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Research Paper

A Comparative Study on The Effect of Chemical & Enzyme Treatments on the Softening of Sisal Fiber

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KEYWORDS : sisal, softening, enzymes, charkha, woven

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ABSTRACT

For thousands of years, natural fibers have been at the core of the textile industry. Minor fibers like flax, jute, banana, sisal, ramie and many other vegetable fibers have been used for more than 8,000 years. Sisal fiber is exceptionally durable with a low maintenance with minimal wear and tear, recyclable, anti-static, does not attract or trap dust particles and exhibits good sound and impact absorbing properties. Though it has strength but also have high stiffness, low cohesiveness which creates problems during end use. So the main purpose of the study was to soften sisal fiber with chemical and enzyme treatment and prepare woven samples from treated fibers. Four different enzymes namely Laccase, Cellulase, Hemicellulase and Pectinase were used to soften the fibers. Enzyme and chemical recipe was standardized for treating fibers. Treated fibers were tested for weight loss, strength, whiteness, SEM, fiber fineness and chemical composition and fibers were spun into yarns on Medleri Charkha. Also yarns were tested for yarn fineness, evenness and for strength. Woven fabric was prepared on table loom. Fabrics were woven in plain weave in which cotton yarns were used in warp direction and yarns prepared from treated fibers were used in weft direction.

Introduction

Over the last 50 years, natural fibers have started to become displaced by synthetic, man-made material such as polyester, acrylic and nylon which are much cheaper and easier to manufacture in bulk. These materials are strong contributor to carbon emissions and waste. (http://blogs.worldwatch.org/). Unlike synthetic fiber, natural fibers are renewable, carbon neutral, biodegradable and eco-friendly and not only come from the environment, but also benefit it. Minor fiber like flax, Jute, Banana, Sisal, Ramie and many other vegetable fibers have been used for more than 8,000 years. All these minor fibers are sourced from nature. Sisal is a leaf fiber which occupies 6th place among fiber plants and represent 2% of the world's production of plant fibers (http://www.fibre2fashion.com/industry-article/2/135/sisal-fibre-an-overview1.asp). Sisal with the botanical name ***Agave sisalana*** is a species of Agave native to southern Mexico but widely cultivated and naturalized in many other countries. In India, it's mainly grown in arid & semi-arid regions of Andhra Pradesh, Orissa, Karnataka, Maharashtra, Bihar, Chhattisgarh and West Bengal. The sisal plant produces approximately 200 - 250 leaves throughout its productive period. The life span of sisal plant is 7-10 years. The shape of sisal leaves is like sword and is about 1.5 to 2 meters tall. Sisal fiber is exceptionally durable with a low maintenance with minimal wear and tear. It is recyclable, anti-static, does not attract or trap dust particles and does not absorb moisture or water easily; the fine texture takes dyes easily and offers the largest range of dyed colors of all natural fibers. It exhibits good sound and impact absorbing properties. Its leaves can be treated with natural borax for fire resistance properties (http://textilelearner.blogspot.in/2013/01/sisal-fiber-properties-of-sisal-fiber.html). Also it not only has strength but also have high stiffness, low cohesiveness which creates problems during weaving & other fabric construction methods like non-woven. So the fiber needs to be softened first & then send for weaving and non-woven **(Shroff A.,et.al 2015)**. Softening of fiber might make it suitable for multiple end uses or application.

Fibers like Ramie, Sisal and Banana has outstanding features. Earlier research by Shroff A. (2014) on bio-softening of these minor fibers like Ramie, Sisal and Banana showed best results after treatment with enzymes like cellulase, hemicellulase and pectinase. Among all these fibers Sisal had problem while softening treatment due to its lignin content which is about 11%. Due to lignin Sisal has less spinability, less cohesiveness, less entanglement and fiber is stiff. For using Sisal fiber effectively it is necessary to soften it by removing lignin content. Therefore the main purpose of this study was to soften Sisal fiber by treating it with different chemicals & enzymes and comparing the physical properties of these treated fibers.

