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Effect of Alkalization on the Quality of Helicteres Isora Fibre

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ABSTRACT

Helicteres isora **is a plant that grows naturally as a shrub and ligno-cellulosic bast fiber is extracted from the stem of the plant by water retting. Alkali treatment is one of the chemical treatments for natural fibres to modify and clean the fibre surface of the impurities like waxes, lignin and pectin's which bind the fibre bundles together making the fibres stiff and coarse. In the present study isora fibres were treated with alkali sodium hydroxide with different percent concentrations at different time durations at room temperature to study the effect of the alkali treatment. The isora fibre shows unique physical and chemical properties after alkali treatment. As the concentration of alkali increases the isora fibres become more yellowish in colour. SEM images of the alkali treated isora fibre show more roughness and surface modification due to the removal of impurities. FTIR shows decrease in the transmittance of the hydroxyl peaks at 3300cm^{-1} suggesting, removal of the hydrogen bonding in the fiber network as a result of alkali treatment. The removal of lignin increases as the NaOH concentration increases making the fibre soft and flexible and widens its suitability for applications in engineering and textile industries.**

Keywords: bast fibre, alkali treatment, properties

INTRODUCTION

India is endowed with a vast resource of natural fibres and isora fibres are the focus of research in terms of its eco-friendly nature and properties such as renewable, biodegradable, light weight, low density and good moisture absorption. Although isora fibres have compatible strength they have inherent drawbacks, isora fibre are coarse, stiff and shows low extensibility. The isora fibres are given alkali treatment to improve certain properties along with soft hand and spinnability.

Sodium hydroxide (NaOH) has been used on various plant fibres like kapok, sisal, jute, and hemp fibres for the removal of non-cellulosic components and for improving chemical and mechanical properties of the fibre (Ansell et al, 2002, Li et al, 2007). Alkali treatment is a common chemical treatment carried out for natural fibres to remove the unwanted impurities to chemically clean and modify the fibre surface. The degumming process removes certain amount of lignin, wax and oils covering the external surface of the fibre cell wall, depolymerises cellulose and exposes the short length crystallites.

Alkali treatments can be considered in modifying the properties of natural fibres in order to improve their textile properties and make them suitable for further wet processing treatments or spinning. In the study, the changes occurring in the properties of isora fibre after the alkali treatment are investigated.

EXPERIMENTAL PROCEDURE

Alkali treatment

Sodium hydroxide solution of different concentrations (2%, 4% and 6%) was prepared by dissolving calculated amount of alkali in 500 ml of distilled water. (Table 1)

Table 1: Preparation of NaOH Stock Solution

Stock solution	NaOH Concentration (%)	Weight of NaOH (gm)	Volume of distilled water (ml)
1	2.0	10.0	500.0
2	4.0	20.0	500.0
3	6.0	30.0	500.0

The isora reeds were cut to 50 cm length, 10 g sample was weighed and scoured in various NaOH concentrations (2%, 4% and 6%) maintaining a material to liquor ratio of 1:15. The fibres were kept immersed in the alkali solution for three different time durations; 1 hour, 2 hours and 3 hours at room temperature. Concentration of NaOH in the after-treatment alkali solution was measured by titration. The samples were soaked in double distilled water in order to remove all the adhering alkali sticking on the fibre surface. The liquor was tested with digital pH meter to check neutralization. The samples were again rinsed with double distilled water. The fibres were then dried at room temperature for 24 hours drying at 100°C (Table 2)

Table 2: Alkali treatment of isora fibres

Isora fibre	10 grams
NaOH solution	2 %, 4 % and 6 %
MLR (Material to Liquor Ratio)	1:15
Temperature	room temperature
Time	1.0 hour, 2.0 hours and 3.0 hours

