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Morphological Study of Bhimal Fibres

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Abstract—*Bhimal fibres are bast fibres found in the Himalayan region of India, mainly in the Doon valley of Uttarakhand. These fibres are extracted from the bark of bhimal (Grewia optiva) tree on which limited research has been carried out so far. It is available in abundance and can aid in reducing the burden of other popularly used textile fibres. Hence, it is imperative to carry out research on the properties of these fibres and ascertain appropriate uses in future. There have been various studies on traditional extraction of bhimal fibres and its composites, but none has been documented so far on surface properties of the fibres. This paper is an attempt to present research carried out to study the morphology of bhimal fibres. The fibres were extracted using chemical method and then analyzed using standard test methods and formulae to delineate morphological properties of the fibres. The methods adopted to study morphology of bhimal fibres were- Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) method. Different stages of extraction were tested for SEM and XRD and best conditions in each of them was optimized. The results were presented as images in case of SEM and figures in case of XRD analysis and compared. The best conditions of both were finalized. Researchers can use this result for further studying morphology of bhimal fibres and positioning the fibres for suitable end use.*

1. INTRODUCTION

Grewia optiva (bhimal) fibres are traditionally extracted by retting by local people in the Himalayan regions. It is multi-utility tree grown basically for fodder as it is an evergreen perennial tree, catering to fodder requirement during winter months. The process used by them is quite lengthy as it takes about 30-45 days to complete natural retting by microbial degradation. Chemical extraction was therefore, used to extract bhimal fibres from the branches of bhimal tree using urea at different concentrations-2.5% owm, 5%owm, 7.5% owm and 10% owm. Out of these, 5 % owm was finalized on the basis of yield and bundle strength. 5% owm urea retted samples were then treated with different concentrations of hydrogen peroxide for bleaching. The process of bleaching along with imparting required degree of whiteness to the fibres also helped to further individualize the fibres. Best of this combination was then given a polysiloxane commercial softener treatment. The morphology of these different combinations, including softener treatment sample along with manually separated bhimal fibre layer from the branch and that extracted by retting by an organization-AAGAAS based in Dehradun, was compared.

Two methods of studying morphology were used here in this study to investigate the morphology of bhimal fibres in detail- Scanning Electron Microscopy and X-Ray diffraction. The following sections describe the procedures used and provide detailed results by the use of images and graphs.

1.1. Scanning Electron Microscopy

Scanning Electron Microscopy is an excellent technique for the study of surface morphology of fibres. It was carried out at University Science Instrumentation Centre (USIC) at North Campus, University of Delhi. Different stages of extraction of bhimal fibres were taken.

2. Determination of Crystallinity

Crystallinity index (CI) measurement is a historic method of investigating crystallinity of cellulose materials. It measures the amount of crystalline cellulose with respect to the total amount of amorphous materials. Sehgal et al. (1959), as a means of determining the relative crystallinity of different samples, proposed a measurement-Crystallinity Index (CI) [1]. Natural fibres are not completely crystalline; their X-ray diagram gives sharp and diffused peaks. The helical arrangement of molecules in cellulosic fibres also leads to broadening of the arcs. Analysis of sharpness and broadening can lead to estimation of crystalline order and the molecular arrangement of the structure which can further help to gain information regarding the crystalline orientation and crystalline content of the fibres [2].

2. MATERIALS AND METHODS

2.1 Scanning Electron Microscopy

Fibre samples were cut to an approximate size of 8mm. and mounted onto a cylindrical aluminium stub with a small square piece of adhesive tape. Next, they were put in an auto-fine coater-JEOL JEC-3000 FC for coating with gold-palladium. Vacuum is created for 60 seconds and pressure of 3 Pa was applied. The machine stopped automatically when coating is done. The samples on the aluminium stub are then mounted onto a sample holder and put into the microscope. The microscope used was JEOL Scanning Electron Microscope, Japan, Model-JSM 6610LV with Tungsten or LaB_6 as the source of electrons, operating voltage as 1-30 kV

and Peltier stage as -25ºC to +50 ºC. It has a resolution of 3 nm. With high vacuum mode and a magnification capacity in the range of 5X to 3,00,000 X times. Vacuum is created in the instrument. The microscope is connected to a computer which shows the live images. We can zoom in and out of particular areas and save the most relevant images at different magnification powers. In the case of bhimal fibres, magnification power of 200, 2000, 8000 and 15000 were used. Out of these, two powers were used for comparison only-200 and 2000.

