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Extraction and structural characterization of cellulose from milkweed floss

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ABSTRACT

The aim of this study was to investigate the utilization of milkweed fruit floss residues as a source for the isolation of cellulose. Cellulose was extracted by acidified sodium chlorite and sodium hydroxide treatments. Characterization of the pristine milkweed floss and extracted cellulose was performed by chemical composition analysis, Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), X-ray diffraction (XRD), and thermogravimetric analysis (TGA). The extracted cellulose had mainly α-cellulose as the other components hemicellulose and lignin were significantly removed during cellulose extraction process. The FTIR spectra also indicated that the chemical treatments extensively removed hemicellulose and lignin from the pristine milkweed floss. SEM technique was used to investigate the surface morphology of the pristine milkweed floss and extracted cellulose. The intensity of the crystalline peak in the X-ray diffractograms of the extracted cellulose was higher than that of pristine milkweed. Further, the XRD results indicated a structural transformation of cellulose I (pristine milkweed) to cellulose II (extracted cellulose) because of the chemical treatments. The extracted cellulose, which is a high biomass, had better thermal stability than the pristine milkweed floss owing to removal of non-cellulosic components.

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Cellulose; crystallinity; extraction; milkweed floss; spectroscopy; thermal stability

Introduction

In recent decades, the emergent recognition of the stipulation to conserve the environment has resulted in an augmented use of environmentally friendly materials and renewable resources for diverse applications.-[1,2] Cellulose is one of the most abundant biopolymers originating in nature, mostly as the main structural component of woody plant cell walls, and is a linear polymer consisting of β-(1–4) glucopyranose unit.[3] Cellulose has fascinated attention due to remarkable physical properties, biodegradability, biocompatibility, chemical reactivity and low cytotoxicity.[3] It created diverse applications such as paper technology, composite materials, textiles, food, drug delivery systems, and cosmetics or pharmaceutical products.[4–8] These days, cellulose research is receiving additional importance in developing novel materials based on the application of knowledge in nanotechnology.[9–12] Cellulose is thus bound to play a key role in the technological changes vault to take place in the way of this century and future.

Though wood is obviously the most important industrial source of cellulose, recent scientific interest in other sources such as agricultural residues and forestry waste is increasing.[13] In vision of environmental protection and ecological economics, the utilization of non-wood plants biomass is of great interest.[14–16] In addition, challenging, reusing and recycling waste can diminish environmental problems associated with its accumulation. In this connection, the authors attempted to explore the possibility of using milkweed floss as a new source of cellulose isolation. *Calotropis gigantea* (Fig. 1a), belonging to the asclepiadaceous family, is a high biomass, fast-growing perennial shrub growing as a weed in China, India and Malaysia and has been found in most of Asia and large parts of Africa and South America.[17] It is commonly known as Indian milkweed and has been claimed in traditional literature to be precious against a wide variety of diseases.[18] Further, recently milkweed plants parts such as bark fiber, floss, seeds, and so on are used for various applications.[19–22]

However, the short lengths and low elongation limit the use of milkweed pods floss in other technical applications.[23] A massive quantity of milkweed plant pods containing cellulosic fibers is generated

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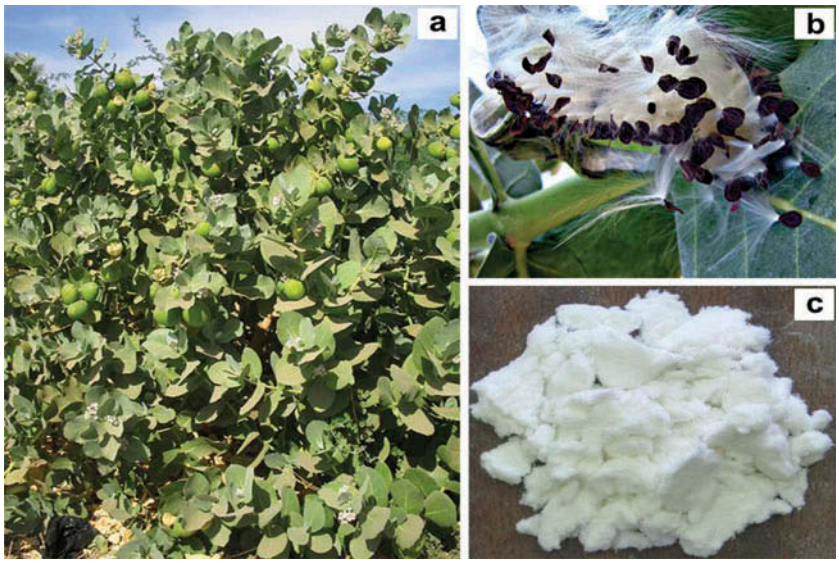

