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Pooja Bhatt
Research Scholar, Department of Clothing and Textiles, College of Home Science, G. B. Pant University of Agriculture and Technology Pantnagar, U. S. Nagar, Uttarakhand, India

Anita Rani
Professor, Department of Clothing and Textiles, College of Home Science, G. B. Pant University of Agriculture and Technology Pantnagar, U. S. Nagar, Uttarakhand, India

Manisha Gahlot
Professor, Department of Clothing and Textiles, College of Home Science, G. B. Pant University of Agriculture and Technology Pantnagar, U. S. Nagar, Uttarakhand, India

RK Srivastava
Professor, Department of Environmental Sciences, College of Basic Sciences and Humanities, G. B. Pant University of Agriculture and Technology Pantnagar, U. S. Nagar, Uttarakhand, India

Corresponding Author:
Pooja Bhatt
Research Scholar, Department of Clothing and Textiles, College of Home Science, G. B. Pant University of Agriculture and Technology Pantnagar, U. S. Nagar, Uttarakhand, India

FTIR spectroscopic analysis of delignified *Sesbania aculeata* (*Dhaincha*) Fibres

Pooja Bhatt, Anita Rani, Manisha Gahlot and RK Srivastava

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Abstract
The natural fibres offer sustainable option to textile industry in terms of reduced impact on environment as well as social acceptance due to improved health and safety aspects and economic perspectives. The bast fibres obtained from the stalks of *Sesbania aculeata* (*dhaincha*) comprise of tissue involving biomaterials like lignin. *Dhaincha* fibres are long, brittle and rough which limit their consumption to coarse end uses like yarns and cordage. Therefore, the study was conducted to soften fibres through the process of delignification and analyze the effect of delignification on *dhaincha* fibres using Fourier Transform Infrared Spectroscopy. The IR spectra indicated change in vibrational bands at 3685.51 cm^{-1}, 2855.90 cm^{-1}, 2380.64 cm^{-1}, 1729.73 cm^{-1}, 1510.31 cm^{-1}, 1363.08 cm^{-1}, 1239.34 cm^{-1}, 1101.58 cm^{-1}, 1101.58 cm^{-1} and 898.50 cm^{-1} and were mainly attributed to C-H stretching, C-O stretching and C=O stretching in the vibrational bands 1200-3000 cm^{-1} which depicted change in composition of biomaterials like lignin and hemicellulose after delignification process. These changes would ultimately help to widen scope of application of *dhaincha* fibres especially in yarn and fabric manufacturing.

Keywords: Bast fibres, *dhaincha* fibres, alkali treatment, delignification, FTIR analysis

Introduction
Natural fibres have found irreplaceable use in our daily lives and have dominated the textile industry in the world fibre production for a long period of time. The natural fibres, with their long history of serving the humanity, are very significant in wide range of applications. These are also a naturally replenishing resource which can be used without harming or damaging the environment. Despite this, the natural fibres have been losing ground and have been presently replaced by synthetic fibres such as nylon and polyester which have acclaimed versatility in use due to cost, serviceability and reproducibility.

However, with the gradual depletion of petroleum resources in the last decade worldwide, fibres derived from plants have engaged renewed interest and attention of researchers with the possibility of replacing synthetic fibres based on petrochemical resources because of their advantages of renewability, biodegradability, abundance, low cost, and reduced health hazard.

Bast fibres thus, have an opportunity to enter into high quality textile market through application of appropriate processing techniques that would enhance the physical and chemical properties of fibres. The leguminous plant *Sesbania aculeata* belongs to fabaceae family and grows in diverse climatic conditions. The plant is native to countries like India, Bangladesh, Pakistan, China, Sri Lanka, Africa, Southern United States and Philippines. The fibrous plant with pithy stem and long pinnate leaves is commonly grown in India usually in the month of July and August and plant serves variety of utility purposes mainly as green manure, intercropping crop, animal feed, medicine, cordages, etc.