Titrimetric test

Titration test was conducted with the after treatment NaOH solution in the burette. Potassium hydrogen phthalate, $C_8H_5KO_4$ (Molar mass = 204.22 g/mol) is a weak acid and a primary standard for acid-base titration with sodium hydroxide solution. When KHP and NaOH combines a positive hydrogen ion leaves the KHP and a negative hydrogen atom leaves the NaOH to form (H_20) water. In the reaction one mole of KHP completely reacts with one mole of sodium hydroxide as seen in the equation: KHP+ NaOH $\rightarrow$ KNaP + H_2O

KHP 5.1 g was weighed and transferred in clean and dry 250 ml beaker. The powder crystals were dissolved in 150 ml double distilled water. The clear solution was quantitatively transferred in a clean and dry 250 mL standard volumetric flask. The solution was finally diluted to 250 ml using double distilled water and was made homogeneous on mixing. The resultant solution was 0.1 N Potassium Hydrogen Phthalate (KHP). 10 ml standard KHP solution was pipette out in a 150 ml conical flask containing a drop of phenolphthalein indicator and was titrated against the burette solution. (Table 3)

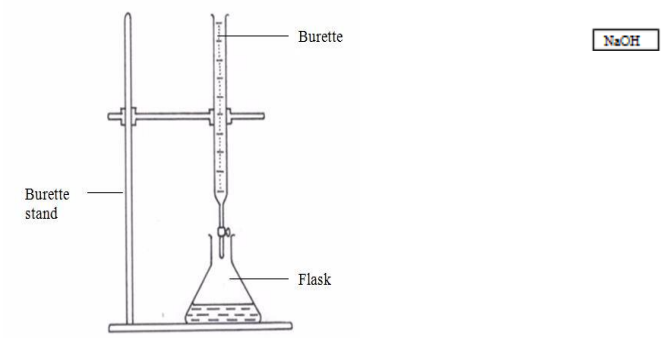


Figure 1: Titration of KHP against used alkali solution

Characterization of isora fibres

The isora fibres were evaluated for the aspects like burning, colour, morphological structure, fibre fineness, chemical composition and FTIR.

Scanning Electron Microscopy (SEM)

The untreated and alkali treated isora fibres were observed under Zeiss Evo 18 special edition scanning electron microscope. The samples were treated with 15 nm gold splutter coating using gold plated K850 Critical Point Drier, Quorum Technologies UK before scanning to make them conducive. Samples were inserted in the SEM machine; vacuum was created and micrographs were taken.

Fibre fineness (tex)

Fineness of isora fibres was measured using the gravimetric method (Booth,1996). A single strand of the isora reed were cut to constant length (20cm).about 50 numbers were counted and then weighed in an electronic weighing balance. Fibre fineness calculated using the formula below:

Fibre fineness =W/L x1000

Where,

W is weight of the known length of fibre mass

L is the total length of the reed

Estimation of lignin content

Lignin content in the isora fibres treated with different concentrations of NaOH was determined using the TAPPI standard test method T- 222. Isora fibre sample was powdered, 40 ml 72% sulfuric acid added gradually in small quantities while macerating the material with a glass rod for 2 hrs adding distilled water. The solution was then boiled for 4 hrs maintaining constant volume by using a reflux condenser. The supernatant solution was decanted without stirring up the precipitate, through a filtering crucible. Then the lignin was transferred to the filter using hot water. The lignin was washed free of acid with hot water. The crucible with lignin was dried in an oven at 105^0C, cooled in a desiccator and weighed to a constant weight.

$$\% \text{ Lignin} = \frac{A - B \times 100}{W}$$

Where,

A = Weight of crucible + insoluble fraction
B = Weight of empty crucible
W = Oven-dry weight of test specimen in grams

FTIR spectroscopy

The infrared spectra of isora fibres treated with different percentages of NaOH were measured on Perkin Elmer Spectrum Version spectrophotometer in the range 4500–400cm^{-1}.

