Extraction and Evaluation of OKRA Fibres

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Abstract

The mother earth is filled with huge amount of fibrous material to satisfy all needs of mankind. Till date, even the highest level of technological innovation has not taped every source considerably. Considerably this, a research is framed on okra fibres. To overcome the problem of shortage of natural fibres and to utilize the underutilized fibres, on the earth, a study on extraction and analysis of fibres from Okra plant was carried out. The world has turned its attention to renewable and sustainable resources. The preparation of characterization of cellulose – based materials is important in research and development area. It is botanically called as Abelmoschus esculentus. The Okra fibres were extracted from the stalk of the plant which was given alkali and acid treatments to study the salient fibre properties such as chemical constituents, weight loss, burning behavior and moisture content. The Scanning Electron Microscopy (SEM) study was also carried out to assess the morphological changes in ,the fibres on processing. Fourier transform infrared spectroscopy (FTIR) test was also carried out to find the presence of the chemical groups in the fibres before and after treatments. The study has thrown light to give better scope for okra fibres in the use of textiles

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Keywords - *Okra, Fibre Property, Fibre, Chemical Constituent, Moisture Content, Scanning Electron Microscopy, Fourier Transform Infrared Spectroscopy and Weight Loss.*

I. INTRODUCTION

In tropical regions of India, there are large varieties of regenerative plants and trees with fiber content. Some of them are cultivated over the generations and some are wild plants, trees and creepers that grow in forests and woods. It is an established fact that any material in its fibrous form is stronger than in bulk form. These strong fibers are used to reinforce the weak materials[1]. *Abelmoschus esculentus* is one of the popular vegetable in India. It is cultivated extensively all the year round for its immature fruits as these are used as a vegetable. The stem of the plant is used for the extraction of the fiber. India is the largest producer of Okra in Asia as well as in the world. It is also used as vegetable in Brazil, West Africa in many other countries in Asia. In India major producing states are West Bengal, Bihar and Uttar Pradesh[2]. This study was carried out to elicit information about the utilization of the non edible portion of the plant, collect the okra plants, extract the fibres from plant source and analyse the properties of fibres.

II. MATERIALS AND METHODS

The methodology of this study is expressed under the following heads.

A. Utilization of non edible portion of the Plant after Harvest

A survey was conducted in Konneripatti (Mettur) village, Edappadi, Salem, Tamil Nadu, India to find out the utilization of the nonedible portion of the plants after harvest. From this survey it was noted that the respondents utilized the non edible portion of the plants for various purposes. As far as these results were concerned the maximum of 58 percent of the farmers used the non edible portion of food crops as animal feed followed by 37 percent of waste used as fuel; and 3and 2 percentages of them used it for rope making and shelter respectively. It was understood from the survey that the maximum of respondents utilized the plant wastes for animal feed followed by the respondents who used it for fuel. Huge amount of okra plant stem is discarded on the field annually after collecting vegetable, without proper utilization[3]. So an attempt was made to extract and analyse the fibres from okra plant.

B. Procurement of Raw Material

The Okra plants were procured from as the Edappadi village. The stem of the plant generally grows to about 90 cm to 200 cm high. The harvested plants were collected in green condition and subjected to extraction process. (Plate – 1 & 2).

Plate: 1. Okra field after harvest

Plate: 2. Cutting stem of Okra plant

They are collected in green condition, and the stems were separated from plant. These were then bundled for retting process.

C. Extraction of Fibers

The method used was stagnant water retting. This process of retting removes the waxy epidermal tissue, adhesive pectin and hemicelluloses that bind the fiber bundles to each other. The okra plants were bundled comprising of 350-400 plants, Each of these bundles were immersed in a concrete tank containing soft water for ten days. Later the stalks of the plant were tapped slightly with wooden hammer for removal of soft pulp. In order to separate the pulp it was scrapped with the help of a knife. This was once again immersed and left in the tank for five days. Then the fibers were separated thoroughly from the pulp, washed, combed and exposed to sunlight for two days until the odour was removed from fibers. The yield per cent was calculated as per the equation expressed by Chakma[2], that the yield of fibers (R%) is measured by the percentage of the ratio between the final mass of the fibers after extraction process (Mf) and that of the plant before extraction process (Mi), which is given in Equation[(4)] R (%) = (Mf /Mi) x100. Fibre yield was only four percent of the weight of green plant.

