Effect of Alkali treatment on jute fibre Composites

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology
in
Metallurgical & Materials Engineering

By

S. PAVAN KUMAR

Department of Metallurgical & Materials Engineering
National Institute of Technology
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Under the Guidance of

Prof. B. B. VERMA

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CERTIFICATE

This is to certify that the thesis entitled, "EFFECT OF ALKALI TREATMENT ON JUTE FIBRE COMPOSITES" submitted by Sri S. Pavan Kumar in partial fulfillments for the requirements for the award of Bachelor of Technology Degree in Metallurgical & Materials Engineering at National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Date: 30-04-2007

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I would like to express my gratitude to Dr. G. S. Agarwal (Head of the Department), Prof. K.N.Singh and Prof. A.K.Panda for their valuable suggestions and encouragements at various stages of the work. I am also thankful to all staff members of Department of Metallurgical & Materials Engineering NIT Rourkela.

I am also thankful to Mr. Sameer Pradhan Mr. S. Heymram and Mr. Rajesh Pattnaik and others in the Department for providing all joyful environment in the lab and helping me out in different ways.

30th April 2007 (S. Pavan Kumar)

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CONTENT

ABSTRACT

The aim of our project was the improvement of the mechanical properties of natural-fibre-reinforced composites, as a result of optimization of the properties of jute fibres by the use of an NaOH treatment process. Jute fibres were subjected to a 5% alkali (NaOH) solution treatment for 0, 2, 4, 6 and 8 h at 30°C. The composites reinforced with alkali treated fibres showed improved mechanical properties. The improvement was maximum for the composites prepared with 4 h treated fibres. The ILSS improved by 26.56% and Bending Strength by 27.53%. An improvement in the crystallinity in the jute fibres increased its tenacity by 12%, 68% and 79% after 4, 6 and 8 h of treatment respectively. The rate of change of flexural strength and modulus were found to be linear with wt% of alkali treated fibres and the %breaking strain was reduced by 28% for composites prepared using 8-hr treated jute fibre. On plotting the different values of slopes obtained from the rates of improvement of the flexural strength and tenacity, against the NaOH treatment time, two different failure modes were apparent before and after 4 h of treatment. In the first region between 0 and 4 h, fibre pull out was predominant whereas in the second region between 6 and 8 h, transverse fracture occurred with a minimum fibre pull out. This observation was well supported by the SEM investigations of the fracture surfaces.

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Chapter 1

GENERAL INTRODUCTION

1.1 Introduction

The most important types of natural fibres used in composite materials are flax, hemp, jute, kenaf, and sisal due to their properties and availability. Jute is an important bast fibre with a number of advantages. Jute has high specific properties, low density, less abrasive behaviour to the processing equipment, good dimensional stability and harmlessness. Jute textile is a low cost eco-friendly product and is abundantly available, easy to transport and has superior drapability and moisture retention capacity. It is widely being used as a natural choice for plant mulching and rural road pavement construction. The biodegradable and low priced jute products merge with the soil after using providing nourishment to the soil. Being made of cellulose, on combustion, jute does not generate toxic gases. An organigram with a classification of the various fibres is presented in Fig. 1.1.

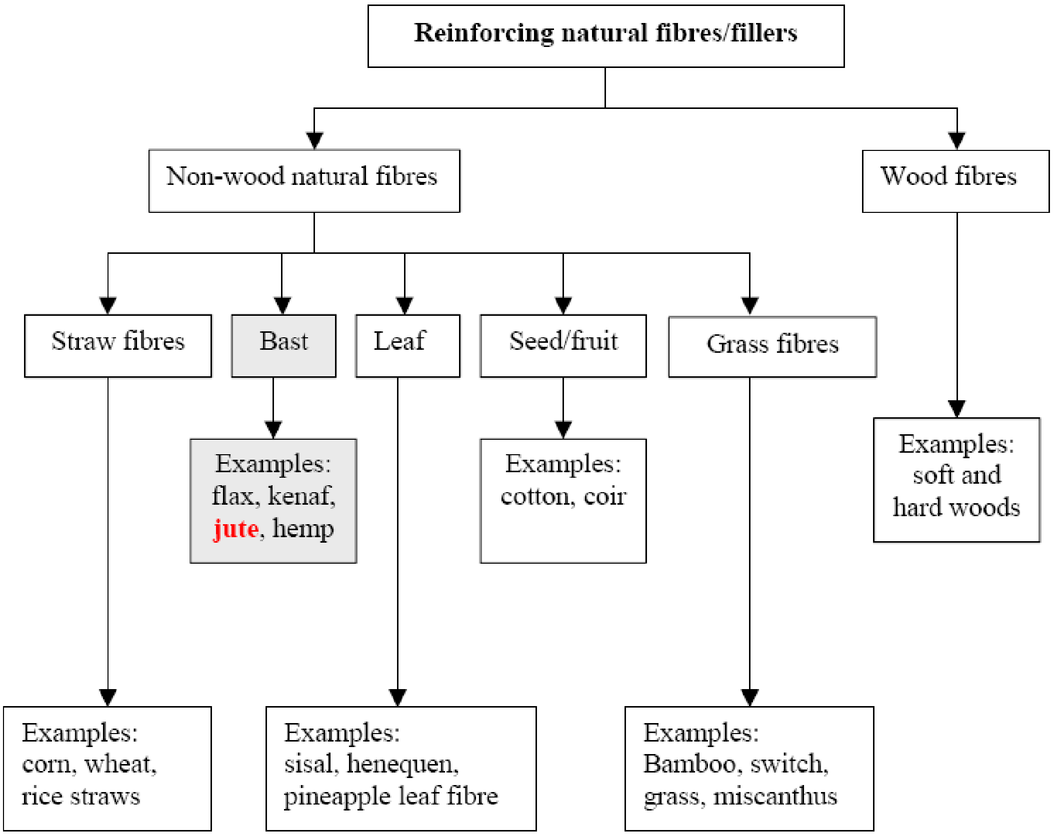
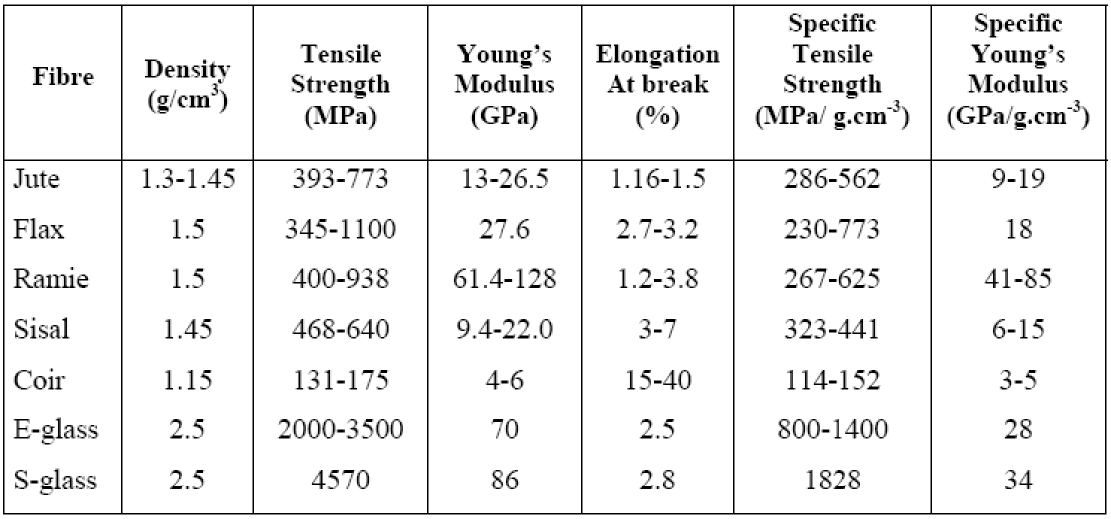


Fig.1.1. Classification of natural fibres which can be used as reinforcement or fillers in polymers [1]

Due to jute's low density combined with relatively stiff and strong behaviour, the specific properties of jute fibre can compare to those of glass and some other fibres (Table 1.1).

Table1.1. Properties of jute fibre in comparison with other fibres



Fibre	Density (g/cm^3)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation At break (%)	Specific Tensile Strength (MPa/ g.cm^{-3})	Specific Young's Modulus (GPa/g.cm^{-3})
Jute	1.3-1.45	393-773	13-26.5	1.16-1.5	286-562	9-19
Flax	1.5	345-1100	27.6	2.7-3.2	230-773	18
Ramie	1.5	400-938	61.4-128	1.2-3.8	267-625	41-85
Sisal	1.45	468-640	9.4-22.0	3-7	323-441	6-15
Coir	1.15	131-175	4-6	15-40	114-152	3-5
E-glass	2.5	2000-3500	70	2.5	800-1400	28
S-glass	2.5	4570	86	2.8	1828	34

The natural fibres can be used to reinforce both thermosetting and thermoplastic matrices. Thermosetting resins, such as epoxy, polyester, polyurethane, phenolic, etc. are commonly used today in natural fibre composites, in which composites requiring higher performance applications. They provide sufficient mechanical properties, in particular stiffness and strength, at acceptably low price levels. Compared to compounds based on thermoplastic polymers, thermoset compounds have a superior thermal stability and lower water absorption. However, in the case of the demand for improved recycling and in combination with new long fibre reinforced thermoplastic (LFT) processing, thermoplastic polymers have been expected to substitute the thermoset polymers.

Considering the ecological aspects of material selection, replacing synthetic fibres by natural ones is only a first step. Restricting the emission of green house effect causing gases such as CO2 into the atmosphere and an increasing awareness of the finiteness of fossil energy resources are leading to developing new materials that are entirely based on renewable resources.

The natural fibre composites can be very cost effective material for following applications:

- Building and construction industry: panels for partition and false ceiling, partition boards, wall, floor, window and door frames, roof tiles, mobile or pre-fabricated buildings which can be used in times of natural calamities such as floods, cyclones, earthquakes, etc.
- Storage devices: post-boxes, grain storage silos, bio-gas containers, etc.
- Furniture: chair, table, shower, bath units, etc.
- Electric devices: electrical appliances, pipes, etc.
- Everyday applications: lampshades, suitcases, helmets, etc.
- Transportation: automobile and railway coach interior, boat, etc.
- Toys

The reasons for the application of natural fibres in the automotive industry include [1, 2]:

- Low density: which may lead to a weight reduction of 10 to 30%.
- Acceptable mechanical properties, good acoustic properties.
- Favourable processing properties, for instance low wear on tools, etc.
- Options for new production technologies and materials.
- Favourable accident performance, high stability, less splintering.
- Favourable ecobalance for part production.
- Favourable ecobalance during vehicle operation due to weight savings.
- Occupational health benefits compared to glass fibres during production.
- No off-gassing of toxic compounds (in contrast to phenol resin bonded wood and recycled cotton fibre parts).
- Reduced fogging behaviour.
- Price advantages both for the fibres and the applied technologies.

