheath extracted from the plant stems, whereas a single fiber is a cell bundle forming one of the links of the mesh. When a longitudinal section of a single fiber was examined under scanning electron a microscope (figure 4 1.), the overlapping of the cells along the fiber is formed. The cells are firmly attached to one another laterally, and the regions at the interface of the two cells are termed as the middle lamella. Separation of the cells can be effected by chemical means, and they are then seen to be threadlike bodies ranging from 0.75 to 5 mm in length, with an average of about 2.3 mm. the cells are some 200 times longer than they are board, and in common terminology are referred to as ultimate cells. A single fiber thus comprises a bundle of ultimates

(a)   (b)
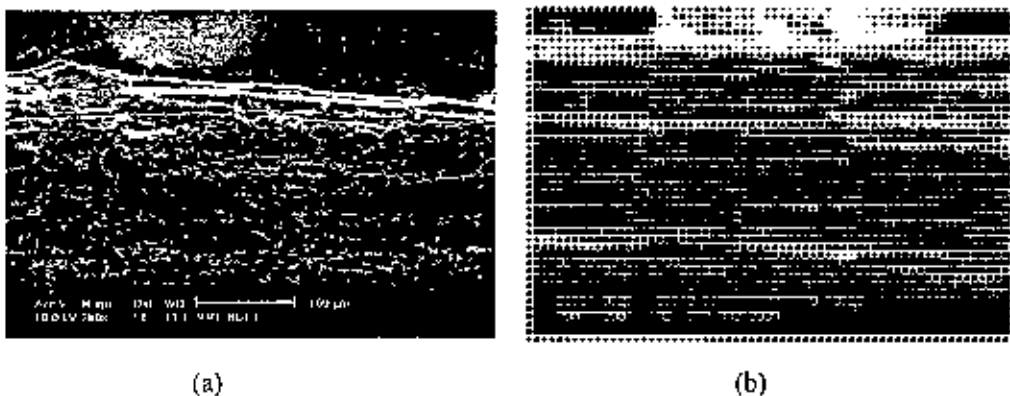
Figure 4.1. SEM image of raw jute fiber showing (a) waxy materials (b) clean surface

4.1.2 Maleic anhydride treated raw jute fiber

The location of the three main chemical components of the fibers is reasonably well established. α-cellulose forms the bulk of the ultimate cell walls, with molecular chains lying broadly parallel to the direction of the fiber axis. The hemicellulose and lignin, however, are located mainly in the area between neighboring cells, where they form the cementing material of the middle lamella, providing strong lateral adhesion between the ultimates Figure 4.2 shows the SEM micrograph of maleic anhydride treated raw jute fiber. There are numerous impression of cell along the fiber in regular pattern. This figure indicate that maleic anhydride removed hemicellulose and lignin to some extent from the ultimate cell. But probably maleic anhydride cannot remove pectin and other gummy substance. Presence of these components makes the fiber surface rougher than raw jute fiber.
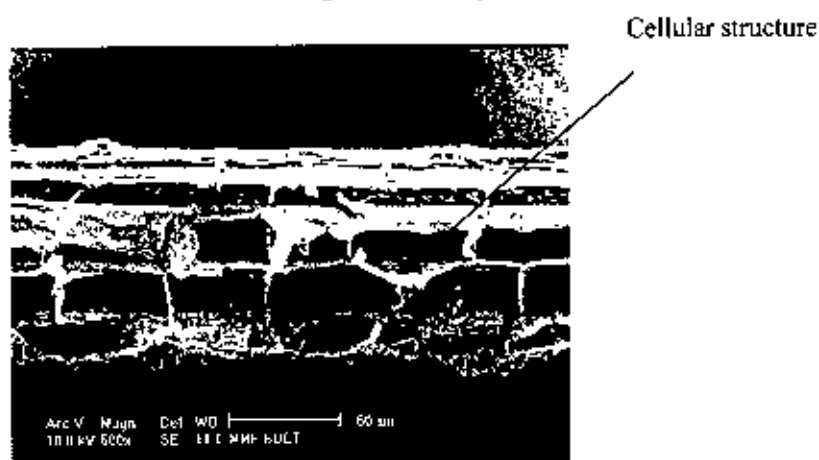
Figure 4.2. SEM image of MAH raw jute fiber

4.1.3 NaOH treated raw Jute fiber

As discussed earlier the NaOH dissolves lignin, hemicellulose and other alkali soluble compounds from the raw jute fiber surface. Since raw jute fiber surface is rough as revealed in fig. 4.1 dissolution of the above mentioned substances makes the fiber surface makes rougher (fig 4 3).
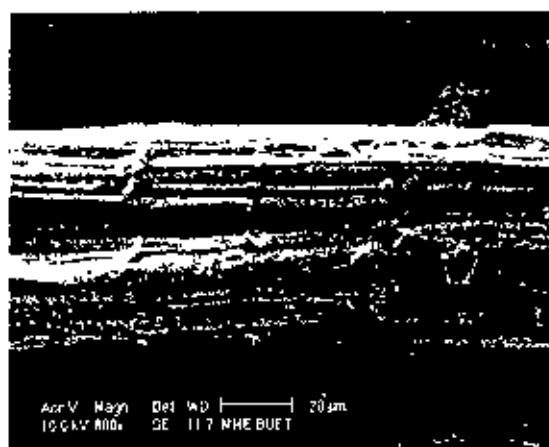
Figure 4.3. SEM image of NaOH raw jute fiber

4.2 SEM EXAMINATION OF THE UNTREATED AND TREATED RETTED JUTE

4.2.1 Retted jute fiber

The SEM micrograph of retted jute is shown in figure 4.4 All the micrographs reveal that; the surface of retted jute is heterogeneous with a relatively low micro-roughness compared to raw jute. This may be explained by the fact that the bacterial action removes the some constituents of the fiber such as pectin, lignin and other gummy and waxy materials during retting in different portions in different areas and, hence, the fiber surface becomes smoother than raw jute fiber.

This bacterial activity causes enzymatic action that facilitates retting process. The fiber bundles are also exposed because of this bacterial action and quite clearly identified in the figure 4.4, which results from the combined action of the microbes and the under water retting atmosphere. Figure 4.4 of the retted jute fiber show the presence of fiber binder materials

The rougher portion clearly shows the binding materials embedded within the fiber bundles. However, it is also observed in figure 4 4 that the fiber surface seemed to be covered by a thin layer of wax or simply the fiber surface itself appears smooth. This assumption is supported from spectroscopic results, which are discussed elsewhere [2]. This layer may be attributed to the presence smoother fiber surface, which has been formed during retting The pre-acetone wash ensures the removal of dust particles from the fiber surface except the

binding materials that appeared as dust particles in all figures.

Micrographs (figure 4.4) shows that there is some coverage of the fiber as well in different regions, but it is unclear whether or not it is due to the presence of an unremoved waxy layer or the binders of the fibrous body. However, the fiber losses weight because of the treatment, which therefore implies that, more enzymatic action of the microbes on the fiber surface that removed the binder materials [2] The amount of weight loss was measured in the study.

Fiber binding materials
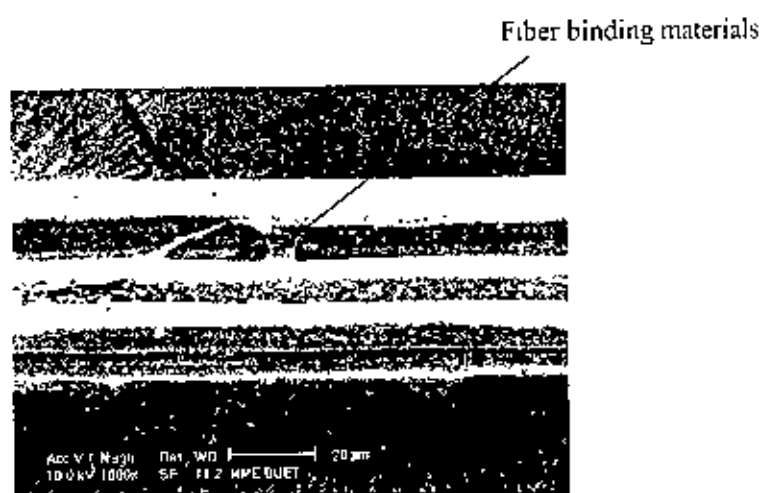
Figure 4.4. SEM image of retted jute fiber

4.2.2 MAH treated retted jute fiber

The maleic anhydride group forms both covalent and hydrogen bonds with the hydroxyl groups of the fiber [10] Retted fiber consist of α-cellulose, hemicellulose, lignin with a relatively smooth surface topography as seen in figure 4 5 The maleic anhydride group reacts with the hydroxyl group and leaving a more or less smooth appearance as shown in figure 4 5

Fiber binding materials
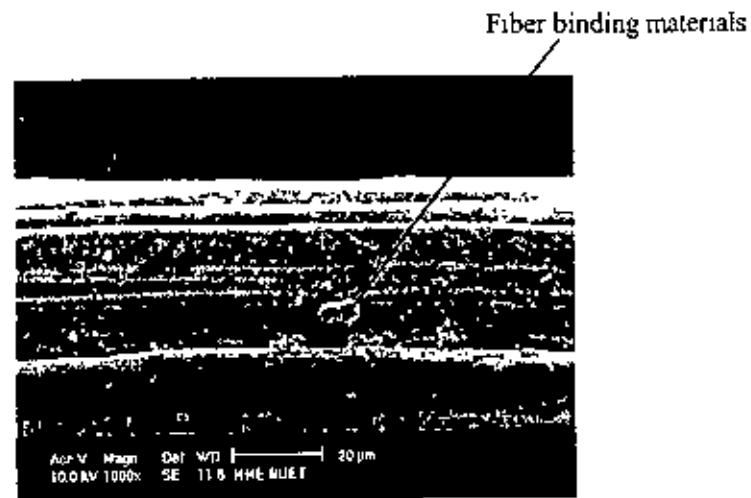
Figure 4 5. SEM image of MAH retted jute fiber

4.2.3 NaOH treated retted jute fiber

When jute fiber is treated with strong alkali, profound changes occur in its physical structure. Lateral swelling occurs together with considerable shrinkage in length Because of which the fiber is softened and develops a high degree of crimp or waviness. The crimp gives wool like appearance to the fiber and much attention has been given to assessing the commercial possibilities for this chemical modification. On stretching the fibers to break, the crimp is straightened and thereby the extensibility of the fiber is increased.

The sheathing of the ultimate cells by a lignified membrane affects the free swelling of the cells and produces tension, and the irregular shape of fibers in cross section will lead to folding under tension once the middle lamella material is weakened by the treatment. But since the alkali concentration was high in our treatment this has acted on the fiber for crimp formation. The tenacity changes are no doubt linked with the losses in weight that occur with alkali treatment, but there may be more profound changes taking place internally within the ultimate cells as shown in the figure 4.6. Such changes are not well understood Moreover, the crimp is a "once-only" effect and some small degree of elasticity may be introduced into the fiber The stability of the crimp is poor. Once the crimped fiber has been straightened under tension, there is no tendency to revert to the crimped state when the tension is removed. That is to say, the woolenizing treatment does not confer elasticity on the fiber.

A strong NaOH treatment has been successfully used to remove lignin, hemicellulose and other alkali soluble compounds from the surface of the fiber, thus increasing the number of reactive hydroxyl groups on the fiber surface available for chemical bonding [19]. The increase in the percentage crystallinity index of alkali-treated fibers occurs because of the removal of the cementing materials, which leads to better packing of the cellulose chains [2, 4]. This may be due to reaction of alkali with the cementing materials of the fiber, which is shown in figures 4.6. This reaction leads to the destruction of the mesh structure of the fiber and splitting of the fibers into finer filaments. The breaking down of the fiber bundle helps to increases the effective surface area available for wetting by the matrix. Thus bonding between the fiber and the matrix at the interface may be improved The increase in stiffness is thought to be due to an increase in crystallinity of the fiber cellulose making the fiber more rigid and brittle. It was observed in figure that all types of jute fibers were somewhat leached and were finer

Loss in weight was observed after the alkali treatment of the fibers, due to heavy dissolution of the hemicellulose content, since alkali is more attacking to hemicellulose than the other components of the fibrous portion of jute. This may resulted in the drop of linear density of the fibers when treated with 20% alkali for 10 minutes at room temperature; due to the creation of voids in the fiber structure, the strands become well separated and dispersed.

Figure 4.6 reveals the crimp or wavy nature of the fibers due to the alkali treatment. Although low concentration of alkali may create the same appearance of the fiber when treatment is carried out for long time. As shown in figure 4.6 the fiber is curved and the binders are well leached out of the fiber surface and the interior body. Although findings about the crystallinity of the fibers were not carried out during the course of the present work but many workers have reported that the crystallinity of the fibers have increased It was imperative that the fibers became stiff and brittle because of its high strength and low extensibility [19] This may be because; the alkali reacts with the cementing materials of the fiber, particularly hemicellulose. Alkalization of plant fibers changes the surface topography of the fibers and their crystallographic structure. However, care must be exercised in selecting the concentration of caustic soda for alkalization, as results show that some fibers at high NaOH concentrations have reduced thermal resistance The modification of cellulose fibers, therefore, develops into changes in morphology and increase in hydroxyl groups. These changes will effectively resul. in improved surface tension, wetting ability, swelling, adhesion and compatibility with polymeric materials.
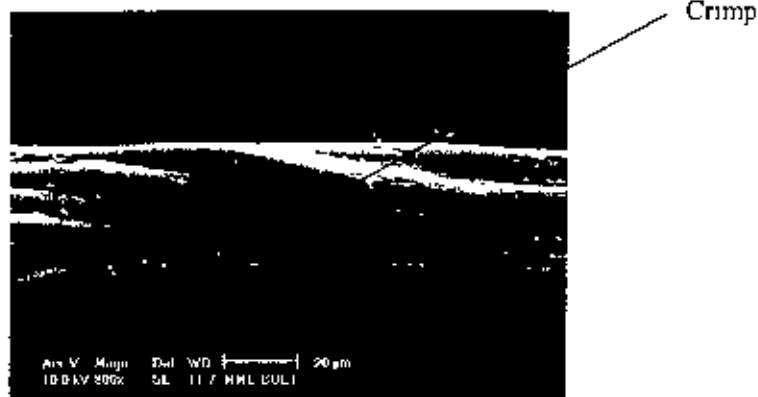
Figure 4 6 SEM image of NaOH retted jute fiber

4.3 SEM EXAMINATION AS RECEIVED, MAH TREATED AND NaOH TREATED SCOURED JUTE FIBER

Other than three principal chemical constituent α-cellulose, hemicellulose, and lignin, jute contains minor constituents such as fats and waxes, inorganic matter, nitrogenous matter, and traces of pigments. Na_2CO_3 treatment removes only fat and wax portion. Since these fat and wax are only of small percentage removal of these constituent produce no effect on surface topography. The scoured fiber surfaces shown in figure 4.7 to figure 4.9 appear as retted jute fiber.

