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Extraction of cellulose from Lignocellulosic biomass and their application in Handmade Paper making

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ABSTRACT

In the present study, Chickpea (*Cicer arietinum* L.) stems are used for the extraction of cellulose and successfully applied for handmade paper making. The obtained cellulose characterized using chemical analysis, Fourier transforms infrared spectroscopy (FTIR), X-ray diffraction (XRD), Differential scanning calorimetry (DSC), and Scanning electron microscopy (SEM). Chemical analysis evidenced that treated chickpea stem has 49.23% cellulose content. The FTIR analysis revealed changes in functional groups indicate removal of non-cellulosic constituents. The XRD analysis evidenced that extracted cellulose has a 53% crystallinity index. DSC studies demonstrate extracted cellulose stand at higher thermal degradation temperatures from 337 °C to 368 °C. The morphology of the extracted cellulose showed a coarse and fibrous surface. The physical properties of the hand made paper produced from chickpea cellulose pulp indicate the suitability of chickpea stem cellulose as an alternative non-wood source for the pulp and paper industry.

Key words: *Cellulose, Characterization, Chickpea stem, Handmade paper, Physical properties, Pulp*

Introduction

The world's population and economic growth are increasing its required high energy demand in the future. To meet this energy demand need to utilize alternative as well as renewable energy resources (EIA 2019). Lignocellulosic biomass is a tremendous renewable energy source on the earth. Because it's generated in huge amount throughout the year is around 120×10^9 ton, which is equivalent to 2.2×10^{21} Joule, 300 times higher than existing global energy demands (Abraham *et al*., 2020). The cell wall structure of lignocellulosic biomass mainly consists of three kinds of polymers; cellulose, hemicellulose, lignin, and other extractives in a less amount. However, the composition and content of these three components vary due to differences in species, types, and sources of lignocellulosic biomass (Konishi, 2020). Cellulose is the major structural and integral part of lignocellulosic biomass,which is a linear homopolysaccharide of β-1, 4- linked anhydrous D-glucose units with the repeating unit of cellobiose. These are held together by intra and intermolecular hydrogen bonds, which increases the crystallinity of cellulose and also makes it highly insoluble and resistant to many organic solvents (Baruah *et al*., 2018).

Cellulose demand is increasing due to it has some novel properties like biodegradable in nature, comparatively cheap, and fully or partially recyclable after use. Obtaining pure cellulose from lignocellulosic biomass is essential due to its potential

in various applications, such as building material, preparation in cellulose-based threads, and films, as textile fibers, as thickeners and stabilizers in process foods, as a gelling agent, as adsorbent, and as packaging and wrapping material (Husin *et al.*, 2019).

With the growing trend of environmental friendliness, need for the handmade papers produced from natural cellulose are rising. Presently, Paper mills need to produced 500 million tons of paper and paperboard (Kumar *et al.*, 2019). The increasing price of traditionally used cellulosic raw materials like cotton rags and hosiery waste, forcing the handmade paper industry to explore new cellulosic raw materials (Kumar *et al.*, 2013).

Chickpea (*Cicer arietinum* L.) is also known as Bengal gram or Gram, belonging to the Fabaceae family. This plant is fast-growing, branched, and reaches a height of 20 to 60 cm. It has a deep taproot, stems are hairy, and branched (Heuzé *et al.*, 2015). Currently, India is the largest producer and consumer of chickpeas (Brouazin *et al.*, 2017). Chickpea stem is an abundant source of biomass. Most of the chickpea stems are burnt in the field after seed harvest. These causes air, and soil pollution. Hence, there is a great concern to convert these stems into valuable products. The main objective of the present study was to obtain a paper using chickpea stems as a source of cellulose. Chemical characterization of chickpea cellulose was conducted to determine its potential for pulp and paper production. The physical properties of the prepared paper sheets were performed to check the quality of the paper.

Materials and Methods

Chickpea stems used in the experiment collected from the local farm of the Amreli district,Gujarat, India. The chemicals used in this study are Hexane (99% Merck), Methanol (99.8% HIMEDIA), Sodium hydroxide pellets (98% HIMEDIA), Maleic acid (99% HIMEDIA), Hydrogen peroxide (50% Astron chemicals India), Citric acid (99.7% HIMEDIA), Guar gum powder (HIMEDIA) and Cationic starch (Astron chemicals India) were of standard analytical grades and used without further purification.