Figure 1. Digital image of (a) Calotropis plant, (b) floss with seeds, and (c) extracted cellulose.

annually as natural waste and that can form an alternative raw material for cellulose extraction. To the best of our knowledge, there are no reports available on a detailed investigation on the extraction of cellulose from milkweed floss. The main goal of this study was to evaluate the isolation potential of cellulose from milkweed followed by acidified sodium chlorite and alkaline treatment process. The milkweed floss and extracted cellulose were characterized by chemical composition analysis, Fourier transform infrared (FTIR) spectroscopy, wide angle X-ray diffraction(WAXD), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM) in order to investigate its potential value-added utilization as a new source of cellulose fiber based materials.

Experimental

Materials

Acetic acid, ethanol, sodium bisulphite, sodium hydroxide pellets (Merck Chemicals), nitric acid, sodium chlorite, and toluene (Sd-Fine Chemicals) chemicals used were of analytical grade. The matured pods collected from milkweed plant were kept at room temperature for 24 h, and then the pods were opened manually, and the fluffy white floss and brown seeds were separated (Fig. 1b). The separated floss was cleaned with tap water to remove the unwanted materials from it. Finally, the extracted fibers were dried at room temperature for a week and stored in polyethylene bags.

Cellulose extraction

Extracted floss was chopped and then dewaxed by refluxing with toluene–ethanol (2:1, v/v) for 6 h in a Soxhlet apparatus. The dewaxed floss sample was then delignified with 0.7% sodium chlorite at 100°C for 2 h in acidic solution (pH 4–4.2 adjusted by 10% acetic acid) using a fiber to liquor ratio of 1:50. After being filtered and extensively washed with 2% sodium bisulphite and distilled water, the residue was dried at 105°C in an oven until constant weight. The residue was treated with 17.5% (w/v) NaOH solution at 20°C for 45 min, filtered, and washed with 10% acetic acid, distilled water, and sequentially with 95% ethanol to remove excess of acid. Finally, the purified extracted cellulose was dried at 105°C in an oven until constant weight. The extracted cellulose is shown in Fig. 1c. In this process, the final yield was calculated as percentage of initial weight of the used raw (pristine milkweed floss) material.

Chemical analysis

The chemical composition of milkweed floss and extracted cellulose was determined using the standard Technical Association of the Pulp and Paper Associations (TAPPI) methods for different components, namely: T 204 cm-07 for extractives, T 203 cm-99 for α-cellulose, T 222 om-06 for lignin, and T 211 om-07 for ash content. The holocellulose was determined according to the method described by Wise et al.[24] The hemicellulose fraction was calculated as the difference between the holocellulose and α-cellulose content. The percent contents of extractives, α-

cellulose, hemicellulose, lignin, and ash were determined, and based on five samples, the average and standard deviation values are reported.

Morphology

Morphology of the pristine milkweed floss and extracted cellulose was examined using a scanning electron microscope (JEOL, model JSM 820); an acceleration voltage was fixed at 5 kV. All the test samples were gold-coated using a vacuum sputter coater (model SC 500) prior to analysis.

FTIR spectroscopy

Possible changes in functional group were monitored with FTIR spectroscopy. Of each sample, 5 mg was pulverized and introduced into a hollow chamber and pressed to make a small pellet. The pellet was run on a Smart iTR ATR Nicolet iS 10 FT-IR spectrophotometer that has an attachment to record the spectra in transmission mode also. Background scanning and correction were performed before testing the samples. All the spectra were recorded in the 4000–500 cm^{-1} region with 32 scans in each case, at a resolution of 4 cm^{-1}. All spectra were smoothed and fitted to an automatic base line correction and normalized by Thermo Nicolet's OMNIC® Software. The background was subtracted from the spectra automatically by the software each time.

X-ray diffraction analysis

Wide-angle X-ray diffractograms of milkweed floss and extracted cellulose were recorded on a RigakuUltima IV X-ray diffractometer. The system has a rotating anode generator with a copper target and a wide angle powder goniometer. The generator was operated at 40 kV and 30 mA with Cu Kα (λ = 1.5406Å), and the samples were scanned in the 2θrange of 5–30°.

The crystal size of cellulose I and II samples, t(nm), was determined perpendicular to the (200) planes by the Scherrer Eq. (1):

$$t = \frac{K\lambda}{\beta cos\theta} \quad (1)$$

where K = 1.0 is the shape correction factor, λ is the X-ray wavelength, and β is the line broadening (in radians) at the half maximum intensity after subtracting the instrumental broadening.

