

# Sisal Fiber: A Potential Raw Material for Handmade Paper

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## ABSTRACT

Sisal fibers have been subjected to proximate analysis and its potential to be used in paper making has been studied. Fibers collected from two different types of land patterns are found to have insignificant differences in physical and chemical characteristics. Analysis of chemical treatments on the sisal fiber reveals that better separation of fibril can be achieved by acid treatment, when compared to treatment with alkali, sodium dodecyl sulfate and acetone. Mechanical beating resulted in better defibrillation. Pulping of sisal fibers by mechanical ( $\geq 90\%$ ) and semi chemimechanical ( $\geq 75\%$ ) methods showed good pulp yields. Results of the various physical tests conducted on papers prepared from sisal pulp were encouraging. Paper made from sisal fibers exhibited high tear strength and high opacity. Chemical treatments of the fibers while pulping resulted in improved properties of the ensuing paper sheets. Comparison of properties from sisal pulps and rag pulps (conventional raw material for paper manufacturing in the handmade sector) further substantiates the suitability and possibility of using sisal fibers for papermaking.

**Key words:** Sisal fiber, pulping, proximate analysis, handmade paper, rag pulp.

## Introduction:

Sisal is a strong leaf-fiber obtained from the leaves of the plant *Agave sisalana*. The plant is a monocotyledonous perennial shrub that grows in the tropical and sub-tropical regions of the world. It is one of the most extensively cultivated hard fibre in the world due to the ease of cultivation of sisal plants, and is quite easy to grow in all kinds of environments<sup>1</sup>. The major producer of sisal fibres are Mexico (120 ktons), Brazil (125 ktons), Tanzania (26 ktons), Kenya (22 ktons), Madagascar (10 ktons), China (25 ktons) per annum. In India, it is mainly grown or cultivated in the arid and semi-arid regions of Andhra Pradesh, Bihar, Orissa, Karnataka, Maharashtra and West Bengal. In Orissa, it is abundantly available and randomly distributed in its central and western belts but highly concentrated in the Bamra region of the State. The sisal plant looks like an overgrown pineapple plant with a pineapple-like bole (a short, stocky trunk) from which the leaves extend. For a matured plant, the bole is about 50cm in height and about 20cm in diameter. The leaves can attain a length of up to 2m, but are usually about 1.0 – 1.2m long. The leaves which may be as broad as 12cm are tipped with sharp, highly lignified spines of about 1.0-1.5cm long. The outside of the sisal leaf consists of a well-developed epidermis with a waxy surface. This epidermis contains cutin, waxes and carbohydrates. Initially, all leaves grow vertically on the plant but with age, they fan out gradually to an angle of about 45<sup>o</sup>. The matured leaves are those closest to the ground containing the coarsest and the longest fibers.

Traditionally, sisal had been cultivated for “line fiber” which is used in the manufacturing of natural ropes, twine, sacking and carpet making. Other uses of sisal had been in the making of coarse textile materials like nets and mats<sup>2</sup>. Sisal has also been used in making handbags, wall coverings and also very durable automobile floor mats. Because of their high cost, use

of sisal pulps is confined to certain small specialty products, such as tea bags, certain filters, and sausage skins with stringent requirements for high strength and fine texture<sup>3</sup>.

Though development of synthetic fibers has eroded the traditional sisal markets, new avenues have opened up in this technological era for its diverse applications. The use of sisal as reinforcement fiber in cement or concrete<sup>4-6</sup>, composite resins<sup>7-8</sup>, polymer matrix<sup>9-15</sup>, bionanocomposites<sup>16-18</sup>, bitumen and plasterboard has also been explored and exploited in the recent past. Several researchers have also reported the use of sisal fibers in thermoplastic<sup>19-21</sup> and synthetic as well as natural rubber composites<sup>22-25</sup>. Most of them have observed an improvement in the bonding at the fiber-polymer interface which is suggested to have taken place due to chemical treatment of the fiber. Specifically, chemicals treatment using sodium hydroxide, isocyanate, permanganate and peroxide have been found to improve the bonding at the fiber-polymer matrix interface, which results in a considerable enhancement in the tensile and other physical properties of the composites to varying degrees. In the present investigation, an attempt has been made to evaluate the chemical characteristics of the sisal fibers abundantly available in the western belt of Orissa and examine its feasibility as a paper-making raw material especially, in the handmade sector.