As mentioned above maleic anhydride treatment forms covalent and hydrogen bond with fiber surface and NaOH removes some substances. So the microstructures remain more or less unaltered.
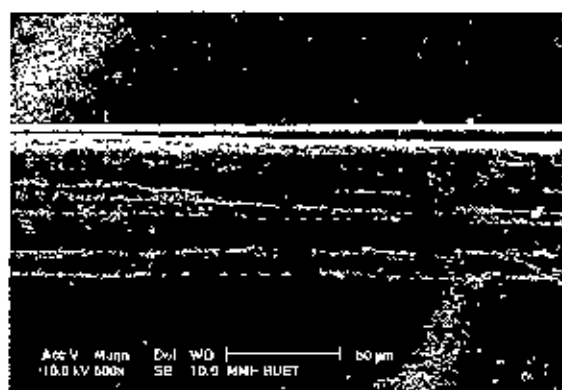
Figure 4 7 SEM image of scoured jute fiber
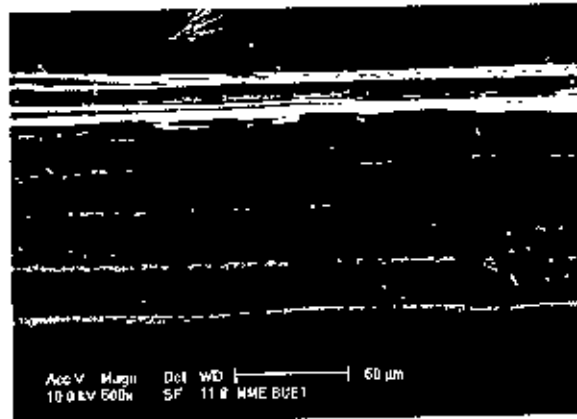
Figure 4.8. SEM image of MAH scoured jute fiber
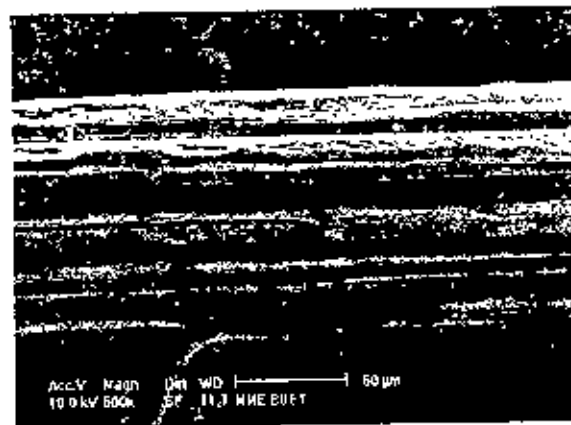
Figure 4 9. SEM image of NaOII treated scoured jute fiber

4.4 FIBER TENSILE STRENGTH

The tensile strength of untreated and treated jute fibers were obtained by dividing max load by cross sectional areas obtained using the microscopy method. Table 4.1 shows the test result of approximately 45 specimens of each type of fiber.

Table 4.1: Tensile strength of different jute fibers

Fiber condition	Diameter µm±sd	Tensile Strength, MPa σ±sd	Tensile Modulus, N/tex	Weibull distribution parameter.m	
				Weibull modulus, m	Characteristics strength, σ_o
Raw jute	139.23 ± 42.2	133.67 ± 90.56	35.31	1.60	150.76
MAH raw jute	171.11 ± 67.95	173.61± 149.47	22.71	1.41	176.17
NaOH raw jute	121.61 ± 44.1	235.94 ± 205.78	1.314	1.2	181.6
Retted jute	72.02 ± 14.5	253.32 ± 119.7	22.43	2.39	275.13
MAH retted jute	50.29 ± 12.49	540.84± 355.68	24.01	1.89	579.43
NaOH retted jute	47.20 ± 13.76	533.42± 260.54	5.57	1.86	559
Scoured jute	96.39 ± 36.3	153.67 ± 84.77	1.23	1.9	168.02
MAH scoured jute	94.52 ± 32.33	171.14 ± 148.32	32.2	1.32	176.17
NaOH scoured jute	50.09 ± 15.72	210.01 ± 119.5	6.29	1.86	229.73

Weibull probability plots of the tensile strengths of the fibers are made. This approach is appropriate for materials such as natural fibers where strengths are variable and depend on the distribution of surface flaws The probability plots for strength of different fibers (figure 4.10 to 4 12) show a reasonable fit to a classic Weibull distribution. The higher value for the m exponent reflects the lower standard deviation for strength.

As expected, the tensile strength of natural fiber in the fiber direction depends on the fiber length and exhibits considerable scatter, due to the statistical distribution of flaws such as pits and nodes and non-uniform cross-section of fiber. The Weibull modulus calculated using the strength distribution is in the range of 1.2 to 2.39. Generally speaking, a fiber with larger cross-section area should have greater chance to have a bigger flaw; therefore, it is weaker than that with smaller cross-section area. For natural fibers, there is a distribution of individual fiber cross sectional area, shape within both a roving and individual single fiber,

so that the tested volume will vary even at a fixed gauge length. Thus, the measured strength distribution will be created by the overlap of the distribution of strength due to volumetric distribution of flaws and due to the variable fiber volume.

It can be seen that the plot for treated fiber shows better accordance to Weibull distribution with showing higher tensile properties than untreated fiber. It is reported that jute fiber has cellulose content of more than 60% and a micro-fibrill angle in the range of 7-12° to the fiber axis [31]. These structure characters are strongly maturity dependent and in turn, affect significantly the overall mechanical properties of natural fibers. MAH and alkali both treatment reduce the defects and critical flaws on the fiber surface. The maleic anhydride group forms both covalent and hydrogen bonds with the hydroxyl groups of the fiber, which are responsible for, increased strength than untreated fiber. The improved properties of the fibers with alkali treatment were the result of dissolution of hemicelluloses and development of crystalinity and fibrillation From Table 4.1 it is evident that the MAH treatment increases the strength of retted jute almost two times than that of untreated retted jute
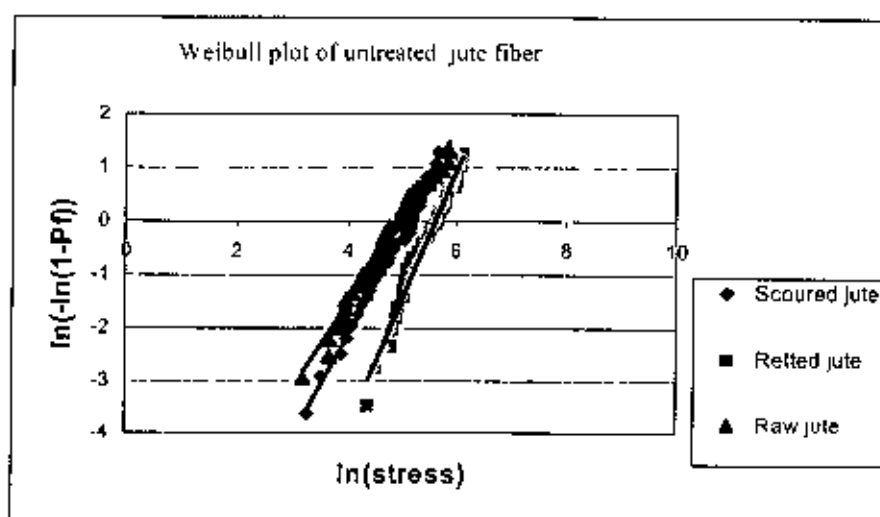
Types of jute	Slope	R² Value
Raw	1 6	0.97
Retted	2.39	0.97
Scoured	1.9	0 98

Figure 4.10. Weibull plot of untreated jute fiber

When the hemicelluloses are removed the interfabrillar region is likely to be less dense and less rigid, thereby makes the fibrils more capable of re-arranging themselves along the direction of tensile deformation. When jute fibers are stretched, such rearrangements among the fibrils would result in better load sharing by them, hence higher stress development in the fiber [9]. The alkali dissolves or modifies the critical defects in the outer layer thus reducing their length and sharpness, which in turns reduces stress concentrations.
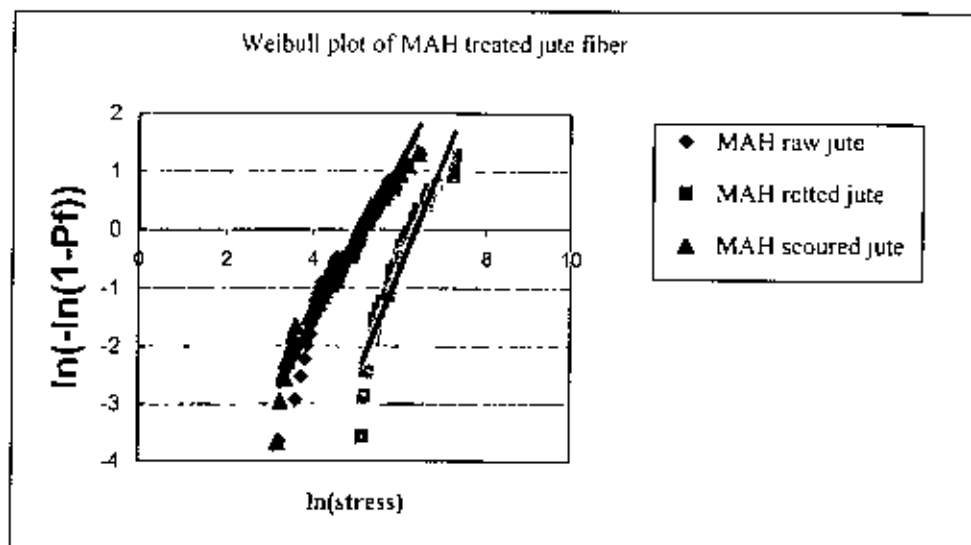
Types of jute	Slope	R^2
MAH raw	1 4	0.93
MAH Retted	1.89	0.91
MAH Scoured	1 32	0.96

Figure 4.11. Weibull plot of MAH treated jute fiber

Weibull plot of NaOH treated jute fiber

Types of jute	Slope	R^2
NaOH raw	1.2	0.96
NaOH Retted	1.8	0.99
NaOH Scoured	1 9	0.98
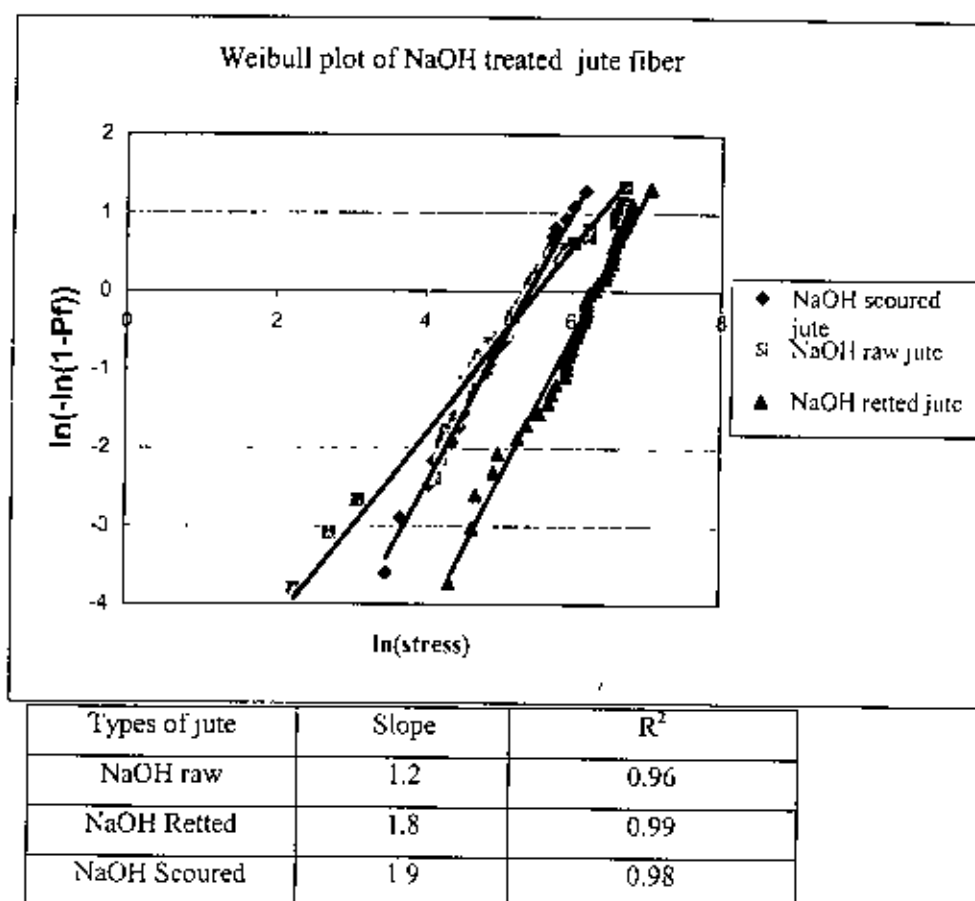
Figure 4.12. Weibull plot of NaOH treated jute fiber

4.5. WATER ABSORPTION, LINEAR DENSITY AND WEIGHT LOSS OF FIBERS DURING TREARMENT

The presence of many hydroxyl groups on the fiber structure makes the jute fibers more hydrophilic. Agro based fibers change dimensions with changing moisture content because the cell wall polymers contain hydroxyl and other oxygen —containing groups that attract moisture through hydrogen bonding. The hemicelluloses are mainly responsible for moisture absorption. As a result of water absorption swelling of cell wall occur and the fiber expands until the cell wall is saturated with water. Beyond the saturation point, moisture exists as free water in the void structure and does not contribute to further expansion

This process is reversible, and the fiber shrinks as it loses moisture. Cellulose, noncrystalline cellulose, lignin, and surface of crystalline cellulose also play major roles.

-Table 4.2 shows the percentage absorption of water. As expected the percentage water absorption decreases with treatment of fibers because in these treatment the chemical bond occur between available hydroxyl molecule and anhydride group. Moisture plays a significant role in influencing the mechanical behavior, the long-term durability of polymer and polymer matrix composites degrade with time. Water absorption is used to determine the amount of water absorbed under specified conditions.

Table 4.2. Linear density, % water absorption and % weight loss of different fibers during treatment

	Raw jute			Retted jute			Scoured jute		
	%Water absorption	%Wt loss	Linear Density, tex	% Water absorption	%Wt loss	Linear Density, tex	%Water absorption	%Wt loss	Linear Density, tex
Untreated	53 37	--	53 56	55 64		2.5733	53.2	--	2 034
MAH treated	43	8.81	49 64	53 43	0.0253	2 003	47	5 42	2 032
NaOH treated	53 3	7.78	27 94	43	5 2	2.4656	45	26 6	2 027

Loss in weight was observed after all treatment. Loss in weight was observed after alkali treatment of fibers shown in table due to heavy dissolution of hemicellulose content The loss occurred primarily within 2 hours of treatment [8]. In case of MAH treatment the raw jute loss more weight than retted jute. The loss in weight is thought to be due to dissolution of hemicellulose and pectin and other gummy substance that are present in as received condition. Scanning electron micrograph (figure 4.2) also support the above dissolution.

Linear density means mass per unit length. Tex is the unit of linear density equals to the mass in grams of 100 meters of fiber, yarns, or other textile strand. Any treatment like MAH or NaOH causes loss in weight of fibers, which is also resulted in the drop in linear density of the fibers.