Thermogravimetric analysis

The thermal stability of milkweed floss and extracted cellulose was established using a thermogravimetric analyzer (Perkin Elmer TGA-7). The amount of sample for each measurement was about 10 mg. All the measurements were performed under a nitrogen atmosphere with a gas flow of 100 mL/min and heated from 50 to 700°C at a rate of 10°C/min.

Results and discussion

The leading work concerned in the current research was to extract cellulose from matured milkweed pods and characterize it. The yield of the extracted cellulose from milkweed floss was found to be 41.6%. The chemical analysis results indicated that milkweed floss contained 10.6 ± 0.8% of extractives, 49.2 ± 1.4% of α-cellulose, 20.1 ± 1.3% of hemicellulose, 16.5 ± 1.0% of lignin and other amorphous impurities, and 3.5 ± 0.3% of ash. The extracted cellulose contained extractives, α-cellulose, hemicellulose, lignin, and ash contents in 2.2 ± 0.2, 91.1 ± 0.9, 2.2 ± 0.4, 3.1 ± 0.3, and 1.3 ± 0.5 percentage, respectively. From the chemical analysis, it is evident that the α-cellulose content increased while the extractives, hemicellulose, lignin, and ash content decreased in the case of extracted cellulose when compared to the pristine milkweed floss. The chemical analysis results proved that during the sodium chlorite and sodium hydroxide treatments, considerable breakdown of the lignocellulosic structure took place in the form of hydrolysis of hemicellulose fraction and depolymerization of lignin. Figure 2 reveals the morphological changes between the pristine milkweed floss and extracted cellulose. The surface of pristine milkweed floss was found to be smooth, with cylindrical shape (Fig. 2a), while the extracted cellulose indicates rough and non-uniform surfaces. The morphological changes in the fiber surface were attributed to the removal of the hemicelluloses and lignin by chemical treatments. Hence, a rough surface of fibers leads adhesion in a fiber-polymeric matrix interface in the production of composites as well as in the papermaking process.

FTIR spectra of pristine milkweed floss, dewaxed milkweed floss, delignified milkweed floss, and extracted cellulose are shown in Fig. 3. From Fig. 3, it is evident that the spectra had well-defined common bands at 3401, 2916, 2850, 1372, 1316, 1158, 1031, and 897 cm^{-1} corresponding to cellulose.[25–30] Further, an additional common band at around 1626 $cm^{-1}$was found in all the three cases that was attributed to the bending mode of the absorbed water. However, in the case of pristine milkweed floss and dewaxed milkweed

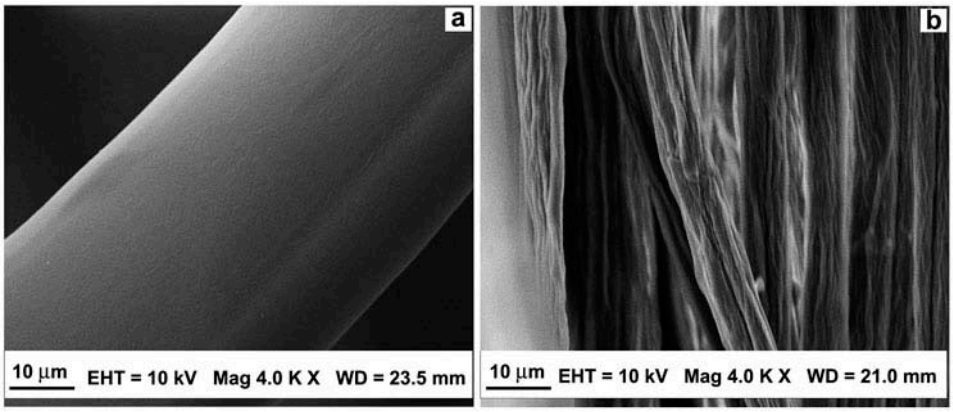

Figure 2. Scanning electron micrographs of (a) pristine milkweed floss and (b) extracted cellulose.