## Materials and Methods

Due to the geographical variation of the natural fibers, sisal fibers have been collected from two different locations approximately 100kms apart, one with irrigation facilities and the other is a rain-fed area. Decorticated fibers<sup>26</sup> were procured from Bamra, Sundargarh district (rain-fed area) designated as Sisal-1 and the other type designated as Sisal-2 were procured from Nildungri (irrigated area), in Sambalpur district of Orissa.

## Proximate analysis of sisal fibers

Some of the characteristics of the fibres viz., chemical constituents (cellulose, pentosan, lignin, pectin, ash & silica) and solubility in various solvents viz. water, ethanol, benzene, acetone and different concentrations of alkali, have been investigated and the results are tabulated (Table 1) for comparison.

**Table 1 Proximate analysis of sisal fibers**

Sl. No.	PROPERTIES		SISAL TYPE	
			SISAL-1	SISAL-2
1.	Moisture (%)		10.11	9.89
2.	Length (cm)		80-115	80-110
3.	Width (mm)		0.3	0.25
4.	Density	(Pycnometer method)	1.453	1.360
		(width method)	1.645	1.544
5.	Tensile strength, B.L. (km)		6.1	5.86
6.	Solubility in alkali (%)			
	1% NaOH		22.7	22.76
	3% NaOH		23.8	23.92
	5% NaOH		30.0	29.72
	7% NaOH		32.55	34.34
	Solubility in organic solvents (%)			
	ethanol	cold	0.65	0.66
		hot	0.8	0.78
	benzene	cold	1.15	1.22
		hot	3.79	3.82
	ethanol-benzene (1:1)	cold	0.85	0.85
		hot	1.75	1.85
	in water	cold	0.25	0.33
		hot	0.54	0.59
7.	Acetone Extractives (%)		4.79	5.23
8.	Holocellulose (%)		87.05	86.34
9.	-Cellulose (%)		61.76	67.19
10.	Lignin % (Acid insoluble)		11.7	10.22
11.	Ash (%)		1.128	1.270
12.	Silica (%)		0.33	0.47
13.	Pentosan (%)		22.34	19.49
14.	Pectin (%)		1.2	0.9

The sisal fibers were thoroughly cleaned by removing the residual pithy materials attached to the fibers after decortication (extraction of fibers from the leaves), washed with water and air-dried. The fibers were then cut to uniform length of approximately 2-4 cm with the help of a pair of scissors for using them in various laboratory-scale investigations. Results of a few preliminary investigations carried-out upon the sisal fibers procured from two different locations as mentioned above, have been summarized in table-1. Moisture percentage of the fibers was calculated by weighing the oven-dried (5-6 hr at 100<sup>o</sup> C) fibers three to four times and taking the average of all the readings. The density of sisal fiber was determined by obtaining the fiber width<sup>27</sup> with the help of a compound microscope at 40x resolution and liquid pycnometry technique<sup>28</sup>.

Chemical analysis has been performed according to TAPPI standard methods<sup>29</sup> as follows: T 223 CM-84-pentosan, T 222 OM-88-lignin (acid insoluble), and T 207 OM-88 for hot and

cold water solubility, T 212 OM-88 for 1% alkali solubility. Solubility was also determined with varying concentrations of the alkali viz. 3%, 5% and 7%. The holocellulose content was evaluated with the method adopted by Wise et al<sup>30</sup>. The  $\alpha$ -cellulose was determined according to the modified method of Sarkar et al.<sup>31</sup> and the pectin content was estimated with 0.5% ammonium oxalate solution<sup>32</sup>. The ash and silica contents were determined gravimetrically. For determining the ash content, fibers were taken in a porcelain basin and ignited for 6-7 hrs on a Bunsen burner till the contents of the basin turned, initially from black, to grayish amorphous powder (ash). Silica (SiO<sub>2</sub>) content in the fibers was analyzed by the conc. HCl method. The results of the various physical and chemical analysis have been summarized in Table 1. All the experiments were repeated more than three times and the values were averaged within an error of 6%.