Applications of natural fibre reinforcement for automotive parts are shown in Fig 1.2.

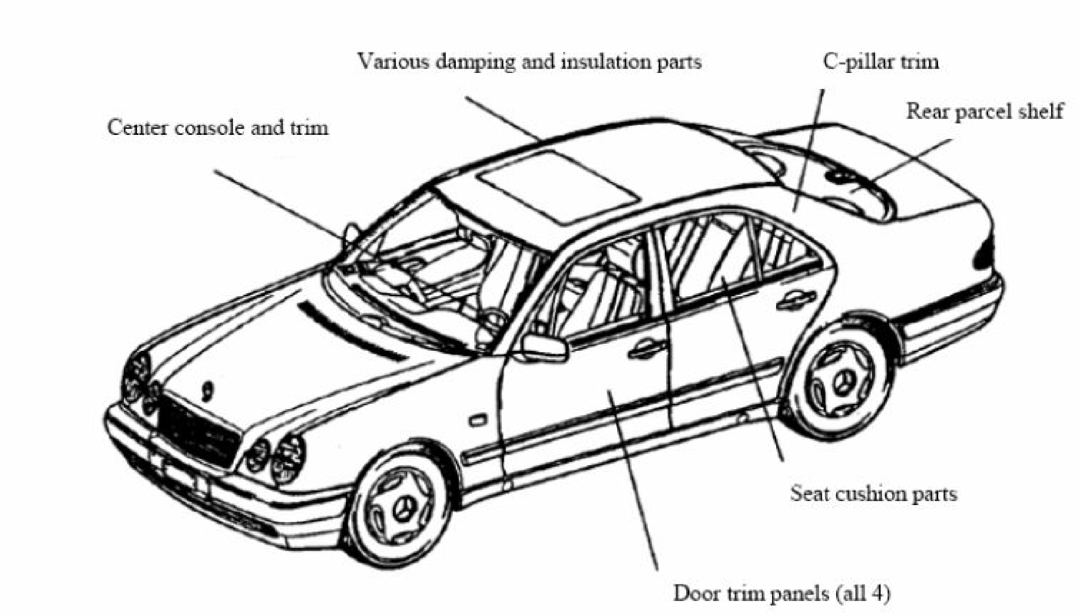


Fig.1.2. Plant fibre applications in the current Mercedes-Benz R-class [2]

Besides the advantages mentioned above, the natural fibre composites possess also some disadvantages. The main disadvantage is the poor compatibility between a hydrophobic polymer matrix and the hydrophilic fibres. This leads to the formation of weak interfaces, which result in poor mechanical properties of the composites. Other important disadvantages of the natural fibre composites are the high sensitivity of natural fibres towards water and the relatively poor thermal stability. Water absorption on composites is an issue to be considered since the water absorbed by the fibres in the composite could lead to swelling and dimensional instability and to a loss of mechanical properties due to the degradation of the fibres and the interface between the fibre and matrix [3, 4, 5, 6, 7, 8].

Therefore, to improve the adhesion between the matrix and the fibres, a third component, called compatibiliser, has to be used for matrix modification or the fibres have to be surface modified prior to the preparation of the composites [9, 10, 11]. Several studies have shown the influence of various types of chemical modifications on the performance of natural fibres and fibre reinforced composites. The different surface chemical modifications of natural fibres such as alkali treatment, silane treatment, isocyanate treatment, latex coating, permanganate treatment, acetylation, monomer grafting under UV radiation, etc. have achieved various levels of success in improving fibre strength and fibre/matrix adhesion in natural fibre composites.

For the composites based on thermoset matrices such as epoxy, polyester, phenol formaldehyde, etc., alkali treatment of natural fibres is one of the most common treatments of the natural fibres [12, 13] as it improves the interfacial adhesion between the fibre and matrix [14, 15, 16, 17]. Gassan and Bledzki reported that treating the fibre surface by NaOH 26 wt% for 20 minutes at 20°C improves the mechanical properties of unidirectional jute/epoxy composite up to 60% compared to untreated fibre composite, at a fibre content of 40 vol% [18]. Prasad et al. studied the alkali treatment of coir fibre by NaOH 5% for 72-96 hours at 28°C. An improvement of the tensile strength and Young's modulus of the fibre by 10-15% and 40%, respectively, was observed. The alkali treatment of the coir fibre improved the flexural strength of the polyester resin composites by 40% [19]. Thais et al. showed an increasing shear strength from 2.6 to 6.9 MPa in the case of fibre surface treatment by NaOH 2% for 1 hour at room temperature of sisal/polyester composites [20]. Ray et al. used a solution of NaOH 5% to treat the jute fibre for 0, 2, 4, 6 and 8 hours at 30°C. For the vinyester resin composites reinforced by 35 wt% jute fibre treated for 4 h, an improvement of 20% for the flexural strength, of 23% for the flexural modulus and of 19% for the laminar shear strength was observed [12].

Chapter 2

LITERATURE REVIEW

2.1. Natural Fibres

Natural fibres are subdivided based on their origins, coming from plants, animals or minerals. Generally, plant or vegetable fibres are used to reinforce plastics. Plant-fibres may include hairs (cotton, kapok), fibre-sheafs of dicotylic plants or vessel-sheafs of monocotylic plants, i.e. bast (flax, hemp, jute, ramie) and hard-fibres (sisal, henequen, coir).

Tanzania and Brazil are the two largest producers of sisal. Henequen is produced in Mexico, abaca and hemp in the Philippines. The largest producers of jute are India, China and Bangladesh.

The availability of large qualities of such fibres with well-defined mechanical properties is a general prerequisite for the successful use of these materials and the lack of this is one of the drawbacks at the moment. Additionally for several more technical orientated applications, the fibres have to be specially prepared or modified regarding:

- Homogenization of the fibre's properties,
- Degrees of elementarization and degumming,
- Degrees of polymerization and crystallization,
- Good adhesion between fibre and matrix,
- Moisture repellence, and
- Flame retardant properties.

2.2. Natural fibres and their chemical compositions

Climatic conditions, age and the digestion process influences not only the structure of fibres but also the chemical composition. Component mean values of plant-fibres are shown in Table 2.1. With the exception of cotton, the components of natural fibres are cellulose, hemi-cellulose, lignin, pectin, waxes and water soluble substances, with cellulose, hemi-cellulose and lignin as the basic components with regard to the physical properties of the fibres.

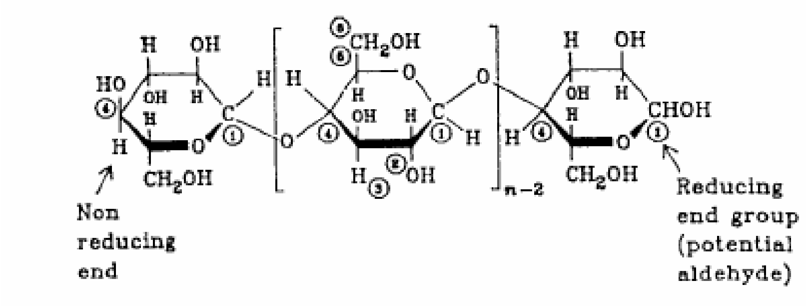
Table 2.1: Composition of different cellulose based natural fibres

	Cotton	Jute	Flax	Ramie	Sisal
Cellulose	82.7	64.4	64.1	68.6	65.8
Hemi-cellulose	5.7	12.0	16.7	13.1	12.0
Pektin	5.7	0.2	1.8	1.9	0.8
Lignin	-	11.8	2.0	0.6	9.9
Water soluble	1.0	1.1	3.9	5.5	1.2
Wax	0.6	0.5	1.5	0.3	0.3
Water	10.0	10.0	10.0	10.0	10.0

2.2.1. Cellulose

Cellulose is the essential component of all plant-fibres. In 1838, Anselme Payen suggested that the cell walls of large numbers of plants consist of the same substance, to which he gave the name cellulose.

It is generally accepted that cellulose is a linear condensation polymer consisting of -anhydroglucopyranose units (often abbreviated as anhydroglucose units or even as glucose units for convenience) joined together by β-1,4-glycosidic bonds. It is thus a 1,4-β- -glucan. The pyranose rings are in the 4C1 conformation, which means that the –CH2OH and –OH groups, as well as the glycosidic bonds, are equatorial with respect to the mean planes of the rings. The Haworth projection formula of cellulose is given by.

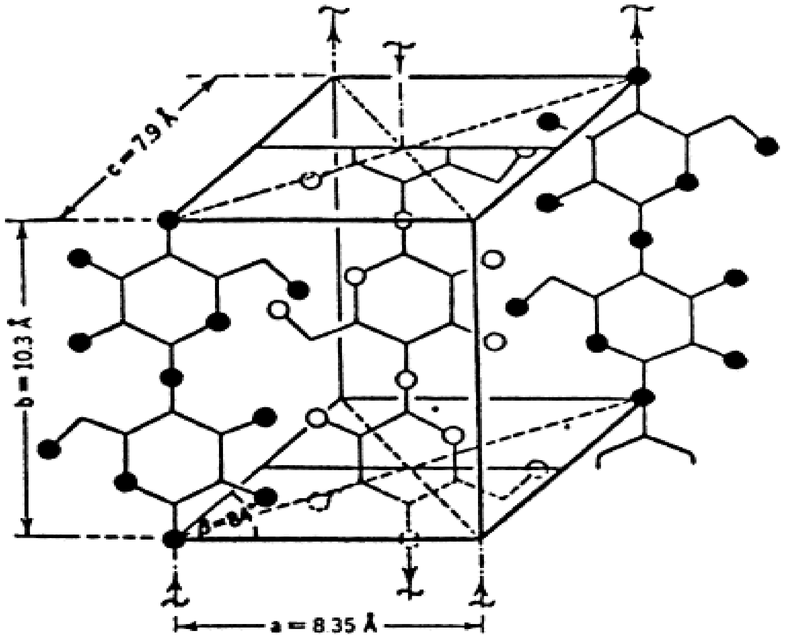


The molecular structure of cellulose is responsible for its supramolecular structure and this, in turn, determines many of its chemical and physical properties. In the fully extended molecule, adjacent chain units are orientated by their mean planes at an angle of 180° to each other. Thus, the repeating unit in cellulose is the anhydrocellulobiose unit and the number of repeating units per molecule is half the DP. This may be as high as 14 000 in native cellulose, but purification procedures usually reduce it to something in the order of 2500.