Extraction of cellulose from chickpea stem

The cellulose extraction process has followed the method described by Hapani, Highland, and George (2020).

Pulp and Handmade paper preparation

Recovered cellulose used for pulp preparation. Chickpea cellulose blended with guar gum: cationic starch (ratio 1:1) and a little amount of water were added for a smooth mixture. Guar gum is amphoteric, form a binder complex with cationic starch that increases the strength of the paper sheet (Bhardwaj *et al.*, 2019). The pulp mixture poured into a round sieve. After a few minutes, paper on the sieve was transferred in absorptive cloth and placed on a platform for pressing with blocks (Anyanwu *et al.*, 2012). Later, it was kept under the sun to dry up. Figure 1 is showing the complete steps of handmade paper preparation.

Chemical analysis

The cellulose, lignin, and hemicellulose content of the chickpea stem were measured using the method explained by Jayaramudu, Guduri, and Rajulu (2010). The ash content is quantified as per the

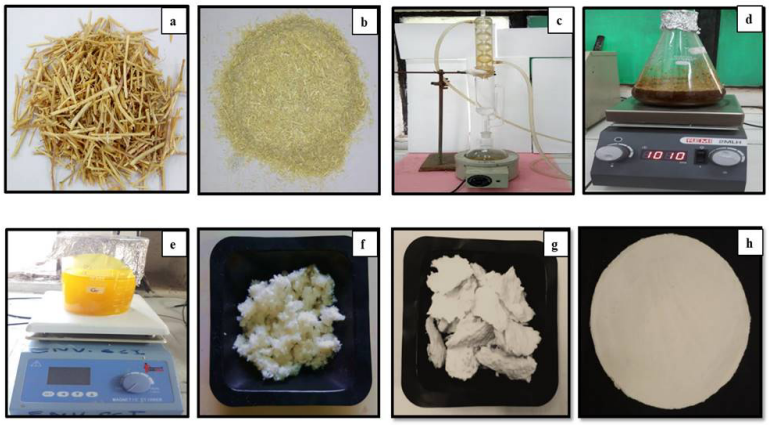

Fig. 1. Photographs of (a) chickpea dry stems (b) grounded stems powder (c) soxhlet apparatus set-up (d) alkali treatment set-up (e) bleaching treatment set-up (f) extracted cellulose (g) obtained pulp (h) handmade paper

TAPPI standard (T211 om-02) (Tappi, 2002).

Pulp analysis

The pulp yield gravimetrically determined as the weight of the dry pulp recovered minus initial dry cellulose took. The physical properties of hand sheets prepared in the laboratory tested for, Tear index (T414 om-98) (TAPPI, 1998), Tensile index (T494 om-01) (TAPPI, 2006), Burst index (T403 om-97) (TAPPI, 1997), Folding endurance (T511 om-02) (TAPPI, 2006) and Brightness (T 525 om-02) (TAPPI, 1992).

FTIR study

The functional group and chemical structure of the treated and raw chickpea 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.

XRD study

The X-ray diffraction was carried out to study the crystallinity of treated and raw chickpea samples using the RIGAKU ULTIMA IV Powder X-Ray Diffractometer (Tokyo, Japan), with CuKα radiation (λ=1.54A°). The crystallinity index of the samples determined by using the following equation (Segal *et al.*, 1959):

$$\text{CrI}\ (\%) = \frac{I_{(002)} - I_{(am)}}{I_{(002)}} \times 100 \quad (1)$$

Where, CrI is the relative degree of crystallinity, $I_{(002)}$ is the intensity of the highest intensity peak which arises from the crystalline region and $I_{(am)}$ is the intensity of the amorphous peak.

DSC study

Using the Shimadzu DSC-60 Plus differential scanning calorimeter (Kyoto, Japan), the thermal behavior of treated and raw chickpea samples was studied. Each sample was heated from 30 °C to 400 °C at a heating rate of 10 °C min under the nitrogen atmosphere. 7 mg sample weighed using hermetically sealed pans with a pinhole in the lid.

