

**MODIFICATION OF JUTE FIBRE BY CHEMICAL
MEANS AND BLENDING OF THE MODIFIED JUTE
FIBRE WITH COTTON, RAYON, POLYESTER AND
SILK.**

**A DISSERTATION IN PARTIAL FULFILMENT
OF THE REQUIREMENTS FOR THE DEGREE
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To Whom It May Concern:

This is to certify that Md. Nurul Islam, Principal Scientific Officer, BCSIR Laboratories, Dhaka worked on "Modification of jute fibre by chemical means and blending of the modified jute fibre with cotton, rayon, polyester, and silk."



He has done the work himself under my supervision. He has not submitted this work anywhere else. His results are submitted in the form of dissertation of Doctor of Philosophy (Ph.D)



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April, 2003

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D E D I C A T E D
TO
MY BELOVED WIFE SAYEDA KHATUN
&
AFFECTIONATE DAUGHTERS LUBABA AND NABILA

400937



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ABSTRACT

Etherification of jute fibre with butyl chloride was carried out at 30⁰C (Temperature was 100⁰C when water was used as a solvent). Sodium hydroxide was choosen as a swelling agent for its economy and easy availability. The effects of solvents were studied. Petroleum ether was found better and hence it was used as a solvent in the present investigation. The degree of substitution, the percent elongation, bundle strength and percent moisture regain of the butyl substituted jute fibre were determined. It was observed that the bundle strength of butyl substituted jute fibre decreases than that of the control. Percent moisture regain of the butyl substituted jute fibre decreases with the increase of substitution. The extensibility of butyl substituted jute fibre increases up to about 10 times of its original value of 1% extention at break with the increase of substitution.

The blending of the etherified jute fibre with cotton, rayon, polyester and silk was another successful attempt. A blend of 50:50 for each fibre component was prepared with 50% of the etherified jute fibre respectively and 30 tex yarns from each blend was produced using the ring spinning frame. The physical properties of these yarns were determined with respect to the same numbered yarns of cotton. The 50% of the etherified jute fibre in the blends produced yarns having tenacities between [10, 9-11, 9 (g/tex)] which are very much comparable to tenacity 11 5(g/tex) of the same numbered cotton yarns. The breaking elongation

(6.2-6.4%), moduli [181-184 (g/tex)] and CSP (1605-1638) of the yarns from etherified jute/cotton and etherified jute/rayon in the ratio of 50:50 were almost similar to the respective properties [6.6%, 171 (g/tex) and 1694] of the same numbered cotton yarn. The breaking elongation of yarns from etherified jute/polyester and etherified jute/silk were much higher between (12.4-12.7%) which were due to highly extensible polyester and silk fibres in the combination.

Different fabrics samples were prepared by plain weaving with the blended yarns made of etherified jute fibre in the ratio of 50:50 with cotton, rayon and polyester. The geometrical properties of the fabrics from the blended yarns were favourably compared with those of the cotton fabrics made of yarns of same count (30 tex). The mechanical properties of these fabrics were determined to observe their service ability in practical usages and to ascertain their suitability as jute blended cotton and synthetic fabrics. The softness and handling characteristics represented by the bending length and flexural rigidity of blended fabrics were very much comparable to those of cotton fabrics with identical fabric structure. The results of the strength properties of the etherified jute blended fabrics have shown that the durability and serviceability of those fabrics under any sort of stress and deformation during use were not much less than the cotton fabrics. It has also been observed that the blending of the etherified jute with cotton or any other flexible fibre improved the draping properties of the cloths made there from and that these fabrics in actual use have gracious look and firmness almost to same extent of those draping properties of the cotton fabrics.

INTRODUCTION

PART-I

INTRODUCTION

1. GENERAL INTRODUCTION

1.1 Jute plants and jute fibres

Jute is called the golden fibre of Bangladesh. There are over thirty species of jute under the genus *Corchorus* [1] of Tiliaceae family. Among them *Corchorus capsularis* and *Corchorus olitorius* are grown abundantly in Bangladesh.

The jute plant is composed of two distinct parts- the outer fibrous bark and the inner stick. Jute fibre is produced in large quantity by retting the mature plants under clean water for 2-3 weeks. The fibre is then separated from the stick by hand, washed with sufficient water and stored after drying in the sun.

1.2 Jute as textile fibre source

Jute plant produces fibre which are woody and coarse in nature having been suitable for making cordage, ropes, camouflage nets etc. from the very dawn of the human civilization [2]. The muslin, the reputed

fine cloths of erstwhile Bengal and the noted “Patta” cloth of the ancient India were said to be made of jute fibre. These jute products were indigenously constructed from the fibre by rolling, plaiting, processing or weaving with the help of hands [3, 4]. Unfortunately, despite all of its potentialities, jute fibre being hard, brittle, unyielding in contrast to cotton or linen had been neglected as textile fibre source for long days.

In the early forties of the nineteenth century some Scottish Scientist at Dundee treated jute fibre with oil-water emulsion and developed softness in it, which reduces breakage and brittleness of the fibre during mechanical processings- Carding, drawing, spinning and weaving. Jute thus got recognition as a textile fibre and occupied almost the entire world market for coarse packing cloths like hessian and sacking, carpet backing clothes, carpets, mats etc.

It was a tremendous success in the textile field, because after a few years in the middle of that century, the world demand for jute and jute goods went up to about 3000 million pounds. The traditional flax fibre lost its European market to jute. The triumph of jute was due only to the exceptionally low price of it as raw materiel. Now, after about 160 years, the situation has been changed. This time, jute is to face serious competition from the synthetic polypropylene and other synthetic fibres and is now alarmingly losing its traditional market in the world.

1.3 Defects in jute products

The emulsion added on to jute acts not only as emollient but also changes partially the physico-chemical structure and properties of the fibre by the action of the water component of the emulsion, as it reduces the surface tension of water penetrating the capillary pores and pits of the fibre articulation. Water bonded with the fibre components dries out leaving the hatching oil. This residual oil is the major contaminant in the jute products and creates greater problems in addition to the natural and inherent defects. These defects in jute goods had so far been overlooked till coming of synthetic fibres in the early sixties of the nineteenth century. The users and buyers have now started complaining of the defects in jute goods. Some of the complaints are listed below.

- Fibre falls from jute products.
- Exudation of emulsion oil from jute products.
- Harshness and coarseness of yarn and fabires.
- Loss of resiliency of jute carpet in usage.
- Decline of fibre strength in usage.
- Browning/fading of Colour.
- Heavier product with less cover area.
- Contamination in material packed in jute goods.
- Shrinkage on wetting.
- Difficult to get fine spinning.
- Difficult to make versatile use.
- Bad smell from jute products due to rancidity of residual emulsion oil.

1.4 Importance of Jute in the economy of Bangladesh.

Jute is the main cash crop of Bangladesh. Here over 5 million people are one way or other dependent on jute from its cultivation to marketing in the form of raw jute and jute goods. About 7 million bales (1 bales = 180 kg) are produced by Bangladesh annually amounting over 50 percent of the world production. Bangladesh earns, despite depression in the international jute market, over 50 percent of her foreign exchange from jute alone. Besides Bangladesh other jute producing countries are India, Sri Lanka, Thailand, Nepal and China.

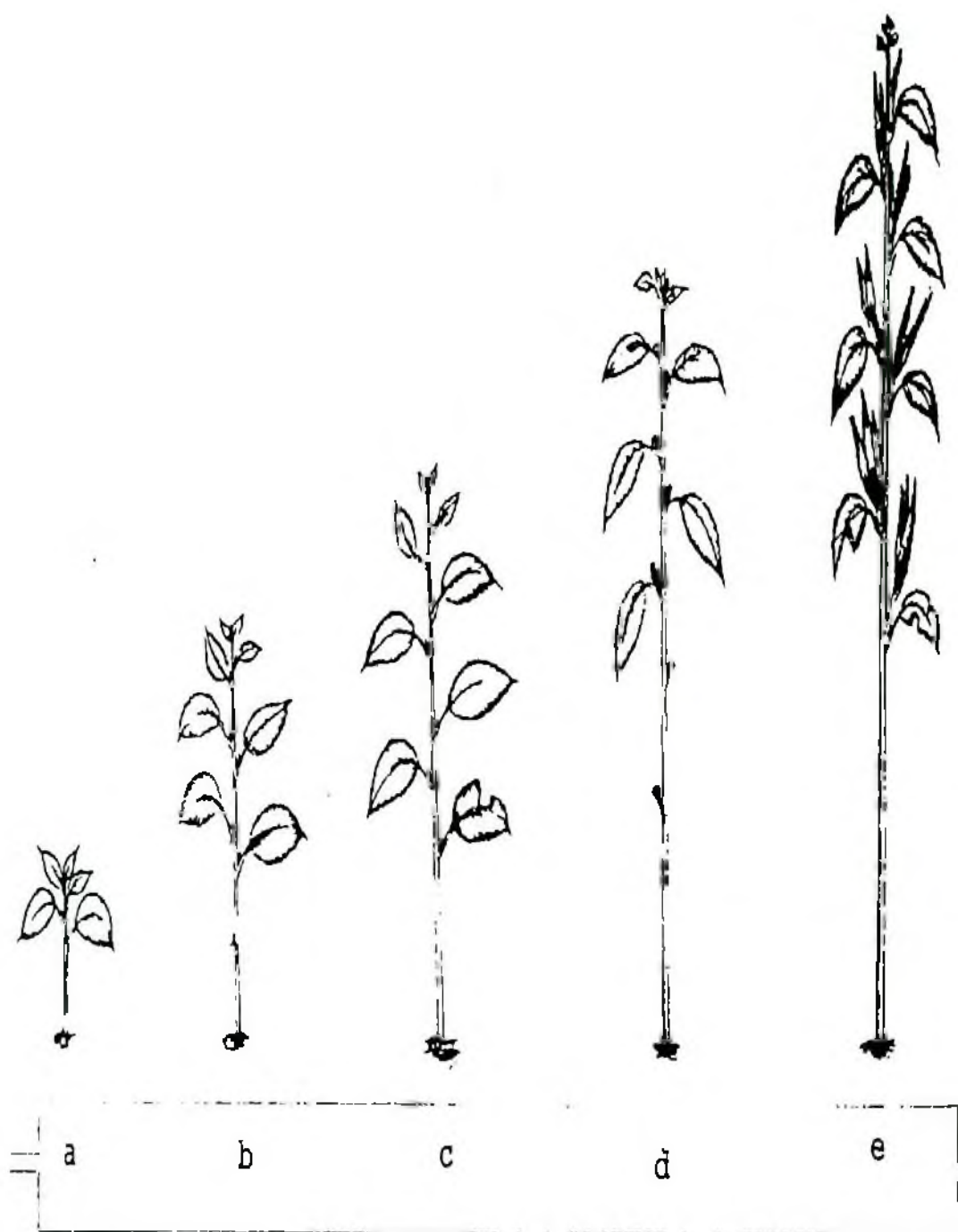
2 COMPLEXITY IN JUTE FIBRE

Scientist concerning to improve the defects in jute products to any possible extent need to realise the limitation imposed by the native properties of the fibre. Although jute is a fibrous bio-materials obtained from jute plants, it differs from cotton in the mode of occurrence and deviates from it and other vegetable fibres by its chemical composition, structure and properties. Cotton is constituted with 98% cellulose, but jute is compounded with cellulose, hemi-cellulose, lignin, pectic substances and fatty materials that make the fibres inconvenient for handling and making of fine and soft jute goods. The cotton fibre are separated from the seeds of cotton plant, to which they are attached, but the jute fibres are embedded in other tissues of the plant. Thus, the quality of the fibres depends to a greater extent on the method employed for their separation

from the neighboring tissues. Jute is not simple fibre like cotton. It forms as the bundle of fibres of multiple cells through a complex cytological process depending on the history of the plant and agronomical conditions. The morphology of the fibre is very complicated. The composition and characters of the components become more intricate as the fibres are developed and matured with the age of the plant. Here is described some salient features about the complexity in jute.

2.1 Agro-botanical

Two species of jute plants are grown, *Corchorus capsularis* and *Corchorus olitorius*, distinguished by their fruits, the former being a capsule, a third of an inch in diameter and the latter a pod of 2 inch long. They are annual plant and grow up to 10 or more feet high with a straight cylindrical stem of about 1 inch thick at the base, having some branches at the top. The various growth stages of the plant are shown in the figure (1)



(a) 10 days, (b) 30 days, (c) 60 days, (d) 90 days, (e) 120 days

Fig-1: Growth rate with the age of the jute plant

The plants are harvested at the time indicated by the fading of the flowers. The fibres become coarse and woody if the plants are left too long in the ground and they are weak if harvested too early. The property of the fibre also depends on the species and varieties of the plants and on the condition of the climate, soil and season.

The jute fibres start forming at the early stage of the plant growth and go on developing their shapes and sizes, structures and properties, composition and constitution till the plant attains the flowering stage [5,6, 7]. When the cross sections of the green plant stem are seen under the microscope, the patches of fibres cells in bundles having 10-30 cells clustered together in each bundle are found located within the various vascular tissues of the cortex. The number of fibre bundles in a stem cross-section varies from 20 to 75 depending on the girth or diameter of the stem and age of the plant. These fibre bundles do not maintain a straight course, but bend, branch and are often rearranged into new groups at different levels. This process leads to the formation of a network. The degree of meshiness is determined by the tangential expansion and radial growth of the stem. The fibre bundles formed at the earlier stage are pushed apart as the bark of the stem is tangentially expanded due to the age of the plant with the formation of the new ray cells and fibrous tissues. At the latter stage, these fibre cells and soft phloem tissues continue their secondary growth for a comparatively longer period in such a way as to develop fibre tissues more compact and more meshy towards the base of the stem [8].

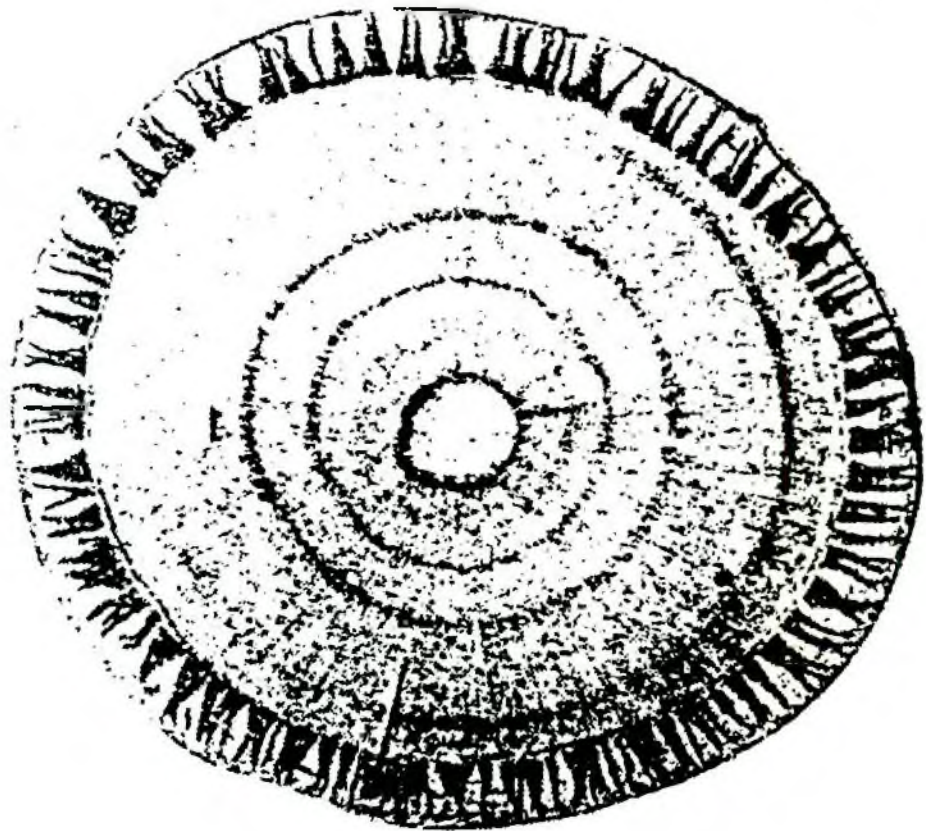


Fig-2 : Cross-section view of jute stem.