The degree of polymerization shows, that the length of the polymer chains varies depending on the type of natural fibre.

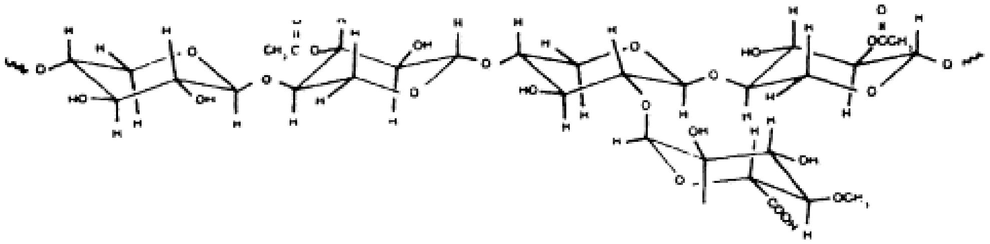
The mechanical properties of natural fibres depend on its cellulose type, because each type of cellulose has its own cell geometry and the geometrical conditions determine the mechanical properties.

Solid cellulose forms a microcrystalline structure with regions of high order, i.e. crystalline regions, and regions of low order, i.e. amorphous regions. Naturally occurring cellulose (cellulose I) crystallizes in monoclinic sphenodic structures. The molecular chains are orientated in fibre direction:

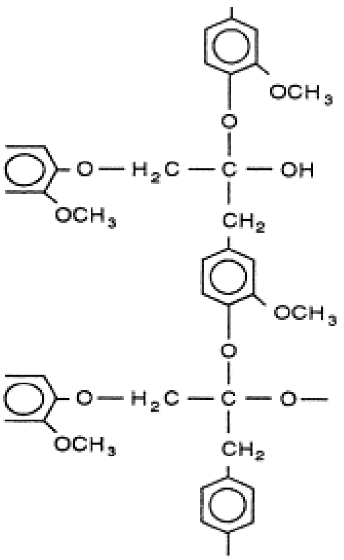


2.2.2. Further components

Hemicellulose is not a form of cellulose at all and the name is an unfortunate one. They comprise a group of polysaccharides (excluding pectin) that remains associated with the cellulose after lignin has been removed. The hemicellulose differs from cellulose in three important aspects. In the first place they contain several different sugar units whereas cellulose contains only 1,4-β- -glucopyranose units. Secondly they exhibit a considerable degree of chain branching, whereas cellulose is a strictly linear polymer. Thirdly, the degree of polymerization of native cellulose is ten to one hundred times higher than that of hemicellulose. Unlike cellulose, the constituents of hemicellulose differ from plant to plant.



Lignins are complex hydrocarbon polymer with both aliphatic and aromatic constituents.



Their chief monomer units are various ring-substituted phenyl-propanes linked together in ways, which are still not fully understood. Structural details differ from one source to another.

The mechanical properties are lower than those of cellulose. At the value of 4 GPa, the mechanical properties of isotropic lignin are distinctly lower than those of cellulose.

Pectin is a collective name for heteropolysaccharides, which consist essentially of polygalacturon acid. Pectin is soluble in water only after a partial neutralization with alkali or ammonium hydroxide.

Waxes make up the part of the fibres, which can be extracted with organic solutions. These waxy materials consist of different types of alcohols, which are insoluble in water as well as in several acids (palmitic acid, oleaginous acid, stearic acid).

2.3. Mechanical properties of natural fibres

Natural fibres are in general suitable to reinforce plastics (thermosets as well as thermoplastics) due to their relative high strength and stiffness and low density (Table 3). The characteristic values for flax and soft-wood-kraft-fibres reach levels close to the values for glass-fibres, types E ("E" because of their early use in electronic applications and today the most typically used glass-fibres reinforcing plastics in non-aggressive media). Nevertheless and also obvious in*(Table 3)*, the range of the characteristic values, as one of the drawbacks for all natural products, is remarkably higher than those of glass-fibres, which can be explained by differences in fibre structure due to the overall environmental conditions during growth. Natural fibres can be processed in different ways to yield reinforcing elements having different mechanical properties. The elastic modulus of bulk natural fibres such as wood is about 10 GPa. Cellulose fibre with moduli up to 40 GPa can be separated from wood, for instance, by chemical pulping processes. Such fibres can be further subdivided by hydrolysis followed by mechanical disintegration into microfibrils with an elastic modulus of 70 GPa. Theoretical calculations of the elastic moduli of cellulose chains have given values of up to 250 GPa, however, there is no technology available to separate these from microfibrils.

Table 2.2: Mechanical properties of natural fibres as compared to conventional reinforcing fibres

Fibre	Density(g/cm3)	Elongation (%)	Tensile strength(MPa)	Young's modulus (GPa)
Cotton	1.5-1.6	7.0-8.0	287-597	5.5-12.6
Jute	1.3	1.5-1.8	393-773	26.5
Flax	1.5	2.7-3.2	345-1035	27.6
Hemp	-	1.6	690	-
Ramie	-	3.6-3.8	400-938	61.4-128
Sisal	1.5	2.02.5	511-635	9.4-22.0
Coir	1.2	30.0	175	4.0-6.0
Viscose(cord)	-	11.4	593	11.0
Soft wood kraft	1.5	-	1000	40.0
E-glass	2.5	2.5	2000-3500	70.0
S-glass	2.5	2.8	4570	86.0

Aramide(normal)	1.4	3.3-3.7	3000-3150	63.0-67.0
Carbon(standard)	1.4	1.4-1.8	4000	230.0-240.0

As in the case with glass-fibres, the tensile strength of natural fibres also depends on the test length of the specimens which is of main importance regarding reinforcing efficiency. The actual tensile strength of a single fibre is typically given for a test length of zero as in the case for glass-fibres. The tensile strength of flax-fibres is significantly more dependent on the length of the fibre than for the case of glass-fibres. Compared to this, the tensile strength of pineapple-fibres is less dependent on the length, while the scatter of the measured values for both is located mainly in the range of the standard deviation. This dependence, strength vs. test length, could be seen as degree or the homogenity or amounts of defects of a fibre or a material in general.

Hydrophilic nature is a major problem for all cellulose-fibres if used as reinforcement in plastics. The moisture content of the fibres, dependent on content of non-crystalline parts and void content of the fibre, amounts up to 10 wt. % under standard conditions. The hydrophilic nature of natural fibres influences the overall mechanical properties as well as other physical properties of the fibre itself.

2.4. Jute fibres

2.4.1. Properties of jute fibres

The need for using jute fibres in place of the traditional glass fibre partly or fully as reinforcing agents in composites stems from its:

- Lower specific gravity (1.29) and higher specific modulus (40 GPa) of jute compared with those of glass (2.5 & 30 GPa respectively),
- Jute fibre is 100% bio-degradable.
- It has high tensile strength, low extensibility, and ensures better breathability of fabrics,
- It has good insulating and antistatic properties, as well as having low thermal conductivity.

2.4.2. Advantages of jute fibres

Apart from much lower cost and renewable nature of jute, much lower energy requirement for the production of jute (only 2% of that for glass) makes it attractive as a reinforcing fibre in composites.

Jute fibre is recyclable and thus environment friendly starting from the seed to expired fibre, as the expired fibres can be recycled more than once.

Jute fibres are easily available and these are environment friendly.

2.4.3. Uses of jute fibres

Jute has entered various diversified sectors, where natural fibres are gradually becoming better substitution. Among these industries are paper, celluloid products (films), nonwoven textiles (for car interiors and other uses), composites (pseudo-wood), and geotextiles.

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, post-boxes, roof tiles, grain storage silos, panels for partition & 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 such as floods, cyclones, earthquakes, etc.

2.4.4. Limitation of jute fibre composites

Unfortunately, all mechanical and other physical properties of natural fibres are influenced by their growing conditions, fibre processing technique and, as for other fibre types, by the fineness of the fibre and sample test-length.

Although, as with most of the other plant-based natural fibres, cellulose forms the main structural component of jute, the non-cellulosic components e.g., lignin and hemicellulose, also play an important part in determining the characteristic properties of the fibres. Jute hemicellulose, which is thought to consist principally of xylan, polyuronide and hexosan, has been shown to be very sensitive to the action of caustic soda, which exerts only a slight effect on lignin or α-cellulose.

There is, however, a major drawback associated with the application of jute fibres for reinforcement of resin matrices. Due to presence of hydroxy and other polar groups in various constituents of jute fibre, the moisture uptake is high (approx. 12.5% at 65% relative humidity & 20^{o} C) by dry fibre and 14.6% by wet fibre. All this leads to (i) poor wettability with resin and (ii) weak interfacial bonding between jute fibre and the relatively more hydrophobic matrices. Environmental performance of such composites is generally poor due to delamination under humid conditions. With increase in relative humidity upto 70%, the tenacity and Young's modulus of jute increases but beyond 70%, a decrease is observed. To reduce the moisture regain property of jute fibre, it is essential to pre-treat the fibre so that the moisture absorption is reduced and the wettability of the resin is improved.

A recent paper on the alkali treatment of jute fibres reports on the removal of lignin and hemicellulose which affects the tensile characteristics of the fibres. When hemicelluloses are removed, the interfibrillar region is likely to be less dense and less rigid and thereby makes the fibrils more capable of rearranging themselves along the direction of tensile deformation. When jute fibres are stretched, such rearrangements among the fibrils would result in better load sharing and hence in higher stress development in the fibre. On the other hand, softening of the inter-fibrillar matrix adversely affects the stress transfer between the fibrils and, thereby, the overall stress development in the fibre under tensile deformation. As lignin is gradually removed, the middle lamella joining the ultimate cells is expected to be more plastic as well as homogeneous owing to the gradual elimination of microvoids, while the ultimate cells themselves are only slightly affected.

Furthermore, for coir and flax fibres some authors report changes in the crystallinity as a result of alkali treatment. The increase in the percentage crystallinity index of alkali-treated fibres occurs because of the removal of the cementing materials, which leads to better packing of the cellulose chains. In addition, treatment with NaOH leads to a decrease in the spiral angle and an increase in molecular orientation. A fair amount of randomness is introduced in the orientation of the crystallites by the removal of noncellulosic matter. The elastic moduli of fibres, for instance, are expected to increase with increasing degree of molecular orientation. In addition to the modification of orientation and the consolidation of weak points, other important factors with regard to the mechanical properties could be the crystallite length and degree of crystallinity as well as the removal of fractions of cellulose of a very low degree of polymerization.