4.6. THERMAL ANALYSIS OF JUTE FIBER

The TG and DTG curves of the untreated and the treated retted and raw jute fibers are shown in Figure 4.13. In the DTG curves of the jute fibers the first peak below 100°C was the result of evaporation of moisture. For retted jute the moisture loss peak for NaOH- and MAH-treated fibers shifted to a lower temperature. This tendency toward releasing moisture at a

lower temperature might be caused by an increase in the surface area of the split fibers (as observed in SEM figure 4.4) facilitating easier evaporation of moisture at lower temperature. Overall surface structure of untreated raw jute fiber evaporates moisture at lower temperature but after treated with NaOH and MAH, moisture-loss peak temperatures remain same for raw jute. Raw jute under untreated and treated conditions show two degradation peaks of α-cellulose and the hemicellulose at around 320-340°C and 260-270°C respectively where as a single degradation peak at about 334-338°C for α- cellulose was observed for both treated and untreated retted jute fibers and the percentage degradation for the α-cellulose of retted jute under treated and untreated conditions was always higher than that of raw jute fibers.

The DSC curves of the raw jute and the retted jute fibers are shown in fig 4.14. The endothermic peak below 100°C is due to the evaporation of moisture. The exothermic peak due to hemicellulose decomposition of MAH treated raw jute fiber shifted to higher temperature as compared to NaOH and untreated jute fiber. But for retted jute fiber this peak remains same for all three conditions. Similar trend was also observed for the endothermic peak temperature for untreated and treated raw jute fibers.

Third endothermic peak in Table 4.4 are for α-cellulose decomposition. In case of alkali treated retted fibers, the α-cellulose decomposition peak changed from endothermic to exothermic. Similar inversion was reported by Mitra et.al in the case of PF and CNSL-PF treated fibers and by Sikdar et.al for the bleached jute fiber, but no such inversion was observed for acetylated or cyanoethylated jute fibers. It is, therefore, apparent that the removal of the noncellulosic constituents such as hemicellulose during alkali treatment leading to the destruction of the chemical linkage between the constituents might have some influence in inverting the α-cellulose degradation peak from endothermic to exothermic. In the case of raw jute due to high percentage of constituents compared to retted jute (losses during retting) such inversion not observed

In case of acetylation where only the hydroxyl groups took part in the chemical reaction, no such inversion was observed

Table 4 3. TGA data of jute fiber

Sample	1st Peak Temp (°C)	2nd Peak Temp (°C)	3rd Peak Temp (°C)	Weight loss 1st peak, %	Weight loss 2nd Peak,%	Weight loss 3rd Peak,%
Raw jute	42.39	263.07	323.25	2.31	6.434	41 06
MAH raw jute	42.39	268.54	340.58	1.648	7.207	47 02
NaOH raw jute	42.99	243 54	333 88	2 061	10.78	45 33
Retted jute	48.89		334.38	1.742	56.60	
MAH Retted jute	39 81		339.52	1.749	57.27	
NaOH Retted jute	42.17		335 49	2.101	54 46	

Table 4.4. DSC data of jute fiber

Sample	1st Peak Temp (°C)	2nd Peak Temp (°C)	3rd Peak Temp (°C)	Nature of 1st peak	Nature of 2nd peak	Nature of 3rd peak
Raw jute	66.34	342.89	378 08	endo	exo	endo
MAH raw jute	51 81	367.91	390.18	endo	exo	endo
NaOH raw jute	65.52	353.35	378.19	endo	exo	endo
Retted jute	59.52	401.32	438.15	endo	exo	endo
MAH Retted jute	53.53	343 07	361.91	endo	exo	endo
NaOH Retted jute	59.05	358 62	401.09	endo	endo	exo
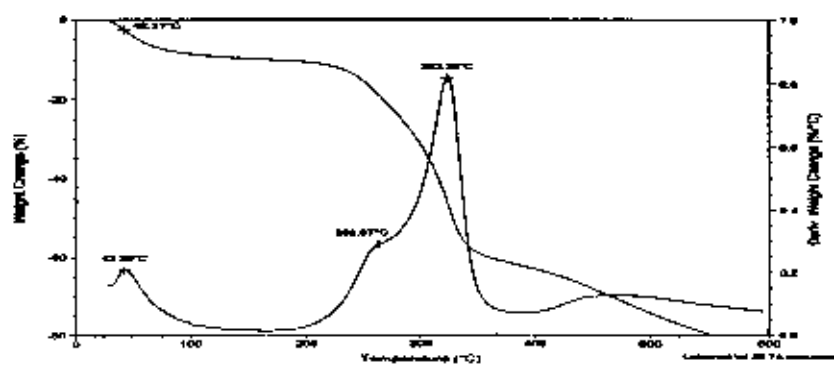
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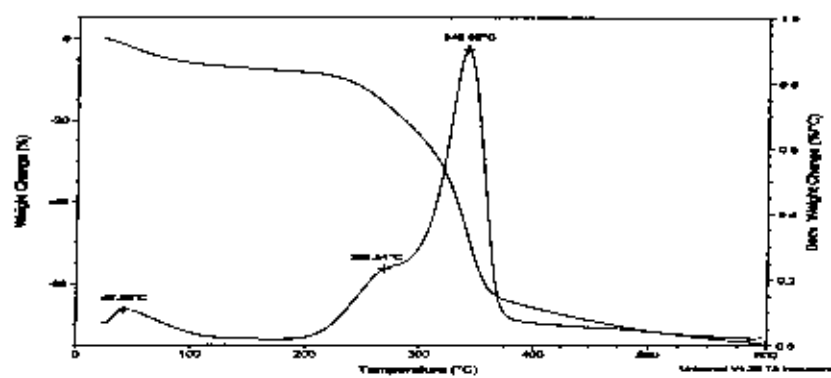
(b)
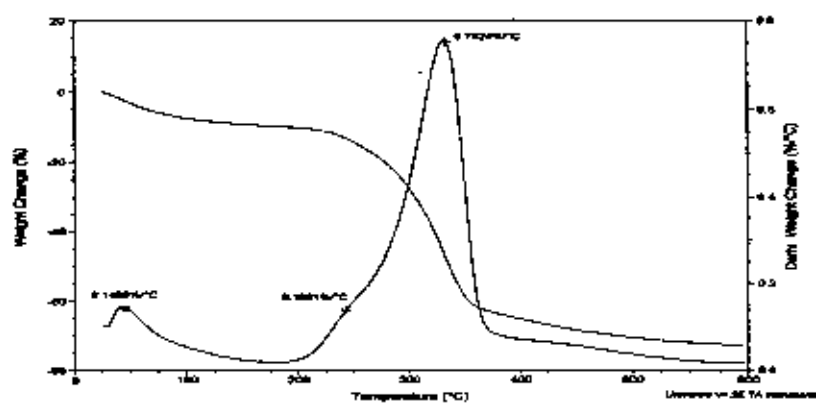
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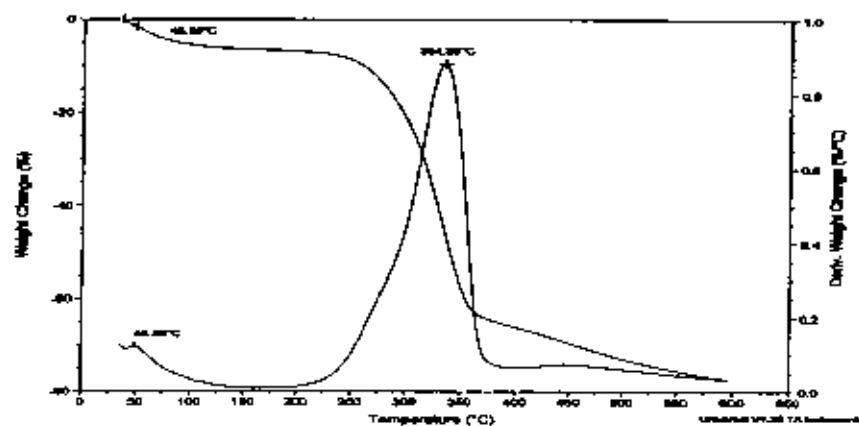
(d)
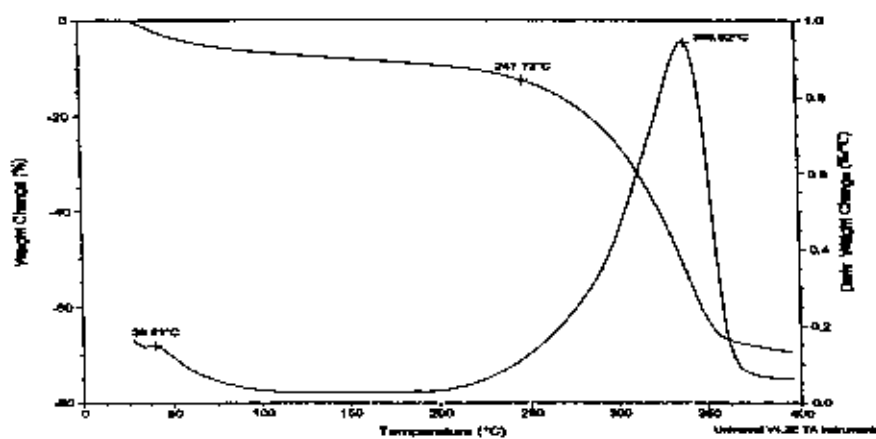
(e)
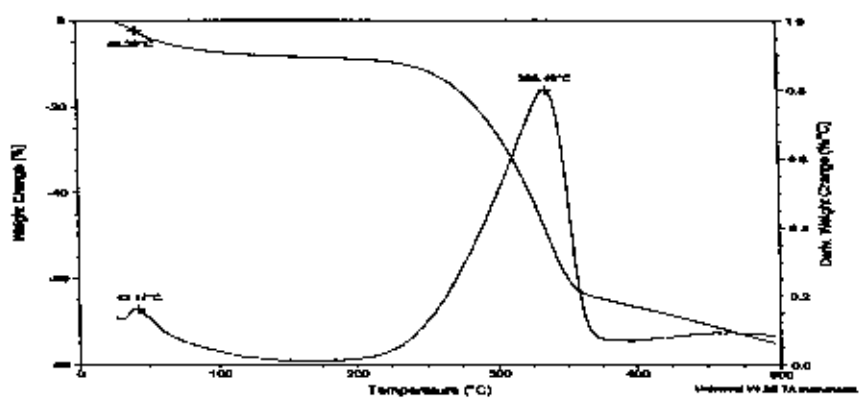
(f)

Figure 4.13 TG/DTG curve of (a) raw (b) MAH raw (c) NaOH raw (d) retted (e)
MAH retted (f) NaOH retted jute fiber.
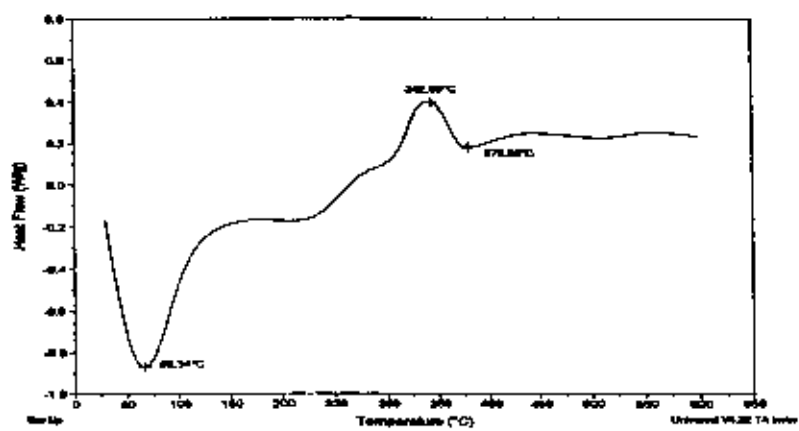
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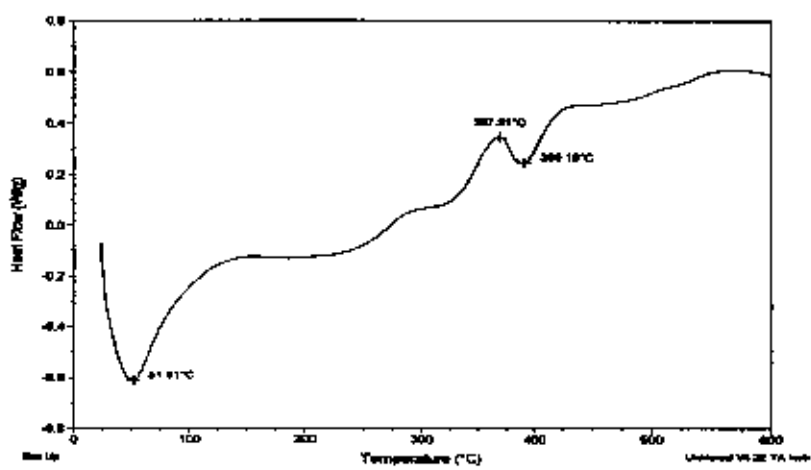
(b)
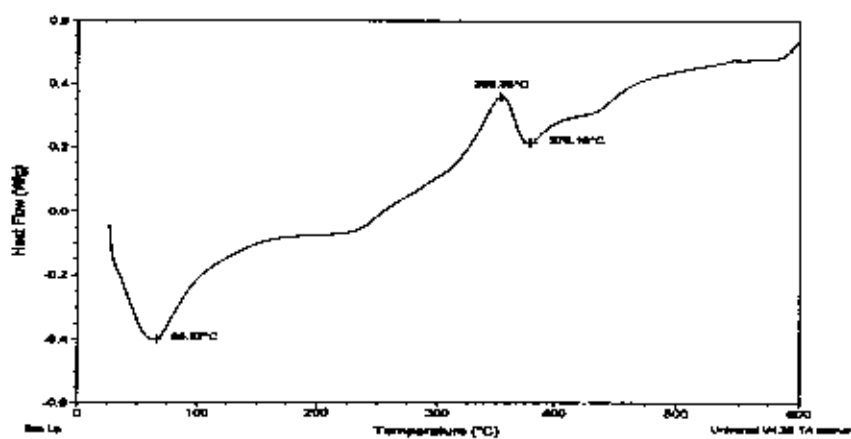
(c)
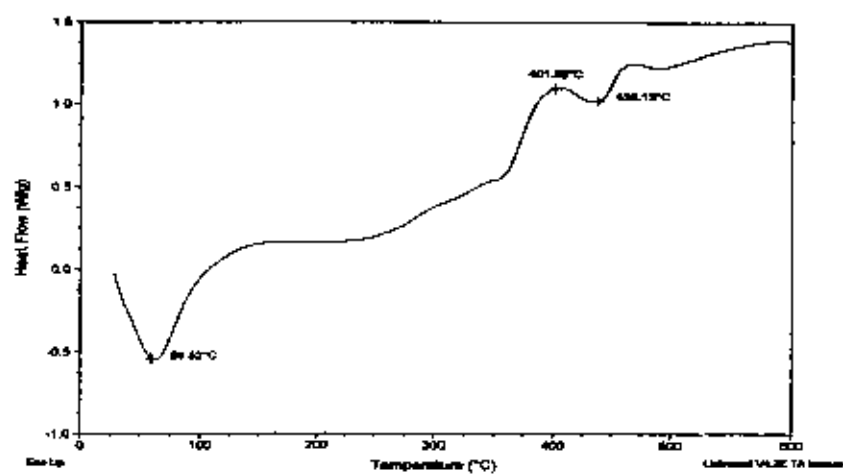
(d)
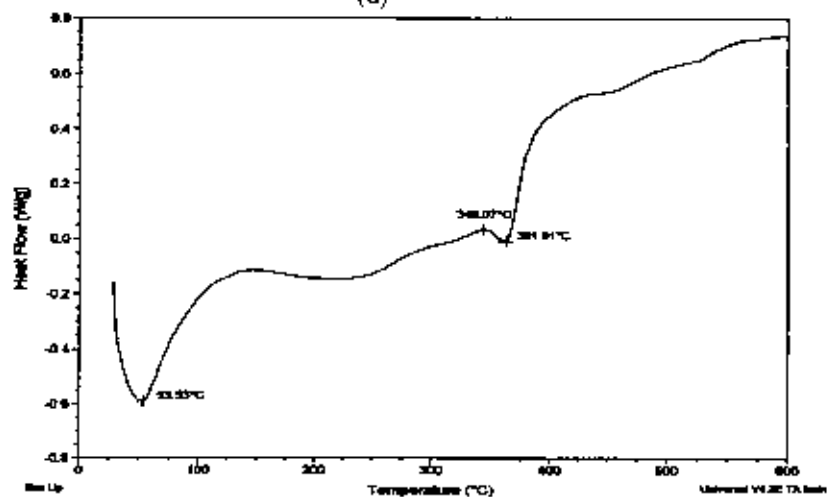
(e)

Figure 4.14 DSC curve of (a) raw (b) MAH raw (c) NaOH raw (d) MAH retted (e) NaOH retted jute fiber.