SEM was carried out for six samples varying in the treatments given to them. The first sample represented by 'A' was of fibrous layer of bhimal manually pulled and removed without any other treatment. It was in the form of joined fibres with cementing materials intact, appearing as a tape. The second one denoted here as 'B' was treated to a solution of urea at the rate of 5% owm. The third one 'C' was fibre treated to 5% owm urea followed by a bleaching cum individualisation process using 5gpl of hydrogen peroxide. The fourth one 'D' was similar to 'C', only difference being in the concentration of the second treatment with hydrogen peroxide which was 20 gpl here. The fifth sample 'E' was the fibres extracted by local people of Dehradun by water retting. The last sample was that of 5% owm urea treated and 5 gpl bleached lab extracted bhimal fibres treated with a siloxane based cationic softener. The scanning was carried out at four magnifications-200, 2000, 8000 and 15000 were used. Out of these, two powers -200 and 2000 have been compared.

2.2 Determination of Crystallinity

In this method, powder sample is fed into an X-ray diffractometer and its crystallinity index is calculated. The basis of the Sehgal method is that there are two components of cellulosic material: crystalline and non-crystalline or amorphous. The amount of crystalline material is represented by the height of the highest diffraction peak and the amount of amorphous material is represented by the height of the minimum intensity between the major peaks. CI is basically the difference between these two intensities, divided by the highest peak. Table II enlists crystallinity indices of some fibres below. CI varies significantly depending on the selection of method employed for calculating it out of the methods- X-ray diffraction, solid state ^{13}C Nuclear Magnetic Resonance (NMR), Infrared spectroscopy (FTIR- Fourier Transform Infrared spectroscopy) and Raman spectroscopy. The most simplest and used method which utilizes measurement of two heights in the X-ray diffractogram, produces slightly higher values than other methods. In general, X-ray diffraction and NMR provide a more accurate measurement of crystallinity as compared to other methods. FTIR measurement is also simple but it gives only relative values, as the spectrum always contains contributions from both crystalline and amorphous regions. Therefore, FTIR is not an absolute measurement technique [3].

Table 1: Percentage crystallinity of some fibres. [4,5,6]

Fibre	Percentage crystallinity (%)	Degree of crystallinity
Cornhusk[4]	48-50	Medium
Pineapple leaf fibre[4]	44-60	Medium to high
Coir[4]	27-33	Low
Banana[4]	45	Medium
Wheat straw[4]	55-65	Medium to high
Rice straw[4]	40	Medium
Hemp[5]	88	High[6]
Jute[5]	71	High[6]

The XRD (X-Ray Diffraction) analysis was carried out at IIT (Indian Institute of Technology), Delhi using Philips X'Pert Pro diffractometer system (powder X-Ray diffraction) with monochromatic intensity of Cu Kα (1.54060 Å) radiation, Ni filter, recorded from 5º to 60º to 2Θ. XRD is a method of determining the arrangement of atoms within a crystal. The source of radiation was a copper X-ray tube with operating voltage and current as 45 kV and 40 mA respectively. Scans were performed over the 5˚-60˚ full scale at a step width of 0.05°and 50 seconds as scan step time. X-rays when diffracted from a perfectly crystalline material give sharp peaks following the Bragg's Law. Amorphous materials also diffract X-rays but the peak is more diffused. Percentage crystallinity and crystallinity index was calculated using the peak height method developed by Segal *et al.* as represented by the equation given below:

$$CrI = \frac{(I_{002} - I_{am})}{I_{002}} \quad [1]$$

$$\%Cr = \frac{I_{002}}{(I_{002} + Iam)} X\ 100 \quad [4]$$

where, CrI represents crystallinity index, %Cr represents percentage crystallinity, I002 represents the intensity (height) of the 002 crystalline peak at 22° and I_{am}, the height of the minimum peak representing amorphous areas between the 002 and the 101 peaks, as shown in Figure 1.