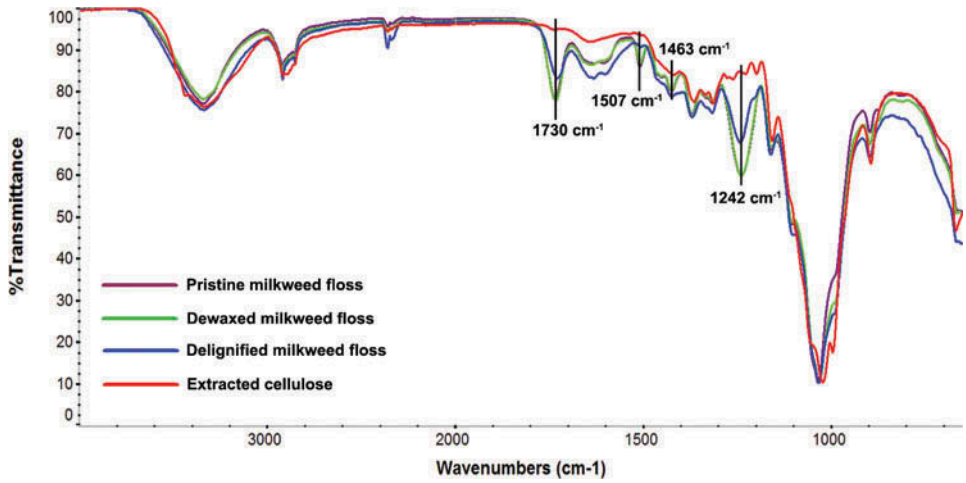

Figure 3. FTIR spectra of pristine milkweed floss, dewaxed milkweed floss, delignified milkweed floss, and extracted cellulose.

floss, additional bands at around 1730, 1507, 1463, and 1242 cm^{-1} were also present that were attributed to hemicellulose and lignin.[26,28] The intensity of these bands decreased in the delignified milkweed floss due to the removal of lignin by acid chlorite treatment. However, in the extracted cellulose, intensity of these bands significantly decreased, and this clearly indicated the maximum removal of hemicellulose and lignin during the alkali treatment by cellulose extraction process. This FTIR analysis results suggests that there was removal of significant amount of hemicellulose and lignin during the cellulose extraction process.

XRD studies of the milkweed floss and extracted cellulose were performed to investigate the crystalline behavior of both the samples, and the corresponding X-ray diffractograms are shown in Fig. 4. The X-ray diffraction curve of pristine milkweed floss shows an intense peak at 2θ = 22.4°, and the broadened shoulder peak at 2θ = 15.9° that can be assigned to (200) and a mixture of (1–10) and (110) reflections, respectively,

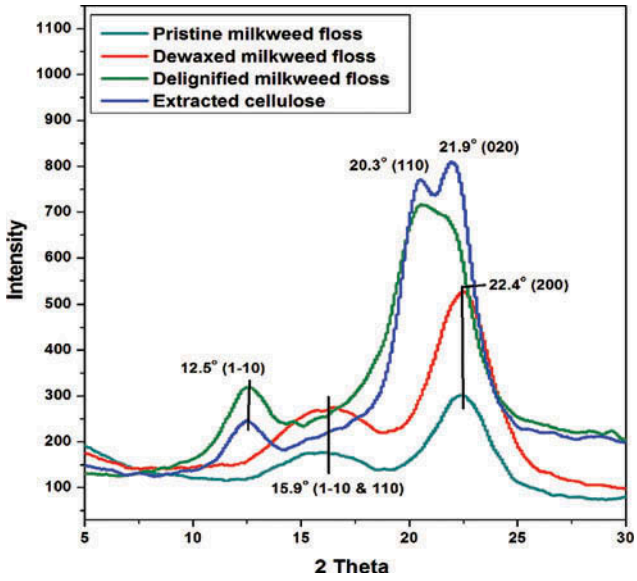

Figure 4. X-Ray diffractograms of pristine milkweed floss, dewaxed milkweed floss, delignified milkweed floss, and extracted cellulose.

which are representing a typical cellulose I crystal structure.[27] Dewaxed milkweed floss diffractograms were similar to pristine milkweed floss, while the peaks intensity increased due to removal of surface impurities by dewaxing process. For delignified milkweed floss and extracted cellulose, the peak at 2θ = 15.9° disappeared, and a new peak appeared at 2θ = 12.5°. The main diffraction peaks located at 2θ = 12.5° (1–10), 20.3° (110), and 21.9° (020) resulted from a cellulose II crystal structure.[31] For extracted cellulose, the peak at 2θ = 15.9° disappeared, and a new peak appeared at 2θ = 12.5°. The main diffraction peaks located at 2θ = 12.5° (1–10), 20.3° (110), and 21.9° (020) resulted from a cellulose II crystal structure.[31] This indicates that the crystal structure of cellulose I has been changed to Cellulose II during the cellulose extraction process using sodium chlorite and sodium hydroxide chemical treatments. This may be due to the strong oxidizing properties of sodium chlorite that could partly destroy intermolecular or/and intramolecular hydrogen bond network at the time of removing lignin and then the strong alkaline solution treatment removing hemicellulose leading to the rearrangement of cellulose chains. The crystallite diameter obtained from X-ray diffractograms using Eq. (1) for pristine milkweed floss was found to be 3.35 nm while for extracted cellulose 5.42 nm. A higher crystallite diameter of the extracted cellulose (cellulose II crystal structure) indicates agglomeration of the chains or arrangement of strong intermolecular hydrogen bond occurred during the cellulose extraction process from pristine milkweed floss (cellulose I crystal structure).