4.7. THERMAL ANALYSIS OF JUTE –PP COMPOSITES

Figure 4.15 and 4.16 shows the TG/DTG and DSC curve of Jute polypropylene composites respectively. Alkali treatment reduced the hemicellulose to a considerable extent giving rise to a lignin-cellulose complex, thereby making the product more stable (457.03 °C) than the untreated sample (442.65 °C). Final degradation temperatures for composites are higher than those of jute fibers revealed in table 4.5 and 4.6. At elevated temperature agro based fibers burn because the cell wall polymers undergo pyrolysis reactions with increasing temperature to give off volatile, flammable gases The hemicellulose and the cellulose polymers are degraded by heat much before the lignin is. The lignin component contributes to char formation, and the charred layer helps insulate the composites from further thermal degradation. Table 4.6 shows MAH treated jute composites showed higher decomposition temperature for 1st peak whereas NaOH treatment lowers this peak temperature for retted jute. All composites showed lower 2nd decomposition temperature than that of jute fibers.

Table 4 5. TGA data of jute pp composites

Sample	1st Peak Temp (°C)	2nd Peak Temp (°C)	3rd Peak Temp (°C)	Weight loss 1st peak, %	Weight loss 2nd Peak,%	Weight loss 3rd Peak,%
Raw jute PP	39 66	322.34	439.97	0 7423	15.83	72 33
MAH raw jute PP	54 25	347.87	454.57	0.7067	12.59	56 88
NaOH raw jute PP	42.39	329 64	448.18	0.8558	11 73	74.42
Retted jute PP	49.69	346.05	442.71	0.5296	12 05	77.80
MAH Retted jute PP	143.61	350.61	455.48	1.606	9.937	77.16
NaOH Retted jute PP	46.04	344.23	457.30	0.7287	15.98	71.52

Table 4.6. DSC data of composites

Sample	1st Peak Temp (°C)	2nd Peak Temp (°C)	3rd Peak Temp (°C)	Nature of 1st peak	Nature of 2nd peak	Nature of 3rd peak
Raw jute PP	52.67	163.18	441.58	endo	endo	endo
MAH raw jute PP	83.51	162.32	451.86	endo	endo	endo
NaOH raw jute PP	58.67	162.32	459.57	endo	endo	endo
Retted jute PP	53.53	162.32	458.71	endo	endo	endo
MAH Retted jute PP	158.89	358.49	459.57	endo	endo	endo
NaOH Retted jute PP	49.25	164.89	454.43	endo	endo	endo
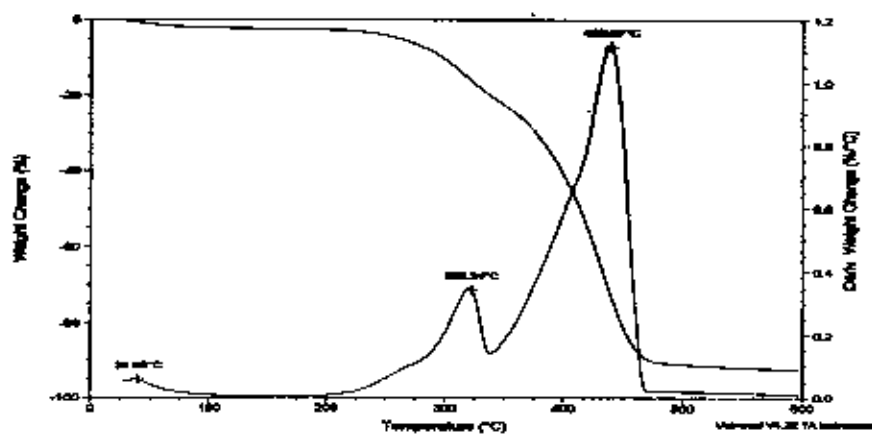
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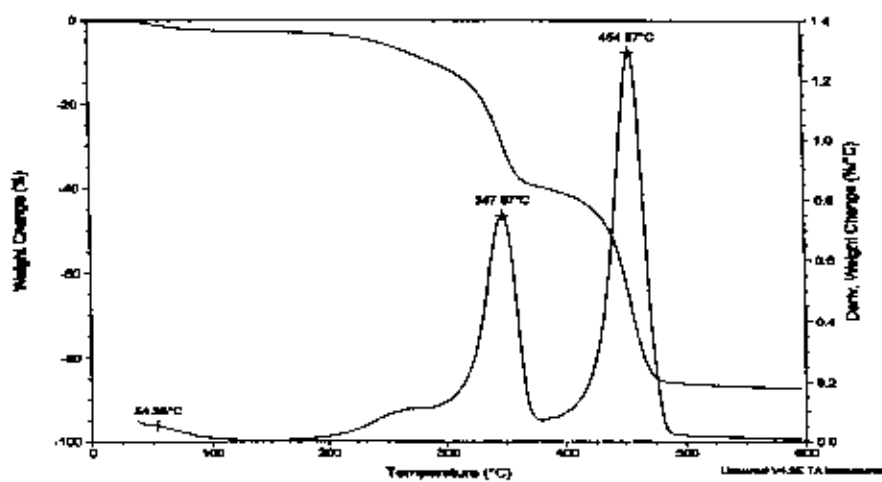
(b)
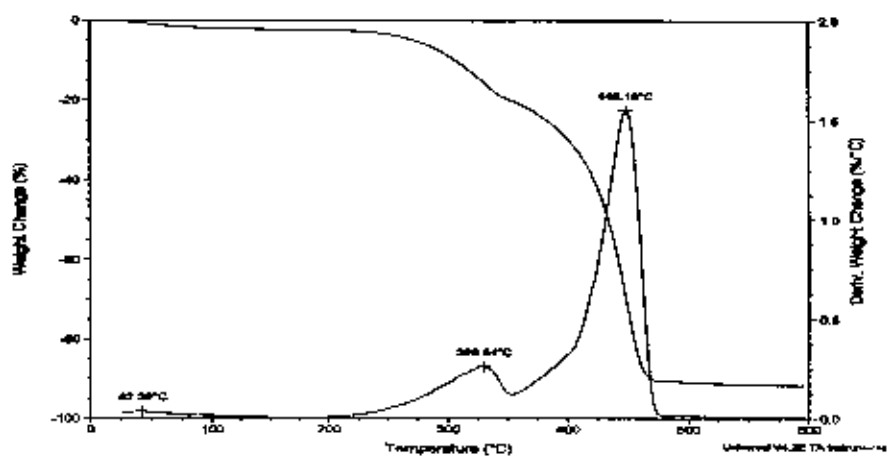
(c)
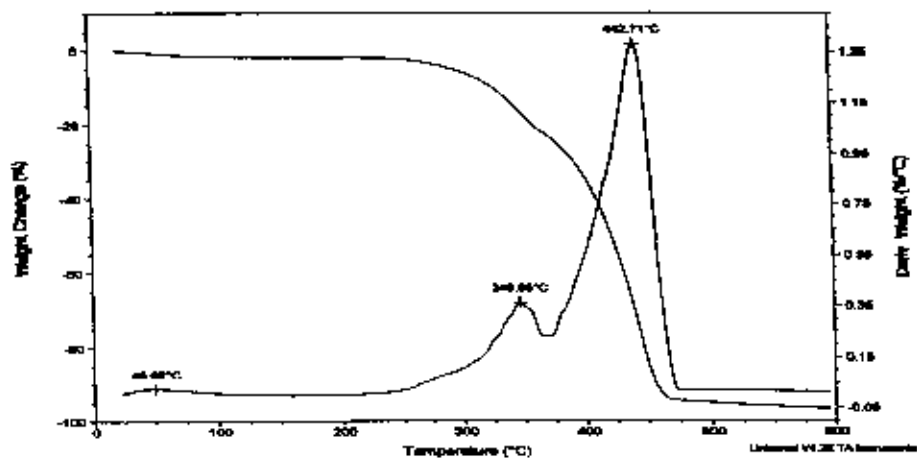
(d)
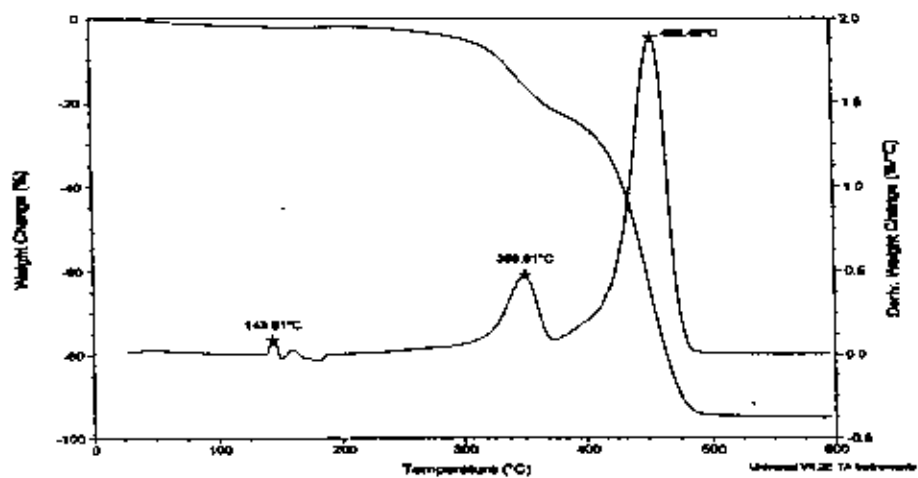
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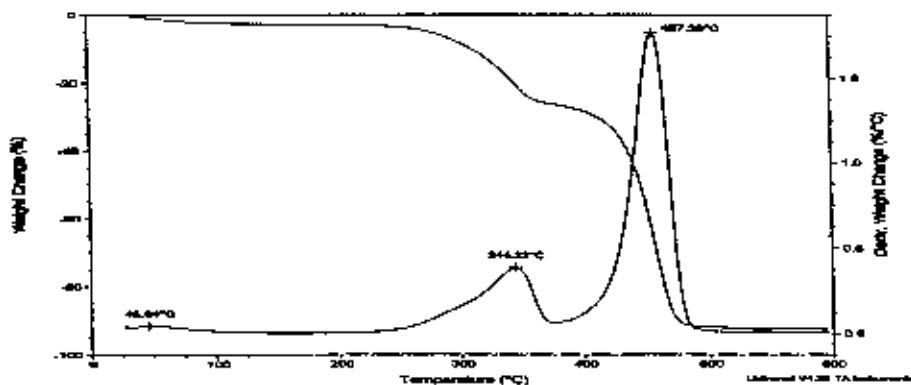
(f)

