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STUDIES ON THE CHARACTERIZATION OF ISOLATED CELLULOSE MATERIAL FROM HEMP MATERIAL

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ABSTRACT

The rapidly expanding fields of conducting polymer nanocomposites are generating many exciting new materials with novel properties. It is therefore, of immense significance to explore the performance of nanostructures of conducting polymers. Thus, synthesis of conducting polymer composites in order to provide the base for both research and applications Hence, the present investigation focused on conducting polymer cellulose nanocomposite which has high processability, high stability. The main scope of present work, it is proposed to characterization of cellulose from Hemp materials and analysed by various experimental methods. In recent days many research groups have a special attention to cellulose because it is an eco-friendly biomaterial product and it has ultrafine network structure and it has extraordinary properties like porosity, crystallinity, good thermal properties, and good mechanical properties etc., peculiar biodegradability and biocompatibility. Cellulose place a major role in contrives 1D hybrid nanomaterials in biomedical, biosensing, catalysis and electronic applications.

KEYWORDS: Isolated cellulose, hemp materials, FT-IR spectral studies, X-ray diffraction, Thermal analysis etc.

INTRODUCTION

This study is concerned with the extraction of cellulose from agricultural waste materials such as hemp. Cellulose, a polysaccharide composed of a linear chain of hundreds to thousands of (1, 4-linked D-glucose units), is the most common biopolymer, and it is a major component of plant cell walls [1]. The molecular formula for cellulose is $C_6H_{10}O_5$, and it is primarily found in wood, rice straw, cotton, and other plant materials [2].

The cell wall's external layer consists of 9-25% cellulose microfibrils, an interpenetrating matrix of 25-50% hemicellulose, and 10-35% pectin. Carbohydrates comprise approximately 90% of the cell wall, and they keep the cellulose fibers together [3].

The distribution of cellulose microfibrils in the main wall is random, and the inner layer of plant cell wall is made up of 40-80% cellulose, 10-40% hemicellulose, and 5-25% lignin, with cellulose microfibrils embedded in lignin. Cellulose and hemicellulose are two components that appear to be organized in the primary cell wall. Lignin is an important component of the secondary cell wall. The basic cell wall of green plants, algae, fungus, tunicates, and bacteria has less cellulose than the secondary cell wall. The intra and inter chain hydrogen bonding network permits cellulose fibrils to be stable and have a high axial stiffness. In certain regions, the cellulose chain is well structured, resulting in a crystalline form [4].

In other locations, the cellulose chains are disordered, resulting in an amorphous structure. Cellulose nano crystals are derived from the crystalline region of cellulose microfibrils.

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The cellulose morphology of a natural fiber cell is characterized by a well-organized architecture of fibrillar components, as well as a large distribution of crystalline regions (crystallites) throughout the materials.The main structural components of the plant cell wall are known as micro fibrils, and the linear connections between these crystallites appear as thread-like structures. Native cellulose microfibrils are thought to be the smallest, well-defined morphological unit. According to current electron microscopic studies, its breadth varies from 3 to 35 nm depending on the cellulose supply used.

The cellulose molecule's pyranose rings contain every one of the groups bigger than hydrogen bound from the rings' perimeter (equatorial locations). The stereo chemical structure of the molecule of glucose is stable at carbons two, three, four, and five; however, if glucose forms a pyranose ring, the hydroxyl at C-4 will approach the carbonyl at C-1 from either side, resulting in two distinct stereochemistries at C-1. The hydroxyl group at C-1 is deemed to be inside the configuration when it is on the same side of the ring as the C-6 carbon. The C-1 chemical element is in the reverse, or b, configuration in polyose (cellulose is poly [b-1, 4 - Danhydroglucopyranose]. This b configuration, which has all functional groups in equatorial positions, allows the cellulose molecular chain to grow as many or as few lines as needed, resulting in a fine fiber-forming polymer [5].