RESEARCH FINDINGS

Titration inference

Titrimetric analysis was used to determine the concentration of NaOH left over in the after- treatment liquor. It was found that the concentration of NaOH in the after-treatment liquor was lesser than before treatment liquor (Table 3). This suggests that some amount of NaOH formed a complex with the cellulose present in isora fibres during the treatment under various conditions. There was a change in the NaOH solution colour from colourless before treatment to yellow after treatment that is indicative of non-cellulosic material being removed from the isora fibre. A similar result was noticed by Ali Arshad et al, (2015). Rumbold J.S, (1930) mentions that when a sample of cellulose is steeped in an aqueous solution of sodium hydroxide, the concentration of the sodium hydroxide is diminished by an amount which depends on the temperature and the concentration of the alkali. Cellulose combines with NaOH during alkalization process to form oxycellulose compound as in the equation:

$$\text{Cell-OH} + Na^+OH^- \rightarrow \text{Cell-O}^-Na^+ + H_2O + \text{suface impurities}$$

Table 3: Titration of NaOH

Sample No.	Before treatment NaOH (%) (A)	Time (hr)	Burette Reading (ml)	After treatment NaOH (%) (B)	Difference
1	2.0	1.0	2.6	1.5	0.5
2	2.0	2.0	2.7	1.48	0.52
3	2.0	3.0	2.6	1.5	0.5
4	4.0	1.0	1.2	3.3	0.7
5	4.0	2.0	1.2	3.3	0.7
6	4.0	3.0	1.2	3.3	0.7
7	6.0	1.0	0.8	5	1.0
8	6.0	2.0	0.8	5	1.0
9	6.0	3.0	0.8	5	1.0

Burning test

The burning behavior of the isora fibres was observed. Like all cellulosic the isora fiber ignites and burns quickly. The odour is of burning paper. The untreated isora fibres out of flame continued to burn resulting in a grey ash and the alkali treated isora fibres burnt with a black ash.

Table 4: Burning test of untreated and alkali treated isora fibre

Sample	Isora fibre	Inflame	Odour	Out of flame (Ash)
	Untreated	Burns quickly with a bright yellow flame	Burning paper	Feathery light grey ash 
	NaOH treated	Burns quickly with a bright yellow flame	Burning paper	Black ash 

Colour of isora fibre

As evident from the visual observation, the isora fibres are pale yellow in colour and became deeper yellow in colour as the alkali concentration was increased (Figure 2). This may be due to the removal of waxy layer and impurities from the fibre surface. Similar results were found with other bast fibres like banana and kenaf (Vardhini et al, 2019, Lam et al,2015). The untreated isora fibres are harsh and coarse but the alkali treated fibres are soft to touch.

Morphological analysis of isora fibre

Scanning electron microscopy images of untreated and alkali treated isora fibres suggest that they have different morphologies and proves that alkali treatment has an effect on the surface of isora fibre. The untreated isora fibres have impurities such as hemicelluloses, lignin, and waxes and appear to be smooth in the SEM images. Alkali treated isora fibres have a rough appearance indicating the loss of non-cellulosic components waxes, fatty acids, lignin and hemicellulose from the extracellular matrix.

Isora fibre treated with 2% NaOH (1 hr, 2hr, 3hr) exhibit a similar morphology as the untreated isora fibre suggesting that this concentration at room temperature is insufficient to dissolve all the non-cellulosic impurities from the fibre. At higher concentrations of 4% and 6% alkali the fibres appeared cleaner with less impurities suggesting that delignification process is more efficient at these concentrations at room temperature (Figure2).The efficiency of delignification process increases with the increasing alkali concentration which is in agreement with the recent findings of various groups (Lam et al, 2015).