2.5. Methods for surface modification of natural fibres

The quality of the fibre–matrix interface is significant for the application of natural fibres as reinforcement fibres for plastics. Physical and chemical methods can be used to optimize this interface. These modification methods are of different efficiency for the adhesion between matrix and fibre.

2.5.1. Physical methods

Reinforcing fibres can be modified by physical and chemical methods. Physical methods, such as stretching, calandering, thermotreatment, and the production of hybrid yarns do not change the chemical composition of the fibres. Physical treatments change structural and surface properties of the fibre and thereby influence the mechanical bondings to polymers.

Electric discharge (corona, cold plasma) is another way of physical treatment. Corona treatment is one of the most interesting techniques for surface oxidation activation. This process changes the surface energy of the cellulose fibres and in case of wood surface activation increases the amount of aldehyde groups.

The same effects are reached by cold plasma treatment. Depending on type and nature of the used gases, a variety of surface modification could be achieved. Surface crosslinkings could be introduced, surface energy could be increased or decreased, reactive free radicals and groups could be produced.

An old method of cellulose fibre modification is mercerization, it has been widely used on cotton textiles. Mercerization is an alkali treatment of cellulose fibres, it depends on the type and concentration of the alkaline solution, its temperature, time of treatment, tension of the material as well as on the additives. At present there is a tendency to use mercerization on natural fibres as well. Optimal conditions of mercerization ensures the improvement of the tensile properties and absorption characteristics, which are important in the composing process.

2.5.2. Chemical methods

Strongly polarized cellulose fibres are inherently incompatible with hydrophobic polymers. When two materials are incompatible, it is often possible to bring about compatibility by introducing a third material, which has properties intermediate between those of the other two. There are several mechanisms of coupling in materials:

- **Weak boundary layers** – coupling agents eliminate weak boundary layers,
- **Deformable layers** – coupling agents produce a tough, flexible layer,
- **Restrained layers** – coupling agents develop a highly crosslinked interphase region, with a modulus intermediate between that of substrate and of the polymer,
- **Wettability** – coupling agents improve the wetting between polymer and substrate (critical surface tension factor),
- **Chemical bonding** – coupling agents form covalent bonds with both materials, and
- **Acid–base effect** – coupling agents alter acidity of substrate surface.

The development of a definitive theory for the mechanism of bonding by coupling agents in composites is a complex problem. The main chemical bonding theory alone is not sufficient. So the consideration of other concepts appears to be necessary, which include the morphology of the interphase, the acid–base reactions at the interface, surface energy and the wetting phenomena.

2.5.2.1. Change of surface tension

The surface energy of fibres is closely related to the hydrophility of the fibre.Some investigations are concerned with methods to decrease hydrophility. The modification of wood-cellulose fibres with stearic acid hydrophobizes those fibres and improves their dispersion in polypropylene. As can be observed in jute reinforced unsaturated polyester resin composites, treatment with polyvinylacetate increases the mechanical properties and moisture repellence.

Silane coupling agents may contribute hydrophilic properties to the interface. Especially when amino-functional silanes, such as epoxies and urethanes silanes, are used as primers for reactive polymers. The primer may supply much more amine functionality than can possibly react with the resin at the interphase. Those amines which could not react, are hydrophilic and therefore responsible for the poor water resistance of bonds. An effective way to use hydrophilic silanes is to blend them with hydrophobic silanes such as pheniltrimethoxysilane. Mixed siloxane primers also have an improved thermal stability, which is typical for aromatic silicones.

2.5.2.2. Impregnation of fibres

A better combination of fibre and polymer is achieved by impregnation of the reinforcing fabrics with polymer matrixes compatible to the polymer. For this purpose polymer solutions or dispersions of low viscosity are used. For a number of interesting polymers, the lack of solvents limits the use of the method of impregnation. When cellulose fibres are impregnated with a butyl benzyl phthalate plastified polyvinilchloride (PVC) dispersion, excellent partitions can be achieved in polystyrene (PS). This significantly lowers the viscosity of the compound and of the plasticator and results in co-solvent action for both PS and PVC.

2.5.2.3. Chemical coupling

An important chemical modification method is the chemical coupling method, which improves the interfacial adhesion. The fibre surface is treated with a compound, which forms a bridge of chemical bonds between fibre and matrix.

2.5.2.4. Graft copolymerization

An effective method of chemical modification of natural fibres is graft copolymerization. This reaction is initiated by free radicals of the cellulose molecule. The cellulose is treated with an aqueous solution with selected ions and is exposed to a high energy radiation. Then the cellulose molecule cracks and radicals are formed. Afterwards the radical sites of the cellulose are treated with a suitable solution (compatible with the polymer matrix), for example vinyl monomer, acrylonitrile, methyl methacrylate, polystyrene. The resulting co-polymer possesses properties characteristic of both, fibrous cellulose and grafted polymer.

After this treatment the surface energy of the fibres is increased to a level much closer to the surface energy of the matrix. Thus, a better wettability and a higher interfacial adhesion is obtained. The polypropylene (PP) chain permits segmental crystallization and cohesive coupling between the modified fibre and the PP matrix.The graft copolymerization method is effective, but complex.

2.5.3. Alkali treatment of natural fibres

The effects of mercerization on the suitability to mechanical treatment, notable tensile strength, of cotton fibres have been extensively studied. The standard definition of mercerization proposed by ASTM D 1965 is: the process of subjecting a vegetable fibre to an interaction with a fairly concentrated aqueous solution of a strong base, to produce great swelling with resultant changes in the fine structure, dimension, morphology and mechanical properties.

As discussed earlier cellulose forms the main structural component of vegetable natural fibres, the non-cellulosic components, e.g. lignin and hemicellulose, also play an important part in the characteristic properties of the fibres. Hemicellulose, which is thought to consist principally of xylan, polyuronide and hexosan, has shown to be very sensitive to the action of caustic soda, which exerts only a slight effect on lignin or α-cellulose. Later studies about the alkali treatment of jute-fibres, for instance, reports about the removal of lignin and hemicellulose which affects the tensile characteristics of the fibres. When the hemicelluloses are removed, the interfibrillar region is likely to be less dense and less rigid and thereby makes the fibrils more capable of rearranging themselves along the direction of tensile deformation. When natural fibres are stretched, such rearrangements amongst the fibrils would result in better load sharing by them and hence result in higher stress development in the fibre. In contrast, softening of the inter-fibrillar matrix adversely affects the stress transfer between the fibril and, thereby, the overall stress development in the fibre under tensile deformation. As lignin is removed gradually, the middle lamella joining the ultimate cells is expected to be more plastic as well as homogeneous due to the gradual elimination of microvoids, while the ultimate cells themselves are effected only slightly.

Further, some authors reported about changes in the crystallinity through alkaline treatment on coir and flax fibres. The increase in the percentage crystallinity index of alkali treated fibres occurs because of the removal on the cementing materials, which leads to a better packing of cellulose chains. Additionally, treatment with NaOH leads to a decrease in the spiral angle, i.e. closer to fibre axis, and increase in molecular orientation. A fair amount of randomness is introduced in the orientation of the crystallites due to the removal of non-cellulosic matter. The elastic moduli of fibres, for instance, are expected to increase with increasing degree of molecular orientation. Well oriented cellulosic fibres such as flax have much higher Young's modulus than fibres with medium orientation, such as cotton. In addition to the modification of orientation and the consolidation of weak points, other important factors with regard to the mechanical properties could be the crystallite length and degree of crystallinity as well as the removal of fractions of cellulose at a very low degree of polymerization.

Chapter 3

EXPERIMENTAL WORK

3.1. Alkali treatment of jute fabrics

- The jute fabrics were cut to 17x17 sq.cm and were soaked in a 5% NaOH solution at 30°C maintaining a liquor ratio of 15 : 1. The initial weight of fabric pieces(W1) was noted.
- The fabrics were kept immersed in the alkali solution for 1,2, 4, 6 and 8 h. The fabrics were then washed several times with fresh water to remove any NaOH sticking to the fibre surface.
- Subsequently neutralized with dilute acetic acid.
- Finally washed with distilled water.
- Then the fibres were dried at room temperature for 48 h followed by oven drying at 100°C for 2 h.
- The fabrics were finally once again weighed(W2)
- Weight loss was calculated

% Weight loss = $[(W1 - W2)/W1]$ x 100

3.2. Preparation of composite

By hand laying technique:

- Resin=60% of fabric weight
- Hardener=2.6% of resin weight
- Accelerator=2% of resin weight

The mixture of polyester resin with the given weight %s of hardener and accelerator were applied in between treated jute fabric pieces to get alternate layers of resin matrix and jute fabric reinforcement.

- The laminate prepared was subjected to a load of 2 ton in the HYDRAULIC PRESS.
- Then it was left under the load for 15 min so that uniform distribution of resin mixture was ensured.
- The green composite then is left out to atmosphere for 48 h under a load of 8kg for avoiding relaxation.
- The specimens were then cut from the composite for flexural tests and tensile test with the help of hacksaw.

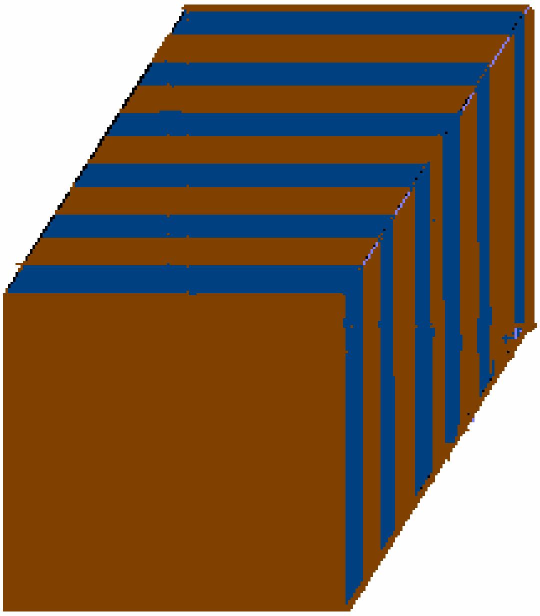


Fig. 3.1: Schematic representation of the laminate

3.3. Flexural test

Three-point bend tests were performed in Instron 1195 machine in accordance with ASTM D790 to measure the flexural strength of the composites. The breaking energy was calculated from the area under the load/displacement curve to the break point and the toughness was obtained by dividing the energy to break by the volume of the test specimen. The flexural interlaminar shear strength (LSS) of the composites which is the maximum shear stress that a material can withstand before it ruptures, was calculated in the stress units based on the area of the sheared edge.

