The Pharma Innovation Journal 2022; SP-11(10): 26-31

www.ThePharmaJournal.com

The Pharma Innovation

ISSN (E): 2277-7695
ISSN (P): 2349-8242
NAAS Rating: 5.23
TPI 2022; SP-11(10): 26-31

www.thepharmajournal.com
Received: 01-08-2022
Accepted: 06-09-2022

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Effect of wet processing on chemical characterization of sugarcane bagasse

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Abstract
The present study was carried out to investigate the possibility of utilizing sugarcane fibre for developing diversified products. The chemical extraction of Sugarcane fibres was done and the effect of wet processing such as scouring and bleaching on the extracted fibres was observed. Chemical constituents of Sugarcane Bagasse fibre were characterised for their ash, lignin, fat and wax, cellulose, hemi-cellulose, moisture content. The percentages of chemical constituent found out were highest in raw sugarcane fibre and lowest was recorded in bleached sugarcane fibre. The solubility percentages in different solvents (cold water, hot water and dilute alkali) were also characterized. Furthermore, Scanning Electron Microscopy and Infra-red (IR) Spectroscopy were characterised. The Infra-Red spectrum and Scanning Electron Microscope (SEM) test depicted the removal of non cellulosic material from fibres after wet processing treatment. The effect of wet processing treatment was clearly seen on the extracted sugarcane fibres. After scouring and bleaching, fibres became smooth and fine with lesser diameter and lesser wall thickness. The surface appearance of raw, scoured and bleached sugarcane fibres were observed under Scanning Electron Microscope. Presence of gummy substance was clear in the SEM image of raw sugarcane fibre. After scouring and bleaching the fibre appeared as cylindrical with smooth thin outer wall.

Keywords: Sugarcane bagasse, chemical characterization, wet processing

1. Introduction
Manmade fibres are generally non-degradable, whereas natural fibres are degradable and ecofriendly. The concept of ecofriendly and recyclable products which are highly recognized brings natural fibres into our sharp focus. The less explored natural fibres are nothing but stem fibres. The vegetable fibres are classified according to their origin and the parts of the plant, i.e. leaf, seed, bast, stem, fruit, grass and stalk. Their properties naturally become affected by factors, such as climate, maturity, harvesting or physical or chemical treatments (Garzón 2014) [2].

Saccharum officinarum is a large, strong-growing species of grass in the genus *Saccharum*. It is one of the most productive and most intensively cultivated kinds of sugarcane and belonging to the family poaceae. Sugarcane is a tropical, perennial grass that forms lateral shoots at the base to produce multiple stems, typically 3 to 4 m high and about 5 cm in diameter (Singh et al. 2015) [7].

Asagekar and Joshi (2013) [1] studied on "Characteristics of Sugarcane fibre" and concluded that residue left after the extraction of juice called bagasse was collected for fibre extraction the soft-core part pith was removed from the bagasse manually to get outer hard rind. Cut the rind across the length so that it should be free from nodes and then subjected to hot water treatments (material, liquor ratio 1:50). In this process the sample were kept in hot water at around 90 °C for 1 hour for removal of coloring matters and sugar traces. The samples were then dried under the sunlight. Then samples were subjected to chemical extraction. In this extraction process, samples were treated with 0.1% NAOH solution, at boiling water temperature for 4 hour. Material, liquor ratio 1:100. The well separated fibres were then dried.

Yadav *et al.* (2015) [13] in their study "A Review on Composition and Properties of Bagasse Fibers" found that physical properties of bagasse fibers with the highest aspect ratio will exhibit highest tensile properties and provide high surface area which are advantageous for reinforcement purposes, with Diameter10-34 (µm), Length 0.8-2.8 (mm), Moisture content 49 (%). Reddy and Yang (2014) conducted a study on –"Fibers from Sugarcane Bagasse" and concluded that Bagasse is a lignocellulosic material consisting of 45–55% cellulose, 20–25%

hemicellulose, and 18–24% lignin. Sugarcane stems consist of three major parts: the pith (5%), fibers (73%), and the rind (22%).

Sugarcane bagasse is one of the largest cellulose based agro industrial by -products and fibrous residue left after the sugarcane is crushed in the factories of the sugar and alcohol industry. Now a day it is used as a biofuel and as a renewable resource in the manufacture of pulp and paper products and building material. Bagasse is also used as a source of renewable power generation and for the production of bio-based materials. The bagasse can be made into soft boards, medium density fiber boards or particle boards, as well as high density hard boards using dry or wet chemical processing. Textile learner (2017) [11].

