

Processing of Ramie Fiber with Enzymes for Nonwovens



Textiles

KEYWORDS : Ramie fiber, Hemicellulase, Pectinase, Cellulase, Needle punch, Nonwoven

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ABSTRACT

Ramie is commonly known as china grass, green ramie or rhea. It is referred to as bast fiber. The fiber has limited application in textiles due to its high stiffness and less cohesive properties. The aim of the study was to remove non-cellulosic gummy materials from the fiber thus softening it and prepare nonwovens through needle punch method. Grey ramie fibres were treated with hemicellulase, pectinase and cellulase enzyme individually by varying its concentration and time. This optimised treatment was further treated with different combination of enzymes, which was later standardised. The effect of enzymes on the fibers was measured in weight loss, strength loss and whiteness index of the fiber. Subjective analyses by touch and feel method, SEM analyses was also done. The best softened fibers were processed for preparation of nonwovens through needle punch method at NIRJAFT, Kolkata

Introduction

Nature offers us a lot of material that can be called fibrous. Vegetable fibers are also called as cellulosic fibers which are completely renewable and biodegradable. Natural cellulosic fibers have occupied a very important place as textiles since mankind developed on earth. Due to the global awareness for eco-friendly products, natural fibers are again gaining importance. These vegetable fibres are strong, light in weight, abundant, non-abrasive, non-hazardous and inexpensive. Ramie (*Boehmeria Nivea*) is a flowering plant in the nettle family *Urticaceae*, native to eastern Asia. It is one of the oldest fiber which is cellulosic in nature. It is bast fiber and the part used is the bark of the vegetative stalks.

The reason for limited use of this fiber is its stiffness and very less cohesiveness. The individual cells are cemented with hemicellulose and lignin and thus form a complex fibre. Therefore there is no spinnability of the yarn into fine Denier. As they are not able to spun, hence the fibers can be used in fibrous state. Thus non-woven was the best option to give a new dimension to the fibers.

To produce nonwoven through needle punching method, the fibers were softened with enzymes which could prove to be the best alternative as it is regarded as environmentally friendly and the reactions catalysed are very specific with a focussed performance. In contrast, chemical processes are less specific and often result in side effects, mostly undesired. These enzymes help in removing the unwanted components from the fiber and thus making it soft.

In this study, the researcher has attempted to remove the non cellulosic with an enzymatic treatment and thus making the fiber soft.

Materials and Methods

Untreated ramie fibers were procured from Ramie Research Station, Odisha. Enzyme BGLU with hemicellulase activity (E1) and Palcoscour enzyme with pectin activity (E2) was supplied by Ros-sari biotech PvtLtd., Bangalore, India and Biofast SBE with cellulase activity (E3) was supplied by Maps (India) Ltd, Ahmedabad, Gujarat. Enzyme solution (1%) was prepared by dissolving 1.0 gram of enzyme in 100ml of 1ml glacial acetic acid solution prepared in distilled water at room temperature. Two grams of ramie fibers were treated with enzyme solution of particular concentration at pH 5 at 50-55°C at 1:60 M:L ratio. After the treatment the samples were washed with distilled water and deactivated. Treatment conditions were optimised at different enzyme concentration (10 and 12% dwf) and time (8,12, and 14 hours) with respect to weight loss.

The chemical compositions of the gray fiber were determined

using A.J. Turner's scheme. Percentage weight losses were calculated from the different weight of samples (conditioned at 27±1°C and 65% for 24 hours) before and after the treatment and expressed in percent over the original weights of the sample. Strength loss of the fibers was also calculated. Whiteness and yellowness indices of the fibers were measured at standard conditionson Spectrascan 5100A Spectrophotometer (premier colour scan, India). Further the optimised concentration and time were used to treat the fiber with the combinations of the enzyme to form a standardise recipe.

Subjective analysis by touch and feel method was done for the softness of the fiber. The best softened fiber was SEM tested and further treated on 10kg fiber for preparing nonwovens through needle punch method. The fabrics were prepared at NIRJAFT, Kolkata. The machine and the fabric parameters to prepare non-wovens were selected by the NIRJAFT depending upon the softness and properties of the fibers.

