Morphological Structure of Ramie for Acoustic

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Abstract:

Ramie a flowering plant in the nettle family Urticaceae, is considered as minor cellulosic fiber. Having beautiful brown colour, lustrous and good strength, these fibers are used for clothing since ages. As the extraction process is tedious and quality may differ depending upon the maturity, there are limitation in products. With the enzyme treatment the fibers were soften and changes in the structure will increase the commercial value of these fibers by using it for technical textile product. To identify the potentiality of the fibers for sound absorbing materials further an assessment was carried out to know structural changes by analysing both the untreated and treated fibers using various ASTM test methods – Bundle strength, SEM, FTIR, XRD and EDS. On comparing the results with previous studies, it was observed that the fibers are more aligned and porous which is needed for developing sound absorbing samples. Thus, these fibers were taken further to spinning and fabric manufacturing levels for the development of woven sound absorbing materials.

Keywords: *Eco-friendly products, Enzyme Treatment, Ramie Fiber, Sound Absorbing Materials, Structural Properties*

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1. Introduction

Sustainable and recyclable products are the need of the hour, as pollution levels are increasing day by day in India due to various factors. Noise pollution has become one of the major concerns for the physical and mental health both at work and residential. With the demand, one of the minor cellulosic fiber – Ramie was explored as acoustic solution. Ramie or Boehmeria nivea (L.) Gaud has a perennial shrub possesses good tensile strength, durability, lustre, microbe resistant property, but are glued together by gummy substance to hold the fibers together and are known as bundled fibers [1]. The mentioned natural characteristics alongwith the inherent hollow structure of the fiber is important factors to develop sound absorbing materials and so was explored to create woven fabrics having elements of aesthetics also.

Ramie, an earthy colour lustrous fiber can have another product line and thereby the utilization will increase. A non-hazardous pre-treatment procedure is needed to reduce the gummy substances of the fibers so that pliability increases. Thus, an experiment with a motto of commercial viability enzyme treatment was conducted which helps in water conservation as well as saves energy too. The treatment increased the softness, lustre and pliability of the fibers and further analysis was conducted to create functional products using the inherent properties.

2. Materials and Methods

2.1 Materials

Fiber Selection

To develop eco-friendly sound absorbing woven samples, ramie fiber was purposively selected based on the fiber structure, inherent properties and its availability. The procurement of un-degummed fibers was from Ramie

Research Station, (CRIJAF – ICAR), Sorbhog, Barpeta, Assam. For the removal of all the impurities scouring process was conducted, which also helped in penetration of enzymes in the fiber structure.

Enzymes

Enzymes are a class of proteins that function as biocatalysts by lowering the activation energy of a reaction making it much faster. The enzymes were used for improving the feel of the fabric. Four different enzymes – Greenboost liquid (Pectinase), Biocool Z-20 powder (Hemicellulase), G-zyme axe liquid (Cellulase) and Denilite® || S (Laccase) were selected for the study and procured from Rossari biotech Ltd. and Novazymes A/S [3]. The enzymes during the treatment procedure penetrates into the fiber, act and reacts with each layer of the fiber and weakness lignin which is the main component that keeps the fiber stiffer. These enzymes interrelate with bonds and thereby the modification in the structural properties takes place by breaking or weakening the bonds. Table 1describes all the different stages of treatment with its process sequence [11].

Table 1: Coding of the raw and treated ramie fibers

Sr. No.	Codes	Description
1.	Rr	Ramie Raw
2.	Rhcbc	Ramie - High per cent concentrated – combing - beating – combing
3.	Rhcbc4	Ramie - High per cent concentrated (4hrs treatment without changing water) – combing -beating – combing

2.2 Methodology

Enzyme treatment

With the conventional lab method, the treatment was conducted in which the fibers were soaked completely and was easy to rotate with less of entanglements. Standardized recipe and procedure of previous research was applied onto the fibers with M: L ratio i.e. 1:40, temperature 55℃ and 5 pH are as constant. Other variables like per cent concentration of the enzymes and treatment methods like padding mangle, beating and combing of the fibers were experimented. The optimized recipe was finally applied and considered for the study. The details of optimized recipe and treatment steps are mentioned in Table 2.