Amylose, a starch component, is a connected polymer of glucose with a C-1 oxygen in its structure. This structure allows the connection to the next glucopyranose ring to become axially oriented and coil rather than expand. Despite its long chemical chains, amylose is not an effective fiber maker. Because of their equatorial position on the cellulose chain, the hydroxyls protrude laterally from the enlarged molecule. Because of their positioning, they are readily available for hydrogen bonding. Because of these hydrogen bonds, the chains form very ordered (crystalline) structures. Because the chains can be longer than the crystalline areas, they pass through a variety of crystalline regions, with intervals of disorder in between, resulting in the "fringed-micelle" theory. The inter-chain element bonding inside the crystalline regions is robust, resulting in fibers of exceptional strength and quality in most solvents. They also prevent carbohydrate from melting (they are not thermoplastic). In less organized places, the chains are wider apart and easier to connect to other molecules, such as water [6].

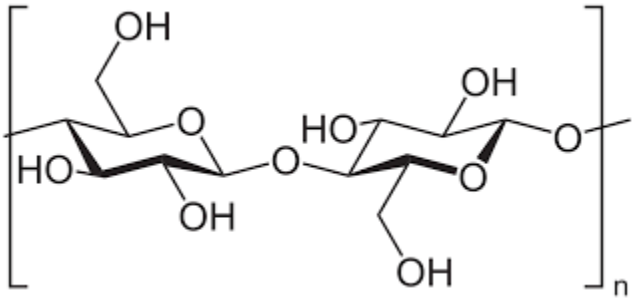

FIGURE 1: STRUCTURE OF CELLULOSE

ISOLATION OF CELLULOSE

The extraction of cellulose from its source takes two phases. The initial step may be to purify and homogenize the raw material so that it can respond more systematically in subsequent treatments. For the second stage treatments, the actual pretreatment is determined by the cellulose source material and, to a lesser extent, the shape of the starting cellulose particle. Wood

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and plant pretreatments involve the removal of matrix components (hemicelluloses, lignin, and so on), resulting in the isolation of individual complete fibers (wood fibers, plant fibers) [7].

The second stage involves separating such "purified" cellulose molecules into micro fibrillar and/or crystalline components. There are numerous ways to isolate cellulose particles. The three main separation procedures are fiber swelling, mechanical treatment, and acid hydrolysis. These processes can be performed alone, but many of them are used in succession or together to get the required particle shape.

Cellulose has no flavor or odor. Because of its strong hydrogen bonding and tendency to form crystalline forms, it is insoluble in water and most organic solvents. It is, however, soluble in a number of odd solvents, such as CdO/ethylenediamine (cadoxen), the binary compound N-alkyl morpholine - N-oxide (NMNO), LiCl/N, N-dimethylacetamide, near-critical water, and various ionic liquids [8].

A polymer's chain length, often known as its degree of polymerization (DP), is a significant feature. The length of the cellulose chain determines a variety of factors. The lengths of cellulose chains differ significantly depending on the source. Natural wood has a DP of roughly 10,000, but cotton has one that exceeds 15,000 [9].

Chain length from the same source may vary from one section of the plant to another. Chemical and mechanical degradation during the purification and analysis processes might also result in shorter chain lengths. Cellodextrins are small molecules that are typically soluble in water and most organic solvents. High hydroxyl group concentrations cause strong intra and intermolecular pressures, which have a substantial impact on the hydrophilic and swelling properties, which are strongly reliant on hydrogen bonding. Cellulose may be a hydrophilic material that expands when exposed to high humidity. Although swelling is somewhat reversible, cellulose activity curves have higher wet contents than adsorption curves [10].

This physical phenomenon effect was attributed to the inaccessibility of cellulose hydroxyl groups in the dry state. In other words, hydroxyl groups are more difficult to obtain in the dry state because they satisfy one another, but radical teams are easier to obtain in the wet state because absorbed water disrupts the hydrogen bonding between the polymer chains.

