Enhancement of Physical, Mechanicaland Morphological Properties of Coir by Mercerization and Acetylation

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Abstract:- Intent of the research is surface treatment of the coir fibre for bestowing hydrophobic properties and augmenting its strength for contriving coir products and composites. Sodium hydroxide and acetic acid of varying percentages of 5 to 20 were employed in surface treatments. An increment around 35% for mercerization and 24% for acetylation is discern in tensile strength, cutback of 15% is perceived in flexural rigidity after incorporation of 10% NaOHin mercerization whilst acetylation increases, and a reduction of 20 % in hydrophilicity is surveilled for both treatments compared with control. Fibres picked up for dyeability and check the colour fastness, the outcome stipulate that the treated fibres improve colour fastness towards light. Variables such as scorage, runnage and twist of 2 ply yarn spawn from treated fibres were scrutinized along with control and from scanning electron microscopic studies it's obvious that even a minimal of 10% addition of NaOH/CH_3COOHshows vouchsafe properties that could be employed in production of coir products and composites.

Keywords:- *Dyeability, Flexural Rigidity, Runnage, Scorage, Spinnability, Tensile Strength, Twist.*

I. INTRODUCTION

Coir fibre procured from the husk of the coconut (*Cocosnucifera*), enjoys a premium position among various natural fibres on grounds of its strength, toughness, durability, natural resilience and versatility in application. Coir is a multicellular bio polymer with crystalline cellulose methodized helically in a matrix comprising of non-crystallinelignocellulosic complex[1]. The high crystalline alpha cellulose and lignin content of coir fibre makes it strong and resistant to borer, termite and other natural elements. Due to hydrophilic nature and low thermal stability coir fibres have got certain constraints. Diverse chemical methods can be used for ameliorating the properties of coir fibre such as softness, fastness properties, tensile properties and affinity towards the binders for upgraded performance of the coir fibre for industrial applications. Naturalfibres have the meritssuch as bio-degradability, less noisy, renewable, low density, reduced wear of tooling, high degree of flexibility, high specific strength and moreover own no health hazards[2, 3, 4].

Mercerization is commonly used as a finishing treatment in cotton with strong caustic alkaline solution in order to enhancelustre, hard and other properties[5,6].In coir fibre, the process promotes the removal of partially amorphous components such as hemicellulose, lignin, waxes and oil soluble in alkaline solution there by lower the level of fibre congregation, making the surface bumpy[7]. During the alkaline treatment, the OH groups present in the fibres react with NaOH had a long-lasting effect on the mechanical properties of flax fibers, mainly on fiber strength and stiffness.

$$\text{Fibre-OH} + \text{NaOH} \rightarrow \text{Fibre-O-Na}^{+} + H_2O$$

As a result, mercerization had a long-lasting effect on the mechanical properties of flax fibers, mainly on fiber strength and stiffness. Acetylation treatment on natural fibre is generally known as esterification method for plasticizing of cellulose fibres[7]. Fibres are acetylated with an acid to graft acetyl groups on to the cellulose structure.

$$\text{Fibre-OH}+CH_3\text{CO-OH}\rightarrow\text{Fibre-O-COCH}_3+H_2O$$

Treatment comes up with rough surface topography withless number of voids that gives a sophisticated distinctive interlocking with the matrix. It reacts with hydrophilic hydroxyl groups and undulate the fibre cell wall. As a result, the hydrophilic nature of the fibre diminishes, leading to improvement in dimensional stability of coir Composites[8,9].

II. MATERIALS AND METHODS

2.1 Materials

Mechanically extracted coir fibres were congregated from Alleppey, Kerala. Sodium Hydroxide (NaOH), Acetic acid (CH_3COOH), Hydrochloric acid (HCl) and Sodium chloride (NaCl) used were of analytical grade. For dyeing acid orange, acid green, malachite green, rhodamine, direct sky blue &direct brown used were of commercial grade.

2.2 Methodology

The fibres were initially cleansed in willowing machine and sort out using combing board.