Results and Discussions

Ramie fiber under microscope reveals that the cells had radial cracks. These cracks were nothing but dislocation folds in the straight continuity of the fibrils in the secondary wall.

Being cellulosic in nature, it dissolves only in 72% concentrated sulphuric acid when subjected to heat. It burns quickly when subjected to heat and gives paper burn smell. The physical properties of the fiber were tested at standard conditions which are presented in Table 1.

Table 1: Physical properties of the ramie fiber under study

Sr. No	Property	Value
1	Length of the fiber (cm)	51
2	Fineness of the fiber (Denier)	201
3	Moisture regain (%)	7.99
4	Tensile strength (gm/tex.)	42.48

The chemical composition of the gray ramie fiber was analysed by A.J. Turner's scheme (Table 2)

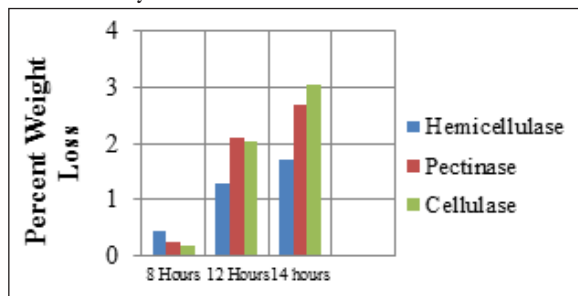
Table 2: Chemical composition of untreated ramie fiber

Sr. No	Content	Ramie fiber (%)
1	Cellulose	77.5
2	Hemicellulose	9.9
3	Lignin	4.45
4	Pectin	4.45
5	Fats and waxes	3.2

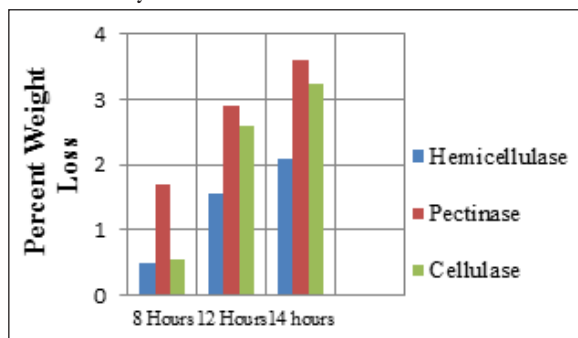
Ramie fiber behaves differently (as expressed by weight loss) at different reaction conditions with enzymes. Therefore it was treated with different enzyme concentrations for various time periods. The percent weight losses of the fiber are given in Graph 1 and 2.

The results from the graph 1 and 2 indicate that the percent weight loss increases with increased severity of the treatment conditions of enzymolysis. This weight loss occurs due to the removal of impurities from the ramie fiber.

Graph 1: Percent weight loss of Ramie fiber at 10% concentration of enzyme.



Graph 2: Percent weight loss of Ramie fiber at 12% concentration of enzyme



During the enzyme treatment, hydrolysis of hemicellulose occurs along with the removal of other impurities. The BGLU has a hemicellulose degrading activity, which consists of several xylan- hydrolysing components with exo and endo activity as well as side chains cleaving glucuronides and acetyl xylan esterase's detaching o-acetyl groups. Endo types attack hemicellulose chain, decreasing the degree of polymerisation of the substrate. Cellulases hydrolyse the β -1,4-glycosidic linkages of cellulose. Traditionally, they are divided into two classes referred to as endoglucanases and cellobiohydrolases. Endoglucanases (endo-1,4-b-glucanases, EGs) hydrolyses the internal bonds (preferably in cellulose amorphous regions) releasing new terminal ends. Cellobiohydrolases (exo-1,4-b-glucanases, CBHs) acts on the existing or endoglucanase-generated chain ends. Both enzymes degrade the amorphous cellulose but, Cellobiohydrolases are the only enzymes that efficiently degrade crystalline cellulose. (10)

While pectinase enzyme (E2) is a mixed enzyme which effectively removed pectin, fats and waxes. From Graph 2, the maximum weight loss of 3.24% was observed in the fiber when treated with pectinase enzyme at 12% concentration for 14 hours. The maximum noncellulosic constituent seen in the ramie fiber is pectins, fats and waxes and other impurities. Therefore, pectinase enzyme helped more in removing the impurities from the fiber. Therefore the weight loss in ramie both at 10% and 12% concentration was much due to pectinase (E2) as compared to hemicellulase (E1) and cellulase (E3).

