Journal of Experimental Biology and Agricultural Sciences

<http://www.jebas.org>

ISSN No. 2320 – 8694

ECO-FRIENDLY EXTRACTION AND CHARACTERIZATION OF CELLULOSE FROM FENUGREEK (*Trigonella foenum-gracum* L.) STEM

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Received – June 10, 2020; Revision – July 28, 2020; Accepted – August 20, 2020

Available Online – August 25, 2020

DOI: [http://dx.doi.org/10.18006/2020.8\(4\).479.488](http://dx.doi.org/10.18006/2020.8(4).479.488)

KEYWORDS

Cellulose

Fenugreek stem

FT-IR

XRD

DSC

SEM

ABSTRACT

Recently, cellulose and its application acquire more attraction in the research and industrial sector due to its desirable properties like its a biopolymer, environment-friendly, inexhaustible, and its thermal and chemical stability are also high when fused with composites preparation. The present study reports an eco-friendly process that involved de-waxing, alkali, and bleaching treatments to extract cellulose from the Fenugreek stems (FS). The fenugreek stems are the chief agricultural wastes in India and can consider as absolute raw material for cellulose extraction. The obtained cellulose was characterized along with commercial cellulose to compare the efficiency of the extracted cellulose for its various properties viz., purity, presence or absence of hemicellulose, and lignin, crystallization, thermal stability, and surface morphology. The FT-IR (Fourier transform infrared spectroscopy) analysis revealed the successful removal of lignin and hemicellulose functionality. The crystallinity index obtained from XRD (X-ray diffractograms) for commercial and fenugreek cellulose found to be 55.11% and 54.68 %. The DSC (Differential scanning calorimetry) analysis confirms that extracted cellulose has (318°C to 352°C) better thermal stability than of raw fenugreek sample (314°C to 346°C). The SEM (Scanning electron microscopy) confirms that extracted cellulose has a rough and less bulky surface, which indicates the removal of non-cellulosic constituents. The results confirm that applied method gives high-grade quality and a 74% yield of cellulose, which can apply in the preparation of future biopolymer composites.

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Peer review under responsibility of Journal of Experimental Biology and Agricultural Sciences.

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1 Introduction

Agricultural waste is generated in large amount throughout the year remains the most abundant renewable resource on the globe. Agricultural wastes mainly include stalk, stem, leaf, hull, shell, bark, legumes, hay, pulp or stubble from fruits, bagasse generated from sugarcane, and many others, which is generated after crop harvesting. These agricultural wastes are highly comprised of cellulose, hemicellulose, lignin, sugars, fibers, proteins, pectin, and minerals (Abdullah et al., 2020; Huaa et al., 2020; Song et al., 2020). There is a great interest in the reuse of agricultural wastes, both from environmental and commercial viewpoints. The environmental concern is when it's disposed to the nature or burn in the field may cause soil, water, and air pollution. The economic view is to produce cellulose fibers from locally available agricultural waste to avoid high transportation costs of the raw materials (Palma et al., 2011; Sundarraj & Ranganathan, 2018; Kassab et al., 2020). Due to the limitation of non-renewable resources in recent years, the demand for cellulose-based renewable materials has been increasing (Mohanty et al., 2005).

Cellulose is a prominent component of all green plants. It is polysaccharides comprise of a linear chain of several thousands of β (1 $\rightarrow$ 4) linked D-glucopyranose monomer units (Wyman et al., 2005). Cellulose is extensively utilizing as commercial material due to its attractive characteristics such as biocompatibility, biodegradability, lightweight, renewable, thermal, and chemical stability (Sharma et al., 2019; Ma et al., 2020). Cellulose has also served as construction material, mainly in the form of intact wood and textile fibers or in the form of paper and board. A variety of stable cellulose derivatives like cellulose esters and cellulose ethers reported, which used in numerous areas of pharmaceuticals and cosmetic industries. In the pharmaceutical, it's used as delayed-release coated dosage forms, controlled release matrices, osmotic drug delivery systems, bioadhesives, as thickening agents, and as stabilizers in liquid dosage preparation. These derivatives have also used as filler, taste masker, free-flowing agents, and pressure-sensitive adhesives in the cosmetic industry (Shokri & Adibkia, 2013; Kavitha et al., 2020; Xu et al., 2020).