The requisites for Fiber Extraction: The retting process required 10 days for initial soaking

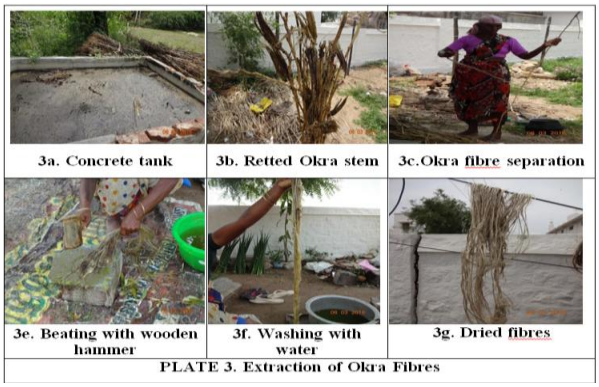


PLATE 3. Extraction of Okra Fibres

and 5 days for final retting. For drying it took 2 days in bright sun light and open air. So the complete fiber extraction process required 17 days. About 200 litres of water and 9 labours were involved for the complete process of seventeen days. The steps for extraction of okra fibres from the stem are clearly exhibited in the Plate 3.

D. Treatment of Fibres

The fibres were processed by scouring, bleaching and dyeing techniques. (Plate 4)

1. Scouring Process

The scouring process was carried out for raw okra fibres using Material Liquor ratio1:10, water -5 litres, Sodium Hydroxide (NaOH) -2-5gms/litre, Wetting agent 1 gm/lit, Detergent-1-2 gm/lit at the temperature 100-150°C for period of 6-8 hrs. The chemical Sodium Carbonate is also added to maintain the pH at 10.5.[5]

2. Bleaching Process

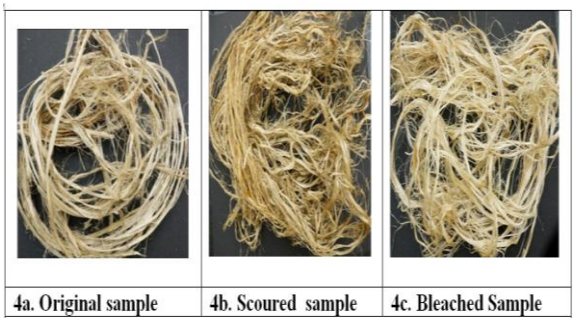


PLATE 4 Untreated and Treated Okra Fibres

Scoured okra fiber samples were bleached with hydrogen peroxide universal bleaching agent. The parameters and chemicals used for bleaching were Hydrogen peroxide- 0.5%, Soda ash (Sodium Carbonate)-1.0%, Sodium Silicate-1-1.5%, Wetting Agent-0.1%, Temperature-70-80°C, Time -8 hrs, pH-10 and the Material Liquor Ratio was 1:4.

3. Evaluation of Fibres

The fibres were evaluated for the aspects like chemical constituents, Scanning Electron Microscopic appearance, weight loss, moisture content and FTIR. All the fibre samples were assessed objectively after preconditioning as per ASTMD 1776.

a. Visual Evaluation

The original and processed fibres were subjectively assessed by visual inspection. About 25 persons were asked to evaluate the fibres visually. The results were recorded and tabulated.

b. Burning Behavior of Okra Fibres.

The burning behavior of the fibres was observed on approach to flame, in flame and after taking way from flame. It was observed and recorded.