HEMP MATERIALS

Natural and organic fibers are gaining popularity in recent years. Most people understand that nature, suppleness, and health are the most important characteristics of textiles. Hemp fiber is naturally one of the most eco-friendly and oldest fibers. Hemp is dubbed the "fiber of a thousand uses." The importance of hemp in our forebears' economic and daily life is increasingly becoming apparent. It was essential for textile, paper, rope, and oil production. Hemp, like flax, kenaf, and jute, is a useful fiber plant. Bast plants are identified by their long, slender primary fibers on the stalk's outer surface. Cellulose, a glue-like soluble gelatin-like carbohydrate, binds the main hemp fiber to the core fiber. Hemp fibers were initially used in composites, reinforcements, and specialty pulp and paper. The core is built from wood. Hemp fibers are commonly used in animal bedding, garden mulch, and a wide range of building materials. Hemp also produces an oil seed that contains 25-35% oil by weight and is high in essential fatty acids, which are thought to be beneficial to health.

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TABLE 1: CHEMICAL COMPOSITION OF HEMP FIBERS

COMPOSITION	WEIGHT(%)
Alpha–cellulose	62-67%
Hemisellulose	8-15%
Lignin	4%
Ash	5%
Wax	1%

Hemp is a natural resource that grows quicker and easier than trees, making it a more cost-effective alternative to waiting decades for trees to mature before using them in fiber goods like Lyocell and wood pulp fabric. The bark of the hemp stalk contains the best fibers, which are among the world's longest natural soft fibers and rich in cellulose.

APPLICATIONS

Cellulose has long been utilized in construction, typically in the form of intact wood and textile fibers, as well as paper and board. At the same time, it is utilized as a versatile starting material for chemical conversions, the production of synthetic cellulose-based threads and films, and a variety of stable cellulose derivatives used in a wide range of industrial and domestic applications. Cellulose may be recycled into plastic wrap, cellophane, a thin transparent film, and rayon, a key fiber used in textiles [11].

Cellulose is the basic material used in the manufacture of cellulose derivatives. Cellulose is used to manufacture water-soluble adhesives and binders, including methyl cellulose and carboxy methyl cellulose, both of which are included in wallpaper paste. Crystalline and powdered cellulose are inert fillers used in tablets, as well as thickeners and stabilizers in processed foods.

MEDICAL FIELD

The biodegradable synthetic function surgical implants are employed in medical science surgery as implanted matrices for controlled long-term medicine release in an organism, as absorbed surgical sutures, and for eye treatment. The term "biomaterial" is now defined as a non-living material utilized in medical device applications to interact with a biological system, such as chitosan, cellulose, starch, and so on. It is vital that the term "biocompatibility" be coined since it governs how a tissue reacts to external material. Biocompatibility refers to a substance's ability to withstand particular host responses during a specific application [12].

The synthetic, absorbed sutures were widely used in general and tracheobronchial surgery. The most common sutures are multifilament with sophisticated handling qualities. Sutures made from polyglycolic acid (PGA), polylactic acid (PLA), and their copolymers are the most common and commercially accessible. Furthermore, polycaprolactone polymers are bio-absorbable, elastic materials used in medicinal applications [13].

Metal fixation is an efficient method of bone treatment, however removing metal implants causes bone weakness and refractures. In contrast, biodegradable implants will adapt to dynamic bone regeneration processes by lowering the amount of weight-bearing material. The injected substance degrades over time, thus there is no need to treat the patient to remove it. This field has the potential to create PGA, PLA, and polydioxanone [14].

Several biodegradable polymers, as well as synthetic polymers such as polyvinyl alcohol (PVA), polyvinyl pyrollidone (PVP), polylactic acid, and natural components such as polysaccharide (CL), chitosan (CH), and starch, may be advantageous in this respect. Some soluble polymers, such PVP and polypropylenediol, are used as pharmaceutical carriers [15]. The drug targeting was performed via bonds that can only be broken under certain conditions.

AGRICULTURE

Plastics, coatings, elastomers, cellulose fibers, and water-soluble polymers are increasingly used for controlled pesticide and nutrient release, soil the fertilization process, seed coatings, and plant protection. Biodegradable polymers are also valuable as crop mulching materials in fields and agriculture plant containers [16].