1. Methodology

1.1 Material:

Sisal fiber was used in the present study. It was supplied by Sisal Research Station (CRIJAF) Bamra, Sambalpur, Odisha. Total four different enzymes were used in the study. Hemicellulase, Pectinase, and Laccase enzymes and were procured from Rossari Biotech Ltd., Kanjurmarg (W.), Mumbai and Palkofeel C (Cellulase) was procured from Maps Enzymes Limited, Ahmedabad.

1.2 Standardizing the recipe for softening of Sisal Fibers

Enzyme and chemical treatments were standardized for the present study. Enzyme treatment was finalized after the pilot study that was carried out by the researcher (Table 1).

Table 1: Standardized Enzyme Recipe

	Time	Temperature	M:L	Concentration	Enzyme	pH
Step 1	45 mints	50-60^{0}C	1:30	10% owf	Laccase Enzyme	5-6pH
Step 2 Combination of enzymes	4hrs	50-55^{0}C	1:30	7% on total liq.	Cellulase	5-5.5pH
				5% owf	Hemicellulase	
				2% on total liq.	Pectinase	

For chemical treatment the recipe was of an ongoing research in department of Clothing and Textile on banana fibers (Table 2). Banana and sisal, being minor cellulosic leaf fibers, have almost similar chemical composition; hence, the recipe used for softening of banana was used for sisal as well.

Table 2: Standardized Chemical Recipe

	Time	Temperature	Concentration	M:L	Chemical	pH
Step-1 Alkali treatment	90mins	80-90^{0}C	4% owf	1:40	NaOH	10-12

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Step-2 Bleaching	30mins	80^{0}C	0.75%	1:40	H_2O_2:NaOCl 2:1 Alkali added to the bath NaOH(4%owf)	9-10
Step-3 Alkali Treatment	60mins	80-90^{0}C	4% owf	1:40	NaOH	10-12
Step-4 Neutralization	5mins	Room Temperature	1%	1:40	Acetic Acid	3-4

Also oiling treatment was finalized which was followed after treating fibers with enzyme and chemical treatment each shown in (Table 3).

Table 3: Standardized Oiling Recipe

	Time	Temperature	M:L	Concentration	Oil
Step 1 Boiling in Emulsion	30 min	70-80^{0}C	1:40	25% on total liquor	Rice Bran Oil
Step 2 Batching with Oil	24hrs	40^{0}C	N.A	N.A	Rice Bran Oil

Treatment of Sisal fiber

After standardizing the recipes, fibers were treated in bulk. 200 grams of fibers were treated with enzyme and chemical treatment each. In enzyme treatment, fibers were treated with Laccase enzyme at the concentration of 10% owf. Treatment was carried out for 45minutes at 50-60^{0}C using 1:40 as liquor ratio and maintaining the pH at 5-6. Fibers were then washed with distilled water and dried. In the next step fibers were treated with combination of enzymes for 4 hours and the concentrations were Cellulase 7% on total liquor, Hemicellulase 5% owf and Pectinase 2% on total liquor. Liquor ratio used for treatment was 1:30. Treatment was carried at 50-55^{0}C and pH was 5-5.5 pH. After the treatment fibers were washed and dried. After the enzyme treatment fibers were subjected to oiling treatment. Fibers were boiled at 70-80^{0}C in Water/Oil (Rice Bran Oil) emulsion solution of 25% on total liquor for 30 minutes using 1:40 liquor ratio. After emulsion treatment fibers were batched for 24hours.

For chemical treatment, in first step fibers were treated with 4% owf with NaOH for 1 and ½ hour at 80-90^{0}C and at 10-12 pH. Liquor ratio used for the treatment was 1:40. Alkali treatment was followed by bleaching with Hydrogen Peroxide (H_2O_2) and Sodium hypochlorite (NaOCl) in ratio of 2:1 and concentration was 0.75%. Bleaching was done at 80^{0}C for half an hour using 1:40 as liquor ratio. Alkali was added to the solution (NaOH 4% owf) and pH was maintained at 9-10. Bleaching was followed by third step which was again an alkali treatment similar to first step only difference was this treatment was carried out for 1hour. Last step was Neutralization. In this step fibers were dipped in 1% Acetic Acid solution to neutralize it after the alkali treatment. It was done at room temperature for 5 minutes and pH of the solution was 3-4. After the chemical treatment oiling treatment was done in similar manner used in enzyme treatment.