Photographs of the unmodified sisal fiber and the fibers with various treatments and modifications have been taken with the help of a compound microscope at 40x magnification.

## Pulping and paper making

The sisal fibers collected from Nildungri, near Sambalpur (Sisal-2) were selected for the pulping due to the proximity of this area to the research field. The fibers cut into uniform size of approximately 2-4 cm in length with the help of a hand operated straw cutter were thoroughly cleaned to remove the residual pithy materials attached to the fibers after decortication (separation/ extraction of fibers from the leaves). The pithy materials which mainly consist of the parenchyma cells obstruct the beating process and may also affect the properties of the paper sheets prepared from these fibers. Hand sheets were prepared by both mechanical as well as semi chemi-

mechanical processes of varying basis-weight and were evaluated for several physical properties especially, the strength properties, by testing their burst, tensile or tear strengths. Their Cobb values were also determined.

### Sheets Prepared by Mechanical Pulping

The cleaned fibers were subjected to the pulping and paper-making process. They were kept in water for nearly 1 hr prior to beating for softening the fibers to facilitate fiber swelling and hydration, which is one of the fundamental requirements and purpose of the beating process during pulping. The fibers were then beaten in an experimental beater (Hollander type of 25l capacity) for varying time intervals at 2-3% pulp consistency (initially in a high and then at medium load). Freeness (which measures the extent of hydration in fiber by measuring the adsorbed water) of the pulps were examined with the help of a Schopper Reigler Freeness Tester at different stages of beating and the strength properties of the resulting sheets examined. Paper sheets were prepared by the handmade

**Table 2 Properties of Sheets at different stages of beating**

Beating time (min)	60	75	90	105	120
Freeness, <sup>0</sup> SR	10-11	12-14	15-17	18-20	24-25
Unscreened pulp yield, %	? 90	? 87	? 85	? 82	? 79
Burst Factor, gfc <sup>2</sup> m <sup>2</sup> /g	8.12	9.75	10.37	12.56	12.89
Breaking Length, mtr	1187	1400	1691	1854	1753
Tear Factor, gfm <sup>2</sup> /g	122	147	124	114	136
Double Fold (M.I.T)	0	3	4	4	6

technique of lifting the uniformly distributed pulp from a vat containing pulp at a lower consistency, on a self fabricated mold of 30 mesh size followed by couching, pressing, drying in air and calendaring, with the help of a small power operated calendaring machine. The properly conditioned paper sheets were then subjected to various types of tests and the data are tabulated in table 2. All the tested samples were of same basis weight with variation of ±10 GSM.

**Hand-sheets prepared by semi chemimechanical process**

ratio was maintained at 1:5 (w/v) in all cases. The cooked fibers were washed free of liquor, and subjected to the beating process. It was found that the cooked fibers consumed less beating time than the uncooked fibers to reach the same degree of freeness. Burst Factor and 1 min-Cobb size values were also determined and are tabulated (Table-3), along with their basis weight (GSM) values for sheets, prepared by the mechanical process without any use of chemicals. Samples were also tested for these properties, prepared by the semi chemimechanical pulping process.