Figure 4 15 TG/DTG curve of (a) raw (b) MAH raw (c) NaOH raw (d) retted (e) MAH retted (f) NaOH retted jute fiber Polyprolene composites
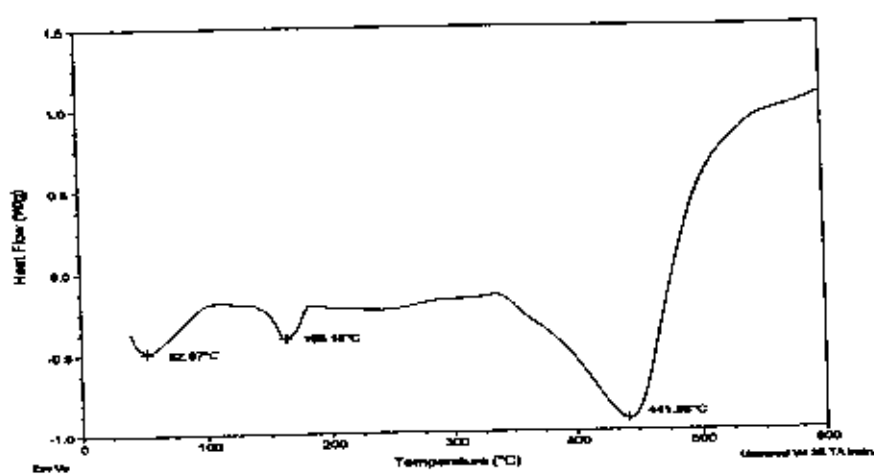
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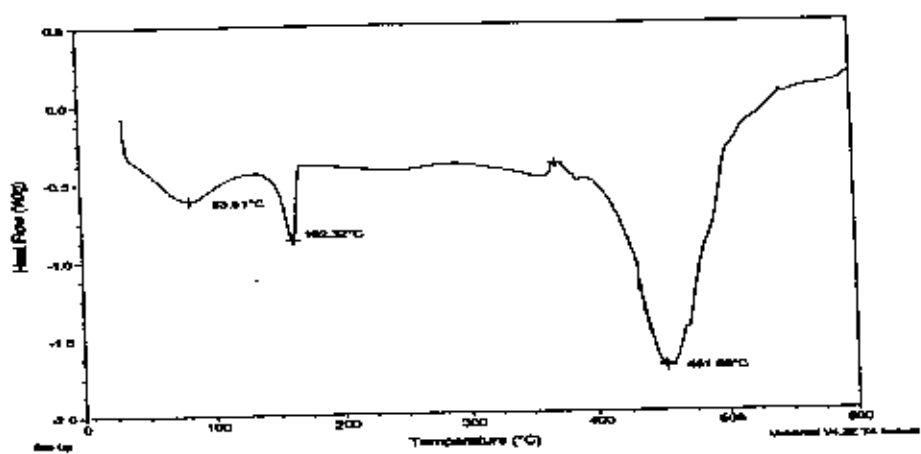
(b)
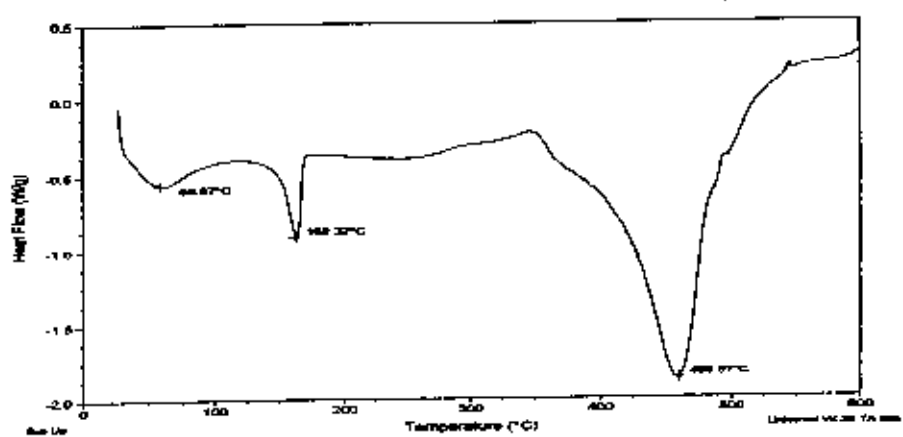
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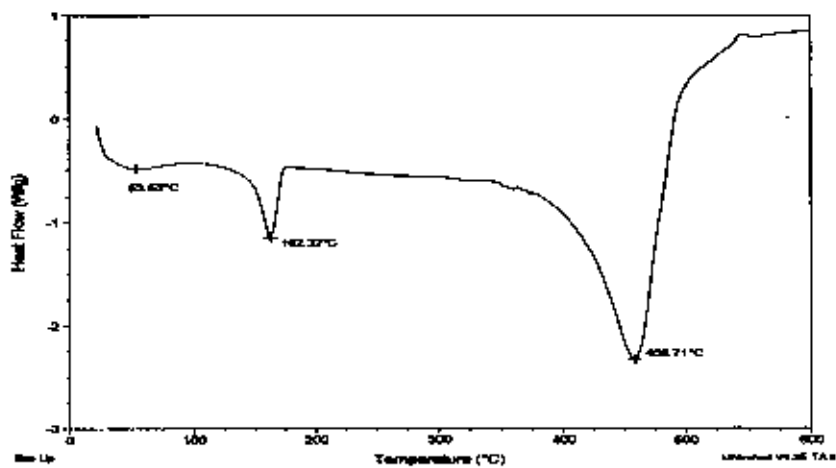
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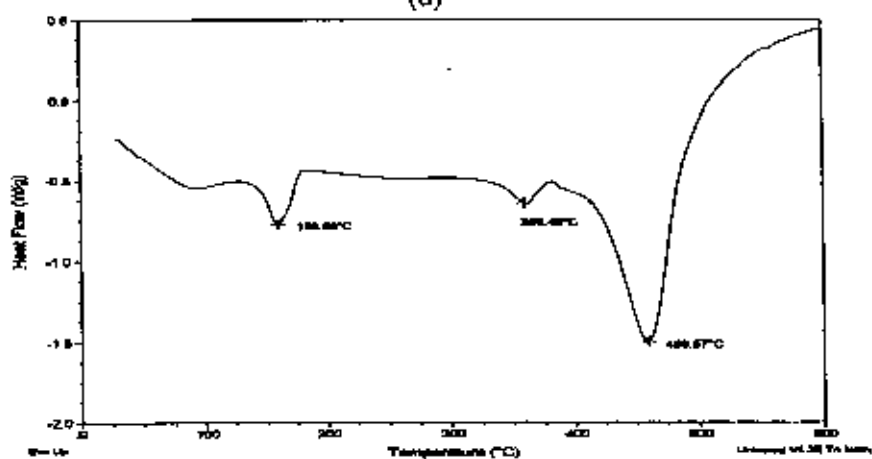
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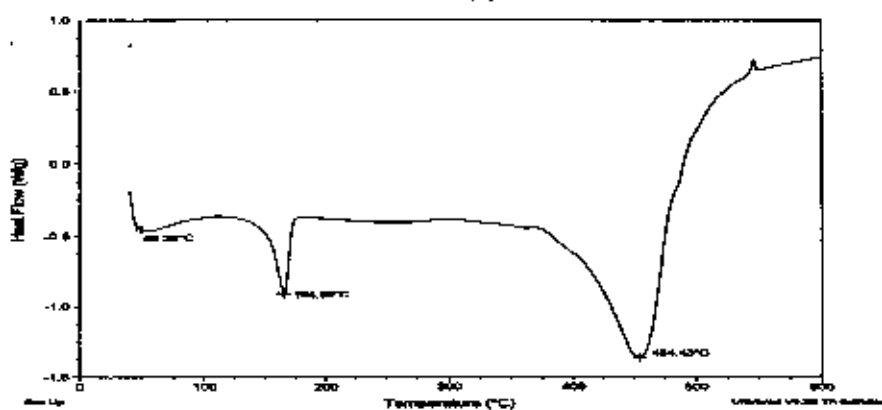
(f)

Figure 4 16 DSC curve of (a) raw (b) MAH raw (c) NaOH raw (d) retted (e) MAH retted (f) NaOH retted jute fiber polypropylene composites.

4.8 TENSILE TESTING OF COMPOSITES

Unlike the situation with metals, the failure of fiber reinforced polymer matrix composites under loading condition has, until quite recently, been poorly understood Due to the inherently anisotropic structure of the material, its strength under certain loading conditions critically depends on how well the loads match the fiber directions in the material under test Interfacial behavior between the fibers and polymer matrix has long been recognized as a key factor influencing the overall properties of composites materials. Fiber-matrix interfacial phenomenon control stress transfer between fiber and matrix, stress redistribution as well as mechanisms of damage accumulation and propagation. Generally, composite materials with weak interfaces have relatively low strength and stiffness but high resistance to fracture if crack grows perpendicular to the fibers, whereas materials with strong interfaces have high strength and stiffness but are somewhat brittle.

Since the focus of the project was to develop natural fiber reinforced composite in the prospect of Bangladesh so both raw jute fiber and retted jute fiber composited were tested to compare better option. This section comprises of the tensile test results concerning (ASTM D 3039/ D 3039M) the chopped raw jute fiber and retted jute fiber composite. For each data point minimum 5 samples were tested and the value given is the mean of those results

4.8.1 Tensile Properties of Chopped Retted Jute Fiber Reinforced Composite

The following section comprises of the presentation of the tensile test data of chopped retted jute fiber reinforce polypropylene composite. Table 4 7 represents the tensile property data summary of three sizes of chopped jute fiber reinforced polypropylene composite.

Table 4.7. Data summary of tensile test of chopped jute fiber reinforced polypropylene composite

Name	Symbol	Fiber size	Volume %	Average tensile strength, MPa	Average tensile stiffness, MPa	%Elongation
Reference polymer	Ref PP	--	0	28.5 ± 0.43	1581 ± 103	8.46
chopped retted	R PP	5	25	28.6 ± 2.5	2937 ± 1118	2.23
			20	25.7 ± 1.6	3117 ± 550	1.66
			15	25.2 ± 0.82	2906 ± 1302	2.47
			10	19.4 ± 1.9	2015 ± 214	2.47
		4	25	27.1 ± 2.24	4305 ± 1507	1.15
			20	21.4 ± 2.13	3313 ± 1473	1.83
			15	22.9 ± 1.7	2458 ± 750	2.07
			10	18.8 ± 0.99	2024 ± 295	2.21
			25	22.6 ± 0.72	3694 ± 1657	1.74
		2	20	19.2 ± 0.99	2680 ± 1107	1.96
			15	19.5 ± 0.98	2955 ± 943	2.02
			10	21.3 ± 0.96	2381 ± 430	2.58
MAH treated chopped retted jute	MAH R PP	5	25	16.3 ± 2.1	3209 ± 1017	0.94
			20	17.8 ± 1.6	6625 ± 3090	0.79
			15	20.9 ± 0.34	3600 ± 1011	1.55
			10	21 ± 2.3	3063 ± 341	2.4
		4	25	11 ± 2.4	4717 ± 2054	0.65
			20	14 ± 0.97	5783 ± 3171	0.8
			15	20 ± 0.53	2592 ± 341	2.34
			10	19.5 ± 0.14	2812 ± 842	2.12
		2	25	14.4 ± 0.81	6544 ± 3102	0.81
			20	15.5 ± 1.23	2385 ± 435	1.46
			15	14.5 ± 1.9	2732 ± 1088	2.05
			10	19 ± 0.51	2167 ± 652	2.70
NaOH treated chopped retted jute	NaOH R PP	5	25	24.5 ± 0.46	2318 ± 509	5.4
			20	21.7 ± 1.02	2395 ± 702	4.29
			15	24.9 ± 0.52	2632 ± 817	6.36
			10	26.3 ± 0.66	2305 ± 241	5.11
		4	25	24.3 ± 1.04	3498 ± 1080	4.56
			20	23.1 ± 0.82	2307 ± 647	4.5
			15	23.6 ± 0.726	1921 ± 270	4.24
			10	24.7 ± 1.12	2254 ± 255	4.19
		2	25	20.3 ± 1.3	2326 ± 584	3.82
			20	20.1 ± 1.02	3636 ± 1482	2.68
			15	24 ± 0.58	1883 ± 345	4.97
			10	22.5 ± 0.80	1841 ± 318	5.85

From table 4.7 it is clear that the stiffness of the composite specimen has been increased and the ductility is decreased compared to reference polymer. This may be due to ineffective load transfer between the fiber and the matrix with in these volume fractions of fibers. Composite strength is sensitive to variation in fiber length when the average fiber length was less than the critical fiber length and the degree of fiber alignment. A good trend of increasing strength is observed due to addition of increasing size of jute fiber.

Figure 4.17 shows the tensile strength of chopped retted jute fiber (both treated and untreated) reinforced polypropylene composites of different volume fraction at a fiber size of 5 mm length The strengths of composites are lower than that of the reference polymer.
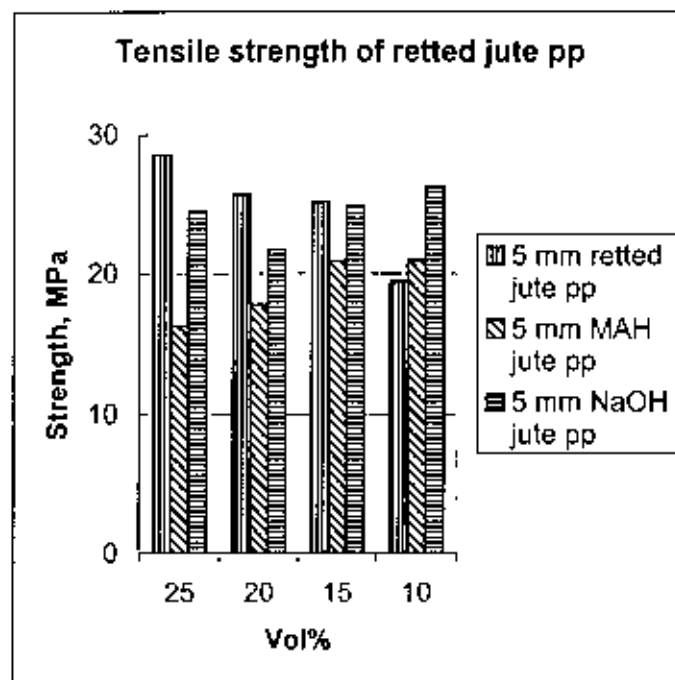
Figure 4 17 Tensile strength of retted fiber composite

Untreated retted jute polypropylene composites show maximum strength (fig 4 17). NaOH treated retted jute fiber composites show greater strength than that of MAH treated jute fiber polypropylene composites.

Figure 4 18 (a), (b) shows the comparison of tensile stress and tensile stiffness data of untreated retted jute fiber composites for three different fiber sizes. The plot reveals that the strength is greater for larger fiber length. Tensile strengths and tensile modulus of untreated retted jute fiber composites increases with increase in fiber volume percent and the increase in more prominent in case of tensile stiffness.
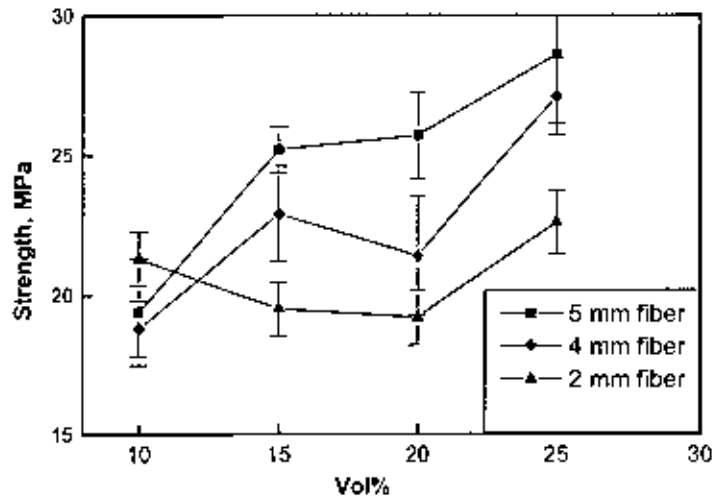
(a)
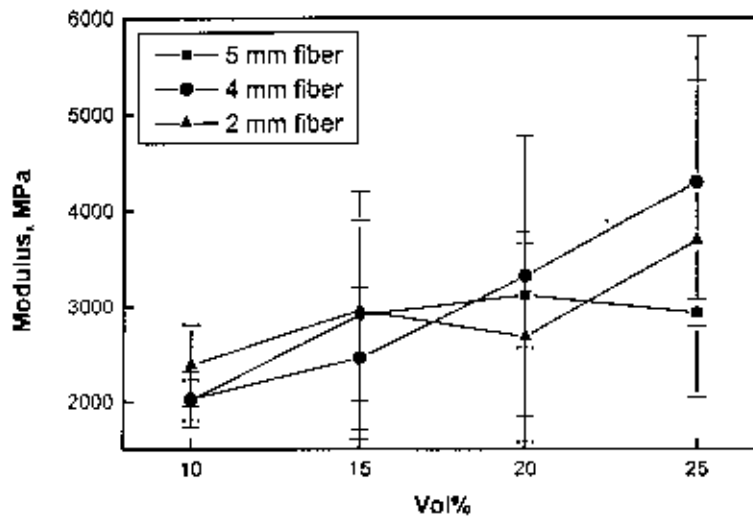
(b)

Figure 4 18 (a) Tensile strength (b) tensile stiffness of untreated retted jute fiber composites

4 19 (a), and (b) show the tensile test data for MAH jute pp composites After maleic anhydride treatment the strength decrease but stiffness increases, as the fiber volume is higher
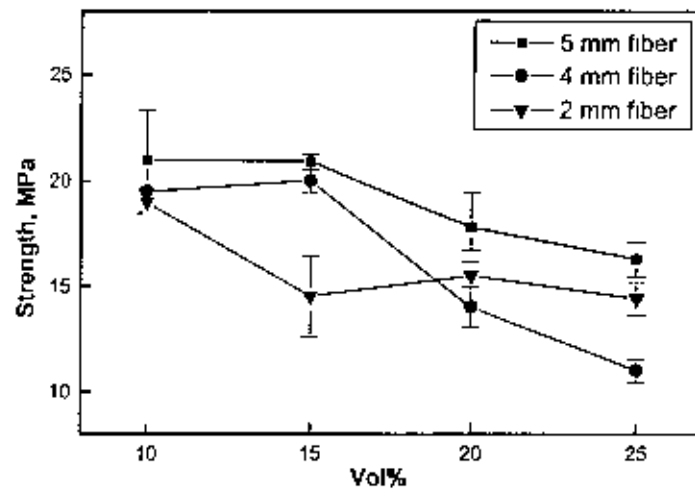
(a)
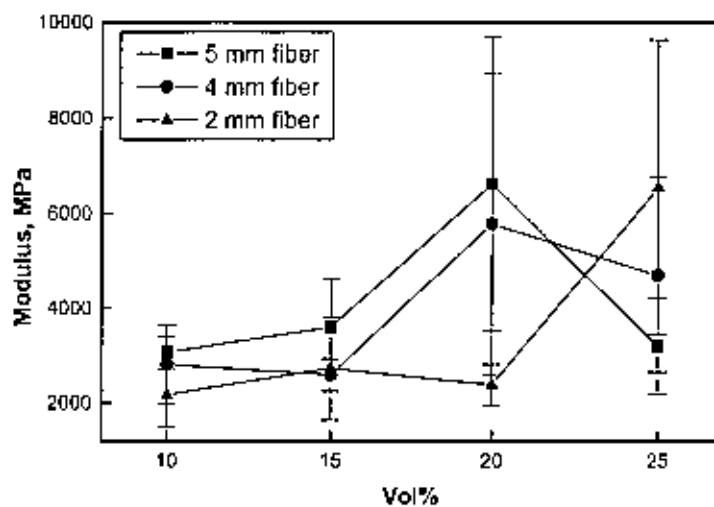
(b)

Figure 4.19: (a) Tensile strength (b) tensile stiffness MAH treated retted jute fiber composites.