Table 2: Process of enzyme treatment on Ramie fibers

Sr. No.	Steps	Rhcbc	Rhcbc4
1.	Beating	10mins	10mins
2.	Combing	Combing of the fibers in small bundles	
3.	Pectinase	2%	2% (Stepwise addition of enzymes in single bath)
4.	Laccase	10%	10%
5.	Cellulase	7%	7%
6.	Hemicellulase	5%	5%
7.	Oil	25%	25%
8.	Batching	Overnight	
9.	Combing	Combing of the fibers in small bundles	

With the ongoing concept of water conservation, researcher treated two lots of fibers with same recipe but change in steps i.e. treating one lot (Rhcbc4) with all the four enzymes but without changing water. Then the fibers were immersed into the oil emulsion followed by batching treatment which improves the pliability by smoothing the surface texture of the fiber.

After standardizing the treatment procedure, further experiment was conducted for easy bulk treatment process alongwith proper rotation of the fibers so that enzymes penetrate equally in the fiber structure. Both the process was conducted using Infracolour and Launder-O-Meter machines. Finally, Rhcbc4 treatment procedure using Launder-O-Meter machine was carried out for the study based on two factors - the concept of water and time management alongwith the quality of fiber.

Evaluation of fiber properties

Subjective analysis (feel and touch method) and pliability test of all the three samples - raw ramie (Rr - standard sample), Rhcbc and Rhcbc4 (enzyme treated samples) were done. Porous structure of the fiber is one of the major factors to trap the sound. Thus, certain physical and structural properties of the fibers were analysed using ASTM methods.

Tensile strength with ASTM D 3822 was carried out on Llyod Instron Tensile Test for identifying the change in the strength of fiber after the treatment. The whiteness Index test was determined using CIE and ASTM D 1925 standard on Spectrophotometer instrument to identify the change in fiber colour. While to identify the morphological changes after the treatment four tests were conducted on Scanning Electronic Microscope **(**SEM), Fourier-transform infrared spectroscopy **(**FTIR), X-ray Diffraction **(**XRD) and Energy-dispersive X-ray spectroscopy (EDS). From the analyses one best sample was further selected for developing woven sound absorbing samples.

3. Results and Discussion

Physical properties

Ramie, a rough bundled fiber has nodes at various intervals when observed under the microscope. The brown lustrous fiber was found to be of 48 cm in length and each bundle of fiber consists of many sub fibers. The average diameter and denier measured between 79 µm and 739 respectively. The fiber is known for its good tensile strength and absorbent characteristics owing to its inherent hollow structure.

Tensile properties

The durability of any product depends on the strength, thus bundle strength of fibers using Instron machine was analysed and the details are given in Table 3.

Table 3: Tensile Strength of ramie fiber

Sample code	Maximum Load (gf)	Extension at Max (mm)	% Strain	Tex	Stress in gm/Tex
Rr	5407	0.39	12.87	242	22.34
Rhcbc	3167	0.69	23.13	153.2	20.67
Rhcbc4	3626	0.48	16	146.8	25.21

Ramie fibers are soft and surrounded by the rigid cellulose component. This cellulose structure is crystalline and porous but the removal of impurities was needed for smooth surface and good spinnability. Thus, the enzyme treatment given to the fibers might have removed the impurities and thereby the bonds have been aligned, which has converted the enzyme treated fibers stronger than the raw ramie.