2.2 Microbial

As a common practice, jute fibres are separated from the adhering tissues by retting-a microbial process [9, 10, 11] to dissolve out soft plant tissues and cuticular substances excepting the fibre mass which is obtained as a tuft of fibre being more coarse, compact and meshy towards the base and finer and thinner towards the upper end. Unfortunately, retting does never ensure complete or true separation of the fibre from the cortex of the plant. The reason lies in the fact that retting starts from the upper part of the plant and the effect is that when fibre separation is complete at the upper part, retting at the bottom part

begins, thus resulting heterogeneity of the characters in jute fibre. More over, the fibres lose to some extent their inborn properties due to disintegration of tissues associated with the native fibre cell during retting, which is more pronounced if they are over-retted. Thus the degree of variability in the fibre properties and structure, if retting is concerned, is dependent upon the conditions of retting in addition to the living fibre cells during the growth of the jute plants.

2.3 Morphological

The morphology of jute fibre describes the complex structural property of the fibre cells which originate from protoxylem tissues and developed into fibre bundle by the activity of the cambium [12]. Fibre bundles constitute the strands or filaments of jute fibre, which are made separated on a card machine before processing to yarn. A transverse section of the typical jute strand is shown in the figure- 3.

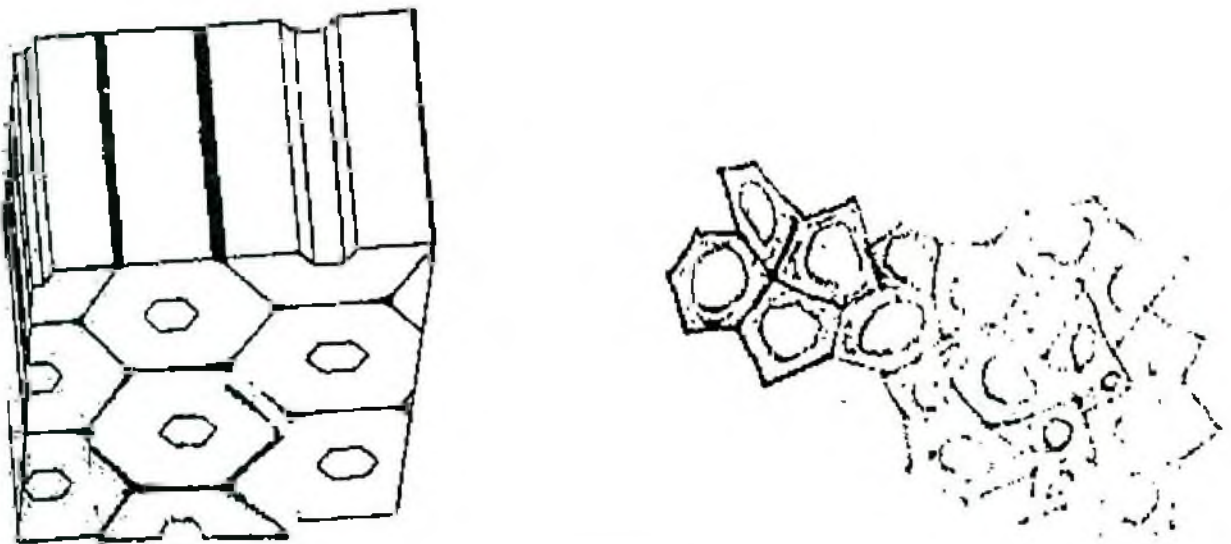


Fig-3 : Cross-section view of a typical fibre filament.

2.3.1 Fibre strand/filament

A well carded textile filament of jute is a dead tissue consisting of fibre cells numbering about 20 to 80 and having dimension of about 10 to 100 mm in length and 0.03 to 0.06 mm in breadth [13]. It appears from the cross sectional view that the fibre cells are more or less polygonal in outline with defined angles and are attached to one another through the middle lamella, a dense space, between the cells to form a wedge-shaped bundle. The fibre cell known as the ultimate fibre is elongated in the direction of the stem axis and associates with the other fibre cells of the bundle in such a way as to form a string of fibre known as the fibre strand or filament. Due to the variability in number of cells in a bundle the filaments of fibre are of variant length, breadth and shape and are often loosely attached to one another to form a net work predominantly towards the lower part of the plant. This attachment is, at places, so strong or runs so closely along a significant length of the other strand that the filaments or strands are hardly got separated during retting and hence, are comparatively coarser and thicker at the place of their joint causing irregularities in the fibres. This the entire length of the fibre tuft come out after retting consists of fibre filaments with a wide range of geometrical variations.

2.3.2 The fibre cell.

The fibre cells can be separated from the fibre filament or bundle by mercerisation alternatively with mild acid and

alkali [13, 14, 15]. These fibre cells are of variable length from 0.5 to 5 mm being 2 mm in general and diameter from 0.01 to 0.03 mm, having tapered ends.

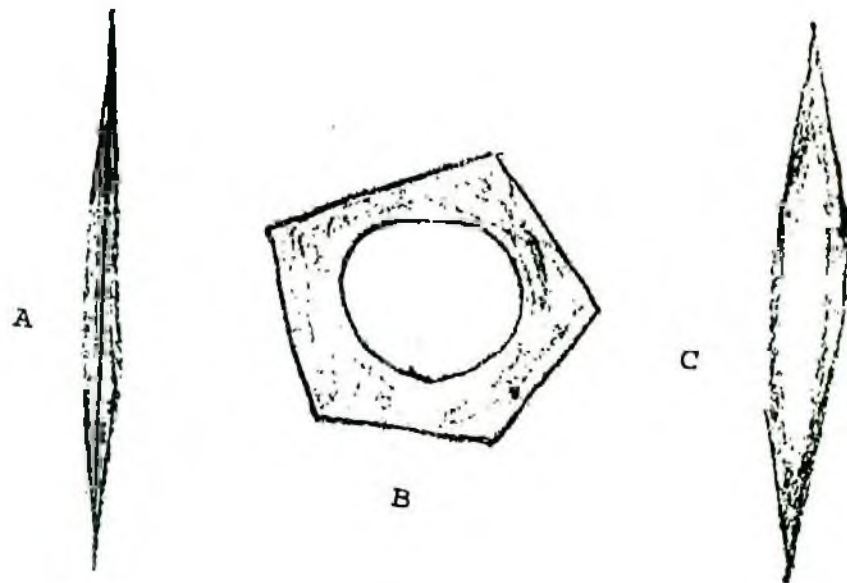


Fig- 4: Fibre cell (A), cross-section of the cell (B) and Longitudinal section (C)

The dimensions of these fibre cells are of such magnitude that their length breadth ratios are about 100 to 150, which is very low for a textile fibre. A high length breadth ratio is typical of nearly all fibres that are spun and woven by traditional methods, being of the order of 1000-2000 for wool, cotton and flax [16]. This is said to be the reason why jute is used for coarse fabrics only. The jute fibres used for textile purpose are not the ultimate fibre cells taken separately, rather they are so cemented together in a filament that any treatment that removes the cement completely disrupts the fibre quality and that the property of the fibre cells is the sum total properties of the cells present in a filament.

2.3.3 The middle lamella

The space heavily constituted between the fibre cells is called the middle lamella. The middle lamella is not the part of the fibre cell, but it surrounds the entire cell, as if it acts like a capsule, hard and non-elastic, holding all the components of the fibre cell inside. It is likely shared and bonded with the adjacent walls of the fibre cells. The lamella is formed at the time of cell division by folding of the primary longitudinal wall of the mother cells along with the plasmalemma and the so called 'cell plate' components [17, 18, 19].

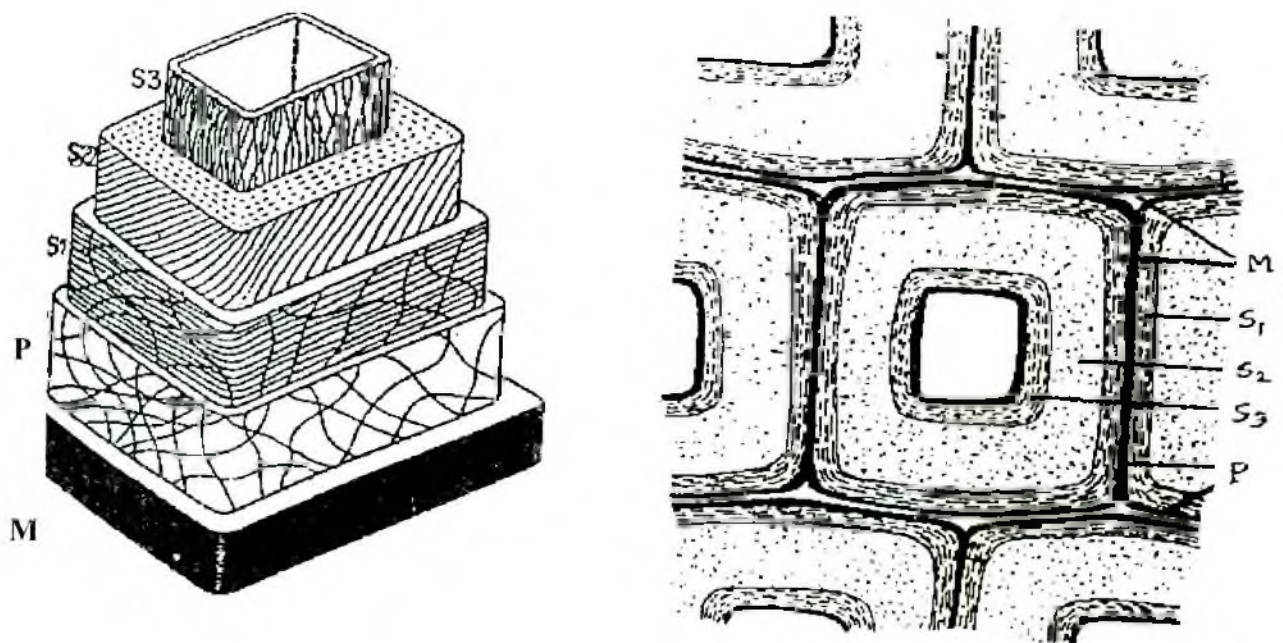


Fig- 5 : Cell-wall architecture, schematically. M middle lamella, P primary wall, S1 transition lamella, S2 main layer of secondary wall, S3 tertiary wall or tertiary lamella of secondary wall.

It is about 1-2 μ thick, varying with species and growing condition [20]. It is amorphous, dense and some what more porous than the fibre wall, the packing density being about 80% - 90% of the secondary wall [21]. The space of the middle lamella is initially filled up with pectic and hemicellulosic substances containing uronic acids or uranides which are partially or entirely disintegrated at the latter stage, becoming heavily lignified during the phase of fibre maturation [22]. The kind and concentration of lignin and hemicelluloses determine the character of the middle lamella.

With the age of jute plant, the space of the middle lamella is densely packed of by these constituents specially by lignin at a high rate making the fibre gradually more rigid, coarse, brittle, irregular, heavy, woody and hydrophobic. The composition of the middle lamella that resembles that of the mortar in brick work links up the fibre cells to form a fibre for textile use and hence the middle lamella is the most vulnerable and sensitive place in fibre, because any crack or damage at this place may destroy the articulation of the fibre.

2.3.4 The cell wall

The young cell walls of the ultimate fibre consist only of a primary wall lamella, which becomes thickened by the apposition of additional lamella which form the secondary wall. The primary wall is so thin being about 0.1 μ in thickness that it is hardly differentiated from the

middle lamella. The primary wall contains cellulose microfibrils forming a thin framework of a woven or dispersed texture, with little orientation, embedded in an amorphous substance of pectic hemicelluloses and much lignin [23]. Because of its lignin content as well as its disperse fibrillar texture, the primary wall has very limited swelling capacity and therefore, bursts on treating the fibre with swelling agents. It seems that the middle lamella and primary wall altogether occupy about 25% of the volume of the jute fibre with a higher concentration of non-cellulosic constituents and by virtue of their compactness in the periphery they play the prime role of determining the visco-elastic and physico-chemical properties of the fibre.

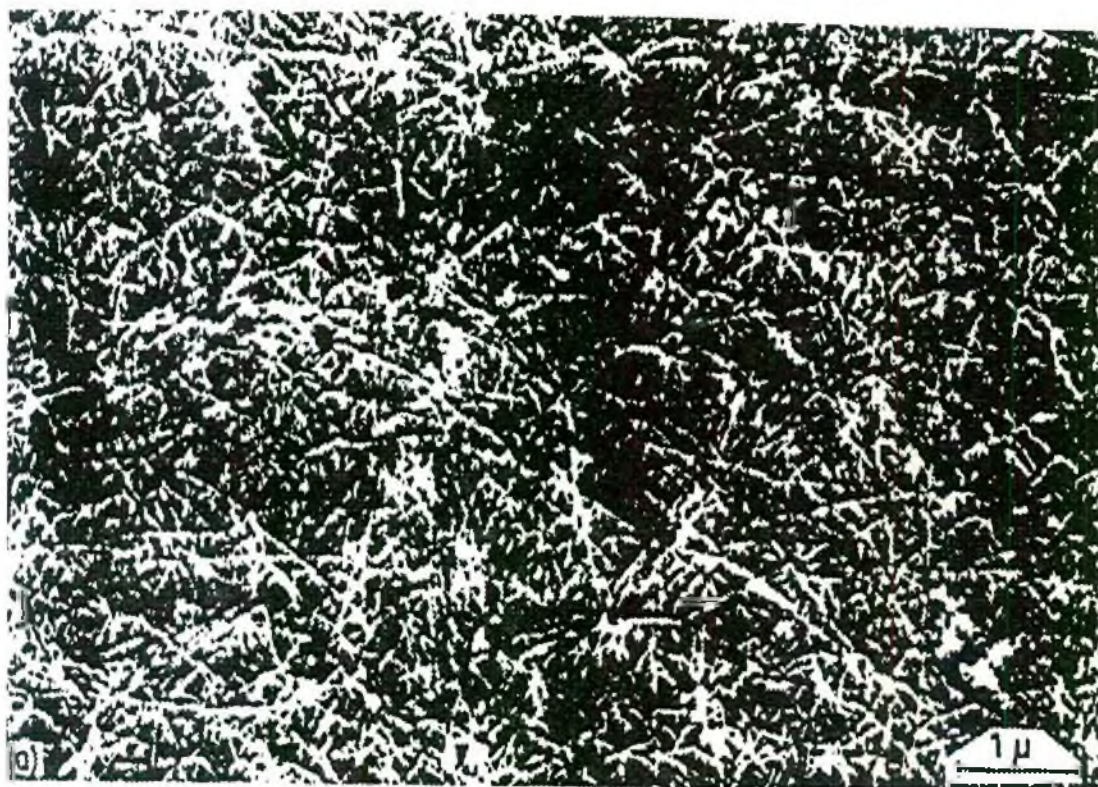


Fig-6 : Orientation of micro-fibrils dispersed texture in primary wall.