Hossain *et al.* (2013) [3] opined that Sugarcane bagasse fibre is one of the most promising natural fibre with its eco-friendliness, renewability, flexibility, low density and biodegradable properties. When the residue of bagasse is burnt off, it creates air pollution. Here the concept of re-using the fibre in both hot and cold environments. Sugarcane fibre is used as a replacement of synthetic fibre in reinforced polymer composites (FRPC).

The main product of Sugarcane is sugar, however, there are many by products of sugarcane industry such as bagasse, molasses, press mud and green top, which are used by various industries. Bagasse based industries mainly produce pulp, paper, particle boards using bagasse as a fuel, cattle feed, medium for cultivation of edible mushroom, production of furfural, etc. (Plaisier *et al.,* 2017) [5].

In this study, the extractions of Sugarcane Bagasse fibres were carried out by chemical extraction method using alkaline solution in an Autoclave Rotary Digester. Chemical constituents of Sugarcane Bagasse fibre were characterised for their ash, lignin, fat and wax, cellulose, hemi-cellulose, moisture content. The solubility percentages in different solvents (cold water, hot water and dilute alkali) were also characterized. Furthermore, Scanning Electron Microscopy and Infra-red (IR) Spectroscopy were characterised.

2. Materials and Methods

2.1 Raw materials: The waste sugarcane Bagasse was utilized for the present study. It was collected from Sugarcane Research Station of Assam Agricultural University situated at Buralikson, Golaghat. The collected sugarcane Bagasse belongs to Lohit (COBLN-9104) variety.

2.2 Extraction of fibre: The extraction of fibres was carried out by chemical extraction method. The soft core part called pith was removed from the Bagasse manually to get outer hard rind. Cut the rind across the length so that it should be free from nodes and then subjected to hot water treatments with material to liquor ratio 1:50 (Asagekar and Joshi, 2013) [1]. In this process the samples were kept in hot water at around 90 °C for one hour for removal of colouring matters and sugar traces. Samples were washed and dried under the sunlight and then subjected to chemical extraction method using alkaline solution in an Autoclave Rotary Digester.

2.3 Wet processing treatments of extracted fibers: Two different wet processing treatments, i.e. scouring and bleaching were done to study their effect on the extracted fibres.

2.3.1 Scouring: Scouring was done by treating the raw sugarcane bagasse fibre with alkali solution of 3% NaOH solution and 1% detergent ULTRAVAN JU at 100 °C for 60 minutes. After 30 minutes the fibres were washed in the running water. 10% acetic acid was used for neutralization, fibres were then washed and dried in sunlight.

2.3.2 Bleaching: Bleaching was done by using 5% H_2O_2 with material to liquor ratio 1:30 at 80-95 ºC for 45 min. After bleaching the fibres were washed properly and dried in sunlight.

2.4 Chemical analysis of Sugarcane Bagasse fibre

Chemical constituents of Sugarcane Bagasse fibre such as ash, lignin, fat and wax, cellulose, hemi-cellulose, moisture content and solubility percentage in different solvents (cold water, hot water and dilute alkali) were done.

2.4.1 Determination of moisture content

Moisture content of Sugarcane fibres were evaluated by following the TAPPI T-264 cm (TAPPI, 1980) [9] standard method. Air dried fibre of 1gm was weighed in a tarred weighing balance. The fibres were dried for 2 hours in an oven at 105C±3 °C and then cooled in desiccators. The stopper was replaced and opened momentarily to equalize the air pressure and weighed again. The bottle was again put in the oven for an hour and repeated cooling and weighing was done as above for successive hourly period until constant weight was reached. The percentage of moisture in the fibre to the nearest 0.1% was calculated by the following formula:

$$\text{Moisture content\%} = \frac{\text{Weight of the test sample - Weight of the oven dried sample}}{\text{Weight of the test sample}} \times 100$$

2.4.2 Determination of ash content

Ash content of Sugarcane fibre was evaluated by following the TAPPI standard method T.211 cm -86 (TAPPI, 1980) [9]. Three samples of oven dried raw material (1 gm) were weighed into a platinum crucible and then placed in a drying oven and reweighed until constant weight was reached The crucibles were then put into a muffle furnace at 575±25 °C. After ignition for 4 hours the ignited crucibles were cooled slightly and placed in desiccators. They were then cooled to room temperature and weighed on an analytical balance (0 to the nearest 0.1 gm). The results were expressed as % of the moisture free fibre substance as follows:

$$\text{Ash content (\%)} = \frac{\text{Over dry weight of test specimen(gram) - Oven dry weight of ash(gram)} \times 100}{\text{Weight of the test specimen(gram)}}$$