From the agricultural wastes, pure cellulose can be extracted by applying different pretreatments. It includes physical, chemical, and biological treatments. The physical treatment involves biomass size reduction, steam explosion, hydrothermal, and microwave-assisted. The chemical treatment includes the usage of alkaline, acidic, ionic liquid, and organic solvents. The biological treatment includes the usage of bacteria and fungi. These pretreatments help in eliminating non-cellulosic constituents and reduce the particle size of agricultural wastes (Bhatia et al., 2020; Rezanian et al., 2020; Sankaran et al., 2020). Various agro-wastes that have been

already used for cellulose extraction are wheat straw, mulberry bark, coconut husk (Johar et al., 2012), garlic stalk, onion skin (Reddy & Rhim, 2018), bagasse, bamboo, hemp, jute, oil palm, sisal (John & Anandjiwala, 2008).

Fenugreek (*Trigonella foenum-gracum* L.) is cultivated worldwide as a semi-arid crop. Its seeds and leaves are used as ingredients in food as well as in medicinal applications such as antibacterial, anticancer, antiulcer, antioxidant, hypoglycaemic, and antidiabetic agents (Murlidhar & Goswami, 2012; Srivastava et al., 2020). Major fenugreek producing countries are Afghanistan, Argentina, Bangladesh, Egypt, France, India, Iran, Morocco, Nepal, Pakistan, Spain, and Turkey. India is the largest producer of fenugreek in the world by the production of 45,000-55,000 tons per year (Vidyashankar, 2014; Yao et al., 2019). India's major producer states are Gujarat, Haryana, Madhya Pradesh, Maharashtra, Punjab, Rajasthan, Uttar Pradesh, and Uttarakhand. Rajasthan accounts for over 80% of India's total output (Lal et al., 2014; Parthasarathy et al., 2008).

The main aim of the present study is to design a new and low-cost eco-friendly method for the extraction of cellulose from the fenugreek stems. In the present study, for the first time fenugreek stems used as starting material for the extraction of cellulose. Not yet an early study reported on fenugreek stem, which used as raw material for cellulose extraction. It is inexpensive and locally available in large quantities in India. In the part of the study, commercial cellulose was used as reference material to check the purity of extracted cellulose in order to extend the validity of our conclusions.

2 Materials and Methods

2.1 Materials

Fenugreek stems utilized in the experiment were collected from the local farm of the Amreli district, Gujarat, India. Stems were extensively washed with distilled water to remove impurities mainly dust. The materials were shaded dry and chopped into small pieces and milled to pass through 2mm mesh. The milled samples then stored at room temperature. The reagents and chemical used in the study such as n-Hexane (99%, Merck), methanol (99%, Astron chemicals India), sodium hydroxide (HIMEDIA), commercial cellulose powder (97%, HIMEDIA), maleic acid (99%, HIMEDIA), citric acid (99%, HIMEDIA), hydrogen peroxide (50%, Astron chemicals India), and other chemicals were of standard analytical grades.

2.2 Extraction of Cellulose from Fenugreek stem

A various study reported that the lower alkali concentration was favorable for cellulose extraction from agricultural wastes. In the alkali solution, lignin and hemicellulose are hydrolyzed and

becomes water-soluble. Mandal & Chakrabarty (2011) reported that, addition of acid in the bleaching treatment helps to disintegrate cellulose fibers. Similar facts were reported by Mwaikambo & Ansell (2002) and Cherian et al. (2010). In the present work, we drafted a new eco-friendly method to extract cellulose from the fenugreek stem. For the production of the maximum yield of cellulose from the fenugreek stem, in current study different concentration of NaOH solutions was added in the alkali and citric acid solutions of the bleaching treatment. On the based of the results, the optimized method is given in figure 1. Extracted cellulose kept in an airtight container for further analysis at room temperature. The cellulose yield (%) was determined by using Leão et al. (2017) equation.

$$\text{Cellulose Yield (\%)} = \frac{M_p}{M_s} \times 100 \quad \dots (1)$$

Where as M_p is the mass of final product

M_s is the mass of raw sample

2.3 Characterization

The extracted cellulose was characterized using following techniques which confirms the purity of fenugreek cellulose.

