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Enzyme Softening of Sisal Fiber: A Sustainable Approach for the Future

KEYWORDS
Sisal fiber, Hemicellulase, Pectinase, Cellulase, Needle punch, Nonwovens.

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ABSTRACT *Sisal occupies 6th place among the plants, representing 2% of the world's production of plant fibers. The fiber has limited application in textiles due to its high stiffness and less cohesive properties. The aim of the study was to remove noncellulosic gummy materials from sisal fiber thus softening it and prepare nonwovens through needle punch method. Grey sisal fibers 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, and was standardised. The effect of the enzymes on the fibers was measured in weight loss, strength loss and whiteness index of the fiber. Subjective analyses by touch and feel method, and SEM analyses were also done. The best softened fibers were then processed for preparation of nonwovens through needle punch method at NIRJAFT, Kolkata.*

Introduction

Natural cellulosic fibers have occupied a very important place as textiles since mankind developed on earth. Minor Fibres like Flax, Jute, Banana, Sisal, Ramie and fibres of similar plants have been used for more than 8,000 years. Agave sisalana Perrine (Agavaceae), popularly known as sisal in is a monocotyledonous plant from Mexico. Sisal derives its name from a small port in the Yucatan peninsula of Mexico through which the earliest supplies of Agave fibers were exported and it became known to commerce as Sisal or 'sisal hemp'. Sisal is a leaf fiber mainly grown in India in arid and semi-arid regions of Andhra Pradesh, Bihar, Orissa, Karnataka, Maharashtra, Chhattisgarh and West Bengal. It is now the world's most important leaf fiber, consisting more than half of the total commercial production of all leaf textile fiber. The reason for limited use of this fiber is its stiffness and vey 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 nonwoven was the best option to give a new dimension to the minor 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 noncellulosics from the fiber with an enzymatic treatment to soften it.

Materials and Methods

Untreated sisal fibers were procured from Sisal research station, Odisha. Enzyme BGLU with hemicellulase activity and Palcoscour enzyme with pectin activity was supplied by Rossari biotech PvtLtd., Bangalore, India and Biofast SBE with cellulase activity was supplied by Maps (India) Ltd, Ahmedabad, Gujarat. Enzyme solution (1%) was prepared by dissolving 1.0 gram of enzyme in 100ml of 1 ml glacial acetic acid solution prepared in distilled water at room temperature. Two grams of sisal 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% owf) 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 conditions on 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 further SEM tested. The best softened fiber selected was 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 nonwovens were selected by the NIRJAFT depending upon the softness and properties of the fibers.

Results and Discussions

Sisal fiber under microscope reveals that the fiber had straight striations. No cross markings were observed. 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.

Table 1: Physical properties of the sisal fiber under study

Sr. No	Test	Sisal fiber
1	Length of the fiber (cm)	84
2	Fineness of the fiber (Denier)	186
3	Moisture regain (%)	4.6
4	Tensile strength (gm/ tex.)	43.36

The physical properties of the fiber were tested at standard conditions which are presented in Table 1.

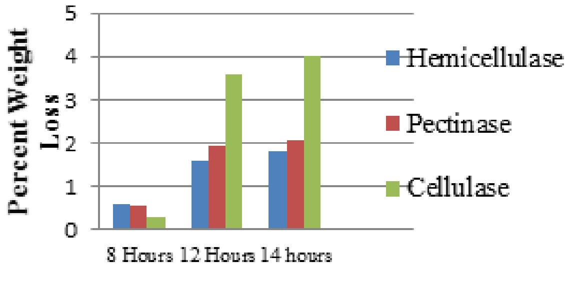
The chemical composition of the gray banana fiber was analysed by A.J. Turner's scheme (Table 2)

Table 2: Chemical composition of untreated sisal fiber

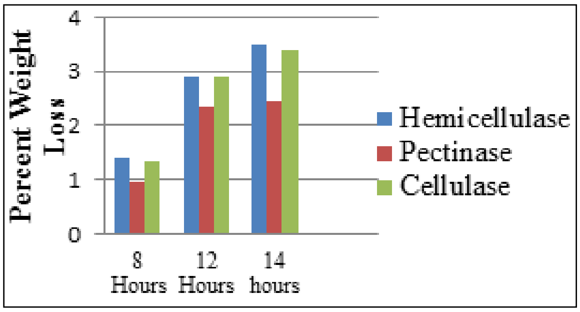
Sr. No	Content	Sisal fiber (%)
1	Cellulose	75.55
2	Hemicellulose	9.45
3	Pectin	1.6
4	Lignin	5.5
5	Fats and waxes	3.95

Sisal 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.