**Table-3 Properties of un-sized and sized paper sheets cooked at varying time intervals**

Cooking time (hr)	Unscreened Pulp yield (%)	Use of sizing chemicals/ fillers (rosin/starch)	Properties		
			GSM	B.F. (gfc <sup>2</sup> m <sup>2</sup> /g)	1min Cobb value (g/m <sup>2</sup> )
1.00	87	Rosin/starch	85	19.81	35.8
1.00	87	Rosin/starch	141	20.72	41.2
1.30	85	Rosin/starch	88	22.03	37.8
1.30	85	Rosin/starch	152	22.51	40.5
2.00	83	Rosin/starch	79	24.63	26.6
2.00	83	Rosin/starch	150	25.72	30.5
2.30	80	Rosin/starch	82	23.61	35.5
2.30	80	Rosin/starch	149	22.79	28.7
3.00	75	Rosin/starch	85	23.55	27.54
3.00	75	Rosin/starch	144	24.72	31.87
--	?90	--	60	2.45	52.07
--	?90	--	70	2.86	52.34
--	?90	--	102	8.43	65.75
--	?90	--	150	10.475	56.76
--	?90	--	200	10.24	123.21
--	?90	--	300	10.11	131.65

**Table-4 Comparison of Physical Properties of sheets prepared from sisal and rag pulps**

Tests	Composition					
	Sisal (200g)				Cotton rags (200g)	
Basis weight (GSM) ±5	80	120	200	80	120	200
Porosity (ml/min)	2000	3840	4081	1850	2012	2930
Stiffness (Taber)	9	8	8	5	6	5
Breaking Length, mtr	1400	1540	1488	1187	1267	1345
Tear Factor, 100gfm <sup>2</sup> /g	147	135	139	111	105	114
Double Fold, M.I.T	0	4	4	0	3	3
Opacity, %	100.00	100.00	100.00	98.78	99.45	98.76
Burst Factor, gfc <sup>2</sup> m <sup>2</sup> /g	3.0	10.178	10.14	1.93	9.00	9.98

In the semi chemimechanical pulping method, cleaned and uniformly cut (4-5cm) sisal fibers were steam digested in a 5-l capacity pressure cooker for different intervals of time from 1-3 hr in 10% sodium hydroxide concentration on weight of oven dry fibers and 10 ml hydrogen peroxide.

The material to liquor ratio was maintained at 1:5 (w/v) in all cases. The cooked fibers were washed free of liquor, and subjected to the beating process. It was found that the cooked fibers consumed less beating time than the uncooked fibers to reach the same degree of freeness. Burst Factor and 1 min-Cobb size values were also determined and are tabulated (Table-3), along with their basis weight (GSM) values for sheets, prepared by the mechanical process without any use of chemicals. Samples were also tested for these properties, prepared by the semi chemimechanical pulping process.

Various properties of the hand sheets prepared from sisal fibers as well as cotton rags (textile wastes), the conventionally used raw material in the hand made paper industries, have been tested and tabulated (Table-4) for the sake of comparison. Both the types of fibers were pulped by the mechanical pulping process to the same degree of Freeness at same pulp consistency. The paper sheets prepared from the unsized pulps were examined for various physical properties. It was observed during the beating of both types of fiber (experimental sisal and the conventional cotton wastes), that the sisal fibers consumed more beating time than the rag fibers, probably due to high stiffness and lignin content of the sisal fibers in comparison to the rag fibers.

## Results and Discussion

Physical characteristics and chemical composition of the two types of sisal fibers (sisal-1 and sisal-2) are quite comparable and exhibit very little variations as visible from the data presented in Table-1. The density of the fibers obtained from the calculation by using the width of the fibers is found to be high when compared to pycnometer technique and are in the range of 1.365–1.630. In the liquid pycnometry technique, measuring the volume of fibers by liquid displacement method was done by taking both water and hexane separately to avoid any swelling of the fibers in water through hydrogen bonding. However, the difference in the volumes of a fixed weight of material is within 6%.

Photographs of the unmodified sisal fiber and the fibers with various treatments and modifications (Figures 1–10) have been taken to study the morphological characteristics of the fiber and also to analyze the effects of various chemicals, physical modifications and dyes on the fiber surface. Some valuable conclusions have been drawn from the photo-analysis, which not only reveals the structural and morphological features of the fiber but also renders support towards the suitability of these fibers as a paper-making raw-material.