SEM study

The surface morphology of the treated and raw chickpea samples was investigated by using a Scanning Electron Microscope XL 30 ESEM EDAX (Philips, Netherland). The samples coated over double side carbon tape. The accelerating voltage was 10 kV.

Results and Discussion

Chemical analysis

The chemical composition of treated and raw chickpea stems shown in Table 1, with other lignocellulosic biomass for comparison. It is observed from Table 1; treated chickpea stem has higher cellulose content compared with raw chickpea stem, and other biomass like olive prunings, wheat straw, sunflower stalk, sorghum stalks, and rice straw. After alkali treatment, the treated chickpea has a less amorphous materials. Hemicellulose content also decreases from 14.26 to 7.84%. Ash content increasing from 2.34 to 5.08 % typically shows the elimination of amorphous elements from the treated chickpea stem (Saravanakumar *et al.*, 2014).

Fourier transforms infrared spectroscopy (FTIR) analysis

FT-IR spectra of the treated and raw chickpea

Table 1. Chemical composition of treated and raw chickpea stem with other lignocellulosic biomass

Fibers	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Extractives* (%)	Ash (%)	Reference
Bagasse	32-48	21	19.9-24	1.73	2.10	(Sun *et al.*, 2004)
Cotton stalks	58.48	14.38	21.45	1.42	2.17	(Jiménez *et al.*,2006)
Olive prunings	35.67	25.80	19.71	10.36	1.36	(Jimenez *et al.*,1990)
Wheat straw	39.72	36.48	17.28	4.01	6.49	(Jimenez *et al.*, 1990)
Sunflower stalk	37.6	6 ˜	10.8	4.07	7.90	(Lopez *et al.*, 2005)
Sorghum stalk	41.50	24.43	15.64	7.99	4.85	(Jiménez *et al.*, 1993)
Rice straw	41.20	6 ˜	21.9	0.56	9.2	(Rodríguez *et al.*, 2008)
Raw Chickpea stem	43.22	14.26	17.11	6.12	2.34	Present study
Treated Chickpea stem	49.23	7.84	8.76	2.17	5.08	Present study

*Solubility in hexane:methanol

samples are shown in Figure 2. The disappearance of some peaks in the spectra of the treated sample indicates the removal of unwanted components. The absorption at 3330 cm^{-1} corresponds to the O-H stretching vibration and hydrogen band of the hydroxyl group. The band at 2898 cm^{-1} represents the C-H stretching (Kumneadklang *et al.*, 2019), which is present in the treated chickpea sample. The intensity of the hemicellulose peak occurred at around 1730 cm^{-1} presence in the raw chickpea stem, and this peak is not visible in the treated chickpea sample, which indicates that hemicellulose eliminated completely (Martínez-Sanz *et al.*, 2020). The band at 1636 cm^{-1} relates to the bending mode of the absorbed water (Alam *et al.*, 2020). The peaks at 1607 cm^{-1} and 1240 cm^{-1} found in the spectrum of raw sample correspond to the aromatic skeletal vibrations of lignin (De *et al.*, 2020). The absorption band at 1426 cm^{-1} in the treated sample was attributed to the (CH_2) groups of cellulose. The presence of a peak at 1363 cm^{-1} is associated with (C-H) groups of cellulose. The absorption band at 1160 cm^{-1} corresponds to the C-O-C asymmetrical bridge stretching. The strong peak at 1053 cm^{-1} arises from the C-O-C pyranose ring skeletal vibration of cellulose (Kalpana and Perarasu, 2020). The peak at 897 cm^{-1} of the β-glucosidic linkage was attributed to the O-C-O stretching during the C-H deformation of cellulose (Jinitha *et al.*, 2018). The FT-IR results indicate that the hemicellulose and lignin removed from the treated chickpea stem.

X-ray diffraction analysis

X-ray diffractograms (XRD) is commonly used to determine the crystallinity index of materials. The X-ray diffractograms of the treated and raw chickpea samples are shown in Figure 3. Both diffractograms show two well-defined peaks at 2θ = 15° (I_{110}) arising from the amorphous and 22° (I_{002}) arising from the crystalline region respectively. The raw chickpea sample is oriented within the amorphous materials; therefore it gives a narrow and less sharp peak in I_{002} planes. After alkali and bleaching treatment, the amorphous materials dissolved, leaving pure crystalline cellulose. As can be seen in figure 3-a, the XRD patterns of treated chickpea stem sample give a sharper diffraction peak, which indicates a higher crystallinity degree (Sabiha-HanimandAziatul-Akma, 2016). The calculated crystallinity indices of raw and treated chickpea samples found to be 42% and 53%, respectively. Thus, the X-ray diffractograms demonstrate that different pretreatments took place preferentially in the amorphous region and the rearrangement of the crystalline region in such a way that the treated chickpea sample exhibited in more crystalline nature.