Their biodegradation technique is critical because it allows diverse biodegradable components to be combined and converted into useful products, hence improving soil quality. Agricultural mulches help farmers grow their crops. They are frequently used to manage weeds, maintain a consistent moisture level, accelerate plant development, and extend soil temperature. Biodegradable films made from starch, cellulose, and chitosan are being developed, with the addition of polymers such as vinyl polymer, polythene (PE), and vinyl resin (PVC). Polylactone and polyvinyl alcohol films break down quickly in the absence of soil microorganisms [17].

Effective fumigant mulches require low porosity sheets that limit the potential escape of volatile chemicals (insecticides, herbicides), allowing for smaller quantities to be applied.

INDUSTRIAL

Natural fiber films are compostable and biodegradable. It enhances dimensional stability in cold settings. This high-gloss, high-transparency film has anti-static features and is semi-permeable to moisture, providing improved anti-mist properties. Nature Flex films are made of cellulose, which is derived from wood pulp harvested from plantations that use sustainable forestry management techniques. Nature Flex films work well on the packaging line and have a wide range of heat sealing temperatures from 70 oC to 200°C. This twenty-seven shows that packaging film is used on quicker production lines while maintaining seal performance [18].

The thermoplastic cellulose acetate might be clear granulates produced at 170 oC and changed by the addition of substantial amounts of liquid plant-based plasticizers to increase the overall biodegradability of acetyl cellulose. Bioceta degrades slowly but totally biologically. It is created by injection, pressing, calendaring, or film processing in the case of film making.

RESEARCH OBJECTIVE

- Characterization of isolated cellulose materials from hemp waste material

INSTRUMENT USED

- FT-IR spectroscopy
- X-ray diffractometer
- Thermogravimetry analysis
- Scanning Electron Microscopy

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METHODOLOGY

The cellulose extracted from hemp used here as a solid support template. 5 g of hemp taken and ground well and sieved (30 mesh). To decolorize the hemp, first it should soaked in 5% of NaOH solution for 2 days and washed thoroughly with ethanol and water several times till the filtrate become colorless for the complete removal of all NaOH and then dried well to remove sticking of fibers and given acid washing for the neutralization of the hemp using dilute acetic acid.Finally thepH was adjusted to neutral. Then the compound was dried at 105˚C for 3 h.

FIGURE 2: CELLULOSE FROM HEMP

RESULT AND DISCUSSION WITH DATA INTERPRETATION

Cellulose, a natural biopolymer, has sparked significant research interest in materials science. Its ecologically favorable origin and qualities play an important role in research. The beneficial qualities include biodegradability, renewability, biocompatibility, high specific strength, abundance, and non-abrasiveness during processing. As a result, we concentrated on understanding the structure-property correlations and adapting cellulose-derived products for new particular uses that need less chemical use. The process of extracting cellulose from sugarcane bagasse is divided into three steps. The sugarcane bagasse was processed with weak hydrochloric acid to eliminate the hemicellulose.[19]

The residual extract was processed to separate cellulose material using two methods:

(i) delignification and
(ii) bleaching

There are two phases to isolating cellulose from hemp fibers. Initially, the hemp fibers were treated with a weak alkaline solution. Repeated washing with distilled water eliminated the alkali that had accumulated on the fibre's surface. The fibers were then neutralised with weak acetic acid and carefully rinsed with distilled water to maintain a neutral pH.[20]

The compound was dried at 105 ºC for three hours. Furthermore, the separated cellulose material is characterized using several analytical techniques and employed to encapsulate polyaniline and polypyrrole.

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CHARACTERIZATION

Analytical methods such as Fourier Transform Infrared (FT-IR) spectroscopy, X-ray diffraction (XRD) and thermal analysis (TG/DTA) were used to validate the structure of cellulose separated from two distinct sources.

FT-IR SPECTRAL STUDIES

The use of IR spectroscopy to cellulose is significant due to the particular nature of molecular groupings inside the cellulose, as well as the utility of a selection method for identifying bands in the IR spectrum. Many studies have acquired detailed structural data on cellulose using infrared spectroscopy. The IR spectra of cellulose derived from hemp material were collected and analyzed. Figure 3 depicts the Fourier transform infrared spectrum (FT-IR) acquired for hemp cellulose, while Table 2 presents the related spectral data. The stretching of -OH groups and hydrogen bonds causes an absorption peak between 3200 and 3600 cm^{-1} [21].