Table 3 shows the percent strength loss of the ramie fiber when treated with enzymes individually. The results indicate that the percentage tensile strength of the fiber decrease with the increase in the weight loss. Noncellulosic materials impart strength to the fibers which are removed with the progress of the enzymatic treatment. This lowers the tensile strength of the fibers. The strength loss in the fiber at 12% concentration is maximum due to pectinase enzyme.

Table 3: Percent strength loss of ramie fiber

Sr. No	Percentage of enzyme (%)	Enzyme	Percent Strength loss		
			8 Hours	12 Hours	14 Hours
1	10	E1	4.4	5.36	9.5
2	10	E2	8.5	9.2	15.2
3	10	E3	8.14	9.6	10.8
4	12	E1	6.8	6.9	12.7
5	12	E2	9.1	9.6	16.41
6	12	E3	8.95	9.4	12.9

Enzyme Code: E1- Hemicellulase, E-2 Pectinase, E3- Cellulase

Enzymes help the fiber to remove the pectin, fats and waxes. This helps in reducing the yellowness colour of the fibers and thereby increases the whiteness of the fibers. Therefore whiteness and the yellowness indices if the fibers were also measured and is presented in Table 4. The result for 12% concentration for 12 hours showed a good result with maximum brightness and minimum yellowness.

Table 4: Whiteness index of Ramie Fiber

Sr. No	Name	Whiteness	Yellowness	Brightness
1	Untreated	43.421	59.373	14.108
2	E1 12% 8 HOURS	42.318	63.715	13.18
3	E2 12% 8 HOURS	42.999	58.899	13.958
4	E3 12% 8 HOURS	38.207	63.642	10.737
5	E1 12% 12 HOURS	42.742	51.45	14.373
6	E2 12% 12 HOURS	46.412	49.7	17.035
7	E3 12% 12 HOURS	43.276	53.116	14.527
8	E1 12% 14 HOURS	45.438	49.055	13.333
9	E2 12% 14 HOURS	46.905	50.797	18.174
10	E3 12% 14 HOURS	45.223	50.379	16.209

Enzyme Code: E1- Hemicellulase, E-2 Pectinase, E3- Cellulase

From Table 4, it was concluded that 12% concentration for 12 hours gave the best results on the fibers when treated with pectinase enzyme. Hence the enzymatic treatment was optimized at 12% concentration for 12 hours. The noncellulosic impurities present in the grey fibers are hemicellulose, pectin, lignin, fats and waxes. These have specific activity towards various constituents of the fibers. Therefore, an individual enzyme cannot impart adequate level of performance even though it gives weight loss in the required range. This is because a single enzyme cannot

remove all noncellulosic constituents uniformly from the gray fibers. Therefore in order to achieve satisfactory enzymatic performance, three enzymes were mixed together.

The fibers were further treated with different combinations of enzymes. The weight loss of the fibers represents the removal of the noncellulosic components in the fibers. Therefore, weight loss of the each combination was measured.

Table 5 shows that the maximum weight loss in the ramie fiber was observed when the enzyme combination was 0% hemicellulase, 5% pectins and 7% cellulase. As the percentage of hemicellulase in the ramie fiber is only 9.9% hemicellulase enzyme was not used in the combination. When the cellulase enzyme is treated with the fiber for 12 hours, it would give the surface softening and also would weaken the hemicellulose bond to some extent. And pectinase enzyme would enter inside the fiber and remove the pectins efficiently.