The secondary wall is formed in the secondary process of fibre maturation. The wall is not homogeneous, but used as a collective term for all fibre layers apposition during secondary growth and is mainly divided into S_1 , S_2 and S_3 lamella.

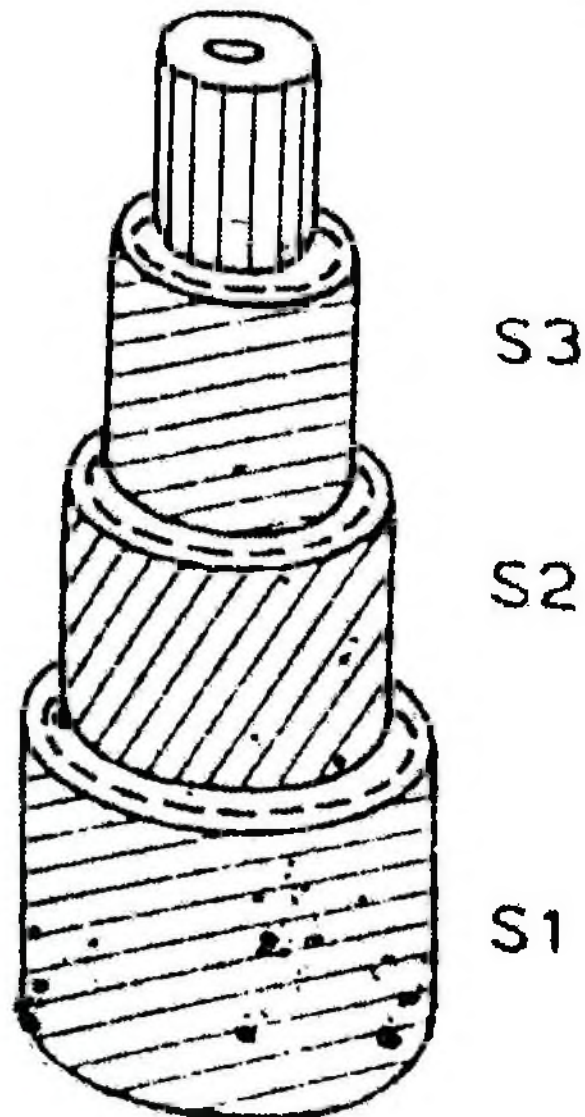


Fig-7: Orientation of micro-fibrils in secondary wall indicated by striation.

S_1 is the transition lamella or the outer secondary wall. It has a thickness of about 0.15μ and consists of two counter rotating helices of cellulose microfibrils. It also contains fair amount of lignin and hemicelluloses, some of which seems to form a thin outer layer.

The main secondary wall is the S_2 lamella which forms the bulk of the fibre, with a thickness of $1-10\mu$ varying with species and growing condition. The main secondary wall in many fibre cell, is subdivided into many sub-light microscopic lamella. Cotton hair-cell wall, for example, consists of as many as 25 lamella each being 0.4μ in width [24, 25] (Fig-8).



Fig-8 : Sections across cotton hairs normally cultivated with daily growth rings.

Each lamella occurs as secondary deposition of micro-fibrils with relatively high content of cellulose, forming a parallel texture or fibrous texture round around the lumen at a fairly low angle to the fibre axis, 6° for jute, 30° for cotton and $10-35^{\circ}$ for other wood fibres.

The S_3 lamella is called the tertiary wall or the inner secondary wall which constitutes the limiting structure of the fibre wall toward the lumen. It consists of one single lamella, $0.07 - 0.08\mu$ thick. The tertiary wall contains a frame work of cellulose micro-fibrils, arranged in a Z helix. It is remarkably resistant to chemical, especially towards alkali reagents which may be a consequence of its dense structure as well as its special chemical composition, where glucuronoarabinoxylan is believed to dominate.



Fig-9 : Orientation of micro-fibrils, crossed parallel texture in secondary wall.

The cellulose micro-fibrils are mostly bands of $100\text{-}250\text{\AA}$ wide and about $35\text{\AA}$ thick and are made up in association with three elementary fibrils each having a diameter of about $35\text{\AA}$ [26, 27]. These elementary fibrils possess a homogeneous chain lattice of cellulose and are brittle like crystalline needles. The association of elementary fibrils into thicker micro-fibrils develops a heterogeneous ultra capillary system in the secondary cell wall, on the one side the finest hollow spaces ($<10\text{\AA}$) between the elementary fibrils and on the other, coarser capillaries (Ca. $100\text{\AA}$) between their micro-fibrillar bundles. Only micro molecules like H_2O and I_2 are able to penetrate into the first, while colloidal particles like congored or colloidal silver and gold can be infiltrated into the coarser ones. The micro-fibrillar framework of cellulose in the secondary wall is incrustated with amorphous hemicelluloses and lignin in the inter fibrillar spaces to a dense structure.

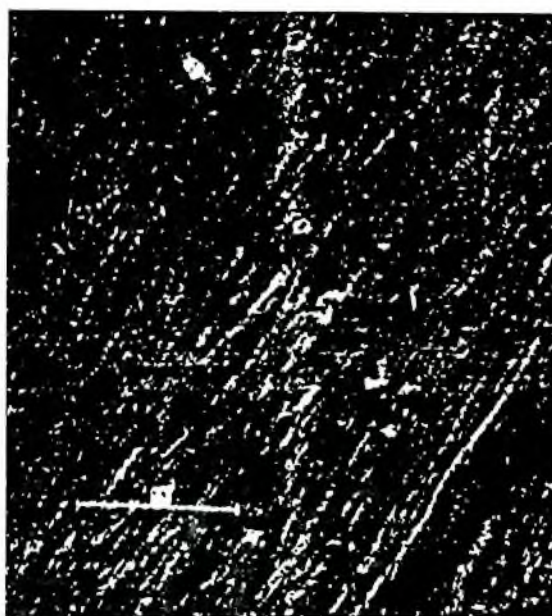


Fig-10 : Electron micrograph of the secondary wall.



Fig-11 : Electro micrograph of secondary wall after delignifications.

2.3.5 The lumen

The lumen is empty and constitutes about 1/5th part of the fibre cell. This void space in the fibre cell originates from drying out of the protoplasm and other liquid elements of the living cell and having these attached to tertiary wall. The study of the lumen would be very interesting, because the high percentage of water imbation, better insulation to heat, less compressibility and other related properties may be attributed to the vacant space available within the fibre cell.

2.4 Model of jute fibre filament

If we consider a fibre filament to be resembled with a cylinder of length 5 mm and of an uneven diameter of about 0.04 mm, it would hold about 60-80 tiny cylinders of fibre cells being 2 mm in length and 0.015 mm in diameter on average and having 3 to 6 sides tapered at both ends. If we make a transverse section of the filament cylinder, we would find cutin, pectin, tannin and pentosans constitute the outer or surface wall of it. It protects the inner fibre cylinders. After the protection wall, there lies the wall of the fibre cell, then lumen, the vacant place and then again the cell wall. The middle lamella always lies between the cylindrical fibre cells lying one after another.

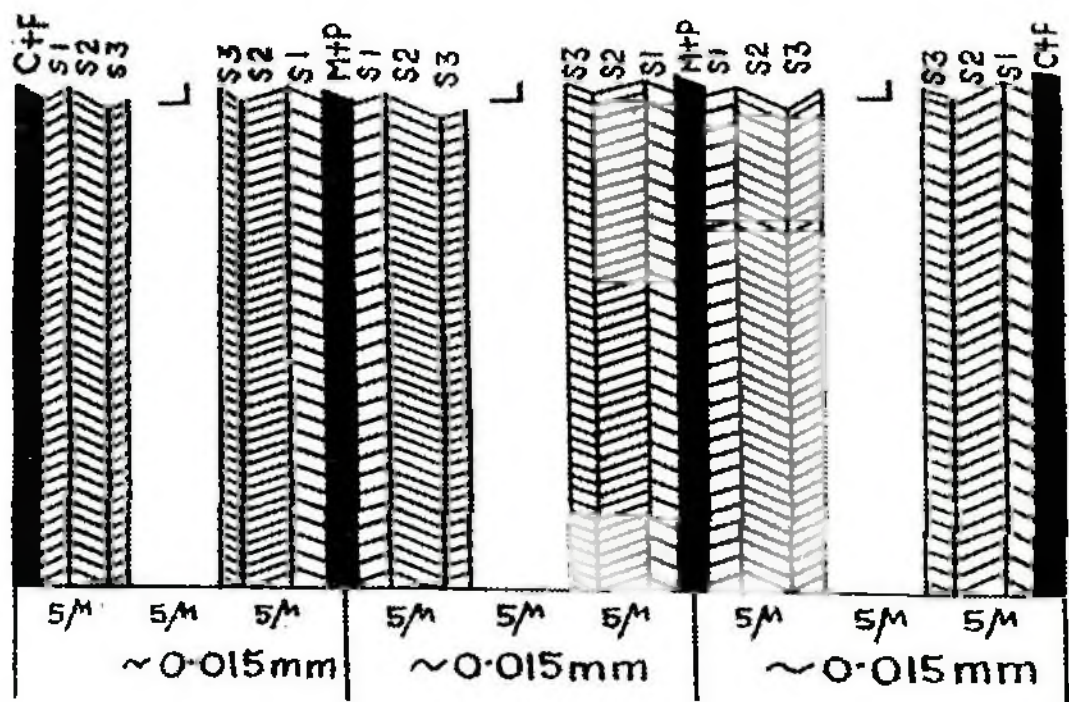


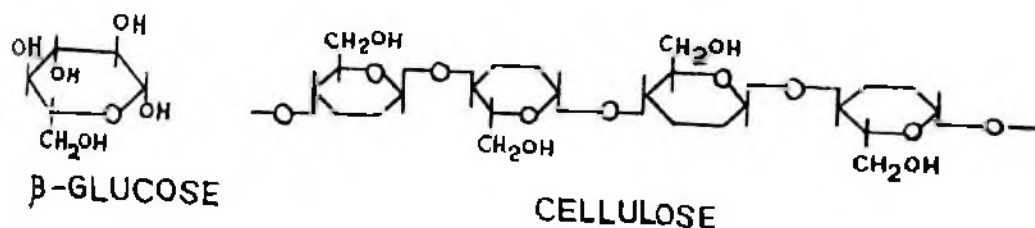
Fig-12 : Schematic L section of jute fibre (Model structure of jute fibre)
 C+P = Cutin and primary layer, L = Lumen, M+P = Middle lamella and primary wall, S₁, S₂, S₃ = Secondary wall layer.

2.5 Compositional

The jute fibre cell walls like all other wood cell walls contain several groups of compounds which consist of fibrous frame substances, ground substances (matrix), incrusting substance and adcrusting substances. The mechanical properties and the technological value of the fibres in textile processing are due to the presence of fibrous materials of cell wall.

2.5.1 Cellulose

The fibrous components are the high polymeric polyglucan cellulose molecules containing 1, 4- β -D glycosidic linkage between the anhydroglucose monomers.



The chain lengths of cellulose molecules present in the fibre cell wall have a wide range of variation. According to Staudinger [28] cellulose molecules with some 10 glucose residues linked together to a

chain go into solution, and these having about 100 units dissolve to form sols in 10% sodium hydroxide. When the degree of polymerisation exceeds 100 and approaches 800, the celluloses are not attacked by 1% sodium hydroxide and find application for regenerated celluloses which swell in the 10% sodium hydroxide yielding viscous gel-solutions. The native celluloses (the so called α -cellulose) is distinguished by a higher degree of polymerisation and form a very stable component of the cell wall.

Table- I

The homologous series of cellulose polymers [28, 29].

Series	D.P	Chain length	Mechanical properties & forms	Formation of fibrils	Solubility in 10% NaOH
Celludextrin (δ -cellulose)	1-10	5-50	Pulverised crystalline pdr.	None	True solution
Hemicolloid (β -celluloses)	10-100	50-500	Pulverised short fibres	Slight	Formation of Sol.
Mesocolloid (regenerated cellulose)	>100	500-2500	Fibres	Large	Formation of gel
Native Cellulose (α -cellulose)	00-10,000	500-50,000	Long fibres	Very long	Limited swelling

These cellulose chains do not occur as free molecules in nature

and in cell wall, they always appear to be more or less crystallised into a chain lattice. These lattices yield excellent x-ray patterns, from which the unit cell represented to a monoclinic crystal lattice, containing four glucose residues of two anti-parallel cellulose chains with the dimensions $a = 8.3 \text{ \AA}$, $b = 10 \text{ \AA}$, $c = 7.9 \text{ \AA}$, $\beta = 84^\circ$ is illustrated in the fig- (13). It is peculiar that the unit cell does not contain whole molecules, but only the repeating unit i.e, the disaccharide of the cellulose chain [30, 31, 32].

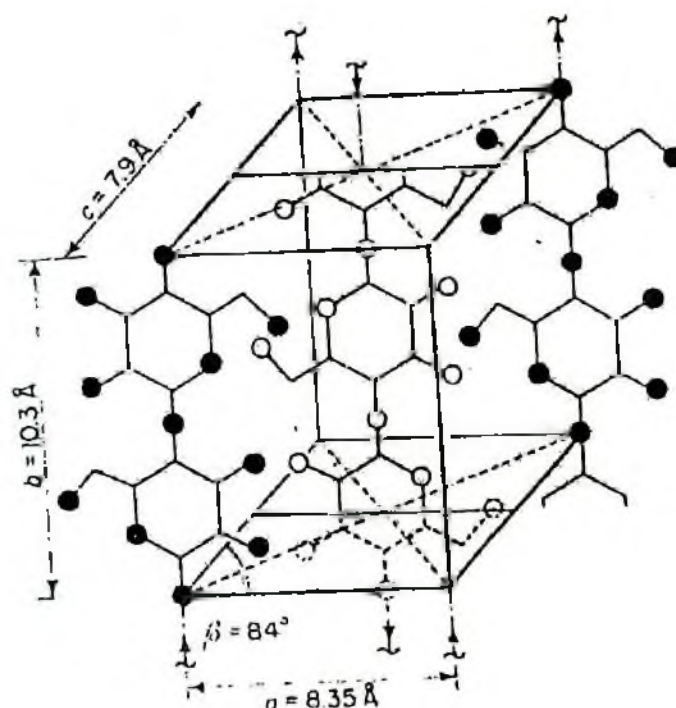


Fig-13: The monoclinic elementary cell of cellulose

The cellulose lattice is held together by various types of bonds.

