

Extraction of Cellulose from Plant Waste (Wheat Straw_Corn Waste)

¹Sabah Baghdadi, ²Dr. Ayyham Al-Jundi, ³Dr. Moadar Al-Ikla

^{1,2,3}Department of Chemistry, Faculty of Sciences, University of Idlib, Syria

Abstract - Cellulose was extracted from plant waste (wheat straw, corn cobs, leaves, and stalks) using three simple, applicable, and environmentally friendly chemical methods. The cellulose extracted by the first method (pressure cooking method) was the most pure and fastest. The cellulose extracted by the second method (soaking method) had good purity, though less than the first method, but its yield was higher. It does not require energy or special equipment and is easier to handle and apply. The cellulose from the third method (organic solvent method) was not pure. The third method is economically costly, lengthy, has a lower yield than the previous ones, and produces solutions that are more polluting to the environment.

Keywords: Environmentally friendly, pressure cooking and plant waste.

I. INTRODUCTION

With the increase in global production of agricultural food products, the percentage of agricultural waste has increased. Researchers and industrialists have sought to utilize this waste in various ways, among which is the extraction of natural cellulose from this waste for its use in various organic industries, such as fiber manufacturing, carboxymethyl cellulose, and methyl cellulose. Approximately 731 million tons of agricultural waste are produced annually, including about 354 million tons of rice straw, 203 million tons of wheat straw, and 180 million tons of corn straw and bagasse, respectively [1].

Citrus production exceeds 120 million tons annually; more than half of this fruit is inedible and is discarded as waste. The global volume of citrus processed annually in juice factories alone exceeds 31 million tons, with 50-60% of it being waste [2], this enormous abundance of agricultural waste has caused significant environmental concerns, including pollution, pest attraction, greenhouse gas emissions, and soil acidity, all of which pose serious threats to both humans and the environment. Furthermore, increasingly stringent waste disposal regulations have led to higher costs and increased energy requirements for traditional waste treatment methods, driving demand for alternative approaches. The idea of

converting industrial agricultural waste into value-added products has emerged as a waste management strategy [3].

The chemical content of biomass consists of cellulose, hemicellulose (xylan, mannan, galactan, and arabinogalactan), lignin (phenylpropene polymer), starch (amylase and amylopectin), secondary organic components (fats, protein, acetyl, boric acid), inorganic materials, and other elements (pigments, wax, secondary metabolites). Plant biomass is the first and largest biomass present on Earth after bacteria, representing about 80% of the total biomass, equivalent to 450 gigatons of carbon. This indicates a significant opportunity to study plant biomass and use it in making industrial chemicals [4].

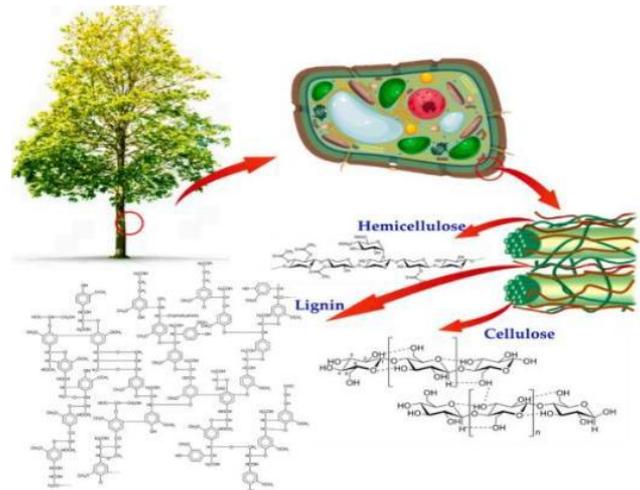


Figure (1): Represents the chemical structure of cellulose, hemicellulose, and lignin

However, only 10% of lignin-containing agricultural waste is used to produce valuable materials [1], that is, with increasing population and industrialization in countries, the demand for energy increases rapidly. This problem has two important consequences:

1. Declining oil, natural gas, and coal resources.
2. Serious pollution resulting from rising temperatures.

Therefore, agricultural waste can be a suitable and ideal alternative to fossil fuels, given its low cost, easy availability, renewability, and high biodegradability [1].

