Development of Natural Fiber Reinforced Polymer Composite

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

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DECLARATION

This is to certify that this research work has been carried out by the author, under the supervision of Dr. Qumrul Ahsan, Associate Professor, Department of Materials and Metallurgical Engineering, BUET, Dhaka, and it has not been submitted elsewhere for the award or degree of any kind.

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To the departed souls of
March 26th
For the independence of Bangladesh
ABSTRACT

Natural fiber reinforced polymer composite has gained paramount importance in the composite research field because of versatility and diversified nature of application field and environmental concern. Retted, woolenized, retted and bleached jute fibers were collected from Bangladesh Jute Research Institute. Retted, retted and bleached variety of woven jute mats were collected from the Bangladesh Jute Mills Corporation (BJMC). Fibers were cut into 5±1 mm length and the woven jute fabrics were cut to 350mmx300mm dimension and dried for 12 hours in an oven at 100°C. Jute fiber surface was analyzed under scanning electron microscope (SEM). Different morphology of the fiber surface was detected. Woolenization treatment severely removed the fiber binding materials e.g. lignin, hemicellulose etc. and bleached out some portion of fibrous body of jute fiber. Bleaching treatment in turn removed the lignin in between the fibrous bundle of jute fiber. Composite specimens were fabricated at room temperature with standard cold press molding and hand lay-up utilizing methyl ethyl ketone peroxide (MEKP) as initiator and ortho phthalic unsaturated polymer resin as matrix. Resin over flow was always allowed for best wetting of the fiber. Maximum of 20–25-volume percent of fiber can be incorporated with this fabricating regime, with current instrumentation facility. The woven jute fabrics showed constant resin impregnation factor during composite fabrication. Water absorption property of chopped jute and woven jute composite was analyzed by ASTM D 570. Water absorption characteristics varied according to the fiber treatment and reinforcement orientation. Density and void content of composites were also carried out. Mechanical characterizations of these fabricated composites were done using Universal Testing Machine. 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. At higher (15 – 20) volume percentage of chopped jute fiber composite showed higher tensile strength than the reference polymer but in case of transverse loading these composite strength just reached the reference value of the polymer. Woven jute fabric reinforced composite showed higher strength behavior in either case of tensile and transverse loading. Failure profile and failure surfaces were observed under a scanning electron
microscope (SEM) to view 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. Effect of volume fraction of these composites on failure mode was also analyzed. Chopped jute composite showed interface and fiber orientation dependence in both case of flexural and tensile loading with large number of fiber pullout. Woven jute laminates showed interface and fabric node ( a 90° cross point of yams within woven fabric) dependence with fiber pullout for tensile loading. When the composites were loaded transversely shearing was a predominant mode of failure along with the interface and fabric node dependence with severe fiber bridging and pullout. These mechanisms helped the laminates to sustain higher load than that of the reference polymer at volume percentage lower than that of the chopped jute composite. Comparison of the SEM micrographs to analyze interface of the fracture surface was carried out. Different types of interface were observed, which are supposed to have direct relationship with the fiber treatment procedure and mechanical properties of the jute fiber reinforced composite. Maximum polymeric residue was observed on the woolenized fiber and minimum was seen on the retted jute, in both case of chopped jute and woven jute reinforced composite. Bleached jute contained moderate amount of polymeric residue in either case of chopped jute and woven jute reinforced composite. Analysis of density, void percentage and strength of composite specimen were also carried out. Higher density and lower void percentage was observed along with higher strength for chopped jute composite. Woven jute fabric laminates showed no density and void content dependence on the strength of composites and its strength is primarily governed with the strength of the yarn. Composite powders were analyzed by Fourier Transformation Infra Red (FTIR) spectrometry to evaluate fiber-matrix interfacial bonding nature or compatibility of the fiber and the matrix. The spectral data of composites were compared with the reference FTIR spectra of jute and cured polymer and it was found that the carbonyl (- CO) environment of jute and the carboxyl (COO-) environment of cured polyester had been changed in the composite. But these tests do not infer about good fiber matrix chemical interaction and mechanical compatibility. Spectral data was compared with tensile and flexural
strength to study the impact of interfacial bonding on the mechanical properties of composites.
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5. SUMMARY AND CONCLUSION

5.1 FUTURE TRENDS
Chapter 1

Introduction
1. INTRODUCTION

Monolithic materials often find limitations on increasing performance requirements in various environments. Hence there has been a trend of finding new materials with tailor made properties for use in various environments or field application. Composites are materials, which meet these challenges. These new materials called composites particularly fiber reinforced plastic, which have been getting increased attention since the World War II. In recent years, interest in natural fiber composites for engineering material applications has increased significantly. This is partly due to the environmental concern about limitations of recyclability of man made composites, but mostly due to their potential in cost reduction. 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. Current understanding regarding the availability of some of the lignocellulosic fibers, their structure and properties, preparation & characteristic of composite incorporated with these natural fibers in polymer, interfacial problems and solution thereon along with potential application made a vast area to explore [Eichhorn, 2001; Rowell, 1998].

Bangladesh has a long history of jute fiber production and the jute fiber is mainly used for producing gunny bags, carpet backing, ropes etc. The vast potential for the use of natural fibers viz. jute in fiber-reinforced composites has lead to an increasing interest for high value added products, such as in household, packaging, structural materials for housing, railways, automobile products etc. The idea of producing long lasting composite materials made of jute fibers has not been considered in the past, because of its rotting, swelling and burning properties. It is now found that application of jute fibers for composites has many advantages. Jute fiber is renewable, versatile, non-abrasive, biodegradable, and compatible [Rowell, 1998].

The main goal of this project is to fabricate natural fiber-reinforced polymer composites and to investigate physical properties (density, void content and water absorption), tensile and flexural properties, and interfacial bond characteristics.
compared to that of the glass fiber reinforced polymer composite. The specific aims related to this orientation are:

- Development of fiber plastic blending techniques for good fiber/matrix compatibility in semi finished, finished products.
- Development of methodology for fabricating polymer composites using glass and jute fibers.
- Evaluation of composite performances by determining its mechanical properties.

In chapter 2 a brief literature review is presented to help the forerunners to get some idea about the ongoing progress of natural fiber reinforced polymer composite and current interest of the project. The third chapter is the experimental with a descriptive indication of the work i.e. SEM characterization of jute fibers, composite fabrication, mechanical characterization (tensile and flexural tests) of fabricated composites and composite bond characterization or fiber matrix compatibility was analyzed by Fourier transformation infrarred (FTIR) spectroscopy; and that is so far completed in the project. The results & discussion of the work starts from the fourth chapter up to end of the document. The fourth chapter comprises of brief description of jute fiber characterization and effects of treatment, which has been explained by scanning electron microscopy (SEM) method. Fabricated composites are shown in section 4.2. Mechanical characterization of chopped discontinuous fiber reinforced composite and woven fabric laminates are explained in detail in section 4.3 and 4.4. Density, void percentage and water absorption properties and relationship with the tensile and flexural properties of composites are discussed in section 4.5. Fourier Transformation Infrar - Red spectroscopic studies and explanation are explained in section 4.6 to analyze the fiber-matrix compatibility and bonding behavior. Summary of the out come of the current project and future research trends are discussed in detail in chapter five.
Chapter 2

Literature Review
2. THEORETICAL BACKGROUND

2.1 INTRODUCTION

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. For example, glass fiber is used to improve the stiffness and strength of plastics, although there are several disadvantages associated with its use. Glass-fiber production requires a great deal of energy-processing temperature and these temperatures can exceed 1200°C. During processing the glass fiber to fabricate composite, polymer composite tends to abrade processing equipment and increase the density of the plastic system.

The use of lignocellulosic fibers in thermoplastics and thermoset has received a lot of interest due to their low densities, low cost, and non-abrasive nature. The natural fiber composites can be very cost effective material especially for building and construction industry (panels, false ceilings, partition boards etc.), packaging, automobile and railway coach interiors and storage devices. There are many examples of the use of cellulosic fibers in their native condition like sisal, coir, jute, banana, palm, flax, cotton, and paper for reinforcement of different thermoplastic and thermosetting materials like phenol formaldehyde, unsaturated polyester, epoxy, polyethylene, cement, natural rubber etc. Different geometries of these fibers, both singly and in combination with glass, have been employed by many researchers for fabrication of uni-axial, bi-axial and randomly oriented composites. Amongst this jute lignocellulosic fibers contain a high proportion of stiff natural cellulose [Eichhorn, 2001].

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. The use of these materials in composites has increased over the last few years due to their relative cheapness compared to conventional materials, their ability to recycle and for the fact that they compete well with in terms of strength per unit of weight of material. This is partly due to the environmental concern about
limitations of recyclability of man made composites, but mostly due to their potential in cost reduction. 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, geotextiles, and building composites [Eichhorn, 2001].

Glass, carbon, boron & 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 [Eichhorn, 2001].

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 [Eichhorn, 2001]. 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 [Eichhorn, 2001].

The physical and mechanical properties of these natural fibers depend on geographic origin, climatic growth condition, and processing technique. Natural fibers are classed according to their source, plants, animals or minerals. Many varieties of plant fiber exist such as hairs (Cotton, Kapok), fiber sheaf of dicotylic plants or vessel-sheaf, of monococylc plant (Flax, Hemp, Jute), and beard fibers (Sisal, Henequen, Coir) and large amount of fiber obtained from trees. The abundance of raw material is also a concern, and the pressure on it to produce the greener technologies has made the area of research of worldwide interest.

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 [Eichhorn, 2001]. 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 [Eichhorn, 2001].

Several studies of fiber composition and morphology have found that cellulose content and microfibril angle tend to control the mechanical properties of cellulosic fibers [http://www.tifae.org.in/index1.htm]. Higher cellulose content and lower microfibril 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 [Eichhorn, 2001].

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 [Rowell, 1998].
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 [Rowell, 1998].

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 [Rowell, 1998].

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, advanced technology, the jute industry looks to broader utilization of its products [Rowell, 1998].

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 10 to 12 feet (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 3 to 4 inches (75 to 100 mm) during the growing season. The plant's light green leaves are
4 to 6 inches (10 to 15 cm) long, about 2 inches (5 cm) wide, have serrated edges, and taper to a point [Rowell, 1998].

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 [Rowell, 1998].

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 [http://www.jute.org/retting.htm]

There are several different retting process; Water Retting, Dow 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 [Rowell, 1998].

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 [Rowell, 1998].

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 [Roger M. Rowell, 1998].
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 [http://www.eap.mcgill.ca/CPH_6.htm].

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 [http://www.eap.mcgill.ca/CPH_6.htm].

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 [http://www.eap.mcgill.ca/CPH_6.htm].

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, scutchted, hacked, 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 [http://www.mafes.msstate.edu/Variety/Kenaf/kenaf18.htm].

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 [Austen, 1984].

2.3.3 Bleaching of Jute Fiber

The bleaching process of jute and allied fiber in modern composite preparation technology is not only concerned with brightening fiber yarn 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 [Rowell, 1998].

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 and 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.
Jute bleaching is carried out at Bangladesh Jute Research Institute following the above procedure. Required amount of jute fiber is treated with a solution containing 10g/l hydrogen peroxide, 6g/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 1 hr. It was then washed several times with water and dried. Sodium silicate and metasilicate are stabilizers, which are added to control the decomposition of hydrogen peroxide (H₂O₂) to water (H₂O). 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.

Hydrogen peroxide can be used on all vegetable fibers and its bleaching effect is permanent [Pauck, 2002]. 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 [Rowell, 1998; Pauck, 2002].

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.

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. They mix with grease and water to reduce the adhesion of soils. Surface-active substances are frequently used as auxiliaries in bleaching. If they entail the enhancement of fiber or fabric wet ability, they 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.

2.3.4 Wuplenization of Jute Fiber

Jute fibers are treated with 20% caustic soda solution for 10 minutes at room temperature. The fibers are 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 [Roger M. Rowell, 1998].
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% [Eichhorn, 2001; Rowell, 1998].

Water wash is done several times to remove and collect the excess NaOH solution followed by 2g/l sulfuric acid solution treatment for the neutralization of excess NaOH. This treatment provides neutralized fiber, which is again followed by water extraction to remove sulfuric acid from 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 [Eichhorn, 2001; Rowell, 1998].

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 [http://www.nyp-corp.com/Jute_to_Burlap.htm].

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 borky portions adhering to the fiber [Rowell, 1998; http://www.nyp-corp.com/Jute_to_Burlap.htm].

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 [http://www.nyp-corp.com/Jute_to_Burlap.htm].

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 [http://www.nyp-corp.com/Jute_to_Burlap.htm].

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 [http://www.nyp-corp.com/Jute_to_Burlap.htm].

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 itself 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 [http://www.nyp-corp.com/Jute_to_Burlap.htm].

The chemical treatment procedures of jute fiber and to that of woven fabric are the same. Although access about the treatment procedure of jute fiber failed from BJRI, BJMC also follow the same chemical treatment procedure as the BJRI.

2.5 EXTRACTION, CHEMICAL STRUCTURE AND 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. 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 [Rowell, 1998].

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 [Eichhorn, 2001; Rowell, 1998].
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 [Rowell, 1998].

The changes in chemical composition at different stages shows that there is little difference in cellulose, holocellulose, 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 [Rowell, 1998].

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 [Rowell, 1998].

The surface characteristics of jute fiber at different stages of growth shows that at the early stages of growth, there was an incomplete formation 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 [Rowell, 1998].
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 [Rowell, 1998].

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" [Eichhorn, 2001; Rowell, 1998].

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 crystalline 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 [Eichhorn, 2001].
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 [Eichhorn, 2001].

There is a particular relation ship between the fiber orientation and extension to that of the spiral angle. X-ray measurements showed 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 [Eichhorn, 2001].

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% [Eichhorn, 2001; Rowell, 1995].

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 [Rowell, 1998].

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 [Roger M. Rowell, 1998]. The mechanical 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 [Eichhorn, 2001].

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Density (g cm⁻³)</th>
<th>Elongation at break (%)</th>
<th>Tensile strength (MPa)</th>
<th>Young's Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cotton</td>
<td>1.5-1.6</td>
<td>7.0-8.0</td>
<td>287.597</td>
<td>5.1-12.6</td>
</tr>
<tr>
<td>Jute</td>
<td>1.3</td>
<td>8.1-18</td>
<td>383.773</td>
<td>26.5</td>
</tr>
<tr>
<td>Flax</td>
<td>1.5</td>
<td>2.7-3.2</td>
<td>345.103</td>
<td>27.6</td>
</tr>
<tr>
<td>Hemp</td>
<td>-</td>
<td>-</td>
<td>690</td>
<td>-</td>
</tr>
<tr>
<td>Ramie</td>
<td>-</td>
<td>3.5-3.8</td>
<td>400.938</td>
<td>61.4-128</td>
</tr>
<tr>
<td>Silk</td>
<td>1.5</td>
<td>2.0-2.5</td>
<td>511.635</td>
<td>9.4-22.0</td>
</tr>
<tr>
<td>Cor</td>
<td>1.2</td>
<td>30.0</td>
<td>175</td>
<td>4.0-6.0</td>
</tr>
<tr>
<td>Viscose (cord)</td>
<td>-</td>
<td>11.4</td>
<td>593</td>
<td>11.0</td>
</tr>
<tr>
<td>Soft wood</td>
<td>1.5</td>
<td>-</td>
<td>1000</td>
<td>40.0</td>
</tr>
<tr>
<td>Kraft</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E-glass</td>
<td>2.5</td>
<td>2.5</td>
<td>2000-3500</td>
<td>70.0</td>
</tr>
<tr>
<td>S-glass</td>
<td>2.5</td>
<td>2.8</td>
<td>4770</td>
<td>86.0</td>
</tr>
<tr>
<td>Aramid</td>
<td>1.4</td>
<td>3.3-3.7</td>
<td>3000-3150</td>
<td>63.0-67.0</td>
</tr>
<tr>
<td>(normal)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbon</td>
<td>1.4</td>
<td>1.4-1.8</td>
<td>4000</td>
<td>230.0-240.0</td>
</tr>
<tr>
<td>(standard)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The two main disadvantages of using jute and kenaf fibers in thermoplastics are the high moisture absorption of the fibers and composites and the low processing temperatures permissible. Natural fibers are strongly hydrophilic materials. Moisture absorption by fibers leads to significant deterioration of the mechanical properties of polymer composites. Furthermore, many polymers are hydrophobic and due to this divergent behavior of the fiber and polymer matrix, the interface in natural fiber composite is rather poor. Jute as well as other natural fibers used as reinforcement in polymer composite causes imperfect bonding at fiber-matrix interface. The moisture absorbed by the composite and the corresponding dimensional changes can be reduced dramatically if the fibers are thoroughly encapsulated in the plastic and there is good adhesion between the fiber and the matrix. If necessary, moisture absorption of the fibers can be significantly reduced by acetylation of the hydroxyl groups present in the fiber, although at some increase in cost. Selecting applications where high moisture absorption is not a major drawback can minimize the disadvantage of the high moisture absorption of the composite [Rowell, 1998; Beckermann, 2004].

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 [Rowell, 1998].

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, 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 [Rowell, 1998].

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 [Rowell, 1998].

The inherent polar and hydrophilic nature of the jute and kenaf fibers and the non-polar 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 [Rowell, 1998].

All lignocellulosic fiber in thermoplastics is limited due to potential fiber degradation at higher temperatures. Thus the plastics that can be used with it are limited to those with low melting temperatures. In general, no deterioration of properties due to fiber degradation occurs when processing temperatures are maintained below about 200°C for short periods [Rowell, 1998].

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 [Rowell, 1998].

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, hemicellulose and lignin, is known as lignocellulosic fibers [Rowell, 1998].

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 (S₁, S₂ and S₃). In all lignocellulosic fibers these layers contain cellulose, hemicellulose 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 S₂ layer (about 50%) and lignin is most concentrated in the middle lamella (about 90%), which, in principle, is free of cellulose. The S₂ layer is usually by far the thickest layer and dominates the properties of the fibers [Rowell, 1998]. In the S₂ layer the microfibrils 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 [Rowell, 1998].

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) [Rowell, 1998].

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) [Rowell, 1998].

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 will biodegrade 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 also 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 [Rowell, 1998].

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 [Rowell, 1998].

Figure 2.1: Jute stem (combined transverse section and longitudinal section). Magnification x 70. (Courtesy of Dr. C. G. Jarman, Tropical Development and Research Institute, London, U. K.) [Hand Book of Fiber Chemistry] [Rowell, 1998].

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 is evidently necessary to provide a coherent and continuous overlapping structure. The cells are some 200 times longer than their width [Rowell, 1998].

The ultimate cell dimensions of kenaf and many other fiber-bearing dicotyledons are similar to those of jute. A distinction must be made between jute-like fibers and flax [Rowell, 1998].

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, woollenization, bleaching and plasma activation graft polymerization with vinyl monomers, which are very well described in the literature. These, however, will increase the fiber cost [Eichhorn, 2001; Rowell, 1995].

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 [Eichhorn, 2001; Rowell, 1998]. 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 [Eichhorn, 2001].

Figure 2.2 shows the hydrogen bonding system in cellulose i.e. the main constituent of the natural fiber. The thinner lines mean 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. These differences in hydrogen bonding mean that the 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 [Rowell, 1998; Beckermann, 2004].

Cellulose micro fibrils contain two crystalline forms, cellulose α and β, in which the chains are packed slightly differently. 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. Cellulose α or 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 allomorphs* [Rowell, 1998; Beckermann, 2004].

* A change in crystalline form that occurs without a change in chemical composition.
2.8.1 Acetylation

One simple method that can be used to overcome this problem 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 [Rowell, 1998].

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 [Rowell, 1998]. 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 [Rowell, 1998].

Chemically modified jute and kenaf cloth have been tested against brown-, white, and soft-rot fungi and tunneling bacteria in a fungal cell. It was observed that the cloth was destroyed in less than 6 months, while cloth made from acetylated fiber above 16-weight percent gain (WPG) showed no attack after 1 year [Rowell, 1998]. 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 [Rowell, 1998].

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 [Rowell, 1995].

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 [Rowell, 1995].

Herrera and Escamilla [2002] has found that the compatibility can be improved by grafting a matrix-compatible polymer onto the fiber surface. 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 cellulose chain in a redox system. In this reaction, oxidation of the anhydroglucose units occurs along the cellulose chains and macrocellulosic 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 fourcroydes) cellulose fibers grafted with methyl methacrylate (MMA) has been examined by this group. They also tried 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 [Eichhorn, 2001; Zafeiriopoulos, 2002].

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 [Eichhorn, 2001; Rowell, 1998; Ray, 2001].

Note that mercerization or alkalization is a process familiar to the textile scientist where the fiber is chemically treated to increase its luster and to increase the affinity towards dyeing [Berger 1999; Escamilla, 2002] has discussed the nature of the chemical changes occurring in jute on mercerization.

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 & temperature effects are reported to influence the values of (D) and (n) [Rowell, 1998].

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 [Rowell, 1998].

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 [Pauck, 2002]. 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 (WAXS) 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 of 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 [Eichhorn, 2001; Rowell, 1998].

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 [Eichhorn, 2001; Rowell, 1998].

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 [Rowell, 1998; Rowell, 1995].

Experiments with jute yarn at the British Jute Trade Research Association, have shown that, untreated yarn subjected to hydrolysis with 0.2 N H₂SO₄ 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 [Rowell, 1998; Rowell, 1995].

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 [Rowell, 1998; Rowell, 1995].

Jute is chemically treated with isopropyl tristearyloxy titanate (abbreviated as titanate), γ-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 [Rowell, 1998; Rowell, 1995]. Zafeiropoulos [2002] explained the effect of acetylation and stearic acid treatments upon the fiber strength. 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 [Rowell, 1998].

### 2.9 RESIN MATERIAL

There are two types of polymers; they are thermoplastic and thermoset. Thermoset polymers are groups of polymeric material that softens when heated and harden again when cooled. These are mainly of olefin or hydrocarbon based high molecular weight substances. Thermosetting resins are usually low molecular weight monomers or oligomers having functional groups for cross-linking reaction. The
polymerization of these resins (curing reaction) can be carried out either by addition reaction, without evolution of volatiles (e.g., epoxy resin, unsaturated polyester or addition polyamides) or by condensation reaction (e.g., phenolic resin) to yield a highly cross-linked three-dimensional network structure, which is infusible and insoluble in most of the organic solvents. In order to prepare void-free molded parts, it is desirable to use resins that do not produce volatile products during curing. Among the widely used engineering plastics, unsaturated polyester has wide acceptance to the composite fabricators and the engineers for its mechanical characteristics, ease of fabrication of composites and cost. Nowadays it is used as lightweight civil construction materials, automotive body parts combining with glass fiber as reinforcement.

Most polyester resins are viscous, pale colored liquids consisting of a solution of polyester in a monomer, which is usually styrene. The addition of styrene in amounts of up to 50% helps to make the resin easier to handle by reducing its viscosity. The styrene also performs the vital function of enabling the resin to cure from a liquid to a solid by 'cross-linking' the molecular chains of the polyester, without the evolution of any by-products. These resins can therefore be molded without the use of pressure and are called 'contact' or 'low pressure' resins. Polyester resins have a limited storage life as they will set or 'gel' on their own over a long period. Often small quantities of inhibitor are added during the resin manufacture to slow this gelling action.