2.3.1 Fourier Transform Infrared Spectroscopy (FTIR)

The functional group and chemical structure of samples were evaluated by FT-IR spectroscopy using the BRUKER ALPHA-II ATR-FTIR (Germany) spectrometer in the region from 4000 to 400 cm^{-1} and a total of 32 scans for each sample.

2.3.2 X-ray diffraction (XRD)

The X-ray diffraction of the samples were carried out using RIGAKU ULTIMA IV Powder X-Ray Diffractometer (Tokyo, Japan), with $\text{CuK}\alpha$ radiation ($\lambda=1.54\text{\AA}$). The crystallinity index (CI) of the samples was calculated according to the Segal et al. (1959) equation.

Figure 1 Extraction of cellulose from fenugreek stem

Table 1 Optimization of fenugreek cellulose extraction process

Sample No.	Concentration of NaOH	Acid ^a	Temp.(°C)	Time (hr)	Cellulose Weight (gms)	Cellulose yield (%)
1	1% wt NaOH	Citric acid 5%	10	1	5.8	58
2	2% wt NaOH	Citric acid 10%	20	2	6.2	62
3	3% wt NaOH	Citric acid 15%	30	3	6.7	67
4	4% wt NaOH	Citric acid 20%	40	4	7.4	74
5	5% wt NaOH	Citric acid 25%	50	5	7.0	70

Results have been taken on the basis of average of minimum triplet repeated process; Initial weight of the raw fenugreek sample = 10g.

^aDifferent concentrations of citric acid taken into bleaching process.

$$Crl(\%) = \frac{(I_{002} - I_{am})}{I_{002}} \times 100 \quad \dots (2)$$

Where: *Crl* the relative degree of crystallinity, *I*₀₀₂ is the intensity of the highest intensity peak which arises from both crystalline and amorphous regions and *I*_{am} is the intensity of the amorphous peak.

2.3.3 Differential scanning calorimetry (DSC)

Using Shimadzu DSC-60 Plus (Kyoto, Japan) differential scanning calorimeter the thermal behaviour of samples were studied. Each sample was heated from 30°C to 400°C at a heating rate of 10°C/min under the nitrogen atmosphere. For this 7 mg sample was weighed using hermetically sealed pans with a pinhole in the lid. The thermograms indicated the onset of melting temperature and crystallization temperature of the samples.

2.3.4 Scanning electron microscopy (SEM)

The surface morphology of the samples were investigated by using scanning electron microscope (SEM). The photographs of sample surfaces were captured using Scanning Electron Microscope XL 30 ESEM EDAX (Philips, Netherland). The samples were coated over double side carbon tape. The accelerating voltage was 10 kV.

3 Results and Discussion

3.1 Yield of Cellulose (%)

The cellulose extraction process was optimized in five different reactions. The reaction parameters like concentration of NaOH, time, temperature, and concentration of citric acid examined to evaluate the optimum reaction conditions. The obtained results presented in table 1. In the fenugreek stem, cellulose is surrounded by hemicellulose, lignin, wax, and other cementing materials. For the removal of these materials, alkali treatment performed using sodium hydroxide (NaOH). The concentration of sodium hydroxide, time of the treatment, and operating temperature mainly affect on the yield and properties of cellulose. From table 1 it is clearly observed if alkali treatment performed at a high temperature for a long duration

with a high concentration of sodium hydroxide, cellulose crystal parts start to degrade which results in a lower yield of cellulose. Citric acids taken into bleaching treatments helps in the elimination of the rest of hemicellulose and improve the properties of cellulose (Bartos et al., 2020; Baskaran et al., 2020; Xia et al., 2020). The optimum conditions of the reactions were 4% wt NaOH, for 4 hr, at 40°C with 20% citric acid, where maximum yield 74% of cellulose was recovered.