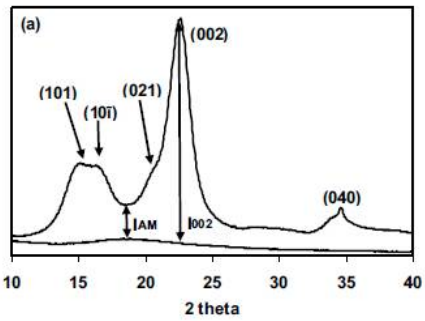


Figure 1: Typical X-ray diffraction graph. [3]

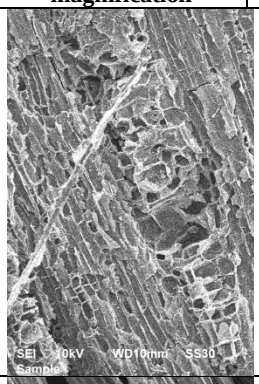
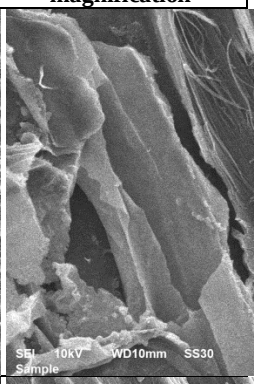
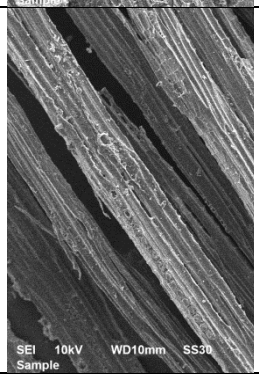
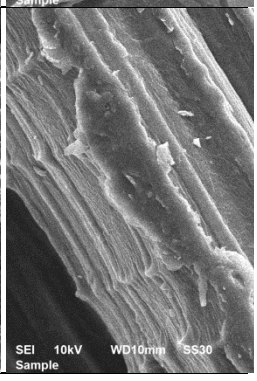
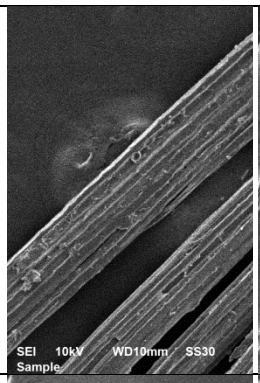
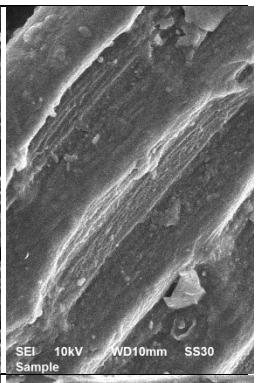
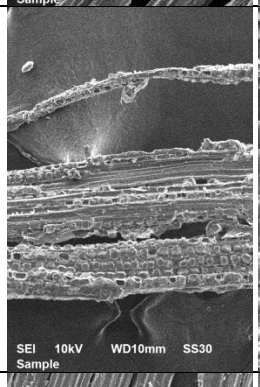
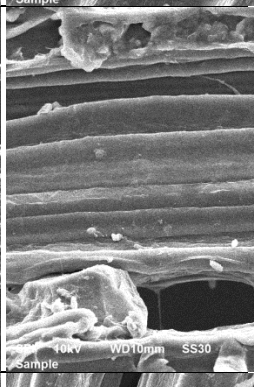
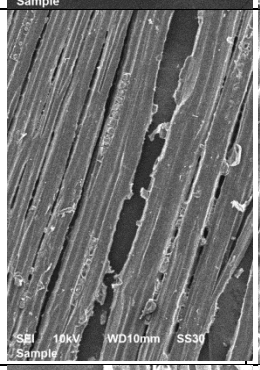
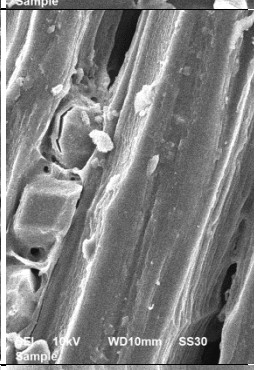
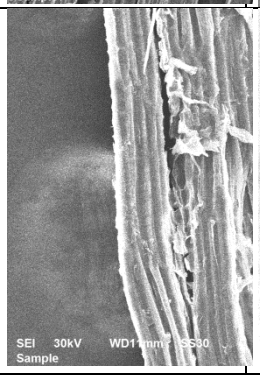
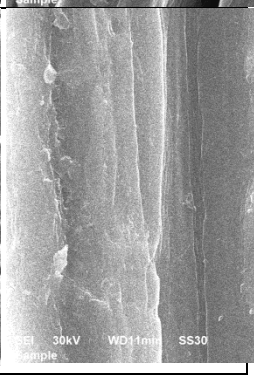
The counter reading at peak intensity of 22° represents the crystalline material and at 18° corresponds to non-crystalline/amorphous material in cellulose. The height of the highest diffraction peak observed represents crystalline material and the height of the minimum intensity between the two major peaks corresponds to amorphous regions. Crystallinity index is basically the difference between these two intensities divided by the intensity of the highest peak [7].