For use in molding, a polyester resin requires the addition of several ancillary products. These products are generally:

- Catalyst; methyl ethyl ketone peroxide or benzyl peroxide
- Accelerator; cobalt naphthenate or cobalt octoate
- Additives: Thixotropic; Pigment; Filler; Chemical/fire resistance

Catalysts are added to the resin system shortly before use to initiate the polymerization reaction. The catalyst does not take part in the chemical reaction but simply activates the process. An accelerator is added to the catalyzed resin to enable
the reaction to proceed at workshop temperature and/or at a greater rate.

Great care is needed in the preparation of the resin mix prior to molding. The resin and any additives must be carefully stirred to disperse all the components evenly before the catalyst is added. This stirring must be thorough and careful as any air introduced into the resin mix affects the quality of the final molding. This is especially so when laminating with layers of reinforcing materials as air bubbles can be formed within the resultant laminate, which can weaken the structure. It is also important to add the accelerator and catalyst in carefully measured amounts to control the polymerization reaction to give the best material properties. Too much catalyst will cause too rapid a gelation time, whereas too little catalyst will result in under-cure.

The starting materials for a thermoset polyester matrix is an unsaturated polyester resin that contains a number of C=C bonds. It is prepared by reaction of maleic anhydride and ethylene or propylene glycol. Saturated acids, such as isophthalic or ortho phthalic acids are also added to modify the chemical structure between the cross-linking sites; however they do not contain any C=C double bond. The resulting polymeric liquid is dissolved in a reactive diluent such as styrene, which reduces the viscosity and makes it easier to handle. The diluent also contains C=C double bonds and acts as cross-linking agent by bridging adjacent polyester molecules at their unsaturation points. Trace amount of inhibitor, such as hydroquinone or benzoquinone, are added to the liquid to prevent premature polymerization.

For curing of such unsaturated polyester resin with fiber, azo type initiators (R-N=N-R) and organic peroxides (R-O-O-R) are generally used.

Unsaturated polyesters are linear polycondensation products based on unsaturated and saturated acids/anhydrides and diols or oxides. These resins are generally pale yellow colored oligomers with low degree of polymerization. Depending on the
chemical composition and molecular weight (1200-3000 g mol\(^{-1}\)) these oligomers may be viscous liquids or brittle solids. The unsaturation in the backbone provides sites for reaction with vinyl monomers using free radical initiators, thereby leading to the formation of a three dimensional network. The solutions of unsaturated polyesters and vinyl monomers (reactive diluents) are known as unsaturated polyester resins (UPE resins). The dilute UPE resin solution thus has a viscosity in the range of 200 – 2000 cP at room temperature. One of the major problems with these resins is their high shrinkage during curing reaction (>7%). This shrinkage results in the fiber reinforcement to become raised from the surface of the molding leading to poor surface finish. By selection of different acids/anhydrides and glycols and changes in the ratio of saturated/unsaturated component and the reactive diluent, the nature of the final network can be varied significantly to meet different performance requirements. Addition of low profile additives (thermoplastics) in the resin formulation compensates for the thermal and polymerization shrinkage of UPE resin, thereby leading to low shrink or zero-shrink resin systems. The advent of low profile, low shrink technology has allowed molding compounds to compete successfully with steel in exterior automotive applications.

There are several types of unsaturated polyester resin - general purpose, flexible, resilient, low-shrinkage (low profile), weather resistant, chemical resistant and fire resistant varieties. There is a whole range of polyesters made from different acids, glycols and monomers, all having varying properties. There are two principle types of polyester resin used as standard laminating systems in the composites industry. Orthophthalic polyester resin is the standard economic resin used by many people. Isophthalic polyester resin is now becoming the preferred material in industries such as marine where its superior water resistance is desirable.

Polyester resins may be classified on the basis of their structure into the following groups:

- Ortho-resins
- Iso-resins
- Bisphenol-A fumarates
- Chlorendics, and
- Vinyl ester resins

Figure 2.3 shows the typical unsaturated polyester resin formed from the reaction of propylene glycol, maleic anhydride and phthalic anhydride. It is observed that the reaction of polyester is solely condensation type polymerization reaction. The figure also shows the idealized chemical structure of typical polyester. The arrow indicates that the (CO - O - C), and -OH groups react to form long chain to facilitate chain growth and the (C= C*) within the molecular chain is the position of unsaturation that facilitates cross-linking.

Ortho-Resins: Ortho-resins, also known as general-purpose resins, are based on phthalic anhydride (PA), maleic anhydride (MA), or fumaric acid (FA) and glycols. Phthalic anhydride is relatively low in price and provides inflexible link in the backbone. However, it reduces thermal resistance of laminates. Figure 2.4 shows the ortho type resins that are the same as that of the figure 2.3, which is also known as general-purpose resin and has got wide range of application. Limited chemical resistance and processibility are other problems associated with these resins. Among the glycols, 1,2-propylene glycol is the most important. Due to the presence of pendant methyl group, the resulting resins are less crystalline and more compatible with commonly used reactive diluent (styrene) than those obtained using ethylene glycol, diethylene glycol and triethylene glycol, and gives products with inferior electrical properties. Using neophenyl glycol or hydrogenated bisphenol-A produces resins with high heat and chemical resistance.
Bisphenol - A fumarate resin: Figure 2.5 shows a typical Bisphenol-A fumarate resin. They are synthesized by reacting ethoxy-based bisphenol-A with fumaric acid.
**Chlorodene resins:** In order to enhance flame retardancy, chlorine/bromine containing anhydrides or phenols are used in the preparation of UP resins.

![Figure 2.6: Chlorodene resin](image)

For example, as presented in figure 2.6 the reaction of chlorodene anhydride/chlorodene acid with maleic anhydride/fumaric acid and glycol yields the following resin with better flame retardancy than general purpose UP resin. Other monomers used include tetrachloro or tetrabromophthalic anhydride. The bromine content must be at least 12% to make self-extinguishing polyester.

**Vinylester resin:** Vinylester resins are similar in their molecular structure to polyesters, but differ primarily in the location of their reactive sites, these being positioned only at the ends of the molecular chains. As the whole length of the molecular chain is available to absorb shock loadings this makes vinylester resins tougher and more resilient than polyesters. The vinylester molecule also features fewer ester groups. These ester groups are susceptible to water degradation by hydrolysis which means that vinylesters exhibit better resistance to water and many other chemicals than their polyester counterparts, and are frequently found in applications such as pipelines and chemical storage tanks.

The figure 2.7 shows the idealized chemical structure of a typical vinylester. Note the positions of the ester groups and the reactive sites (C$^\equiv C^+$) within the molecular chain. The dot represents the site that is attacked by the polymerization initiating system and is a type of electrophile, implies the carbon-carbon double bond is electron hungry site. As the peroxide initiator breaks it supplies a free electron that initiates the cross linking reaction.
Figure 2.7: Typical vinyl ester resin

With the reduced number of ester groups in a vinyl ester when compared to polyester, the resin is less prone to damage by hydrolysis. The material is therefore sometimes used as a barrier or 'skin' coat for a polyester laminate that is to be immersed in water, such as in a boat hull. The cured molecular structure of the vinyl ester also means that it tends to be tougher than polyester, although to really achieve these properties the resin usually needs to have an elevated temperature post cure.

Table 2.2: Typical Composition of unsaturated polyester resin

<table>
<thead>
<tr>
<th>No</th>
<th>Name</th>
<th>Weight %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Propylene Glycol</td>
<td>26</td>
</tr>
<tr>
<td>2</td>
<td>Methyl Ethyl Glycol</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>Phthalic Anhydride</td>
<td>30</td>
</tr>
<tr>
<td>4</td>
<td>Maleic Anhydride</td>
<td>16.5</td>
</tr>
<tr>
<td>5</td>
<td>Styrene Monomer</td>
<td>30 - 32</td>
</tr>
<tr>
<td>6</td>
<td>Methyl Methacrylate</td>
<td>7</td>
</tr>
<tr>
<td>7</td>
<td>Aerosol (Trade Name)</td>
<td>Not specified</td>
</tr>
</tbody>
</table>

Table 2.3: Typical composition of MEKP initiator

<table>
<thead>
<tr>
<th>Components</th>
<th>Volume %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methyl Ethyl Ketone Peroxide</td>
<td>32 - 34</td>
</tr>
<tr>
<td>Dimethyl Phthalate</td>
<td>43</td>
</tr>
<tr>
<td>2,2,4-Trimethyl-1,3-pentanediol dilaurate</td>
<td>26 - 30</td>
</tr>
<tr>
<td>Hydrogen Peroxide</td>
<td>01</td>
</tr>
<tr>
<td>Methyl Ethyl Ketone</td>
<td>02</td>
</tr>
<tr>
<td>Hexylene Glycol</td>
<td>06</td>
</tr>
<tr>
<td>Water</td>
<td>01</td>
</tr>
</tbody>
</table>
**Initiator:** Methyl Ethyl Ketone Peroxide (MEKP) is the most widely used catalyst system. MEKP is used with promoters, usually 6% Cobalt Naphthenate or 6% Cobalt Octoate and dimethylamine (DMA) or diethylamine (DEA). The MEKP used most often is supplied at 9% active oxygen.

CoNap/Oct - Cobalt Naphthenate/Octoate promoter is a dark, purplish liquid sold in solutions of various concentrations in active cobalt. The properties of cobalt octoate and cobalt naphthenate are similar, although a 20 to 30% higher amount of cobalt octoate may be needed for the same gel time as with cobalt naphthenate.

Diethylamine, (DEA), dimethylamine (DMA), dimethylacetamide, (DMAA) and diethyl acetamide (DEAA), are used as accelerator in curing unsaturated polyester resin.

2,4-pentanedione (2,4-P) can be incorporated into any cobalt promoted cure system at a level of 0.05 to 0.50% and above, to retard the gelling. The gel time can be extended as much as tenfold without adverse effects on the final cure or the corrosion resistance of the composite provided there is sufficient cobalt present. This material can be formulated to give an extended pot life or can be used for fabrication when very long gel times are required. Promoters, accelerator and in some special cases inhibitors should be mixed thoroughly with the resin, prior to initiator addition to avoid explosion hazard. A thixotrope, UV - stabilizer and air release agents are occasionally used for special purpose.

2.10 COMPOSITE COMPOUNDING FUNDAMENTALS

Transformation of uncured or partially cured fiber reinforced thermosetting polymers into composite parts involves curing the material at ambient or elevated temperature. These materials stem from early materials including glass-reinforced polyesters or more generally fiber reinforced plastics. The reinforcement phase may consist of fibers of glass, carbon or aramid fibers or cheaper and biodegradable natural fibers of cellulosic base in a matrix of resins such as polyesters, vinyl esters, epoxies or phenolics. The fiber phase may be used in forms such as woven, chopped, strand and continuous filament [http://www.baaqmd.gov/pmt/handbook/s1lc12pd.htm].
Unlike thermoplastic composites, once set, they cannot be heated and reshaped and the possibility for recycling this material is very limited. High cure temperature is required to initiate the polymerization reaction that is induced either by mixing a peroxide initiator that dissociates to initiate polymerization, and generate heat to assist reaction or by applying heat from external source. High cure pressure is applied to ensure uniform flow of high viscous resin through the fibers or mats in the mold, as well as consolidation of individual unbonded plies into a bonded laminate. The magnitude of this two-process parameter with the duration affects the performance of the product [http://www.baaqmd.gov/pml/handbook/s11c12pd.htm].

The length of time required for proper cure is known as cure cycle. It is desirable to achieve proper cure at shortest interval of time. It should be noted that the cure cycle depends on a number of factors, including resin chemistry, catalyst or initiator reactivity, curing temperature, and the presence of inhibitor or accelerator [http://www.baaqmd.gov/pml/handbook/s11c12pd.htm].

Reinforced plastics products are fabricated using any of several processes, depending on their size, shape and other desired physical characteristics. The principle molding processes are open molding and closed molding. The primary types of open molding fabrication processes include spray lay-up (resin spray-up), hand lay-up, continuous lamination and pultrusion. Bag molding primarily represents closed molding processes. Process descriptions for these molding processes are given below.

Molds for cold press molding are generally simpler than their injection mold counterparts. There is no spur and runner system in a cold press mold, and the process itself is generally limited to simpler part geometries due to the lower flow capabilities of the starting thermosetting materials. However, no provision was there to heat the mold, but if needed then it could be usually accomplished by electric resistance heating, steam, or hot oil circulation. Compression molds can be classified as hand molds, used for trial runs; semiautomatic, in which the press follows a programmed cycle but the operator manually loads and unloads the press; and automatic, which operate under a fully automatic press cycle (including automatic loading and unloading).

Materials for cold press molding include polyesters, unsaturated polyester, phenolic, melamine, urea-formaldehyde, epoxies, urethanes, and elastomers. Advantages noted
for cold press molding in these applications include: molds that are simpler, less expensive, and require low maintenance; less scrap; and low residual stresses in the molded parts (thus favoring this process for flat thin parts. A typical disadvantage is longer cycle times and therefore lower production rates are common.

Closed, such as compression or injection molding operations involve the use of two matched dies to define the entire outer surface of the part. When closed and filled with a resin mix, the matched die mold is subjected to heat and pressure to cure the plastic. For the most durable production configuration, hardened metal dies were used. A process similar to compression molding of thermosetting resins, except that no heat is applied during the molding cycle.

Hand lay-up is a method of producing composite structures by hand applying composite materials in successive layers on a tool or mold that defines the part geometry. It is a cost-effective manufacturing method for simple structures. It is usually employed in connection with the processes of spray-up and hand lay-up molding, when such processes do not include the application of pressure during curing, also known as Open Mold Processes. Open contact molding in one-sided molds is a low-cost, common process for making fiberglass composite products. During the project, the mold is first treated with mold releasing agent. The following considerations are necessary for fabrication of composite:

- Proper amount of resin incorporation is a factor because if not enough resin is placed in the mold cavity, the part will not be formed completely. If too much material is applied into the mold, it may not melt completely or excess flash may be produced.
- The fluid plastic is held under pressure, often ranging upwards from 140 atm, for a sufficient length of time for the material to undergo polymerization or cross-linking, which renders it hard and rigid. Different materials require different forces to mold them. High-viscosity materials require more pressure than materials with low viscosities.
- A down stroke machine consists of a compression press in which the platen moves and upstroke machine consists of a compression press where the platen moves upward to close the mold.
• The mold is subjected to high levels of heat, approximately around 300°F to 375°F. Appropriate heating is crucial to allow the resin to soften for it to flow into the mold cavity. If the temperature is too low, the plastic material may not melt completely and produce an unfinished part.

2.10.1 Curing of Unsaturated Polyester

The molecular chains of the polyester can be represented as follows, where 'B' indicates the reactive sites in the molecule as shown in the figure 2.8 [http://www.netcomposites.com/education.asp?sequence=10].

The figure 2.8 shows the schematic view of a polyester resin, which is premixed with the styrene, and at the beginning of polymerization initiator is used. With the addition of styrene 'S', and in the presence of a catalyst, the styrene cross-links the polymer chains at each of the reactive sites to form a highly complex three-dimensional network as follows:

```
 A   B   A   B   A   B   A
```

Schematic Representation of Polyester Resin (Uncured)

Figure 2.8: Schematic polyester resin

Figure 2.9 explains how cross-linking occurs when the styrene (S) is reacted to form a network structure, and at this stage the polyester resin is then said to be 'cured'. It is now a chemically resistant (and usually) hard solid. The cross-linking or curing process is called 'polymerization'. It is a non-reversible chemical reaction. The 'side-by-side' nature of this cross-linking means that polyester laminates suffer from brittleness [http://www.netcomposites.com/education.asp?sequence=10].

```
  A   B   A   B   A   B   A
  S   S   S   S
A   B   A   B   A   B   A
```

Schematic Representation of Polyester Resin (Cured)

Figure 2.9: Schematic cured state of polyester.
The curing reaction of unsaturated polyester resin (UPR) is a free radical chain growth cross-linking polymerization between the reactive diluents (e.g., styrene monomer) and UP resin. Polyester molecules are the cross-linkers while styrene serves as an agent to link the adjacent polyester molecules. Depending on the temperature and other processing conditions some of the species remain unreacted after curing in the form of residual monomer and soluble polymer that do not contribute to the network structure. A wide range of peroxides, azo, and azine compounds can be used as initiators depending on the curing temperature. For room temperature curing, as in the case of large hand lay-up structures, methyl ethyl ketone peroxide (MEKP) and for moderate temperature (60-90°C) curing benzoyl peroxide is used. For hot press or oven curing (130-150°C) di-t-butyl peroxide or t-butyl perbenzoate is used. A mixture of initiators is used when a large temperature increase is expected. To accelerate the decomposition of peroxides, some metal compounds, tertiary amines, and mercaptans can be used. Cobalt naphthenate (CoNap) and cobalt octoate (CoOc) are the most widely used accelerators [http://www.netcomposites.com/education.asp?sequence=10].

CoNap is used at about 0.01%. The excess of it causes too much acceleration, resulting in darkening in color and bubble formation in the cured products. A 3-D network is formed as a result of copolymerization between the unsaturated monomer present in the resin (e.g., styrene) and the maleate and fumarate double bonds of the polyester [http://www.netcomposites.com/education.asp?sequence=10].

Great care is needed in the preparation of the resin mix prior to molding. The resin and any additives must be carefully stirred to disperse all the components evenly before the catalyst is added. This stirring must be thorough and careful as any air introduced into the resin mix affects the quality of the final molding. This is especially so when laminating with layers of reinforcing materials as air bubbles can be formed within the resultant laminate, which can weaken the structure. It is also important to add the accelerator and catalyst in carefully measured amounts to control the polymerization reaction to give the best material properties. Too much catalyst will cause too rapid a gelation time, whereas too little catalyst will result in under-cure [http://www.netcomposites.com/education.asp?sequence=10].
2.10.2 Degree of Cure

The curing reaction for polyester resins is initiated by adding small quantities of a catalyst, such as organic peroxide or an aliphatic azo compound, to the liquid mix. With the application of heat (in the temperature range of 107-163°C), the catalyst decomposes rapidly into free radicals, which react (mostly) with the styrene molecules and break their C = C bonds. Styrene radicals, in turn, join with the polyester molecules at their unsaturation points and eventually form cross-links between them. The resulting material is a solid polyester resin [http://www.netcomposites.com/education.asp?sequence=10].

The curing time for polyester resins depends on the decomposition rate of the catalyst, which can be increased by increasing the curing temperature. However, for a given resin-catalyst system, there is an optimum temperature at which all of the free radicals generated from the catalyst are utilized in curing the resin. Above this optimum temperature, free radicals are formed so rapidly that wasteful side reactions occur and deterioration of the curing reaction is observed. At temperatures below the optimum, the curing reaction is very slow. Small quantities of accelerator can increase the decomposition rate of a catalyst so the cross-linking reaction becomes faster [http://www.netcomposites.com/education.asp?sequence=10].

The type of ingredient also influences the properties and/or processing characteristics of polyester resins. For example, terephthalic acid generally provides a higher heat deflection temperature than either isophthalic or orthophthalic acids, but it has the slowest reactivity of the three phthalic acids; adipic acid, if used instead of any of the phthalic acids, lowers the stiffness of polyester molecules, since it does not contain an aromatic ring in the backbone. Thus, it can be used as a flexibilizer for polyester resins. Another ingredient that can also lower the stiffness is diethylene glycol. Propylene glycol, on the other hand, makes the polyester resin more rigid, since the pendant methyl groups in its structure restrict the rotation of polyester molecules [http://www.netcomposites.com/education.asp?sequence=10].

The amount and type of diluent are also important factors in controlling the properties and processing characteristics of polyester resins. Styrene is the most widely used diluent because it has low viscosity, high solvency, and low cost. Its
drawbacks are flammability and potential (carcinogenic) health hazard due to excessive emissions. Increasing the amount of styrene reduces the modulus of the cured polyester resin since it increases the space between polyester molecules. Because styrene also contributes unsaturation points, higher styrene content in the resin solution increases the total amount of unsaturation and, consequently, the curing time is increased. An excessive amount of styrene tends to promote self polymerization and causes polystyrene-like properties to dominate the cured polyester resin [http://www.netcomposites.com/education.asp?sequence=10].

Unsaturated polyester is thermosetting resin made by the condensation reaction between difunctional acids and glycols. The resulting polymer is then dissolved in styrene [C₆H₅CHCH₂] or other vinyl unsaturated monomer. The structures of the acids and glycols used and their proportions, especially the ratio of the unsaturated versus the saturated acid, and the type and amount of monomer used, are all tailored for each resin to balance economy, processing characteristics, and performance properties [http://www.netcomposites.com/education.asp?sequence=10].

High molecular weight fluids, such as polymer melts, have high viscosities and flow only under stress and is dependent on the temperature and shear rate. For all fluids, the viscosity increases with the increase in temperature. Shear rate has influential effect on the high molecular weight compound rather than the low molecular weight compound. The viscosity of liquid thermoset polymer tend to increase (shear thickening) or decrease (shear thinning). Shortly after the curing reaction begins, the resin viscosity increases very rapidly owing to the increasing number of cross – links formed by curing reaction and attains a nearly constant value when the curing reaction is complete [http://www.netcomposites.com/education.asp?sequence=10; Kiasat, 2000].

Proper flow through fiber network or lay - ups is a critical in producing void - free specimens and good fiber wet - out. In thermoset resins, curing may take place simultaneously with resin flow, and if resin viscosity rises too rapidly due to curing, its flow may be inhibited, causing voids and inter-laminar adhesion. At moderate pressure the fiber bodies are entangled and act as a resistance to flow of resin in between the void places. This is explained by the 3 – D Darcy equation of flow of viscous fluid through a porous resistance. The resin gel time and the increase in
viscosity also affected the resin flow. If the fibrous body is humid to some degree and the liquid polymer or melt has sufficiently low viscosity then resin flow competes with the moisture present in the fiber [Kiasat, 2000; http://www.netcomposites.com/education.asp?sequence=10].

Thermosetting resins show a considerable shrinkage during curing. A consequence is that a considerable constraint on shrinkage is present in composites, due to the presence of stiff fibers. The constraint on shrinkage causes stresses in the resin. The resin stress shows relaxation due to viscoelastic material behavior. However, new shrinkage stresses occur simultaneously due to further curing [Kiasat, 2000; http://www.netcomposites.com/education.asp?sequence=10].