The primary thermogravimetric (PTG) and its corresponding derivative thermogravimetric (DTG) analyses of pristine milkweed floss and extracted cellulose were carried to analyze their thermal stability (Fig. 5a and b). Both pristine milkweed floss and extracted cellulose PTG thermograms show a minor weight loss in the range 50°C–150°C that was attributed to evaporation of absorbed moisture in the samples[32], and the mass loss approximately in this region was about 4.1% for pristine milkweed floss and 1.5% for extracted cellulose. Decomposition of the pristine milkweed floss occurred in several stages, indicating the presence of different components that decompose at different temperatures. For the pristine milkweed floss, the PTG and DTG curves showed two main decomposition steps. The first weight loss stage in the range of 200–320°C was attributed to the hemicelluloses or pectin decomposition.-[32] The second weight loss stage in the temperature range of 320–430°C was attributed to cellulose decomposition. Figure 5b demonstrates that the DTG curve of pristine milkweed floss is broad and seems to consist of two peaks around 260°C and 375°C, which were assigned to the degradation of hemicellulose and cellulose.[33] Besides, lignin was the most difficult to decompose among the three components. Its decomposition occurred slowly under the whole temperature range from 200 to 700°C, and absence of its characteristic peak is due to the wide range of temperatures.[32] Further, for the extracted cellulose, only single-step degradation occurred in the region 275–460°C, and the corresponding DTG peak was observed at around 395°C accounted for the pyrolysis of cellulose. However, the decomposition temperature of extracted cellulose is found to be higher than for pristine milkweed floss, and there is no shoulder around 260°C indicating the removal of hemicellulose portions. The chemical treatments attack the non-cellulosic components and amorphous region in the cellulose and increase the degree of crystallinity. Higher crystallinity values resulted in greater resistance toward heat and increase in the maximum temperature for thermal degradation. These results are very consistent with results obtained from chemical, FTIR spectral, and X-RD analyses.

Conclusions

Based on the conclusions in this work, cellulose was successfully extracted from milkweed floss by chemical treatments such as sodium chlorite and alkali. Chemical

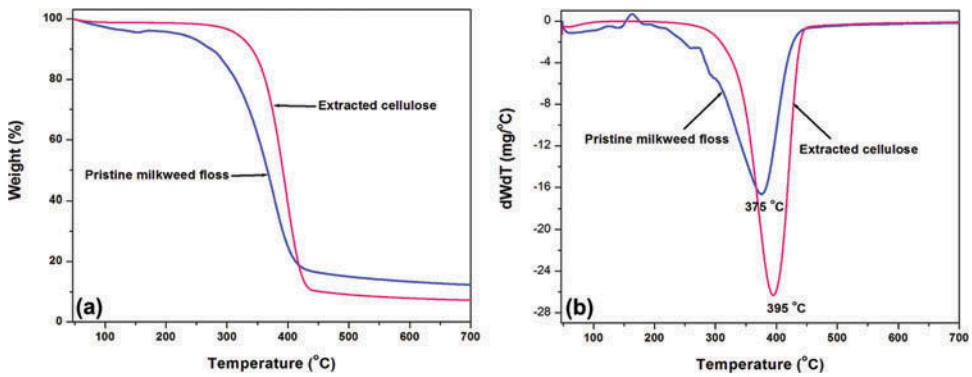

Figure 5. Primary thermograms (a) and derivative thermograms (b) of pristine milkweed floss and extracted cellulose.

and FTIR spectral analyses on the milkweed floss revealed the significant amount of hemicellulose and lignin removal during the cellulose extraction process. XRD results indicated an increase of crystallinity and polymorphic transformation (cellulose I to cellulose II) of the milkweed floss on chemical process. TGA results revealed that this extracted cellulose exhibited a higher thermal stability than the original milkweed floss. This work demonstrated that the milkweed floss under study can be potentially used as raw material for the production of cellulose. Also, the characterization results validate the promising usage of milkweed floss for various industrial applications such as pulp and paper, bio-fuel, packaging, composites, pharmaceuticals, and so on.

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Declaration of interest

The authors declare no conflict of interest.

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