Table 5: Effect of optimum enzymatic treatment conditions on weight loss and strength loss of the Ramie fiber

Sr. No	Concentration of Enzyme (%owf)			Weight loss (% owf)	Strength loss (%)
	E1	E2	E3		
1	2	10	0	6.2	8.3
2	5	7	0	7	9.12
3	0	5	7	9.1	9.8

Enzyme Code: E1- Hemicellulase, E-2 Pectinase, E3- Cellulase

In order to prepare nonwoven fabric, it was necessary to maintain the strength of the fibers. Therefore, bundle strength test of the fibers was tested. Strength loss increased with the increase in weight loss. Table 5 showed that the fiber had minimum strength loss at 2:10:0 enzyme combination and the maximum strength loss was observed when the fiber was treated with 5:7:0 enzyme concentration.

The fibers when treated with single enzymes showed very minimal effect on the whitening of the fibers. Therefore the effect of the combination of the enzyme on the whitening of the fibers was determined.

Table 6: Whiteness index of the combinations of enzyme on the ramie fiber

Sr. No	Name	Whiteness	Yellowness	Brightness
1	Untreated	43.421	59.373	14.108
2	12% 2:10:0 12 HOURS	50.745	46.157	20.8
3	12% 5:7:0 12 HOURS	57.114	37.41	27.683
4	12% 0:5:7 12 HOURS	48.771	46.578	19.163

Hemicellulase: Pectinase: Cellulase

Table 6 revealed that the whiteness and brightness had increased and yellowness had decreased when the ramie fiber was treated with 5% hemicellulase, 7% pectinase and 0% cellulase enzyme.

The fibers treated with the combinations of the three enzymes where given to panel of five judges along with the untreated fiber. The majority chosen combination by touch and feel method for the fiber was selected as the best softened fiber. The best combination of the enzymes selected for the fiber was 0% hemicellulase, 5% pectinase and 7% cellulase.

Further SEM tests of the untreated and the best soften fiber were done at the magnification of 100X and 550X.

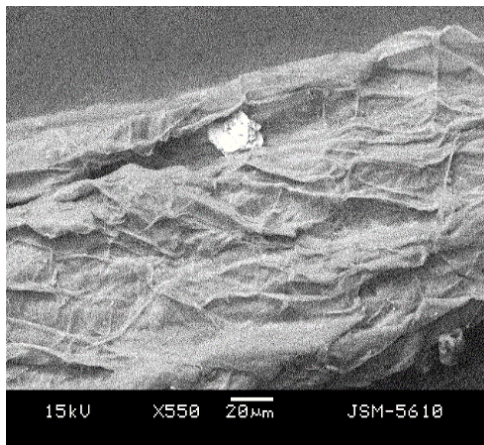


Plate 1: SEM test of untreated ramie fiber at 100X

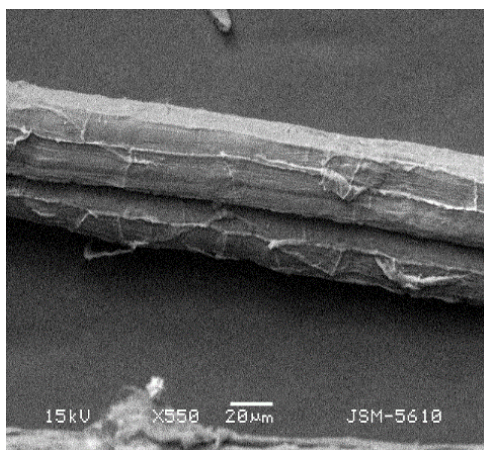


Plate 2: SEM test of treated ramie fiber at 100X

The primary function of fiber softening is to make the fibers surface smooth. Plate 1 showed that the untreated sisal fiber had rough and irregular surface. While from the Plate 2 it was observed that the surface of the fiber after treatment was smoothen to some extent.

From the above analysis done by the weight loss, strength loss, whiteness index, SEM test and softness test, fibers treated with 0% hemicellulase, 5% pectinase and 7% cellulase enzyme combination was selected best for the preparation of the nonwovens.

The best softened fibers selected were further treated on 10kg of fiber for preparing nonwovens through needle punch method. The fabrics were prepared at NIRJAFT, Kolkata. The machine and the fabric parameters to prepare nonwovens were selected by the NIRJAFT depending upon the softness and properties of the fibers. The nonwoven fabrics prepared were 100% ramie with cross laid of different GSM. Combination of the fabric was also prepared by blending 60:40% ramie and bananafiber of different GSM.