➢ Dimensions of Test specimens were

- Length=16xthickness+12 mm,
- Width = 12.7 mm,
- Thickness = 4 - 5 mm
- Span length=70 mm,
- Crosshead speed=2mm/min

3-dimensional view of specimen

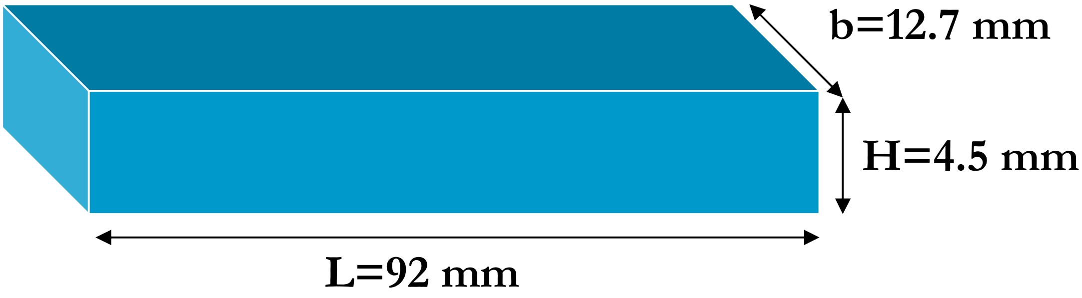
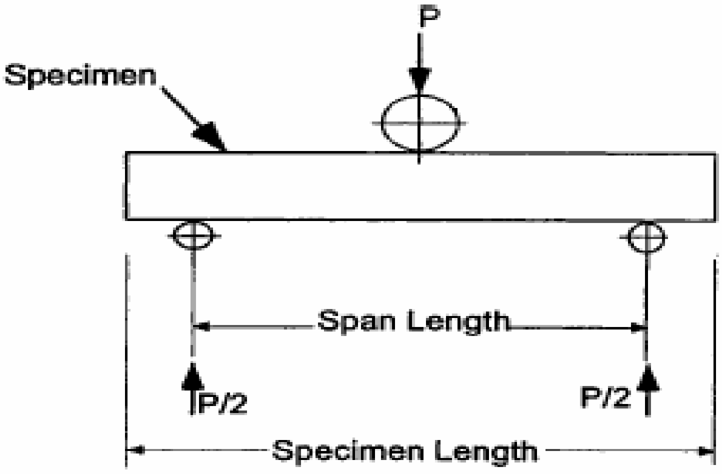


Fig 3.2: 3-dimensional view of specimen

Front view



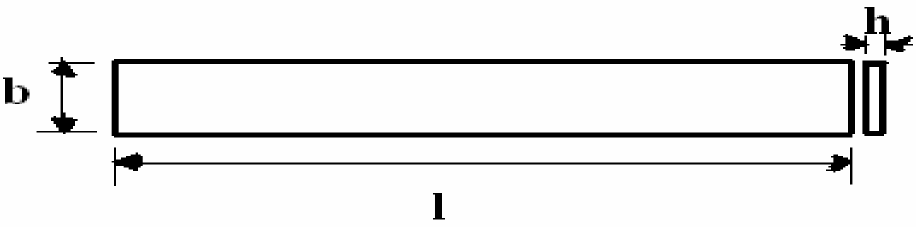


Fig 3.3: Front & Top View of the specimen

3.4. Strength measurements

- **Interlaminar shear strength** (ILSS): It is the maximum shear stress that a material can withstand before rupture.
- It was calculated on the stress units based on the area of the sheared edge

$ILSS = 0.75P/(b*h)$,

P-load, b-width & h-thickness

- **Bending Strength** = $3PL/2bh^2$

L-span length, P-load, b-width & h-thickness

3.5. Tensile test

Among the many mechanical properties of plastic as well as composite materials, tensile properties are probably the most frequently considered, evaluated, and used throughout the industry.

These properties are an important indicator of the material's behavior under loading in tension.

Tensile testing provides these useful data: tensile yield strength, tensile strength at break (ultimate tensile strength), tensile modulus (Young's modulus), and elongation at yield and break. The tensile strength (σ) is given by

$$\sigma = F/ b\, h$$

Where

F: load

b: width of the sample

h: thickness of the sample

Strain or elongation is defined as:

$$\varepsilon = l_o / \Delta l$$

Where

Δl: is the extension

l_o: the initial gauge length

The Young's modulus in tension (Et) is the slope of the stress vs. strain curve evaluated at small strains, where the response is linear.

Tensile tests were performed in an Instron 1195 machine in accordance with ASTM D3039 to measure the UTS, Breaking energy, Yield stress of the composites.

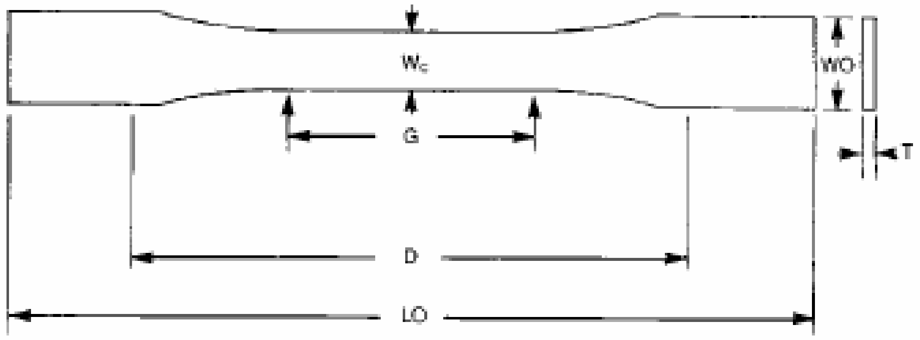


Fig 3.4: Specimen for tensile testing

Wc- Width of narrow section = 15mm,

Wo- Width overall = 25mm,

T- Thickness = 3 – 4mm,

G- Gauge length = 70mm,

Lo- Length overall = 120mm,

D- Distance between grips = 80mm.

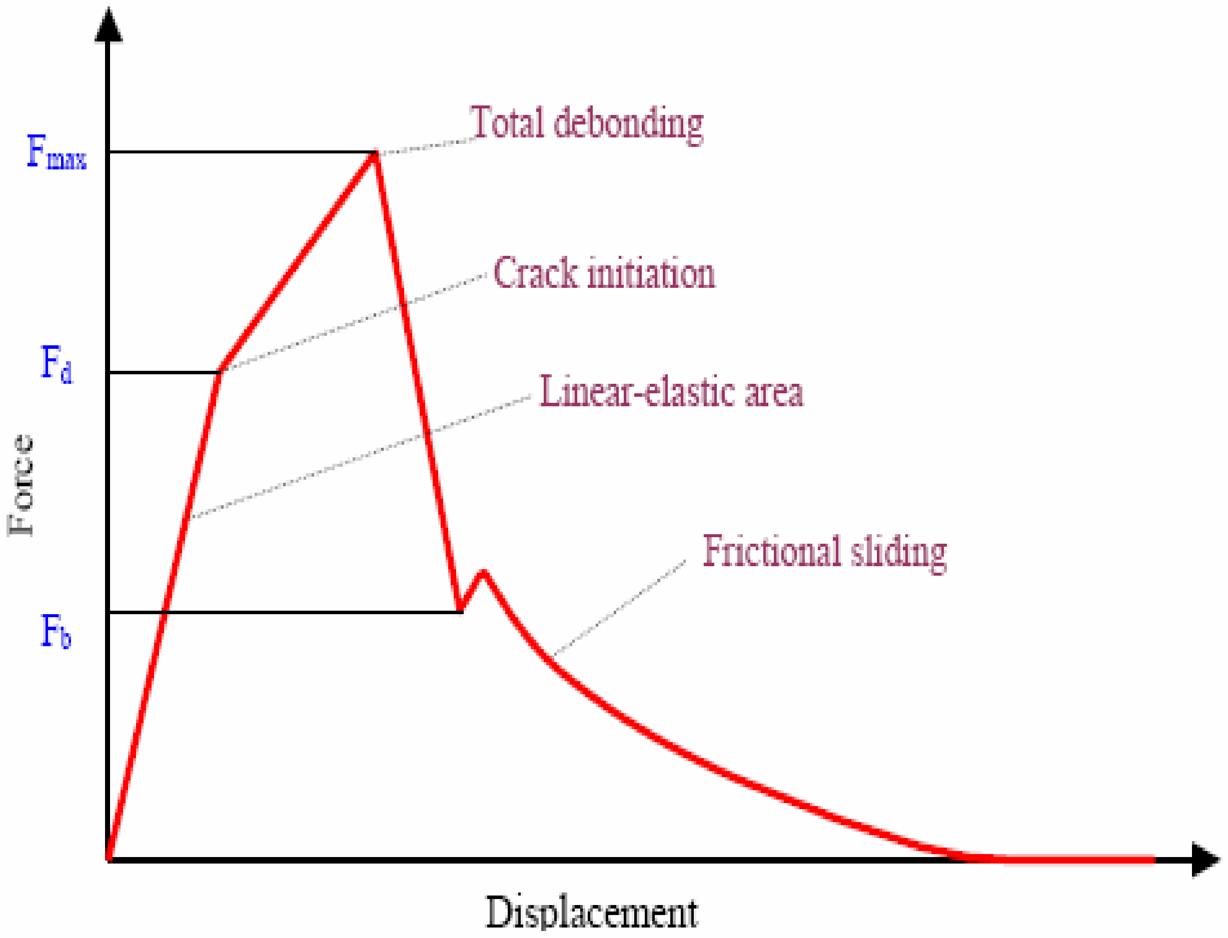


Fig. 3.5: A typical plot of pull-out force versus displacement

Chapter 4

RESULTS AND DISCUSSION

Loss in weight was observed after the alkali treatment of the fibres, due to heavy dissolution of the hemicellulose content. The loss occurred primarily within 1 h of treatment. This had resulted in the drop of linear density of the fibres when treated between 1 and 6 h due to the creation of voids in the fibre structure, the strands became well separated and dispersed. The crystallinity of the fibres was observed to have increased only after 6 h treatment. It was imperative that the fibres became stiff and brittle on account of its high strength and low extensibility [21].

The NaOH treatment of jute fibres leads to changes in shrinkage or mechanical properties like tensile modulus and tensile strength (under isometric conditions) dependent on the NaOH concentration. NaOH concentration and allowed shrinkage during the alkali treatment are the most significant treatment parameters, as far as mechanical fibre properties are concerned. Other treatment parameters with comparatively smaller effects are temperature and time, if treatments are carried out for more than approximately 5 min. The 3-point bend test results for jute composites are presented in Table 4.1. For each data point, minimum five samples were tested and the value given is the mean of those results.