Along the b-axis of the lattice i.e. parallel to axis of the fibre cell the bonds are covalent, whilst the thirty times weaker hydrogen bonds are exerted perpendicularly to this axis. The covalent bonds must be broken to overcome the tensile strength of such a fibre, whilst in lateral swelling the hydrogen bonds are weakened.

The cellulose chains containing indefinite number of glucose residues are invisible only because their width is amicroscopic, each glucose residue measuring $5 \text{ \AA}$.

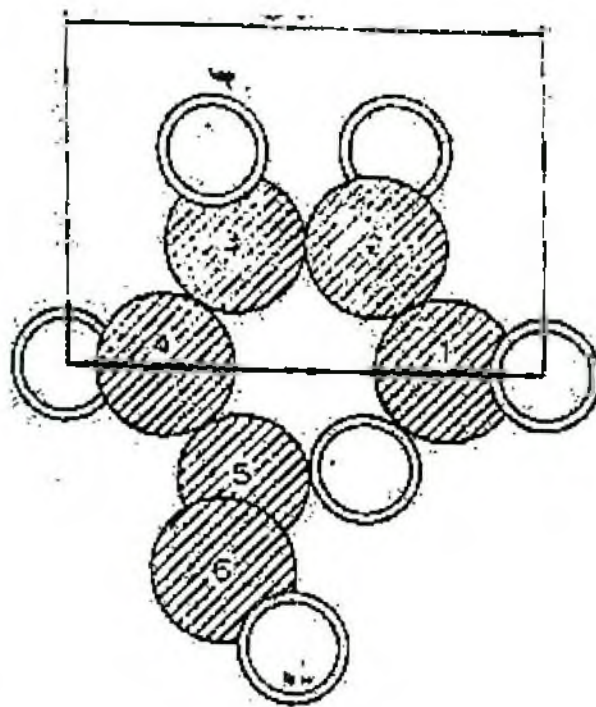


Fig-14 : The molecular structure of glucose C-atoms hatched, O- atoms surrounded

These chains are linked to form crystalline elementary fibrils which are about $30 \text{ \AA}$ thick and $35 \text{ \AA}$ wide, some fraction of these are being occupied by amorphous region.

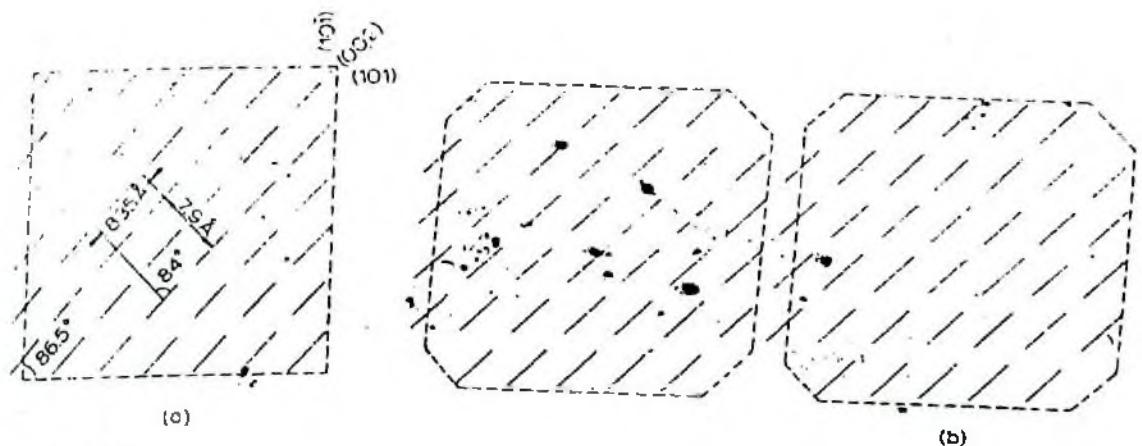


Fig-15 : Cross-section of crystalline elementary fibril showing the arrangement of the cellulose chains (a) number of chains in fibrils of $35 \text{ \AA}$ diameter, (b) fasciation of elementary fibrils to microfibrils, $35 \text{ \AA} \times 100 \text{ \AA}$

The cross section of an elementary fibrils measures $30 \times 35 = 1050 \text{ \AA}^2$. Since the area occupied by a single cellulose chains is $1/2 \times 7.9 \times 8.35 \times \sin 84^\circ = 32.8 \text{ \AA}^2$, an elementary fibril contains only about 32 cellulose molecules and possesses a pronounced tendency to aggregate laterally by two, three or more such units to form microfibrils [33]. The cellulose micro fibrils are mostly bands 100 (to a maximum of 250) $\text{ \AA}$ wide and about $30 \text{ \AA}$ thick. [34]. Single elementary fibrils are mostly

observed in primary walls, whilst in secondary walls they are mostly aggregated to thicker micro-fibrils.

2.5.2 Ground substances (matrix)

Except cellulose all amorphous carbohydrates form the ground substances of the cell wall. It consists of hemicelluloses and pectic materials which can be removed from the cell wall by varying treatment with dilute acids and alkalis. The cellulose fibrils are embedded in this ground substance or matrix. It forms a soft plastic mass reinforced by the fibrils.

Carbohydrates which in distinction from cellulose are soluble in concentrated alkali (17.5% NaOH, 24% KOH), are known as hemicelluloses, while the uronic acid containing hemicelluloses known as uronides constitutes the pectic substances. On hydrolysis the hemicelluloses lead to different hexoses (in addition to glucose, mannose and galactose), pentoses (xylose, arabinose) and uronic acid (galacturonic acid, glucuronic acid). These components appear in widely differing relative amounts and in numerous combinations. In the uronic acids, the primary alcohol group CH_2OH of the corresponding hexoses is oxidised to an acid group COOH , which can then be lost by decarboxylation to give the homologous pentoses. Thus components of polymeric chains like glucose-glucuronic acid-xylose or galactose-galacturonic acid-arabinose or the two series mixed among one another are expected to occur as

amorphous carbohydrates in the cell wall.

2.5.3 Distribution of carbohydrates in cell walls.

The carbohydrate composition is the major component of jute fibre strand. The total mass distribution of carbohydrate matter appears approximately to be uniform throughout the fibre wall, but the relative concentrations of celluloses and hemicelluloses are different in different layers of the cell wall. Cellulose is virtually absent in the middle lamella, but its concentration increases towards lumen, where as the hemicelluloses are predominately located in the middle lamella and primary wall recending in the secondary walls.

From the analysis of primary cell walls of different vegetable fibres such as cotton hairs, leaves and stems (Table-II) compiled by Setterfield and Bayley, [35] it is obtained that over 50% of the dry cell wall is the ground substances and if the pectic substances are included (because of their similarity to hemicelluloses) the matrix amounts to almost 60%, against only 30% of the skeletal substance cellulose.

Table-II

Composition of primary walls.

	Dry %	Fresh %
Water	-	60
Hemicelluloses	53	21.2
Pectic substances	5	2
Cellulose	30	12
Protein	5	2
Lipids	7	2.8

Meir [36] examined the different layers of the cell walls of wood fibres for their carbohydrates throughout their growth. The contents of the primary wall (p) were considered together with those of the middle lamella (m) as the primary wall grows simultaneously with the middle lamella and is hardly differentiated. Carbohydrate composition m+p wall and that of S_1 , S_2 and S_3 layers of secondary wall is shown in the table below.

Table-III

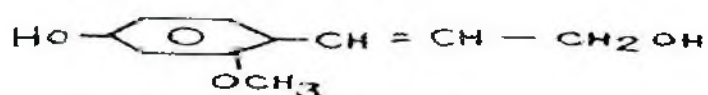
Polysaccharides in different wall layers of spruce trachids.

	M+P	S ₁	S ₂	S ₃
Galactose	16.4	8.0	0.0	0.0
Arabinan	29.3	1.1	0.8	0.0
Glucurono arabinoxylan	13.0	17.6	10.7	12.7
Glucomannan	7.9	18.1	24.2	23.7
Total Hemicelluloses	66.6	44.8	35.7	36.4
Cellulose	33.4	55.2	64.3	63.6

The cell wall of jute fibre which resembles wood trachids & fibres in the nature of carbohydrate composition, is likely to have similar kind of distribution of hemicelluloses and cellulose in different layers of it.

2.5.4 Incrusting substance

The most important of the incrusting substances embedded in the cell wall is lignin, which arises in the cell as a macromolecular substance by polymerisation of coniferyl alcohol [37].



Coniferyl alcohol

As can be seen, this is a methylated C_9 compound in which a diphenol bears an unsaturated C_3 chain. The methyl group substitutes one of the two phenol groups. Owing to its double bond in the side chain, this compound can polymerise in different ways, so that in contrast to the linear polymerisation of polysaccharide and polyuronic chains, cross-linking can occur in different directions in space by the so-called reticular polymerisation.

In jute fibre, the cell wall contains close to 12 to 18% lignin. The cell wall also contains additional incrusting substances which belong to the class of tannins, the polymerising phenols.

Besides these organic compounds, some mineral substances left behind by water are incorporated in the cell wall as incrusting substances.

2.5.5 Distribution of lignin

The relative concentration of lignin with respect to other constituents at different wall layers of the fibre cell is not uniform. With the qualitative staining reaction a high accumulation of lignin is observed in the middle lamella and primary wall, whilst the secondary wall is less lignified. From the quantitative photometric analysis of absorption curves (Fig.16) measured in the UV microscope, it has concluded that the lignin content decreases from a maximum in the primary wall to minimum in the outer secondary wall [38].

A model of the lignin distribution as seen in fig (16) yields a calculated intensity curve exactly as measured in the densitometer [39]. The lignin content, thus, seems to be constant and gives a value of 3.4% lignin throughout the secondary wall and 7.3% lignin in the primary wall.

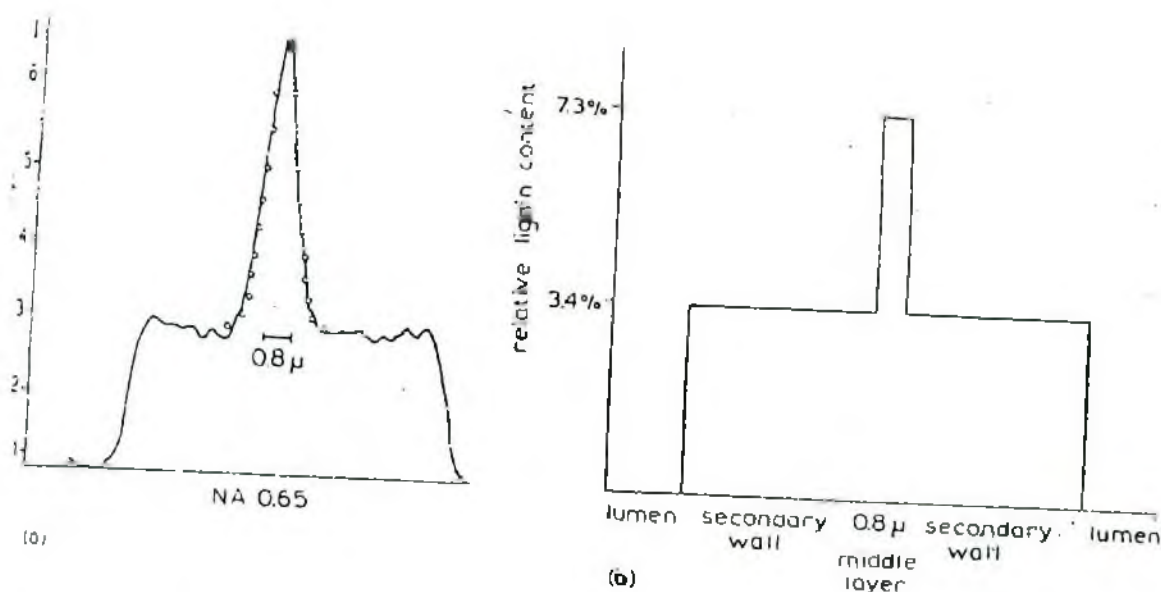


Fig-16 : Distribution of lignin in primary and the secondary wall of jute fibres: (a) intensity (I) of lignin fluorescence shown by densitometer curve across the wall of adjacent cells, o calculated values based on diffraction at the phase boundary primary/secondary wall; (b) actual lignin distribution, without obscuration from diffraction effects.

The jute fibres used in the experimentation only slightly lignified as being taken from the jute plant at its early stage of growth. The concentration of lignin goes on increasing with the age of the fibre and is

as high as 80-90% in the middle lamella of well matured fibre, but only about one fifth of the total lignin is present in this region. The bulk of lignin about 80% is located in the secondary wall [40].

Lignification starts in the primary wall and then in the middle lamella and secondary wall [41]. The middle lamella and primary wall are more heavily lignified than the secondary wall. A space problem is probably involved. Since the middle lamella and primary wall is poor in cellulose, a much greater amount of lignin is incrustated to produce in it a solid mass, than in the secondary wall with its high percentage of cellulose micro-fibrils. With incrustation of lignin the volume of the matrix substances is reduced by dehydration and their amount by chemical degradation, resulting in a hardened cell wall in the later stage in combination with polymerising phenols and mineral substances.

2.6 Length wise distribution of fibre properties

The tuft of fibre obtained after retting from a jute plant does not have the same quality of fibres at different length of it. One observing it from the base to the top will see that it is hard and stiff at the base owing to the fibres stuck in other plant tissues and mucilage while in the other parts the fibre are in general, well separated and flexible. After having cut off some length as stiff base known as hard cuttings in commerce, the fibre tuft is used in commerce and industry, but does not still have the equal distribution of the physical and chemical properties along the length

of it and hence, the yarns and fabrics contain heterogeneous fibrous elements which are evaluated on their composite or average properties.

The fibre properties of jute in terms of its length, diameter, colour, strength, extension, moisture regain, chemical composition and ultimate fibre dimension have been investigated. A length wise distribution of these properties may be expressed in the table IV showing only the bottom, middle and top parts of the fibre tuft without having hard cuttings.