C. Weight Loss

Weight loss of untreated and treated samples was assessed using weighing balance, compared and recorded.

d. Fiber Length and Density

The Length of raw okra fiber was found out by measuring the fibers using meter scale. About 100 fibers were measured and the mean value was calculated and recoded for the length of the fibers. The weight of the same fibers was assessed using electronic balance. The density of the fibers was calculated as per ASTM D1577 using the formula

Fiber Length Density (D) = 9000*W/L*N, Where W= Weight of the Fibers,

L= Length of Fibers, N= Number of Fibers.

e. Determination of Chemical Constituents of Fibers

Chemical compositions of fibers namely celluloses, hemicelluloses and holocelluloses were estimated according to TAPPI procedures: αcelluloses – T203cm-99, and holocelluloses – Tappi 249-75. The difference between the values of holocelluloses and αcellulose gives the hemicelluloses content of the fibers. The moisture content of the fibers were estimated by T264om-88 procedure. Five grams of sample was mixed with 7.5% NaOH reagent and stirred at 25° C for one hour. After 60 minutes the suspension was filtered. The chemical composition was determined using the filtrate. Twenty five ml of the prepared filtrate and 10 ml of 0.5 N potassium di chromate were taken and 50 ml of concentrated sulfuric acid was added into it under stirring. Fifty ml of water was added to the mixture, followed by two drops of ferroin indicator and then titrated against 0.1 N ferrous ammonium sulfate solution to a purple color. About 12.5 ml of 17.5% NaOH and 12.5 ml of water were used as blank.

α celluloses content was estimated using the following equation: α celluloses (%) = (6.85×(V2 −V1)×N×20)×100/A×W (1) where V1 and V2 are the titre volume of filtrate and blank, N is normality of ferrous ammonium sulfate solution, A is the volume of pulp filtrate, and W is the oven-dry weight of specimen. Two grams of the sample was weighed and kept in hot air oven at 150° C for two hours. The ash residue and the remaining holocellulose was separated and weighed.

From this, the holocellulose content was evaluated using the formula **Holo celluloses (%) = A –B/C× 100 (2) w**here A and B are the oven-dry weight of fibers and of ash in A, and C is an initial weight of the specimen. For the estimation of lignin, 0.75 ml of 0.1 M sodium hydroxide was added to 15 mg of the sample and kept under stirring in hot water bath. After one hour the sample was removed and washed first with distilled water followed by 10% acetic acid. The samples were dried in a hot air oven. Ten mg of this sample was soaked in 0.2 ml of 72% sulfuric acid for two hour and then added 10 ml of distilled water. The sample was filtered, washed and dried under high vacuum.

Lignin content (%) = C – D/C×100

Where C and D are the initial and final weight of fibers.

f. Moisture content

The weighed (A) amount of sample was kept in hot air oven for two hours at 105 ±3 ◦C followed by cooling in a desiccators, replaced the stopper and opened the stopper momentarily to equalize the air pressure and weigh. Bottle was returned to the oven for one hour; repeat the cooling and weighing as above for successive hourly periods until constant weight (B) was reached, that is, until successive weighing do not changed by more than 0.002g. The moisture content was calculated as follows:

Moisture content (%) = A−B/A× 100 [(6)].

The original and the treated okra fibres were for chemical constituents namely Holocellulose, Hemicellulose, α cellulose, Lignin. Also moisture content of the fibres was analysed.

g. Fibre Morphology

The fibre morphology was found and analysed through Scanning Electron Microscopic appearance (SEM) analysis. It was observed at different magnification in both the longitudinal and cross section for raw and treated okra fiber samples.

h.Fiber Samples Fourier Transform Infrared (FTIR)

The FTIR Test done by IR AFFINITY-IS SHIMADZU instrument. The IRAffinity-1S offers the high S/N ratio (30,000:1, 1-minute accumulation, neighborhood of 2,100 cm^{-1}, peak-to-peak), a maximum resolution of 0.5,1,2,4,8,16cm^{-1}, and compact dimensions. Wave number range 7,800 to 350cm^{-1} The okra fibers were studied by FTIR microscopy different treatment led to significant difference in the infrared spectra as exhibited in every molecule.