The strength of the fiber has reduced after the treatment, which could be due to the changes in the nodes or maybe it is the impact of process during the treatment. The stress bearing capacity of the treated fiber Rhcbc4 has increased and thereby %strain increased compare to raw. The elongation being an important factor for spinnability has also increased in Rhcbc4 compare to raw. Thus, based on lustre, softness and spinnability Rhcbc4 showed best result and was selected for the yarn preparation.

Whiteness Index

The change in the colour of ramie fibers were observed after enzyme treatment, which is due to the removal of pectin, fats and waxed from the fiber. To identify the change in yellowness, whiteness and brightness was analysed by whiteness index test. The purpose of this test was to understand the aesthetical properties of the product with the change of fiber colour. The details are given in Table 4.

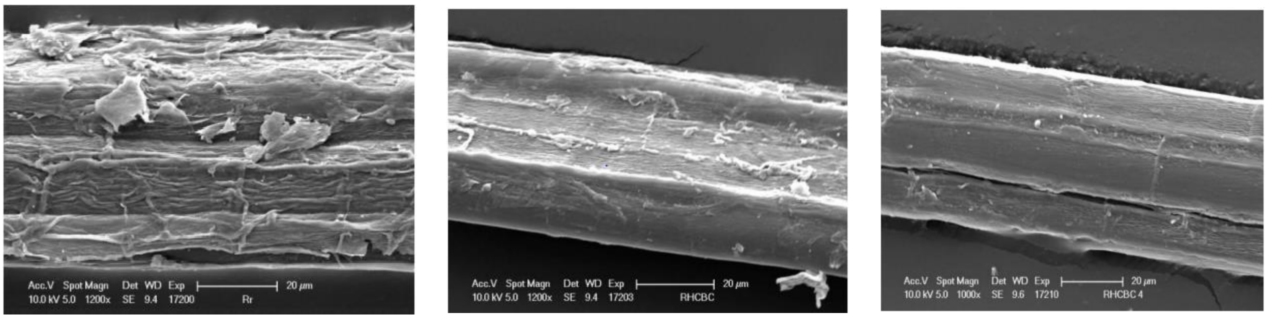
Table 4: Whiteness index data of ramie fiber

Sample Code	Whiteness Index	Yellowness Index	Brightness Index
Rr (Raw ramie)	45.13	52.59	15.65
Rhcbc	49.72	52.88	19.19
Rhcbc4	53.91	41.02	24.20

From the results it was observed that more of whiteness was achieved in Rhcbc4 – enzyme treatment without changing water. While negligible difference was observed in the yellowness of both the fibers, which could be because of cellulase and laccase enzymes.

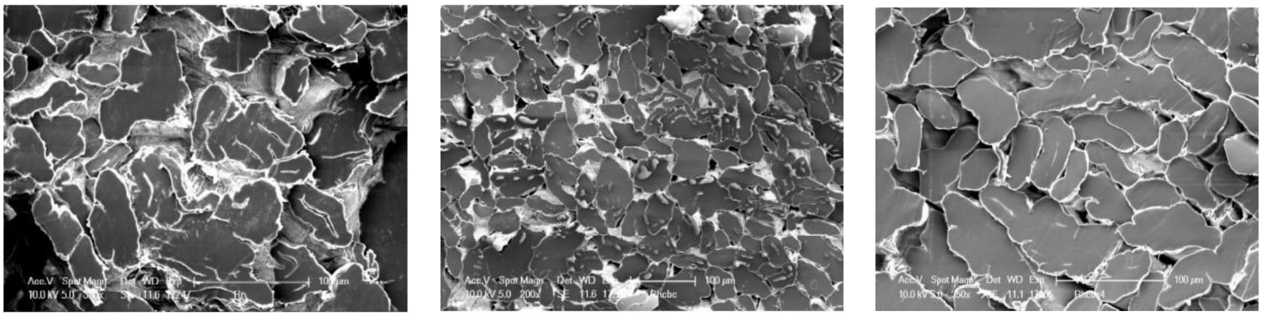
Scanning Electronic Microscope (SEM)

Scanning Electronic Microscope (SEM) was conducted to understand the morphological difference between untreated and treated fibers. In the hydrolysis process each and every enzyme reacts with the fiber, which consists hemicellulose, lignin and pectin. The raw ramie fiber is comparatively less dense with space in between the fibers; oval shape and variation in the size of the fibers were observed along with the lumen in the cross-sectional view of Plate 1.