1.3 Analysis of Treatment

Effect of given treatments on the sisal fibers were evaluated by testing weight loss, fiber strength, fiber whiteness index, SEM test, fiber fineness and chemical composition.

1.4 Preparation and analysis of yarns

Treated fibers were spun into yarns on Medleri Charkha in Navsari Agriculture University, Navasari, Gujarat. Prepared yarns were evaluated for yarn evenness, strength and yarn fineness.

1.5 Application of treated fibers

Woven samples were prepared from the yarns on Medleri Charkha by researcher. Weaving was done by researcher on the table loom in the department of Clothing and Textile, Faculty of Family and Community Science, The Maharaja Sayajirao University of Baroda.

2. Results and Discussion

In order to study the effect of given enzyme and chemical treatments on sisal fiber, raw sisal fibers, enzyme treated sisal fibers and chemically treated sisal fibers were subjected to various test.

2.1 Evaluation of the effect of the enzyme and chemical treatment on sisal fibers

2.1.1 Determination of Weight loss

The effect of given treatments was primarily measured by weight loss test. It was observed that weight loss was higher in enzyme treatment 9.91% as compared to chemical treatment which was 8.04%.

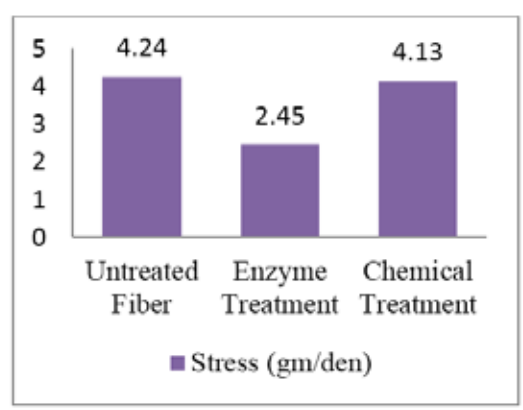
2.1.2 Determination of tensile strength

The strength of the fiber was tested on Instron machine in Textile Engineering Department of The Maharaja Sayajirao University of Baroda, Vadodara. Sample size was 10 cm and test speed was 20mm/min both which were kept constant for all the samples.

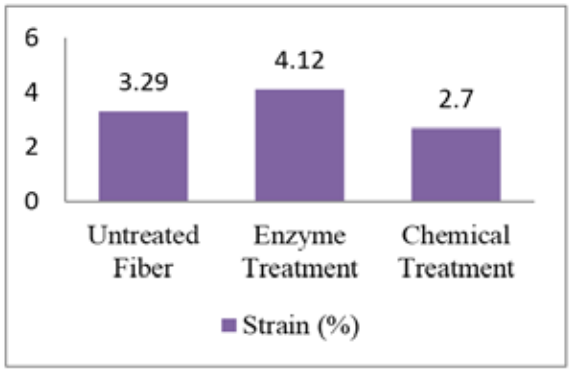
In Table 4 Tensile strength of sisal fiber; showed that untreated sisal fiber had higher strength than enzyme treated fibers. Untreated fibers had strength of 4.24gm/den whereas chemical and enzyme treated had 4.13gm/den and 2.45gm/den respectively. It was observed that after the chemical treatment strength of the fiber was maintained as compared to enzyme treated fiber. Elongation of enzyme treated fibers was increased as compared to chemically treated fibers. Chemically treated fibers became more fibrils and less flexible.