III. RESULTS AND DISCUSSION

The results are expressed in the Tables followed by discussion.

A. Visual Inspection

The untreated and treated fibers were subjected to visual evaluation for color, texture, luster and general appearance.

S.No	Samples	Color %			Brightness %				Texture %			Luster %			General Appearance %		
		Greenish	Ivory	White	Very Dull	Dull	Bright	Very Bright	Soft	Medium	Rough	High	Medium	Low	Good	Fair	Poor
1	Raw	88	12	-	60	40	-	-	-	36	64	4	36	60	40	52	8
2	Scoured	8	92	-	-	8	92	-	8	60	32	-	88	12	52	48	-
3	Bleached	-	20	80	-	-	20	80	84	12	4	92	8	-	96	4	-

From the Table I it is clear that the raw okra fibres were greenish in color. As far as brightness is considered it was rated as very dull by 60 per cent of the judges. But it improved as bright by 92 per cent and 20 per cent in scoured and bleached samples respectively. The texture of the fibre samples of extracted, scoured and bleached were rated as 64, 60 and 84 per cent by the judges as rough, medium and soft respectively. Similarly the judges have rated the lustrre as 60 per cent low for raw fibres, 88 per cent as medium after scouring and 92 per cent as high after bleaching. With reference to general appearance the raw fibres were rated as fair by 52 per cent of the judges, whereas it improved as good by 52 and 96 percentages after scouring and bleaching.

Hence it could be colour, brightness, texture, luster and general appearance of the raw fibres improved after scouring and bleaching processes.

B. Burning Behavior of Okra Fibres

The results of burning test of okra fibre and a comparison with other natural fibres is presented in the Table II.

The burning behavior of the untreated and treated okra fibres was noted to readily ignite on approaching flame, in flame it burnt quickly with a bright yellow flame, it continued to burn even after it was removed from flame, it gave an odor of a burning paper and the ash was black and soft in the original fibre sample whereas it was feathery and grey in both the treated samples. This resembles the burning behavior exhibited by cotton[7].

S.No	Fiber	Approaching flame	In flame	Removed from flame	Odor	Ash	Particulars
1	Okra	Scorches, ignites readily	Burns quickly; with bright yellow flame	Continues to burn rapidly, has after glow	Burning paper	Black	Work done in this research
2	Okra Scoured	Scorches, ignites readily	Burns quickly; with bright yellow flame	Continues to burn rapidly, has after glow	Burning paper	Feathery grey ash	
3	Okra bleached	Scorches, ignites readily	Burns quickly; with bright yellow flame	Continues to burn rapidly, has after glow	Burning paper	Feathery grey ash	

C. Weight Loss in Scoured Okra Fibres

Weight loss in scoured Okra fibres

TABLE III
Weight Loss in Soured Okra Fibres

S.No	Particular	Original Sample (gms)	Scoured Sample (gms)	Bleached Sample (gms)
1.	Weight loss	31.32	27.42	10.43

From the Table II, it is clear that the original sample of okra Okra fiber weighed 31.32 gms. On scouring the fibres weighed 27.42 gms. There was a loss in weight by 12.45 percent. This may be due to the removal of unwanted materials adhering to the fibres. This confirms that the fibre obtained was cleaned on processing. (Figure 1)

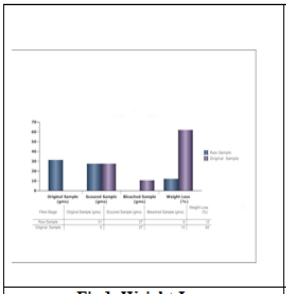


Fig 1. Weight Loss

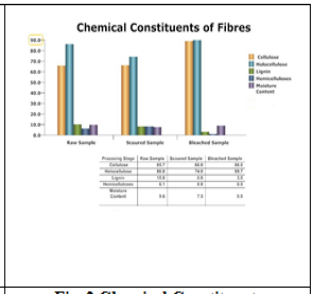


Fig. 2 Chemical Constituents

On bleaching, further loss in weight of 10.43 grams was observed. A loss in weight of was noted in the fibre samples after bleaching over the scoured fibre samples. The effect of chemical treatment and alkalization brings morphological changes, weight and color as said by Khan which is proved in this study also. The loss in weight could be due to the deterioration of fibres by the chemicals used during bleaching process which is clearly viewed in the SEM image.