Rr **Rhcbc** **Rhcbc4**

a) Longitudinal View



Rr **Rhcbc** **Rhcbc4**

b) Cross sectional view

Plate 1: Longitudinal and Cross-section SEM images of enzyme treated ramie fibers with variation in process

Thus, the Rhcbc4 fiber showed changes in lumen part, is more porous in structure, more aligned and visible after the treatment, the pithy materials were comparatively removed by the enzymes. Hence based on the observation and need of the study sample Rhcbc4 were carried further for yarn conversion to develop woven sound resistant materials. The sound that will penetrate into the fibers will be trapped into the lumen or gaps within the fibers and to some extent the scattering of sound will also play a major role in sound resistance.

Fourier –transform infrared spectroscopy (FTIR)

The FTIR spectra of ramie fiber samples shows similar peaks, but more prominent variation in intensity was observed in sample Rhcbc4 (Figure 1). Rhcbc showed a weak peak around 3500cm-1 while broader and deep peak was observed in sample Rhcbc4 which arranges from 3200 - 3500 cm-1 due to the bonded O-H group. Other uneven peaks were observed in the range of 1300-1600 cm-1, which are of C=O stretching, C-H deformation and stretching and C-O deformation bonds. However, in the case of Rhcbc4 a peak was observed nearby 1100 cm-1, which might be due to stretching and deformation of C-O and C-H bonds. All the mentioned ranges were associated with the lignin; hence it could be said that the treatment and process had an impact on the softening of the fiber.

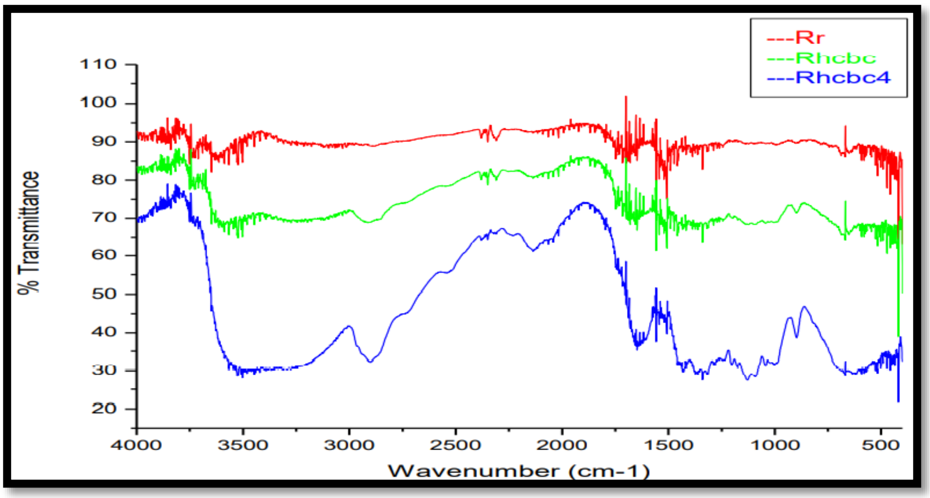


Figure 1: FTIR graph of untreated and treated ramie fibers with different process

Thus results also show that the treatment remarkably decreases certain components, such as cellulose, hemicellulose and lignin. The change in the components leads to the change in other properties, surface area and crystallinity of the fiber.