2.2.1 Mercerization treatment

Fibres were immersed in sodium hydroxide solution of varying percentage of5, 10, 15 & 20 kept overnight with occasional stirring. For neutralisingp Hit'sthen dipped in0.2 N dil. HCl for half an hour, washed with running water and air dried. Water/liquor ratio were maintained at 1: 24 at room temperature[5].

2.2.2 Acetylation treatment

Fibres were immersed in varying concentration of 5, 10, 15 & 20 percentage acetic acid solution for 6 hours with occasional stirring, washed with running water and air dried. Water/liquor ratio were maintained at 1: 24 at room temperature[6,8].

2.2.3 Dyeability

Mercerized fibres along with control were dyed with acid, basic and direct dyes. Dye bath assistant used were 2%H_2SO_4 foracid dyeing, 2% CH_3COOH for basic dyeing and 10% NaCl for direct dyeing in the material liquor ratio 1:24[10,12].

2.2.4 Spinnability

The spinning of mercerised and acetylated coir fibre along with control into 2 ply yarn by hand spinning using motorised spinning wheels and the mechanical and tensile properties were looked over.

III. PHYSICAL PROPERTY EVALUATION

3.1. Tensile properties

Tensile properties of coir fibre were determined using UTM (Universal Testing Machine) Shimadzu AG-X/R as per IS 235-1989 standard. Test parameters; for fibre: strain rate = 10 mm/min, gripping length = 5 cm at atmospheric temperature.

3.2 Flexural rigidity

Flexural rigidity of the fibreswas tested using the flexural rigidity tester. Treated and rawfibres were tied around a PVC pipe of 2-inch diameter to attain the shape of a ring. After 24 hours, the rings were tested using the flexural rigidity tester with and without load (1 g), the ring diameter and deformation of ring on loading was recorded. The average deflection on loading was deliberated[9, 10, 11]. The flexural rigidity was calculated as:

Flexural rigidity

$$=0.0047mg\,(2\Pi r)^{2}\,(\cos\theta/\tan\theta)\ gcm^{2}$$

Where,

mg = Weight of load applied in grams

r = radius of the ring in cm

d = deformation of lower end of ring

θ =493d/2Πr

3.3 Density

Fibre density can be more conveniently determined indirectly by comparing the sample with standards of a known density. The frequently used technique for indirect determination of fibre density is sink float method [13].

For this method, known densities of two liquids were handpicked. The liquids miscible and inert to the fibre are being selected. One liquid must be less dense than the fibre and the other liquid must be denser than fibre. Water (density=1) and chloroform (density=1.48) were chosen. A known volume of liquid having density lesser than the fibre was taken in beaker and the fibre was immersed. Then liquid was added drop wise to the beaker with constant stirring. When the density of the solution precisely equalsthe fibre, the fibre will neither sink nor float but will remain suspended in the liquids[11]. The density of the fibre is evaluatedas:

Density of the fibre (d)

$$= \frac{d_A V_A + d_B V_B}{V_A + V_B}$$

Where d_A, d_B& V_A,V_B are the densities and volumes of the liquids used.

3.4 Water absorption

Water absorption of treated and raw coir fibres were determined by immersing it in distilled water in a beaker at room temperature for different time durations. The specimens were taken out from the water and all surface water was abolished after 24hr immersion.

The water absorption was calculated by using weight difference. The percentage weight gain of the samples was measured[11,15].Water absorption of the treated and raw coir fibre was determined by using the relationship as follows.

Water absorption $WA = \frac{(W_1 - W_2)}{W_2} \times 100$

Where,

W_1=weight of sample after immersing in water

W_2= dry weight of the sample

3.5 Colour fastness

Xeno test was employed to ascertain colour fastness of the sample using 'Blue wool' standard fabrics and this is a consistent stipulation determining light fastness swiftly. The tests were carried out byAATCC-16H standard, humidity -30% and temperature 60^0C[10,11, 14].

IV. MORPHOLOGICAL EVALUATION

4.1Scanning electron microscopy (SEM)

Scanning Electron Microscopy studies of treated and raw coir fibre was carried out on Scanning Electron Microscope (SEM) (JEOL JSM-6390 LV) system with an accelerating voltage of10 KV.