Unsaturated polyester (UP) resin is one of the most commonly used thermosetting resins for composite applications. Glass-fiber reinforced unsaturated polyesters (G/UP) and carbon fiber reinforced epoxies are structural materials, which have widely been used, in the automotive and aerospace industry. The mechanical performance of the composite materials is affected by the processing-induced residual stresses. Residual stresses occur because of curing shrinkage of the resin. Thermosetting resins solidify due to the cross linking of the polymer chains during a chemical curing reaction. A significant volumetric shrinkage occurs during curing of the resin due to the formation of the chemical bonds. The simultaneous occurrence of the curing shrinkage and the solidification of the resin induces the residual stresses when the resin is subjected to dimensional constraints as in a composite material. Although the resin is highly viscoelastic at the curing temperature and thus the stress build-up is partly reversed by the stress relaxation, significant stresses are measured in the resin specimens, who are one-, two-, or three dimensionally constrained. Quite high residual stresses are micro mechanically predicted to develop in the composites during curing. The residual stresses cause deformations, shape inaccuracies, and the initiation of damage in the composite products.


The principal disadvantage of polyesters over epoxies is their high volumetric shrinkage. Although this allows easier release of parts from the mold, the difference in shrinkage between the resin and fibers results in uneven depressions (called sink marks) on the molded surface. The sink marks are undesirable for exterior surfaces.
requiring high gloss and good appearance (e.g., class A quality in automotive body components). One way of reducing these surface defects is to use low-shrinkage (also called low-profile) polyester resins that contain a thermoplastic component (such as polystyrene or polymethyl methacrylate).

As curing proceeds, phase changes in the thermoplastic component allow the formation of microvoids that compensate for the normal shrinkage of the polyester resin. When casting neat (unreinforced) polymers for use as mechanical test specimens, it is critical that voids, inclusions, and similar defects be minimized, both in size and number. Most thermoset polymers used as matrices, even those considered to be toughened, tend to be relatively brittle, and thus their ultimate strengths are strongly dictated by critical flaw size.

Inclusions can be present in impure resin as obtained from the supplier, or introduced during the fabrication process (e.g., inadequately cleaned molds, airborne dirt particles, inadequate mixing of components, etc.). Caution also must be exercised when using release agents, to avoid contamination of the polymer. Defects can be in the form of surface scratches, edge chips, and mold marks. Voids are typically caused by trapped volatiles, which evolve during the initial stages of the curing process. The evolution of volatiles can be suppressed, or at least minimized, by subjecting the polymer to pressure during the curing process. However, it is more common to apply a vacuum during the initial stage of the cure cycle, either while the polymer is still in the mixing container or already in the mold. This is done at one or more points in time as the temperature is being elevated, and while the viscosity is at its lowest.

2.11 POSSIBLE ORIENTATION OF FIBERS IN COMPOSITE AND MECHANICAL PROPERTIES

In case of the fiber reinforced polymer the fibers are more or less arranged in a pattern shown in the figure 2.10 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.
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. Nowadays the bulk molding compound technology (BMC) and sheet molding composite technology (SMC) 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.

Mechanical properties are often the critical factors in predicting polyester resin composite for a specific application. The mechanical properties are substantially influenced by the selection of monomeric diols or dibasic acids (building blocks of polyester), their amounts temperature of processing and the amount of reinforcement used. While tensile and flexural strength characteristics are only slightly affected by different formulations but it is also specifically influenced by the presence of fiber. 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
(http://info.lufarmingdale.edu/depts/met/met205/composites.html).
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 (http://info.lufarmingdale.edu/depts/met/met205/composites.html).

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
(http://info.lufarmingdale.edu/depts/met/met205/composites.html).

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 2.11.

![Figure 2.11: Schematic representation of the mechanical properties of fiber, resin matrix & composite.](http://example.com/image.png)
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 [http://info.lufarmingdale.edu/depts/mct/mct205/composites.html].

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).
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 [http://info.lu.farmingdale.edu/depts/met/met205/composites.html]. 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 [http://info.lu.farmingdale.edu/depts/met/met205/composites.html].

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 [http://info.lu.farmingdale.edu/depts/met/met205/composites.html].

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 [Gao, 1992].

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 [Gao, 1992].

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 [Gao, 1992].

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 [Gao, 1992].

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 [Gao, 1992].

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 [Gao, 1992].

Normally the mechanical property of composite is mathematically expressed as the following equation

\[ X_c = X_f \nu_f + X_m \nu_m \]  \hspace{1cm} (2.1)

\[ \nu_f + \nu_m = 1 \]  \hspace{1cm} (2.2)

Where 'X' is the property of concern, '\nu' 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.

\[ \sigma_c = \sigma_f \nu_f + \sigma_m \nu_m \]  \hspace{1cm} (2.3)
Where the \( \sigma \) 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 equation 2.3 the longitudinal tensile strength of the composite body can be estimated as

\[
\sigma_{lu} = \sigma_f \nu_f + \sigma_m' (1 - \nu_f)
\]

(2.4)

Where \( \sigma_{lu} \) is the longitudinal tensile strength of unidirectional chopped fiber reinforced composite specimen, \( \sigma_f \) is the fiber ultimate tensile strength and \( \sigma_m' \) is the matrix stress at fiber failure strain.

For chopped discontinuous and randomly oriented fiber reinforced composites the equation may be written as:

\[
\sigma_{lu} = \sigma_f \nu_f (1 - \frac{L}{2L}) + \sigma_m' (1 - \nu_f)
\]

(2.5)

Where \( \sigma_{lu} \) is the longitudinal tensile strength of chopped random fiber reinforced composite specimen and \( L \) is the critical fiber length.

For effective reinforcement of the matrix, the fiber volume fraction and length of fiber in the composite must be greater than a critical value, i.e. \( L > L_c \). \( \tau \) is the interfacial shear strength and \( V_f \) is the critical fiber volume in the composite.

\[
V_f = \frac{\sigma_{lu} - \sigma_m'}{\sigma_f - \sigma_m'} \quad \text{and} \quad L_c = \frac{\sigma_f d_f}{2\tau}
\]

(2.6)

2.12 FIBER MATRIX COMPATIBILITY

The mechanical properties of natural fiber reinforced polymer composites in many ways dependent on the interface formed between the fiber and the matrix. To understand the bonding nature between the fiber and the matrix it is essential to know the types of chemical reaction or physical interaction and the types of bonds formed during these chemical or physical reactions at the fiber matrix interface. Both the natural fiber and polymeric resin materials possess covalent bonds, so if the natural fibers and polymer reacts chemically at the interface then the types of bond stretching can be analyzed by the Infra Red (IR) method. Or more precisely the fiber – polymer interfacial bonding of the natural fiber reinforced polymer composite nature can be studied by Fourier Transformation Infra Red spectroscopic method.
In the most basic terms, the infrared spectrum is formed because of the absorption of electromagnetic radiation at frequencies that correlate to the vibration of specific sets of chemical bonds from within a molecule. First, it is important to reflect on the distribution of energy possessed by a molecule at any given moment, defined as the sum of the contributing energy terms. Thus the total energy can be stated by the following equation that has the value of:

\[ E_{\text{total}} = E_{\text{electronic}} + E_{\text{vibrational}} + E_{\text{rotational}} + E_{\text{translational}} \] (2.6)

Molecular bonds vibrate at various frequencies depending on the elements and the type of bonds. For any given bond, there are several specific frequencies at which it can vibrate. According to quantum mechanics, these frequencies correspond to the ground state (lowest frequency) and several excited states (higher frequencies). One way to cause the frequency of a molecular vibration to increase is to excite the bond by having it absorb light energy. For any given transition between two states the light energy (determined by the wavelength) must exactly equal the difference in the energy between the two states (usually ground state \( E_0 \) and the first excited state \( E_1 \)) [http://sis.bris.ac.uk/~sd9319/spec/IR.html#IRspec]. The difference in the energy states closely equals the absorbed light energy, which can be equated by the following equation and has the value of...

\[ E_1 - E_0 = \frac{hc}{\lambda} \] (2.7)

Where \( h \) is the Planck's constant, \( c \) is the speed of light, and \( \lambda \) is the wavelength of incident light.

The energy corresponding to these transitions between molecular vibrational states is generally 1-10 kilo calories/mole, which corresponds to the infrared portion of the electromagnetic spectrum.

In a view to find out information about the bonding characteristics of the jute fiber and unsaturated polyester resin composite Fourier Transformation Infra Red (FTIR) spectroscopic method was carried out.
2.12.1 Considerations for Interpretation of Infrared Spectra

- It provides information about the presence and absence of functional groups.
- Functional group region roughly $4000 - 1350\text{ cm}^{-1}$ and the fingerprint region from $1350 - 450\text{ cm}^{-1}$, for functional groups weak bands are also useful.
- One band assigned to a functional group should be confirmed by absorption band(s) in other part of the spectrum in a FTIR spectrograph. The intensity of absorption bands depends on the number and type of functional groups present.
- Intense absorption usually show weak band at twice the wave number.

The atoms that are sensitive to IR radiation of particular frequency and this absorbed frequency include the vibrational energy state of the absorbing atom. Atoms reach to higher vibrational energy state absorbing IR radiation. Irradiation and absorption is a quantized process. Only particular group of frequency or energy is absorbed by particular functional group. Web numbers vs. %transmittance curves were drawn.

Jute polyester composite may have such an interface, where there could be perfect bonding of chemical nature, may be mechanical interlocking or may be some different types of bonding system. This FTIR studies may help us to explain the bonding characteristics of these jute polyester composite. FTIR spectra of jute and unsaturated polyester resin will be explained and then the composite spectra will be analyzed.

2.13 MECHANICAL PROPERTIES OF COMPOSITE

Zafeiropoulos [2002] focused on the flax/polyolefin composite properties on acetylation & 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 for the treated & untreated fiber; X - ray diffraction, scanning electron microscopy and inverse gas
chromatography. 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 & chemicals.

Zafeiropoulos [2002] 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.

Joseph [2002] fabricated composite with banana fiber & 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 & 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.

Escamilla [2002] investigated the mechanical behavior of rigid thermoplastic matrix-cellulose fiber composite. Investigation concluded that these materials exhibit a brittle behavior, i.e. they possess 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 KN. 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 PBA (grafted cellulose fiber) seem to provide an alternative method of energy dissipation in the composite, thus, showing a better impact behavior than 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.

Ray et al [2001] 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.

Ray et al [2002] 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. 
Gassan [2002] studied the tension - tension 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 possesses 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.

Gassan [2001] 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 [2000] 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 assess 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 & 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 fiber-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.

Cichocki Jr. [2002] 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 & 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.

Kugler [2002] 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 compliant ring high density polyethylene on the outside of the complaint ring. Analysis is presented to calculate the stress and strain in the composite.

Khondker [2004] 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.

Khan [2004] 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 $0^\circ$ (longitudinal) and $90^\circ$ (transverse) directions of the composites. Thermodynamic properties such as storage modulus, loss modulus and $\tan \delta$ of the composite were performed in $0^\circ$ (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 $\tan \delta$ 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.
Pickering et al. [2004] investigated the effect of a maleated polypropylene- (MAPP) coupling agent on the mechanical properties of composites based on 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 307% for Young's modulus were obtained by using 60 (wt)% fibre and 2 (wt)% MAPP, compared to the unreinforced matrix. Fibre 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 of BP, after which a decrease was observed.

Pickering et al. [2004] in another paper stated single fibre tests on four different types of natural fibre. 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 thinnings 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 maleic 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. Slabwood composites had the highest tensile strength and Young's modulus value among the wood fibre composites.

Pickering et al. [2004] also showed that hemp fibre 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-fibre reinforced polypropylene composite by optimising the fibre strength, fibre processing, composite processing and fibre-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 fibre treatment method to separate the fibres from their fibre bundles and to remove lignin and pectin, whilst retaining the fibre strength. An alkali treatment consisting of digesting the fibres in a 10% NaOH solution at a maximum temperature of 160°C and a hold time of 45 minutes was found to produce strong fibres with low lignin content and good fibre separation. Finally, the alkali treated fibres, polypropylene and a maleated polypropylene (MAPP) coupling agent were compounded in a twin screw extruder, and injection moulded into composite tensile test specimens. The strongest composite produced consisted of polypropylene with 40% wt fibre and 2% wt MAP, and had a tensile strength of 38 MPa, and a stiffness of 4 GPa.

Khan [2004] in another work explained that due to biodegradable and ecofriendly behaviors, natural fibres 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.

2.14 CURRENT CONSIDERATION AND OBJECTIVE

Bangladesh has huge jute resource. Jute has long been used as textile and household raw material. But in last two or three decades some exciting properties of jute has
been discovered e.g. strength and stiffness compared with the artificial high cost fibers like glass, aramid asbestos etc. also the nonabrasive properties of jute fiber and strength consideration encouraged the researchers to use jute as a low cost reinforcement in some polymer matrix. Composite materials becoming familiar materials nowadays because of its versatile property and compatibility compared other monolithic materials. The present focus in Bangladesh is to produce artificial fiber e.g. glass fiber, nylon fiber etc. reinforced polymer composite especially for home appliance products. But the emphasis of this industrial revolution is not focused on environment, and it is hard to assess the future impact of these materials on our environmental condition. Jute fiber is hydrophilic in nature and the superficial properties can be altered via chemical treatment to make matrix compatible reinforcing fiber. This low-cost reinforcement can reduce the cost associated with production of structural materials e.g. home appliances, automotive structures, aerospace application etc. The specific aim of this project is to utilize countries natural fiber resources for fabricating natural fiber reinforced polymer composite and to develop the physical and mechanical property of composites through studying the fiber-polymer compatibility of composites. Especially the project was undertaken to create a basis of developing the natural fiber reinforced polymer composite.
Chapter 3
Experimental
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) Retted jute, b) Retted and bleached jute and c) Retted and woolenized jute.

Retting means simply the water retting 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 it is available in literature as well. Table 3.1 provides brief information about the jute fiber types, treatment procedure and place of allocation.

Five types of woven jute fabric namely a) Fine Jute Fabric Natural b) Food Grade Benolla Twill Bag, c) Hessian Fabric d) Fine Jute Fabric Full Bleached Stiff, and e) Carpet Backing Cloth (CBC) Half Bleached were obtained from Bangladesh Jute Mills Corporation (BJMC) and the woven fabric that were collected from BJMC was of same treatment as the fibers, and no information about the treatment procedure of the CBC half bleached woven fabric was provided. The chopped strand mat of glass fiber of 300gram/m² was collected from the local market that was imported from Singapore Highpolymer Company Ltd (SHCP).

3.1.1 Nomenclature of Jute Fiber, Woven Jute Mat

Table 3.1 represents the types of jute fiber along with their respective treatment procedure and nomenclature.
Table 3.1: Types of Jute fiber

<table>
<thead>
<tr>
<th>Name of the Jute Fiber</th>
<th>Treatment</th>
<th>Identification</th>
<th>Source</th>
<th>Pictorial view</th>
</tr>
</thead>
<tbody>
<tr>
<td>Retted Jute</td>
<td>Simple water retting</td>
<td>&quot;JR&quot; implies Jute Retted</td>
<td>BJRI</td>
<td>![Pictorial view of Retted Jute Fiber]</td>
</tr>
<tr>
<td>Retted and Bleached Jute</td>
<td>BJRI Specified Bleaching Procedure</td>
<td>&quot;JRB&quot; implies Jute Retted and Bleached</td>
<td>BJRI</td>
<td>![Pictorial view of Retted and Bleached Jute Fiber]</td>
</tr>
<tr>
<td>Retted and woolenized Jute</td>
<td>BJRI Specified woolenization (alkali treatment procedure).</td>
<td>&quot;JW&quot; implies Jute Retted and woolenized</td>
<td>BJRI</td>
<td>![Pictorial view of Retted and woolenized Jute Fiber]</td>
</tr>
</tbody>
</table>

Table 3.2 represents the types of woven jute mat along with their respective types, wt per square meter and nomenclature. All the fibrous samples were obtained from BJMC.
<table>
<thead>
<tr>
<th>Name of woven jute fabric</th>
<th>Types / thickness &quot;mm&quot;</th>
<th>Unit weight gm/m²</th>
<th>Nomenclature</th>
<th>Obtained from</th>
<th>Photograph</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fine jute fabric natural</td>
<td>15x15 inches², 1mm</td>
<td>260gm/m²</td>
<td>JRa1</td>
<td>BJMC</td>
<td></td>
</tr>
<tr>
<td>Food grade, Benolfa Twill Bag</td>
<td>Not specified by BJMC, 15mm</td>
<td>600gm/m²</td>
<td>JRa2</td>
<td>BJMC</td>
<td></td>
</tr>
<tr>
<td>Material</td>
<td>Description</td>
<td>Weight (g/m²)</td>
<td>Grade</td>
<td>Source</td>
<td></td>
</tr>
<tr>
<td>--------------------------------</td>
<td>------------------------------</td>
<td>---------------</td>
<td>-------</td>
<td>--------</td>
<td></td>
</tr>
<tr>
<td>Hessian fabric</td>
<td>Not specified by BJMC, 1mm</td>
<td>100</td>
<td>JRa3</td>
<td>BJMC</td>
<td></td>
</tr>
<tr>
<td>Fine Jute Fabric</td>
<td>full bleached, stiff</td>
<td>300</td>
<td>JRBx1</td>
<td>BJMC</td>
<td></td>
</tr>
<tr>
<td>Carpet Backing Cloth</td>
<td>half bleached</td>
<td>335</td>
<td>JRBx2</td>
<td>BJMC</td>
<td></td>
</tr>
</tbody>
</table>
3.1.2 Identification of Woven Jute Mat

It was found that the number of yarn that are needed to weave one square inch of woven mat is the type of that mat, regarding this for example fine jute fabric natural is typed as 15/15 that implies each square inch of the woven jute fabric contains 15 yarn / 15yarn placing there position 90° apart each other forming the fabric. The following figure 3.1, will clarify the facts about the types of jute fabric.

As we see in figure 3.1 it is quite clear that the number of yarns that are needed to fabricate a unit area of woven fabric of jute is the type of that jute fabric. This case was observed during the course of work. The types of these fabrics are given in square inch.

For jute mat, grams per square meter was calculated using the following steps:

- Fabrics were cut to a particular length, breadth and with an area of 35/30cm².
- Converted the area of the woven fabric to square meter.
- Taken the weight of the cut piece of the woven fabrics.
- Multiplied the weight of the fabric with the calculated area in meter, which gives the unit gram weight of the woven fabrics.

3.1.3 Identification of Chopped Glass Strand Mat

The chopped glass strand mat of 300gm / m² was obtained from the local market (Almai fiberglass ltd.) that has been imported from SHCP Singapore.
The figure 3.2 shown above is the chopped glass strand mat, also known as E-glass strand mat. There is no weaving in chopped strand mat instead it is randomly oriented and bonded with suitable binder. E-glass bound with a slow wetting binder, or is made from fiberglass strands chopped to length and bonded together with powder binder or emulsion binder, which are most likely the silane polymer or polyester powder that binds the chopped fiber together. It is made of E-glass fiber strand or roving which is chopped into 50mm long and distributed on mat machine uniformly and continuously. Powder polyester is used. All through the text the glass fiber is designated as GFRP.

3.2 SEM IMAGING OF JUTE FIBERS

The Scanning Electron Microscope, or SEM was utilized to find out the 3-dimensional images of the three types of treated jute fibers. The SEM shows very detailed surface topography at much higher magnifications than that of light microscope shows. The images created without light waves are rendered black and white. The project work utilizes 10KeV source for imaging. In order to assess the effect of the treatment upon jute fiber surface, characterization of three types jute fibers was done in an Scanning Electron Microscope (SEM) (Model no XL - 30, SL no - D1276, 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 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 the operating voltage was kept constant and a thicker layer of gold was deposited instead of usual 40nm thickness [Zafeiropoulos, 2002; http://www.msc.mtu.edu/~jwdrelie/LabSEM.pdf; Alexander Bismarck, 2001].

3.2. Sample Preparation for Imaging

The SEM used to produce the images in this project is operated under high-vacuum conditions, and a battery of pumps serves 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 (minerals, semi-conductors, metals, etc.), it is a major concern when one wishes to examine biological samples such as jute. Jute samples contain large amounts 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.

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 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". 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.

Jute Samples were prepared carefully to withstand the vacuum inside the microscope. Since jute is a biological specimen along with hydrophilic and hygroscopic 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±1mm in length and allowed to dry at 100°C in a resistive heating type oven, for 12 hours [Rowell, 1998; Zafeiropoulos, 2002; http://www.msc.mtu.edu/~jwdrelic/Lab6SEM.pdf]. 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 was applied on the jute fiber [Zafeiropoulos, 2002].

It was then 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, was output to a monitor for live-time viewing or to a film-recording device for photographic output.
3.3 FABRICATION OF COMPOSITE

Two types of composites were fabricated. The chopped jute fiber composite and the others are the laminated (jute mat) composites.

3.3.1 Fiber Chopping

Jute fibers (Retted, Retted & Bleached, Retted & Woolenized) were chopped to a length of 5±1mm, using scissors, with care, at Bangladesh Industrial Technical Assistance Center (BITAC). Careful measurement of the fiber length was carried out and the chopped fiber length of 5±1mm was ensured. The chopped fibers were individually stored in an airtight bag.

3.3.2 Woven Fabric Cutting

Fine Jute Fabric Natural, Food Grade Benolla Twill Bag, Hessian Fabric, Fine Jute Fabric Full Bleached Stiff and Carpet Backing Cloth Half Bleached Woven fabrics were cut into 35×30cm² dimension and hand lay-up technique was used to fabricate the woven fabric laminated specimens.

Chopped glass fiber strand mat was cut into 35×30cm² dimension as that of the woven jute fabric. The cut piece of these mats was stored individually inside polythene bag to keep it moisture free.

3.3.3 Composites Preparation

Resin materials were obtained from Singapore Highpolymer Chemical Products Pte Ltd (SHCP). The specification of the resin is general-purpose unsaturated polyester resin of grade SHCP 021073, Type 27191 OM was used to fabricate the composite specimen, which is general purpose (ortho) roof grade and pre accelerated with cobalt naphtthanate promoter (6% active cobalt) of 0.5% (w/w) of resin along with the following additives with unknown percentage; such as Aluminum Trihydrate
(ATH), Antimony Trioxide, Methyl Methacrylate (MMA), Di-Methyl Aniline (DMA), UV Stabilizer, Inhibitors, Low Styrene Emission (LSE), Air Release Agent. The use of jute fiber as a reinforcement medium for thermoset plastics and the application of the resultant composite have come to face nowadays. Reinforced plastic as a finished product also led to the development of a wide range of fabrication technique. Today larger choices of process for FRP coexists, individually suitable to product configuration, volume of production, design considerations, tooling, than it does for standard materials e.g. wood, steel or aluminum. This allows the user to dictate the parameters of quality and quantity. The fundamental method of creating an FRP product, essentially involves impregnation of the reinforcement with a liquid resin and curing the composite under ambient or increased temperature or pressure.

3.3.4 Preparing The Resin

The degree of wetting during the production process is important for a good adhesion between fiber and matrix. When applying thermoset the viscosity can be low, which eases the wetting. Problems that can be encountered are related to moisture and air. The fiber moisture can affect the chemical reaction. In order to prevent this, the fibers were dried. In order to fabricate a specimen the resin was prepared properly and in a safe manner and the following steps were maintained.