Table 4: Tensile Strength of Sisal fiber

Sr. No.	Treatment	Denier	Max. load (gf)	Extension at max. (mm)	Stress in gm/den	Strain %
1	Untreated Fiber	235.00	1013.66	3.29	4.24	3.29
2	Enzyme Treatment	273.60	672.65	4.12	2.45	4.12
3	Chemical Treatment	201.60	883.29	2.70	4.13	2.70

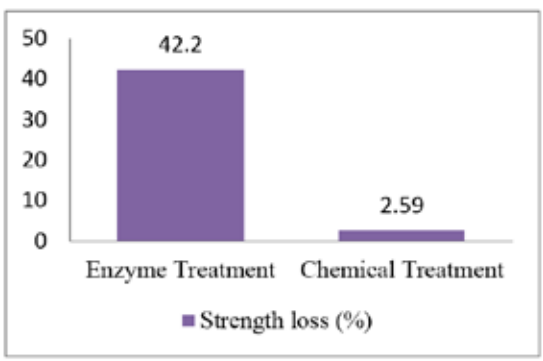


Graph 1: Tensile Strength of Sisal Fiber



Graph 2: Percent Strain of Sisal Fiber

Graph 1 and 2 shows stress and elongation of untreated and treated fibers.

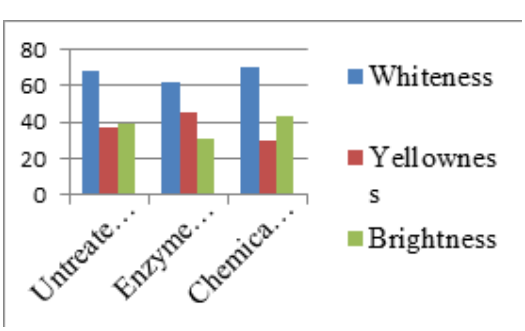


Graph 3: Strength loss of Treated Sisal fiber

Graph 3 showed that a chemically treated fiber has less strength loss as compared to enzyme treatment. This was mainly because of alkali used during the chemical treatment which caused the mercerization without tension due to which fibers got stronger. Also hypochlorite used for bleaching behaves similar to acid pretreatment which increases the degree of crystallinity. (http://ipst.gatech.edu/faculty/ragauskas_art/posters/poster_biotechsymp_600dpi.pdf)

2.1.3 Determination of Whiteness index

Enzymes help the fiber to remove pectin, fats and waxes. This helps in reducing the yellowness of the fibers and thereby increases the whiteness of the fibers **(Shroff A.,et.al 2015).** Whiteness of the untreated fibers and treated fibers were measured on the Spectrascan 5100A Spectrophotometer to see the effect of given treatments on the fibers. It was observed that after chemical treatment whiteness and brightness of fiber was increased and yellowness was decreased. Whereas in case of enzyme treatment whiteness was reduced and there was increase in yellowness of the fiber.



2.1.4 SEM test

SEM test of the untreated and treated fibers were done at the magnification of 250X and 500X. Plate 1 shows the SEM results of untreated Sisal fiber at 500X. Plate 2 shows the SEM results of enzyme treated Sisal fiber at 500X. Plate 3shows the SEM results of chemical treated Sisal fiber at 500X. The primary function of the fiber softening is to make the fibers surface smooth. It was seen in (Plate 1) that untreated sisal fiber was irregular and rough and also much of the pithy material was observed. (Plate 2) shows the enzyme treated sisal fibers in which the fibers were observed to be very smooth and clean. Striations parallel to fiber axis were clearly seen with the cross marking. Whereas in (Plate 3) shows chemical treated sisal fibers which is also resembled clean and the striations were noticeable.