Table-IV

Length-wise distribution of fibre properties of jute collected from various sources including the author's own work. [42, 43]

SL No	Property	Bottom	Middle	Top
1.	Fibre length (cm)	4	7	7
2.	Fibre diameter (μ)	45	35	25
3.	Extension (%)	0.5	1.0	0.8
4.	colour	Brown yellow	Golden yellow	Whitish yellow
5.	Strength (Pressly index) lbs/mg	8	10	9
6.	fineness (gm/tex)	3	2	1.50
7.	Moisture regain (%)	11	10	10
8.	Waxy material (%)	2.5	2.0	1.9
9.	Cellulose (%)	59	65	60
10.	Hemicellulose (%)	22	24	25
11.	Lignin (%)	14	12	11
12.	D.P. of cellulose	3000	1600	500
13.	Ultimate fibre length (mm)	2	2.5	2.8
14.	Ultimate fibre diameter (mm)	.018	.017	0.15

The bottom gives fibres having comparatively higher diameter and less extensibility, which means that coarseness and hardness in jute fibres are more pronounced towards the base and gradually decrease towards the upper part. The irregularity in the fibre property has been developed from the complex process of deposition of chemical components and their morphological character at different age of the plant growth. The apposition of cellulose micro-fibril in the secondary wall is always associated with disposition of amorphous carbohydrates like pectin and hemicellulose and of incrusting and adcrusting substances like lignin, tannin and cutin. With the inceasing D.P or mol. wt of cellulose molecules having greater amount of non-cellulosic substances in the bottom part, the cell walls as well as the middle lamella of the fibre become compact developing more hardness and coarseness of the fibre.

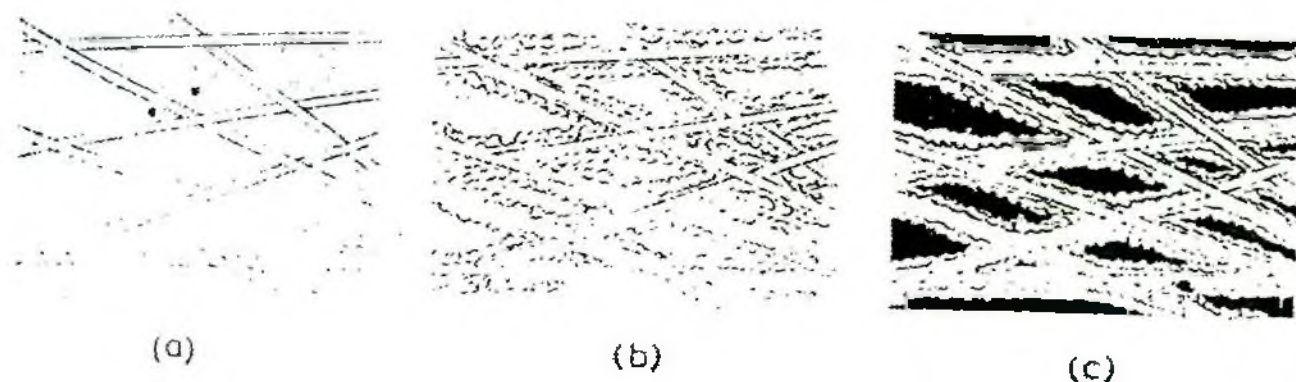


Fig-17 : Incrustation of the cell wall; (a) fibrillar frame work interfibrillary matrix, (b) incrustation of lignin, some matrix is left (loss of extensibility,) further incrustation by phenols and/or mineral salts (hardening).

3 JUTE AND ITS RELATIONSHIP WITH PHOTSENSITIVITY

Jute fibre on exposure to sunlight or on prolonged standing under the shed, changes its colour known as yellowing effect. This is being accompanied by the degradation of its strength properties. This property appears to be in common as is found with paper products manufactured from high yield pulps such as ground, thermo mechanical pulps and high yielding sulphite pulps [44,45].

The photochemical auto oxidation is believed to be initiated by the photochemical conversion of the phenyl propane units of lignin, although polyphenolic extractive components may sometime be a contributing factor, to the corresponding benzyl radicals. These radicals react with atmospheric oxygen forming hydro-peroxy radicals which are capable for abstracting hydrogen atom from surrounding molecules. The resulting hydro peroxides generate coloured chromophoric groups possibly via intermediate dioxatane structure causing the yellow colouration [46] . The hydro peroxides also may decompose with the generated hydroxy radicals. This, in turn, may either accelerate yellowing process or alternatively attract the cellulose component causing its degradation. The latter phenomena is analogous to the photo chemical tendering of the cotton fibres by some anthraquinone [46] dyes. The degradation of cellulose and other polysaccharides can also generate chromophoric groups and thus contribute to the discoloration of the fibre.

So the presence of lignin is the principal cause of photo degradation. Removal of lignin from the fibre is perhaps the way but the removal of lignin from the jute fibre destroys the fibre properties. consequently, the photo sensitivity of the jute fibre can not, by any means, be prevented without drastic reduction in dry and particularly in wet strength of the jute fibres. In fact, the photo degradation can be reduced but it can not be eliminated.

There are two ways to reduce these effects on jute fibre.

- (i) To incorporate inorganic components in the fibre that may either quench the photon-excited aromatic groups in lignin or to scavenge the radical intermediates in the oxidative chain process initiated by photon-excitation process.
- (ii) controlled/partial removal of lignin component and/or its conversion to a photo chemically more resistant form. As the lignin can not be removed completely without seriously injuring the fibre strength, it has been suggested to remove lignin partially from the jute fibre. Obviously, the degree of delignification should not be extended beyond the point where dry and particularly wet strength of the fibre is reduced below acceptable values. The latter proposition seems to be viable and it has drawn the attention of the workers in the field. In fact, recently this proposition is gaining ground slowly.

4 EFFECTS OF UREA-FORMALDEHYDE AND MELAMINE FORMALDEHYDE RESINS ON JUTE FIBRE AND JUTE FABRICS.

Callow [47] attempted to check decolouration of jute by treating it with urea-formaldehyde precondensate containing excess formaldehyde. If subsequent washings of treated jute are avoided, the effect was positive but he pointed out that urea-formaldehyde has no effect if the phenolic end groups in lignin were just blocked by methylation.

Other workers [48, 49] used urea-formaldehyde and melamine formaldehyde resin using organic as well as aqueous solvents and various catalysts. The net results were decrease in tensile strength, rot proof on one hand and increase in wrinkle recovery and to some extent wet strength of the treated jute fibre. Other important textile parameters, like (comparable) dry and wet tensile strength, abrasion resistance, stiffness shrinkage etc were not, however, studied thoroughly.

Recently, effects of ureaformaldehyde and melamine formaldehyde resin precondensates on jute fibre and jute fabrics were studied in detail by Amin et al [50]. They treated bleached jute fibre and fabrics with the foresaid precondensates and studied the effects of resin as well as catalyst concentrations, time and temperature of resin curing. The total resin, bound nitrogen, bound formaldehyde, percent resin add on and percent precondensates reacted with jute fibre and jute fabrics were also

studied.

The treated fabrics showed marked improvement in various physical properties, such as insolubility in cuprammoniumhydroxide, reduction in moisture regain, tensile strength etc. in one hand and in some important textile parameters like crease resistance (wet and dry) abrasion resistance, flexural rigidity, dimensional stability etc. on the other.

There exists, as the worker noted, a direct relationship between the resin add on to the fabrics and significant improvement in the crease resistance and in reduction of moisture regain. The losses of tensile strength and abrasion resistance and increase in flexural rigidity values were found proportional to the amount of applied resin.

It was further observed that fabrics showed reduced flammability when treated with melamine formaldehyde resin in admix with urea and sodium dihydrogen phosphate. The results, as they claim, confirm with the acceptable international standards for flame proof fabrics.

The textile properties of jute cellulose made to react with different types of N-methylol compounds and inorganic salt catalysts were studied [51]. The ammonium salt catalyst gives a more improved wrinkle recovery angle in urea-formaldehyde (UF) and melamine formaldehyde (MF) finishes than metal salt catalysts which are effective in dimethylol ethylene urea (DMEU) and dimethylol dihydroxyethylene urea (DMDHEU) finishes. The author observed that wrinkle recovery angle increases with increase in catalysts concentration up to a level and

further increase in concentration cause decrease in wrinkle recovery angle. The jute fabric gives the optimum wrinkle recovery angle at 150°C curing temperature in 5 minutes curing period. Catalysts used in this study alone caused a higher tensile strength loss than the cross-linked jute fabric. The wrinkle recovery angle of jute fabric increases with the lowering of tensile strength irrespective of the resin catalyst system.

They also studied the effect of acid catalyst [52] at different pH levels on the textile properties of jute fabric modified by DMEU and DMDHEU. The cross-link reaction leads to change in physical properties of jute fabrics such as dry wrinkle recovery angle (DWRA) and tensile strength. DWRA is developed at a curing temperature of 150°C and increases with increasing temperature and curing duration from 5 to 7 minute. They also observed that acetic acid and hydrochloric acid give the highest DWRA compared with that obtained with other acid catalysts. They concluded that values obtained in the presence of acid catalysts are inferior to those obtained by using metal salt catalysts.

5 FIBRE MODIFICATION

Every kind of materials we see around us is destined to have some distinctive and native properties which determine as well as limit its applicability and service ability to human need. To explore wider or

newer usages of any material we, therefore, need to change or modify, if possible, the intrinsic properties of it to an extent to satisfy our specific purpose. Raw rubber is sticky at low temperature and has little use, while vulcanisation i.e, sulphide rubber modifies its properties making suitable for so many purposes that modern civilization can not go without it. Similarly, modification of nylon to kevlar for making space suits is an example of an achievement of a specific purpose of the cosmic research. Jute, too, has got its characteristic native properties being unfavourable for textile processings, which have been improved to the present status to make coarse yarns and fabrics by the addition of oil-water emulsion to raw jute fibre before processing. This technique of softening practiced by all jute mills for processing of jute fibres is infact a physico-chemical modification of jute fibres. But it is temporarily developed in jute fibre for the specific purpose, because the fibre is soon reverted to its original characters on drying, with more additional complications. So, an effective modification in relation to properties and structure of the fibre by all possible ways is necessary in order to expand and diversify its usages.

5.1 Methods of modification

The approaches for modification of jute fibre may be considered in four different broad areas:

- a) Argo-genetical methods to improve fibre quality and to increase

yield.

- b) Improvement of the effectiveness of enzymic processes utilized in retting and pilling .
- c) Physical processes utilized in the conversion of crude fibre to yarn and fabric.
- d) Chemical methods of fibre modification.

5.1.1 Agro-genetical method.

This is a biological process which may be very effectively utilized to improve the fibre quality and increase the yield per acre of land. It is expected that by introducing genetical factors and improved hybridization the fibre cell geometry, cell composition and fibrillar arrangement may be changed towards the desired fibre properties.

5.1.2 Enzymic approach.

It is known that retting of jute involves enzymes which hydrolyze pectic substances and plant tissues and thus separate the fibre strands from adhering plant tissues [53]. The fibre quality may be improved by having enzymes characterised and specified to hydrolyze jute constituents, but this is a cost effective approach.

5.1.3 Physical approach

Jute suffers from certain undesirable viscoelastic properties. Its moduli of elasticity and compressibility are quite high in comparison with flax, cotton and synthetic fibres. These defects in jute however may be, to some limited extent, overcome by modifying the structure of yarns and fabrics, which is achieved by blending jute with highly extensible fibres like cotton, viscose or acrylic at the time of mechanical processings. This physico-mechanical processes could be a better process to minimize shredding, improve feeling, increase composite elasticity and so on, but would not much effective due to the inherent visco-elastic behaviour of jute fibre which makes it incompatible with those fibres.

5.1.4 Chemical modification

Jute fibre is typically composed of the following chemical constituents shown in the table-V

Table-V

Average chemical composition of jute fibre [54,55,56].

1	Cellulose	57%
2	Hemicellulose	20.5%
3	Lignin	11.5%
4	Pectic Substances	0.3%
5	Tanins, protein, pigments and inorganic matters	0.7%
6	Waxy and fatty materials	0.5%
7	Moisture at room condition	9.5%

It may be reiterated that cellulose is the only fibrous component masked by the non-fibrous ground and incrusting substances which are about 40% of the total mass of the fibre. Again, the jute fibre ultimate cells are of the dimension of 2mm x 0.02 mm which are fused or attached very closely to one another at the middle lamella through the said incrusting materials in such a way as to form the fibre strand. So, the chemical processes for modification of jute must not be the same as those followed for cotton which is a single cell of 98% cellulose without non-cellulosic incrustation. However, any mild or controlled chemical treatment that render partial removal of incrusting and ground substances from the fibre is seemingly the proper approach to modify jute fibre chemically. Perhaps, etherification is a viable approach to modify jute fibre. In fact, etherification is an well known process in protecting or blocking hydroxy groups in lignocellulosic materials. Extensive studies

were made but limiting to methyl [57], ethyl [58] and to an extent propyl [59] radicals to determine the various types of linkages present in those substrates. The etherification studies also helped to ascertain the relative reactivities of the hydroxyl groups in monomeric units of the polysaccharides [60]. There is, practically, no attempt to assess the changes taken place in the fibre properties of jute or cotton or any other fibre due to etherification having C_1 - C_3 carbons.

Rahman's [61] works on the etherification of jute (*Corchorus olitorius*) fibre are briefly described below. Etherification of jute fibre with relatively higher alkyl chlorides of carbon atoms (C_4 - C_{12}) was carried out. The etherification based on some factors such as reaction time, reaction temperature and alkali concentration was studied. The effects of solvents were also studied.

The degree of substitution and the percent elongation of alkyl substituted jute fibres were determined. Butyl and dodecyl substituted fibres gave better percent elongation compared to others. The bundle strength of alkyl substituted jute fibre was also determined.

Microscopic and photo micrographic studies of untreated and treated jute fibres were carried out. The change in length and diameter of the fibres resulted for etherification were studied. Butyl substituted jute showed maximum increase in diameter and shrinkage in length.

Moisture regain of alkyl substituted jute fibre was determined and

it is found that it decreases with the increase of substitution.

Finer yarn was made from butyl and dodecyl substituted jute fibre and some textile properties like tex (count), tenacity, extensibility etc. were determined. The results indicate that a better quality yarn can be made from alkyl substituted fibre. It is observed from the table VI that after treatment with wetting agent the textile

Table –VI

Effect of degree of substitution on some textile properties of yarn made from etherified jute fibre after treatment with wetting agent.

Sample	Tex (wt. of 1 km in g)	Tenacity (g-wt-tex)	Extensibility (%)	Moisture regain (%)
Control	258.37	4.2	1.02	8
Butyl	137.80	10.9	2.62	7.58
Dodecyl	134.0	9.4	2.24	7

parameters of the yarn have significantly been improved which is probably due to the impregnation or add-on effect of wetting agent in the yarn.

Ali [62] carried out sulphonation reaction on jute (*Corchorus capsularis*) fibre for the modification of its intrinsic properties, following the principle of pulping reactions with the neutral sulphite-anthraquinon cooking liquor under different reaction conditions. The sulphonation treatment in the optimal condition improved the fineness in terms of linear density and developed flexibility, softness and compressibility of jute fibre. These values were measured in order to assess the textile importance of sulphonated jute fibre compared to cotton and other textile fibres for fine spinning. It was observed that sulphonation did not impair the tensile properties of the fibre as its tenacities were not changed, rather were improved in certain cases. It was further observed that the extensibility of jute fibre increased up to about four folds of its original value of 1% extension at break and that the fibre moduli decreased and the fibre toughness increased.