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



Graph 2: Percent weight loss of sisal fiber at 10% concentration of enzyme



The results 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 sisal fiber. 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 glucoronides 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 ß-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.

While pectinase enzyme (E2) is a mixed enzyme which effectively removed pectin, fats and waxes. Graph 2 revealed thatthe maximum weight loss of 3.5% was observed when treated with hemicellulase enzyme at 12% concentration for 14 hours and 3.4% with cellulase enzyme at 12% concentration for 14 hours. It means that the maximum impurities were removed from hemicellulase and then cellulase enzyme. As sisal fiber contains more percentage of lignin which is present all over the fiber and fills the spaces in the cell wall between the cellulose, hemicellulose and pectin's. Therefore the removal of lignin is very difficult as its location is unknown. The upper layer of the fiber comprises of cellulose and hemicellulose, the fiber when treated with enzyme hemicellulase (E1), pectinase (E2) and cellulase (E3), reacts first with hemicellulase, weakens the chains and then reacts with the cellulose. Therefore the weight loss in sisal fiber both at 10% and 12% concentration is more due to hemicellulase and cellulase enzyme.

Table 3 shows the percent strength loss of the sisal fiber when treated with enzymes individually.

Table 3: Percent strength loss of sisal fiber

Sr. No	Percentage	Enzyme	Percent Strength loss		
			8 Hours	12 Hours	14 Hours
1	10	E1	5.76	7.05	9.8
2	10	E2	4.47	5.57	8.21
3	10	E3	6.01	7.78	11
4	12	E1	12.8	12.4	14.1
5	12	E2	8.9	9.9	12.6
6	12	E3	9.5	12	13.4

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

From Table 3 the results indicate that the 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 sisal fiber both at 12% concentration is more due to hemicellulase and cellulase enzyme.

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. From Table 4, it was concluded that 12% concentration for 12 hours of cellulase enzyme gave maximum whitening on the fiber.

Table 4: Whiteness index of Sisal Fiber

Sr. No	Name	Whiteness	Yellowness	Brightness
1	Untreated	68.992	42.891	38.743
2	E1 12% 8 HOURS	70.728	32.542	43.649
3	E2 12% 8 HOURS	68.018	37.291	39.404
4	E3 12% 8 HOURS	69.426	33.721	41.779
5	E1 12% 12 HOURS	66.893	33.671	38.71
6	E2 12% 12 HOURS	70.87	34.714	42.954
7	E3 12% 12 HOURS	74.556	27.302	49.725
8	E1 12% 14 HOURS	68.567	31.756	40.836
9	E2 12% 14 HOURS	71.115	33.27	43.746
10	E3 12% 14 HOURS	72.61	29.734	46.461

Enzyme Code: E1- Hemicellulase, E2- Pectinase, E3- Cellulase

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:Effect of optimum enzymatic treatment conditions on weight loss of the sisal fiber

Sr. No	Concentration of Enzyme (%owf)			Weight loss (% owf)
	E1	E2	E3	
1	2	0	10	8.23
2	5	0	7	8.52
3	5	2	5	9.63

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

Maximum weight loss in the fiber was when the enzyme combination was 5% hemicellulase, 2% pectinase and 5% cellulase. The maximum impurity in the sisal fiber is lignin. This makes the fiber more stiff and less cohesiveness in nature. When the enzyme concentration was in the ratio of 5:2:5 the high percentage of hemicellulase and cellulase enzymes would weaken the hemicellulose bond and would give surface itching to the fiber while the pectinase would enter inside and remove the pectin's. The minimum weight loss in the sisal fiber was observed when the enzyme combination was in the ratio of 2:0:10. For this specific combination, concentration of hemicellulase enzyme was less and thus the hemicellulose bonds was affected to certain extent and the high concentration of cellulase enzyme only gave surface itching to the fibers.

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.