Dhaincha a fast growing, succulent crop which is easily decomposable and yields maximum amount of organic matter and nitrogen as it fixes atmospheric nitrogen with the help of its root nodules. Traditional applications of *dhaincha* includes use of leaves, seeds and fruit as astringent, anthelminthic and anti-tumor agents in the medical field. The beans obtained from the plant have the ability to treat ringworms and other skin infections. The leaves yield pinitol, which acts as anti-diabetic agent along with being used for fodder, paper and biomass, cordage, fish nets, sackcloth, thickening agent and promising source of paper manufacturing. The *dhaincha* fibres had been found to be long, coarse and brittle which limit their uses (Jahan and Rahman, 2011) [7].

The *dhaincha* fibres have also found useful as non-woven and could have prospects in the field of geotextiles (Singh and Rani, 2013) [17]. The chemical characterization of the plant revealed that it is composed of 21.94%, ash content 2.15%, fats and waxes 1.34%, holocellulose 73.6%, cellulose 42.2% hemicellulose 30.9% (Negi, 2017) [11]. The encrusting substance lignin glue the fiber cells together and provides strength to the plant and stiffness to fibres. The presence of lignin greatly affects the fibre properties like stiffness, which needs to be minimized to yield fibre with better property set. This would expand the possibilities of introducing this fibre to wider field of applications. The chemical components of *dhaincha* fibres was established through use of FTIR technique.

Infrared (IR) spectroscopy or FTIR is non-destructive technique in which absorption of different IR frequencies by the sample is measured. The main objective of Fourier Transform Infrared Spectroscopy is determination of chemical functional groups present in the sample. The IR radiation absorbed by different functional groups are distinctive and characteristic frequencies. FTIR could thus be considered as a tool for compound identification and structural elucidation. The IR absorption positions are determined using wavenumber (cm^{-1}). The IR absorption information is presented with the help of spectrum which helps to analyze the compounds present in the sample (Abidi *et al.*, 2014; Hsu. 1997 and Celino *et al.*, 2014) [1, 5]. Thus, the study was conducted to remove lignin from *dhaincha* fibres to expand use in textile applications. The effect of delignification on *dhaincha* fibres was analyzed using Fourier Transform Infrared Spectroscopy.

Materials and Methods

The stems of *dhaincha* plant about two and half months old were collected from Crop Research Centre, Govind Ballabh Pant University of Agriculture and Technology (G.B.P.U.A.T.), Pantnagar, Uttarakhand, India. The ribbons of the *dhaincha* stalks were retted for 15 days and further treated chemically (Fig. 1) using delignification process (Ilyas *et al.*, 2017) [6]. The treatment focussed on removal of components like lignin, hemicellulose, pectin, etc. The chemical reagents used in the study for lignin removal from *dhaincha* ribbons were sodium chlorite ($NaClO_2$), acetic acid (CH_3COOH) and sodium hydroxide (NaOH).

Fig 1: Steps in processing of *dhaincha* fibres

The delignification process involved two phases, phase I and phase II. The fibres were washed thoroughly to remove impurities and foreign particles. The phase I of the treatment focussed on removal of lignin through use of acetic acid and sodium chlorite [ASTM D1104-56 (1978)]. The parameters of chemical treatment were optimized using Box-Behnken design of Response Surface Methodology of Design Expert Software. The optimum process for phase I of chemical treatment included acetic acid (13.719%), sodium chlorite (15.583%) for 3 hours using MLR 1:30 at 70 ^{0}C temperature. The phase II of experimental process mainly focussed on bleaching of holocellulose fibres [ASTM D1103-60 (1977)] obtained from phase I using sodium hydroxide (0.5%) for 5.5 minutes time duration. The fibres obtained after phase II were referred to as α-cellulose and neutralized by soaking in dilute acetic acid by stirring for 30 seconds and then allowed to settle for 5 minutes, rinsed with water and oven dried at 103 ^{0}C temperature.

The delignified fibres were analysed using Fourier Transform Infrared Spectroscopy Phase- II Spectrometer (Fig. 2). The powdered sample of *dhaincha* fibres was used for analysis. The FTIR spectra was obtained using Bruker software and was further analysed for establishing functional groups in *dhaincha* fibres before and after delignification process.

Fig 2: Spectrometer

Results and Discussion

The FTIR spectra of untreated and treated *dhaincha* fibres are shown in Figures 3. As shown in Fig 3, the infrared spectra for dhaincha fibres before and after treatment were recorded for range (600-3900 $^{cm-1}$) using Bruker software as the range signifies mid infrared range which depicts the specific vibrational bands related to functional groups. These groups help to determine composition of sample based on this mid-IR spectral fingerprint.

