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Extraction of Cellulose from Calotropis procera Plant and Characterization Using FOURIER TRANSFORM INFRA-RED (FT-IR)

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Abstract

The increasing demand for sustainable and environmentally friendly materials has intensified interest in biopolymers derived from renewable resources. In this study, cellulose was successfully extracted from the stems of Calotropis procera, an abundant and underutilized Lignocellulosic plant, using chemical treatment methods. The extraction process involved dewaxing with a toluene–ethanol mixture, followed by alkaline delignification using hydrogen peroxide and subsequent purification with acetic acid and nitric acid. The extracted cellulose yield was determined to be approximately 42% of the total stem weight. Fourier Transform Infrared Spectroscopy (FT-IR) was employed to characterize both untreated Calotropis procera stems and the extracted cellulose. The (FT-IR) spectra of the raw stems showed characteristic absorption bands corresponding to cellulose, hemicellulose, and lignin at (3415.31 cm-1, 1716.34cm-1 and 1536.99cm-1) respectively. In contrast, the extracted cellulose exhibited typical cellulose functional groups, including O–H stretching at (3419.17cm-1), C–H stretching at (2921.63 cm-1), C–O–C vibrations (1031.73 cm-1), and β-glycosidic linkages at (896.737cm-1), with the absence of peaks associated with lignin and hemicellulose. These results confirm the effective removal of non-cellulosic components and the efficiency of the applied extraction method. The findings suggest that Calotropis procera stems represent a promising and sustainable alternative source of cellulose for potential industrial and biomedical application. This study recommended that, comparative studies between cellulose extracted from Calotropis procera and conventional sources, conversion of cellulose into cellulose derivatives such as cellulose acetate or carboxymethyl cellulose and evaluation the *feasibility of large-scale production, considering the plant's abundance and low agricultural requirements in arid and semi-arid regions.*

Keywords: Calotropis procera, Cellulose extraction, Alkali treatment, FOURIER TRANSFORM INFRA-RED (FT-IR) characterization, Natural fibers

1. Introduction

Sound environmental management and wise usage of natural renewable resources for the production of industrial and consumer materials and energy are important themes since 1990s (Wang and Tao, 1995).

The rise of biopolymers in advanced applications is inevitable due to the environmental and health hazards posed by fossil-based polymers, which exacerbate global energy issues through high energy consumption during production and the emission of greenhouse gases. In response to this problem, biopolymers are now addressing industrial needs by offering sustainable solutions to replace fossil-based polymers. Concurrently, the demand for biopolymers is increasing in biomedical fields such as tissue engineering, wound healing, and drug delivery, owing to their numerous advantages over fossil-based materials (Salleh, 2025)

Cellulose is an unbranched, natural polymer composed of repeating glucose units $(C_6 H_{10} O_5)n$. It is the most abundant organic material and polysaccharide on Earth, contributing approximately 1.5 × 1012 tons to the total annual biomass production (Widjaya, 2018). This biodegradable polymer is primarily found as micro fibrils within the cell walls of wood, plants, algal tissues, and the membranes of tunicate epidermal cells. Furthermore, it is synthesized by bacteria such as Acetobacter xylinum (Xu, 2020). Over the last decades, the use of cellulosic fibers has intensified, which increased the market demand of such raw material. Thus, to satisfy the large demand of natural fibers, it becomes challenging to supply the needs of all the users at reasonable costs, with required quantities and qualities (Mansouri, 2012)

The reutilization of natural organic residues to obtain an applicable product is a very useful practice that has many advantages. There has been an increasing trend towards more efficient utilization of agro-industrial residues, such as Calotropis Procera wood, sugarcane bagasse, wheat straw and rise straw as raw materials for industrial applications (Silva et al., 2012).

Lignocellulosic biomass including Calotropis Procera wood is the most abundant organic material in the world, and it has the potential to be a very promising alternative source of fuels and chemicals (Maitan et al., 2015). About 40 – 50% of wood is the glucose polymer cellulose, much of which is in a crystalline structure. Another 25 – 35% is hemicelluloses, an amorphous polymer usually composed of xylose, arabinose, galactose, glucose, and mannose. The remainder is mostly lignin (18– 24%) plus lesser amounts of mineral (1– 4%), wax (<1%), and other compounds (Sun et al.,2004) (Wirawan et al., 2012). Table (1) shows Composition of cellulose in different sources.

Table (1): Composition of cellulose in different sources (Heinze et al., 2006)

Composition of Cellulose %	Sources
40 -50	Bagasse
32 – 43	Coir (fiber from the outer husk of the coconut)
45	Corn cobs
35	Corn stalks
95	Cotton
71	Flax (rettened)
63	Flax (unrettened)
43 – 47	Hardwood
70	Hemp
78	Henequen (fiber used for twine and paper pulp).
36	Kenaf
76	Ramie
73	Sisal
40 – 44	Softwood
30	Wheat and rice straw

Cellulose derivatives are modified forms of cellulose obtained through chemical reactions that introduce functional groups into the cellulose backbone. These modifications alter the native properties of cellulose, such as solubility and reactivity, enabling its use in a wide range of industrial, pharmaceutical, and biomedical applications.