The -OH band was detected at 3480 cm^{-1}. The absorption frequency at 2919 cm^{-1} was caused by the asymmetrical stretching vibrations of -CH and -CH_2 bending. This C-H stretching vibration is presumably caused by the presence of methyl or methylene groups in the cellulose molecule.[22]

The peaks detected around 1460 cm^{-1} correspond to the -CH_3 asymmetric deformation of lignin, whereas the tiny strong peaks at 1283 cm^{-1} belong to the C-H symmetric deformation of lignin, as described in previous studies.[23]

The carbon-oxygen (-O-C-O-) stretching modes produced powerful and complicated multiple peaks in the 1000–1300 cm^{-1} range. Additionally, the absorption band about 1132 cm^{-1} in the spectra of all hemp composites was linked to the cyclic alcohols of lignin. The bands were ascribed to aromatic C-H inplane deformation and C-O deformation of lignin's main alcohol, respectively. The peak at 716 cm^{-1} indicates out-of-plane bending of C-H.[24]

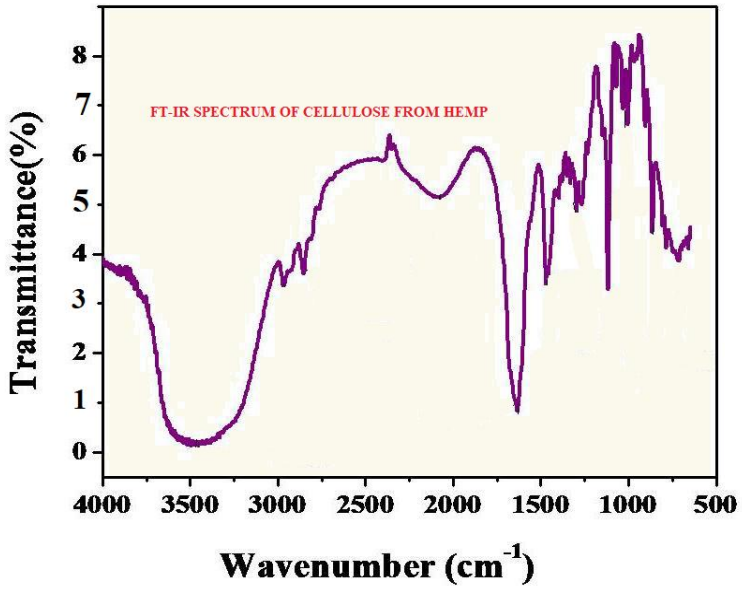

Figure 3: FT-IR spectrum of cellulose from hemp

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Table 2: FT-IR absorption frequencies of cellulose from hemp waste

WAVENUMBER (cm^{-1})	CELLULOSE(HEMP)
3481	OH stretching
2918	Asymmetrical stretching vibration of CH & CH_2 bending
2856	KBr peak
1635	Asymmetric Stretching vibration of O-H bending
1461	$–CH_3$ asymmetric deformation
1284	–CH asymmetric deformation
1133	C-O-C asymmetrical stretching
858	C-H stretching out of plane of aromatic ring
715	C-H out plane bending

X-RAY DIFFRACTION ANALYSIS

Figure 4 shows the X-ray diffraction spectrum of cellulose extracted from hemp. The X-ray diffraction analysis was used to identify the primary elements of the cellulose composite.The diffraction peaks at $2\theta = 19.5^0$ correspond to (110), $2\theta = 25^0$, and 36^0 correspond to (200) and (004) lattice planes, respectively. It indicates the presence of cellulose derived from hemp. [25]