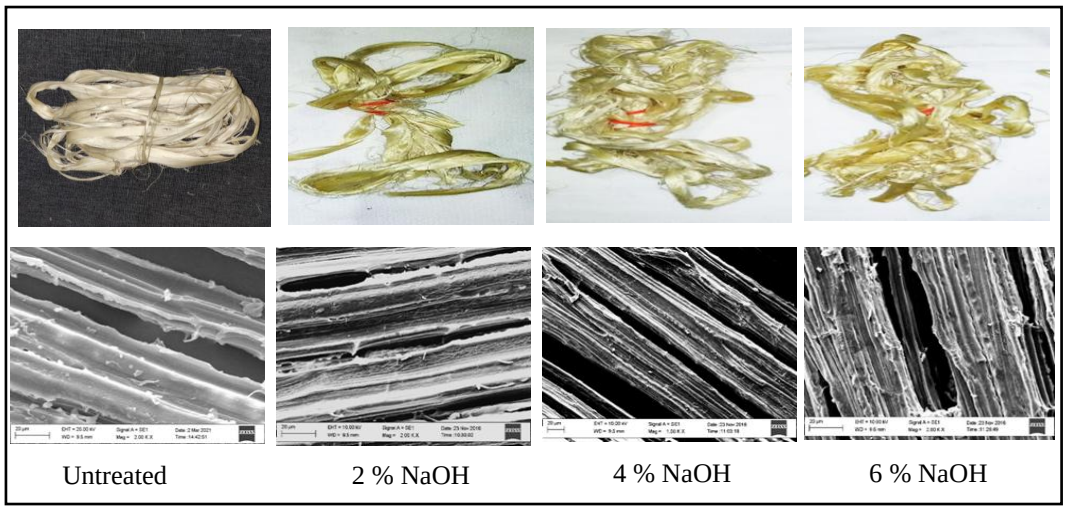


**Figure 2: Top panel: Colour of isora fibres before and after alkali treatment.
Bottom panel: SEM images of isora fibres before and after NaOH treatment at 3hrs with 2%,4% and 6%.**

Effect of alkali concentration and fibre fineness

Table 5: Fineness of isora fibres

NaOH	Untreated	2%			4%			6%		
Time		1hr	2hr	3hr	1hr	2hr	3hr	1hr	2hr	3hr
Fineness (tex)	23	21	20.7	20.5	20.5	20.4	20.2	19.6	18.6	18.2

There is a difference between the untreated and alkali treated isora fibres (Table 5). Low concentrations of alkali 2% at 1hr and 2 hr treatment time does not influence fibre fineness. The fineness of isora fibres increase with increase in alkali concentration and time duration. Isora fibre fineness is slightly increased with 4% alkali and treatment time of 3 hr.

In all 2%, 4% and 6% cold sodium hydroxide alkalization, there is a notable reading for 3 hr treatment. This shows that time is a factor and sufficient time has to be given for scouring i.e leaching out or to decompose the impurities like wax and lignin from the fibre surface. Cold alkali affects only the inter-crystalline regions resulting in swelling, but fails to reach the crystalline regions which can be reached only by concentrated sodium hydroxide (Ali Arshad, 2013).

Lignin content

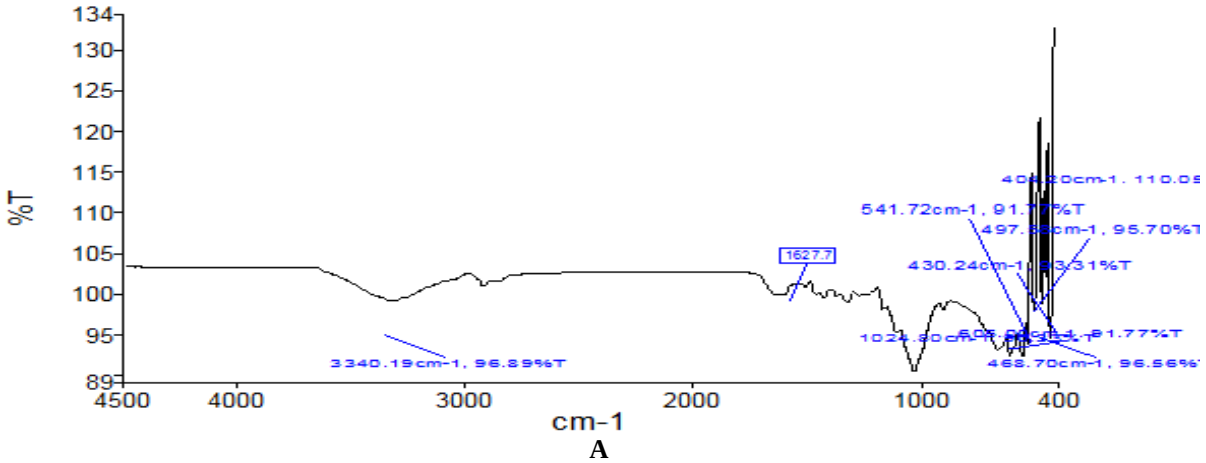
Table 6: Lignin content of NaOH treated isora fibres