Common Types of Cellulose Derivatives: (Shokri, and Adibkia., 2013), (Doelker., 2005)

1. Cellulose Ethers:

Carboxymethyl cellulose (CMC) is water-soluble; used as a thickener, stabilizer, and binder in food, pharmaceuticals, and cosmetics.

Methyl cellulose (MC): Exhibits thermal gelation; used in food products and controlled drug release.

Hydroxyethyl cellulose (HEC): Used in coatings, paints, and personal care products.

Hydroxypropyl methylcellulose (HPMC): Widely used in pharmaceutical formulations and film coatings.

2. Cellulose Esters

Cellulose acetate (CA): Used in fibers, membranes, photographic films, and biodegradable plastics.

Cellulose nitrate (CN): Applied in lacquers, inks, and propellants (historically).

Cellulose sulfate: Investigated for anticoagulant and biomedical applications.

Methods of Preparation of cellulose derivatives:

Cellulose derivatives are typically synthesized via Etherification (e.g., reaction with alkyl halides or epoxides).

Esterification (e.g., reaction with organic or inorganic acids) These reactions are usually carried out in alkaline media to activate cellulose hydroxyl groups.

Cellulose esters are widely produced as commercial products. Typical products of cellulose acetate are films, fibers, particle filters and reverse osmosis membranes. (Yuan et al., 2005).

Calotropis procera is a perennial shrub commonly found in arid and semi-arid regions. It grows rapidly, requires minimal agricultural input, and contains a high proportion of fibrous material. Despite its abundance, it remains largely underutilized.

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leaves opposite, grey-green, large up to 15 cm long and 10 cm broad, with a pointed tip, two rounded basal lobes and no leaf stalk; flowers waxy white, petals 5, purple-tipped inside and with a central purplish crown, carried in stalked clusters at the ends of the branches; fruit grey-green, inflated, 8 to 12 cm long, containing numerous seeds with tufts of long silky hairs at one end. (Kaur, et al., 2021)

Calotropis procera is a species of flowering plant in the family Apocynaceae that is native to North Africa, tropical Africa, Western Asia, South Asia, and Indochina. The green fruits contain a toxic milky sap that is extremely bitter and turns into a gluey coating which is resistant to soap. (Hassan, et al., 2015)

The present study aims to extract cellulose from Calotropis procera stems using chemical methods and to characterize the extracted cellulose using Fourier Transform Infrared Spectroscopy (FT-IR), with the objective of evaluating its suitability as a sustainable cellulose source.

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Fig (1-A) *Calotropis procera*

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Fig (1-B) *Calotropis procera* Plant Fruit

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Fig (1-C) *Calotropis procera* Plant Flowers Fig (1-D) *Calotropis procera* Plant

Materials and Methods

Materials:

The sample:

The sample of this study was Calotropis procera plant stems was collected from deferent areas of Rufaa town, Gezira state, Sudan.

Reagents and Solvents:

The solvents were used in this study are:

Toluene obtained from Lap Tech-chemicals, ethanol absolute from CARLO.ERBA Milan, Italy; acetic acid and 70% nitric acid obtained from Lap Tech-chemicals. Hydrogen peroxide, obtained from Oxford Lab Chem.

Instruments:

Fourier transform Infra-red (FOURIER TRANSFORM INFRA-RED (FT-IR)) spectra were performed for determination of the structures of cellulose and cellulose esters using Fourier Transform Infra-Red (FT-IR) instrument, Model Name FT-IR-4600 type A- Serial Number C007261786 - Measurement Date 21/05/2017 - Company Spectrum Research Systems Co. at National Research Center, Cairo, Egypt.

Methods:

Sampling:

The collected Calotropis procera plant stems was washed thoroughly with distilled water to remove dirt and impurities, then air-dried for several days. The dried material was cut into small pieces and ground into powder using a mechanical grinder.

Dewaxing of Calotropis procera plant stems:

A fifty grams of dried powder of Calotropis procera plant stems was extracted with toluene/ethanol (2:1, V/V) using a Soxhlet apparatus for 6 hrs. then, it was dried in an oven at 60 °C for 16 hrs.

Delignification and purification of Calotropis procera plant stems:

The method of delignification of Calotropis procera plant stems described by (Sun, 2010) was followed:

The sample of dewaxed Calotropis procera plant stems was treated with 2.0% H2O2 at pH of 11.8 (adjusted by 5M NaOH) at 48oC for 16 hrs. with stirring. After filtration, the insoluble residue (delignified cellulose) was purified with a mixture of 80% acetic acid and 70% nitric acid (1:1, V/V) at 100oC for 20 min, then it was washed with 95% ethanol, and distilled water until neutralization (detected by litmus red paper). Delignified, purified cellulose was dried in an oven at 60oC for 16 hrs.

Calculation of Cellulose Extraction Yield:

The extracted cellulose yield was determined bay weight /weight percentage from the total weight of the stems. The weight of obtained cellulose was (20.8 g).