Sisal fibers consist of micro-fibrils arranged parallel to each other along the fiber axis to form a bundle with gummy materials (or other substances) in the inter-fibrillar region as visible from Fig.-1. When refluxed with alkali, the gummy and easily extractable materials are removed from fibers and so the fibrils seem to be more separated (Figure 2). With acid, however, the removals of these materials are more prominent with scattered fibrils (Figure 3). To remove the organic soluble materials, the sisal fibers were refluxed in dioxane in a soxhlet for 8 hours. No significant change in the microscopic picture

(Figure 4) is noted indicating the presence of less amount of organic cementing materials for inter fibrillae binding.

Treatment of the fiber with sodium dodecyl sulphate (SDS), which is a surfactant, does not seem to delignify the fiber significantly; instead it appears to have formed a protective covering on the fiber surface as may be seen from Figure 5. The formation of a protective layer on the fiber surface may also be gauged from the fact that even after beating the SDS treated fiber with a mortar and pestle for about 4-5 hr, no change could be detected on the fiber morphology as evident by comparing figures 5 and 9. The interaction of SDS with sisal fiber surface is mostly due to the adsorption of the surfactant on the hydrophilic surface of the sisal fiber. We could remove the sticky materials present with the fibrils by scratching the fiber with a sharp blade (Figure 6), providing support to the mechanical (beating) process of defibrillating and delignifying the fibers, while pulping in papermaking. But the modifications appear to be taking place only at the peripheral region of the fiber surface rather than the core portion of the fiber.

The sisal fiber seems to possess good adsorption capacity towards dyes/colours as clearly visible from the photograph-10. The fiber had been dissolved in an orange colour dye solution for an hour and then was washed with water for several times before taking the photograph. The dye appears to have adhered to the fiber surface firmly.

The proximate chemical analyses were performed by using standard techniques and the results are tabulated in Table 1. High cellulose (61-68%) and low lignin (10-12%) contents in comparison to the conventional pulping fibers of hardwoods, which contain 38-49% cellulose and 23-30% lignin and the unconventional agro-residues of cereal straws, which contain 28-36% cellulose and 12-20% lignin<sup>33</sup> show a positive