Table 6: Strength loss of the combinations of the sisal fiber

Sr. No	Concentration of Enzyme (%owf)			Strength loss (%)
	E1	E2	E3	
1	2	0	10	11
2	5	0	7	11.33
3	5	2	5	10.12

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

Table 6 revealed that for the sisal fiber the strength loss of 2% hemicellulase, 0% pectinase and 10% cellulase combination and 5% hemicellulase, 0% pectinase and 7% cellulase enzyme combination was approximately similar. And the minimum strength loss of the sisal fiber was observed when treated with 5% hemicellulase, 2% pectinase and 5% cellulase.

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 7: Whiteness index of the combinations of enzyme on the sisal fiber

Sr. No	Name	Whiteness	Yellowness	Brightness
1	Untreated	69.803	39.035	40.516
2	12% 2:0:10 12 HOURS	70.934	27.608	44.915
3	12% 5:0:7 12 HOURS	69.787	31.27	42.636
4	12% 5:2:5 12 HOURS	71.419	27.753	45.471

Hemicellulase: Pectinase: Cellulase

Table 7 indicated that the yellowness of the sisal fiber decreased much when treated with 5% hemicellulase, 2% pectinase and 5% cellulase enzyme combination. And also the whiteness of the fiber increased when treated with the same combination.

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 5% hemicellulase, 2% pectinase and 5% cellulase.

SEM tests of the untreated and the best softened fibers were done at the magnification of 100X and 550X.

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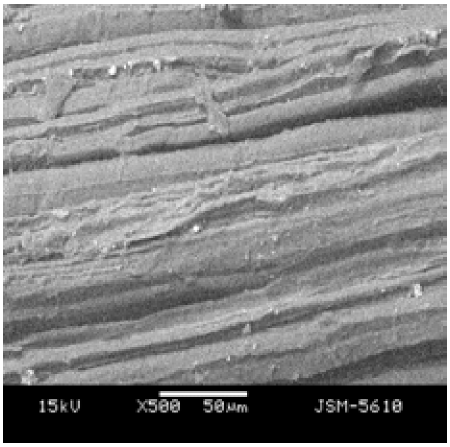

Plate 1: SEM test of untreated sisal fiber at 550X

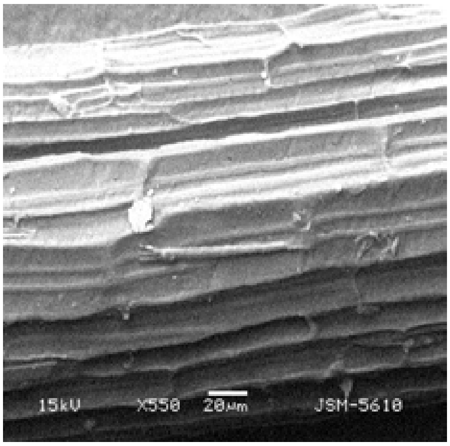

Plate 2: SEM test of treated sisal fiber at 550X

The primary function of fiber softening is to make the fibers surface smooth. Plate 1 and 2 showed that the untreated sisal fiber had rough and irregular surface. While from the Plate 3 and 4 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 5% hemicellulase, 2% pectinase and 5% 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% sisal with parallel laid and cross laid.

For preparing sisal nonwoven fabric, in the manual needle punch machine the fibers were cut into staple lengths of 3 inches. These fibers were laid first in the parallel direction and then passed through the needle board consisting of 1332 number of needles. The fibers was then again laid in the opposite side of the fabric in the horizontal direction and then passed through the needle board. In the similar way the fibers were laid in the cross direction and punching was done from the both side of the fabric. The machine parameters were kept constant. The nonwoven fabrics produced are shown in Plate 3,4,5,and 6 below.

Table 8: Constructional details of experimental fabrics

Blended composition	Fiber laying technique	Needle penetration	Punch density-Punches/cm^2	Stroke frequency Stroke/min	GSM
100% Sisal	Parallel laid Cross laid	9mm	150	240	566 608 527 634

Plate 3: 100% sisal nonwoven fabric of 566 GSM (Parallel laid)

Plate 4: 100% sisal nonwoven fabric of 608 GSM (Parallel laid)

Plate 5: 100% sisal nonwoven fabric of 527 GSM (Cross laid)

Plate 6: 100% sisal nonwoven fabric of 634 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 hemicellulase and cellulase enzyme removed more impurities from the sisal 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 5% hemicellulase, 2% pectinase and 5% cellulase.

100% sisal of parallel laid and cross laid with different GSM were prepared with different GSM. The fabrics were made manually in manual needle punch machine.

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