1.1 Cellulose as an Available Natural Polymer

Cellulose is the most abundant organic polymer in the world, produced primarily by plants [1], it is an important structural component of plant cell walls [5], as well as by microorganisms [6], its structure consists of crystalline and amorphous regions [7], and cellulose has good mechanical properties. Especially in recent years, interest has grown in developing new polymeric materials that are non-toxic, biodegradable, and derived from renewable resources [8].

Cellulose can be obtained from countless sources, including citrus processing waste from the citrus juice industry, which is low in lignin and available in huge annual quantities, making it an ideal raw material for cellulose [2], other sources include peanut shells, rice husks, banana stems, soybean hulls, sugar beet pulp [6], empty palm fruit bunches [3], oil palm fibers [9], cocoa pod husks, rice straw, wheat straw, corn straw [10], bagasse [1], algae, fungi, tunicates, bacteria, marine animals (tunicates), and plants (flax, hemp, cotton, etc.), the primary source of cellulose is forest wood. Table 1 shows the percentage of cellulose from various sources. As shown in the table, cotton has the highest cellulose content, followed by wood [12].

Table (1): Percentage of cellulose from its main sources

Source	Cellulose(%)
Cotton	90-99
Hemp	75-80
Wheat	49-54
Flax	70-75
Jute	60-65
Leaf fiber	40-50
Corn cobs	45
Kenaf	47-57

Cellulose is an organic compound belonging to polysaccharides, consisting of D-glucopyranose units linked by β -1,4-glycosidic bonds [12], the repeating unit of the cellulose polymer is anhydroglucose with the formula $(C_6H_{10}O_5)_n$, where n is the average degree of polymerization, which can be more than 10000 or 1000 (depending on the source). These units are linked by covalent oxygen bonds between C1 and C4 of adjacent units to form a long chain, as shown in Figure 2. This figure shows that cellulose contains three hydroxyl groups: secondary on C2 and C3, and primary on C6. These hydroxyl groups show reactivity when treated with sodium hydroxide to form sodium alkoxide [12], as shown in Figure (2):

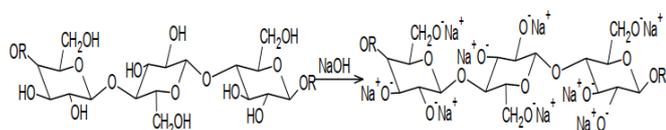


Figure (2): Represents the activation of cellulose with sodium hydroxide

Cellulose is found abundantly in plants and plays an important role in their structure and strength. The hydroxyl (OH) groups in cellulose form a strong and complex network of hydrogen bonds between molecules [1], the presence of this hydrogen bond network within and between cellulose chains gives it special properties, such as stability even at high temperatures and hardness. It also contains two regions that distinguish it from other natural polymers like starch: crystalline and amorphous regions [12], the size of the cellulose chain depends on its source. Table 2 shows the average degree of polymerization of cellulose from different sources [12].

Table (2): Represents the degree of polymerization of cellulose from some sources

Source	Average Degree of polymerization
Cotton fibers	8000-14000
Cotton linters	1000-6500
Bagasse	700-900
Bacterial cellulose	2700
Wood fibers	8000-9000
Flax fibers	7000-8000

Cellulose is a white powder, a linear polymer with high molecular weight [13], insoluble in water and common organic solvents [1], hygroscopic, solid, biodegradable, tasteless and odorless. Its melting point is between 270-260°C, and its density ranges between 0.5-0.2 g/cm³ [12].

1.2 Importance and Status of Cellulose

Recently, global agricultural research has focused greatly on non-woody materials for use in the environmental industry. Agricultural waste is considered one of the most important alternative raw materials for the pulp and paper industry, due to increasing forest product consumption and deforestation [14].