b. Length, Weight and Density of fibres

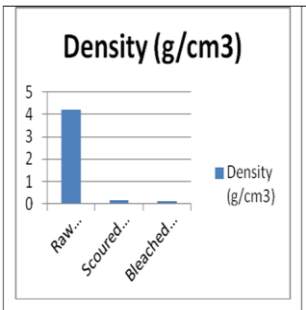


Figure – 3

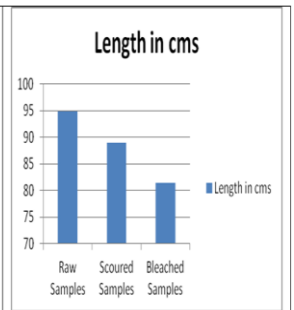


Figure - 4

Length, and Density of fibres

From the Figures 3 and 4, it is is clear that the length of the raw fibers was 94.83cms. which reduced to 89.0 and 81.5 cms in scoured and bleached samples respectively. The fiber density was noted to be 4.195 g/cm^3 in the raw fiber, which reduced to 0.151 g/cm^3 in scoured and to 0.110 g/cm^3 in bleached samples.

TABLE V
Chemical Constituents of Fibres

S.no	Processing Stage	α Cellulose (%)	Holocellulose (%)	Lignin (%)	Hemicelluloses (%)	Moisture Content (%)
1.	Raw Sample	65.7	86.0	10	6.12	9.64
2.	Scoured Sample	66	74.0	8	8	7.53
3.	Bleached Sample	88.8	89.72	3	0.92	8.88

From the Table V it is clear that the α Cellulose content in the raw fibers was 65.7% which increased in scoured and bleached samples to 60 and 88.8 percentages respectively. The holocellulose was noted to be 86 percent which showed as reduction in scoured sample to 74 percent but an increase in bleached samples to 89.72 percent. The hemicellulose was found to be 6.12 percent in the raw fiber which increased to 8 percentages in the scoured

sample and a drastic decrease was observed in the bleached sample to 0.92 percent. As for the lignin content in the raw okra fibers, it was 10 percent which also increased on scouring to 11.11 percent but decreased drastically in the fiber samples on bleaching to three percent. The removal of lignin was higher after chemical treatment in sisal fibres[8]. The same observation was also noted in the okra fibres. All the cellulose fibers have several compound namely cellulose, hemicelluloses, lignin, pectin and inorganic matters. Fiber chemical constituents are the most important parameters which influence their processing and application. (Figure 2)

Comparison of chemical constituents obtained results of original okra fibres with a literature survey. (Table VI)

TABLE VI
Comparison of Chemical constituents with literature

S.No	Particulars	α cellulose	Lignin	Hemi cellulose
1.	Source Khan *et.al* 2009	60-70%	5-10%	20%
2.	Obtained result	65.7 %	10%	6.12%

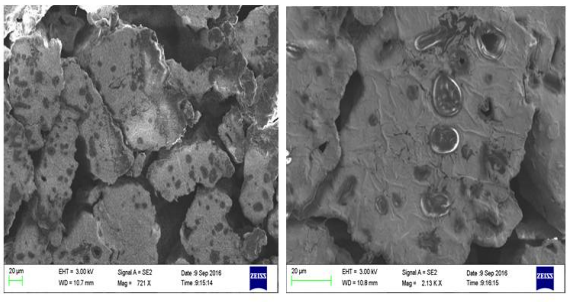
The okra bast fiber posses an excellent quantity of cellulose and it also contains low percentage of lignin. The cellulose and lignin contents of the obtained results of extracted okra fibres are as per the expression of Khan[6] 2009.