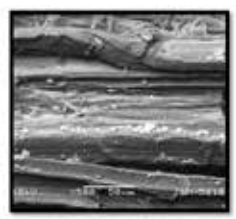


Plate 1: SEM test of untreated sisal fiber at 500X

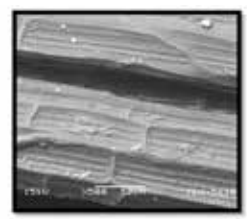


Plate 2: SEM test of Enzyme treated Sisal fiber at 500X

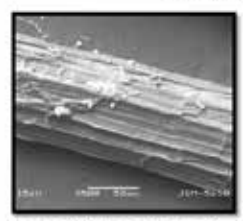
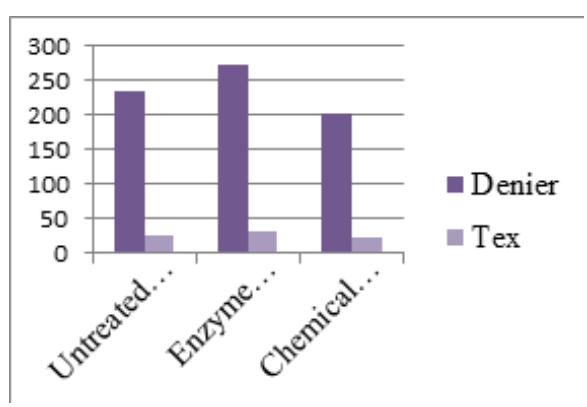


Plate 3: SEM test of Chemical treated sisal fiber at 500X

1.1.5 Determination of Fiber Fineness

It was observed that there were slight variations in denier of fibers. Untreated fiber had denier of 235.8 whereas enzyme treated had 273.6 and chemical 201.6. Graph 5 shows denier and tex of all three fibers. From the graph it can be concluded that denier had been increased after the enzyme treatment. That means after the enzyme treatment on sisal fibers the fibers were swollen.



Graph 5: Fineness of Sisal fiber

1.1.6 Determination of Chemical Composition

The chemical composition of these three fibers i.e. untreated sisal fibers, enzyme treated and chemical treated sisal fibers were determined by following the scheme recommended by Turner and Doree. The chemical composition was determined to find out the effect of given enzyme and chemical treatment on the chemical structure of the sisal fibers. The (Table 5 Chemical Composition of the Sisal fibers) shows the chemical composition

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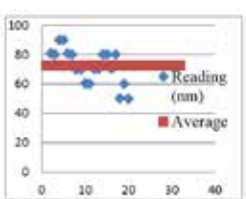
of untreated and treated sisal fibers. It was observed that after chemical treatment fibers became softer mainly because lignin was degraded (3.35%) as compared to enzyme treated fibers. There was not much difference in Cellulose and Hemicellulose percent in both treatments. Main observation was removal of lignin was higher after chemical treatment.

Table 5: Chemical Composition of the Sisal fibers

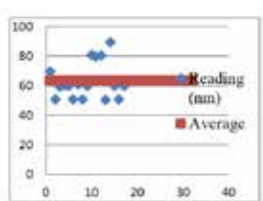
Sr. No.	Content	Untreated Fiber (%)	Enzyme Treatment (%)	Chemical Treatment (%)
1	Cellulose	80.35	77.9	78.9
2	Hemicellulose	9.8	5.2	5.0
3	Lignin	6.15	5.45	3.35
4	Pectin	1.7	1.3	1.1
5	Fats and Waxes	1.5	0.40	0.85
6	Water soluble components	8	7.9	8.5
7	Ash Content	0.9	0.75	0.9

1.1 Evaluation of the properties of the prepared sisal yarns
After the preparation of the yarn from treated fibers, yarns were evaluated for their strength, fineness and evenness.

3.2.1 Determination of Yarn Evenness
Evenness of the yarn was determined by taking 20 readings and plotting a graph from these readings. The evenness of the yarn was determined by using micrometer microscope. Yarns were viewed under the microscope using micrometer at 45X power.

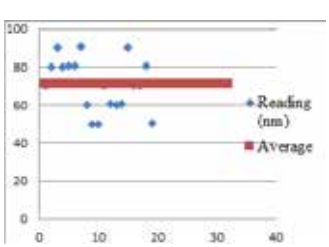


Graph 6: Evenness of yarns prepared from untreated sisal fibers



Graph 7: Evenness of yarns prepared from Enzyme treated fibers

(Graph 6) represents the yarn evenness prepared from untreated sisal fibers. It was observed that yarns prepared from untreated sisal fibers were more even than prepared from enzyme treated and chemically treated sisal fibers. From the (Graph 7 and 8) represents the evenness of yarns prepared from enzyme treated sisal fibers and chemically treated sisal fiber. It was observed that both these yarns were very uneven and showed a lot of variation. It was mainly because of the unevenness of fibers and also because of the technique used for spinning of the yarns. It is dependent on the feeding of the fibers during spinning.