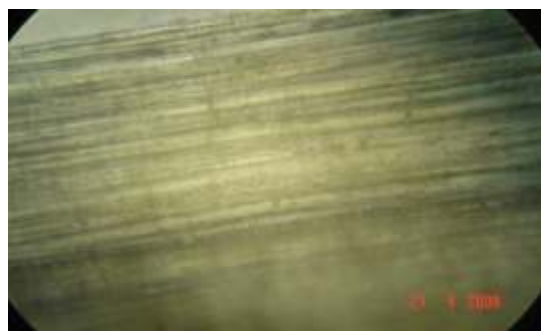


Fig. 1. Sisal Fibers

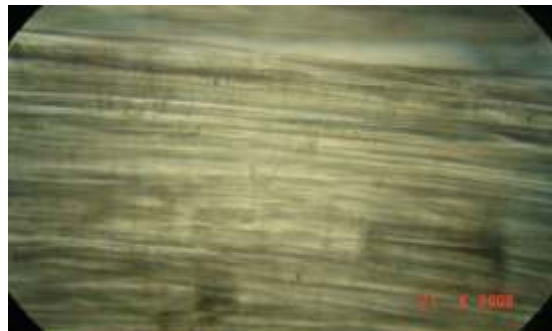


Fig. 2. Fibers treated with 7% NaOH

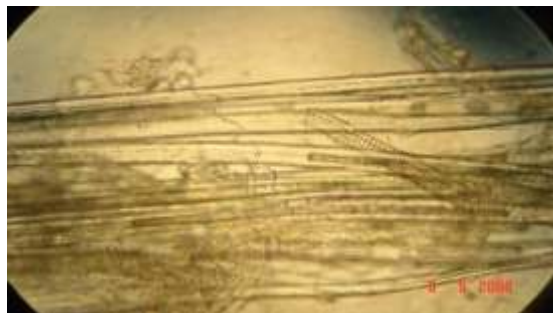


Fig. 3. Dilute hydrochloric acid treated fiber

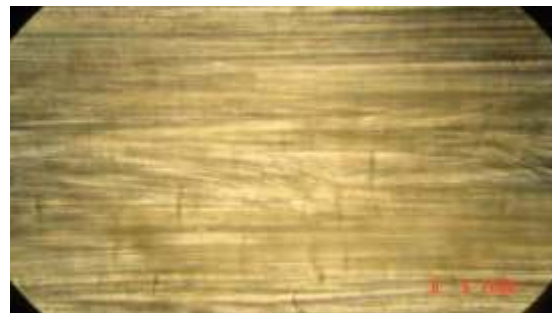


Fig. 4. Dioxane treated fiber

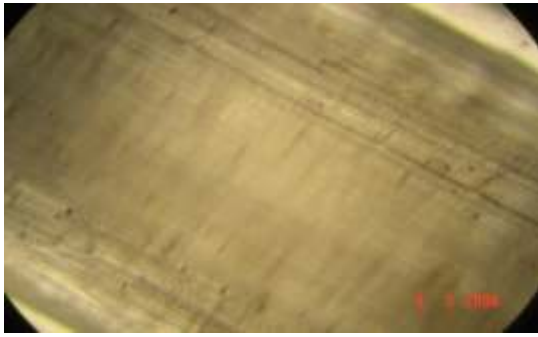


Fig. 5. Surfactant (SDS) treated fiber

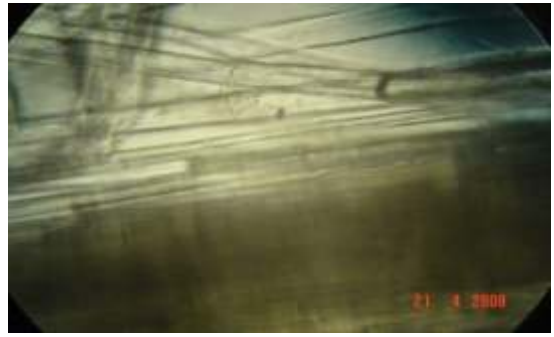


Fig. 6. Fiber scratched with a blade

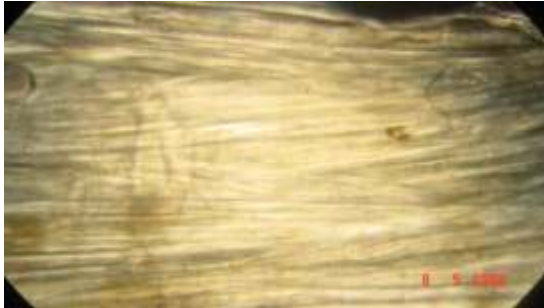


Fig. 7. Fibers beaten in a mortar and pestle (2-3hr)



Fig. 8. Fibers beaten in a beater (1.30hr)