Fourier Transform Infra-Red (FT-IR) Characterization:

The FT-IR analysis was carried out using an FT-IR spectrophotometer in the range of **4000–400 cm⁻¹**. Crowd stems of Calotropis procera plant sample (A) and the extracted cellulose sample (B) was prepared using the KBr pellet method. The obtained spectra were analyzed to identify functional groups and to confirm the removal of lignin and hemicellulose.

Results and Discussion

Visual and Physical Observation:

After alkali and bleaching treatments for the sample (B), the fiber color changed from brownish to white, indicating effective removal of lignin and other non-cellulosic components.

The percentage of Cellulose in The Sample:

The percentage of cellulose in Calotropis procera stems sample was approximately (42%) calculated from the total weight of the stems (50 g).

Fourier Transform Infra-Red (FT-IR) Analysis:

Crude Calotropis procera stems:

The Fourier Transform Infra-Red (FT-IR) spectrum of crude Calotropis procera stems (sample (A)) is shown in table (2) and figure (2) which exhibited absorbance at 3415.31, 2923.56, 1643.05, 1384.64, 1043.3, 1043.3, 1716.34 and 1536.99cm-1.

The absorption at 3415.31cm-1 is due to stretching of the hydroxyl group (OH). The peak at 2923.56cm-1 corresponds to C-H stretching. The band at 1643.05 cm-1 is attributed to the bending mode of absorbed water. The intensity of the peak at 1043.3cm-1 is related to (C-O-C) stretching, the peak at1716.34cm-1 is attributed to the bending of hemicellulose and the intensity of the peak at 1536.99cm-1 is related to lignin aromatic rings.

Extracted Cellulose from Calotropis procera stems:

The Fourier Transform Infra-Red (FT-IR) spectrum of Extracted Cellulose from Calotropis procera stems (sample (B)) is shown in table (3) and figure (3) which exhibited absorbance at deferent peaks as follows:

The absorption at 3419.17cm-1 is due to stretching of the hydroxyl group (OH). The peak at 2921.63 cm-1 corresponds to C-H stretching. The band at 1650.77cm-1 is attributed to the bending mode of absorbed water. The intensity of the peak at 1031.73cm-1 is related to (C-O-C) stretching and the characteristic band at 896.737 cm^{-1} corresponding to β-glycosidic linkages, confirming the cellulose structure.

The absence of peaks around 1730 cm^{-1} (hemicellulose) and 1510 cm^{-1} (lignin aromatic rings) confirmed successful delignification and purification of the Calotropis procera stems.

Table (2): Characteristic Fourier Transform Infra-Red (FT-IR) Absorption Frequencies of Crude **Calotropis procera stems** Functional Groups

Types of Vibration	Functional group	Intensity	The peak	
stretching	O–H	43.3881	3415.31	
stretching	C–H	54.5948	2923.56	
stretching	C–O–C	70.7145	1043.3	
Bending	Absorbed H2O	65.1175	1643.05	
Bending		hemicellulose	61.7454	1716.34
Bending		lignin aromatic rings	77.672	1536.99

Fig (2): Characteristic Fourier Transform Infra-Red (FT-IR) Absorption Frequencies of Crude **Calotropis procera stems** Functional Groups

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Table (3): Characteristics of Fourier Transform Infra-Red (FT-IR) Absorption Frequencies and Extracted Cellulose Functional Groups:

Types of Vibration	Functional group	Intensity	The peak
stretching	O–H	38.3399	3419.17
stretching	C–H	55.2205	2921.63
stretching	C–O–C	58.8532	1031.73
Bending	Absorbed H2O	65.6649	1650.77
Bending	β-glycosidic linkages	83.7977	896.737

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Fig (3): Characteristic Fourier Transform Infra-Red (FT-IR) Absorption Frequencies of Extracted Cellulose Functional Groups

Figure (4) illustrates a comparison between the peaks of untreated Calotropis procera stems and the cellulose extracted from it. This shows a significant difference between the two curves, indicating the effectiveness of the method adopted for the extraction and isolation of cellulose.

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Fig (4): Comparison between crude stems and extracted cellulose Fourier Transform Infra-Red (FT-IR)- curves.

Conclusion:

This study concluded that, Fourier Transform Infra-Red (FT-IR) analysis confirmed the removal of lignin and hemicellulose and verified the presence of characteristic cellulose functional groups

Recommendations:

Further characterization of the extracted cellulose is recommended using additional analytical techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA) to evaluate its crystallinity, surface morphology, and thermal stability.

Optimization of the extraction parameters, including reagent concentrations, reaction time, and temperature, is suggested to improve cellulose yield and purity.

Comparative studies between cellulose extracted from Calotropis procera and other conventional cellulose sources should be conducted.

The conversion of the extracted cellulose into cellulose derivatives (such as cellulose acetate or Carboxymethyl cellulose) is recommended to explore its suitability for advanced industrial and pharmaceutical applications.

Investigation of the biocompatibility and biodegradability of the extracted cellulose is recommended, particularly for potential biomedical applications.

Scale-up studies are suggested to evaluate the practicality of large-scale cellulose production from Calotropis procera, especially considering its abundance and low agricultural requirements in arid and semi-arid regions.

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