2.4.3 Determination of lignin content

Lignin content of Sugarcane Bagasse fibre was evaluated by following the TAPPI standard method T.13 m-54(1980). 1g of the sample was taken and mixed with 15ml of 72% H_2SO_4 at

20±1 ºC and kept in a water bath for 2 hour maintaining constant temperature 20±1 ºC. The material was transferred to a beaker and water was added to make the total volume become 575 ml. The solution was boiled for 4 hour and the beaker was kept overnight to settle the insoluble material. The final residue was then filtered, washed, dried at 105±3 ºC and weighed. The result was expressed as percentage as follows:

$$\text{Lignin content (\%)} = \frac{\text{Oven dry weight of the acid insoluble lignin in gram}}{\text{Oven dry weight of the test specimen in gram}} \times 100$$

2.4.4 Determination of fat and wax content

Fat content of Sugarcane Bagasse fibre was evaluated using a Soxhlet apparatus as described in BIS: IS: 199, 1971. 5gm of dried sample was transferred to a thimble and pinged with a wad of fat free cotton. The thimble with the sample was dropped into the fat extraction tube as Soxhlet apparatus and attached the bottom with the Soxhlet flask. Approximately 80ml of anhydrous ether was poured through the sample in the tube to the flask. After connecting the top of the fat extraction tube to the condenser the sample was extracted for 20 hours on a water bath. At the end of the extraction period (when ether in the flask reached a small volume) poured the content of the flask into a small dry, previously weighed beaker through a small funnel containing a plug of cotton. The flask was ringed with several portion of ether and filtered thoroughly. After evaporation of the ether under low temperature, dried the beaker at 100 °C for 1 hour, cooled and weighed again. The difference in the weight represents the ether soluble material present in the sample.

$$\text{Fat and wax content (\%)} = \frac{\text{Weight of the ether soluble material}}{\text{Weight of sample}} \times 100$$

2.4.5 Determination of cellulose content

Cellulose content of Sugarcane Bagasse fibre was analyzed by the method given by Updegraff, 1969. Take 3 ml acetic/nitric acid reagent and 1 gm of the sample in a test tube and was mixed in a vortex mixer. The tube was placed in a water bath at 100 °C for 30 min. The contents were then cooled and centrifuged for 15-20 min and then supernatant was discarded. The residue was washed with distilled water and 100 ml of 67% sulphuric acid was added and allowed to stand for 1 hr. Then 1 ml of the prepared solution was diluted to 100 ml. To 1 ml of diluted solution, 10 ml of Anthrone reagent was added and mixed well. The test tubes were then heated in a boiling water bath for 10 min and then cooled and the colour was measured at 630 nm. A blank was set with Anthrone reagent and distilled water. To prepare cellulose standard graph take series of volumes 1ml of the diluted solution (say 0.4-2 ml corresponding to 40-200 µg of cellulose) and develop the colour.

The result was expressed as percentage as follows

$$\text{Cellulose content (\%)} = \frac{\text{Absorption reading of the fiber sample}}{\text{Absorption reading of the prepared sample}} \times 100$$

2.4.6 Determination of alpha cellulose content

Alpha cellulose content of Sugarcane Bagasse fibres were analyzed by the method given by the TAPPI standard method T-429 cm-10 (TAPPI, 1980) [9]. 1 gm moisture free sample was taken in 250ml beaker. The beaker was put in a thermostatic water bath, maintained at 20 ºC. A solution of 17.5% NaOH was prepared before it was kept in the water bath at 20 ºC. The sample was then wetted with 15ml of NaOH solution and macerated gently with a flattened glass rod for 1 minute. Then 10ml NaOH was added for three times.