1. Pre-promoted and pre-accelerated resin system was used that was supplied and the additive levels were pre-adjusted from the company.
2. The time required for fabrication was estimated, taking into account the resin and air temperature.
3. The required amount of pre-promoted, pre-accelerated and pre blended resin system was weighed.
4. Appropriate amount of MEKP initiator was taken usually 1% v/w to that of resin blend.
5. The initiator was thoroughly mixed in resin system with a stirrer. When gel time is too short due to working condition promoter and accelerator level can be adjusted in order to lengthen gel time and must not exceed the MDS.
Materials safety data sheet: Specification. Promoter and accelerator was thoroughly mixed with resin blend system to avoid the explosion hazard. Mixing promoter and accelerator directly with MEKP initiator system can cause violent explosion.

3.3.5 Jute preparation

Utilization of chopped jute and jute mat was undertaken during the course of the project. Chopped jute fiber composite specimens were prepared with 5, 10, 15, and 20 volume percentage of chopped jute using cold press molding. It is to be noted that jute fiber and fabric all were put in to an oven at 70 - 100°C for 12 hours prior to fabrication for the composite specimen to ensure moisture removal of the fiber and fabric [Ray, 2001].

3.3.6 Cold Press Molding

In order to be used in the fabrication of products, the liquid resin must be mixed with a catalyst to initiate polymerization into a solid thermoset. Catalyst concentrations generally range from 1 to 2 percent by original weight of resin; within certain limits, the higher the catalyst concentration, the faster the cross-linking reaction proceeds. Common catalysts are organic peroxides, typically methyl ethyl ketone peroxide (MEKP). Resins may contain inhibitors, to avoid self-curing during resin storage, and promoters, to allow polymerization to occur at lower temperatures.

Cold press molding presses are oriented vertically and contain two platens to which the mold halves are fastened but during the project, a mold was used, as shown in figure 3.3. The mold body was so prepared to facilitate proper clean up and ease of isolation of its component. The presses involve either of two types of actuation: (1) upstroke of the bottom platen or (2) down stroke of the top platen, the former being the more common machine configuration.

Figure 3.3 shows the cold press setup used for the present study, which is suitable for relatively simple shaped component processing. Incorporating a male and female mold of steel construction prepared for quick release, this process allows for medium
volume output of repetitive at low capital investment. The chopped jute is dried in an oven for 12 hrs at 100°C. The oven-dried fibers were packed airtight bag prior to molding. The chopped jute fiber reinforcement is spread in female mold half incorporating a prelayer of resin; next another layer of resin along with the rest of the chopped jute fiber reinforcement, which is then fully covered with the rest of the prepared resin.

The process needs 2 to 3 min to complete the resin – reinforcement incorporation in female mold. After filling the male mold half on the female mold half and three plates of one inch thick and approx 12 kg of wt was placed at the top of male mold. The application of pressure assists the resin to force and penetrate in all parts of the mold and the exothermic heat generated by the hardener action during polymerization reaction enhances curing. The total cycle time for processing is as follows

- Cleaning the mold - 15 min.
- Waxing the mold thoroughly to assist smooth discharge of the composite specimen - 10 min.
- Weighing resin and oven dried reinforcement individually according to the required volume or weight percent of the reinforcement / resin ratio - 5min.
- Initiator mixing - 1 min.
- Resin reinforcement incorporation in mold - 3min.
• Pressure plate setting on mold - 0 5 min.
• Pressing begins at the 5th min, just after the initiator mix-up.
• Pressing up to the marked portion within 1 min.
• Holding for 5 min.
• Relaxation of pressure allowed for 5 min.
• Increasing pressure for the second and last time within 1 min.
• Relaxation of pressure allowed for 5 min.
• Gel time completion in between 25th to 30th min after hardener mixing, sensed by constant indication of load from the molding machine.
• Curing time 2 hr allowed to avoid bulging effect of the fabricated composite.
• Specimen discharge within 15 min.

Total space-time needed 3hr 45min up to 4hr. for fabrication of composite specimen.

3.3.7 Hand Lay-up

Figure 3.4 shows the typical hand lay-up procedure for laminated composite fabrication where resins are impregnated by hand into fibres, which were in the form of woven, knitted, stitched or bonded fabrics. Rollers or brushes, with an increasing use of nip-roller type impregnators for forcing resin into the fabrics by means of rotating rollers and a bath of resin, usually accomplish this. Laminates were left to cure under standard atmospheric conditions.

Gloves, respirator mask and eye protection were used.

Hand lay-up requires a clean flat surface as the working surface; it forms the bottom texture of the plate.
• A clean plastic sheet was placed on the flat surface and waxed on the surface with normal car polishing wax (Japanese wax), for easy removal of the part.

• The fibers were cut (both Mat and Woven fibers). Care was taken while handling the fibers. Improper handling will cause damaged and distorted fibers and tools for hand lay-up like brush, serrated roller should be clean and non sticky and was accomplished by acetone wash.

• The resin was mixed in a beaker with the proper ratio (inside the fume hood), as per the given instruction of mixing in the material safety and data sheet. The quantity of the resin depends on the size of the plate made.

• A first coat of the resin was applied on the waxed plastic sheet. The Mat was placed on top of that and the resin was applied using the brush. The serrated roller was used to even out the resin and remove any entrapped air in the resin. The woven fabric layer is placed on top of the first layer, and the above process was repeated.

Further layers were piled up on top of others with resin and serrated roller was used to drive the air out and to ensure proper compaction. After the final layer (Mat always) is laid, it is covered with a waxed plastic sheet, and a flat plate is placed on the top with a weight to ensure a smoother surface. The whole set is allowed to sit for the required time for cure.

The table 3.3 shows the name and identification of the fabricated specimen. A total number of seventeen specimens were fabricated and as mentioned earlier, the chopped fiber reinforced composites were fabricated using cold press molding and the laminated ones were fabricated by hand lay-up procedure.

During fabrication of composite it was always found that required volume of fiber/mat couldn't be maintained due to process loss of resin. In case of higher volume fraction of fiber, if the amount of resin is low, increased pressure is necessary along with proper control of gel time of the liquid resin. As a result higher amount of resin was taken to facilitate squeeze out of excess resin from the mold.
<table>
<thead>
<tr>
<th>SI</th>
<th>Matrix</th>
<th>Jute treatment condition and reinforcement name</th>
<th>Fabrication procedure</th>
<th>No of ply / Volume %</th>
<th>Nomenclature</th>
<th>Pictorial reference</th>
<th>Dimension</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>Retted</td>
<td>Cold press molding</td>
<td>Y</td>
<td>0</td>
<td>Reference</td>
<td>350×300×11 mm³</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>Retted and Bleached</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>350×300×12 .5mm³</td>
</tr>
<tr>
<td>3</td>
<td>Polyester</td>
<td>Chopped retted</td>
<td>Y</td>
<td></td>
<td>6.50%</td>
<td>JR5</td>
<td>294×292×12 mm³</td>
</tr>
<tr>
<td>4</td>
<td>Polyester</td>
<td>Chopped retted</td>
<td>Y</td>
<td></td>
<td>10.63%</td>
<td>JR10</td>
<td>294×292×11 .5mm³</td>
</tr>
<tr>
<td>5</td>
<td>Polyester</td>
<td>Chopped retted</td>
<td>Y</td>
<td></td>
<td>19.98%</td>
<td>JR20</td>
<td>294×292×12 .5mm³</td>
</tr>
<tr>
<td>6</td>
<td></td>
<td>Chopped retted and woolenized</td>
<td>Y</td>
<td></td>
<td>5.46%</td>
<td>JW3</td>
<td>294×292×10 .5mm³</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td>Chopped retted and woolenized</td>
<td>Y</td>
<td></td>
<td>9.46%</td>
<td>JW10</td>
<td>294×292×11 mm³</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td>Chopped retted and woolenized</td>
<td>Y</td>
<td></td>
<td>26.49%</td>
<td>JW20</td>
<td>294×292×10 .5mm³</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Chopped retted and bleached</td>
<td></td>
<td>Y</td>
<td></td>
<td>7.24%</td>
<td>JRD6</td>
</tr>
<tr>
<td>---</td>
<td>---</td>
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<td>---</td>
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<td>------</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td>Chopped retted and bleached</td>
<td></td>
<td>Y</td>
<td></td>
<td>13.72%</td>
<td>JRB13</td>
</tr>
<tr>
<td>11</td>
<td></td>
<td>Chopped retted and bleached</td>
<td></td>
<td>Y</td>
<td></td>
<td>14.96%</td>
<td>JRB15</td>
</tr>
<tr>
<td>12</td>
<td></td>
<td>Chopped retted and bleached</td>
<td></td>
<td>Y</td>
<td></td>
<td>23.73%</td>
<td>JRD23</td>
</tr>
<tr>
<td>13</td>
<td></td>
<td>Fine jute fabric natural</td>
<td></td>
<td></td>
<td>Y</td>
<td>13 ply</td>
<td>18.9877%</td>
</tr>
<tr>
<td>14</td>
<td></td>
<td>Food grade bengal twill bag</td>
<td></td>
<td></td>
<td>Y</td>
<td>8 ply</td>
<td>23.28%</td>
</tr>
<tr>
<td>15</td>
<td></td>
<td>Hessian fabric</td>
<td></td>
<td></td>
<td>Y</td>
<td>13 ply</td>
<td>15.7%</td>
</tr>
<tr>
<td>16</td>
<td></td>
<td>Fine jute fabric full bleached stiff</td>
<td></td>
<td>Y</td>
<td>13 ply</td>
<td>21.9%</td>
<td>JRa1</td>
</tr>
<tr>
<td>17</td>
<td></td>
<td>Carpet backing cloth half bleached</td>
<td></td>
<td>Y</td>
<td>13 ply</td>
<td>19.52%</td>
<td>JRD2</td>
</tr>
</tbody>
</table>
Woven fabrics have constant ability to absorb liquid resin up to its saturation point, within the first few minutes of gel time of the liquid thermosetting resin. This is known as impregnation factor, which has a constant value and is known as the characteristics of these individual woven fabrics. Application of high pressure on these laminates has some impact on these specimens. The impregnation factor increases when pressure is applied during fabrication of these composites, but at higher pressure, other than normal hand lay-up process, empty space between fibers decreases, higher pressure assists in embedding higher number of ply within polymer matrix but that does not imply higher impregnation factor. Table 3.4 represents the details of fabricated specimen.

3.4 MECHANICAL CHARACTERIZATION OF COMPOSITES

3.4.1 Standardization and Practices

Mechanical characterization of these fabricated composite was done using Universal Testing Machine (UTM, UH – 500kNA, Capacity 500kN, Shimadzu Corporation, Japan). 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.
Table 3.4: Specimen naming, identification, and respective fabrication procedure

<table>
<thead>
<tr>
<th>Name</th>
<th>Symbol</th>
<th>Fabrication procedure</th>
<th>No of Tensile specimen tested</th>
<th>No of Flexure specimen tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference polymer</td>
<td>Ref</td>
<td>Hand lay-up</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Glass fiber reinforced polyester</td>
<td>GFRP</td>
<td>Hand lay-up</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted jute fiber reinforced polyester (5 vol%)</td>
<td>JR5</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted jute fiber reinforced polyester (10 vol%)</td>
<td>JR10</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted jute fiber reinforced polyester (20 vol%)</td>
<td>JR20</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (6 vol%)</td>
<td>JRB6</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (13 vol%)</td>
<td>JRB13</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (15 vol%)</td>
<td>JRB15</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (23 vol%)</td>
<td>JRB23</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (5 vol%)</td>
<td>JW5</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (10 vol%)</td>
<td>JW10</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (20 vol%)</td>
<td>JW20</td>
<td>Cold press molding</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Fine jute fabric natural, type 15 x 15 (Retted Variety)</td>
<td>JRA1</td>
<td>Hand lay-up</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Food grade Bennila twill bag (Retted Variety)</td>
<td>JRA2</td>
<td>Hand lay-up</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Hessian fabric (Retted Variety)</td>
<td>JRA3</td>
<td>Hand lay-up</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Fine jute fabric full bleached stiff, type 15 x 15</td>
<td>JRB41</td>
<td>Hand lay-up</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Carpet backing cloth half bleached type 15 x 13</td>
<td>JRB52</td>
<td>Hand lay-up</td>
<td>4</td>
<td>4</td>
</tr>
</tbody>
</table>

The above nomenclature will be followed throughout the text.
3.4.2 Tensile Specimen Preparation

Table 3.5: Standard dimensions for tensile specimen for fiber-reinforced polymer composite (ASTM D 3039)

<table>
<thead>
<tr>
<th>Fiber Orientation</th>
<th>Width, mm</th>
<th>Overall Length, mm</th>
<th>Thickness, mm</th>
<th>Tab Length, mm</th>
<th>Tab thickness, mm</th>
<th>Tab Bevel Angle, °</th>
<th>No of Test specimen for individual composite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Balanced and symmetric</td>
<td>25</td>
<td>250</td>
<td>2.5</td>
<td>Emery cloth</td>
<td>Specimen thickness</td>
<td>None</td>
<td>4</td>
</tr>
<tr>
<td>Random-discontinuous</td>
<td>25</td>
<td>250</td>
<td>2.5</td>
<td>Emery cloth</td>
<td>Specimen thickness</td>
<td>None</td>
<td>4</td>
</tr>
<tr>
<td>Reference</td>
<td>25</td>
<td>250</td>
<td>2.5</td>
<td>Emery cloth</td>
<td>Specimen thickness</td>
<td>None</td>
<td>4</td>
</tr>
<tr>
<td>Glass fiber reinforced composite</td>
<td>25</td>
<td>250</td>
<td>2.5</td>
<td>Emery cloth</td>
<td>Specimen thickness</td>
<td>None</td>
<td>4</td>
</tr>
</tbody>
</table>

It is clearly seen from the table 3.5 that the dimensions for the tensile specimen of composite systems are specified. Since composites were fabricated from chopped discontinuous jute fiber and from woven fabric so the composites falls in the category random – discontinuous and balanced symmetric respectively. According to that the tensile specimen would look like the following figure 3.5. It is clearly specified that the specimen dimension must fulfill the recommended dimension of 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.

![Figure 3.5: Schematic drawing for tensile specimen](image-url)
The dimensions of the tensile specimen are specified in the table 3.4. Constant Head-Speed Tests: A standard head displacement rate of 2mm/min was used for test and the strength and elongation were calculated using the following equations:

Ultimate Tensile Strength (UTS) \( \sigma_{\text{UTS}} = \frac{P_{\text{load}}}{A} \) ..................................................(3.1)

Where \( P_{\text{load}} \) is the load needed to fail the specimen and \( A \) is the cross sectional area of the composite specimen.

\[ \%\text{Elongation} = \frac{\text{Final gage length} - \text{Initial gage length}}{\text{Initial gage length}} \times 100 \] ..................................................(3.2)

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.

### 3.4.3 Flexure Test Specimen Preparation

Figure 3.6 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 \( \text{L/d}=16 \) is acceptable. Some materials require \( \text{L/d}=32 \) to 64 to keep the shear stress low enough. During the project the ratio of \( \text{L to d} \) 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 and it has the value of...

Rate of crosshead motion (mm/mm) \( R = \frac{ZL^2}{6d} \) .................................................. (3.3)

Where \( Z \) is the rate of strain of the outer fiber, mm/mm/min; \( L \) is the length of the support span and \( d \) is the specimen depth.

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) \( \sigma_f = \frac{3PL}{2bd^2} \) ........................................ (3.4)

Where \( P \) is the load at a given point on the load deflection curve, \( L, b \) and \( d \) are the length of the support span, width and depth of the specimen and is expressed in mm. For composite specimens made of brittle polymer \( \sigma_f \) is sometimes stated as \( \sigma_m \) and is known as the flexural stress at break.

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

Strain in the outer surface (mm/mm) \( \varepsilon_f = \frac{6Dd}{L^2} \) ................................................ (3.5)

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 depth.

Figure 3.6: Typical flexure test setup of polymer composite.
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 1:4 to that of the span. The support span and the compression end diameter was 10mm.

3.5 SCANNING ELECTRON MICROSCOPY OF THE FAILED SPECIMEN (TENSILE AND FLEXURAL)

The Scanning Electron Microscope, or SEM was utilized to characterize the fracture surface, crack profile of chopped random discontinuous jute and woven jute laminated composites. The project work utilizes 10KeV source for imaging.

Two types of test were carried out on the entire composite specimens namely tension test and flexure test and so both the types of fracture surface were characterized individually.

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 – D1276, Philips, The Netherlands). A vacuum unit sputter coater (Model no – R7341, 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 [Zafeiropoulos, 2002]. 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.

3.6 FOURIER TRANSFORMATION INFRARED STUDIES OF COMPOSITE

This test was carried out with a view to analyze the bonding characteristics, which is more clearly defined as compatibility in between the fiber and the matrix. The powder of individual specimens were mixed with potassium bromide (KBr) and disc type pellets were made in a vacuum type pelletizer and scanned in computer interfaced
Fourier transformation infrared machine (Model FTIR 8400S, Shimadzu Corporation Japan) at the Institute of Food Science and Technology (IFST), Bangladesh Council for Scientific and Industrial Research (BCSIR). The spectrum obtained was analyzed individually for the fibers, resins and composites and compared with each other to find some idea about bonding characteristics.

3.6.1 Procedure

Samples were ground and "diluted" with KBr powder, with pure KBr powder was used as the blank. Spectral distortions are minimized by keeping the powder granule size of the KBr blank, KBr "diluent", and sample roughly the same. Excessive grinding promotes water absorption by the sample and the KBr [http://sis.bris.ac.uk/~sc9319/spec/IR.htm#third].

First, heated the anvils and the body of the die set before making pellet. This makes them as dry as possible and then dry KBr powder was use. Third, making the anvils, the die set and the powder were all at the same temperature. Hot powder and cold anvils will produce a cloudy wet pellet. Heating the KBr powder in a dry environment will dry it out and storing it in a heated case or desiccator will keep it dry. Used as little KBr powder as possible - usually just barely enough to coat the anvils of the die set so that the face of the anvil is completely and evenly covered with a very thin coat of powder. Grinded sample together with KBr, in a ratio of about 10:1 to 20:1 and avoided using excessive amounts of sample or KBr. The mixed powder was stored in a labeled vial in the desiccator until ready to use. To compress an excessive amount of powder requires considerably more force on the press than is required if the proper amount of powder is used, and if immoderate force is not applied to the excessive powder charge, the pellet will either wedge or contain white spots.
3.6.2 Sample Handling Problem in FTIR

The main sample handling problem in FTIR analysis of solid and semi-solid materials is that nearly all materials are too opaque in their normal forms for direct transmission analysis in the mid-infrared spectral region. Traditionally, the opacity problem has been remedied by reducing the optical density of samples to a suitable level by various methods of sample preparation [http://sis.bris.ac.uk/~sd9319/spec/IR.htm#inspec]. Vacuum press was employed to make the sample disc, so proper equipment orientation and operation is necessary to ensure standard practice. It is usual that when trying to make FTIR sample disc of polymeric and composite materials, either natural or synthetic tend to stick to the dyes, and this problem was solved by vacuum technique.

3.7 DENSITY AND WATER ABSORPTION TEST OF COMPOSITES

Densities of composites were calculated using rule of mixture, which is stated in the chapter 2 in equation 2.1.

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. Factors affecting water absorption include: type of plastic, additives used, temperature and length of exposure. ASTM D-570 was used to find out the water absorption behavior of composites.

For the water absorption test, the specimens are dried in an oven for a specified time and temperature and then placed in a desiccator to cool. Immediately upon cooling, the specimens are weighed. The material is then emerged in water for 24 hours or until equilibrium is attained. Specimens are removed, patted dry with a lint free cloth, and weighed. Water absorption is expressed as increase in weight percent.

Percent Water Absorption = \[ \frac{(\text{Wet weight} - \text{Dry weight})}{\text{Dry weight}} \times 100\% \]...(3.8)
3.7.1 Test procedure

The test specimens (25 mm / 25 mm) were cut from the molded sheets. All samples edges of the samples were sealed with gel coat (usually polyester resin). Samples were dried for 24 hours at 50°. Conditioned samples were then immersed in distilled water at room temperature. Samples were periodically taken out of the water, surface water was wiped off with tissue paper and weighed to the nearest 0.0001 g and the samples were re-immersed in distilled water immediately. Weights of the samples were taken till the sample reached the equilibrium value [http://www.che.iitm.ac.in/~abhijit/nat_rep.pdf].
Chapter 4

Results and Discussion
4. RESULTS & DISCUSSION

4.1 SEM EXAMINATION OF THE RETTED, RETTED & BLEACHED AND RETTED & WOOLENIZED JUTE FIBER SURFACE

4.1.1 Retted Jute

The SEM micrographs of retted jute of different views are shown in figure 4.1, 4.2 and 4.3. All the micrographs reveal that the surface of retted jute is heterogeneous with a relatively high micro-roughness. This may be explained by the fact that the bacterial action removes the amorphous constituents of the fiber during retting in different portions in different areas and, hence, the fiber surface becomes rougher. This bacterial activity causes enzymatic action that facilitates retting process. The arrow indicated in figure 4.1 shows that the exploded blister on the fiber side surface formed during the bacterial action. The formation of blister is attributed to the fact that, jute fiber is a combination of cellulose, pectin, lignin and other organic components, which adhere with each other to form the fiber body, and microbes that are acting during retting process can not attack the whole fibrous body at a time instead acts according to portion where it can penetrate and implement its biological action. Thus there may be a possibility of microbes acting efficiently underneath the formed blister, bursting the blister and removing the fraction of rotted portion of the fiber body. The fiber bundles are also exposed because of this bacterial action and quite clearly identified in the figure 4.1, which results from the combined action of the microbes and the under water retting atmosphere.

Figure 4.2 reveals the microbial action that has removed some of the inner surface of the fiber bundle significantly. This is possibly due to removal of the fiber's outer surface layer along with the dissolution of the pectin, lignin and other gummy and waxy materials at the first stage of the enzymatic action of the microorganism. The spilled out frontal portion indicated by arrow is the scissor mark and not the effect of retting. All the micrographs (figure 4.1, 4.2) of the retting treatment 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 figures 4.1, 4.2 and 4.3 that the fiber surface
seemed to be covered by a thin layer of wax or simply the fiber surface itself appears smooth. 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.

The micrograph of retted jute in figure 4.3 shows that the fiber is covered with a layer, whose composition probably is of waxy substance. This assumption is supported from spectroscopic results, which are discussed elsewhere [Eichhorn, 2001]. It can be seen from figure 4.3 that after retting the waxy layer is not evenly distributed along the fiber surface, but their thicknesses varies from point to point and withstand dissolution from bacterial enzymatic action.

Micrographs (figure 4.1, 4.2 and 4.3) 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 loses weight because of the treatment, which therefore implies that, more enzymatic action of the microbes on the fiber surface that removed the binder materials [Eichhorn, 2001]. The amount of weight loss was not measured in the study.