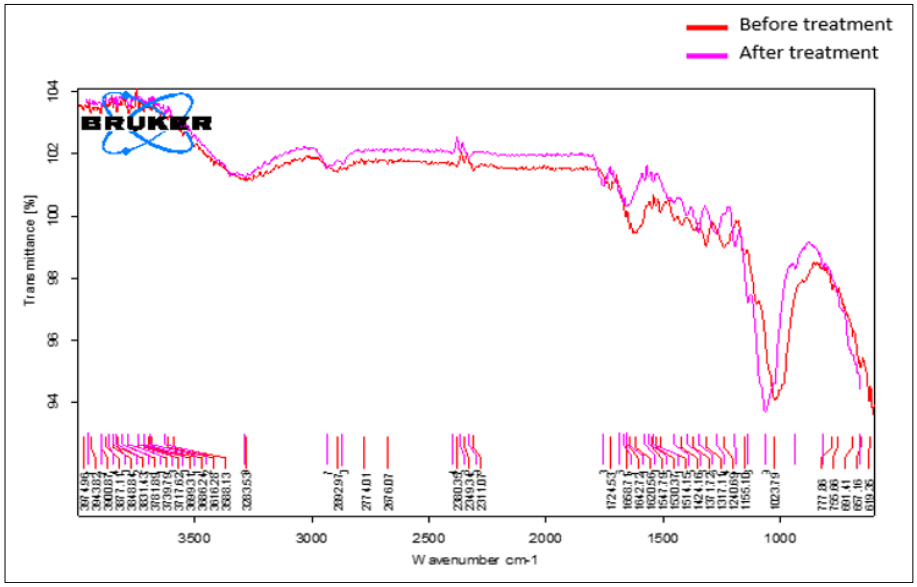

Fig 3: FTIR spectra of *dhaincha* fibres before and after treatment

Table 1: Infrared band assignments for FTIR spectra of *dhaincha* fibres before and after treatment

Wavenumber (cm^{-1})	Vibrations before treatment (cm^{-1})	Vibrations after treatment (cm^{-1})	Assignment
3700-3000	3588.13 3283.53	3685.51 2923.87	CH stretching of cellulose and hemicellulose (Johar *et al.*, 2012; Abidi *et al.*, 2014) [9, 1]
3000-2000	2892.97 2380.35	2855.90 2380.64	CH_2 stretching of cellulose and hemicellulose (Celino *et al.*, 2014; Sun *et al.*, 2005) [16]
1800-1500	1724.53	1729.73	C=O stretching of vibration of carboxylic acid in pectin or ester group in hemicellulose (Abidi *et al.*, 2014) [1]
	1642.72 1620.50	1642.67 1627.01	OH bonding vibration of adsorbed water (Celino *et al.*, 2014)
1500-1200	1547.79 1530.37 1514.15	1548.30 1530.55 1510.31	NH_2 deformation (Abidi *et al.*, 2014) [1] Aromatic ring of lignin (Abidi *et al.*, 2014) [1]
	1424.16	1424.35	Carboxylic acid of pectin and COO^- vibration (Abidi *et al.*, 2014; Celino *et al.*, 2014; Sun *et al.*, 2005) [1, 16]
	1371.72	1363.08	CH bending of cellulose and hemicellulose (Abidi *et al.*, 2014; Celino *et al.*, 2014) [1]
	1317.11	1316.16	CH_2 wagging of cellulose and hemicellulose (Abidi *et al.*, 2014; Olsson and Salmén. 2004) [1]
	1240.69	1239.34	C-O of acetyl in pectin or hemicellulose
1200-650	1155.10	1159.77	Anti-symmetrical deformation of C-O-C band (Abidi *et al.*, 2014; Rosa *et al.*, 2010) [1]
	1023.79	1101.58	C-O stretching and ring vibrational modes (Karimi *et al.*, 2014)
		1028.29 898.50	β linkage of cellulose (Abidi *et al.*, 2014) [1]
	777.86	777.42	CH_2 rocking (Abidi *et al.*, 2014; Celino *et al.*, 2014) [1]
	619.35	617.50	O-H out of plane bending (Abidi *et al.*, 2014) [1]