The sulphonated jute fibres were mechanically processed to spin into fine yarns of 70 tex and 50 tex using the cotton processing lines. The properties of the sulphonated jute yarn of 70 tex and 50 tex have been shown in table- VII

Table-VII

Properties of yarns prepared from sulphonated jute fibre.

Linear density : A= 70 tex and B= 50 tex

Yarn Sample	Tenacity (g/tex)		Elongation at break (%)		Initial modulus (g/tex)	
	A	B	A	B	A	B
Sulphonated jute	10.4	10.1	2.9	2.4	350	309

The blending of the sulphonated jute fibre with cotton, rayon, acrylic, polyester and silk waste was performed. Three different lots of blend such as 50/50, 65/35 and 80/20 for each fibre component were preferably prepared with 50%, 65% and 80% of the sulphonated jute fibre respectively and 30 tex yarns from each lot of the blend were produced using the ring spinning frame. The physical properties of these yarns were determined. Only the physical properties of 65/35 blend for each fibre component have been shown in table- VIII

Table- VIII

Properties of yarns from S.Jute-Cotton, S.Jute-Rayon, S.Jute-Polyester, S.Jute-Acrylic and S.Jute-Silk waste fibre blends.

Linear density = 30 tex (20 Ne)

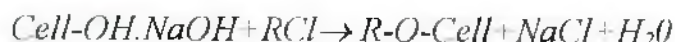
Yarn description	Tenacity (g/tex)	Elongation at break (%)	Initial Modulus (g/tex)	CSP
S.Jute/Cotton (65:35)	10.3	5.8	181	1520
S. Jute/Rayon (65:35)	10.0	5.9	178	1540
S. Jute/Polyester (65:35)	11.0	10.8	150	1590
S. Jute/Acrylic (65:35)	11.2	9.1	169	1540
S. Jute/Silk/Rayon (10:80:10)	11.0	12.5	151	1680

6 ETHERIFICATION

6.1 The principle of etherification

The etherification of cellulose usually consists of the preparation of alkali cellulose by the interaction of cellulose with a base and a

solvating agent and the reaction of the alkali cellulose with the etherifying reagent. The processes in which alkali is not consumed are methylation of hydroxyls of cellulose by diazomethane, addition of alkoxides and addition of olefin activated by polar substituent groups such as nitrile, carboxyl, sulphonyl, to the hydroxyls of cellulose [63]. The etherification reaction may be expressed by the following equation



It is reported [64, 65, 66] that alkylhalides of three or more carbon atoms do not diffuse easily to the reaction zone even at high temperature (140-150° C), instead excessive by products formation and degradation of cellulose occur. If, however, the cellulose is first partially ethylated or methylated, it then reacts readily with the higher alkyl halides to form mixed ethers. Thus case of etherification depends directly upon the rate of diffusion of the reagent to the reaction centre [67,68] and this is in turn depends upon the size of the reagent molecule. Larger molecules can enter if small molecules are used as opening wedges. The efficiency of etherification is poor with the branched chain alkyl halides, because they react only slightly with alkali cellulose.

6.2 Etherification of cellulose with alkyl halides.

Alkyl halides have been used relatively less frequently than alkyl sulphates in commercial practice. The patent [59] by Bayer and Co and

leuchs has shown the method of preparing methyl, ethyl, butyl and other cellulose ethers, apparently for the first time. They showed that the low substituted products were obtained in the fibrous form where as the higher alkyl substituted have lost the original fibrous structure.

7 SOME FACTORS INFLUENCING THE ETHERIFICATION REACTION

7.1 Swelling agent

Cellulose alone does not react appreciably with etherifying reagents. To make the cellulose reactive it is to be treated with a swelling agent and a solvating agent. The usual swelling agent is sodium hydroxide and the solvating agent is water. The various swelling agents other than aqueous sodium hydroxide have been proposed [69]. These include sodium cupricellulose (Norman compound), thallium cupricellulose, organic bases, liquid ammonia and other inorganic bases. In general there is no advantage in cost or performance of other bases over sodium hydroxide. The alternative swelling agents are expensive and they frequently yield products that are difficult to isolate or to purify.

7.2 Concentration of alkali

The degree of substitution increases with the increase of alkali concentration. The etherification reaction depends upon the composition

of alkali cellulose which depends on the concentration of alkali used for immersion and on the degree of pressing to remove the excess of caustic solution. Usually one part of the fibre should yield 3-5 parts, preferably about 4 parts of alkali cellulose [70]. The etherification efficiency decreases as the proportion of water increases [71].

7.3 Reaction temperature

Although substitution increases with the increase of temperature, it is observed that with the rise of temperature the reaction velocity increases more rapidly than the rate of diffusion. The more substituted outer layer forms an insulating jelly shell around unreacted regions, thus preventing diffusion and the reaction comes to stop unless the shell is mechanically broken.

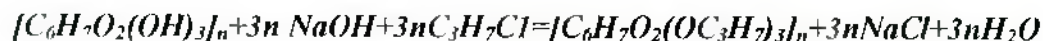
7.4 Solvent

Choice of solvents [72] depends upon the solubility of the carbohydrate derivative. For unsubstituted carbohydrates, MMF or DMS is used, for partially substituted carbohydrate DMF, dioxan or tetrahydrofuran is used.

8 ETHER DERIVATIVES OF CELLULOSE

N.I Nikitin [73] and his workers prepared propyl, butyl and amyl cellulose by the action of the corresponding alkyl halides with ground

filter paper. The following reaction was investigated.



In addition to the above reaction of etherification, a side reaction due to the inter-action of the alkyl halide with sodium hydroxide takes place. This is accompanied by the formation of the corresponding alcohol and a salt or there is a splitting of halogen acid accompanied by the formation of an olefin. Etherification with butyl and isobutyl chlorides yielded only a low degree of etherification while ethyl bromide and isobutyl bromide yielded products containing 2.25 groups per $C_6H_{10}O_5$. Butyl and isobutyl iodide yielded products with only 1 OC_4H_9 group. The etherification of alkali cellulose with butyl bromide gave compounds which corresponded approximately to the formula $C_{12}H_{16}O_6(OC_4H_9)_4$ or $C_{12}H_{15}O_5(OC_4H_9)_5$ and which produced films from benzene solution. Among the isoamyl halides, the bromide gave the best but still an unsatisfactory etherification. Etherification with propyl bromide yield products containing 2.5-3 alkyl groups per $C_6H_{10}O_5$ while the other halides did not yield satisfactory results.

Mishina and his co-worker [74] studied the effect of the general conditions of mercerisation on reactivity of cellulose during ethylation. The swelling of alkali prior to ethylation of cellulose determines the rate of degree of its ethylation. The increase of sodium hydroxide concentration in 10-70 percent range increases cellulose swelling. The addition of urea or zinc oxide to sodium hydroxide solution and also

milling of cellulose increase the degree of ethylation.

They studied the activity of catalysts. They reported that sodium hydroxide and potassium hydroxide were the best effective catalysts for alkylation of cellulose, with sodium carbonate and pyridine being less effective. Acid catalysts were not useful for etherification reaction due to strong cellulose degradation and initiation of side reaction.

An Attempt was made by S. N Danilov et al [75] to prepare water soluble glycerol ethers of cellulose from alkali cellulose and (i) glycerin monochlorohydrin (ii) epichlorohydrin and glycidol. There was no reaction at low temperature in pyridine and charring occurred at higher temperature. They studied the different chemical and physical properties of these ether derivatives.

Plisco and his co-worker [76] studied the effect of the nature of substituents in aromatic sulphonates on the homogenous alkylation of cellulose. They showed that the degree of substitution of cellulose during alkylation with ethyl, heptyl, or decyl aromatic sulphonates depends upon the type of the substituents attached to the aromatic ring and the reaction condition.

Hydroxy ethyl cellulose [77] with good solubility in hot or cold water was prepared by treating alkali cellulose with ethylene oxide. The product having hydroxy ethyl group content 21.4 percent and D.P 550 is completely dissolved in water to give a clear 2 percent solution with

viscosity 400-600 CP.

1, 2 Dihydroxy propyl [78] cellulose was prepared by the treatment of alkali cellulose with glycidol in acetone. The products having D.S.O 20 were obtained.

Kulkarni and his co-workers [79] carried out the etherification of lignocellulose materials, such as jute, wood and the like or its sodium derivatives with an ether forming agent, like monohalo substituted alkanes, monohalo substituted alkyl acids, ethylene chlorohydrin and ethylene oxide. The resultant ethers so obtained were found to claim the use for a multiplicity of end uses such as vehicles for paints and varnishes, soil binders in detergents and as coating and finishing materials for textiles, paper and the like.

9 SOME PROPERTIES OF CELLULOSE ETHER

Compared with cellulose esters, cellulose ethers are stable to acids and alkali [80] depending on the degree of etherification. In addition, these ethers are more resistant to the action of ultraviolet [81] light and possess relatively lower inflammability [82].

The hygroscopic properties of cellulose ethers are mainly a function of the nature and size of the ether radical and of the degree of substitution. The hygroscopicity decreases with the increasing number of carbon atoms in the substituted groups from methyl to ethyl, propyl, butyl

etc. as well as with increasing degree of substitution [83]. Whether more importance must be attribute to the degree of substitution or to the nature of the substituents [84] is still the subject to be investigated.

The influence of the increasing size [84] of the substituents upon the water binding capacity of cellulose ether is shown below:

	Ethyl Cellulose	Butyl Cellulose	Amyl Cellulose
Degree of substitution (Groups per glucose residue)	2.15	2.28	1.91
Moisture absorbed	3.00	1.673	0.975

10 MODIFICATION OF JUTE BY OTHER BLOCKING AGENTS.

The changes in the chemical properties and the fine structure of jute fibres mercerised with liquid ammonia were studied [85]. It is reported that the mercerisation of jute fibre with liquid ammonia increased the extensibility of fibre without loss of strength and is, therefore, more desirable swelling agent than sodium hydroxide. Sodium hydroxide also produces improvement in extensibility but causes loss of strength due to removable of part of hemicellulose. The x-ray, IR and moisture absorption data for the treated fibres were determined.

Chatterjee, S.M [86] studied the effect of processing variable on

benzoylation of jute. They observed that the extent of benzoylation of jute increased progressively with increase in the concentration of benzoyl chloride, reaction temperature and reaction time, reaction media also affected the extent of reaction. It was further observed that dewaxing and decoiling of the jute was the ideal pretreatment for achieving maximum benzoylation with minimum adverse effect on tensile strength and the best results were obtained by treating dewaxing jute with benzoyl chloride in varying concentration in pyridine at $60\pm 2^{\circ}\text{C}$ for a period of 45 minutes.

Esterification of jute fibre with a long chain fatty acid is reported by some workers [87]. Lignified cellulose does not respond to organic base catalysed esterification reaction. However, partial esterification may be effected if the jute fibre is pretreated with alkali at concentrations between 5 and 15 percent and then treated with a fatty acid chloride. The degree of acylation depends on the concentration as well as the total amount of alkali, the amount of moisture, the ratio of the acid chloride to the total alkali and the reaction time. Maximum acylation results when jute fibre is pretreated with 10 percent alkali and the alkali cellulose containing 100 percent moisture is reacted for 3 hours with acid chloride at a molar ratio of about 0.5 of the total alkali present.

11 AIM OF THE PRESENT WORK

Since jute fibre can be spun to make jute yarn and jute yarn can be blended with natural and synthetic fibres, the present work is to see

further blending with other fibres. So the aim of the work is to see the modification of fibres with newer method and the blending of the fibre so obtained.

In view of the above consideration, the following work-plans have been undertaken:

- I. To carry out the etherification of jute fibre with n-butyl chloride at a temperature of 30°C (Temperature is 100°C , when water is used as a solvent) for a period of 2 hours in different solvents.
- II. To investigate the effect of modification due to etherification on the physico-chemical and physico-mechanical properties of jute fibre, which will include the study of degree of etherification, bundle strength, percent moisture regain and percent elongation etc. of the butyl substituted fibres.
- III. To prepare yarns by developing the technology of blending of the etherified jute fibre with cotton, rayon, polyester and silk fibre in order to improve the quality of jute-based yarns comparing with cotton yarns.
- IV. To fine out some textile parameters like tex (count) tenacity, extensibility, initial modulus and C.S.P. etc. of the blended yarns.
- V. To weave the jute-based yarns made from the etherified jute fibre blended with natural or synthetic fibres into light weight fabrics and to determine the fabric construction, fabric properties comparing with cotton fabrics.

EXPERIMENTAL

PART – II A

FIBRE

12 EXPERIMENTAL

12.1 Material

12.1.1 Jute fibre: Raw jute fibre (*corchorus capsularis*) was collected from BJRI (Bangladesh Jute Research Institute) Dhaka. After rejecting the bottom portion (12 cm) the rest of the fibre was cut into pieces (6 cm).

12.2 Preparation of alkyl chloride [88]

12.2.1 Preparation of n-butyl chloride (ZnCl_2/HCl method)

Concentrated hydrochloric acid (200 ml) was taken in a distilling flask (1 litre) and anhydrous zinc chloride (340 g) were added to it. A reflux condenser was fitted into the mouth of the distilling flask and a tube connected to an inverted funnel was attached to the top of the

condenser. The funnel was dipped just below the surface of water (250 ml) in a breaker. n-Butyl alcohol (115 ml) was introduced into the distilling flask and refluxed gently on a wire gauge for 2 hours. The flask was kept inclined during the refluxing period (2 hours). After cooling it was distilled and the distillate was collected up to the temperature of 115°C . The upper layer of the distillate was separated and mixed it with an equal volume of concentrated sulfuric acid and the mixture was refluxed gently for 30 minutes. The n-butyl chloride was distilled from the acid which passed over at 76°C - 79°C . The distillate was washed successively with distilled water (100 ml), 5 percent sodium hydroxide solution (50 ml) and distilled water (100 ml). It was then dried over anhydrous calcium chloride, filtered and distilled. The n-butyl chloride was collected at 75°C - 78°C . The yield was 85%.

13 DETERMINATION OF ALKOXYL GROUP [89, 90] AND DEGREE OF SUBSTITUTION.

13.1 Procedure (Zeisel method)

Moisture free sample (50 mg) was introduced into the reaction flask. Acetic anhydride (3-4 drops), distilled phenol (5-6 drops) and a small bit of red phosphorus were added into the flask. After that freshly distilled hydriodic acid (10 ml; d. 1.7) was added into the reaction flask and attached immediately to the condenser with water supply. Carbon

dioxide was passed through the reaction mixture at the rate of 2-3 bubbles per second and the flask was heated under micro burner, care was taken to ensure that the reaction mixture boiled only very gently during the heating period. The water was run out of the condenser during the last 45 minutes so that the alkoxyl groups might pass over easily to the absorber. The absorption vessel was removed and the contents of the vessel were quantitatively transferred into the iodine flask. The vessel was washed well with distilled water and then with aqueous sodium acetate solution. The washings were collected into the iodine flask. The excess of bromine was destroyed by adding drop by drop formic acid until it was free from bromine (tested with methyl red indicator, colour persisted when bromine was completely destroyed). The sides of the flask were washed with distilled water, potassium iodide (1g) was added into the flask followed by sulfuric acid (10 ml, 10% W/V). The flask was stoppered, shaken well, and allowed to stand for 5 minutes, and the liberated iodine was titrated with standard sodium thiosulphate solution (0.05N) using starch solution (1%) as indicator. The process was carried out identically for the control.