C. SEM Appearance Cross section of Okra fibres

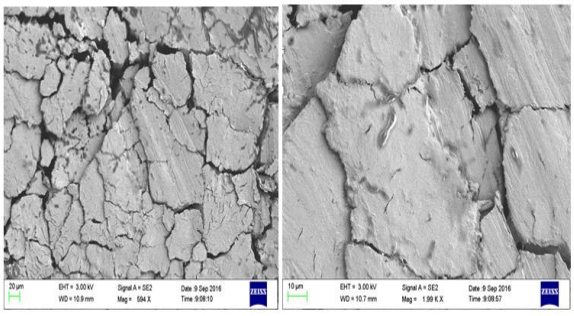
Fiber Morphology

The study of Scanning Electron Microscopy exhibits the appearance of the raw and treated fibers. The raw okra fiber at the magnification of 17.17 kx showed very rough surface and showed more hairiness at 10.87 kx magnification. The scoured okra fiber showed a smoother surface. The bleached samples showed even more smother surface than scoured samples. Perfect uniformity in the surface was observed in the bleached fiber samples over the scoured and bleached samples. The fiber bundle of untreated okra fibers are covered by non cellulosic materials and this may be reason for the uneven.

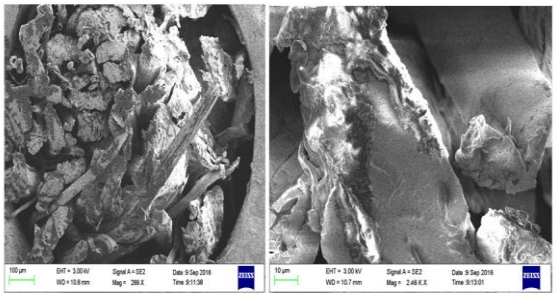
The alkaline and acidic treatment remove the waxy epidermal tissue from the okra fiber bundle with the agreement of the result expressed by Cao *etal* [(8)], The uniformity improvement was also noted in the cross sectional views of the treated fibers over the raw fibers.[(9)] The Cross Section of the Okra fibres showed irregular Circles & Scales. On scouring the surface was clear. Bleached samples showed lesser scales. As far as the longitudinal view was concerned, more hairiness was observed in the raw fiber. This was reduced on Scouring but small depressions were noted. This became very clear without hairiness in the bleached state.



Raw Okra fiber Sample view-1 Raw Okra fiber Sample view-2



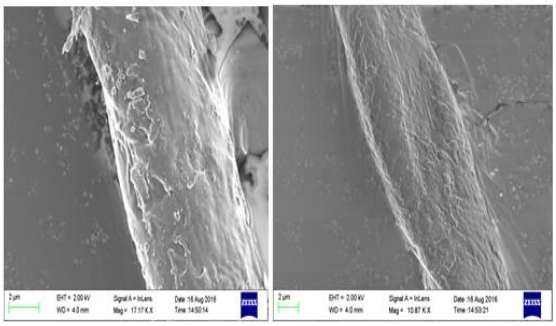
Scoured Okra fiber Sample view-1 Scoured Okra fiber Sample view-2



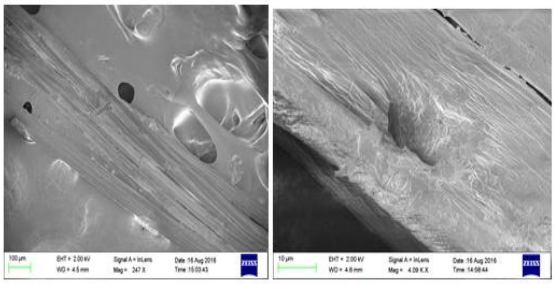
Bleached Okra fiber Sample view-1 Bleached Okra fiber Sample view-2