At the end of 10 minutes, the beaker was covered with a glass and allowed the mixture to stand for 30 minutes. After 30 minutes, 100 ml of distilled water of 20 °C was added quickly to the fibre and again allowed to stand for another 30 min. After the end of this process, the content of the fibres were poured into a tarred crucible on a clean suction flask. The beaker and the residue were then rinsed with 25 ml of 8.3% NaOH solution at 20 °C and quantitatively transferred all the fibre to the crucible. The filtered residue was then washed with 50 ml portion of distilled water. The residue on the crucible was then washed free of alkali and then heated with 2N acetic acid. The acid was sucked out after 5 minutes and pulp was washed with distilled water till free of acid. It was then dried in oven at 105±3 °C to a constant weight. The alpha cellulose was calculated as % based on moisture free cellulose.

$$\text{Alpha cellulose \%} = \frac{\text{Weight of the sample in gram}}{\text{Oven dried weight of the test specimen in gram}} \times 100$$

2.4.7 Determination of hemi cellulose content

Hemi cellulose was determined by a standard method of biochemical analysis by (Soest and Wine, 1967) [8]. 1 gm of the powdered sample was taken in a refluxing flask and 10 ml of cold neutral detergent solution was added. 2 ml of decahydronaphthalene and 0.5gm sodium sulphite was added to the above solution. The solution was heated to boiling and refluxed for 60 min. The contents were filtered through sintered glass crucible (G-2) by suction and washed with hot water with acetone. Finally, two washings were done with acetone. The residue was transferred to a crucible, dried at 100 °C for 8hr. The crucible was cooled in a desiccator and weighed.

$$\text{Hemi cellulose (\%)} = \frac{(\text{Wt of the crucible} + \text{Wt of the residue}) - \text{Wt of the crucible}}{\text{weight of the sample}} \times 100$$

2.4.8 Solubility of Sugarcane Bagasse fibre in cold water
Solubility of Sugarcane Bagasse fibre in cold water was determined by the TAPPI standard method T-207 m-54 (TAPPI, 1980). 1gm oven dried sugarcane fibre was digested at room temperature with 300ml of distilled water with frequent stirring for 48 hour. The mixture was subsequently filtered and the residue was washed with cold distilled water and then dried at 100-105 ºC and weighed in a Stoppard, weighing bottle to constant weight. The loss in weight of the fibre substance was calculated as % cold water soluble material.

$$\text{Cold water solubility (\%)} = \frac{A - B}{A} \times 100$$

Where, A = Initial oven dried weight of the specimen in gram
B = Oven dry weight of the specimen after extraction in gram

2.4.9 Solubility of Sugarcane Bagasse fibre in hot water
Solubility percentage of Sugarcane Bagasse fibre in hot water was determined by the TAPPI standard method, T-207 m-54 (TAPPI, 1980) [10]. gm oven dried Sugarcane fibre was digested with 100 ml distilled water in water bath under a reflux condenser for 3 hour and the mixture was then filtered, the residue being washed with hot water and dried at 100-105ºC to a constant weight. The loss in weight of the fibre substance was calculated as % hot water soluble material as follows:

$$\text{Hot water solubility (\%)} = \frac{A - B}{A} \times 100$$

Where
A = Initial oven dry weight of the specimen in gram
B = Oven dry weight of the specimen after extraction in gram

2.4.10 Solubility of Sugarcane Bagasse fibre in dilute alkali
Solubility percentage of Sugarcane bagasse fibre in dilute alkali was determined by the TAPPI standard method, T-212 m-54 (TAPPI, 1980) [10]. 1 gm of oven dried raw material was stirred with 100 ml of a 1% solution of NaOH, in a covered beaker, which was placed in a boiling water bath for exactly 1 hour with intermediate stirring and then the residue was washed in succession with hot water, 50 ml of 10% acetic acid and again with hot water. The final residue was then dried at 100-105 °C and weighed. The result was expressed as % on the oven dry weight of fibre samples.

$$\text{Dilute alkali solubility (\%)} = \frac{A - B}{A} \times 100$$

Where, A = Initial oven dry weight of the specimen in gram
B = Oven dry weight of the specimen after extraction in gram

2.5 Scanning Electron Microscopy: The Scanning electron microscopy (SEM) of Sugarcane Bagasse fibre was observed at CSIR-NEIST, Jorhat. Fibres were separated and mounted on specimen holder with the help of electro conductive tapes. The samples were coated with gold in an ion sputter coater (JFe 100, JEOL, Japan) in low vacuum with layer 150-200nm thick and observed in JEOL, JSM-35M-35CF electron microscope at 15 KV an accelerating potential.