In case of MAH treated retted jute pp composites low strength value is may be due to low fiber-matrix adhesion of the modified jute and polypropylene Strength of composite also decreases with fiber volume percent of jute fiber. In case of greater volume percent the

wetting of all fiber do not occur so there is a possibility of generating more flaws inside the composite system and resulting poor adhesion and lower strength.

The mechanical properties of NaOH treated retted jute fiber composites with varying proportions of fibers (10-25%) are presented in figure 4.20(a) and (b). The improvement in strength becomes more dominant with lower fiber percentage
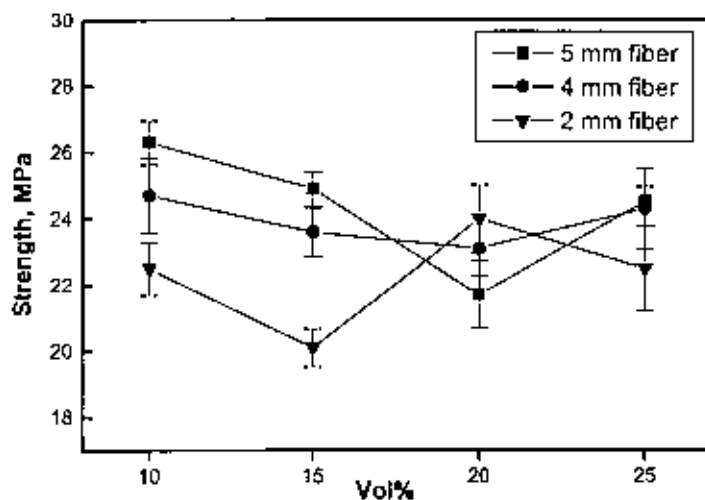
(a)
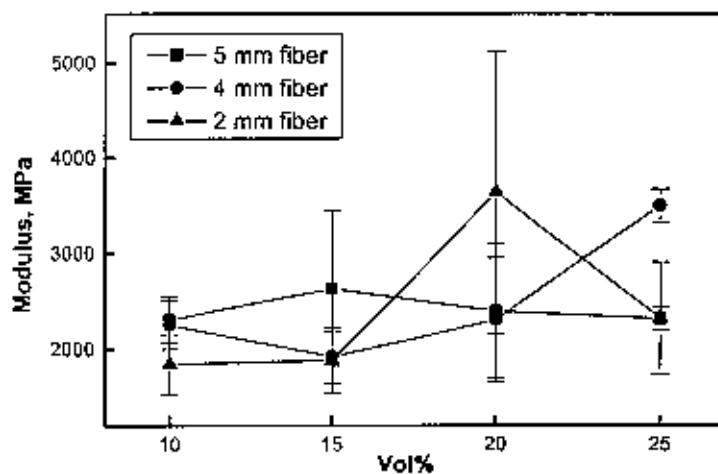
(b)

Figure 4 20. (a) Tensile strength (b) tensile stiffness of NaOH treated jute fiber pp composites.

In general NaOH treatment increase tensile strength and increased Young modulus of the composites compared to untreated jute polypropylene composites. This may be because; the alkali reacts with the cementing materials of the fiber, particularly hemicellulose. This lead to the destruction of the mesh structure of the fiber and splitting of the fibers into finer filaments. The breaking down of the fiber bundle increases the effective surface area available for wetting by the matrix. Thus bonding between the fiber and the matrix at the interface may be improved [8, 22].

Figure 4.20 shows the tensile properties of chopped NaOH treated retted jute fiber reinforced polypropylene composite. The strength value of NaOH treated jute reinforced composites increase than that of maleic anhydride treated retted jute fiber reinforced polypropylene but lower that that of untreated jute fiber composite and reference polymer. Stiffness of NaOH treated retted jute polypropylene composites are lower than that of MAH treated retted jute polypropylene composites.
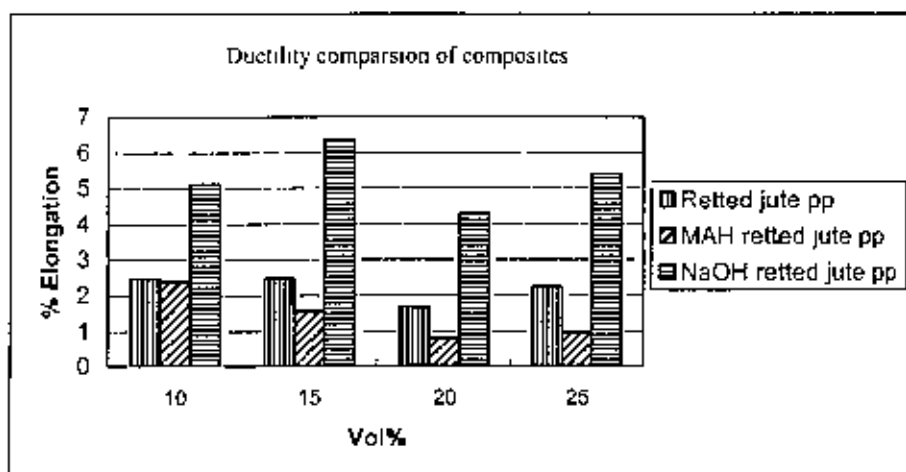
Figure 4.21. Ductility in terms of percentage elongation of composites (5 mm)

Ductility in terms of % elongation of MAH treated jute polypropylene composites decrease from that of untreated retted fiber polypropylene composite as well as reference polymer. NaOH treated retted jute increases the percentage elongation of all composites as compared to other treated and untreated retted jute composites (fig 4.21). This may be due to development of crystallinity by NaOH treatment causing higher extensibility of fibers. The pullouts of fibers from the matrix as shown in SEM fig 4 24 also contribute to the increase in percentage elongation of NaOH treated jute polypropylene composites.

4.8.2. Tensile Properties of Chopped Raw Jute Fiber Reinforced Composite

Table 4 8 and figure 4.22 and 4.23 illustrate the tensile test data of raw jute pp composites. Strength as well as elongation of treated and untreated raw jute polypropylene composite show lower value than that of treated and untreated retted jute polypropylene composites and reference polymer also. In all case raw jute polypropylene composites show greater stiffness than that of reference polymer

The prominent increase in tensile stiffness occurs in case of MAH treated raw jute pp composites (fig.4.23) and NaOH treated raw jute polypropylene composites shows greater tensile stress (fig. 4 22). MAH treatment produce rougher fiber surface in case of raw jute fiber. For rough fiber surface as in the MAH treated jute fiber it is expected that the matrix will occupy the cellular pores on the fiber surface resulting more mechanical bonding between matrix and fiber

Table 4.8: Data summary of tensile test of raw jute pp composites

Name	Symbol	Fiber size	Volume %	Average tensile strength, MPa	Average tensile stiffness, MPa	% Elongation
Ref polymer	Ref PP	--	0	28.47 ± 0.43	1581 ± 103	8.46
Raw jute pp	Raw jute-PP	5	25	16.72 ± 0.64	2925 ± 462	1.43
MAH Raw Jute pp	MAH raw pp	5	25	12.09 ± 0.6	5410 ± 2132	0.86
NaOH raw jute pp	NaOH raw pp	5	25	19.56 ± 0.91	2871 ± 590	2.1

Tensile strength of composites
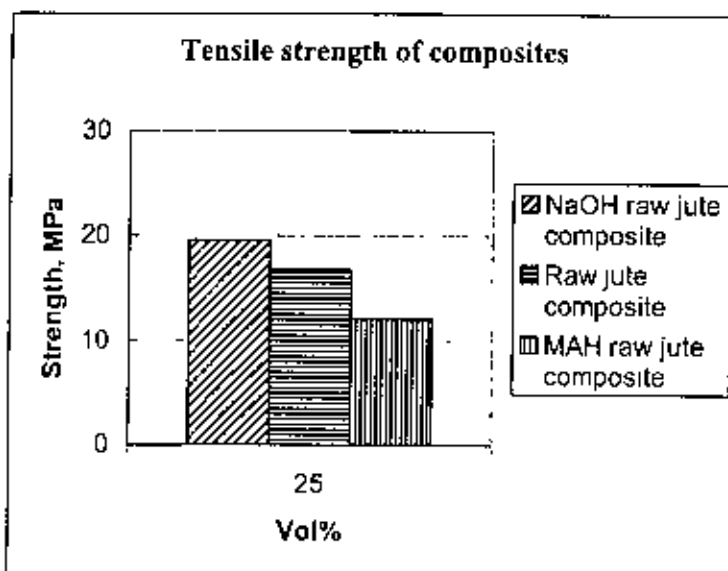
Figure 4.22 Tensile strength of raw jute polypropylene composites

Tensile stiffness of composite
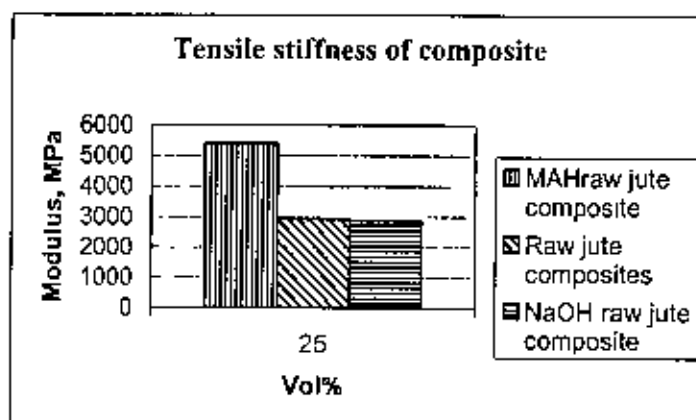
Figure 4 23 Tensile stiffness of raw jute polypropylene composites

4.9 SEM OF TENSILE FRACTURE SURFACE OF CHOPPED JUTE FIBER COMPOSITES

Scanning electron microscopy of the fracture surface of tensile specimens indicate that there is considerable difference in the fiber-matrix interaction between the treated and untreated jute fiber composites. Figure 4 24 shows the fracture surface of a retted jute fiber reinforced composite with some fiber pullout and clean fiber surface when the fibers are aligned to

tensile axis of composites, but in some fracture area tensile separation between the matrix and the fibers are observed when fibers are parallel to tensile force.

Figure 4.25 (a) and (b) are the scanning electron micrograph of NaOH treated retted jute fiber polypropylene composites. A large number of fibers have pulled out from the matrix and considerable amount of polymer residue remains on the fiber surface.

Fracture of fibers is observed in case of raw jute polypropylene composites (fig 4.26). Fiber pullout also occurred in case of raw jute polypropylene composites as shown in figure 4.27(a) and (b) Splitting of raw fibers occurs during processing of fibers, which are appearing in fig 4.27(c). MAH treatment improves the wettability of raw jute surface by the polymer. The improved bonding between fiber surface and polypropylene and a fair amount of polymer residue adheres on the fiber is clearly seen in fig 4.28 (a) and (b). This improved wettability due to increase in microroughness of MAH treated jute fibers as shown in figure 4 2.

The failure of tensile specimen of the chopped jute fiber reinforced composite showed directional behaviour according to the fiber orientation. This implies that the interfacial strength between the fiber and the matrix is lower. The failure initiated at the site where presence of micro flaw is larger and where the fiber orientation is parallel to the applied load.

The tensile failure of fiber-reinforced composites is generally dominated by failure of the fiber bundle. The matrix material, serves mainly to transfer the load among the fibers through the elasticity, or yielding, debonding with sliding friction between the fiber and matrix. The matrix can carry some load in a polymer matrix composite but, after matrix cracking, carries almost zero load in composites. The two factors controlling fiber failure are (1) the statistical fiber length and (2) the stress distribution along the fiber direction. The stress along a fiber depends on the applied stress, but also on precisely how stress is transferred from a broken fiber to the surrounding intact fibers and matrix environment. The stress transfer is governed by the elastic properties of the constituents and by the fiber/matrix interface, and is difficult to obtain in the presence of more than one broken fiber. [19].
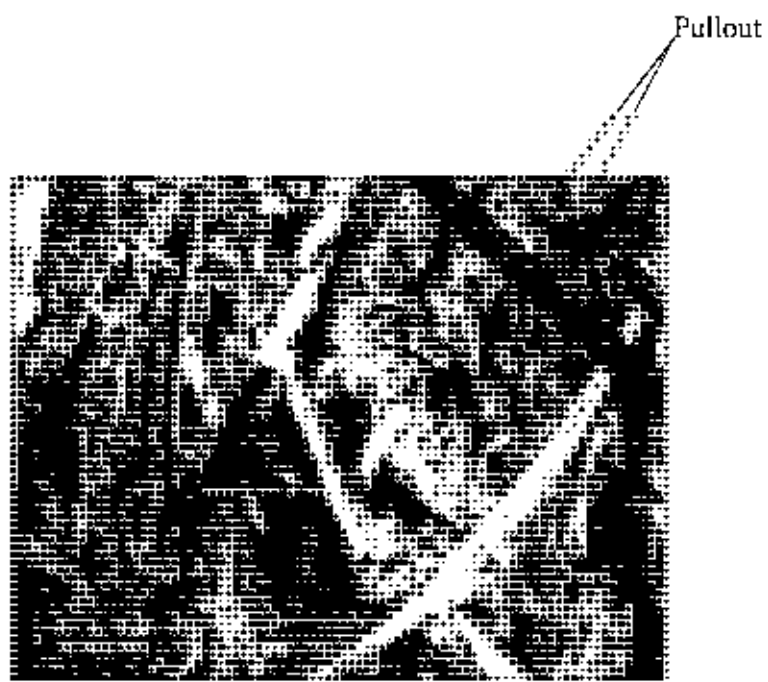
Figure 4.24 SEM figure of fracture surface of a retted jute pp composite