Table 4.1: Observations from 3-point bend test

Treatment Time (Hrs)	ILSS (MPa)	Bending strength (MPa)	Energy to yield point (J)
0	2.71	0.07619	0.4508
1	2.89	0.09204	0.4369
2	3.08	0.09250	0.5193
4	3.43	0.09717	0.5536
6	2.91	0.07345	0.3506
8	2.77	0.06932	0.3192

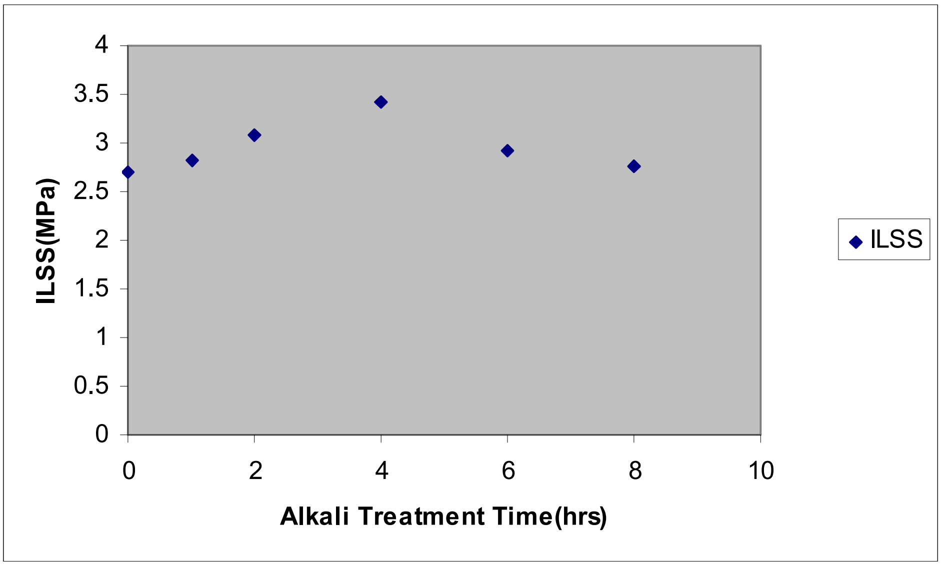


Fig. 4.1: Influence of the fibre treatments on Interlaminar shear strength (ILSS) of unidirectional jute/polyester composites.

The mechanical properties of composites prepared from Fibres treated with alkali (5% NaOH solution) are included. The composites prepared with fibres treated for 4 h showed maximum improvements. The ILSS of the composites after alkali treatment was 238.9 MPa in contrast to 199.1 MPa for the composites with untreated fibres. An improvement of 26.56% was measured. The improvements however, were 6.749% and 2.214% only for composites prepared with 2 and 8 h treated fibres respectively. The maximum improvements were with 4 h treated fibre composites at 40 wt% reinforcements. It has been reported that jute fibres exposed to longer time periods of alkali treatment (8 h) exhibited maximum strength properties, but the composites prepared with them showed lower strength values. In the present case the maximum composite strength was obtained from the 4 h treated fibres. The fibres became rigid and brittle owing to the development of crystallinity causing high strength and low extensibility [21].

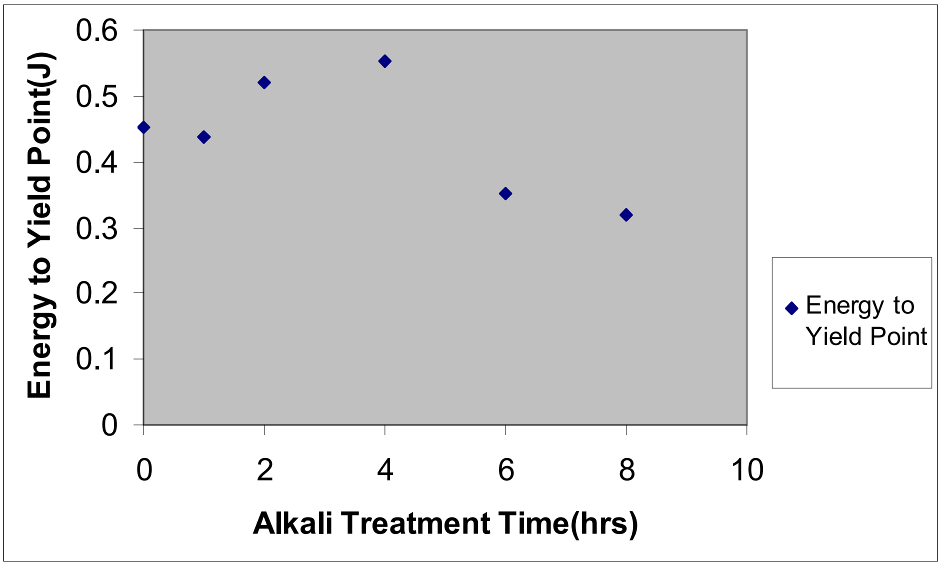


Fig. 4.2: Influence of the fibre treatments on Energy to Yield Point of unidirectional jute/polyester composites.

On application of stress, these fibres suffered breakage due to increased brittleness and could not take part in effective stress transfer at the interface, thus lowering the strength of the composites. A deflection at 4 h treatment was apparent imperative of the occurrence of two different failure modes before and after 4 h of NaOH treatment. Taking the strength values of fibres from Bledzki and Gassan (1999) the composite strengths were estimated applying the rule of mixtures.

This was compared with the experimentally obtained flexural strengths of the composites prepared with 4 h treated and untreated fibres. The theoretical estimation of strength and modulus from the rule of mixtures considers defect free fibres and composites.

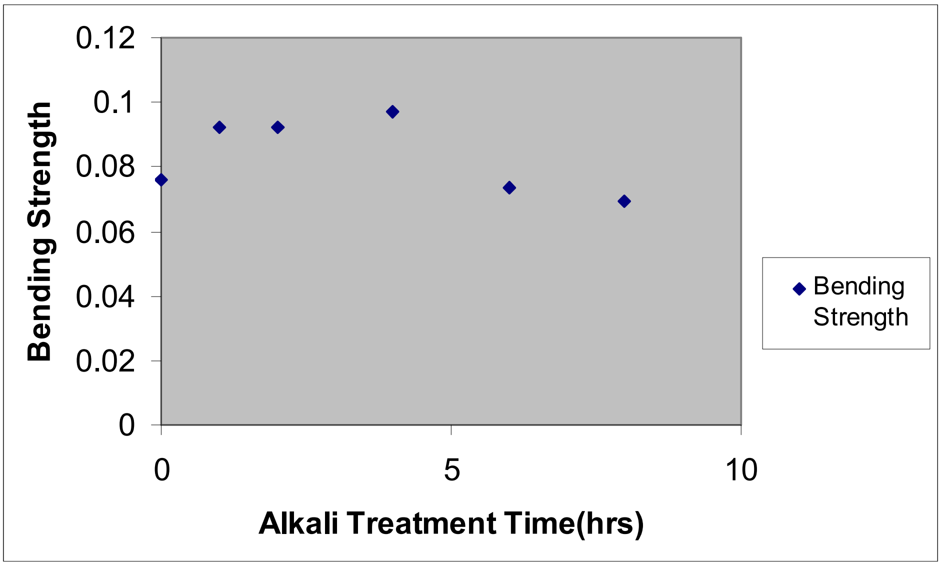


Fig. 4.3: Influence of the fibre treatments on Bending Strength of unidirectional jute/polyester composites.

4.1. Tensile test results:

Table 4.2: Observations from tensile testings

Treatment Time (Hrs)	UTS (MPa)	Young's Modulus	Stress at 0.2% Yield (MPa)	Stress at Break (MPa)	Energy to Yield Point (J)	Energy to Break Point (J)
0	11.49	2032.	11.08	7.39	0.214	0.3496
1	24.73	2892.	18.25	20.96	0.365	1.12
2	28.59	3308.	22.77	24.41	0.4498	0.9384
4	57.15	4334.	29.52	57.15	0.4189	1.979
6	26.99	3445.	18.91	23.34	0.3235	1.166
8	19.46	2567.	15.24	14.03	0.2937	0.7496

The influence of the alkaline treatment of jute fibres on the transverse tensile modulus and the transverse tensile strength of the unidirectional composites are shown in Fig-4.4.

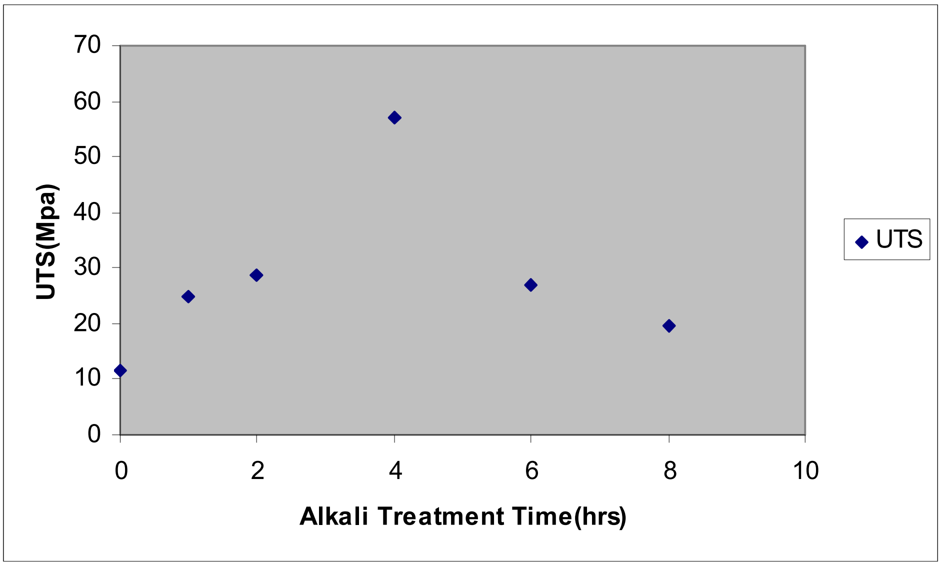


Fig. 4.4: Influence of the fibre treatments on the tensile strength of jute/polyester composites.