V. MECHANICAL PROPERTY EVALUATION

5.1 Scorage, Runnage Twist and Tensile strength of yarn

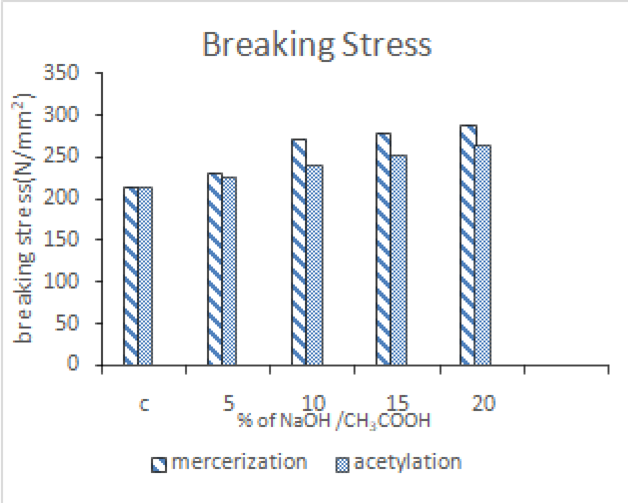
Scorage of yarn indicates fineness or coarseness of coir yarn which was obtained by dividing the number of strands that could be laid very close to each other without overlapping in a length of 0.914 metre or 36'' or one yard by 20[12]. Runnage is the length of yarn in meter per kg or feet per pound[12]. Twist is the number of turns about the axis of a yarn based on its nominal gauge length before untwisting[12]. Tensile properties of coir yarn were determined using UTM (Universal Testing Machine) Shimadzu AG-X/R. At test parameters; for yarn strain rate= 10 mm/min, gripping length = 30 cm at atmospheric temperature. Parameters such asscorage, runnage twist and tensile strength of coir yarn were determined as per IS 14596-1998 standard.

VI. RESULTS AND DISCUSSIONS

6.1 Physical properties of surface modified fibre.

6.1.1 Tensile strength

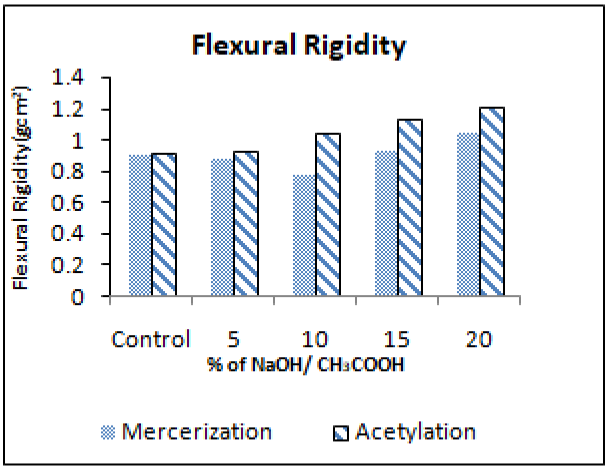
The tensile properties of treated and raw fibre were determined by taking a minimum of 50 coir fibres with known diameters. It's clearly shown that there is an appreciable improvement in the breaking stress of treated fibres compared to the control owing to surface treatment. There is 24.4% improvement in the breaking stress of acetylated fibres and 35.2% improvement in mercerized fibres in correlation with untreated one. The increase in breaking stress is from 213 N/mm^{2}to 288 N/mm^2 for control and mercerized fibre whereas265N/mm^2 for acetylated fibre. The increase in breaking stress of the treated fibre may be due to orderly arrangement of NaOH and CH_3COOH units on the cellulose backbone of the coir fibre.



Fig(1)breaking stress of mercerized and acetylated fibres

6.1.2 Flexural properties

Flexural rigidity (FR) test indirectly measures the degree of softness by evaluating the flexural rigidity of the fibre, when subjected to deformation. During mercerization NaOH addition reduces flexural rigidity but excess addition increases FR values. FR value obtained was0.905gcm^2 for control fibre and 0.775gcm^{2}for 10% mercerized fibre.Virtually15% reduction in FR of the fibre was remarked. Whereas in acetylation FR value slightly increases indicates the fibre become hard. Acetylation Andover inclusion of chemicals rupture the surface of the fibre and becomes rough. Reduction in flexural rigidity is directly proportional to improvement in softness.