Tappi-T-222	2%NaOH			4%NaOH			6%NaOH		
Untreated	1hr	2hr	3hr	1hr	2hr	3hr	1hr	2hr	3hr
18.04	18.00	17.92	17.59	17.57	17.29	16.64	17.03	16.43	15.89

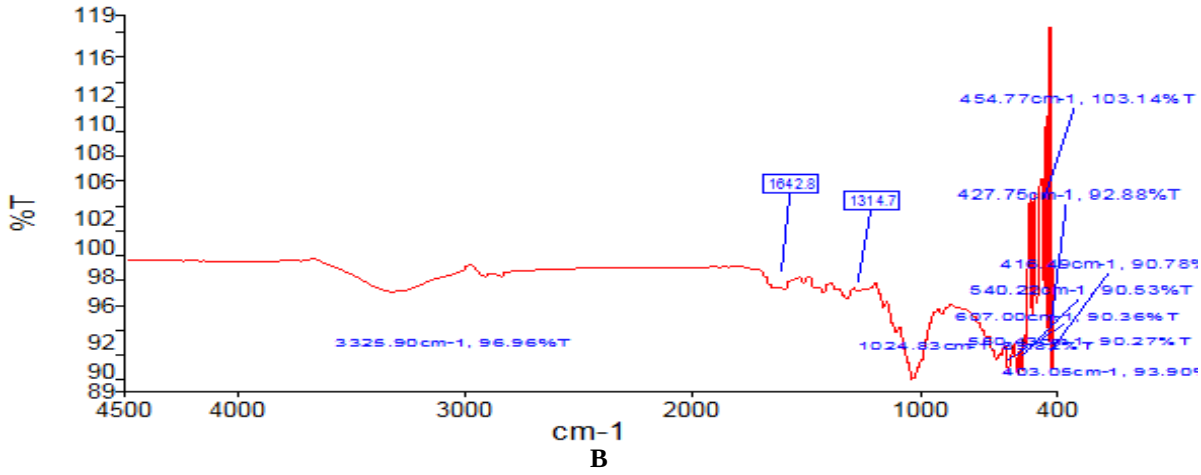
Lignin content in the natural fibres influences the structure, properties, morphology and flexibility of the fibre. Ref: Table 6. from the lignin estimation of all the 9 alkali treated isora samples the 3 hrs treatment showed good removal of lignin and it is optimized. In the untreated isora fibre lignin was 18.04 % percent. Alkali treatment at 2% concentration for 3 hrs was 17.59 percent. At 4% alkali was 16.64 percent. and 6% 15.89 percent. Similar decreasing trend of lignin values was recorded for okra fibres. Pectin's and other waxy materials were removed from the okra fibres making them soft and pliable, the moisture content in the fibres was reduced with the alkali treatment (Vasugi. N et al, 2019).