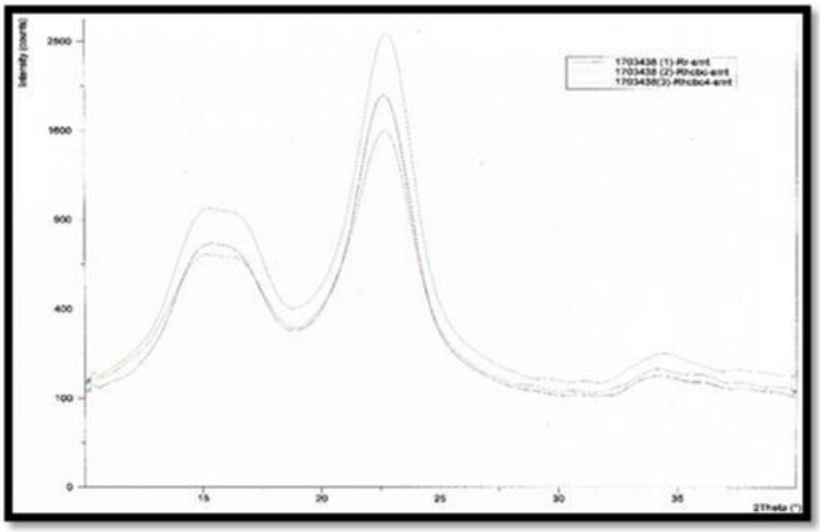
X-ray Diffraction (XRD)

XRD analysis was conducted, from the data in table 5 it seems that destruction in the chains has an impact on crystallinity, crystalline size and orientation angle at 2θ, in both the treated fibers compare to untreated fibers. Due to enzyme treatment the swelling in the chains was found in the SEM and thus the sample Rhcbc showed highest crystalline size. Additionally, the surface modification had a positive impact on pliability and this change in the property might assist in sound absorption also. Hence, with the removal of non-cellulosic compounds, crystallinity has increased and softness of the fiber was the resultant.

Table 5: XRD of bundle fibers scanned in Transmission mode 10-40°

Sample Code	Crystallinity (%)	Orientation angle at 2θ (°)	Crystallite Size (°A)
Rr (Raw Ramie)	81.77	14.3	32.42
Rhcbc	85.59	11.56	34.06
Rhcbc4	84.90	13.46	32.44

The X-ray diffraction of untreated (Rr) and treated ramie (Rhcbc & Rhcbc4) fibers are shown in Graph 1. Both the Rhcbc4 and Rhcbc shows rising peaks compare to Rr at 16° and 22°. Rhcbc4 shows peak at 16°, where the intensity of the peak has increased compare to the other samples with uneven broader width suggests the destruction in the amorphous region.