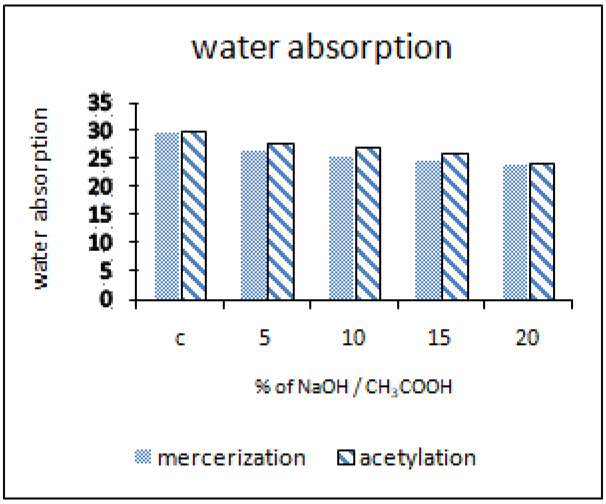
Fig(2)flexural rigidity of mercerized and acetylated fibres

6.1.3 Density

Density of coir fibre may vary with change in diameter. Using sink float method density of the control fibre was 1.2g/cm^3 whereas surface treated fibres seems to 1.005& 1.119g/cm^3 respectively for mercerization and acetylation. Slightly decreased density of coir fibre was the resultant of surface treatment, due to the leaching of the extractives from coir fibre during the surface treatment.

6.1.4 Water absorption

Water absorption values were found to be 29.8 % of the control, after surface treatment it was reduced to be 24-23.8 %. It was found that around 20% reduction in water absorption value of both the surface treated fibres, because fibres come by dimensional stability by mercerization and acetylation, thereby decreasing the hydrophilic nature of the fibre. It is due to the blockage of active sites on surface treatment. Water absorption result reveals that surface treatment increases hydrophobic nature of the fibre.



Fig(3)water absorption of mercerized and acetylated fibres

6.1.5 Colour fastness

Control and mercerized fibre were subjected to Xeno test in which control fibre Graded I &II with respect to dyes and treated fibre were graded II &III. Results evidently portrays that surface treatment enables improvement in colour fastness towards light, the resultant fibre when compared to control have improved colour fastness because of surface modification and light resistance of the fibre.

Dyes	Control	Treated
Acid Orange	II	III
Acid Red	I-II	II-III
Rhodamine	I	II
Malachite Green	I	II
Direct sky blue	II	III
Direct Brown	II	III

Table(1)colour fastness of control & treated samples

6.2 MORPHOLOGYCAL PROPERTIES

6.2.1 Scanning electron microscopy

The effect of chemical treatment on surface morphology of coir fibre was determined using scanning electron microscopy. Fig 4(a) shows the image of control fibre. It is visible that surface of the fibre was smooth due to waxy layer that were present on the surface of fibre. The SEM image of mercerized coir fibre is shown in fig 4(b). A comparison between the untreated and mercerized coir fibre reveals topographical changes because of the removal of low molecular weight compounds. This reduces moisture absorption and provides good surface area for adhesion with the matrix. The acetylation process fig4(c) of coir fibre showed that the surface was rougher and wavier as compared to the untreated coir fibre. Upon acetylation treatment the hydrophilicity of coir fibre reduced by the interaction with acetyl group. The fibrillation is also found to arise as the binding materials were removed and some micro-pores appear in the acetylated fibre surface and shows a large number of pits on the surface. Hence this acetylation treatment increases effective fibre surface area for better adhesion with the matrix. The removal of surface impurities on coir fibre was advantageous for fibre-matrix adhesion since it facilitated both mechanical interlocking and the bonding reaction.