PLATE - 5

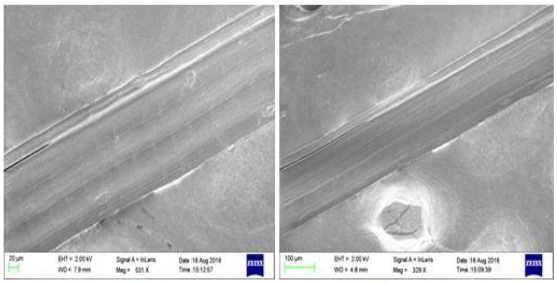
3.3.d. SEM Appearance Longitudinal View of Okra fibres



Raw Okra fiber Sample view-1 Raw Okra fiber Sample view-2



Okra fiber Sample view-1 Scoured Okra fiber Sample view-2



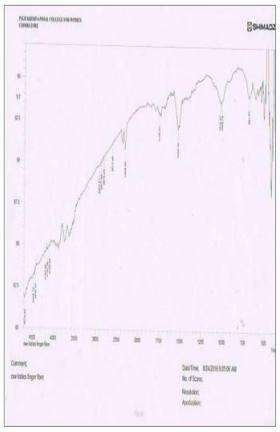
Bleached Okra fiber Sample view-1 Bleached Okra fiber Sample view-2

PLATE – 6

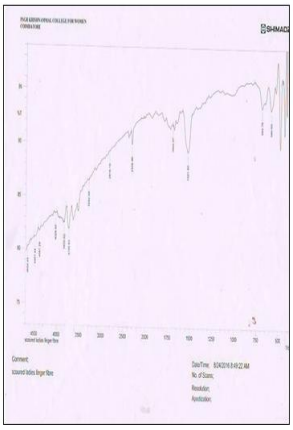
Hence the SEM analysis supported the weight loss in the fibres after bleaching where in the hairs are removed to a great extent.

E.Fourier Transform Infrared Spectrometry (FTIR)

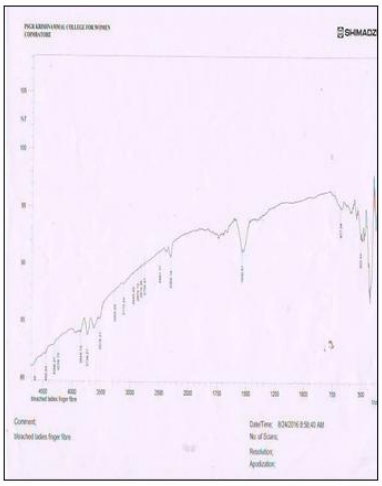
The Fourier Transform Infrared Spectrometry of the samples are expressed in the figure 5a,5b and 5c.



5a. Raw okra Fiber Sample



5b.Scoured Okra Fiber Sample



5c. Bleached okra Fiber Sample

FIGURE - 5

Fourier Transform Infrared Spectrometry (FTIR) analysis shows the peak in certain predominant groups. This shows a difference in the original, scoured and bleached samples. The peak varies between them which may be due to the treatments the fibres underwent. Fig 1-4 the FTIR spectra of scoured okra fibers indicate that intensity of absorption band of (N-H) at 3,600 cm^{-1} presence of no prominent peaks were noted between 3000-3100cm^{-1} in the case of bleached and raw samples were where as small peaks were observed at 3100cm^{-1} (C-H group) in the second fiber samples. At the 2400 cm^{-1}, prominent peaks were noted in all the fiber samples depicting the presence of OH group (acids). Peaks were noted in raw, scoured and bleached samples of which the prominence was higher in sample scoured sample exhibiting Carboxyl group (C=O) between 1600 and 1750cm^{-1}. A stretch was observed between 1200-1300cm^{-1} express C-O group. The peak was observed at 1550 in all the samples raw, scoured and bleached. In the sample scoured the prominence was the highest over other samples.

IV. CONCLUSION

From this study it is obvious that the dirt and the waxy material from the fibres are removed from the okra fibres on scouring process. The bleaching of the fibres has brightened the fibres which would enhance the dyeing. The morphological variations are also noted in the treated fibres. Moisture content in the fibres samples have reduced on scouring and bleaching which may help in the conversion of the fibres into composite structures for which this property is essential.

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