Fiber
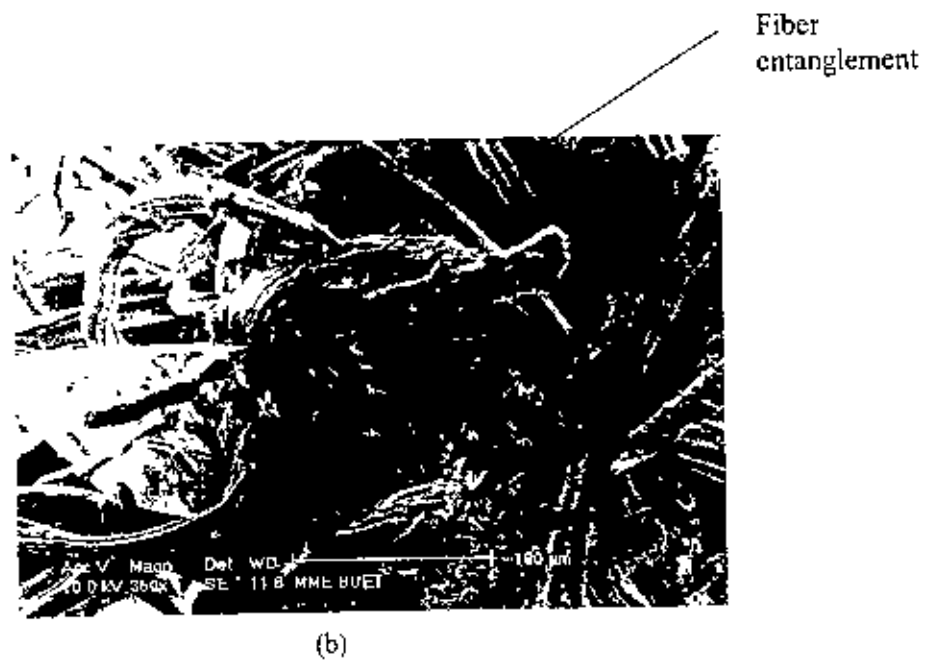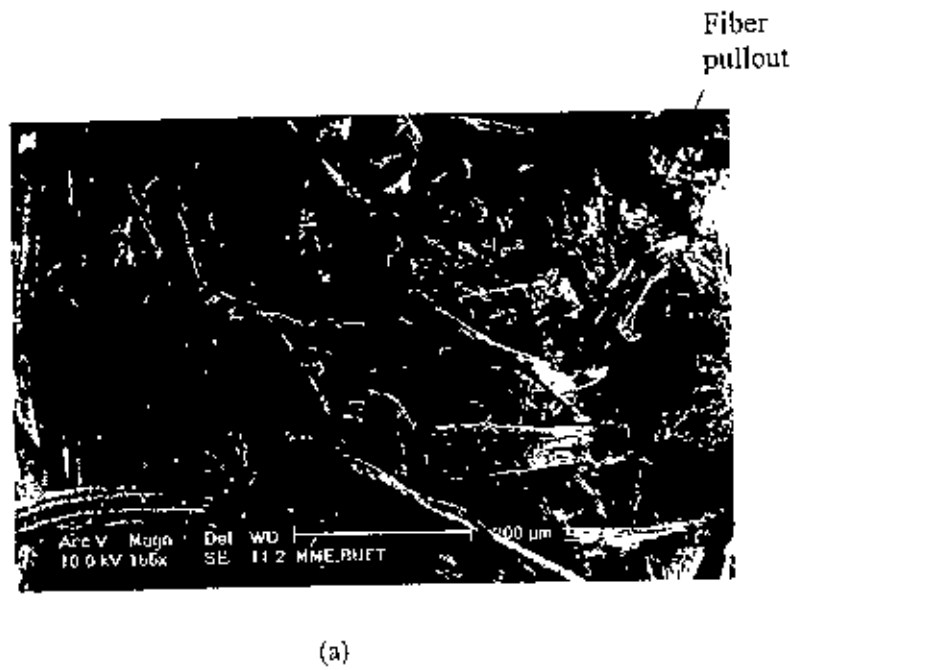pullout

(a)

Fiber
entanglement

(b)

Figure 4.25. SEM micrographs of fracture surface of NaOH treated retted jute pp composites show (a) fiber pullout (b) fiber entanglement on the tensile fracture surface.
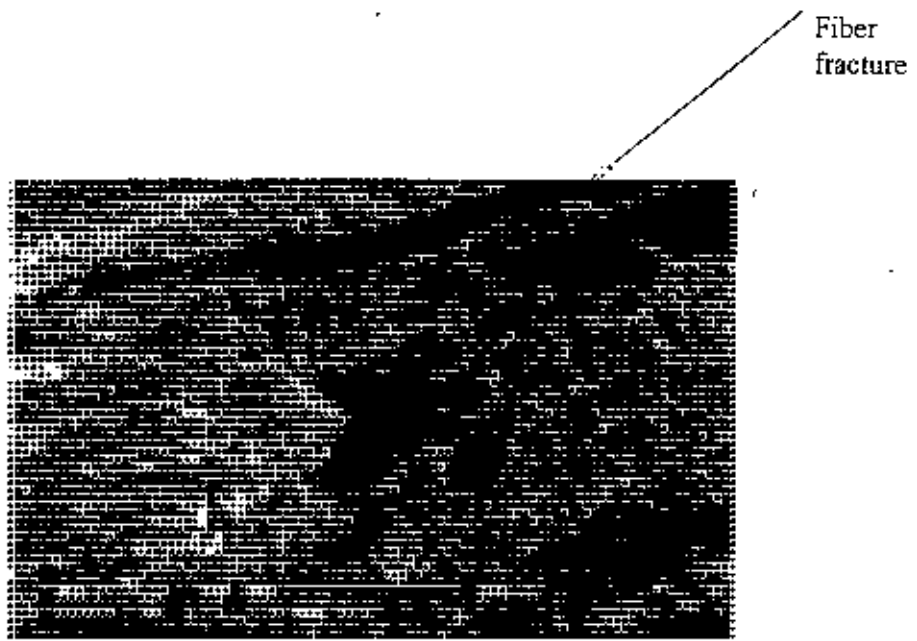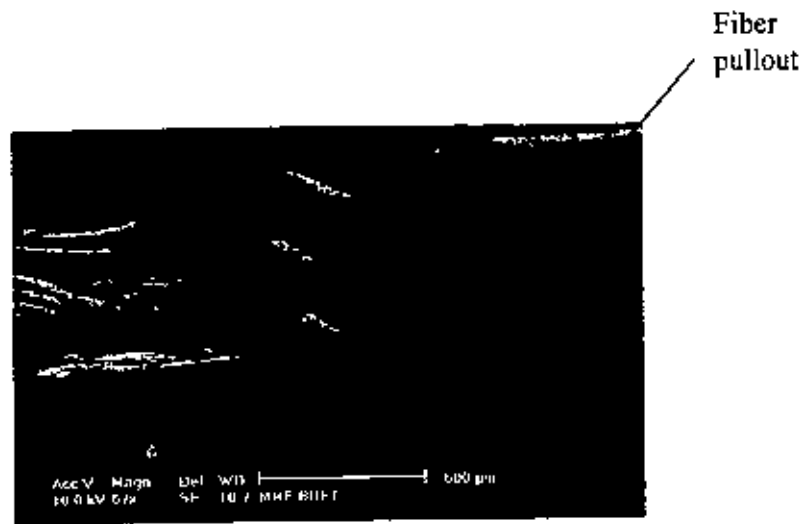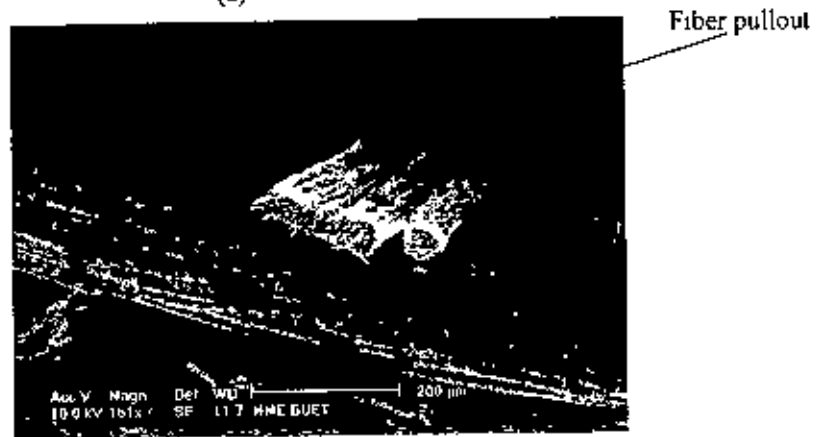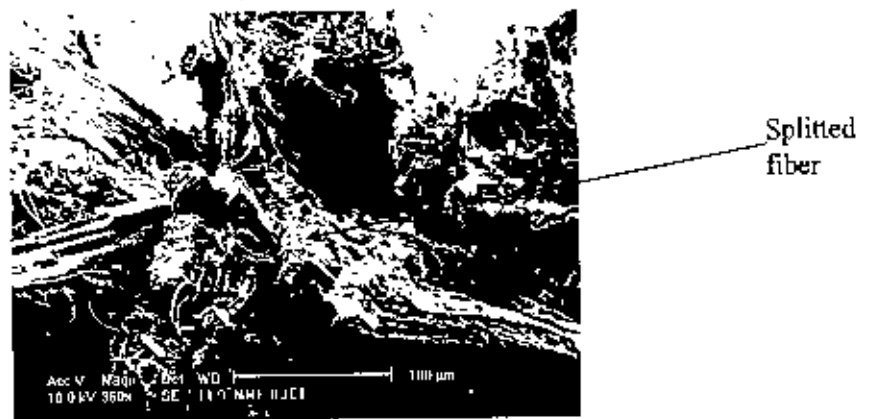
Fiber
fracture

Figure 4.26 SEM micrograph of raw jute pp composites

Fiber
pullout

(a)

Fiber pullout

(b)

Splitted
fiber

(c)

Figure 4.27 SEM micrographs of NaOH raw jute pp composites showing (a) and (b) fiber pullout and (b) splitted fiber on tensile fracture surface

(a)

(b)
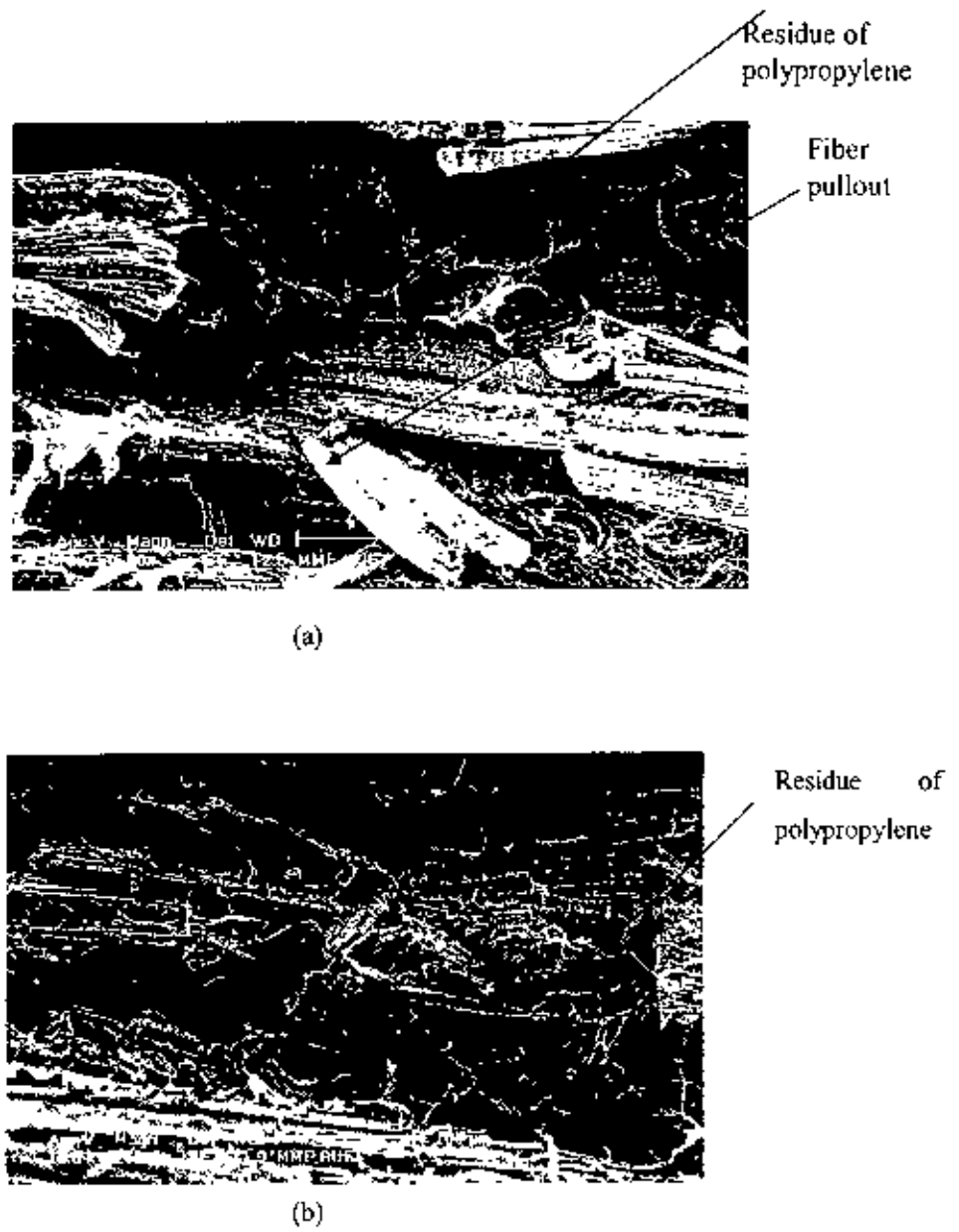
Figure 4.28 SEM micrographs of MAH raw jute pp composites showing (a) fiber pullout and (b) residue of polypropylene on fiber surface

4.10. FLEXURE PROPERTIES OF CHOPPED RETTED JUTE FIBER POLYPROPYLENE COMPOSITE

Table 4.9 shows the flexure test data summary of the chopped retted jute fiber reinforced polymer composite. For each data point minimum 5 samples were tested and the values given are the mean of these results

The flexure modulus of composites was comparatively lower than the corresponding tensile modulus of composites. These two loading conditions exhibit different kind of stresses in the specimen under test. Whereas the stresses in a tensile test are uniform throughout the specimen cross-section, the stress in flexure varies from zero in the middle to maximum in the top and bottom surfaces. The simple tension and flexure moduli measurement can differ significantly when the material is in homogeneous and anisotropic. A flexure test is highly influenced by the properties of the specimen closest to the top and bottom surfaces whereas a simple tension test reflects the average property through the thickness.