2.6 Infra-red (IR) Spectroscopy: The IR-Spectroscopy of Sugarcane Bagasse fibres were investigated at CSIR-NEIST, Jorhat following the KBr (potassium bromide) disk technique (Hustubise *et al*., 1960) [4].

3. Results and Discussion

3.1 Chemical Composition

The fibres obtained after chemical extraction, scouring and bleaching were evaluated for their chemical composition, such as ash, lignin, fat and wax, cellulose, alpha-cellulose and hemi-cellulose content. The experimental data thus obtained is presented in Table1.

Table 1: Chemical analysis of Sugarcane fibre

Chemical Constituents (%)of Sugarcane fibre	Raw	Scoured	Bleached
Moisture	8.90	8.71	8.02
Ash	2.20	2.00	1.80
Lignin	25.00	24.12	23.35
Fat and wax	1.46	1.02	0.82
Cellulose	47.00	45.00	44.62
Alpha cellulose	50.00	49.30	49.00
Hemi cellulose	32.72	30.20	29.52

Table 1 shows the chemical composition of sugarcane fibre. The chemical constituents' percentages were found high in raw fiber in comparison to scoured and bleached fiber. Cellulose content in natural fibre is considered to be main component, for strength, stiffness, and structural stability and was found high in raw fiber. The lignin content in the fibres contributes to the rigidity and its value is found greater in raw fiber.

3.2 Solubility of Sugarcane fibre

The solubility of sugarcane fibres in different solvents such as cold water, hot water and dilute alkali, before and after wet treatment was determined and presented in the table2.

Table 2: Solubility of Sugarcane fibre

Solubility (%) in different solvents			
Fibres	**Cold water**	**Hot water**	**Dilute alkali**
Raw fibre	6.00	11.06	19.01
Scoured fibre	7.28	12.02	23.42
Bleached fibre	7.96	13.08	25.04

Results reported in the Table 2 showed variations in solubility of raw, scoured and bleached Sugarcane fibre in different solvents. Cold water solubility was found maximum in bleached fibre (7.96%) and minimum solubility in raw Sugarcane fibre (6.00%). Hot water solubility was found maximum in bleached Sugarcane fibre (13.08%) and minimum in raw fibre (11.06%). Among all the solvents dilute alkali showed maximum solubility in bleached Sugarcane fiber (25.04%) and minimum in raw Sugarcane fibre (19.01%).

3.3 Scanning Electron Microscopy (SEM) of Sugarcane fibre

The SEM image of raw sugarcane fibre showed as rod like structure with serrated outer wall. The cracks on the surface of raw **s**ugarcane fibre revealed the presence of gummy substance which was clearly visible. The scoured sugarcane fibre appeared as cylindrical with thin wall thickness, which might be due to decrease in non cellulosic materials and gummy substances. On the other hand, smooth outer wall lining was found in case of bleached Sugarcane fibre. The Scanning Electron Microscopy (SEM) of raw, scoured and bleached Sugarcane fibres was shown in figure 1.

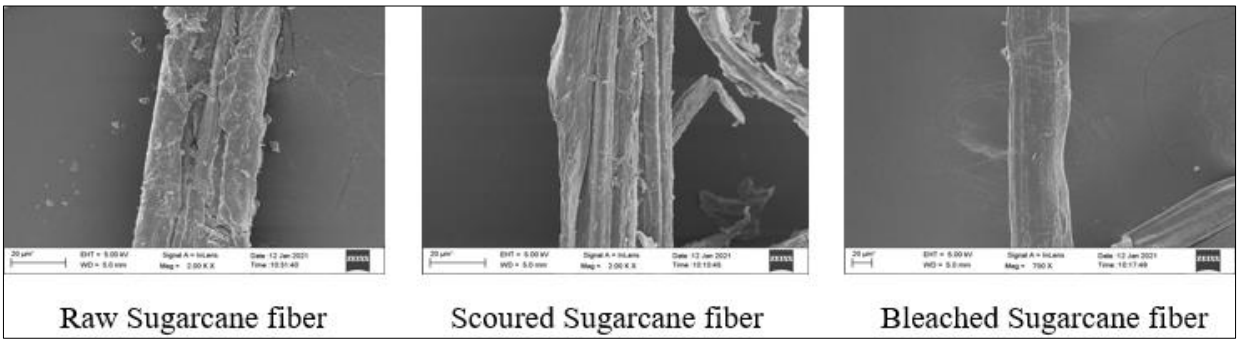

Fig 1: Scanning Electron Microscopy (SEM) of raw, scoured and bleached Sugarcane fibres