4.1.2 Retted & Woolenized Jute

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 or 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 4.4. Such changes are imperfectly 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 [Ray, 2002]. 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 [Eichhorn, 2001; Rowell, 1998]. This may be due to reaction of alkali with the cementing materials of the fiber, which is shown in figures 4.4, 4.5 and 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 4.4 that the 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\(^1\) 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.

\(^1\) Although treatments effected on the linear density of the jute fiber but jute fiber has a range of density, and for ease of our calculation the density 1.3g/cm\(^3\) was taken
Figure 4.4: SEM image of retted and woolenized jute, view of leached out section.

Figure 4.5: SEM image of retted and woolenized fiber, view of terminal curved portion.

Figure 4.6: STM image of retted and woolenized jute, middle portion view.
Figure 4.5 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 4.5 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 [Ray, 2002].

This may be because; the alkali reacts with the cementing materials of the fiber, particularly hemicellulose. This 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 increases the effective surface area available for wetting by the matrix.

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 result in improved surface tension, wetting ability, swelling, adhesion and compatibility with polymeric materials.

4.1.3 Retted & Bleached Jute

Bleaching procedure is used to improve the whiteness of the textile by decolorizing it from the gray state. Although cellulose fiber is white in color, due to residual lignin traces remaining on the fibers, the fiber appears creamy or brownish. In the present study jute fibers were bleached by peroxide bleaching technique. The effect of bleaching treatment on jute fiber is not well understood and fewer articles explained the bleaching effect on the composite mechanical properties [Eichhorn, 2001; Rowell, 1998]. Micrographs (figure 4.7, 4.8 & 4.9) of macro- and micro fibrils represent the construction units of the cellulose fiber cell-wall architecture, which is characterized by layers differing in fibril texture. The fibers consist of different layers, with the fibril position giving different densities and textures.
Figure 4.7: SEM Image of Retted & Bleached Jute, middle portion

Figure 4.8: SEM Image of Retted & Bleached jute, fractured view.

Figure 4.9: SEM image of Retted & bleached jute, terminal view
Figure 4.7 shows a separated fiber indicated by the arrow. The fiber surface is effectively cleaned by the chemical action of $\text{H}_2\text{O}_2$ and the smoothness of the surface signifies the increase in crystallinity of jute fiber. The fiber is damaged by the action of the bleaching chemicals as shown in figure.

Figure 4.8 shows a fractured fiber, and the smooth fracture surface indicates that there may be an increase in crystallinity of the fiber due to effective dissolution and removing of the binding materials from the fiber interior.

Figure 4.9 shows the cellular bundle of jute fiber body that has been effectively extracted of the waxy binding materials. Damage of the fiber surface and spreading of fiber lamellar may result from chemical action of caustic soda and hydrogen peroxide is shown in this figure. Inner void in fiber is also observed.

4.2 FABRICATED COMPOSITE

This section comprises of the pictorial presentation of the fabricated composite beginning from reference polymer, GFRP, cold pressed chopped jute specimens and hand lay-up molded woven jute laminated specimen. Ortho phthalic unsaturated polyester resin matrix was used to fabricate composite specimens, and that will be termed polyester throughout the text. The figure 4.10 to 4.26 represents the individual fabricated specimen.

Figure 4.10: Reference polymer fabricated from ortho phthalic unsaturated polyester resin
Figure 4.11: Glass fiber reinforced composite

Figure 4.12: Chopped retted jute fiber reinforced unsaturated polyester composite, 5-volume percentage of retted jute (dimension ratio 20 approx)

Figure 4.13: Chopped retted jute fiber reinforced unsaturated polyester composite, 10-volume percentage of retted jute (dimension ratio 20 approx)
Figure 4.14: Chopped retted jute fiber reinforced unsaturated polyester composite, 20-volume percentage of retted jute (dimension ratio 20 approx)

Figure 4.15: Chopped retted and woolenized jute fiber reinforced unsaturated polyester composite, 5-volume percentage of retted and woolenized jute (dimension ratio 22 approx)

Figure 4.16: Chopped retted and woolenized jute fiber reinforced unsaturated polyester composite, 10-volume percentage of retted and woolenized jute (dimension ratio 24 approx)
Figure 4.17: Chopped retted and woolenized jute fiber reinforced unsaturated polyester composite, 20-volume percentage of retted and woolenized jute (dimension ratio 20 approx)

Figure 4.18: Chopped retted and bleached jute fiber reinforced unsaturated polyester composite, 6-volume percentage of retted and woolenized jute (dimension ratio 15 approx)

Figure 4.19: Chopped retted and bleached jute fiber reinforced unsaturated polyester composite, 13-volume percentage of retted and woolenized jute (dimension ratio 20 approx)
Figure 4.20: Chopped retted and bleached jute fiber reinforced unsaturated polyester composite, 15-volume percentage of retted and woolenized jute (dimension ratio 20 approx)

Figure 4.21: Chopped retted and bleached jute fiber reinforced unsaturated polyester composite, 23-volume percentage of retted and woolenized jute (dimension ratio 13 approx)

Figure 4.22: Fine jute fabric natural woven fabric (retted) laminate composite (dimension ratio 25 approx)
Figure 4.23: Food grade henequen twill bag woven fabric (retted) laminate composite (dimension ratio 25 approx)

Figure 4.24: Hessian fabric (retted) laminate composite (dimension ratio 19 approx)

Figure 4.25: Fine jute fabric full bleached stiff woven fabric (retted and bleached) laminate composite (dimension ratio 25 approx)
4.3 TENSILE PROPERTIES OF CHOPPED JUTE 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.

It is essential to mention that woven fabric costs much more higher than the natural plant fiber when it is not at the chopped state, but fiber chopping is costly, more over considering the facts of processibility and mechanical properties and the part requirement laminates are now occupying a great area of interest in the field of natural fiber reinforced polymer composite. Since the focus of the project was to develop natural fiber reinforced composite in the prospect of Bangladesh so both chopped fiber and laminated composites were tested to compare better option. This section comprises of the tensile test results concerning (ASTM D 3039/ D 3039M) the chopped jute fiber reinforced composite and woven jute fabric laminated composite.

4.3.1 Tensile properties of chopped retted jute fiber reinforced composite

The following section comprises of the presentation of the tension test data of chopped retted jute fiber reinforced unsaturated ortho phthalic polyester composite.
<table>
<thead>
<tr>
<th>Name</th>
<th>Symbol</th>
<th>Volume %</th>
<th>Average Tensile strength (MPa)</th>
<th>Average tensile StiIness</th>
<th>%Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference polymer</td>
<td>RefT</td>
<td>0</td>
<td>16.52</td>
<td>3.8</td>
<td>0</td>
</tr>
<tr>
<td>Glass fiber reinforced polymer composite</td>
<td>GFRP</td>
<td>16.67</td>
<td>21.4</td>
<td>12.64</td>
<td>0.35</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 5-volume%</td>
<td>JR5T</td>
<td>6.59</td>
<td>14.24</td>
<td>6.23</td>
<td>0.22</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 10-volume%</td>
<td>JR10T</td>
<td>10.65</td>
<td>13.87</td>
<td>7.13</td>
<td>0.22</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 20-volume%</td>
<td>JR20T</td>
<td>19.98</td>
<td>21.65</td>
<td>7.41</td>
<td>0.43</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 6-volume%</td>
<td>JR36T</td>
<td>7.24</td>
<td>13.01</td>
<td>7.58</td>
<td>0.17</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 13-volume%</td>
<td>JR13T</td>
<td>13.72</td>
<td>14.70</td>
<td>8.19</td>
<td>0.17</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 15-volume%</td>
<td>JR15T</td>
<td>14.96</td>
<td>22.42</td>
<td>8.86</td>
<td>0.17</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 23-volume%</td>
<td>JR23T</td>
<td>23</td>
<td>22.19</td>
<td>7.02</td>
<td>0.43</td>
</tr>
<tr>
<td>Chopped Retted &amp; woolenized Jute fiber 5-volume%</td>
<td>JWS</td>
<td>6.59</td>
<td>6.30</td>
<td>4.55</td>
<td>0.08</td>
</tr>
<tr>
<td>Chopped Retted &amp; woolenized Jute fiber 10-volume%</td>
<td>JW10T</td>
<td>10.65</td>
<td>14.05</td>
<td>3.85</td>
<td>0.22</td>
</tr>
<tr>
<td>Chopped Retted &amp; woolenized Jute fiber 20-volume%</td>
<td>JW20T</td>
<td>19.98</td>
<td>21.67</td>
<td>7.91</td>
<td>0.43</td>
</tr>
</tbody>
</table>

It has been mentioned earlier in the experimental section about the volume percentage of the chopped fibers taken to fabricate composite specimen and the ASTM standards selected for the tension test.
Table 4.1 represents the tensile property data summary of three types of chopped jute fiber reinforced polyester composite. The table shows the symbol of specimen. It is observed from the table that the stiffness of the composite increases upon incorporation of reinforcement fiber.

Figure 4.27 shows the tensile failure characteristics of chopped retted Jute fiber reinforced polyester composites of different volume fraction. The composite specimen failed at lower stress than that of the reference polymer.

![Tensile property representation of chopped retted Jute fiber reinforced polyester composite of varying volume percentage](image)

**Figure 4.27:** Tensile property representation of chopped retted Jute fiber reinforced polyester composite of varying volume percentage

From table 4.1 and the figure 4.27 it is clear that the stiffness of the composite specimen has been increased and the ductility is decreased. This may be due to ineffective load transfer between the fiber and the matrix within these volume fractions of fiber. The increase in %elongation for the composite specimens is higher due to the fiber pull out from the matrix. A good trend of increasing strength is observed due to addition of increasing volume percentage of jute fiber while comparing the strength of the composite specimens alone.

4.3.2: Tensile properties of chopped retted and bleached jute fiber reinforced composite

Figure 4.28 shows the tensile properties of chopped retted and bleached jute fiber reinforced composite.
Figure 4.28: Tensile property representation of chopped retted and bleached Jute fiber reinforced polyester composite of varying volume percentage.

reinforced polyester composite. For low volume fraction the %elongation remains almost similar. For any case stiffness increases from reference polymer, which is confirmed from the table 4.1. The 6 and 13-volume percentage fiber containing samples show similar stiffness. This may be due to random fiber dispersion, and load transfer phenomenon in between the fiber and the matrix.

The 15-volume percentage fiber-containing specimen shows similar stiffness as that of the 6 and 13-volume percentage fiber containing samples, but with increased strength. Possibility of higher extent of flaw in side the composite system is much more predominant at higher volume fraction of fiber. Since esterification and cross-linking reactions are probable. So, when the reaction proceeds after gel time a large number of reaction byproduct may cause flaw within the matrix and fiber matrix interface. But at higher percentage of fiber, uniform dispersion of the fiber throughout the matrix may be assumed and at this condition effective load transfer from matrix to fiber is possible and thus higher volume percentage specimen may withstand higher load.

It may be inferred that the fiber has random dispersion throughout the matrix and that is the reason why some part of the composite body is rich in fiber and some portions are short of fiber. So if there is good fiber matrix compatibility and efficient
load transfer from matrix to fiber then fiber richer portion will tolerate higher load than that of the fiber deficient portion provided the fibers are well embedded within the matrix.

4.3.3 Tensile Properties of Chopped Retted and Woolenized Jute Fiber Reinforced Composite

Figure 4.29 shows the tensile property of chopped retted and woolenized jute fiber reinforced polyester composite. It is observed that the stiffness of the composite increases with increasing fiber volume fraction. Britteness also increases with increasing volume fraction of fiber. Ductility of the composite specimens decreased well below the reference value of polymer due to incorporation of woolenized jute fiber [Ray, 2001].

This is attributed from the fact that, when woolenization treatment is done on jute fiber, crimp develops with increase in modulus of elasticity of jute fiber. Along with this fact the jute fiber have the helical structure microscopically, which enables the fiber to extend to some extent when the fiber is stressed [Rowell, 1998].
More over woolenization treatment enables the binders to leach out of the fiber system, facilitating lesser elongation to brake. This may be the reason of increasing stiffness and decreasing elongation of the composites.

4.4 TENSILE PROPERTIES OF WOVEN JUTE FABRIC REINFORCED COMPOSITE

Table 4.2: Tensile property data summary of woven jute reinforced composite

<table>
<thead>
<tr>
<th>Type</th>
<th>Symbol</th>
<th>Vol%</th>
<th>Ply Depth</th>
<th>Number of Ply</th>
<th>Test direction</th>
<th>Avg Stress MPa</th>
<th>Avg Stiffness MPa</th>
<th>% Elongation mm/mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>Ref T</td>
<td>0</td>
<td>-</td>
<td>0</td>
<td>N/A</td>
<td>18.62</td>
<td>3.87</td>
<td>0.43</td>
</tr>
<tr>
<td>Chopped strand mat 300gsm</td>
<td>GFRP T</td>
<td>16.78</td>
<td>1mm</td>
<td>22</td>
<td>N/A</td>
<td>91.40</td>
<td>12.64</td>
<td>0.35</td>
</tr>
<tr>
<td>Jute fabric natural 15/15 in² 260gsm</td>
<td>JRa1 T</td>
<td>18.98</td>
<td>1mm</td>
<td>11</td>
<td>0-90°</td>
<td>31.74</td>
<td>7.43</td>
<td>0.1</td>
</tr>
<tr>
<td>Food grade jute fabric 100gsm</td>
<td>JRa2 T</td>
<td>25.26</td>
<td>2mm</td>
<td>8</td>
<td>0-90° Twill Direction</td>
<td>28.82</td>
<td>4.71</td>
<td>0.2</td>
</tr>
<tr>
<td>Hessian Fabric 190gsm</td>
<td>JRa3 T</td>
<td>15.7</td>
<td>1mm</td>
<td>13</td>
<td>0-90°</td>
<td>30.59</td>
<td>7.53</td>
<td>0.1</td>
</tr>
<tr>
<td>Bleached jute fabric full bleached stiff 15/15 in² 300gsm</td>
<td>JRa1 T</td>
<td>21.97</td>
<td>1mm</td>
<td>13</td>
<td>0-90°</td>
<td>25.55</td>
<td>7.61</td>
<td>0.1</td>
</tr>
<tr>
<td>Carpet backing cloth half bleached 15/15 in² 335gsm</td>
<td>JRa2 T</td>
<td>19.52</td>
<td>1mm</td>
<td>11</td>
<td>0-90°</td>
<td>21.94</td>
<td>7.69</td>
<td>0.1</td>
</tr>
</tbody>
</table>
The types of woven jute fabric laminated composite fall in the category of the 0 - 90° class. The strength property of the laminated composite can be well standardized by the laminate theory, but the results obtained will be expressed and analyzed on the
context of the strength compared to that of polyester polymer and the glass fiber reinforced composite.

Table 4.2 and figure 4.30 infer an increase in strength and stiffness values of the woven jute fabric reinforced laminated composite specimens than the reference polymer. The decrease in displacement values of composites in the figure 4.30 indicates the decrease in ductility, which is the effect of incorporation of the woven jute fabric. The initial linear portion of the jute laminate curves shows the elastic behavior of the composite. The deviation from linearity is an indication of the beginning of initial matrix cracking, and the first major change in slope in the curve is the sign of a major crack in the matrix or the beginning of fiber failure [Gowda, 1999].

The curve for GFRP at figure 4.30 shows the highest tensile property. As it is known that the glass fiber contains binders also it contains a coupling agent so that the compatibility between the fiber and the polyester matrix is improved, thus increasing the composite strength by effective load transfer from matrix to fiber.

The strength of Woven jute fabric laminated composite remains well below the strength of glass fiber reinforced composite.

It is interesting to see that the bleaching treatment does not conclude increase in tensile strength compared to that of the retted jute fabric reinforced composites. It is seen from the figure 4.30 (a) and (b) that there is no adequate improvement in the tensile strength of the fine jute fabric full bleached stiff and carpet backing cloth half bleached considering the tensile strength property of the retted woven jute fabric. This may be due to bleaching treatment that effectively removes the binding materials of jute fiber as it was indicated in SEM image of bleached jute in figure 4.9, page 98 of this chapter. As illustrated in experimental chapter, it was seen that the hessian fabric is woven loosely and the fabricated composite contains the lowest volume percentage of the hessian fabric among the laminated composites.

4.5 ANALYSIS OF TENSION TEST DATA OF THE CHOPPED JUTE FIBER REINFORCED AND WOVEN JUTE MAT REINFORCED COMPOSITE

The following section comprises of the stress variation of the tensile failure of composite specimen.
The process of composite failure is as follows; initially the matrix fails, then the fibers begin to attain load that is widely distributed in the weaker fibers. As the local stresses build up, however, the fiber failure begins to occur preferentially among certain layers until a critical configuration is formed. With any further increase in the applied stress, this damage configuration propagates throughout the remaining fibers around the most severely damaged layers, then the fibers are pulled out and finally the composite fails. Figure 4.31 shows a comparison between the tensile strength property of chopped jute and woven jute fabric reinforced laminated composites. Jute fiber exhibits stiffer characteristics as compared to jute fabric due to more initial stretching of the fabric than the nature of the fiber. Chopped fiber break at different times as each fiber can stretch independently and break individually when reaching their breaking stress. The failure mode is by progressive breaking of the fibers. The variation in the values of strengths may be due to: (i) the assumption that the cross-sectional area of each yarn is macroscopically circular, (ii) the difference in the strength of chopped fiber reinforced composite arising out of process defects. The tensile strength of a composite material is mainly dependent on the strength and modulus of fibers, the strength and chemical stability of the matrix and the effec-
The tensile strength variation of chopped jute and woven jute fiber reinforced polyester composite shows higher tensile strength at higher volume percentage of reinforcement than the reference polymer. It is observed that the chopped jute reinforced composite showed higher tensile strength at higher volume percentage of reinforcement than the woven fabric reinforced composite. The bleached woven jute reinforced composite showed lower tensile strength than that of the retted woven jute reinforced composite. This implies bleaching treatment does not significantly affect the ultimate tensile strength of woven jute fabric reinforced composite.

Figure 4.31: Tensile strength variation of chopped jute fiber reinforced polyester composite.
Chopped discontinuous fiber reinforced polymer composite property is the cumulative result of fiber length, fiber orientation and strength, fiber volume fraction, fiber matrix compatibility, and interface – interphase interaction. In this respect the chopped retted, retted and bleached jute closely resemble the tensile property of the reference polymer but not the chopped retted and woolenized jute fiber reinforced composite.

4.5.1 Tensile failure mode of chopped jute and woven jute composite

SEM observations of the fracture surface of tensile specimens indicate that there is considerable difference in the fiber-matrix interaction between the treated and non-treated i.e. retted jute fiber reinforced composites. Figure 4.32 (I) (a) 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, but in some fracture area tensile separation between the matrix and the fibers are observed when the fibers are parallel to the tensile force. Bleaching and woolenization appears to produce a significant improvement of the wettability of jute surface by the polymer. The improved bonding is clearly seen in Figure 4.32 (II) and (III) where the fiber has pulled out from the matrix but a fair amount of polymer residue remains on the fiber. Fiber damage normally occurs during blending of fiber and the polymer by the cold press fabrication method. The failure of tensile specimen of the chopped jute fiber reinforced composite showed directional behavior according to the fiber orientation. This implies that the interfacial strength in 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 loading modes imply that only the fiber along the loading direction will have an impact on the tensile behavior of laminated composites and is assumed maximum at that direction.
Figure 4.32: Tensile fracture of chopped jute fiber reinforced composite, arrow indicates the polymeric residue on fiber surface.
Figure 4.33 to figure 4.37 shows the fracture surface of woven jute fabric laminated composite due to tensile action. The smooth surface appearance of the failed specimen indicates the brittle nature of the matrix polymer. Fiber pullout is more or less predominantly observed in all the test specimens. The matrix is seen to adhere more efficiently with fibrous bundles of 'retted and bleached' jute mats rather than the fibrous bundles of retted jute mats.

The crack generated along side the fibrous bundles that lie parallel to the load, and is very much apparent on figure 4.35 and 4.36. This implies the interfacial strength of the fiber and matrix is less than the interfacial strength of the fiber or polymer. Shearing effect is seen in figure 4.36 and 4.37 (b) but it is not the predominant failure mode during tensile action on the laminates.

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 (i) the statistical fiber strength and (ii) 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. This 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. [Gao, 1992].

In a brittle polymer matrix like polyester, the matrix carries most of the load, which is usually compressive and fibers are added only to increase toughness. That is, to increase the time to catastrophic failure by holding the matrix together after cracking. Jute fiber is more ductile than the matrix and the matrix fails first. As the cracks propagate and reach the interface, a weak interfacial bond is desired. This enhances debonding, and the cracks are not stopped, but deflected along the length of the fibers. This effectively delays the time it takes the cracks to propagate through the entire matrix, and thus increases toughness. As in many of the curves it was seen that composite fails as one entity indicating that strong bonding is present rather than weak interface [Gao, 1992].
Figure 4.33: Tensile fracture of fine jute fabric natural woven fabric reinforced polyester composite (arrow indicates polymeric residue on fiber surface)

Figure 4.34: Tensile fracture of Food grade jute fabric twisted woven jute fabric reinforced polyester composite (arrow indicates polymeric residue on fiber surface)

Figure 4.35: Tensile fracture of sisal fabric reinforced unsaturated polyester composite (arrow indicates polymeric residue on fiber surface)
Table 4.4 shows the critical volume fraction data of the three types of chopped jute fiber reinforced composite specimen. It has been seen from the table that the critical volume fraction is well below the 5-percentage range of the composite specimen. The strength was seen to increase after incorporation of 10-volume percentage of chopped jute fiber. It has been seen from the table 4.4 that the critical volume fraction has been increased upon severity of the surface modification treatment of jute fiber.
Table 4.4: Determination of critical volume fraction of chopped jute reinforced composite.

<table>
<thead>
<tr>
<th>Definition</th>
<th>Property</th>
<th>Retted jute</th>
<th>Retted and bleached jute</th>
<th>Retted and woolenized jute</th>
<th>Ref</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber UTS, MPa [Rowell, 1998]</td>
<td>$\sigma_u$</td>
<td>490</td>
<td>490</td>
<td>393</td>
<td></td>
</tr>
<tr>
<td>Strain at Fiber UTS minimum [Rowell, 1998]</td>
<td>$\varepsilon_u$</td>
<td>1.545</td>
<td>1.517</td>
<td>1.5</td>
<td></td>
</tr>
<tr>
<td>Matrix stress at fiber failure strain, MPa</td>
<td>$\sigma_m$</td>
<td>1.40</td>
<td>1.32</td>
<td>1.39</td>
<td></td>
</tr>
<tr>
<td>Maximum TS of composite MPa</td>
<td>$\sigma_{LM}$</td>
<td>21.67</td>
<td>22.44</td>
<td>21.67</td>
<td></td>
</tr>
<tr>
<td>Critical Fiber volume (Vol %)</td>
<td>$V_f$</td>
<td>4%</td>
<td>4.2%</td>
<td>4.1%</td>
<td></td>
</tr>
<tr>
<td>Critical Fiber length (cm) [2017]</td>
<td>$L_f$</td>
<td>0.23</td>
<td>0.21</td>
<td>0.20</td>
<td></td>
</tr>
</tbody>
</table>

4.6 FLEXURE PROPERTIES OF CHOPPED JUTE COMPOSITE

When a 3-point flexure test is done on a brittle material like cured polyester it is often called modulus of rupture (MOR). This test provides flex strength data only, not stiffness (modulus). The following section comprises of the flexure test data analysis of the chopped jute.