3. RESULTS AND DISCUSSION

3.1 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) was carried out at four magnification powers to identify powers at which sufficient conclusions can be drawn and then two were compared. The images obtained for manually pulled, extracted and removed fibres from bhimal branch (sample A), four combinations of urea retting and hydrogen peroxide (sample B, C, D and F) and another sample of fibres as extracted by the local people of Dehradun (sample E) have been summarized in the table below (Table 2):

Table 2: SEM images of different samples at 200 and 2000 magnification powers.

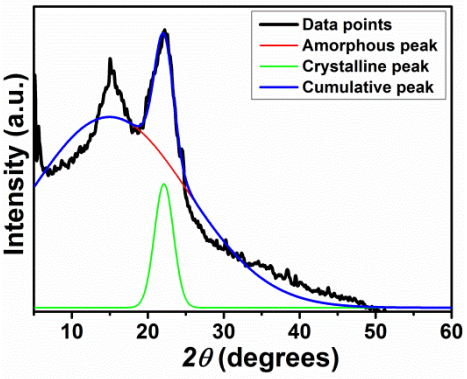
Description of Sample conditions	Scanning Electron Microscope Images @ 200 times magnification	Scanning Electron Microscope Images @ 2000 times magnification
A- fibrous layer manually pulled and removed		
B-Urea Retted @5%owm solution		
C- Urea Retted @5%owm solution and further bleached @rate of 5 gpl hydrogen peroxide		
D- Urea Retted @5%owm solution and further bleached @rate of 20 gpl hydrogen peroxide		
E-Extracted by water retting by the local people of Dehradun		
F- 5% owm urea treated, followed by 5 gpl hydrogen peroxide treatment followed by a softener		

As visible in the table, in the first sample 'A' pectins and other gummy substances, which are found in jute and allied fibres, are visible since no treatment has been given. These are basically non-cellulosic components covering the fibre bundles and are essentially hemicelluloses and pectins [8]. Because of microbial action further augmented by the presence of urea, sample 'B' shows much less visible