It is clearly observed that the NaOH treatment improves the tensile strength and Energy to Break Point. An improvement in the tensile strength of the 3 layer fibre composites was observed with increasing from 11.49 MPa to 57.15 MPa in the case of untreated fibre and fibre treatment by NaOH 5%, 4hours. This can be understood that the removal of the hemicellulose and a part of the lignin by alkali treatment can increase the interfacial adhesion between the matrix and NaOH treated fibre. However, at higher alkali treatment time (6hr,8hr), the tensile strength of the composites was observed to level off. It is possible that the fibres became rigid and somewhat brittle afterwards owing to the development of crystallinity causing high strength and low extensibility. On application of stress, these fibres suffered breakage due to increased brittleness and could not take part in effective stress transfer at the interface, thus lowering the strength of the composites. Therefore, the treatment condition of NaOH 5wt%, 4 hours was assumed the optimal for the treatment.

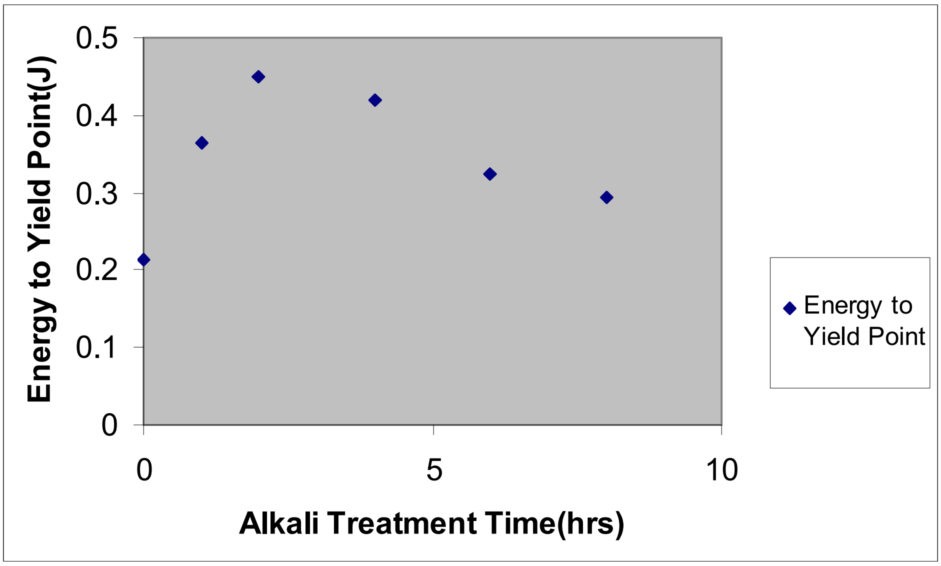


Fig. 4.5: Influence of the fibre treatments on Energy to Yield Point of unidirectional jute/polyester composites.

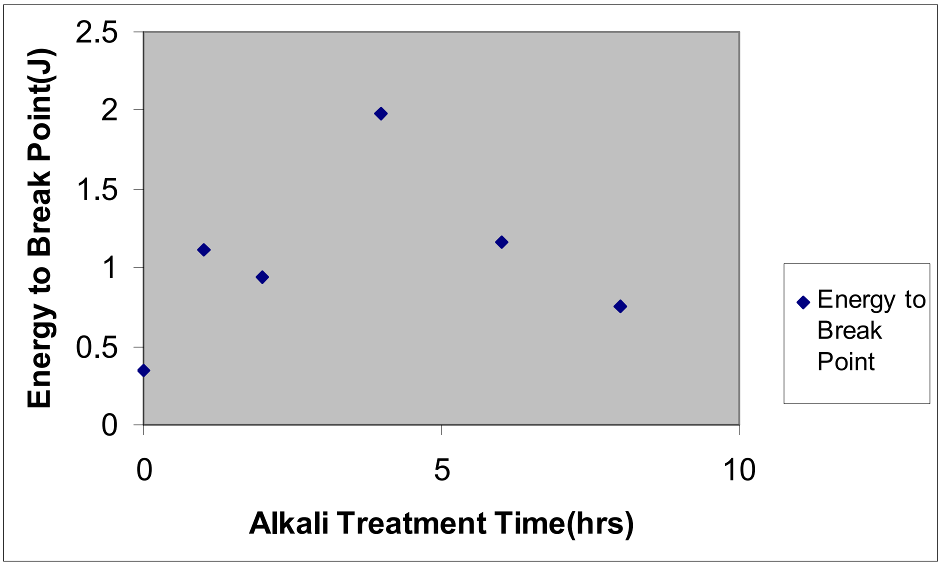


Fig. 4.6: Influence of the fibre treatments on Energy to Break Point of unidirectional jute/polyester composites.

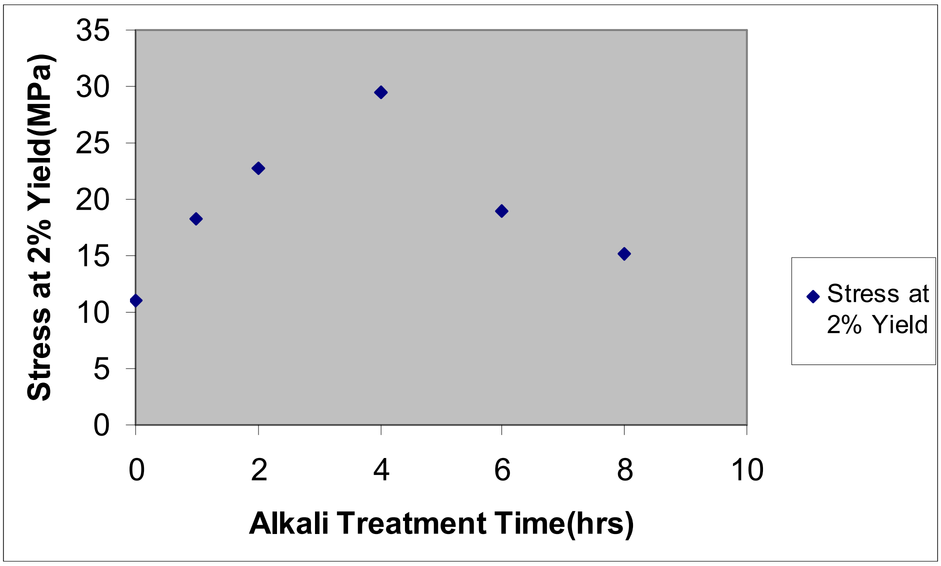


Fig. 4.7: Influence of the fibre treatments on Stress at 2% Yield of unidirectional jute/polyester composites.

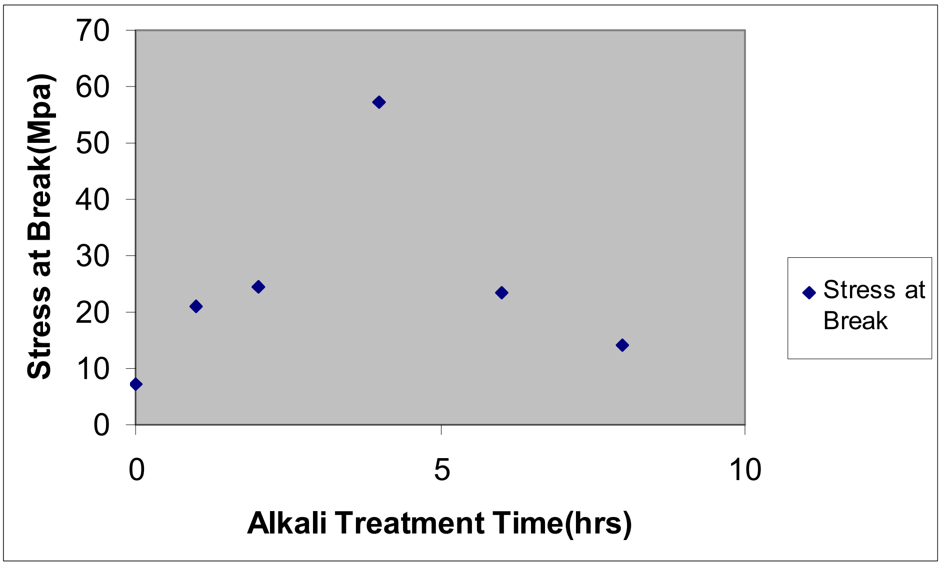


Fig. 4.8: Influence of the fibre treatments on Stress at break of unidirectional jute/polyester composites.

Observed changes in mechanical properties are possibly due to interacting factors such as [25]:

1. The rupture of alkali-sensitive bonds existing between the different components of the fibre as a result of swelling and partial removal of the hemicellulose: the fibre becomes more homogeneous through microvoid elimination, for instance, and the stress transfer between ultimate cells improves,
2. The formation of new hydrogen bonds between certain of the cellulose chains as a result of the removal of hemicellulose, which normally separates the cellulose chains: this may also occur as a result of the release of initial strains and subsequent re-adjustments to the chains after the intra-crystalline swelling action, also resulting in a probable change in the orientation of the non-crystalline cellulose,
3. Changes in parts of the crystalline cellulose, and
4. Changes in the orientation of the molecular chains, as detected for the jute fibres used

4.2. Factography

Morphological analysis of the fractured surface of the tested specimen were carried out using SEM. This is the most widely used surface analysis technique. High resolution SEM has proved an invaluable tool for studying surface topography and failure analysis. The technique enables qualitative three-dimensional (3-D) imaging of surface features, however, it does not easily lend itself to quantitative surface roughness characterization. This can be overcome by complementing SEM investigations with atomic force microscopy. In SEM, a highly focused scanning electron beam bombards the surface causing large numbers of secondary electrons to be generated, the intensity of which is governed by surface topography. The method is suitable for all materials, but nonconducting materials must be given a thin conductive coating sputtered gold, which can alter or mask the true surface morphology. The resolution of topographical features is approximately 5 nanometers. SEM is often used to survey a surface before more specialized techniques are employed.

Scanning electron microscopy analysis of fracture surfaces illustrates the improvement of the interaction between the fibre and matrix in polypropylene composites by alkali treatment.

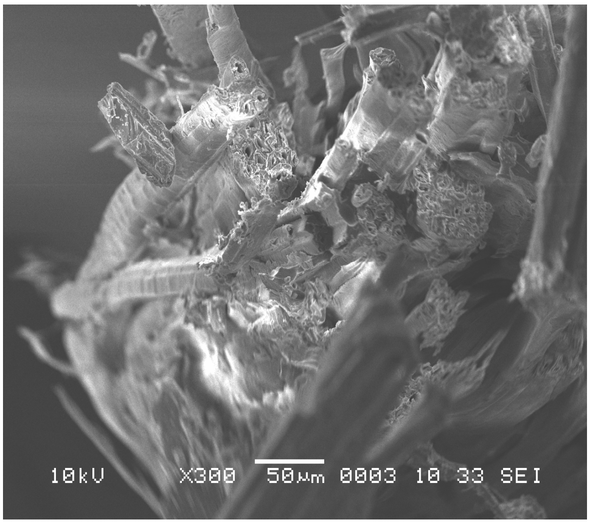


Fig. 4.9: SEM of untreated jute fibre composite at magnification 300X.