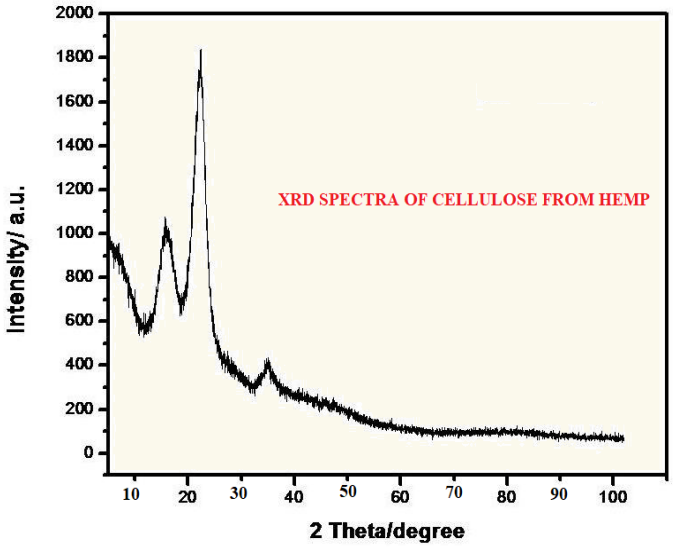

Figure 4: XRD spectra of cellulose from hemp

THERMAL ANALYSIS

Thermogravimetry Analysis (TGA) and Differential Thermal Analysis (DTA) were performed on cellulose isolated from hemp to determine the thermal stability of the materials. Figure 5 shows the TGA results for hemp cellulose. The TGA curve shows three decomposition steps. Because of the presence of moisture in the substance, breakdown occurs at temperatures ranging from 25^0C to 130^0C. The second stage occurred between 130^0C and 380^0C, indicating that cellulose's heat stability improved with purity. The third stage of weight loss, which occurs between 356^0C and 650^0C, verifies the existence of cellulose.

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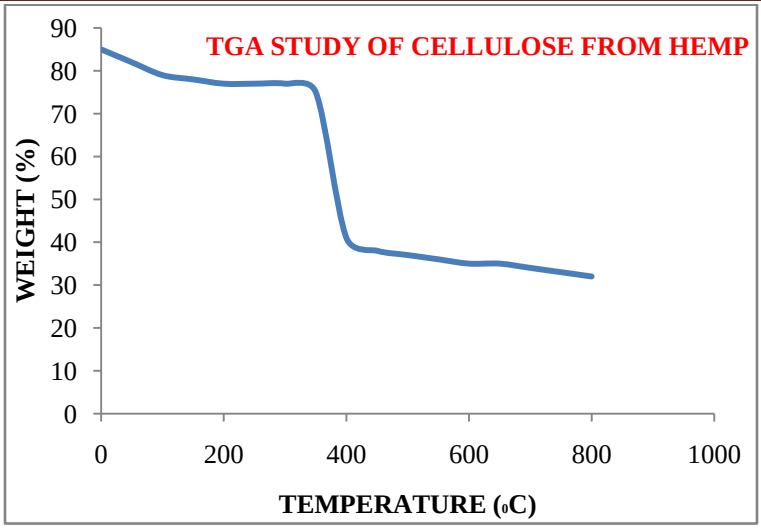

Figure 5: TGA study of cellulose from hemp.

Differential Thermal Analysis (DTA) was performed on cellulose isolated from hemp, as illustrated in Figures 6. Water evaporation produces endothermic peaks ranging from 26°C to 131°C (Shaikh et al., 2009; Yang et al., 2007). The strong peak found at 310^0C is attributable to cellulose dehydration/decomposition, which resulted in rapid devolatilization processes, leaving little or no solid residue.