Table 4.5: Flexure test data summary for chopped jute fiber reinforced polymer composite

<table>
<thead>
<tr>
<th>Name</th>
<th>Vol%</th>
<th>Average Flexure Strength MPa</th>
<th>Average Modulus of Rupture GPa</th>
<th>Avg Flexural strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference polymer</td>
<td>0</td>
<td>47.34</td>
<td>4.8</td>
<td>0.0095</td>
</tr>
<tr>
<td>Glass fiber reinforced polymer composite</td>
<td>7</td>
<td>179.04</td>
<td>7.2</td>
<td>0.02575</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 5-volume%</td>
<td>6.39</td>
<td>24.73</td>
<td>3.6</td>
<td>0.00682</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 10-volume%</td>
<td>10.65</td>
<td>28.12</td>
<td>3.63</td>
<td>0.00781</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 20-volume%</td>
<td>19.98</td>
<td>38.86</td>
<td>3.7</td>
<td>0.0125</td>
</tr>
<tr>
<td>Chopped Retted and Woolenized Jute fiber 5-volume%</td>
<td>5.46</td>
<td>31.52</td>
<td>3.32</td>
<td>0.0099</td>
</tr>
<tr>
<td>Chopped Retted and Woolenized Jute fiber 10-volume%</td>
<td>9.46</td>
<td>35.93</td>
<td>3.37</td>
<td>0.0092</td>
</tr>
<tr>
<td>Chopped Retted and Woolenized Jute fiber 20-volume%</td>
<td>26.49</td>
<td>45.82</td>
<td>3.41</td>
<td>0.013</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 6-volume%</td>
<td>7.24</td>
<td>29.56</td>
<td>3.16</td>
<td>0.0101</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 13-volume%</td>
<td>13.72</td>
<td>36.54</td>
<td>3.28</td>
<td>0.012</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 15-volume%</td>
<td>14.96</td>
<td>57.37</td>
<td>4.9</td>
<td>0.011</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 23-volume%</td>
<td>23.73</td>
<td>47.22</td>
<td>4.5</td>
<td>0.013</td>
</tr>
</tbody>
</table>

*Chapter 2 Literature review*
Table 4.5 shows the flexure test data summary of the chopped jute fiber reinforced polymer composite. Although the flexural strength and modulus of the composites remain below the reference value of the polymer matrix but from table 4.5 it is clearly indicated that the flexural stress, modulus of rupture (MOR) and the flexural strain gradually increased along with the increase in the fiber volume fraction.

4.6.1 Flexure test data analysis of chopped retted jute fiber reinforced polyester composite

![Figure 4.38: Flexure stress strain characteristics of chopped retted jute fiber reinforced polyester composite of varying volume percentage.](image)

Figure 4.38 reveals the flexure stress strain characteristics of chopped retted jute fiber reinforced polyester composite. Flexural stress has been increased with the increase in the volume percentage of jute fiber but fail to reach the reference value of polyester matrix. Brittleness has been decreased with little increase in toughness of the composites to that of the matrix. Ductility is also increased with increase in the volume fraction of jute fiber, since jute fiber itself shows 1-1.5% elongation when stressed under tensile load [Fichhorn, 2001; Rowell, 1998].
Although the composite contains different volume percentage of reinforcement but stiffness remained unchanged. The increase in fiber dosing resulted in increased flexural toughness.

The crack profile of the flexure specimen of the 5, 10 and 20-volume percentage reinforced composite is shown in figure 4.39, 4.40 and 4.41 respectively. The arrow "↓" indicates the applied load direction on the specimen.

Figure 4.39: Flexure crack profile of 5-volume percentage chopped retted jute fiber reinforced composite (magnification 5 - 6 approx.)

Figure 4.40: Flexure crack profile of 10-volume percentage chopped retted jute fiber reinforced composite (magnification 5 - 6 approx.)

Instead of crack propagated unidirectionally, crack bifurcated at one or two points as shown in figures. The crack profile of the flexure specimen of the 20-volume percentage is shown in figure 4.41, which seems to propagate through fiber bridging process.
Figure 4.41: Flexure crack profile of 20-volume percentage chopped retted jute fiber reinforced composite (magnification 5 – 6 approx)

It is observed that the larger the volume percentage of fiber in the matrix the greater the probability of directional crack propagation style with reduction of catastrophic brittle failure.

4.6.2 Flexure test of chopped retted and woolenized jute reinforced polymer composite

Figure 4.42: Flexure Stress Strain Characteristics of chopped retted and woolenized jute fiber reinforced polyester composite of varying volume percentage.
Figure 4.42 denotes the flexure stress-strain characteristics of chopped retted and woolenized jute fiber reinforced polyester composite of various volume %.Britleness has been decreased with little increase in toughness of the composites to that of the matrix, with the increase in ductility. The ductility is increased significantly for the 20-volume percentage jute-containing composite.

If a flexure load is applied on a composite body in which, the reinforcement lies perpendicular to the load path, will adapt more resistance than any other fiber orientation, provided the fiber matrix interface acts as a load transfer source. The brittleness was increased due to fiber incorporation. The flexural strain of these three specimens remains almost the same and indicates similar flexural stiffness with no adequate increase in strength than that of the cured polyester, regardless the variation of the fiber volume percentage. The fibers became rigid and brittle afterwards owing to the development of crystallinity due to woolenization of jute fiber but causes high strength and low extensibility. On application of stress, these fibers suffered breakage due to increased brittleness [Ray, 2002; Ray, 2001].

Figure 4.43, 4.44 and 4.45 represents the crack profile of 5, 10 and 20-volume percentage chopped retted and woolenized jute fiber reinforced polyester composite respectively. The cracks bifurcated at one or two points as indicated in the figures. This is due to the fiber orientation and fiber bridging during the test.

Figure 4.43: Flexure crack profile of 5-volume percentage chopped retted and woolenized jute fiber reinforced polymer composite (magnification 5–6 approx)
Figure 4.44: Flexure crack profile of 10-volume percentage chopped retted and woolenized jute fiber reinforced polymer composite (magnification 5–6 approx).

Figure 4.45: Flexure crack profile of 20-volume percentage chopped retted and woolenized jute fiber reinforced polymer composite (magnification 5–6 approx).

Figure 4.45 show that the crack has been arrested for the 20-volume percentage chopped retted and woolenized jute fiber reinforced polymer composite specimen.

4.6.3 Flexure test data analysis of chopped retted and bleached jute fiber reinforced polymer composite

Figure 4.46 represents the flexure stress strain characteristics of chopped retted and bleached jute fiber reinforced polyester composite. Flexural stress has been increased with the increase in the volume percentage of jute fiber. It is quite apparent that the flexural strain resembles the similar range for the entire specimens. Brittleness has been decreased with little increase in toughness of the composites to that of the matrix with the increase in ductility. It is seen from the figure 4.46 that the flexural strength of 15-volume percent containing specimen is higher than that of the 23-
volume percentage reinforced composite, where as the 6-volume percentage specimen resembles higher strength than that of the 13-volume percentage specimen. It is assumed that at higher volume percentage of fiber the larger force of compaction ensures better fiber matrix interaction to give stiffer material. The probable reason may be the critical volume of fiber that helps to increase the strength of the composites.

![Flexure stress-strain characteristics of chopped retted and bleached jute fiber reinforced polyester composite of varying volume percentage.](image)

Figure 4.46: Flexure Stress Strain Characteristics of 6-volume percentage Chopped Retted and bleached jute fiber reinforced polyester composite

More over it was seen from the SEM image of the bleached jute fiber that effective removal of the fiber binding materials, which is supposed to increase the crystallinity index along with increase in modulus of elasticity, might be incoherent with the matrix at higher volume percentage of jute fiber and acts as source of flaw.

The study of the fracture profile of the 6, 13, 15 and 23-volume percentage of chopped retted and bleached jute fiber reinforced polymer composite is given in the figure 4.47, 4.48, 4.49 and 4.50 respectively. Fiber pull out is apparent in the figures presented below. It is quite evident from the figures that the crack propagation profile is different for individual specimens. Where the distribution of fiber is
locally increased, crack tip has shifted at that point and followed some new path. This fact is evident in the entire figures.

Figure 4.47: Flexure crack profile of 6-volume percentage chopped retted and bleached jute fiber reinforced polymer composite (magnification 5 - 6 approx)

Figure 4.48: Flexure crack profile of 13-volume percentage chopped retted and bleached jute fiber reinforced polymer composite (magnification 5 - 6 approx)

Figure 4.49: Flexure crack profile of 15-volume percentage chopped retted and bleached jute fiber reinforced polymer composite (magnification 5 - 6 approx)
It is seen from Figure 4.48 that the crack tip has followed the direction that is parallel to the fiber orientation. The increase in ductility is attributed to be the fiber pullout from the matrix. As the fiber volume fraction increases in the matrix the distribution of fiber becomes more uniform but proper wetting of reinforcement is hindered by backpressure generated by entanglement of fibrous entity. Obviously no reinforcing characteristics will be achieved if the fiber orientation is parallel to the direction of flexure load, since no resistance force of fiber will be gained at that orientation. Orientation perpendicular to the path of flexure load fails to impose maximum resistance against the transverse loading of composite.

Table 4.6 shows the data summary of flexure test of the woven jute fabric laminated composite.

<table>
<thead>
<tr>
<th>Name</th>
<th>Vol %</th>
<th>Average Flexure Strength MPa</th>
<th>Average Modulus of Rupture GPa</th>
<th>Avg Flexural strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference polymer</td>
<td>0</td>
<td>47.31</td>
<td>4.8</td>
<td>0.0025</td>
</tr>
<tr>
<td>Glass fiber reinforced polymer composite</td>
<td>15.78</td>
<td>179.04</td>
<td>7.2</td>
<td>0.02575</td>
</tr>
<tr>
<td>Fine jute fabric natural</td>
<td>18.98</td>
<td>57.34</td>
<td>3.4</td>
<td>0.025</td>
</tr>
<tr>
<td>Food grade benfali will bag</td>
<td>23.26</td>
<td>53.03</td>
<td>2.9</td>
<td>0.0188</td>
</tr>
<tr>
<td>Persian fabric</td>
<td>15.7</td>
<td>59.09</td>
<td>3.2</td>
<td>0.021</td>
</tr>
<tr>
<td>Fine jute fabric full bleached slitt</td>
<td>21.97</td>
<td>50.24</td>
<td>3.5</td>
<td>0.0198</td>
</tr>
<tr>
<td>Carpet backing cloth half bleached</td>
<td>19.52</td>
<td>69.04</td>
<td>3.7</td>
<td>0.018</td>
</tr>
</tbody>
</table>

Figure 4.48: Flexure crack profile of 23-volume percentage chopped jotted and bleached jute fiber reinforced polymer composite (magnification 5 - 6 approx)
4.7 FLEXURE PROPERTIES OF WOVEN JUTE FABRIC REINFORCED POLYMER COMPOSITE

4.7.1 Flexure Test of Woven Jute Fabric Reinforced Composites

Figure 4.51: Flexure stress strain diagram of woven jute fabric reinforced polymer composite
Figure 4.51 shows the flexure stress-strain diagram of fine jute fabric natural woven fabric reinforced polymer composite. It is seen that the composite hardly reach the flexure property of cured polyester. The flexural strain of the fine jute fabric natural is the maximum and boltilla twill bag and the hessian fabric varied slightly in strain with similar flexural strength behavior. Table 4.6 infers that the fine jute fabric natural reinforced composite posses the highest stiffness among the retted variety of jute fabrics.

Carpet backing cloth half bleached jute fabric reinforced composite shows the highest flexural toughness among the woven jute fabric reinforced composite. It is difficult to identify from the above curves that when fiber debonding and load transfer from matrix to fiber has taken place.
It is seen from the figures 4.52, 4.53, 4.54, 4.55 and 4.56 that the distribution of polymer is uneven, although the impregnation factor of the woven fabrics is
constant. This implies that the ortho phtholic unsaturated polyester has been precured before it entered the twisted fibrous yarn, although standard hand lay-up procedure was implemented for the composite fabrication. Moreover, it is easily observed that the machined portion of the flexure specimen is somewhat deficient in polymer from point to point, hence indicates the fiber twist creates a resistance to flow of liquid polymer inside the twisted fiber bundle before the liquid polymer gets cured.

The failure pattern looks like the unidirectional fiber reinforced polymer composite. All the fractures are characterized by matrix crack, followed by the shifting if the crack encounters the fibrous bundle that lie parallel to the crack direction followed by fibrous bundle pullout from the matrix. The strength of the composite specimen is due to the fibrous bundle pullout and crack bridging. When the matrix fails the stress concentration builds up on the embedded yarn those are perpendicular to the loading direction, and the matrix interface. Thus the stress concentration forces the yarn to pullout from the matrix and then shearing occurs. This shearing force is resisted by the interfacial bonding between the fiber and the matrix and so the composite withstand higher load.

Sequentially the matrix failed first as seen in the above figures then the twisted fiber bundle that lies perpendicular to the crack tip imposes resistance against the propagation of crack. The twisted fiber bundle that lies parallel to those of the propagating crack, under flexure load, acted as the flaw. Thus the effective fiber volume of the composite that acts as the load-bearing portion of the composite is decreased. These parallel-twisted fibrous bundles also act as the shearing site to that of the advancing crack. The crack has been seen to propagate and glided over these twisted fiber bundle. The main sliding of fracture is observed near the fiber bunches that lie parallel to the load path. Generally, failure has occurred within the matrix material, especially at the node point of the embedded fabric. The tensile force acting on the outer portion of the composite body induces delamination between matrix and the fiber.
4.8 ANALYSIS OF FLEXURE TEST DATA FOR CHOPPED JUTE AND WOVEN JUTE FABRIC REINFORCED POLYMER COMPOSITE

Table 4.7 shows the comparison of average flexure strength variation with respective fiber types and fiber volume fraction, upon which the figure 4.57 is built. Table 4.7 indicates that the development of flexure strength property of the chopped jute fiber reinforced composite to that of the reference polymer.

At this developmental stage, it can be assumed that adequate strength property will develop compared to the polymer if composites with higher volume fraction of jute fiber can be fabricated.

<table>
<thead>
<tr>
<th>Name</th>
<th>Symbol</th>
<th>Volume %</th>
<th>Average Flexure Strength, MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference polymer</td>
<td>Ref</td>
<td>0</td>
<td>47.31</td>
</tr>
<tr>
<td>Glass fiber reinforced polymer composite</td>
<td>GRP</td>
<td>16.787</td>
<td>179.64</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 5-volume%</td>
<td>JR5F</td>
<td>6.59</td>
<td>24.72</td>
</tr>
<tr>
<td>Chopped Retted &amp; woollenized Jute Fiber 5-volume%</td>
<td>JWR5F</td>
<td>5.46</td>
<td>28.11</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute Fiber 6-volume%</td>
<td>JRDF</td>
<td>7.24</td>
<td>29.56</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 10-volume%</td>
<td>JR10F</td>
<td>10.65</td>
<td>51.51</td>
</tr>
<tr>
<td>Chopped Retted &amp; woollenized Jute Fiber 10-volume%</td>
<td>JWR10F</td>
<td>9.46</td>
<td>53.92</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute Fiber 13-volume%</td>
<td>JRB13F</td>
<td>13.72</td>
<td>45.82</td>
</tr>
<tr>
<td>Chopped Retted Jute fiber 20-volume%</td>
<td>JR20F</td>
<td>19.98</td>
<td>41.14</td>
</tr>
<tr>
<td>Chopped Retted &amp; woollenized Jute fiber 20-volume%</td>
<td>JWR20F</td>
<td>26.40</td>
<td>36.53</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 15-volume%</td>
<td>JRB15F</td>
<td>14.96</td>
<td>57.36</td>
</tr>
<tr>
<td>Chopped Retted &amp; Bleached Jute fiber 25-volume%</td>
<td>JRB25F</td>
<td>23.73</td>
<td>47.22</td>
</tr>
<tr>
<td>Fine jute fabric natural</td>
<td>JRa1F</td>
<td>18.98</td>
<td>57.34</td>
</tr>
<tr>
<td>Food grade bennolia twill bag</td>
<td>JRa2F</td>
<td>23.26</td>
<td>53.03</td>
</tr>
<tr>
<td>Hessian fabric</td>
<td>JRa3F</td>
<td>15.7</td>
<td>59.09</td>
</tr>
<tr>
<td>Fine jute fabric full bleached stuff</td>
<td>JRBa1F</td>
<td>21.97</td>
<td>50.24</td>
</tr>
<tr>
<td>Carpet backing cloth half bleached</td>
<td>JRBa2F</td>
<td>9.32</td>
<td>69.04</td>
</tr>
</tbody>
</table>

Figure 4.57 shows the stress variation among various types of treated chopped jute fiber and woven jute fabric reinforced polymer composite. It is seen that the composite with chopped jute fiber failed at lower flexural stress than the reference.

A composite has three entities that are susceptible to failure, the reinforcement, the matrix and the interface. The failure of one can initiate the failure of the other, and the actual process that takes place in any particular case is determined by the stress...
required to activate each individual mechanism. The mechanism activated by the lowest stress will normally govern the composite failure [Zhu, 1998].

Ortho phthalic unsaturated polyester shrinks upon curing. When chopped jute polyester composite was made it was macroscopically seen that the shrinkage has been reduced. The shrinking tendency of polyester imposes a compressive stress from all side of individual chopped fiber. This residual compressive stress is prominent just at the out side of the fiber-matrix interface. To minimize the effect of this compressive stress a tensile stress zone is found. This tensile stress ensures microscopic crack development throughout the matrix. So when the composite materials were tested with flexural load the micro-flaw at the surface of the composite body initiates the crack. Since the bottom surface of a flexure specimen always remains in tensile stress, so this tensile stress adds up with the tensile stress surrounding the chopped fiber in side the composite body and so composite fails at lower stress than that of the cured polyester, which is also found in other resin matrix system.

**Figure 4.57: Flexure test data analysis of chopped jute fiber reinforced polymer composite.**
A crack propagating transversely through a composite is more likely to deviate from its path and grow along the fiber matrix interface, if the interface is weak, resulting in debonding and fiber pullout. Both mechanisms contribute to an increase in the composite's fracture toughness [Zafeiropoulos, 2002].

The interphase surrounds the fiber and its property differs from the bulk of the matrix. It may be created by the local variation of the matrix microstructure close to the fiber surface. For example, there may be a variation in cross-link density, which can be influenced by the presence of the fibers as well as fiber surface chemistry. The interphase may also contain microvoids and unreacted solvents or curing agents that tend to migrate toward fiber surface.

Stress transfer between fibers and matrix is possible owing to a mechanical interlocking that arises because of polymerization shrinkage of the matrix as well as thermal contraction of matrix from curing temperature. At elevated temperatures or at high-applied loads, the difference in expansion of fibers and matrix may relieve this mechanical interlocking. Under extreme circumstances, a microcrack may be formed at the interface resulting in reduced mechanical properties for the composite. The interfacial failure is characterized by clean fiber surface, thin cracks (debonding) between fiber and matrix, and clear impressions in the matrix from which the fibers have pulled out.

From figure 4.57 it is clear that the retted jute fiber reinforced composite comprises the lowest strength property but it is interesting that the retted jute fiber reinforced polymer has the highest slope among the three-treated fiber types and shows increase in reinforcing behavior with little increase in reinforcement volume fraction.

The woolenized fiber is reported to be very good reinforcing fiber in case of added coupling agent and if the matrix is epoxy resin [Ray, 2002]. But during the course of the project, it was found that the woolenization seems to increase the strength property to a lesser extent than that of the retted jute fiber. The retted and bleached jute composite showed decrease in strength property when the reinforcement percentage is increased from 6 to 13-volume percentage. At this point, the increase in reinforcement from 13 to 15-volume percentage showed sharp increase in strength of the composite, afterwards integrating higher i.e. 23-volume percentage of reinforcement showed decrease in strength.
4.9 FLEXURE FRACTURE SURFACE ANALYSIS OF CHOPPED JUTE AND WOVEN JUTE FABRIC REINFORCED COMPOSITE

Figure 4.58, 4.59 and 4.60 shows the flexure fracture surface of the retted jute, rotted and woolenized jute, and retted and bleached jute composites respectively. Fiber pullout is the predominant failure mode and shows chopped fiber orientation dependence along with polymeric residues. The arrow in the right indicates the direction of load application.
The polymeric residue on the retted and woolenized is maximum and minimum on the retted and bleached jute. The failed specimen of retted jute composite posses few polymeric residue on the reinforcing fiber. This is because of the fact that the retted jute comprises of fiber binding materials along with waxy substance and bleaching process does not effectively remove these binders thoroughly from the fiber surface, but woolenization treatment effectively removed these binders. This treatment opens up the (–OH) groups to facilitate chemical reaction between the fiber and the matrix. Figure 4.61 to 4.65 shows the SEM micrograph of the woven jute fabric reinforced polymer composite that was failed under three-point flexure load. The smooth fracture surface is the failure characteristics of the cured brittle polyester matrix. It is clearly seen that the fiber bundles has been pulled out from the matrix but the pulling occurred at the center of the fiber bundles rather pulling out from the matrix. This reveals that there may be some mechanical interlocking or chemical bond has been formed that resist the outer layer of the twisted fiber bundle to pulled out of the matrix completely. Obviously the shrinkage of the matrix will always imposes a compressive load that insists the mechanical interlocking in between the fiber and matrix. Fiber pull out is clearly seen in these figures.

Closure look towards the fracture surface shows that the fracture slide through twisted fiber bundle that lie parallel to the load direction. For the figure 4.61 just beneath the parallel twist, on the left side of the fractured surface a deep cavity is observed, which seem to separate the laminate at that position. This cavity implies delamination type failure of the specimen under flexure load at that position.
It is known that during the three-point bend test the mid portion bottom side encounters tensile stress, while the mid top portion encounters compressive stress. The cumulative action generates a shear force that acts on the embedded fibrous body at the fiber-matrix interface and interphase. As the reinforcement is a discontinuity so the shear force tries to delaminate the test body under applied flexural load. The warped fibrous body, parallel to the load direction may act as the source of flaw, where the crack slides over. The tensile action induces the pull out of fiber bundle that supports the composite body prior to initial matrix failure [Zafeiropoulos, 2002]. Also it is previously explained that the fibrous body that lie parallel to the load direction is a potential source of flaw, which is indicated at the bottom portion of the SEM images. "f" indicates loading direction.

Figure 4.61: SEM image of flexure failed jute fabric natural woven jute reinforced polymer composite.