Table 4.9. Data summary of flexure test of chopped retted jute fiber reinforced polypropylene composite

Name	Symbol	Fiber size	Volume %	Average flexure strength, MPa	Average Flexure stiffness, MPa
Reference polymer	Ref PP	--	0	48.6 ± 2.8	1683 ± 115
Chopped retted	R PP	5	25	42.4 ±1.5	2233 ± 373
			20	50.3 ± 2.6	2512 ± 140
			15	45.3 ± 2.4	1789 ± 181
			10	48.8 ± 3.7	1724 ± 238
		4	25	44 ± 6.1	1737 ± 245
			20	47.9 ± 2.3	2037 ± 187
			15	44.9 ± 4.7	1812 ± 302
			10	45.2 ± 4.3	1868 ± 173
			25	44.4 ± 1 9	2063 ± 269
		2	20	43.3 ± 3.13	1968 ± 104
			15	39.2 ± 2.6	1612 ± 201
			10	41.9 ± 3.7	1613 ± 112
MAH treated chopped retted jute	MAH R PP	5	25	34.6 ± 4.1	2159 ± 317
			20	36.8 ± 2.5	1742 ± 218
			15	41 9 ± 2.1	2567 ± 206
			10	41 5± 2.1	2748 ± 502
		4	25	35 ± 2 5	2185 ± 418
			20	37.4 ± 6.5	2175 ± 287
			15	40.4 ± 1.9	2145 ± 392
			10	38.3 ± 4.7	1737 ± 89
		2	25	38.7 ± 1.72	1905 ± 295
			20	31.6 ± 4 15	1715 ± 313
			15	36.7 ± 3.5	1960 ± 368
			10	38 4 ± 1.9	2015 ± 198
NaOH treated chopped retted jute	NaOH R PP	5	25	41.9 ± 4	1658 ± 184
			20	44.5 ± 1.4	1813 ± 309
			15	38.4 ± 0.63	1804 ± 119
			10	37.9 ± 2	1764 ± 94
		4	25	44 ± 6.02	1736 ± 245
			20	42.6 ± 3.7	1904 ± 129
			15	45.6 ± 4.9	1877 ± 243
			10	40.6 ± 2.3	1674 ± 122
		2	25	41.3 ± 1.5	1566 ± 292
			20	43.8 ± 1.5	1618 ± 229
			15	36.5 ± 1.6	1638 ± 154
			10	36.8 ± 2 6	1708 ± 76
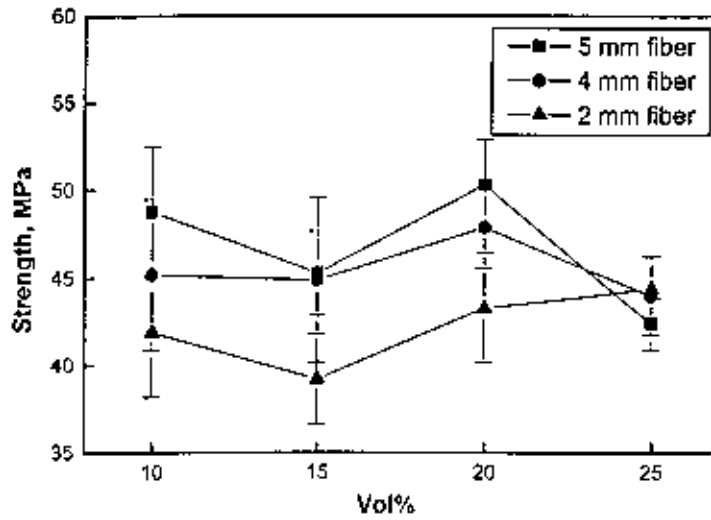
(a)
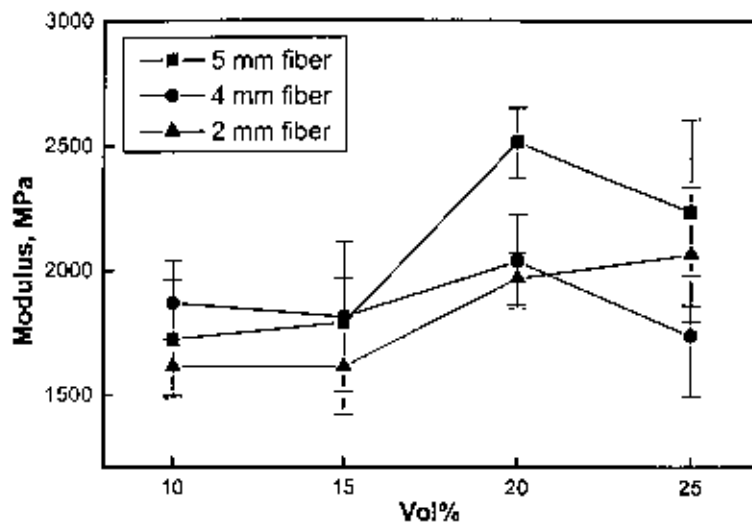
(b)

Figure 4.29. (a) Flexure strength (b) flexure stiffness of retted jute pp composites

The flexure stresses of composites are always lower than that of reference polymer but improvement of stiffness in all composites are well noted. The stress value in some case lower for greater fiber volume percentage (25%). This may be due to fiber agglomeration. The probability of fiber agglomeration increases at higher fiber content, creating region of microscopic flaws (stress concentration) that requires less energy to initiate or propagate a crack

Figure 4 29 (a), (b) shows the flexure stress and flexure stiffness of untreated retted jute fiber polypropylene composites. The other composites such as MAH treated jute fiber composites and NaOH treated jute fiber composites follow the similar trend
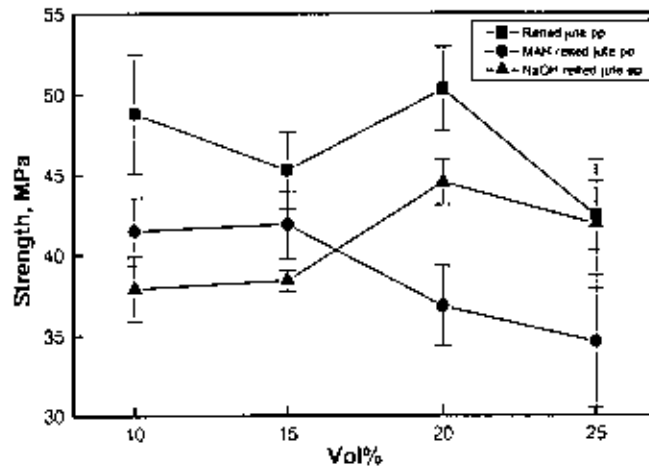
Figure 4 30 Flexure strength comparison of three types composites (5 mm fiber size)

Figure 4.30 shows the flexure stress comparison of three types composites reinforced with retted fiber (5 mm fiber size) in untreated and treated condition. Untreated retted jute pp composites show comparatively better flexure strength.

Comparison of flexure stiffness of three types retted jute fiber (fiber size 5 mm) composites is presented in fig 4.31. MAH treated jute fiber composites show higher flexure stiffness. The increase in flexure modulus may be due to increase in polypropylene adherence to MAH treated rougher fiber surface
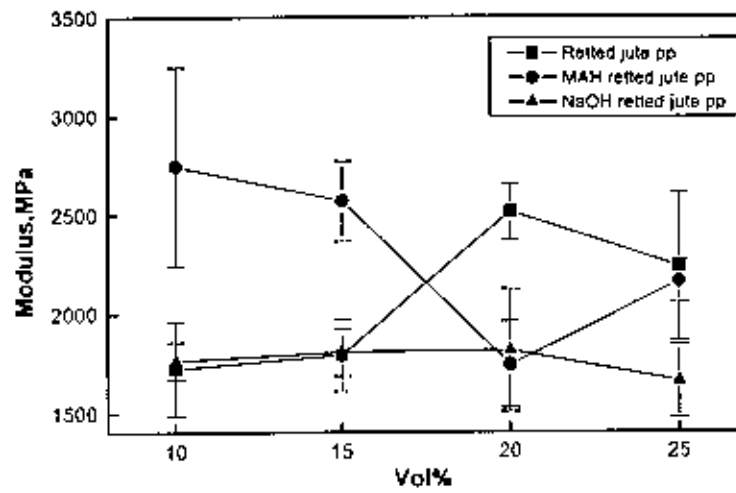
Figure 4.31. Flexure stiffness of three types composites (5 mm fiber size)
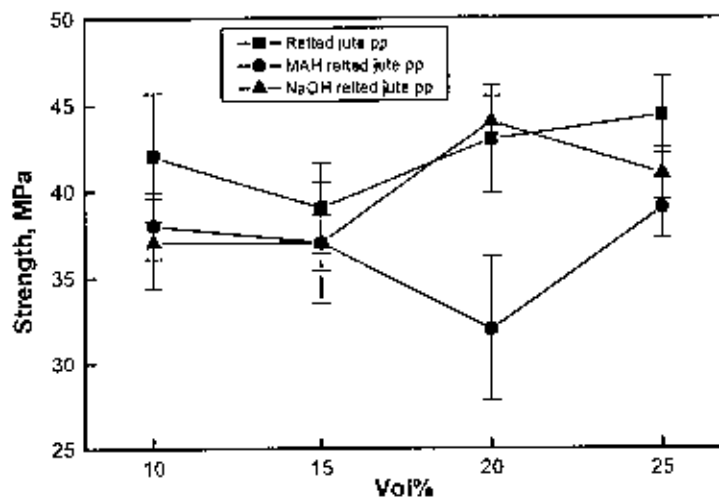
Figure 4.32 Flexure strength of three types composites (2 mm fiber size)
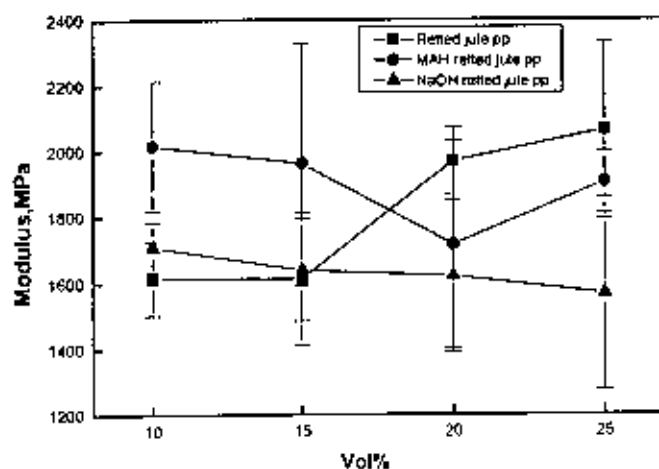
Figure 4.33 Flexure stiffness of three types composites (2 mm fiber size)

For 2 mm retted jute fiber, NaOH treated retted jute fiber composites show greater flexural stress (figure 4 32) and MAH treated retted fiber jute pp composites shows greater flexural stiffness (figure 4.33).

4.11. FLEXURE PROPERTIES OF CHOPPED RAW JUTE FIBER POLYPROPYLENE COMPOSITE

The flexure properties of untreated and treated raw jute polypropylene composites are summarize in table 4.10.

Table 4.10. Data summary of flexure test of chopped raw jute fiber reinforced polypropylene composite

Name	Symbol	Fiber size	Volume %	Average flexure strength, MPa	Average Flexure stiffness, MPa
Raw jute	Raw jute PP	5	25	33.6 ± 4.2	1908 ± 150
MAH Raw jute	MAH Raw jute PP	5	25	29 ± 2.1	1884 ± 94.31
NaOH Raw jute	NaOH Raw jute PP	5	25	36.9 ± 2.1	1972 ± 115.1

Figure 4.34(a), (b) represent the flexure stress and flexure stiffness of raw jute fiber polypropylene composites. NaOH treated jute pp composites shows the maximum flexure stress and stiffness. In general NaOH treatment increase flexure modulus [8, 22].
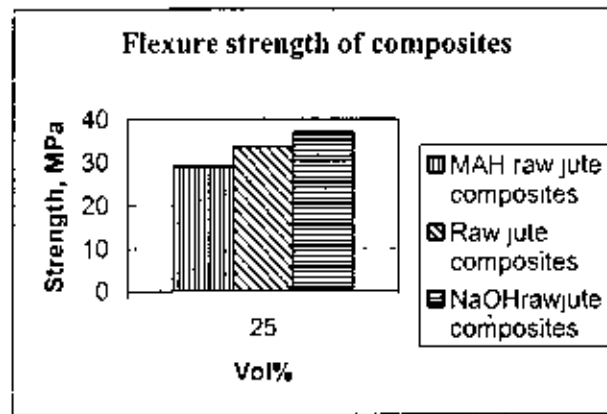
(a)
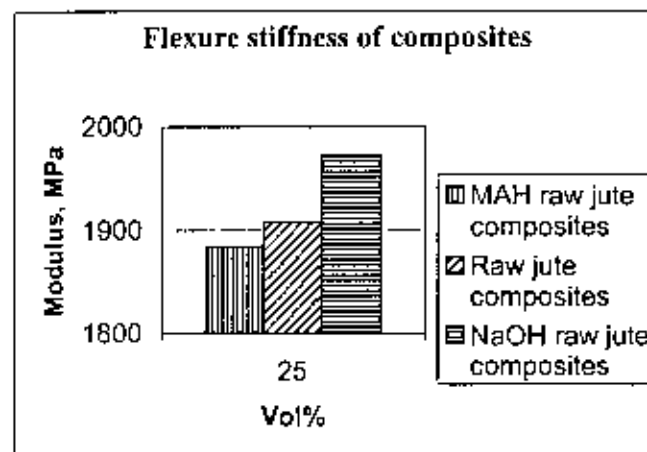
(b)

Figure 4.34. (a) Flexure strength (b) flexure stiffness of raw jute pp composites

CHAPTER – 5
SUMMARY AND CONCLUSIONS

Following conclusion can be made from the present work.

1. Retted jute fiber possesses greater strength as compared to raw jute fiber as the fiber surface of retted jute are smoother than that of raw jute. Strength of the scoured jute fiber is lower than that of retted fiber and greater than that of raw jute fiber. Chemical treatments (MAH, and NaOH) cause weight loss of fiber which lead to decrease in diameter of individual fiber and decrease in linear density of fiber. Both MAH and NaOH treatments increase the strength of all types of jute fiber.

2. Retting causes dissolution of hemicellulose, so in TG curve no peak for hemicellulose was found for retted jute. The increase in fiber fineness (decrease in diameter) facilitates moisture evaporation at a lower temperature in case of retted fibers. In case of raw jute fiber, the presence of rough surface structure facilitates evaporation of moisture at lower temperature. The percentage weight loss due to degredation of α-cellulose of retted fiber under treated and untreated condition was always higher than that of raw jute fiber. In case of NaOH treated retted fibers, the α-cellulose decomposition peak changed from endothermic to exothermic.

3. TG/DTG, DSC curve of composites shows that final degradation temperatures of composites are higher than that of jute fibers. MAH treated jute polypropylene composites showed higher decomposition temperature for 1st peak whereas NaOH treatment lowers this peak temperature for retted fiber. Composites made from treated and untreated jute fibers showed lower values at 2nd peak temperatures as compared to jute fibers of the same

4. Overall composite tensile stresses are lower than that of reference polymer. MAH treated jute fiber composites showed lower stress than that of untreated jute polypropylene composites and NaOH treated jute polypropylene composites. Tensile moduli of all composites are greater than that of reference polymer. In case of MAH

treatment the composites show better tensile modulus Composite with larger fiber size shows greater strength

5. Flexure stresses for three types of composites are lower that that of reference polymer. MAH treated jute fiber composites showed lower stress than that of untreated jute polypropylene composites and NaOH treated jute polypropylene composites. Flexure stiffness increase to a considerable amount than that of reference polymer.

6. Scanning electron micrographs of tensile failure surface show the fiber pull-out and polymeric residue on fiber surface.

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SUGGETION FOR FUTURE WORK

In this study the strength of the fibers increased but the composites do not show any increase in strength value. To improve the strength value different blending technique could be tried to improve uniform fiber distribution in whole composite Different fabrication technique such as extrusion, injection molding could be used to get improve mechanical properties. The interfacial shear strength could be determined by single fiber fragmentation technique so that the actual condition at the interface of composites without and with fiber treament can be charaterized. Acerylation or grafting with various polymering substances (polyvinyl alcohol, poly-methyl-methacrylate, hexa-ethylene-methacrylate, etc). treatment may also adopted to decrease the hydro grospic nature of jute fiber. Different fabricating regime foe incorporating higher volumn fraction of chopped jute could be prebable and woven jute laminated composite could be fabricated.