Fig. 9. SDS treated fibers beaten (4-5hr) in a mortar

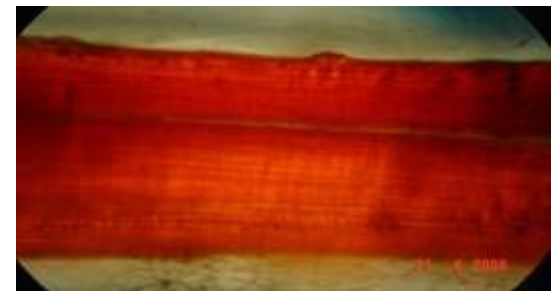


Fig. 10. Alkali (7%) treated sisal fiber soaked in a dye

indication for employing the locally available sisal fibers as a paper-making raw material. Pentosan contents (19-23%) were also comparable or slightly less than the wood (19-26%) and non-wood fibers (23-32% for the agro-residues)<sup>33</sup>. Low silica percentage in the sisal fibers is further a green signal for pulping, as it would cause less damage to the beating equipment. Lower lignin percentage is suggestive of an inexpensive pulping process (low consumption of cook chemicals and/or less beating time), which is an important factor for the economical viability of any ligno-cellulosic material particularly in the small scale industries. To investigate on the lipophilic contents in the sisal fiber, acetone was used as an extractant. After ten hours of reflux in a soxhlet, 4-5% of lipophilic materials could be extracted, this may be present mostly at the surface of the fibres. On analyzing the solubility of sisal fibers at various concentrations of alkali, it may be concluded that more surface chemicals could be extracted with higher concentration of alkali.

Hand made paper has its demand due to its eco-friendly characteristics and socio-economical significance through employment generation in rural or sub-urban areas in developing countries. The paper prepared by the hand made technique chiefly involves the mechanical pulping process and occasionally includes the semi-chemimechanical processes basically, to preserve its eco-friendly nature and make the end products cost effective. In the wake of this, compromise in some properties (especially, the strength and optical properties) of the ensuing paper sheets, as compared to the mill-made papers is a natural consequence. In this light, we have endeavored to prepare paper sheets without adding any additives (fillers or sizing chemicals), and compared the physical characteristics of the paper sheets prepared with additives like starch / rosin and chemically digested fibers.

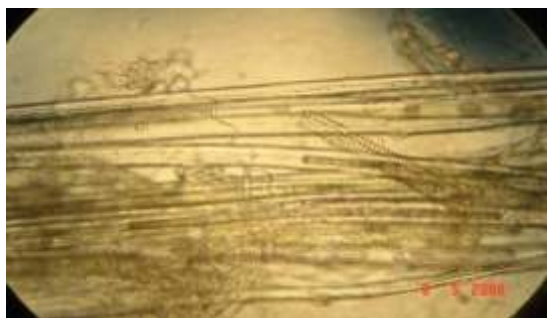
It is observed that with the increase in beating time, freeness values of the pulp increased. This is due to an increase in defibrillation of the microfibrillar bundles (of which the sisal fiber is structurally composed of) and thereby more adsorption of water on the fiber surface, resulting in higher swelling of the fibers. The improvement in the strength properties of the paper sheets prepared at different stages of beating, as evident from values in Table-2, may be attributed to the better swelling of fibers leading to an enhancement in inter-fibrillar hydrogen-bonding.

A considerable improvement in the burst strength values of hand-sheets can be observed when the sheets were treated with mild chemicals viz. rosin (used for sizing) and starch (used as a filler). Further enhancement in the strength property could be detected for the chemically digested (alkaline-peroxide) sisal fibers prior to beating. Moreover, the chemically cooked fibers were found to have reduced the beating time and reduced the pulp yields significantly. Sizing chemicals and fillers tend to fill the voids or vacuum spaces present within the microfibrillar network, which gives rise to the paper web-structure. These substances (rosin/starch) increase the number of hydrogen-bonds within the paper thereby, enhancing its strength properties. Chemical digestion further accelerates the delignification that results in reduction of the beating time. High Cobb values for un-sized papers (Table-3) is indicative of large number of voids and empty spaces within the sheets, weakening its strength.

## Conclusions

On comparing the data obtained by several physical examinations of the sheets, prepared from the experimental sisal fibers as well as the conventionally used rag fibers in the handmade sector, it can be concluded that sisal fiber is quite compatible with the rag fiber for paper manufacturing. In fact, slightly higher values of strength properties (like burst, tear, tensile in terms of breaking length), observed in case of sisal paper as compared to the rag paper is suggestive of utilizing the sisal fibers also as a strength enhancing additive for weak and recycled pulps.

### Sisal fiber: a potential raw material for handmade paper



Sisal fibers are found to have potentiality in paper making through blending with other conventional raw materials.

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