Figure 4.62: Flexure crack SEM image of food grade benolla twill bag woven jute fabric reinforced polymer composite.
Figure 4.63: Flexure fracture surface of Hessian fabric reinforced polymer composite

Figure 4.64: SEM image of the crack surface of fine jute fabric half bleached stiff woven jute reinforced polymer composite

Figure 4.65: SEM image of the flexure fracture surface of carpet backing cloth half bleached woven fabric reinforced polymer composite.
Usually jute fiber is composed of a numerous microscopic fiber bundle as was seen in the SEM imaging of the jute fiber. From the figures 4.61 to 4.65 it is unclear that whether the liquid resin penetrated the micro fibril or not. A large number of micro voids are observed. These are generated by the volatile reaction by-product formation and entrapment of these volatiles by curing and shrinkage of the matrix. The shearing effect is seen to split out the micro fibrils of jute fiber.

4.10 COMPARISON OF THE STRENGTH AND PHYSICAL PROPERTY OF CHOPPED JUTE, WOVEN JUTE MAT AND GLASS FIBER REINFORCED POLYMER COMPOSITE

Figure 4.66 and 4.67 represents the comparative study of the tensile and flexural fracture surface of chopped jute, woven jute mat and the glass fiber reinforced composites respectively. Chopped woolenized jute and fine jute fabric natural woven jute composite has been selected for comparison with glass fiber reinforced composite.

Strength of fiber reinforced polymer composite is dependent on many factors, which ensures effective load transfer from the matrix to the reinforcing fiber. Strength of fiber and the matrix, fiber matrix compatibility and interfacial strength, fiber aspect ratio, critical length, critical volume fraction and fiber orientation etc are the regulatory terms of a fiber reinforced polymer composite. Laminated composite strength is also dependent on the number of ply and its orientation.

Table 4.8: Comparison of tensile and flexural strength property of chopped jute, woven jute and glass fiber reinforced polymer composite.

<table>
<thead>
<tr>
<th>Composite name and symbol</th>
<th>Fiber length/ type</th>
<th>Volume %</th>
<th>No of ply</th>
<th>Ultimate Tensile Strength MPA</th>
<th>Flexural strength MPA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference polymer: Ref</td>
<td>0</td>
<td>0</td>
<td>18.64</td>
<td>47.31</td>
<td></td>
</tr>
<tr>
<td>Glass fiber reinforced polymer composite:</td>
<td>5.5cm</td>
<td>15.78</td>
<td>22</td>
<td>91.40</td>
<td>179.04</td>
</tr>
<tr>
<td>Chopped strand mat: GFRP</td>
<td>Woven</td>
<td>18.98</td>
<td>13</td>
<td>31.74</td>
<td>57.34</td>
</tr>
<tr>
<td>Fine jute fabric natural: Jiral</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chopped Rotted &amp; woolenized Jute fiber 5-</td>
<td>5+1mm</td>
<td>5.46</td>
<td></td>
<td>14.24</td>
<td>28.11</td>
</tr>
<tr>
<td>volume%, JW5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chopped Rotted &amp; woolenized Jute fiber 10-</td>
<td>5+1mm</td>
<td>9.46</td>
<td></td>
<td>13.87</td>
<td>33.92</td>
</tr>
<tr>
<td>volume%, JW10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chopped Rotted &amp; woolenized Jute fiber 20-</td>
<td>5+1mm</td>
<td>26.49</td>
<td></td>
<td>21.65</td>
<td>36.32</td>
</tr>
<tr>
<td>volume%, JW20</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 4.66: Comparison of tensile strength property of chopped jute, woven jute and glass fiber reinforced polymer composite by analyzing fracture surface.
Figure 4.67: Comparison of flexural strength property of chopped jute, woven jute and glass fiber reinforced polymer composite by analyzing fracture surface.
Table 4.8 shows the comparison of tensile and flexural strength property of chopped jute, woven jute and glass fiber reinforced polymer composite. Figure 4.66 and 4.67 shows that there are large numbers of fibers pulled out anisotropically from the matrix of glass fiber reinforced composite. But pulled out yarn at some particular position is seen on the fracture surface of the woven jute composite. Gradual increase in fiber on the fracture surface is seen on the chopped woolenized jute fiber reinforced composite with increasing volume fraction. Thus, fiber length, ply number and compatibility between the matrix and fiber ensure higher strength of glass fiber reinforced composite.

Although the yarn length is higher for the woven jute but since, yarn is made, the surface area of individual fiber is reduced for effective load transfer. So the woven jute composite shows lesser strength than that of the glass fiber reinforced composite. For the chopped jute the effective load transfer area is reduced because of fiber chopping and so it shows lesser strength than the woven jute and glass fiber reinforced composite.

Table 4.9 shows the density, void percentage and percentage water absorption with relative strengths of composites.

Figure 4.68: Density variation of chopped jute and woven jute laminate composites

Figure 4.68 and 4.69 and table 4.9 shows that with increasing density and decreasing void percentage, chopped retted jute and retted and bleached jute composite shows consistent increase in strength. Strength of retted and bleached jute composite is not strictly density and void percentage dependent. This may be due to the load transfer
factor between the fiber and the matrix. Tensile and flexural strength of woven jute fabric reinforced composite is not dependent on its bulk density and the void fraction; rather its strength is solely governed with the stiffness of the yarns regardless of the compactness of the woven fabrics.

Table 4.9: Comparison of density, void fraction and strength of composites

<table>
<thead>
<tr>
<th>(Symbol) Name</th>
<th>Volume %</th>
<th>Calculated Density gm/cc</th>
<th>Bulk Density gm/cc</th>
<th>% Void</th>
<th>Average Tensile strength, MPa</th>
<th>Average Flexural strength, MPa</th>
<th>% water absorption</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Ref) Reference polymer</td>
<td>0</td>
<td>1.04</td>
<td>1.03</td>
<td>1.23</td>
<td>18.62</td>
<td>47.22</td>
<td>9.11</td>
</tr>
<tr>
<td>(GFRP) Glass fiber reinforced polymer composite</td>
<td>9.78</td>
<td>1.29</td>
<td>1.28</td>
<td>0.84</td>
<td>91.40</td>
<td>179.04</td>
<td>0.02</td>
</tr>
<tr>
<td>(JR3) Chopped Retted Jute fiber 5-volume%</td>
<td>6.59</td>
<td>1.05</td>
<td>1.05</td>
<td>0.05</td>
<td>41.27</td>
<td>24.73</td>
<td>0.71</td>
</tr>
<tr>
<td>(JR10) Chopped Retted Jute fiber 10-volume%</td>
<td>10.65</td>
<td>1.07</td>
<td>1.06</td>
<td>0.94</td>
<td>41.98</td>
<td>28.12</td>
<td>1.28</td>
</tr>
<tr>
<td>(JR20) Chopped Retted Jute fiber 20-volume%</td>
<td>19.98</td>
<td>1.09</td>
<td>1.08</td>
<td>0.80</td>
<td>21.67</td>
<td>38.86</td>
<td>1.91</td>
</tr>
<tr>
<td>(JW5) Chopped Retted &amp; woollenized Jute fiber 5-volume%</td>
<td>5.46</td>
<td>1.05</td>
<td>1.03</td>
<td>2.39</td>
<td>6.30</td>
<td>31.52</td>
<td>0.95</td>
</tr>
<tr>
<td>(JW10) Chopped Retted &amp; woollenized Jute fiber 10-volume%</td>
<td>9.46</td>
<td>1.07</td>
<td>1.04</td>
<td>3.93</td>
<td>14.07</td>
<td>33.63</td>
<td>1.40</td>
</tr>
<tr>
<td>(JW20) Chopped Retted &amp; woollenized Jute fiber 20-volume%</td>
<td>26.49</td>
<td>1.11</td>
<td>1.10</td>
<td>1.67</td>
<td>21.67</td>
<td>48.85</td>
<td>1.77</td>
</tr>
<tr>
<td>(JRB6) Chopped Retted &amp; Bleached Jute fiber 6-volume%</td>
<td>7.24</td>
<td>1.06</td>
<td>1.04</td>
<td>1.69</td>
<td>13.05</td>
<td>41.14</td>
<td>1.24</td>
</tr>
<tr>
<td>(JRB13) Chopped Retted &amp; Bleached Jute fiber 13-volume%</td>
<td>13.72</td>
<td>1.08</td>
<td>1.06</td>
<td>1.49</td>
<td>14.70</td>
<td>36.54</td>
<td>1.43</td>
</tr>
<tr>
<td>(JRB15) Chopped Retted &amp; Bleached Jute fiber 15-volume%</td>
<td>14.96</td>
<td>1.08</td>
<td>1.07</td>
<td>1.08</td>
<td>22.34</td>
<td>57.37</td>
<td>1.47</td>
</tr>
<tr>
<td>(JRB23) Chopped Retted &amp; Bleached Jute fiber 23-volume%</td>
<td>23.73</td>
<td>1.10</td>
<td>1.09</td>
<td>0.80</td>
<td>22.21</td>
<td>47.22</td>
<td>1.81</td>
</tr>
<tr>
<td>(JRA1) Fine jute fabric natural</td>
<td>18.98</td>
<td>1.09</td>
<td>1.08</td>
<td>1.00</td>
<td>51.73</td>
<td>57.34</td>
<td>1.43</td>
</tr>
<tr>
<td>(JRA2) Food grade benolla twill bag</td>
<td>23.26</td>
<td>1.09</td>
<td>1.09</td>
<td>0.85</td>
<td>28.88</td>
<td>53.03</td>
<td>1.99</td>
</tr>
<tr>
<td>(JRA3) Hessian fabric</td>
<td>13.7</td>
<td>1.08</td>
<td>1.07</td>
<td>1.37</td>
<td>39.28</td>
<td>59.09</td>
<td>1.47</td>
</tr>
<tr>
<td>(JRBa1) Fine jute fabric full bleached stiff</td>
<td>21.9</td>
<td>1.09</td>
<td>1.09</td>
<td>0.60</td>
<td>23.55</td>
<td>50.24</td>
<td>2.28</td>
</tr>
<tr>
<td>(JRBa2) Carpet backing cloth half bleached</td>
<td>19.52</td>
<td>1.09</td>
<td>1.08</td>
<td>0.94</td>
<td>21.94</td>
<td>69.04</td>
<td>2.50</td>
</tr>
</tbody>
</table>

Figure 4.70 shows that the percentage water uptake by the composite specimens increases with increase in the percentage of jute fiber, which is shown in the table 4.9. The woven jute composite showed higher water absorption property than the chopped jute composite due to the directional nature. The bleached variety of woven jute mat showed maximum water absorption, which implies bleaching treatment.
exposes maximum of (-OH) groups of the cellulose of jute fiber, and the composite fabricated with this fiber or mat becomes more hydrophilic in nature.

![Void percentage of chopped jute composite and woven jute laminates](image1)

**Figure 4.69:** Volume effect on the void percentage of chopped jute and woven jute laminates

![Water absorption properties of chopped jute composite and woven jute laminates](image2)

**Figure 4.70:** Water absorption characteristics of chopped jute and woven jute composite

Figure 4.71 a, b, c and d shows the relationship between the theoretical and experimental tensile strength value of the chopped jute and woven jute laminated composite.
Because of the larger theoretical and experimental tensile strength value of the glass fiber reinforced composite comparison has been avoided in the curves of chopped jute composite. A huge deviation from the theoretical tensile strength of the composites is observed in figure 4.71. These theoretical tensile strength has been derived from the rule of mixture and standard tensile strength value of jute fibers were obtained from table 2.1 of the literature review chapter [Bichhorn, 2001]. Experimental tensile strength decreases, since incorporation of reinforcing fiber introduces flaw inside the matrix and at the fiber matrix interface or interphase due to incoherency and the volatile reaction products formed inside the cured polymer matrix.

Table 4.10: Deviation of theoretical and experimental ultimate tensile strength of chopped jute and woven jute composite.

<table>
<thead>
<tr>
<th>Name</th>
<th>Fiber Volume %</th>
<th>Resin Vol%</th>
<th>Properties Rule of Mixture (UTS) MPa</th>
<th>Average strength (UTS) MPa</th>
<th>% Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ref</td>
<td>0</td>
<td>100</td>
<td>18.6</td>
<td>18.6</td>
<td>0.0</td>
</tr>
<tr>
<td>GFRP</td>
<td>15.87</td>
<td>8.13</td>
<td>428.8</td>
<td>91.4</td>
<td>79.1</td>
</tr>
<tr>
<td>JR5T</td>
<td>6.59</td>
<td>93.44</td>
<td>42.3</td>
<td>14.3</td>
<td>66.2</td>
</tr>
<tr>
<td>JR1OT</td>
<td>10.65</td>
<td>89.35</td>
<td>56.8</td>
<td>13.9</td>
<td>75.5</td>
</tr>
<tr>
<td>JR2OT</td>
<td>19.98</td>
<td>80.02</td>
<td>90.3</td>
<td>21.7</td>
<td>76.0</td>
</tr>
<tr>
<td>JW5T</td>
<td>5.46</td>
<td>94.54</td>
<td>38.7</td>
<td>6.3</td>
<td>83.7</td>
</tr>
<tr>
<td>JW1OT</td>
<td>9.46</td>
<td>90.54</td>
<td>53.5</td>
<td>14.1</td>
<td>73.7</td>
</tr>
<tr>
<td>JW2OT</td>
<td>26.46</td>
<td>72.502</td>
<td>116.3</td>
<td>21.7</td>
<td>85.4</td>
</tr>
<tr>
<td>JRB6T</td>
<td>7.24</td>
<td>93.76</td>
<td>45.7</td>
<td>11.1</td>
<td>71.4</td>
</tr>
<tr>
<td>JRB13T</td>
<td>13.72</td>
<td>86.28</td>
<td>69.9</td>
<td>14.7</td>
<td>79.0</td>
</tr>
<tr>
<td>JRB15T</td>
<td>14.96</td>
<td>83.04</td>
<td>74.5</td>
<td>22.4</td>
<td>69.9</td>
</tr>
<tr>
<td>JRB23T</td>
<td>23.73</td>
<td>76.27</td>
<td>107.2</td>
<td>22.2</td>
<td>79.3</td>
</tr>
<tr>
<td>JRA1</td>
<td>18.9877</td>
<td>81.0123</td>
<td>108.1</td>
<td>31.7</td>
<td>70.6</td>
</tr>
<tr>
<td>JRA2</td>
<td>23.26</td>
<td>76.74</td>
<td>128.3</td>
<td>28.8</td>
<td>77.5</td>
</tr>
<tr>
<td>JRA3</td>
<td>15.7</td>
<td>84.3</td>
<td>92.6</td>
<td>30.6</td>
<td>67.0</td>
</tr>
<tr>
<td>JRBa1</td>
<td>21.97</td>
<td>78.03</td>
<td>102.4</td>
<td>25.6</td>
<td>75.0</td>
</tr>
<tr>
<td>JRBa2</td>
<td>19.52</td>
<td>80.48</td>
<td>93.1</td>
<td>21.9</td>
<td>75.4</td>
</tr>
</tbody>
</table>

Table 4.10 shows the Deviation of theoretical and experimental ultimate tensile strength of chopped jute and woven jute composite. This is due to the processing difficulties that introduces flaw inside the composite body.
Theoretical and experimental tensile property of chopped retted jute composites

(a) Chopped retted jute composite

Theoretical and experimental tensile property of chopped retted and woolenized jute composites

(b) Chopped retted and woolenized jute composite

Theoretical and experimental tensile property of chopped retted and bleached jute composites

(c) Chopped retted and bleached jute composite
4.10.1 Economic consideration of chopped jute and woven jute laminated composite

Figure 4.72 shows the comparison of unit cost of composites. The cost of the reference polymer is the highest. Cost per unit wt of composites decreases with increasing volume percentage of chopped jute or woven jute laminated composites.
So it is easily understood that the cost of jute fiber reinforced composite will
decrease more if industrial process of fabricating composites are selected.

4.11 FOURIER TRANSFORMATION INFRARED ANALYSIS OF JUTE-
POLYESTER COMPOSITES

Fourier transformation infrared or FTIR analysis was carried out on cured polyester,
retted jute and ‘retted and bleached’ jute fiber. FTIR analysis was also carried out on
some composites. The tests were made to compare the bonding nature between
matrix and fiber.

Table 4.11 represents the average tensile and flexure strength data of the entire
composite specimens fabricated during the project. The table also indicates about the
Fourier transformation infrared test that was performed on selected composite
specimens.

<table>
<thead>
<tr>
<th>Name</th>
<th>Symbol</th>
<th>Avg. Flexure strength MPa</th>
<th>Avg. Tensile strength MPa</th>
<th>FTIR Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass fiber reinforced polyester</td>
<td>GFRP</td>
<td>179.04</td>
<td>91.40</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted jute fiber reinforced polyester (5 vol%)</td>
<td>JR5</td>
<td>24.69</td>
<td>14.27</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted jute fiber reinforced polyester (10 vol%)</td>
<td>JR10</td>
<td>28.11</td>
<td>13.90</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted jute fiber reinforced polyester (20 vol%)</td>
<td>JR20</td>
<td>38.80</td>
<td>21.67</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (6 vol%)</td>
<td>JR36</td>
<td>41.14</td>
<td>13.05</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (13 vol%)</td>
<td>JRB13</td>
<td>36.53</td>
<td>14.70</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (15 vol%)</td>
<td>JRB15</td>
<td>57.36</td>
<td>22.44</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted &amp; bleached jute fiber reinforced polyester (23 vol%)</td>
<td>JRB23</td>
<td>47.22</td>
<td>22.21</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted &amp; woolenized jute fiber reinforced polyester (5 vol%)</td>
<td>JW5</td>
<td>31.51</td>
<td>6.30</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted &amp; woolenized jute fiber reinforced polyester (10 vol%)</td>
<td>JW10</td>
<td>33.92</td>
<td>14.05</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Retted &amp; woolenized jute fiber reinforced polyester (20 vol%)</td>
<td>JW20</td>
<td>45.82</td>
<td>21.67</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Fine jute fabric natural, type 15 x 15 (Retted Variety)</td>
<td>JRA1</td>
<td>59.25</td>
<td>28.49</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Food grade Bercollia twill bag (Retted Variety)</td>
<td>JRA2</td>
<td>48.61</td>
<td>30.62</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Hessian fabric (Retted Variety)</td>
<td>JRA3</td>
<td>58.03</td>
<td>30.05</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Fine jute fabric full bleached stuff, type 15 x 15</td>
<td>JRD41</td>
<td>46.44</td>
<td>26.62</td>
<td>FTIR Test</td>
</tr>
<tr>
<td>Carpet backing cloth half bleached type 15 x 13</td>
<td>JRB42</td>
<td>68.09</td>
<td>19.30</td>
<td>FTIR Test</td>
</tr>
</tbody>
</table>
It was observed that the jute fiber becomes brittle when it reacts with initiated polyester resin. It is known that cellulose contains "-OH" groups, and unsaturated polyester oligomer contains ester "- COOR" and "- OH" reactive groups and unsaturated double bonds within its skeleton. The catalyst, which breaks down with a free electron, only attacks the unsaturated double bond by prioritizing its nucleophile nature. This can be best explained by the fact; peroxide that decomposes and produces a free electron by breaking the bonds and creates a reactive site known as the nucleophile and at the same time the ">C=C<" i.e. C - C double bond acts as electrophile. But it is not possible to say that the "-OH" groups of jute fiber react with the unsaturated bonds of the unsaturated polyester. There may be reaction between the "-COOR" and the "-OH" within the polymer itself, or "-OH" group of cellulose of jute fiber [http://www.tifac.org.in/index1.htm; http://www.chem.gla.ac.uk/staff/mikej/Celulose.htm].

Figure 4.73 shows the typical ortho phthalic unsaturated polyester resin. It is observed that an alcoholic -OH group on the left of the figure 4.68 and a -COOH group on the right. Along with this it is also observed that there are seven C - O groups and four C=O groups in the chain. C=C, C - C and C - H bond systems are also observed in the figure.

It can be assumed from the figure 4.73 that none but the -COOH group is susceptible to bond with the cellulose -OH groups to bond of jute fiber. But chance of reacting the cellulose -OH group with C=O, C - O, C=C, C - C and C - H is little or negligible because of the affinity factor and steric hindrance of both the molecular segment of polymer and reinforcement fiber. The following section describes the FTIR characteristics of polymer fiber and composites.
4.11.1 FTIR of reference polymer

FTIR spectrum of the reference polymer is given in figure 4.75 and the spectrum in 4000 – 3500 cm⁻¹ range can be considered as noise and it is not significant in analysis. A peak within 3000 – 2800 cm⁻¹ range indicates aliphatic C – H stretching, but it is difficult to ascertain about the structure of the aliphatic portion of the whole molecule.

Peak within the 2400 – 2200 cm⁻¹ range appears as a doublet, which is due to the presence of the CO₂ molecule present in the test chamber.

Peak at 1733.9 cm⁻¹ is the C=O absorption band of saturated aliphatic esters (except formats) and falls within the 1750 – 1735 cm⁻¹ region and possesses sharp appearance. Peak at 1717.5 cm⁻¹ shows the C=O absorption bands of formats, α-β-unsaturated benzoate esters. Further conjugation has little or no additional effect upon the frequency of carbonyl absorption.

Since esters are composed of carboxylic acid so propagating further in the spectra of reference polymer shows that the peak of C=O stretching bands are more intense than ketonic C=O stretching and usually appears at 1760 cm⁻¹ for the monomers of saturated acid.

The carboxylic dimer has a center of symmetry; only the asymmetrical C=O stretching mode absorbs in the IR region. Hydrogen bonding and resonance weaken the C=O bond resulting in absorption at a lower frequency. The C=O group in dimerized saturated aliphatic acid absorbs in the region of 1720 – 1706 cm⁻¹. Although the polymer was unsaturated polyester (ortho phthalic), a saturated three-dimensional network is formed after the condensation reaction and cross-linking between the polymer chains. Peak at 1700.1 cm⁻¹ resembles the presence of dimerized saturated aliphatic acids that absorbs in this region.

Peak at 1696.3 cm⁻¹ indicates unsaturation in conjugation with carboxylic carbonyl group that decreases the frequency of absorption of both monomer and dimer. In general α-β-unsaturation and aryl conjugated acids show absorption for the dimer in the 1710 – 1680 cm⁻¹ region.
Below the 1500 cm\(^{-1}\) region of IR spectra is called the fingerprint region. In this region a complex patterned spectra appears and is the characteristics of a particular compound, and is very difficult to analyze properly.

Peak at 1260.4 cm\(^{-1}\) and 1119.6 cm\(^{-1}\) is the C – O stretching vibration of esters actually consists of two asymmetrical coupled vibrations C – C (\(=\)O) – O and O – C – C, the former being more important. These bands occur in 1300 – 1900 cm\(^{-1}\) region. The corresponding symmetrical vibrations are of little importance. The C – O stretch correlation are less reliable than the C=O correlations. The C – C (\(=\)O) – O stretch of esters of \(\alpha\)-\(\beta\)-unsaturated acids results in multiple bands in the 1300 – 1160 cm\(^{-1}\) region and is absent in the spectra. Peak at 1260.4 cm\(^{-1}\) and 1119.6 cm\(^{-1}\) signifies the esters of aromatic acid and stretch of lactones respectively.