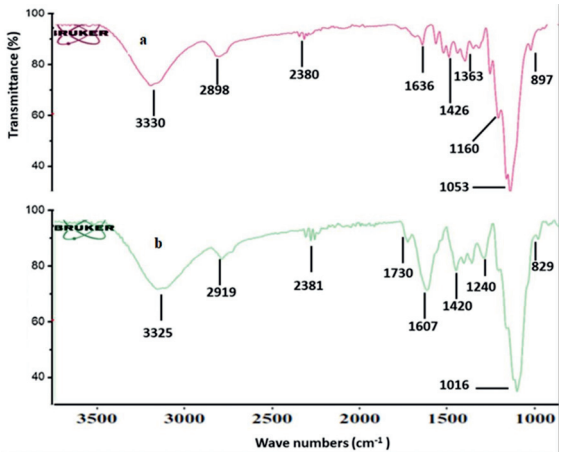

Fig. 2. FT-IR spectra of (a) treated chickpea sample (b) raw chickpea sample

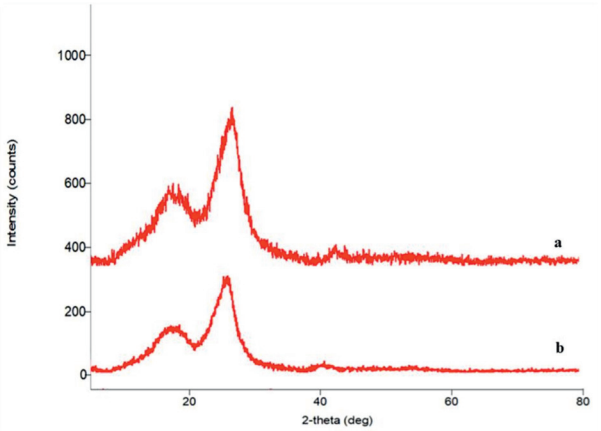

Fig. 3. X-ray diffraction patterns of (a) treated chickpea sample (b) raw chickpea sample

Differential scanning calorimetry (DSC) analysis

The DSC thermograms of the treated and raw chickpea samples are shown in Figure 4. In both the thermograms, the first endothermic peak was observed due to the evaporation of water from 30 °C to 125 °C (Kalpana and Perarasu, 2020). The raw chickpea stem has a more amorphous characteristic, therefore absorbs more water and shows a sharp endothermic peak (Figure 4-b). The above phenomenon is rarely observed with crystalline cellulose,

which shows no or minimal peak lines in the treated sample (Figure 4-a).

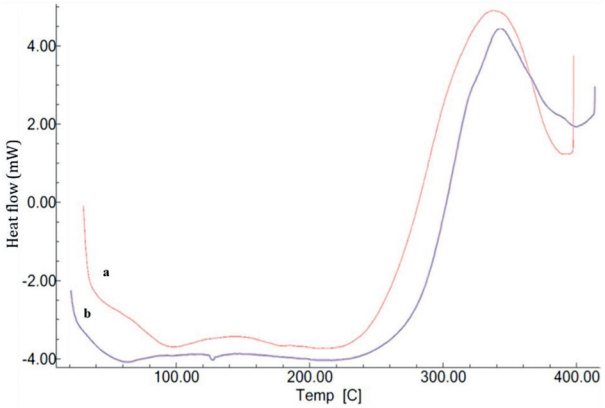

Fig. 4. DSC thermographs of (a) treated chickpea sample (b) raw chickpea sample

Yang *et al.*, (2007) reports that thermal decomposition regions of cellulose are in the range of 315 °C-400 °C. The melting point observed in the second endotherm in both samples describes the nature of the decomposition of the crystallites. The treated chickpea stem has less amorphous character compared to the raw chickpea stem, which is responsible for the higher degradation temperature of treated chickpea stem from 337 °C to 369 °C. The non-cellulosic constituents in the raw chickpea stem make it less stable whereby decreases the melting temperature in the range of 332 °C-359 °C. DSC studies indicate that treated chickpea stem was found to exhibit at high temperatures than the raw chickpea sample.