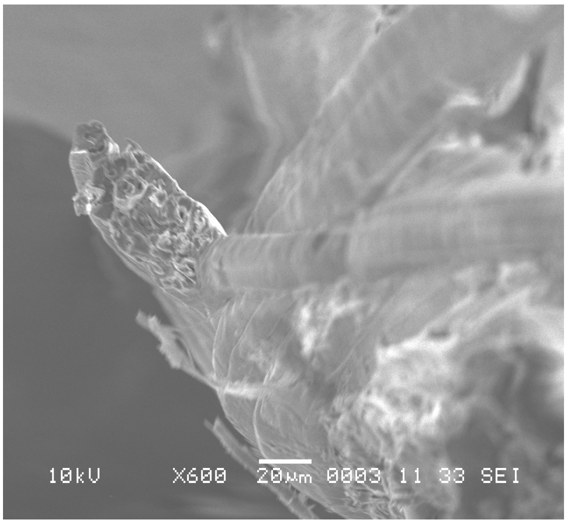


Fig. 4.10: SEM of untreated jute fibre composite at magnification 600X.

The figures 4.9 and 4.10 indicate that fibre breakage was the main reason for the low strength of the composite in untreated state. Observations at high magnifications indicate thin fibre bundle breakage (Fig-4.9) and shearing of a fibre (Fig-4.10).

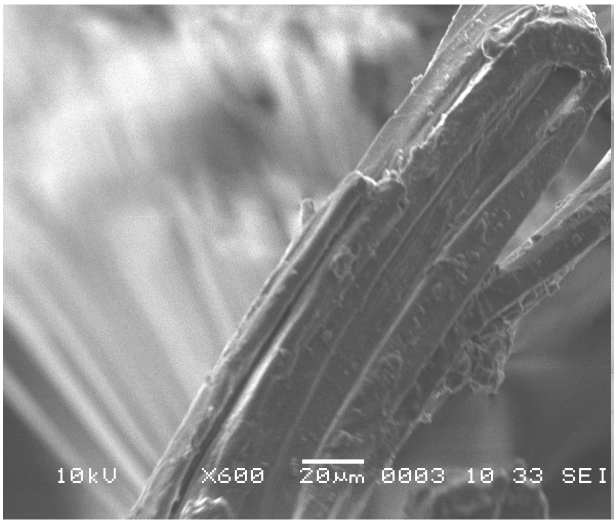


Fig. 4.11: SEM of untreated jute fibre composite at magnification 600X

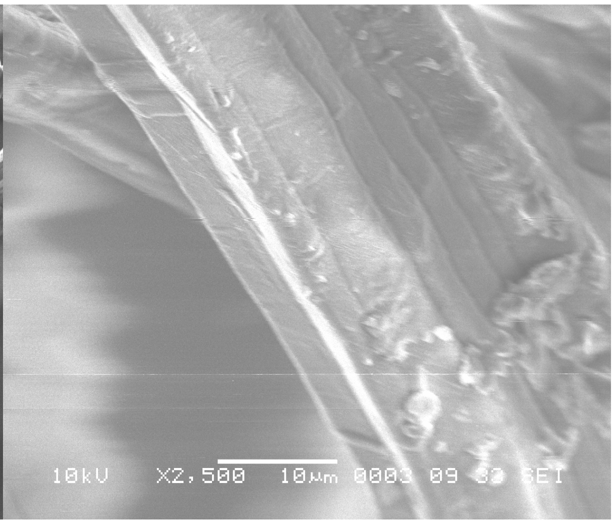


Fig. 4.12: SEM of untreated jute fibre composite at magnification 2500X

Fig. 4.13: SEM of 4h treated jute fibre composite at magnification 300X.

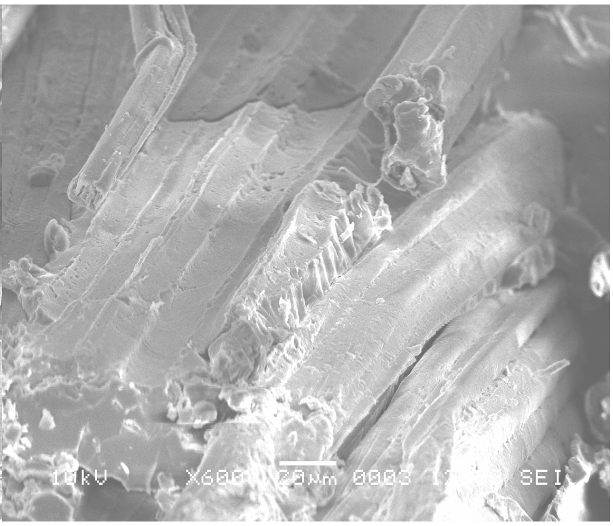


Fig. 4.14: SEM of 4h treated jute fibre composite at magnification 600X.

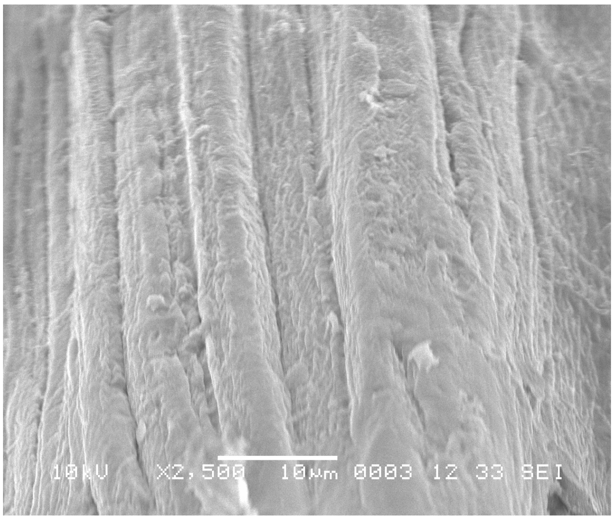


Fig. 4.15: SEM of 4h treated jute fibre composite at magnification 2500X

In case of SEM photographs observed for the fractured surfaces of 4h alkali treated composites only relatively lower amounts of fibre pullout was observed and discontinuity between fibre and matrix (Fig-4.13).at higher magnification levels we can clearly detect matrix cracking and some fibre pullout was observed (Fig-4.14).at a magnification of

2500X we can have a detailed structure of a fibre due to dissolution of cellulose and the individual fibres are clearly visible (Fig-4.15).

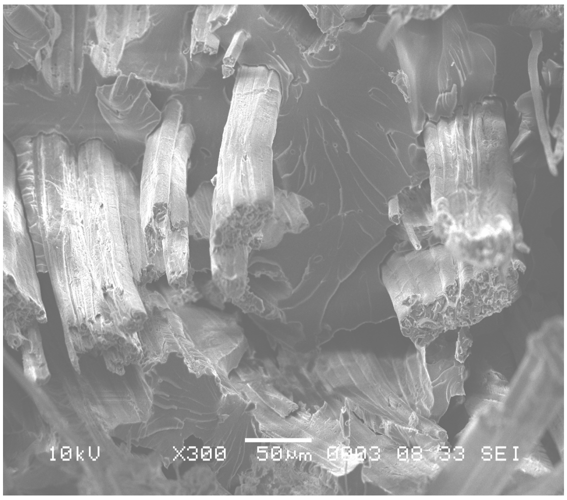


Fig. 4.16: SEM of 8h treated jute fibre composite at magnification 300X.

Fig. 4.17: SEM of 8h treated jute fibre composite at magnification 2500X.

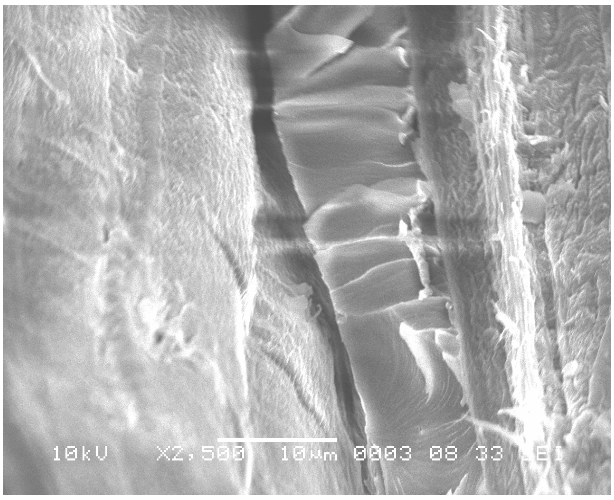


Fig. 4.18: SEM of 8h treated jute fibre composite at magnification 2500X.

In the factographs of fractured surfaces of 8-hr alkali treated composites large fibre pullout and damage of the interface is noticed indicating a weak interface and severe matrix cracking is revealed (Fig-4.16). higher magnifications have indicated fibre surface damage due to long exposure to alkali environment. This may be the result of loss of tensile strength and ILSS in composites prepared from treated jute fabrics.

Chapter 5

CONCLUSIONS AND REFERENCES

The following conclusions were drawn from the experiments conducted and the results obtained.

1. The loss in weight of the fabrics after the alkali treatment was observed, due to heavy dissolution of the hemicellulose content. The loss occurred primarily within 1 h of treatment.

2. The 3-point bend test results revealed that The composites prepared with fibres treated for 4 h showed maximum improvements. The ILSS of the composites showed an improvement of 26.5% with respected to composites prepared from untreated fabrics. In case of 8 h treated fibre composites the fibres became rigid and brittle owing to the development of crystallinity causing high strength and low extensibility.

3. It is clearly observed that the NaOH treatment improves the tensile strength and Energy to Break Point. An improvement in the tensile strength of the 3 layer fibre composites was observed with increasing from 11.49 MPa to 57.15 MPa in the case of untreated fibre and fibre treatment by NaOH 5%, 4hours. This can be understood that the removal of the hemicellulose and a part of the lignin by alkali treatment can increase the interfacial adhesion between the matrix and NaOH treated fibre.

4. Scanning electron microscopy analysis of fracture surfaces illustrates the improvement of the interaction between the fibre and matrix in polypropylene composites by alkali treatment. In case of SEM photographs observed for the fractured surfaces of 4h alkali treated composites only relatively lower amounts of fibre pullout was observed and discontinuity between fibre and matrix . In the factographs of fractured surfaces of 8-hr alkali treated composites large fibre pullout and damage of the interface is noticed indicating a weak interface.

5. A deflection at 4 h treatment was apparent imperative of the occurrence of two different failure modes before and after 4 h of NaOH treatment.

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