3.4 Infra-red (IR) spectroscopy of Sugarcane fibre

The small O-H stretching band at 2999 cm^{-1} might be associated with limited reactive efficiency of the hydroxyl band at the raw sugarcane fibre surface. A significant reduction in the peak was observed after scouring and bleaching. It indicates that, both scouring and bleaching remove lignin from the fibre. The carboxyl stretching vibration of ester and carboxyl groups of hemicelluloses are attributed to the band at 2283 cm^{-1}. Scouring removed the hemicelluloses to certain extent; however, in the spectrum of bleached fibre the peak is almost eliminated, which indicate the complete removal of hemicelluloses from the fibre during bleaching. The peak at 1860 cm^{-1} correspondent to C=O stretching of carbonyl, carboxyl and acetyl groups. The peak at 1548 cm^{-1} correspondent to conjugation of C=O with two aromatic rings.

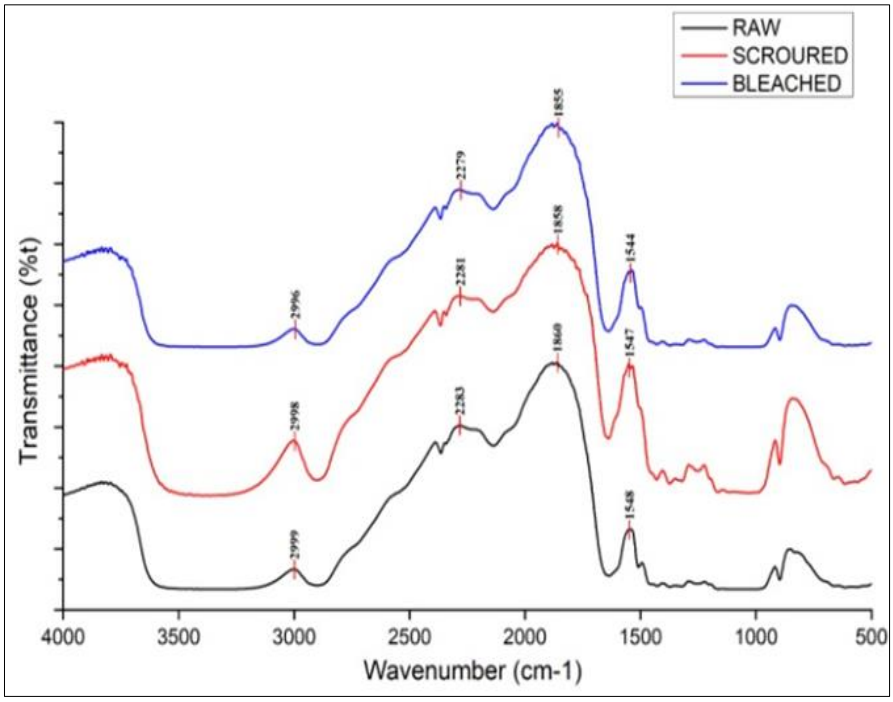

Fig 2: Infra-red (IR) spectroscopy of Raw, Scoured and Bleached Sugarcane fibre

4. Conclusion

The development of environmental friendly products is gaining popularity in order to find practical solutions to environmental problems and an urgent need to utilize agro waste for conserving the environment as well as supplementing the income of farm families. Sugarcane bagasse is one such agricultural waste found abundantly throughout the country and the world, but not being utilized to its full potential. Hence, from the tests and analysis the study highlights the following key points:

1. The percentages of chemical constituent found out were highest in raw sugarcane fibre and lowest was recorded in bleached sugarcane fibre.
2. Bleached sugarcane fibres revealed maximum solubility in all the three solvents such as cold water, hot water and dilute alkali whereas raw sugarcane fibre showed minimum solubility.
3. In IR spectra of sugarcane fibre the small O-H stretching band at 2999 cm^{-1} might be associated with limited reactive efficiency of the hydroxyl band at the raw

sugarcane fibre surface. A significant reduction in the peak was observed after scouring and bleaching. It indicates that, both scouring and bleaching remove non cellulosic matter from the fibre.

4. The surface appearance of raw, scoured and bleached sugarcane fibres were observed under Scanning Electron Microscope. Presence of gummy substance was clear in the SEM image of raw sugarcane fibre. After scouring and bleaching the fibre appeared as cylindrical with smooth thin outer wall.

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