13.2 Calculation of results.

The percent alkoxyl was calculated from the following formula:

$$\% \text{ alkoxyl} = (V-b) \times N \times f \times \frac{100}{W}$$

Where V = Volume (ml) of standard $\text{Na}_2\text{S}_2\text{O}_3$ used for treated sample.

b = Volume (ml) of standard $\text{Na}_2\text{S}_2\text{O}_3$ used for untreated sample.

N = Normality of the standard $\text{Na}_2\text{S}_2\text{O}_3$ solution.

W = Weight (mg) of the sample.

f = factor.

$$\text{Degree of substitution (D.S)} = \frac{1.62 \times \%alkoxy}{M - 0.14\%alkoxy}$$

Where M = Molecular weight of the alkoxy group.

14 DETERMINATION OF MOISTURE REGAIN [91, 92]

The jute fibre was dried at 105°C to constant weight and exposed to a standard atmosphere ($65 \pm 2\%$ R.H., Temperature $20 \pm 2^{\circ}\text{C}$) for 48 hours and again weighted carefully in the conditioned atmosphere. The percent moisture regain was calculated from the following relation.

$$R = \frac{b - a}{a} \times 100$$

Where R = Moisture regain percent.

a = Dry weight of the sample.

b = Moist weight of the sample.

15 PREPARATION OF ALKALI FIBRE [70]

Alkali fibre was prepared by immersing the jute fibre in sodium hydroxide solution of 10% and 20% strength (w/v) separately and allowed

to ripen for 3 hours. Excess of alkali was removed by squeezing so that the weight of the alkali fibre was 3 times of the weight of jute fibre.

16 DETERMINATION OF ELONGATION AND BUNDLE STRENGTH OF JUTE FIBRES BY STELOMETER [93]

The bundle strength and the percent elongation of the jute fibre were determined in the following way.

16.1 Conditioning of the jute sample:

The jute sample was conditioned for 24 hours in the standard atmosphere ($65 \pm 2\%$ R.H., Temperature $20 \pm 2^\circ\text{C}$)

16.2 Preparation of specimen

A group of fibres were grasped on the fibrograph comb using the stelometer clip to grip the fibres at a point at least $\frac{5}{8}$ inch (15 mm) from the teeth of the comb. These fibres were then pulled through the fibrograph comb teeth three to four times to straighten them and the loose fibres were removed using tweezers. The specimen was then ready to be placed in the clamps.

16.3 Placing the specimen in the clamps.

The test specimen was placed in the fibre clamps in the stelometer vise and the clamps were locked in the vise and then opened. The fixed clamp was raised on the vise and inserted the loose ends of the flat bundle specimen held in the sample clip. The sample clip was drawn onward until it fell into place over the hook of the tension lever. Sufficient pressure was applied on the fixed clamp to prevent fibre slippage and released the spring level to apply tension on the specimen. The clamp was tightened furthest from the clip first to ensure correct tension between the clamps and then removed the clamps from the vise.

16.4 Procedure.

The clamps with the specimen were placed in the stelometer (manufactured by the special Instrument Laboratory Inc. Knoxville, Tenn.) previously calibrated and the trigger was released to start both the force and elongation indicators moving across the scales. After the fibres were broken, the force and the elongation indicators were read to the nearest 0.01 kg and 0.1 percent respectively. Sixteen readings for force and elongation for each of the test specimens of same sample were taken at $\frac{1}{8}$ inch gage length and at zero gage length separately. The observed reading was multiplied by a factor of 0.8 to compensate for the slippage of the fibre in the clamps.

16.5 Calculation of results.

The percent elongation and the bundle strength were calculated from the following formulas:

(i) True elongation (%) = Elongation (%) at $\frac{1}{8}$ inch gage length - elongation (%) at zero gage length

(ii) Bundle strength in g per tex = $\frac{\text{Breaking load}}{\text{Weight in mg}} \times 14.9$

17 PRELIMINARY TREATMENT OF JUTE FIBRE

17.1 Treatment of jute fibre with sodium hydroxide solution at different times.

Jute fibre was immersed in sodium hydroxide solution (10% strength, w/v) at 30°C for different times. It was then washed successively with cold and hot water till alkali free. It was dried in air and stored in stoppered bottle. Similar experiments were carried out with 20% alkali. The bundle strength, the percent elongation and the percent moisture regain of the treated fibre were determined. The results are shown in the table IX

17.2 Treatment of jute fibre with sodium hydroxide solution of different strengths.

Jute fibre was treated with sodium hydroxide solution of (% w/v) different strengths at 30°C for 2 hours. After washing and drying percent elongation, bundle strength and percent moisture regain of each treated sample were determined. The results are shown in table X.

17.3 Treatment of jute fibre with n-butyl chloride alone at 30°C at different times.

Jute fibre was treated with n-butyl chloride (the ratio of fibre to n-butyl chloride, 1:1 w/v) at 30°C for different times. It was then dried in air. The percent elongation, bundle strength and percent moisture regain of each treated sample were determined. The results are shown in table XI.

17.4 Treatment of jute fibre with n-butyl chloride using different n-butyl chloride/fibre ratios.

Jute fibre was pretreated with sodium hydroxide solution of 20% strength and then etherified with n-butyl chloride using different n-butyl chloride/fibre ratios. The time of etherification for each sample was 2 hours and the reaction temperature was 30°C. The degree of substitution, percent elongation, bundle strength and percent moisture regain were determined. The results are shown in the table XII.

17.5 Etherification of jute fibre with n-butyl chloride in different solvents.

Jute fibre was pretreated with sodium hydroxide solution of 20% strength and then etherified with n-butyl chloride (the ratio of fibre to butyl chloride 1:1 w/v) at 30°C for 2 hours in different solvents. The solvents were petrol (b.p. 80-120°C), ethyl alcohol (95%) and water. The reaction temperature was fixed at 100°C when water was taken as a solvent. The methods of etherification are described below.

17.5.1 Etherification of jute fibre with n-butyl chloride in petrol medium.

To a steel vessel containing alkali fibre and petrol (volume ratio n-butyl chloride/petrol = 1:5), n-butyl chloride was added. The mouth of the vessel was kept air tight with a lid. The etherification was carried out

at 30°C for 2 hours. The contents of the vessel was stirred occasionally. After two hours the fibres were taken out of the vessel. It was then washed successively with cold and hot water till alkali free and dried in the air. The degree of substitution, percent moisture regains, percent elongation and bundle strength of the treated fibre were determined and these properties of the fibre have been shown in table XIII.

17.5.2 Etherification of jute fibre with n-butyl chloride in alcohol medium:

Alkali fibre was taken in a steel vessel containing ethyl alcohol (volume ratio n-butyl chloride/ethyl alcohol = 1:5) and butyl chloride. The reaction was performed at 30°C for 2 hours. The mouth of the vessel was kept air tight with a lid. The whole mass of the steel vessel was agitated from time to time. The resulting jute fibre was washed first with cold water and then with hot water till alkali free. It was then dried in the air. The degree of substitution, percent moisture regain, percent elongation and bundle strength of the treated fibre were determined and these properties of the fibre have been shown in table XIV.

17.5.3 Etherification of jute fibre with n-butyl chloride in water medium:

n-Butyl chloride was treated with alkali fibre and 3% aqueous sodium hydroxide [volume ratio n-butyl chloride/aqueous sodium

hydroxide (3%) = 1:5] in a digester at a temperature of 100°C and pressure 5 kg/cm^2 for 2 hours. The digester rotated upside down two times a minute. The jute fibre was then taken out of the digester. It was washed with cold water and then with hot water till alkali free and dried in the air. The degree of substitution, percent moisture regain, percent elongation and bundle strength of the treated fibre were determined and these properties of the fibre have been shown in table XV.

PART -II B

SPINNING

PREPARATION OF YARN BY THE BLENDING OF ETHERIFIED JUTE FIBRE WITH THE NATURAL OR SYNTHETIC FIBRE

18 EXPERIMENTAL

18.1 Making of yarn.

Yarn was made by blending of the etherified jute with cotton, rayon, polyester and silk fibre respectively following the standard processing system - cotton system spinning. The jute staples prepared after cutting the long jute into a length of 35 mm, were processed like cotton.

18.2 Cotton system spinning -the blended yarns .

The etherified jute fibres were cut into a length of 35 mm, using a cutting machine. The jute staple so made were processed through the

mechanical processing units of a miniature cotton spinning machinery. Yarns having count (30 tex) was prepared by blending the etherified jute fibre with cotton or synthetic fibres .

18.2.1 Jute /Cotton

The etherified jute staples were blended with cotton in the ratio of 50: 50 respectively. The fibres of the jute staples were individualised by passing through the opener machine and were then mixed up with cotton samples in the required proportions, which were then processed in the mini carding machine. Several passages through the carding machine were made for intimate blending of the component fibres. The card slivers were then fed to the mini drawing machine and were spun to 30 tex yarns.

18.2.2 Jute / Rayon

The etherified jute fibre and rayon filaments were stapled separately into equal length of 35 mm and were processed through the mini carding machine. A blend (50:50) of jute - rayon was prepared and 30 tex yarns were spun.

18.2.3 Jute / Polyester

The spinning of 30 tex yarn from the jute - polyester blend (50:50) was made possible with some modification of the process. The

blend was carded well, but condensing of webs to slivers were done with difficulty. Several passages through the mini carding machine were necessary for better mixing of the fibre components before getting uniform slivers. Some times doubling of the slivers was needed to process in the mini drawing frame in order to get even densities of roving slivers in the mini process machine. The yarn were prepared successfully in the ring frame having the same draft and bobin speed .

18.2.4 Jute/Silk

The silk filaments were collected from Seri-culture Institute. Rajshahi. The staples of the etherified jute fibre were blended with the silk fibre and for better carding some rayon staples were mixed up. The sliver finally contains jute-silk-rayon in the ratio of 10:80:10 respectively, from which 30 tex yarn was spun.

19 MEASUREMENT OF COUNT.

210 yds of yarn in length winding on the laboratory reeling frame were weighed and an average of ten such readings were taken to measure the count of the yarn. The yarn counts measured in both jute system i.e. number of pounds in 14400 yds and cotton system i.e. number of hanks (840 yds) in a pound were expressed in the International unit of 'Tex' which measures number of grams in one kilo meter length of the yarn . The count in tex was taken as a measure of linear density of the yarn.

20 MEASUREMENT OF TENSILE PROPERTIES

The yarns were tested in Instron Tensile Tester for measurement of the tensile properties which included tenacity, breaking elongation and initial modulus at 1% elongation. For each experimental value, 10 yarn samples were tested with the tensile tester machine at 10 cm gauge length with a pretension load of 0.4N and at a constant rate of elongation, 5 cm per minute.

21 THE COUNT-STRENGTH PRODUCT (CSP)

The concept of CSP may be used to derive an index by which the spinning quality of a fibre or the spinning efficiency of a particular system may be assessed. It was obtained as the product of cotton count and Lea strength of the yarn. A hank of yarn, with its starting and finishing ends knotted, was placed over the hooks of the Lea tester, pendulum lever type, made by Good brand & Co Ltd. As the lower hook descend a load was imposed on the loops of the yarns constituting the hank. The maximum load to break all the threads unraveling the hank was indicated on the dial, which was the 'Lea strength' of the hank.

PART -II C

FABRICS

PRODUCTION OF FABRICS WITH ETHERIFIED JUTE BLENDED YARNS

22 EXPERIMENTAL.

22.1 Preparation of fabrics.

Three different samples of fabrics each measuring about 10 meter in length and 1 metre in width were made by weaving on the ordinary power looms of m/s N.K. Textile Mills. Gazipur. The weaving was performed with the blended yarns made of the etherified jute fibre in the ratio of 50:50 with cotton, rayon and polyester respectively. The set of warp and weft yarns was done on the one up and one down principle followed for weaving plain or square cloth. The description of the fabric construction has been shown in table-XXV in the discussion chapter.

23 CLOTH COVER.

The cloth cover describes the fabric construction showing to what extent the warp and the weft yarns are closely woven and was obtained from the relationship

$$K_c = K_1 + K_2 - K_1 K_2 / 28$$

Where K_c = cloth cover

K_1 = warp cover factor

K_2 = weft cover factor

The fraction of space per inch of cloth covered by warp yarn is known as warp cover factor which was obtained as.

$$K = \frac{\text{thread per inch}}{\sqrt{\text{Count}}} = \frac{n}{\sqrt{N}}$$

Similarly, the weft cover factor was calculated. The thread per inch n were counted with the help of a traveling thread counter.

24 STIFFNESS AND FLEXURAL RIGIDITY.

The stiffness of a fabric is a measure of resistance to bending of a strip of cloth of unit width bent into unit curvature in absence of any tension. This was achieved simply by measuring the bending length of a fabric specimen with the help of a Shirely Stiffness tester, 9DL 3A, conforming to the requirements of British Standard 3356: 1961. A strip of

cloth 6 inch $\times$ 1 inch cut to the size of the template (i.e. the scale), of the tester was slid in a direction parallel to its length, so that its end was projected from the edge of the horizontal platform of the tester.

The length of the overhang cloth was measured when the tip of the test specimen was depressed under its own weight to the point where the line joining the tip of the horizontal platform made an angle of 41.5 degree with horizontal plane. The bending length, C was then obtained as follows.

$$C = \frac{L}{2}$$

where L = the overhang fabric length.

Each specimen was tested four times, at each end and again with strip turned over. Mean values for the bending length in warp and weft directions were calculated.

The flexural rigidity which is a measure of stiffness associated with handle of cloth was determined from the following relationship .

$$G = WC^3 \times 10^3 \text{ mg/cm} = \text{mNmm}$$

where C = bending length

W = cloth weight (gm) per square cm.

N = Newton

25 STRENGTH MEASUREMENT

The tensile strengths of the fabric samples were measured on a Tensile Strength Tester with a clamp speed of 30 mm/min and specimen length of 5 cm. Two specimens per sample of fabric were prepared for the test. In the experiments, the Z wick Fabric Tensile Strength Tester was used .

26 DRAPING PROPERTIES

Drape may be broadly defined as the ability of a fabric to give a graceful appearance in use. The warp and weft way characteristics of a fabric interact and produce graceful folding of cloth as it is draped over any circular support. The draping properties of the fabric specimen under test were measured following the procedure of the Fabric Research Laboratories of U.S.A.[94].

The fabric sample was cut into a circular specimen about 10 inch in diameter and was supported on the circular disk about 5 inch diameter of the Drape meter. The circular specimen was draped over the circular disk supporter of the Drape meter and assumed some folded configuration. The line of the projection from the periphery of the fabric specimen was not circular, rather it took the shape of the folded

configuration. The drape was measured as the drape coefficient F which is the ratio of the projected area of the draped specimen to its undraped area, after deduction of the area of the supporting disk. Thus

$$F = \frac{A_s - A_d}{A_D - A_d}$$

Where A_D = the area of the specimen .