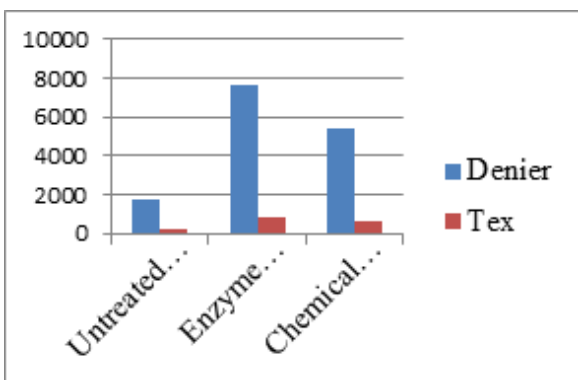
Graph 8: Evenness of yarns prepared from Chemical treated fibers

3.2.2 Determination of Yarn Fineness
There was a lot of variation in the denier cause of uneven yarn. Table 6 shows the readings of fineness of these yarns in denier and Tex.

Table 6: Fineness of Yarns prepared from treated sisal fibers

Sr. No.	Treatments	Denier (D)	Tex
1	Yarns prepared from untreated sisal fibers	1787.9	198.65
2	Yarns prepared from Enzyme treated sisal fibers	7635	848.33
3	Yarns prepared from Chemical treated sisal fibers	5409	601

From Table 6 it was concluded that yarns prepared from untreated sisal fibers are finer as they had lesser denier of 1787.9. Whereas yarns prepared from enzyme treatment was coarser because of their higher denier of 7635. Yarn made from chemically treated fibers was finer compared to enzyme yarns because they had lesser denier i.e. 5409 than enzyme yarn.



Graph 9: Fineness of Yarns prepared from Sisal Fiber

1.2 Preparation of Woven Samples
After the yarns were prepared from enzyme treated sisal fibers and chemically treated sisal fibers, woven samples were prepared by the researcher. These woven samples were prepared on the table loom in the Department of Clothing and Textiles of Faculty of Family and Community Sciences, the Maharaja Sayajirao University of Baroda, Vadodara. These samples were woven with plain weave with cotton yarns in warp direction and yarns prepared from treated sisal fibers in weft direction.

Plate 4 and 5 shows the woven samples prepared from yarns of enzyme treatment and chemical treatment respectively. It shows that fabric prepared from yarns of chemically treated fibers was smoother as compared to enzyme sample. Enzyme sample was very rough and have more of protruding fibers on the surface of the fabric.


Plate 4: Woven Sample prepared from yarns of Enzyme treated fibers


Plate 5 : Woven Sample prepared from yarns of Chemical treated fibers

3. Conclusion

In conclusion it can be seen that

- After enzyme treatment strength was reduced but there was increase in elongation of the fiber. SEM shows smooth fiber than untreated fibers and more clean because the pithy material was removed. Also fibers were swollen. Enzyme treated fibers were softer than raw and untreated sisal fiber. Enzyme treated fiber showed decrease in whiteness and brightness index and yellowness index was increased after treatment.
- After chemical treatment strength of the fiber was maintained. But there was decrease in elongation and fibers were less flexible and more fibril. SEM showed no swelling of fibers and showed small abbreviations. These fibers were less smooth than enzyme treated fibers. Whiteness index is increased due bleaching and brightness was also increased. Also yellowness was reduced.
- Chemical treatment gave better results than enzyme treatment.
- Yarns prepared from treated fibers were very uneven. So more work needs to be done to spinning of fibers to obtain even yarn. Also care has to be taken during spinning of fibers specially while feeding the fiber to produce more even yarns.
- Fabrics prepared from chemically treated fiber were smothering than samples prepared from enzyme treated fibers.
- Fabric made from enzyme treated fibers was rough and have uneven texture. There were many protruding fibers on the surface of the fabric giving it rough surface.
- It can be concluded that fabrics can be used for upholstery, products for handicrafts and ropes, twins etc. Further treatments and finishing can be done for using it into apparels.

Acknowledgment

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