The infrared band assignments for FTIR spectra of *dhaincha* fibres reveals that the structural changes occurred in the *dhaincha* fibres on chemical treatment. The slight structural changes were observed in shifting of position of vibrational bands at IR range after treatment *viz*., 3685.51 cm^{-1}, 2923.87 cm^{-1} which signifies change in CH stretching of cellulose and hemicellulose, change in vibrational peak at 2855.90 cm^{-1}, 2380.64 cm^{-1} which denotes CH_2 stretching of cellulose and hemicellulose, 1729.73 cm^{-1} which relates to C=O stretching of vibration of carboxylic acid in pectin or ester group in hemicellulose), 1510.31 cm^{-1} signifying changes in aromatic ring of lignin, 1363.08 cm^{-1} assigning CH bending of cellulose and hemicellulose, 1239.34 cm^{-1} depicting change in C-O of acetyl in pectin or hemicellulose and 1101.58 cm^{-1}, 1101.58 cm^{-1}, 898.50 cm^{-1} denoting changes related to C-O stretching and ring vibrational modes and β linkage of cellulose present in the sample. Also, after chemical treatment the transmittance of the sample was observed to be increased which reveals that the bonds absorbing IR were decreased after treatment. Some new intensity peaks at 1200-650 cm^{-1} were also obtained due to C-O group (Prusty *et al.*, 2019) [14]. These changes in chemical structure of *dhaincha* fibres were observed due to delignification treatment.

Thus, the data displayed that the structural changes were mainly observed in cellulose, lignin, hemicellulose and pectin content of *dhaincha* fibres. The C-H stretching, C-O stretching and C=O stretching in the vibrational bands 1200-3000 cm^{-1} attribute to change in composition of lignin, hemicellulose and pectin present in fibre sample to desirable extent for use in textile applications. The alkaline treatment was found effective in removal of waxes, lignin and hemicellulose present in external surface of cell wall of the fibrous plants. The important modification obtained with alkaline treatment is disruption of the hydrogen bonding between the constituents of the cell wall (Faruk *et al.*, 2012) [4]. Finding of studies conducted by Jayabal *et al.*, 2012;

Oudiani, *et al.*, 2011; Ilyas, *et al.*, 2017 and Oushabi, A. *et al.*, 2017 [8, 3, 6, 13] also reported that treatment of sodium hydroxide is efficient for removal of lignin, hemicellulose and other soluble compounds from the surface of the fibers. The treatment also allows increase in the reactive groups on the surface of the fiber thus, making the sites available for chemical bonding.

The IR spectra clearly shows changes in the fibre structure after the delignification treatment. The changes in the wavenumber vibrations showed change in the structural components of fibre like lignin, hemicellulose, pectin and cellulose. This clearly indicates the required changes in the fibres which depicted softening of *dhaincha* fibres due to removal of stiffening components like lignin and hemicellulose. The softening of fibres would help to expand possibilities of use of *dhaincha* fibres in different end uses.

Conclusion

Dhaincha fibres have limited use in textiles in terms of yarn and fabric manufacturing. The main reason for the limited use is the coarse nature of the fibres. Thus, the study was conducted to soften fibres by removing lignin and other stiffening components like hemicellulose. The IR spectra clearly indicated change in composition of lignin, hemicellulose and pectin in fibres after chemical treatment in vibrational bands at 3685.51 cm^{-1}, 2855.90 cm^{-1}, 2380.64 cm^{-1}, 1729.73 cm^{-1}, 1510.31 cm^{-1}, 1363.08 cm^{-1}, 1239.34 cm^{-1}, 1101.58 cm^{-1}, 1101.58 cm^{-1} and 898.50 cm^{-1}. The results showed that changes were mainly attributed due to C-H stretching, C-O stretching and C=O stretching in the vibrational bands 1200-3000 cm^{-1} which was observed due to delignification treatment. The change in composition of biomaterials like lignin and hemicellulose would ultimately help to widen scope of application of *dhaincha* fibres especially in yarn and fabric manufacturing.

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