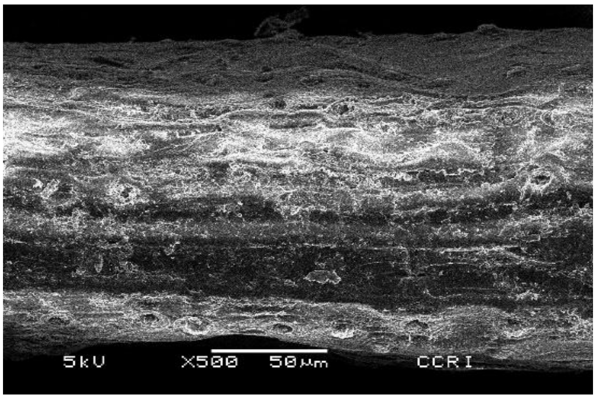
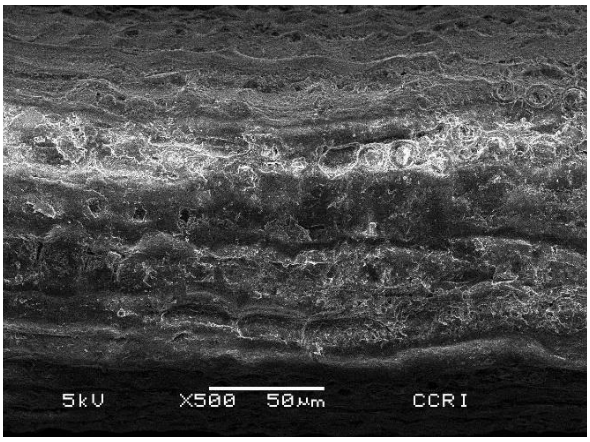
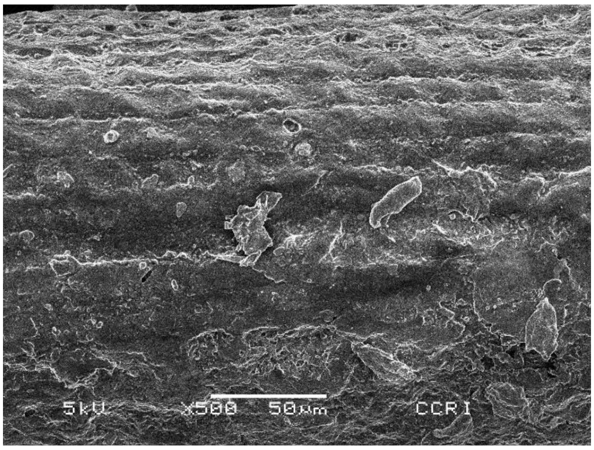


Fig4(a)control



4(b)mercerization



4(c) acetylation

6.3 MECHANICAL PROPERTIES

6.3.2 Scorage, Runnage and Twist

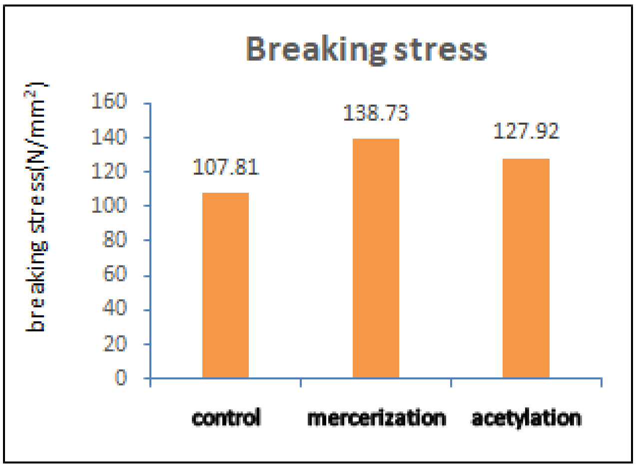
Surface Treatments						
	Scoragee		Runnage (m/kg)		Twist	
control	15		328		66	
%	Mercerization			Acetylation		
5	16	330	63	16	333	64
10	16	330	63	16	333	64
15	17	332	62	17	336	65
20	17	332	62	17	336	65

Table(2) scorage runnage and twist of control & treated samples

From these results slight increment in scorage and runnage whereas reducing twist hence we conclude that surface enhancement reduces hairiness and increase texture due to the smoothening of fibre. Surface treated fibres spinning regular in comparing control one.

6.3.3 Tensile strength of yarn

Surface treatments involving addition of 10% NaOH and CH_3COOH exhibited increase in tensile strength. Breaking stress increment is 29% in mercerization and 19% increment in acetylation compared to control.10% addition of NaOH & CH_3COOH provides vouchsafe properties and hence this minimal usage of chemicals will be very helpful for healthy effluent disposal in environmental point of view.