Graph 1: X-ray diffraction of untreated and enzyme treated ramie fibers

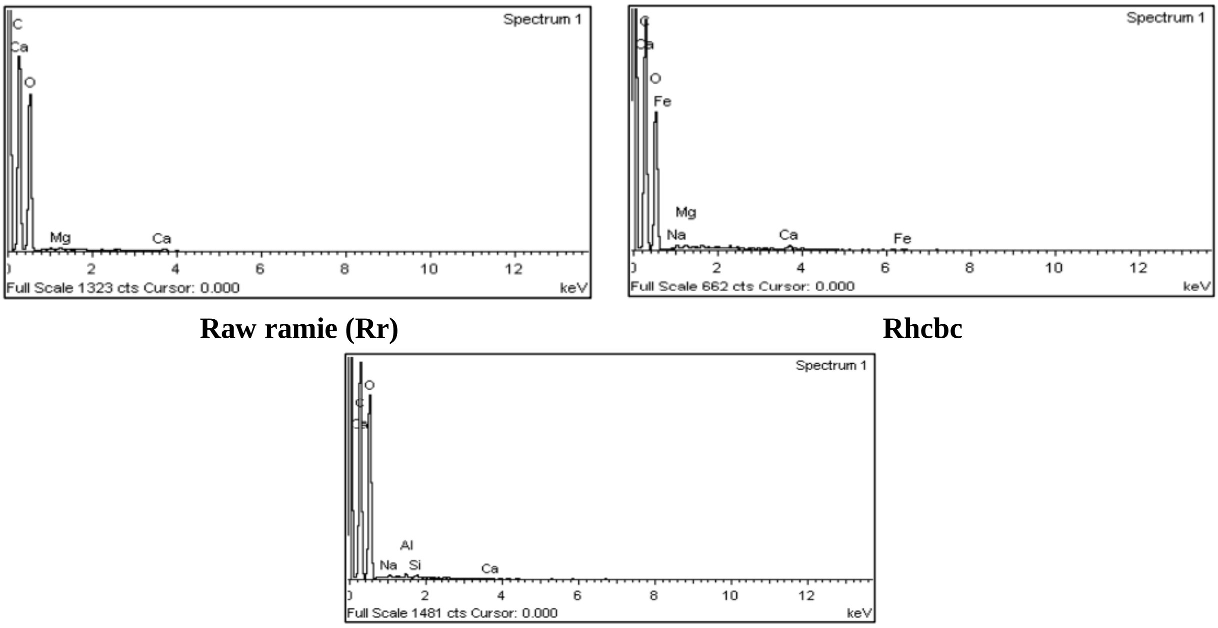
Energy-dispersive X-ray spectroscopy (EDS)

The treatment makes the changes in the bonds of the fiber. Thus, EDS analysis was conducted to know the change in the elements of the treated fiber and the details are mentioned in Table 6.

Table 6: Details of EDS elements of all the ramie fibers

Element	**Rr (Raw Ramie)**		**Rhcbc**		**Rhcbc4**	
	Weight%	**Atomic%**	**Weight%**	**Atomic%**	**Weight%**	**Atomic%**
C K	27.21	33.27	27.07	33.17	27.13	33.19
Mg K	0.07	0.04	0.09	0.05	-	-
Ca K	0.15	0.05	0.16	0.06	0.04	0.02
O	72.57	66.64	72.35	66.55	72.49	66.59
Na K	-	-	0.20	0.13	0.21	0.12
Fe K	-	-	0.13	0.04	-	-
Al K	-	-	-	-	0.08	0.05
Si K	-	-	-	-	0.05	0.03
Total	100.00	100.00	100.00	100.00	100.00	100.00

The elements present in the Ramie untreated fibers are Carbon (C K), Magnesium (Mg K), Calcium (Ca K) and Oxygen (O). Insignificant difference in the Carbon and Oxygen elements has been observed from the Graph 2. In both the treated samples – Rhcbc and Rhcbc4, reduction of calcium was observed. While, Sodium element is due to the use of Soda ash in the scouring recipe, which is in permissible limit. Additionally, elements like Magnesium, Iron, Aluminium and Silicon in the sample Rhcbc and Rhcbc4 respectively could be the result of reaction of enzymes with the metal body of the instrument or containers used during the enzymatic process.



Raw ramie (Rr) **Rhcbc**

Rhcbc4

Graph 2: EDS graphs of untreated and treated ramie fiber

Hence, all the changes and addition of elements were in permissible limits. So, the treatment recipe was effective, for the softening of the fibers and is harmless to the ecology and environment.

4. Conclusion

Ramie a lustrous fiber with hollow fiber structure is rough at the raw stage. To change the rough texture and improve spinnability enzyme treatment was carried out. Morphological changes were observed in the treated ramie fibers during physical and structural analysis.

The changes in lumen part, more porous structure and more aligned fibers will assist sound to penetrate, trap and scatter into the fibers. From the resources point of view, utilization of water and time has been reduced with the use of Launder-O-Meter, thus giving an opportunity to the experts to develop similar instrument to promote the ecological treatment process. Thus, with the sustainable process, a range of sound absorbing woven fabrics can be developed with aesthetical values.

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