For preparing nonwoven fabrics, the fibers were subjected to softening treatment by passing the fibers through softener machine. The fibers were passed through the rollers and the dust material of the fibers if any is removed. Then the fibers were subjected to the treatment of 2% oil-water with emulsion and kept for 24 hours in a closed chamber to maintain 30% moisture in the fibers. The fibers were then processed in a beaker card where slivers are formed. The beaker card ramie and banana slivers were then hand opened thoroughly and processed in a DILO nonwoven plant comprising a card, a camel-back cross lapper and needle loom (DILO OD II/6) for making cross laid nonwo-

ven. For blended fabric, hand opened ramie and banana were mixed thoroughly in 60:40 dry weight proportion following the stack mixing technique and then processes in DILO nonwoven plant as stated above.

Ramie fiber showed good cohesiveness property. Therefore the 100% ramie nonwoven fabric was successfully prepared on DILO nonwoven machine. As the cohesiveness of the ramie fiber was good enough, the fibers when mixed in the proportion of 60:40 ramie and banana, nonwoven fabric was effectively prepared. The number of needle on the needle board of the DILO machine was 2464 while the machine parameters were kept constant.

Table 7: Constructional details of experimental fabrics

Blended composition	Fiber laying technique	Needle penetration	Punch density Punches/cm ²	Stroke frequency Stroke/min	GSM
100% Ramie	Cross- laid	13mm	100	214	452 and 625



Plate 3: 100% ramie nonwoven fabric of 452 GSM (Cross laid)

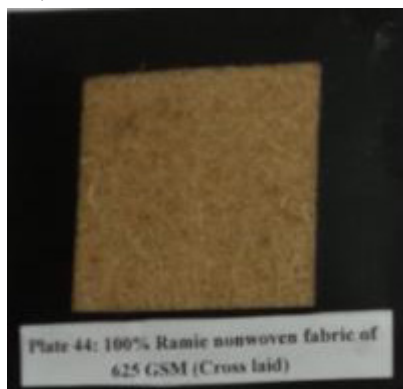


Plate 4: 100% ramie nonwoven fabric of 625 GSM (Cross laid)

Conclusion

The three eco-friendly enzymes used in this study were hemicellulase, pectinase and cellulase enzyme. These were used to soften the fiber by removal of pectins, waxes, lignin and hemicellulose.

From the single enzyme treatment it was also concluded that-

The pectinase and cellulase enzyme removed more impurities from the ramie fiber thus making it softer.

Good results were not seen for improvement of whiteness index.

The single enzyme treatment on the fiber gave best result at 12%concentration (owf) at 12 hours. And hence the enzymatic treatment was optimized at 12% concentration (owf) for 12 hours.

But the fiber when treated with enzymes individually they removed particular impurity only. The impurities were not evenly removed as the enzymes are specific in their action. i.e. Hemicellulase enzyme removes hemicellulose from the fiber, pectinase enzyme removes pectins and cellulase enzyme affects the cellulose and makes the surface soften.

Therefore, the fibers were treated with combination of enzymes. It was concluded that the fibers when treated with the combination of the enzymes gave better results than the fibers treated with the enzymes individually. The impurities i.e. the non-cellulosic particles from the fiber were removed more evenly when treated with combination of enzymes.

It was also concluded that the weight loss in the fibers increased with increase in concentration and time. And more the weight loss in the fiber more was the strength loss as the removal of more amount of impurities and fats would loosen the binding force and thus reduce its resistance to breaking on application of load.

Analysis of the treatment of combination of the enzymes on the fibers gave a standard combination recipe for the fiber. The best combination selected for the fiber was 0% hemicellulase, 5% pectinase and 7% cellulase.

100% ramie nonwoven fabric of cross laid with different GSM were prepared. 60:40% ramie: banana nonwoven fabric of cross laid with different GSM were also prepared. The fabrics were made in the DILO nonwoven mechanical needle punch machine.

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