3.2 Fourier Transform Infrared Spectroscopy (FTIR)

FT-IR spectra of commercial and fenugreek cellulose have been shown in figure 2. A strong band appeared at 3500 cm⁻¹- 3000 cm⁻¹, which is associated with the stretching vibration of O-H groups having strong inter and intra-molecular H-bonding (Kamsonlian et al., 2011; Paniz et al., 2020). The stretching frequency at 2893 cm⁻¹ and 2904 cm⁻¹ was due to the symmetric C-H group of cellulose (Rosa et al., 2012; Galiwango et al., 2019). The band at 1736 cm⁻¹ corresponds to the carbonyl group (C=O) due to the presence of acetyl ester and carbonyl aldehyde groups of hemicellulose (Ventura-Cruz & Tecante, 2019; da Silva & Poletto, 2020; Hussin et al., 2020). This band completely disappeared in the extracted cellulose because of the removal of most of the hemicellulose. The peaks at 1254, 1508 and 1604 cm⁻¹ correspond to the aromatic skeletal vibrations of lignin (Morán et al., 2008; De et al., 2020). These peaks are absent in fenugreek cellulose indicating the complete removal of lignin. The absorption band at 1163 cm⁻¹ corresponds to the C-O antisymmetric bridge stretching of cellulose (Lubis et al., 2019; Khan et al., 2020).

The absorption bands at 1110 and 1055 cm⁻¹ correspond to C-O-C the β-1, 4-glycosidic ring linkages between the D-glucose units in cellulose (Tarchoun et al., 2019; Kalpana & Perarasu, 2020). The peaks at 1061 and 897 cm⁻¹ are associated with cellulose, the C-O stretching, and the C-H rock vibrations of the cellulose (Trilokesh & Uppuluri, 2019; Shaker et al., 2020). The sharp signals at 895 cm⁻¹ of fenugreek cellulose and commercial cellulose reflect the crystalline band of cellulose (Fan et al., 2012).

Figure 2 FT-IR Spectra of - (a) Commercial cellulose (b) Fenugreek cellulose

Figure 3 XRD patterns of (a) Commercial cellulose (b) Fenugreek cellulose

3.3 X-ray diffraction (XRD)

XRD patterns of commercial and fenugreek cellulose have been represented by figure 3. The XRD pattern of commercial cellulose shows three well-defined peaks at $2\theta = 15.80^\circ$, 22.88° , and 35.20° . Similarly, XRD patterns of fenugreek cellulose also showed three peaks at $2\theta = 15.10^\circ$, 22.63° , and 34.53° . Table 2 represents the 2θ peak values of commercial and fenugreek cellulose. The broad peak at $2\theta = 15.10^\circ$ corresponds to the (110) crystallographic plane. This peak is more evident than the sample enclosed with more amorphous material, such as lignin, hemicelluloses, pectin, and oil/wax. The

broad peak at $2\theta = 22.63^\circ$ corresponds to the (002) crystallographic plane (Melikoğlu et al., 2019; Gu et al., 2020). These show the possible relaxation of stress in the cellulose chains as a result of the removal of its amorphous constituents (Li et al., 2009; Sutrisno et al., 2020). The crystallinity index of the fenugreek stem has been increasing after the removal of non-cellulosic constituents. It is possible after alkali treatment, it produces cleavage in the hydrogen bonds, which leads to the removal of cementing materials (Wang et al., 2019). The crystallinity index obtained from X-ray diffractograms for commercial and fenugreek cellulose found to be 55.11% and 54.68 % respectively.