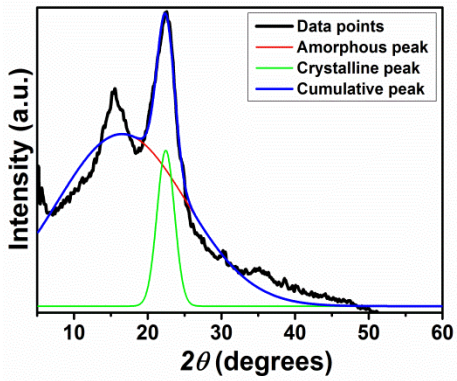
cementing substances and long striations typical of bast fibres are visible. The SEM images are indicative of the fact that retting has been sufficiently done since under-retting shows cortical cells attached to the longitudinal fibres. This image is comparable to the one obtained for sample 'E' which is water retted extracted fibre practised by the local people of Dehradun, thus indicating that urea retting is increasing the speed of separation of fibres without affecting the quality of the fibres. Sample 'C' and 'D' obtained by treating 'C' with 5gpl and 20 gpl hydrogen peroxide solutions respectively. In the magnification power 200, it is quite clearly visible that fibres appear damaged by the action of peroxide in sample 'D'. However, not much difference is seen at magnification power of 2000 times. Softener was applied to 5% owm urea retted, 5 gpl hydrogen peroxide treated fibres later when it was established that 20 gpl peroxide was damaging bhimal fibres. The SEM images show increase in parallelisation and arrangement of fibres as represented by 'E'.

3.2 Determination of Crystallinity

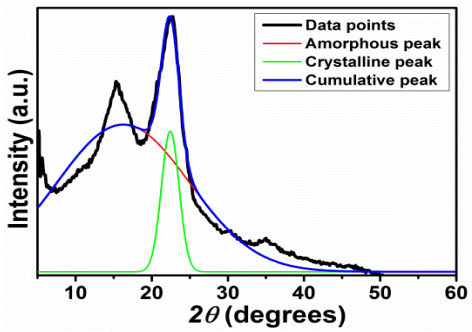
X-ray diffraction patterns and calculated crystallinity indices of bhimal fibres at different stages of extraction have been given below in Table 3 and Figure 2.



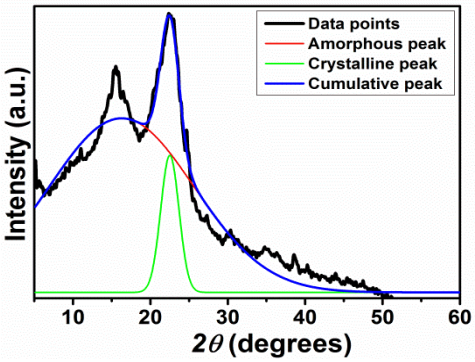
a)



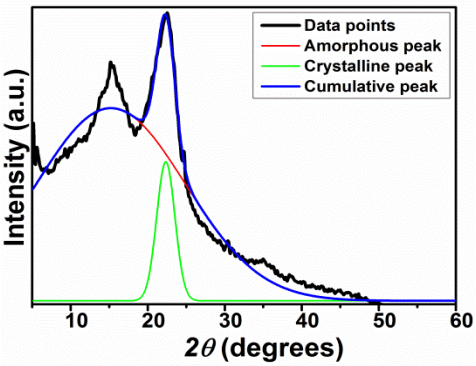
b)



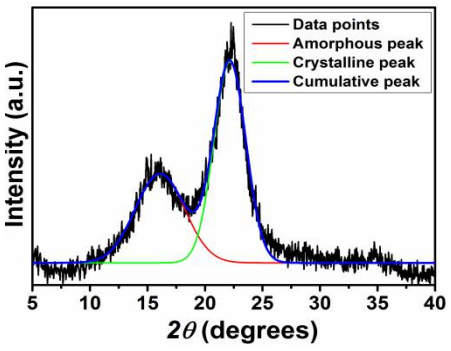
c)



d)



e)



f)

Figure 2. X-Ray diffraction graphs of bhimal fibres at different stages of extraction-a) fibrous layer manually pulled and removed, b) urea retted @5% owm solution c) urea retted

@5% owm solution and further bleached @rate of 5 gpl hydrogen peroxide d) urea retted @5% owm solution and further bleached @rate of 20 gpl hydrogen peroxide e) extracted by water retting by the local people of Dehradun f) - 5% owm urea treated, followed by 5 gpl hydrogen peroxide treatment followed by a softener

Table 3: Percentage crystallity indices of bhimal fibres at different stages of extraction.