Peak at 1066.6 cm\(^{-1}\) shows the resemblance of Si – O stretch. The significance of this band is not clearly understood from the spectra, but it is attributed to be the pre-addition of compatibiliser or may be the glass window used for the pellet holding. Peak at 700.1 cm\(^{-1}\) closely resembles the C – H bending vibration of alkynes or mono-substituted alkynes.

### 4.11.2 Spectra of retted jute and retted and bleached jute

Only the \(\alpha\) - cellulose is the fibrous portion of jute fiber, which seems to interact with the matrix polymer. So finding some information in the IR spectra about \(\alpha\) - cellulose will be significant [http://www.chem.gla.ac.uk/staff/mikej/Cellulose.htm].

![Cellulose](image-url)
Figure 4.74 indicates that there are seven C – O groups in the cellulose chain. It is clearly seen that the C – O – C contributes for the chain of cellulose. Within these seven groups four CH₂OH, and three C – OH, the former being the out of plane position in the cellulose structure and the latter contributes to the membership of the cyclic structure.

Figure 4.76 show the FTIR spectra of retted jute and retted and bleached jute. Both the spectra superimpose each other so an offset image is plotted for the ease of comparison.

It is already known that there are –OH groups in cellulose. The non-hydrogen bonded –OH groups in cellulose or free –OH groups of cellulose absorbs strongly at 3700 – 3584cm⁻¹ range, which is absent in the spectra shown in figure 4.76. Because of the natural hydrophilic tendency of jute the presence of equilibrium moisture in the fibrous sample also absorbs in the 3700 – 3584cm⁻¹ range so it interferes with the –OH groups of cellulose. This is the noise region of the FTIR spectra. Although the presence of –OH groups in cellulose is known but it is not confirmed from the spectra [http://sis.bris.ac.uk/~sd9319/spec/IR.htm#irspec].

Peak at 2920.0cm⁻¹ reveals the C – H stretching of aliphatic compounds. A peak for CO₂ also appears at 2400 – 2200cm⁻¹ region with in the spectra.

Peak between 1733.9 to 1596.9cm⁻¹ range reveals the presence of C – O groups within cellulose chain. Although this is the double bond region and the most common and characteristic of the double bonded group absorbing in this region are the carbonyls, but it is not confirmed from the spectra, since practically C – O is present rather than C = O in cellulose.

Peak at 1541.0cm⁻¹ shows the presence of C=O of amides and 1490.9cm⁻¹ reveals the aromatic compounds with low degree of substitution.

Highly substituted materials of aromatic origin have absorptions of variable intensity near 1400cm⁻¹. The number and position of the substituents can usually be derived from the 670 – 900cm⁻¹ range. Peak at 1419.5cm⁻¹ and 898.8cm⁻¹ resembles the above fact or the latter may arise from dme. Peak at 1375.2cm⁻¹ may be the methyl symmetrical bending absorption. Peak at 1259.4 resembles the C – O stretch and –OH deformation bands in alcohol. Si – O stretch is also seen at 1039.6cm⁻¹. The peak that are shown in the spectra other than C – O and –OH resembles the presence of
fiber body binding materials and other organic additives, which can have a profound effect on the fiber-matrix compatibility and reinforcing mechanism. Also inadequate removal of the jute fiber-processing chemical of organic origin can have its appearance on the IR spectra.

FTIR spectra of retted and bleached jute reveals peak at 2854.5 cm$^{-1}$, which resembles the C-H stretching of aliphatic origin. CO$_2$ peak appears as usually within the 2400 -2200 cm$^{-1}$ range. A broadened peak at 2462.9 cm$^{-1}$ is quite hard to guess and may come from the alkyl halide i.e. chlorine containing stuff. The peak within the range 1733.9 cm$^{-1}$ to 1560.3 cm$^{-1}$ resembles the presence of C-O stretching of cellulose molecule along with this 1541.0 cm$^{-1}$ resembles the C=O stretching.

Although it is stated that the region below 1500 cm$^{-1}$ is the fingerprint region but still then the peaks 1508.2, 1490.9, 1458.1 signifies about some aromatic group with low degree of substitution. Peak at 1419.5 cm$^{-1}$ indicates the presence of highly substituted aromatic group. At 1261.4 cm$^{-1}$ C-O stretch and -OH deformation band of alcohol is seen. Peak at 1066.6 cm$^{-1}$ shows the Si-O stretch.[http://sis.bris.ac.uk/~sd9319/spec/IR.htm#ispec].

If a comparison between the two FTIR spectra of the retted jute to that of retted and bleached jute is made along with their respective SEM images (description of SEM is given in section 4.1 of this chapter) it can be inferred that the bleaching action has leached out the fibrous binding materials and the %transmittance for the retted jute is lower i.e. it absorbs higher energy in the IR range, instead the %transmittance of the bleached jute is higher, which implies the lower degree of IR absorption by the retted and bleached jute. This clearly shows the presence of the more fiber binding material in retted jute rather than retted and bleached jute. Although there is a variation of % transmittance between the two types of fiber but the fingerprint region of both the fiber is similar which implies that same jute fiber of same jute plant origin is supplied for the project by BJRI.

4.11.3 Spectral analysis of chopped retted jute composite

Figure 4.77 shows the FTIR spectral representation of fiber reinforced polymer matrix composite, which contains 5 volume percentage of retted and bleached jute fiber.
Peak at 3060.8 and 3026.1cm⁻¹ resembles the C–H stretching of aromatic and unsaturated groups. Peak at 2925.8 and 2852.5cm⁻¹ resembles the C–H stretching of aliphatic compounds. A peak for CO₂ is present as usual in the spectrum. Window region within 2700.0 to 1850.0cm⁻¹ shows two peaks at 2273.9 and 1944.1cm⁻¹. This may be attributed from the fact that the initiator or accelerator present in the composite system has its own composition that may react to form some products along with the polymerization reaction and signifies the presence of -C=N and since CO₂ has already appeared in the spectrum so there is a great possibility of presence of C=O (carbonyls) for the peak at 1944.1cm⁻¹.

At 1732.0cm⁻¹ resembles the presence of ester in the composite. The double bonded carbonyl groups show most common characteristic peaks within the range 1950–1430cm⁻¹. Strong absorption between 1540–1650cm⁻¹ may indicate the C=O groups of amides; although there are some strong sharp peaks but it is not clear from the fact that whether these peaks are form the matrix polymer, jute fiber or the chemical additives that are added with the composite system [http://sia.bris.ac.uk/~sd9319/spec/IR.htm/#rspec].

The aliphatic C=C stretch absorptions are found in the range 1630–1690cm⁻¹ range. Probably all the unsaturation of the ortho phthalic polyester will react to form a cross-linked structure, so peaks at 1647.1 and 1652.9cm⁻¹ may resemble the presence of unsaturation.

The C–H motions attributes the methylene scissors vibration found near 1467cm⁻¹ and the asymmetrical CH₃ bending modes falls nearly 1460.0cm⁻¹; peak at 1452.3cm⁻¹ may define the presence of ether (–O–) functional group. Singlet at the 1373.2cm⁻¹ resembles the presence of methyl symmetrical bends. Ester in addition to their C=O stretch, also show strong C–O–R stretch absorption around 1200–1300cm⁻¹ region, which resembles the peak at 1282.6cm⁻¹, so it is confirmed that ester has formed with different chemical environment.

C–O–C stretch are found in ethers and esters around 1200–1100cm⁻¹ and the C–O stretch and O–H deformation bands in alcohol are found with in 1260–1000cm⁻¹ range. Although a small peak at 1041.5cm⁻¹ is observed, which resembles the presence of the Si–O stretch but the probability of the former i.e. ester related functional groups are greater. 991.3cm⁻¹ and 908.4cm⁻¹ shows the diene stretch,
since the diene stretch occurs at 1000 – 900 cm\(^{-1}\) region, but it does not reveal the presence of diene in that cross-linked network.

As shown in the figure 4.76 the ester peak was sharp for the matrix polymer and for fiber it resembles the presence of C –O. But the ester absorption region of FTIR spectra in composite shows more intense absorption with broader peak. This fact reveals that the ester formation environment has changed. But since there are –OH groups present in jute fiber that may have more affinity towards reacting with the –COOH end group of ortho phthalic polyester rather than with C –O groups of jute fiber. If reaction occurs with C –O then the fiber will degrade; since, if the cyclic structure of the glucose unit of cellulose degrades then there will be no reinforcing behavior of jute fiber. There is a chance of hydrogen bonding with the –OH group’s of polymer and fiber. The –OH group of polymer is the end group of the propagating polymer chain that reacts with the –COOH end group of ortho phthalic polyester rather than with C –O. Also the steric hindrance of the bigger polymer chain with the cellulose fiber will not allow reacting at any position rather than at –OH site.

The wave number variation and %transmittance has no impact on the strength of composites. Strength has increased along with the increment of volume fraction of fiber for the composites. It was assumed that the variation of percentage transmittance might have some effect on the composite strength, either positively or negatively. Especially when the fiber volume fraction increases, the probability of effective load transfer from matrix to fiber increases thereby facilitating stronger composite materials.

Figure 4.78 shows strong IR absorption peak for the composites compared to the reference polymer. This may be due to the formation of ester group in between the fiber and the polymer. This is due to higher absorption of IR in the –CH and carboxylic stress region and the result is peak broadening.

The C=O absorption band of saturated aliphatic esters (except formats) within 1750 – 1735 cm\(^{-1}\) region and has sharp appearance. Formation of esters is confirmed in the spectra for all the spectra of the chopped retted jute composite within 1000 – 1260 cm\(^{-1}\) finger print region. It is also seen from the figure 4.73 that the finger print region of all composites has the same spectral pattern except the change in intensity of the spectra. The higher volume fraction jute fiber reinforced composite shows
higher transmittance and the lower volume fraction composite shows higher IR absorption. This may be due to the fact that at lower percentage of fiber, there is lower entanglement and lower steric hindrance towards polymer, so it could be obvious that at lower volume fraction the fiber may react with much more affinity towards polymer than at higher volume fraction of jute fiber.

4.11.4 Spectral analysis of woven jute fabric reinforced composite

This section consists of FTIR spectral information of fine jute fabric natural, food grade benolla twill bag and hessian fabric reinforced unsaturated polyester composite. First the FTIR spectral data of fine jute fabric natural woven mat will be discussed in brief and latter, spectral resemblance between the other two woven mats will be compared and discussed.

FTIR spectral data of the fine jute fabric natural woven mat composite is shown in figure 4.79 and it is similar to that of the chopped retted jute fiber reinforced composite. The C=O absorption band within the 1750 - 1735 cm\(^{-1}\) region resembles blunt appearance of saturated aliphatic esters (except formates). Peak within 1000 - 1260 cm\(^{-1}\) range confirms the resemblance of the C–O–C stretch in ethers and esters or C–O stretch and O–H deformation bands in alcohols.

Figure 4.79 shows the comparison of the IR data of fine jute fabric natural, food grade benolla twill bag and Hessian fabric reinforced unsaturated polyester composite. There is a closer match between all the spectral information. The blunt headed intense peak for ester group appeared at 1732.0, 1732.0 and 1718.5 cm\(^{-1}\) for the fine jute fabric natural, hessian fabric and food grade benolla twill bag reinforced unsaturated polyester composite respectively.

The percent transmittance for the hessian fabric reinforced unsaturated polyester composite is higher than that of the other two retted woven mat reinforced composite, although fine jute fabric natural reinforced unsaturated polyester composite showed the same wave number as that of the hessian fabric reinforced unsaturated polyester composite.

The peak 1718.5 cm\(^{-1}\) of the food grade benolla twill bag reinforced composite indicates some environmental change during ester formation.
Peak that fall within the fingerprint region from 1000 – 1260 cm\(^{-1}\) range indicates the presence of C=O – C stretching of esters, other than the ester of reference polymer. But for the prediction of fiber matrix compatibility assumptions were made that the only reaction possible is in between the –COOH groups of ortho phthalic unsaturated polyester and that of the –OH groups of cellulose. Not only the –COOH and –OH groups, but also C=O and –OH and other oxygen containing functional group may undergo hydrogen bonding within composites, which is not clearly verified from the spectrum. Moreover it was observed that jute fibers becomes brittle reacting with resin. This behavior may denote the ester formation reaction between the matrix and the fiber rather than hydrogen bonding [http://www.tifac.org.in/index1.htm; Eichhorn, 2001] and any types of mechanical interaction. Since the woven fabrics have constant impregnation factor, so volume percentage of these fabrics in the matrix cannot be controlled. As the chopped retted jute fiber reinforced composite, the volume fraction effect is apparent on the transmittance of woven jute fabric reinforced composite.

4.11.5 FTIR spectral analysis of retted and bleached woven jute mat reinforced composite

This section consists of the FTIR spectral information of fine jute fabric full bleached stiff, and carpet-backing cloth reinforced unsaturated polyester composite. First the FTIR spectral data of the fine jute fabric full bleached stiff woven mat will be discussed in brief and latter, spectral resemblance between the other two woven mats will be compared and discussed.

Figure 4.80 denotes the FTIR spectral data of fine jute fabric full bleached stiff woven mat reinforced composite. Peak 3246.0, 3060.8 and 3026.1 cm\(^{-1}\) resembles the C=H stretching of aromatic and unsaturated groups. Peak at 2931.6 and 2854.5 cm\(^{-1}\) resembles the C–H stretching of aliphatic compounds. A peak for CO\(_2\) is present in the spectrum. In the window region in between the 2700.0 to 1850.0 cm\(^{-1}\) shows the presence of CO\(_2\) and peak at 1948.0 cm\(^{-1}\) may be attributed to the presence of –C=N group of initiator or accelerator also there is a great possibility of presence of C=O (carbonyls) at that region.
The C=O absorption band of saturated aliphatic esters (except formates) with blunt appearance is present in the 1750 - 1735cm⁻¹ region. The C = O - C or C - O group stretch in ethers and esters and O - H deformation bands in alcohols are inferred within 1000 - 1260cm⁻¹ wave number region. Peak within this finger print region confirms the formation of ester.

The volume fraction of fiber in FIF full bleached stiff and CBC half bleached reinforced unsaturated polyester composite are 21.97 and 19.52 respectively. But if a comparison is made between the two spectra then it is found that the percentage transmittance of the former is lower i.e. higher IR absorption in the ester region, although the latter absorbs at little higher wave number. This indicates the formation of ester is enhanced upon bleaching treatment rather than the least bleached fabric. In addition it is seen that there is a nice match in the finger print region between the two types of composite but with varying intensity. This may be attributed to the fact that higher degree of bleaching ensures good chemical interaction with the resin, but since, the chemical treatment removes the binders of fiber so its tensile modulus increases but transverse strength decreases [Khan, 2005].

Figure 4.80 clearly indicate that the lower volume fraction of fiber-reinforced composites absorbs IR at higher intensity in the 1750 - 1735cm⁻¹ region, which is an indication of good fiber matrix interaction. This interaction may be of chemical origin.

From these FTIR spectra it is not possible to conclude precisely about the resemblance of strength properties of composites.

4.11.6 FTIR comparison of the fiber, matrix, chopped jute and woven jute fabric reinforced composite

Figure 4.81 shows the FTIR comparison of the fiber, matrix, chopped jute and woven jute fabric reinforced composite. Peaks at 2920.0cm⁻¹ region denote the C - H stretching of aliphatic compounds. CO₂ absorption band also appears at 2400 - 2200cm⁻¹ region with in the spectra.

Peak between 1733.9 to 1596.9cm⁻¹ range reveals the presence of C - O groups within cellulose chain. Although this is the double bond region and the most
common and characteristic of the double bonded group absorbing in this region are the carbonyls, but it is not confirmed from these three spectra, since practically C – O is present rather than C = O in cellulose.

The increase in intensity C-H stretch and ester (COOR) stretching of composite in comparison with the reference polymer, may be due to the increase in the number of C-H in the composite system, and higher number of ester may be produce due to the reaction between the –OH group of jute fiber and the –COOH group of matrix.

The ester and carbonyl absorption region shows similar bands with different intensity for individual composites. The CBC half bleached woven fabric reinforced composite gives less intense band pattern among the composites, but the retted jute fabric and chopped jute composite shows bands of similar intensity. This may be due to the chemical reaction between fiber binding materials of retted jute with polymer.

The finger print region of individual composite in the spectra resembles similar pattern but with different intensity. The decrease in FTIR band intensity of CBC half bleached woven fabric reinforced composite may be due to the fiber bleaching treatment that removes the binders from the fibrous body.
Figure 4.75: FTIR Spectra of reference polymer
Figure 4.76: Comparison of IR spectra of retted jute to that of retted and bleached jute.
Figure 4.77: Comparison between the Reference polymer, reinforcement (retted jute 5 vol%) and Composite.
Figure 4.78: FTIR spectral comparison of chopped reeled jute reinforced composite
FTIR spectral comparison of woven jute fabric (retted variety) reinforced Unsaturated Polyester Polymer Matrix composite

Figure 4.79: FTIR spectral comparison of woven jute fabric reinforced Polymer composite
FTIR spectral comparison of retted and bleached woven jute mat reinforced Unsaturated Polyester Polymer Matrix composite

Figure 4.20: FTIR spectral comparison of retted and bleached woven jute mat reinforced Polymer composite
Figure 4.81: FTIR comparison of chopped retted jute and woven jute fabric reinforced polymer composite
Chapter 5
Summary and Conclusion
5. SUMMARY AND CONCLUSION

According to the present work on the development of the natural fiber reinforced polymer composite the following conclusions could be made.

Scanning electron micrographs of retted, bleached and woolenized jute fibers were carried out and different fiber surface morphology was found for different fiber treatment process.

Chopped jute and woven jute reinforced polymer composite fabricating techniques such as cold press molding and hand lay-up or contact molding were adopted during the course of the project. For both chopped jute fibers and mats, fiber volume fraction higher than 25-30% improper wetting of fiber with polymer was observed. Glass fiber reinforced polymer composite was fabricated to compare the strength property to that of the jute fiber reinforced composite.

Increase in tensile and flexural strength was found with increasing volume fraction of chopped jute in the composite. The average tensile strength of chopped jute composite exceeded the average tensile strength of reference polymer at higher volume percentage of fiber loading. Flexure strength of these chopped jute composites remain under the value of reference polymer. The glass fiber reinforced polymer composite showed higher value of tensile and flexural strength than that of jute fiber reinforced composite. This is due to the strong nature of glass fiber and effective load transfer from matrix to fiber at tensile or flexural loading condition. It was observed that fiber compaction and orientation strongly affects the strength of these composites. Woven jute fabric reinforced composite showed higher strength than that of the reference polymer in either case of tensile and flexure test, although the volume percentage of these fabrics in the matrix was below the volume percentage of chopped jute.

The bulk density and calculated density of the composites shows an idea of the void percentage with in any composite specimen. At higher volume of fiber loading the chopped jute composite showed increased density and lower void percentage with increase in strength property. This is not strictly followed by the chopped retted and bleached jute reinforced composite. With increasing density and decreasing void percentage the woven jute composite showed increased tensile strength. Increase in
water absorption characteristics with increasing volume percentage of jute fiber was observed for chopped jute and woven jute laminated composite.

Chopped jute composite showed interface dependence failure mode with good amount of fiber pullout in each case of tensile and flexural test. Shearing effect through crack deflection was also observed during flexural test. SEM studies showed polymeric residue on the fiber surface of the composite specimen. Maximum polymeric residues were seen on woolenized jute fiber and the minimum polymeric residues were seen on retted jute fiber, which implies increased compatibility between fiber and the matrix with treatment procedures.

Failure mode of woven jute fabric was also interface dependent. During tensile test the yarn that lie perpendicular to the load direction acted as flaw and non-load bearing component of the composites. These yarns were also acted as a source of defect when the composites were loaded transversely. During flexural test these yarns lie parallel to the loading direction. Both these fact lowers the effective volume fraction of fiber in woven jute fabric reinforced composite. Flexure fracture profile showed that the crack tip acted through these node (a 0-90° cross point of two yarns in woven jute fabric) points to ensure failure of these composites. Fiber pullout was prevalent mode of failure for tensile test. Severe shearing and fiber pullout was seen in case of flexural test. Bleached variety of woven jute fabric showed little amount of polymeric residue on the fiber surface due to good fiber matrix compatibility and is an effect of bleaching treatment.

Fourier Transformation Infra Red spectroscopy was carried out to compare and find out the bonding nature between matrix and fiber of the composites. The spectrum of chopped jute and woven jute fabric reinforced composites were similar to each other but with different intensity of infrared absorption. Since polyester contains – COOH (carboxylic acid group) and jute fiber contains – OH (hydroxyl group). It was assumed that ester formation is probable mode of compatibilization between the polyester matrix and jute fiber. Cured polyester showed strong IR absorption at 1733cm⁻¹ with a sharp peak but jute fiber showed strong – CO absorption at this region with double peak. All composite specimens showed intense absorption at around 1733cm⁻¹ region, which indicates the formation of different types of ester other than ester of cured polyester. The polymeric residues on the fiber surface
region with double peak. All composite specimens showed intense absorption at around 1733 cm\(^{-1}\) region, which indicates the formation of different types of ester other than ester of cured polyester. The polymeric residues on the fiber surface indicate some types of chemical reaction in between the jute fiber and the polyester matrix. So, ester formation is the probable mode of compatibilization between the jute fiber and the unsaturated polyester matrix.

5.1 FUTURE TRENDS

Treatment procedure other than bleaching and woolenization could be tried to improve the strength and morphological characteristics of jute fiber or improvement of bleaching and woolenization treatment could be made. Acetylation or grafting with various polymeric substances (polyvinyl alcohol, poly-methyl-methacrylate, hexa-ethylene- methacrylate, etc.), treatment may also be adopted to decrease the hygroscopic nature of jute fiber. Therefore improvement of interfacial characteristics between the jute fiber and the polymer is the most important area of current and future work. Jute fibers degrade at the 180-200\(^\circ\) temperature range so, such a chemical treatment must be selected that must have to impart increase in thermal stability, thereby facilitating high temperature fabrication technique and high temperature stability of composite part.

Different fabricating regime for incorporating higher volume fraction of chopped jute could be probable and woven jute laminated composite could be fabricated with compression or cold press molding to increase the volume fraction of woven jute. Voids are potential problems of chopped jute and woven jute laminated composite. The formation of voids are assisted by the formation of thermoset curing reaction so some chemicals must be used with resin to remove the voids from the curing reaction system before the initial gelation time, and is an area of future work of the current research. Polyester thermoset resin was used as matrix material during the project, and thermosets are not recyclable. So, after its useful life it must be incinerated to reduce the volume of waste and to obtain thermal energy for some useful work. From this point of view the jute fiber could be blended with thermoplastics to fabricate composite part of such properties where thermoset like behavior is not necessary and to ensure recyclability of these composite materials after its valuable life.
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