Table 2 Crystallinity index (Crl) of commercial cellulose and fenugreek cellulose

Sample	2-theta (deg)	D (ang.)	Height (counts)	FWHM (deg)	Int. I (counts deg)	Int. W (deg)	Size (ang.)
Commercial cellulose	15.80	5.604	568	4.77	4062	7.2	17.6
	22.88	3.883	1091	2.62	3961	3.63	32.3
	35.20	2.548	73	3.7	582	7.9	23
Fenugreek cellulose	15.10	5.864	1008	2.70	3766	3.74	31.0
	22.63	3.925	2597	1.73	6774	2.61	48.9
	34.53	2.596	180	1.57	359	2.0	55

Figure 4 DSC curve of (a) Raw fenugreek sample (b) Commercial cellulose (C) Fenugreek cellulose

3.4 Differential scanning calorimetry (DSC)

Figure 4 shows the three DSC thermograms of raw fenugreek sample, commercial, and fenugreek cellulose. All three DSC thermograms exhibit two distinct endothermic changes within the temperature range of 30°C to 400°C. However, the nature of endotherms is dependent on the composition of the materials. In all three thermograms, the first endothermic peak observed due to water evaporation from 50°C to near 100°C (Rasheed et al., 2020; Ufodike et al., 2020). Moreover, the amorphous character of the cellulosic material absorbs more water and shows a sharp endothermic peak. The above phenomenon rarely observed with

crystalline cellulose. Therefore shows no or minimal peak lines (Mandal & Chakrabarty, 2011). Similarly, Yang et al. (2007) report that thermal decomposition regions of cellulose are in the range of 315°C -400°C. The melting point observed in the second endotherm in three cases describes the nature of the decomposition of the crystallites. The non-cellulosic constituents in the raw fenugreek sample make it less stable, whereby decreases the melting temperature in the range from 314°C to 346°C.

The fenugreek cellulose loses a substantial amount of non-cellulosic materials and thereby rearrange and reorients into crystalline form resulting in a compact structure. The resultant

Figure 5 SEM micrograph of (a) Raw fenugreek sample (b) Fenugreek cellulose (C) Commercial cellulose

fenugreek cellulose confirms a rise in the melting temperature from 318°C to 352°C. The melting temperature range of fenugreek cellulose obtained from DSC confirms it is close to the commercial cellulose 316°C -359°C.

3.5 Scanning electron microscopy (SEM)

SEM micrographs of the raw fenugreek sample, commercial, and fenugreek cellulose are illustrated in Figure 5. The raw fenugreek sample shows (figure 5a) a very smooth surface indicates the presence of non-cellulosic material such as lignin, hemicellulose, wax, and other cementing materials (Kian et al., 2020). Bleaching with H₂O₂ and citric acid helps to eliminate the rest of lignin-hemicellulose and besides disintegration leading to the development of pure cellulose. This was observed from figure 5b that fenugreek cellulose has loose fibers, rough and less bulky appearance. Alkaline and bleaching treatment eliminates unwanted materials and increases the surface area of cellulose (Pereira et al., 2020). Furthermore, confirmation compares to commercial cellulose micrograph (figure 5 c) similar surface structure shown in the fenugreek cellulose micrograph.

Conclusion

Agricultural waste such as fenugreek stem is an attractive alternative source for cellulose extraction. Cellulose was extracted from the dewaxed fenugreek stem. An optimized process was given a 74% yield of cellulose. The FTIR spectroscopic revealed changes in functional groups of fenugreek cellulose indicate the removal of wax, lignin, and hemicelluloses functionalities. XRD analysis intimated crystallinity of fenugreek cellulose is similar to commercial cellulose. Further, based on the DSC analyses, fenugreek cellulose was found to exhibit better thermal stability than raw fenugreek sample which could stand with higher temperatures when infused with polymeric composites for future applications. The morphological examination by SEM indicated that a rougher and less bulky structure of fenugreek cellulose was obtained after the given treatments. The cellulose obtained from fenugreek stems has similar characteristics to commercial cellulose which has been finding potential applications as raw material for pulp and paper industry, as green composites, as water absorbents, or as raw materials for cellulose derivatives. This work may give a new path for the high utilization of fenugreek stem waste.

Acknowledgments

The authors admirably acknowledge the financial support provided by the INSPIRE programme under the Assured

Opportunity for Research Careers (AORC) scheme, financed by The Department of Science and Technology (DST) (Sanction Order No.: DST/INSPIRE Fellowship/2016/IF160737, New Delhi, India.

Conflict of Interest

No potential conflict of interest was reported by the authors.

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