Sr. No.	Stage of Extraction	At 2Θ scale		%Cr	C. I.
		I002	Iam		
A	Fibrous layer manually pulled and removed	5137	3524	59.31	0.31
B	Urea Retted @ 5% owm solution	6471	3861	62.63	0.40
C	Urea Retted @ 5% owm solution and further bleached @ 5 gpl hydrogen peroxide	4445.15	1201.66	78.72	0.43
D	Urea Retted @ 5% owm solution and further bleached @ 20 gpl hydrogen peroxide	2183.15	641.99	77.28	0.37
E	Extracted by water retting by the local people of Dehradun	2413	761.99	76	0.68
F	5% owm urea treated, followed by 5 gpl hydrogen peroxide treatment followed by a softener	4583	1522.78	75.06	0.67

In the above Table 3, the crystallinity indices of different stages of bhimal fibre extraction are given which vary from 0.31 to 0.67, in fibres manually pulled out from bhimal branch and softener applied fibres, respectively. A low value of crystallinity index in sample A means poor order of cellulose crystals to the fibre axis, indicated by the lowest crystallinity index. After a treatment with urea in sample B, a 30% increase in crystallinity index from sample A to B indicates improvement in order of the crystallites. This is due to the removal of amorphous materials like hemicelluloses, lignin and some other non-cellulosic materials. The removal of surface impurities may facilitate both mechanical interlocking and bonding due to the exposure of hydroxyl groups to chemicals such as resins and dyes. A further increase of only 1% in the index is seen in sample C as compared to sample B, which was further bleached with 5 gpl hydrogen peroxide. This indicates that there was no significant increase in the crystallinity of the fibres on bleaching. However, sample D which was bleached at a higher rate of 20gpl hydrogen peroxide, showed a 10% decrease in C.I. as compared to sample B which was not bleached (control sample). This suggests an increase in percent of hydrogen peroxide has had a derogatory effect on the crystallite structure and it had got destroyed by strong bleaching conditions. The water retted sample, sample A procured from AAGAAS Federation (Alaknanda Ghaati Shilp Federation), extracted by the local people of Uttarakhand, gave 16% better C.I. as compared to manually pulled fibres which were not given any treatment but 16% lower C.I. as compared to sample C which gave the highest crystallinity index out of the above mentioned samples. The best C.I. was observed in sample F which sample C treated to a softener treatment. An increase in crystallinity indices with different treatments suggests increase in cellulose content. The index was as high as 56% higher as compared to best C.I. sample, sample C and 116% increase as compared to sample A-manually pulled and separated fibres.

4. CONCLUSION

In this study on study of morphology of bhimal fibres following points can be concluded:

4.1 SEM analysis of manually pulled, extracted and removed fibres from bhimal branch (sample A) showed a lot of deposits of gummy materials between and on the fibres since no treatment was given. This was confirmed by XRD analysis too as it has the lowest crystallinity index and percentage crystallinity, indicating low arrangement of crystalline materials to the fibre axis.

4.2 Amongst the combinations of urea retting at 5% owm without the use of a softener and hydrogen peroxide treatment (sample B, C, and D), use of 5 gpl hydrogen peroxide gave good results in terms appearance of fibres with lowest cementing material and it was also confirmed with the highest crystallinity index and percentage crystallinity amongst these samples. Use of a higher dosage of hydrogen peroxide @20 gpl gave derogatory effect on the fibres as they appeared to be dismantled in SEM images and crystallinity also dropped.

4.3 In case of Sample F in which urea retting was followed by 5 gpl hydrogen peroxide treatment followed by a softener treatment, highest degree of uniformity was seen in SEM images and lustre was also increased visibly in the fibres. Crystallinity index and percentage crystallinity was also highest amongst all urea retting and bleaching combinations.

4.4 Sample E-fibres extracted by local people of Dehradun also gave similar results to sample F discussed in the last point in terms of SEM images, crystallinity index and % crystallinity. But appearance of sample F was best in terms of lustre, softness, and uniformity. Therefore, a laboratory treatment beginning from urea treatment, followed by bleaching , further followed by a polysiloxane based softener treatment was found to be better to natural retting process as practiced by local people of Dehradun for extracting bhimal fibres.

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