Fig(5)breaking stress of control mercerized and acetylated yarn

VII. CONCLUSION

The experimentation was based on enhancing physical mechanical and morphological properties of coir fibres availing surface treatments. Multitudinous surface treatments proved to be very victorious in conversion of properties of coir fibres, thereby increasing hydrophobic nature and boosting fundamental properties of coir fibres. SEM characterization showed that the surface of the fibre became wavier and rougher due to the removal of the waxy layers and the parenchyma cells from the surface of the fibre by the chemical treatments conducted.

The acetylation shows the least reduction in the waxy layers while the mercerization shows higher reduction of waxy layers with the increase of the alkali concentration. The removal of surface impurities on plant fibre was advantageous for fibre-matrix adhesion since it facilitated both mechanical interlocking and the bonding reaction as a result of the exposure of the hydroxyl groups. The vouchsafe properties assists the fabrication of coir products explicitly coir yarn, mats, mattings, geotextiles, coir products and composites. The research does help greatly in exploring newer areas of application of surface treated coir fibre & and development of diversified coir products.

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REFERENCES

[1]. Graffin M W, Ott E, Spurline H M “Cellulose and cellulose derivatives”, Part II Interscience, Newyork863, 1954.

[2]. Singha A, Rana “Effect of aminopropyltriethoxysilane (APS) treatment on properties of mercerized lignocellulosic Grewiaoptivafiber” J polym. Environ 21;141-150, 2013.

[3]. Matuana L M,Park CB, Balatinecz J “Processing & cell morphology relationships for microcellular foamed PVC/wood- fibercomposites” Journal of PolymEng Sci37;1137, 1997.

[4]. Mohanty, M Misra,L.T Drzal “Natural fibres, biopolymers and biocomposites”Ist edition 2005.

[5]. S. Kalia, B. S. Kaith, and I. Kaur, “Pretreatments of natural fibers and their application as reinforcing material in polymer composites - a review,” Journal of Polymer Engineering and Science, vol. 49, pp. 1253-1272, 2009.

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[6]. M. M. Rahman and M. A. Khan, "Surface treatment of coir (Cocos nucifera) fibres and its influence on the fibres' physico-mechanical properties," Journal of Composites Science and Technology, vol. 67, no. 11-12, pp. 2369-2376, 2007.

[7]. M. Sreekala, M.Kumaran, S.Joseph, M.Jacob, S.Thomas "Oil palm fibre reinforced phenol formaldehyde composites: Influence of fibre surface modifications on the mechanical performance" Appl.Composite.Mater., 2000.

[8]. Ivy A.W. Tan, Wu, W.H., Ron A. Chan and Leonard L.P. Lim. "Effect of Mercerization and Acetylation on Properties of Coconut Fibre and its Influence on Modified Bitumen". *Journal of Civil Engineering*,2012.

[9]. Mishra L, Basu G, Jose S, Samanta A "Accelerated retting cum softing of coconut fibre" J Industrial crops and Products 77;66-73, 2015.

[10]. DasA,Sharma U "Evaluation of bacterial treatment on the flexural rigidity & light fastness properties of coir fibre". Coir News 22-24.2004.

[11]. Lakshmi. N, Sarika. B, Sumy. S, Ravi P "Improvement in Physical Properties of MMA Grafted Coir Fibres". *Cord, International Journal on Coconut R & D*, 2015.

[12]. Ravi, P.K. *INDIAN COIR, a reference book.* Published on the occasion of INDIA INTERNATIONAL COIR FAIR: Coir Board, 2007.

[13]. Beever, R.B. *Experiments in fibre physics.* Butterworth: London 193-197;1970.

[14]. Anita Das Ravindranath, Radhakrishnan, S, Sharma U. "Astudy on bio bleaching of coir using bacterial cultures". International workshop on wet processing of coir, 100-104. Alleppey, India 1997.

[15]. Dixit.S Verma.P "The effect of surface modification on the water absorption behaviour of Coir fibres". Department of Chemistry, MANIT. Bhopal 2012.