Scanning electron microscopy (SEM) analysis

The SEM micrographs of the treated and raw chickpea samples are illustrated in Figure 5. The morphological structure of the treated chickpea sample (Figure 5-a) shows more roughness and a clean surface. Further, it shows epidermal cells broken down at the top of the surface resulting in long fibers and marginal brightness in this region. It can be attributed mainly to the elimination of non-cellulosic constituents (Kathirselvam *et al.*, 2019). The raw chickpea sample has a relatively smooth surface (Figure 5-b), with a compact structure. It indicates the presence of lignin, hemicellulose, and other amorphous materials (Jayaramudu *et al.*, 2010).

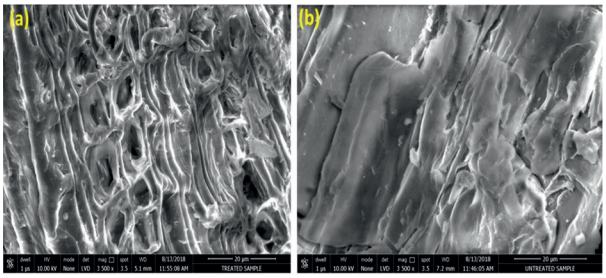

Fig. 5. SEM micrographs of (a) treated chickpea sample (b) raw chickpea sample

Physical properties of handmade paper

The physical properties of the handmade paper produced from chickpea cellulose pulp are compared with those of other lignocellulosic biomass summarized in Table 2. The pulp yield from chickpea cellulose is higher than other lignocellulosic biomass, except cotton stalks. The brightness of the prepared paper is also quite good. The tensile index and burst index values are in the range of TAPPI standards. These indicate paper produced from chickpea cellulose pulp is suitable for wrapping and packaging applications (Ibrahim *et al.*, 2011).

Table 2. Comparison of physical properties of handmade paper sheet of chickpea stem with other lignocellulosic biomass

Fibers	Yield %	Brightness %	Tensile index, Nm/g	Tear index, mN m^2/g	Burst index, kPa m^2/g	Folding endurance	Reference
Bagasse	43.00	42.2	6.50	0.30	1.20	–	(Ibrahim *et al.*, 2011)
Cotton stalks	57.24	26.15	2.34	0.26	2.13	–	(Jiménez *et al.*, 2007)
Wheat straw	42.6	–	–	4.23	4.10	–	(Montane *et al.*, 1998)
Sunflower stalks	38-48	–	43-50.9	3.4-3.7	3.7-6.2	–	(Lopez *et al.*, 2005)
Sorghum stalks	31.7	33.5	12.85	6.25	8.05	560	(Jiménez *et al.*, 1993)
Rice straw	42.82	46.5	2.36	1.45	0.33	–	(Rodríguez *et al.*, 2008)
Chickpea stem	43.46	39	26.02	3.70	3.17	170	Present study

Conclusion

The extraction of cellulose from chickpea stem by effective processes like de-waxing, alkali, and bleaching treatment allows the lignocellulosic biomass into polymeric fractions with reasonable yield (49.23%) and purity. The FTIR spectroscopic revealed variation in functional groups after treatment indicates successful removal of wax, lignin, and hemicellulose functionalities. X-ray diffraction analysis specifies an increase in the crystallinity of the extracted cellulose after treatments from 42% to 53%. Based on the DSC analysis, a treated chickpea sample was found to exhibit better thermal stability than the raw chickpea sample. The SEM micrograph of treated chickpea sample shows a reduction in fiber size, and the rough surface, it can increase the chances of strong bonding in the pulp preparation when guar gum and cationic starch was added. The pulp recovery from extracted cellulose is also quite good (43.46%). The physical properties of handmade paper such as brightness, tensile, bursting, and tear indexes, and double-fold strength are also quite good. Therefore, Chickpea stem waste could be considered an interesting and promising alternative source for low-cost papermaking applications.

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