A_d = the area of the supporting disk, and

A_s = the actual projected area of the specimen.

RESULTS AND DISCUSSIONS

PART-III A

FIBRE

RESULTS AND DISCUSSION

27 PRELIMINARY INVESTIGATIONS ON JUTE FIBRES

Jute fibre (*corchorus capsularis*) was collected from BJRI (Bangladesh Jute Research Institute), Dhaka and the comparatively hard bottom part (12 cm) was discarded. The remaining part was cut into pieces (6 cm). It was cleaned manually to remove the hard patches and sun dried and used as jute fibre for all investigations.

n-Butyl Chloride was prepared by standard method.

The alkoxy groups were determined by Zeisel method and the percentage of alkoxy groups was reported after deduction of the alkoxy content of the jute fibre.

The bundle strength and the percent elongation of jute fibres were determined by stelometer.

The percent moisture regain of jute fibres was determined by standard method. Alkali fibre was prepared for etherification and the following preliminary investigation for jute fibers were carried out to find the suitable parameters.

27.1 Effect of time of swelling on some properties of jute fibre.

Preliminary investigations were carried out with jute fibre by treating the jute fibre with sodium hydroxide (10% and 20% w/v) separately at different times so as to find out the effect of time on swelling of the fibres.

Table-XVI

Effect of time of swelling on some properties of jute fibre.

Time of swelling (hour)	Strength of NaOH (10%)			Strength of NaOH (20%)		
	Elongation (%)	Bundle Strength (g/tex)	Moisture regain (%)	Elongation (%)	Bundle Strength (g/tex)	Moisture regain (%)
Control	1	17.73	8.7	1	17.73	8.7
1	1	16.51	8.9	2.6	16.30	9.3
2	1.7	16.30	9.3	4.1	15.11	10.4
3	2.1	16.10	9.7	4.0	15.0	10.6
5	2.3	15.60	10.1	4.3	14.63	10.8
10	2.7	14.12	10.4	4.4	13.51	11.3

From table XVI it is seen that with 10% alkali treatment there is slight increase in percent elongation of the fibre while the bundle strength decreases with the increased time of swelling. This is compatible in that as the alkali action is increasing, the compactness or crystallinity is decreasing, hence the bundle strength is falling. This is further evidenced with moisture regain properties of alkali treated fibres (*Pages 81, 83*). The behavior of the 20% alkali treated jute fibre is following the same pattern as found with 10% alkali treated fibres. The percent elongation is increased from the first hour and continues to increase as the time of alkali treatment increases. This is due possibly to the increase in fibre diameter and cell wall thickness which sustain to increase the percent elongation.

27.2 Treatment of jute fibre with sodium hydroxide solution of different strength.

The treatment of jute fibre with sodium hydroxide solution of different strengths shows (Table- XVII) that with the increase of alkali concentration, the percent elongation increases while the bundle strength decreases.

Table-XVII

Effect of alkali concentration on jute fibre.

Strength of NaOH (%w/v)	Elongation (%)	Bundle Strength (g/Tex)	Moisture regain (%)
0	1	17.73	8.7
2	1	17.51	9.0
5	1.4	17.0	9.3
10	1.9	16.62	9.8
15	2.5	16.10	10.1
20	4.1	14.89	10.3
30	4.6	13.37	11.5

This is quite obvious that with the increased concentration of alkali the inter molecular/inter chain hydrogen bonds are loosen at the expense of bundle strength and consequent increase in percent elongation. The moisture regain increases with the increase of alkali concentration which is due to increased amorphous character of the fibre.

27.3 Treatment of jute fibre with n-butyl chloride alone at 30°C in different times.

The jute fibre was treated with n-butyl chloride alone at 30°C in different times. From table - XVIII it is seen that time of treatment has practically no effect on elongation and bundle strength although there is a

considerable improvement of moisture regain properties. This indicates that butyl chloride has not reacted in any way with jute fibre.

Table -XVIII

Effect of the treatment of butyl chloride alone on elongation, bundle strength and moisture regain of jute fibre.

Time of butyl chloride treatment (hour)	Elongation (%)	Bundle Strength (g/tex)	Moisture regain (%)
1	1	17.70	6.7
2	1	17.1	6.4
4	1.5	16.8	6.1
8	1.6	16.3	5.7

Loss in moisture regain property is probably due to impregnation or add-on effect of butyl chloride on the fibre.

27.4 Effect of jute fibre and n-butyl chloride ratio

The jute fibre was pretreated with 20% sodium hydroxide. Etherification reactions were then carried out with different proportions of fibre and butyl chloride in petrol medium at 30°C. It is observed (Table - XIX) that the degree of substitution is maximum in the jute-butyl chloride ratio 1:1 and 2:1 in two hours reaction time.

Table-XIX

Etherification of jute fibre with butyl chloride using different fibre: butyl chloride ratios, the reactions time being 2 hours at 30°C

Jute fibre-butyl chloride ratio (w/v)	OC ₄ H ₉ (%)	D.S	Elongation (%)	Bundle strength (g/tex)	Moisture regaing (%)
1:1	0.98	0.021	9.7	13.21	5.6
2:1	0.97	0.021	9.5	13.10	5.8
3:1	0.90	0.020	9.2	12.92	5.9
10:1	0.79	0.017	8.6	12.70	6.3
15:1	0.71	0.015	8.4	12.51	6.5
20:1	0.60	0.013	8.2	12.43	6.7

It is seen that as the proportion of fibre to etherifying agent increases, the degree of substitution decreases. The bundle strength is remaining more or less unaffected. The moisture regain decreases (control has 8.9% moisture) with the increase of substitution. This is due to the increasing hydrophobic character of the substituents on the jute fibre.

27.5 Etherification of jute fibre with n-butyl chloride in different solvents.

Alkali jute fibre (20%) was etherified with butyl chloride in different solvents. The degree of substitution, percent moisture regain,

percent elongation and bundle-strength were determined in each case. It is seen from the table XX that the solvents like petrol, ethyl alcohol and water have marked effect on etherification and consequently on bundle strength, percent moisture regain and percent elongation. In the case of petrol as a solvent a high value of D.S., percent elongation and bundle strength in comparison with that of alcohol and water were obtained. By products formation took place in water and alcohol medium. For this reason a low D.S value was obtained in these media.

Table-XX

Effect of solvents on the degree of substitution, percent moisture regain, percent elongation and bundle strength of the butyl substituted jute fibre

Solvent	Reaction temp. 0°C	OC ₄ H ₉ (%)	D.S	Elongation (%)	Bundle strength (g/Tex)	Moisture regain (%)
Control	-	-	-	1	17.70	8.9
Petrol (b.p. 80-120C°)	30	0.98	0.021	9.75	13.27	5.1
Ethyl alcohol (95%)	30	0.21	0.004	7.20	9.73	7.5
Water	100	0.10	0.003	5.30	8.62	8.3

We have tried three solvents to see whether these solvents are industrially feasible or not. In the petrol medium we have got encouraging results. So petrol is a better solvent than any other tried for. Hence subsequent investigation will be based on petrol medium.

PART - III B

SPINNING

RESULTS AND DISCUSSION

28 FIBRE BLENDS AND BLENDED YARNS.

28.1 Etherified jute fibre blended with cotton.

The etherified jute fibres were stapled to match the cotton staples and were mixed together in the ratio of 50:50. Careful attention was paid to have regular web formation during carding, which ensured uniform blending of the fibre components of the blend. The properties of the blended yarn have been shown in table XXI

Table- XXI

Properties of etherified jute-cotton blended yarn.

Linear density = 30 tex (20 Ne)

Yarn sample	Tenacity (g/tex)	Elongation at break (%)	Initial Modulus (g/tex)	CSP
Jute/Cotton 50/50	11.1	6.2	184	1638
Cotton 100%	11.5	6.6	171	1694

It will be seen from table XXI that 50/50 blend of etherified jute and cotton give yarns having its mechanical properties very much close to 100% cotton indicating better compatibility of the mixed up component fibres.

28.2 Etherified jute fibre blended with rayon

The etherified jute fibre and rayon fibre were mixed up in a comparatively better form than any other blends, which was probably due to the individual fibre characteristics of both the fibre staples in achieving intimate blending. The yarn properties have been shown in table XXII

Table - XXII

Properties of etherified jute and rayon blended yarn.

Linear density = 30 tex (20 Ne)

Yarn sample	Tenacity (g/tex)	Elongation at break (%)	Initial Modulus (g/tex)	CSP
Jute/Rayon 50/50	10.9	6.4	181	1605

It will be seen from table- XXII that the properties of the jute/rayon blended yarn are mostly similar to those of the etherified jute/cotton blended yarn. The rayon staples being regenerated cellulosic fibres seemed to have similar compatibility for inter-mingling with the etherified jute fibre like cotton.

28.3 Etherified jute/polyester blended yarn.

The blending of etherified jute with polyester staples was an unique achievement, although the process was difficult. The success was very effectively made to mix up 50% of the etherified jute fibre with 50% of polyester fibre to produce very regular yarn. The properties of the blended yarn have been shown in table XXIII

Table- XXIII

Properties of yarn from etherified jute fibre and polyester fibre blend.

Linear density of yarns = 30 tex (20 Ne)

Yarn sample	Tenacity (g/tex)	Elongation at break (%)	Initial Modulus (g/tex)	CSP
Jute/polyester 50/50	11.9	12.7	147	1613

28.4 Etherified jute fibre/silk yarn

Mixing of the etherified jute with silk only was difficult, which was over come by adding up to 10% rayon staple with the blend. Finally the blend having 10% etherified jute, 10% rayon and 80% silk seemed to be comparatively more uniform and yarn having count 30 tex was spun on ring frame. The properties of the yarn have been shown in table XXIV.

Table-XXIV

Properties of the yarn from etherified jute/silk fibre blend.

Linear density = 30 tex (20 Ne)

Yarn sample	Tenacity (g/tex)	Elongation at break (%)	Initial Modulus (g/tex)	CSP
E.Jute/Silk/Rayon 10:80:10	11.2	12.4	150	1682

It will be seen from the table- XXIV that the properties of E. Jute/Silk/Rayon blended yarn are very much comparable to those of the blended yarn from E. Jute/Polyester. The yarn from the binary blend of E. Jute/Silk was made with some success, but the quality of this yarn was too irregular to report. This was due to very unequal distribution of the fibre components in the yarn cross section, which was however improved when rayon staples up to 10% were incorporated in the fibre combination.

The etherified jute was prepared by SN_2 mechanism. The method is expensive because of the price of the solvent, petroleum ether. Although the product is beautiful but does not show any remarkable variation as compared to other methods [62]

PART - III C

FABRICS

RESULTS AND DISCUSSIONS

29 The Fabric Construction

The plain weave fabrics made from the etherified jute yarns blended with cotton, rayon, and polyester have shown a very good prospect of performance as they are comparable to the structure of cotton fabrics.

The details of the fabric samples prepared from the etherified jute yarns blended with cotton, rayon, polyester have been shown in table XXV.

Table- XXV

Details of fabric construction from etherified jute yarns blended with other fibres.

Fabric Sample	Yarn Composition (blends ratio)	Yarn count (tex)	Thread per cm	Cover factor	Cloth cover	Wt. g/m ²
A	Etherified jute fibre/cotton (50:50)	warp 30 weft 30	ends 16 pick 16	warp 9.17 weft 9.17	15.34	136
B	Etherified jute fibre/rayon (50:50)	warp 30 weft 30	ends 16 pick 15.5	warp 9.17 weft 8.88	15.15	139
C	Etherified jute fibre/polyester (50 : 50)	warp 30 weft 30	ends 16 pick 15.7	warp 9.17 weft 9.0	15.23	135
D	Cotton 100%	warp 30 weft 30	ends 16.2 pick 16.2	warp 9.28 weft 9.28	15.49	109

The structure of a fabric plays an important part to determine the fabric properties. The plain weave structure was chosen for fabric construction from the etherified jute yarns blended with other fibres, because maximum fabric about 70% are of plain structural pattern. From table XXV it will be seen that each kind of fabric was woven with equal set of warp and weft maintaining optimum spacing in the intersection of the yarns. The variations in the number of threads per unit length of fabric were in correspondence to the yarn tex, but the cover factors for both warp and weft and their resulting effect on the fabric was maintained

almost identical. The weights per unit area were, however different for different cloths in accordance with the quality of yarns used. The geometrical properties of the fabrics from blended etherified jute fibre were favorably compared with those of the fabrics from 30 tex cotton yarns.

30. Mechanical properties.

The results on the mechanical properties of the fabric samples woven with the etherified jute yarns blending with cotton, rayon and polyester have been shown in table XXVI

400937

Table XXVI

Mechanical properties of fabrics prepared from etherified jute fibre yarns blending with cotton, rayon and polyester.

Sample No	Fabric composition	Bending length (cm)	Flexural rigidity (mNmm)	Tensile Strength (kgf)	Drape co-efficient
A	Etherified jute/cotton (50:50) blend, 30 tex yarn	1.8	8.7	20.1	37.1
B	Etherified jute/rayon (50:50) blend, 30 tex yarn	1.9	10.1	20.3	38.03
C	Etherified jute/polyester (50:50) blend, 30 tex yarn	2.0	11.47	21.6	40.25
D	Cotton (100%), 30 tex yarn	1.6	4.36	22.5	35.30

It was shown in the previous chapter that the etherified jute fibre blending with cotton, rayon and polyester respectively has got adequate flexibility to be spun into fine yarns. The light weight fabrics were made with these blended 30 tex yarns. The mechanical properties of these fabrics were determined to observe their serviceability in the practical usages and to ascertain their suitability as jute blended cotton and synthetic fabric. Shirting's and suiting are usually made from cotton, wool, silk and synthetic yarns or the blended yarns there of. The use of jute after having etherified in the field of making such fabrics has been a possibility. The result shown in table XXVI indicate that the softness and handling characteristics represented by the bending length and flexural rigidity of the blended fabric were very much comparable to those of cotton fabrics with identical fabrics structure. The bending length and flexural rigidity of fabric samples prepared from etherified jute fibre blended with cotton were 1.8 cm and 8.7 mNm respectively, which were only 0.2 and 4.3 points higher than those of the cotton fabrics and were in proximity to the values for other blended fabrics.

The strength properties of the fabric samples from fibre blends were in the range of 20.1 to 21.6 kgf which were evidently very near to 22.5 kgf strength of cotton fabrics. It signifies that the durability and serviceability of the fabrics under any sort of stress and deformation during their uses are not much less than the cotton fabrics.

The etherified jute blended fabrics draped very elegantly in almost the same way the cotton fabrics draped over a circular support. The table XXVI shows that the drape co-efficient of the blended fabrics were found in the range of 37 to 40 which were 2 to 5 point higher than that of cotton cloth. This indicated that the blending of etherified jute fibre with cotton or any other flexible textile fibres improved the draping properties of the cloth made there from and that these fabrics in actual use would have gracious look and firmness almost to the same extent of those draping properties of cotton fabrics.

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