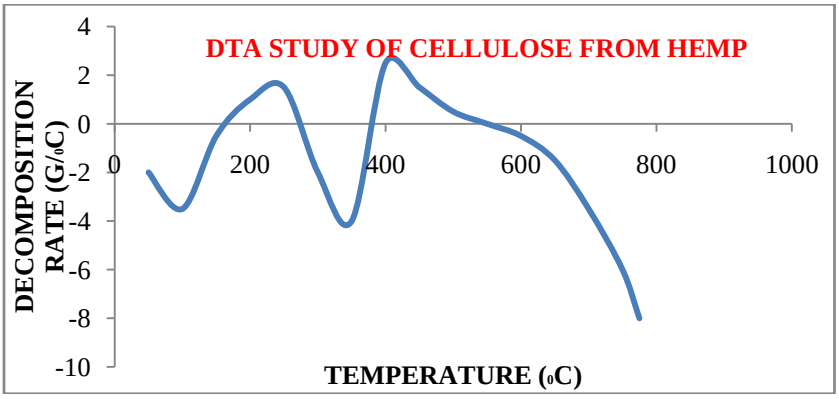

Figure 6: DTA study of cellulose from hemp

SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

Figures 7 depict scanning electron microscopy (SEM) images of cellulose from hemp, The SEM picture of cellulose extracted from hemp revealed a needle-like structure. Furthermore, morphological modifications were seen with various sources (i.e., hemp).

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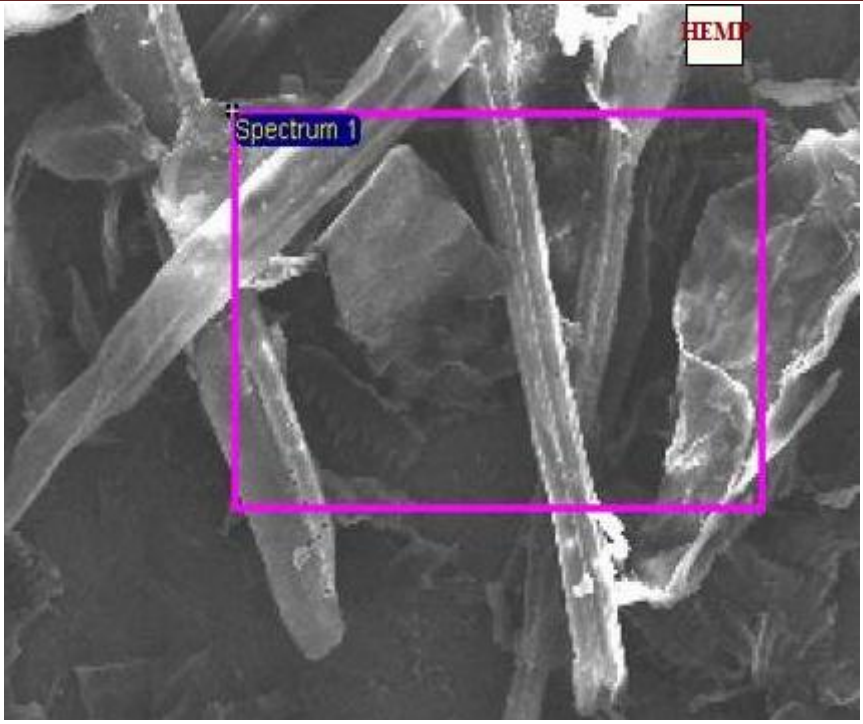

Figure 7: SEM images of cellulose from hemp

CONCLUSON

This study focuses on the separation and characterization of cellulose from agricultural waste materials such as hemp waste materials. The investigation's findings were reviewed and interpreted appropriately in this study. FT-IR spectra were used to establish the structure of the isolated cellulose repeating units. FT-IR spectrum investigations were also used to characterize the structure of the produced conducting polymers, PANI and PPY, as well as their encapsulating cellulose composites. The FT-IR spectra reveal that the peaks in PANI and PPY were preserved in the cellulose nanocomposite. The crystalline nature of PANI, PPY, and their composites was investigated using the X-ray diffraction (XRD) technique. The diffractogram of composites made from hemp material reveals a considerable shift in the diffractograms PANI and PPY, confirming the interaction of cellulose with PANI and PPY conducting polymers. The surface morphology of PANI,PPY, and cellulose, as well as their composites, was examined using SEM. The sensor investigations employing the produced polymeric composites in the study, PANI-C (hemp) and PPY-C (hemp), as modified electrodes yielded findings equivalent to those found in the literature. As a result, it is stated that this study offers significant promise for future sensor development. Until recently, researchers worked on polymer nanocomposites, which are beneficial in a limited number of applications; however, more study is needed on polymeric nanocomposites to fulfill the expanding needs in a variety of disciplines.

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