Fourier Transform Infrared Spectroscopy

FTIR test is carried out to find the presence of chemical groups in the fibres. With reference to Figure:3 the FTIR spectra taken to determine the effect of alkali treatment concentration and time on the isora fibres. In the study by (Joshy et al, 2009) the FTIR spectrum of isora fibre showed a strong broad peak at 3300–3320 cm^{-1} which is because of the hydrogen-bonded -OH stretching vibration. Second peak at 1730 cm^{-1}, which is the characteristic band for ester and carbonyl groups present in lignin and hemicellulose. Third band at 1600-1625 cm^{-1} is due to the C-C stretching of the aromatic ring in the lignin components. In the present study the IR spectra of alkali treated isora fibres show a decrease in the transmittance percentage of –OH peaks at 3300 cm^{-1} indicating the loss of hydrogen bonds in the fibre network leading to more number of free –OH groups that would absorb at 3300 cm^{-1}. There are small peaks at 1730 cm^{-1} and 1600-1625 cm^{-1} in the alkali treated isora fibres indicating some removal of hemicellulose and lignin from the isora fibre.



A



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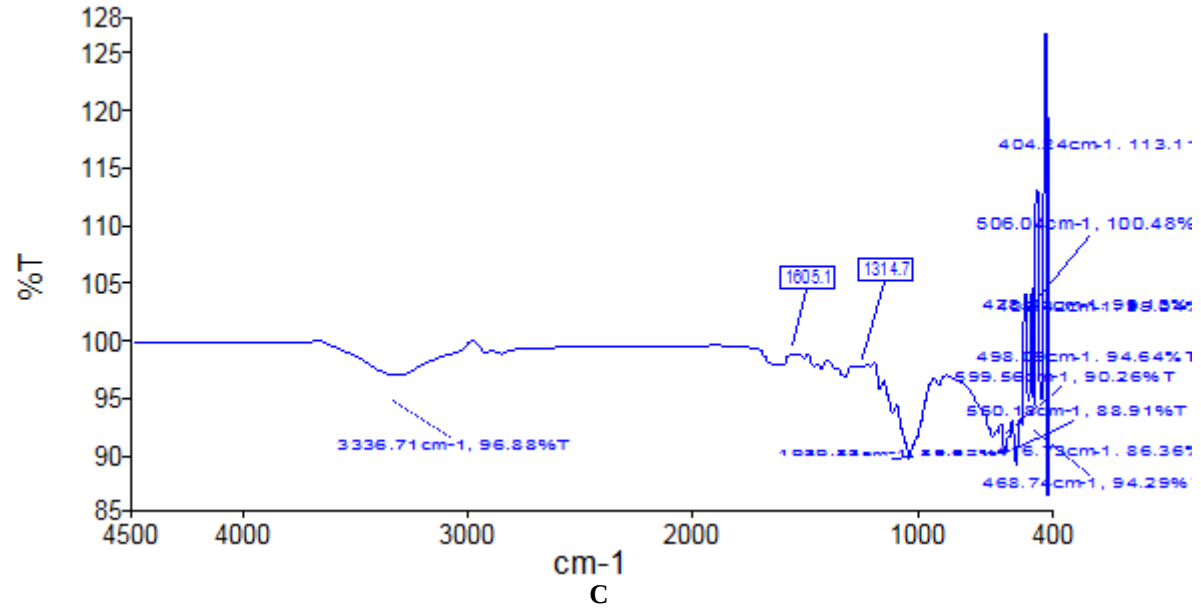

C

Figure 3 (A), (B) and (C): The FT-IR spectra of alkali treated isora fibre
(A) 2.0 % NaOH 3.0 hr (B) 4.0 % NaOH 3.0 hr (C) 6.0 % NaOH 3.0 hr

CONCLUSION

The study suggests that cold alkali sodium hydroxide treatment caused the physical and microstructural changes in the isora fibre.

- With increase in sodium hydroxide concentration and time, isora fibre fineness increases
- Morphological variations are noted in the untreated and sodium hydroxide treated isora fibre
- Removal of lignin and other impurities with increase in concentration and time
- FTIR analysis shows a difference in peaks with increase in sodium hydroxide concentration

Alkali treatment with sodium hydroxide can be used to modify to properties of the isora fibres making them suitable for use in industrial and textile applications.

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