Cellulose derivatives are characterized by many advantages, including recyclability, reproducibility, biocompatibility, biodegradability, cost-effectiveness, and availability in a wide range of forms. It is a promising raw material for producing chemicals for applications in various industries [13].

Biodegradable polymers are materials that undergo chemical, biological, and physical force breakdown in the environment [4], cellulose does not dissolve in water. Introducing hydrophilic groups along the cellulose chain leads to the breakdown of hydrogen bonds, making its derivatives soluble in traditional solvents, thus expanding its application range to include esters and ethers [14].

Cellulose esters and ethers are two main groups of cellulose derivatives with different physical, chemical, and

mechanical properties, these derivatives are widely used in specific formulations, for example, esters of cellulose are stable against biological degradation, heat, hydrolysis, and oxidation [1].

Cellulose plays many important biological roles in plant cell walls, including providing mechanical strength to the cell wall, in plant growth, cell differentiation, cell-to-cell communication, water transport, and immunity [1].

Cellulose has garnered wide interest in diverse applications due to its large number of distinctive properties, including biocompatibility, such as in the cosmetics, food, and pharmaceutical industries [7].

For example, studies indicate that the following chemicals are made from cellulose: methoxymethyl furfural, 5-hydroxymethylfurfural, levoglucosenone, C-levoglucosenone, levulinic acid, formic acid, succinic acid, aromatic carbonates, hydroxycarbonates, and ethanoethylene. Similarly, biobutanol, biodiesel, glucose, xylitol, lactic acid, levulinic acid, formic acid, phenol, and vanillin are generally produced from lignocellulosic materials [4].

In recent decades, nanocellulose fibers have attracted significant interest. The functions and properties of the resulting nanocellulose depend largely on the cellulose source and its preparation process. Typically, nanocellulose refers to particles with at least one dimension in the nanometer range, these nanocellulose particles are usually made through mechanical pretreatment of the source material and acid hydrolysis [15].

1.3 Cellulose Sources and Their Applications

Wheat is one of the most important human food products, native to Central Asia. Its global production reaches 750 million tons, Wheat straw is one of the most important sources of cellulose, containing 40-60% percentage of natural cellulose. It is the second largest lignocellulosic material in the world. Wheat straw is characterized by its abundance and many advantages [1].

Chemical analysis of wheat straw shows it is rich in carbohydrates (cellulose, hemicellulose, and lignin), proteins, minerals (potassium and phosphorus), and silica [14], This resource has important uses, including: direct combustion for power generation, bioethanol production, feed production, and it can be easily utilized to prepare superabsorbent materials and prepare controlled-release fertilizer [14], Water absorption and release test results showed that the product has excellent swelling capacity and slow-release properties, Its addition to soil can significantly reduce nutrient losses and improve water retention capacity.

These hydrogels are defined as a three-dimensional network of hydrophilic polymers, Excellent, its low friction coefficient, high permeability, and high biocompatibility, This material has been used in medical, agricultural, and food industries [1].

Corn is widely cultivated in the world. Regarding waste, about 19.782*107 tons of corn cobs are produced annually; Cobs are used in producing animal feed, polishing materials, and biochar. However, unfortunately, a large quantity of cobs is discarded as agricultural waste after grain harvest [10], similarly, corn husks are a potential source for cellulose extraction [7].

Many studies focus on the high-value use of corn cobs due to their rich carbohydrate composition. Researchers have reported the possibility of using corn cob pulp to obtain monosaccharides, furan, bio-adsorbents, spherical biopolyurethane foams, phenolic hydrocarbons, and polyester resins, Hemicellulose extracted from corn cobs can also be used to produce ethanol, furfural, and succinic/propionic acid, as well as obtaining microcrystalline cellulose from corn cobs [7],[16].

Corn stover is considered the largest agricultural waste in the United States due to high land productivity, with about 100 million dry tons collected annually from corn, making it a renewable, sustainable, and one of the cheapest and most available waste materials in the United States[15].

Nanocellulose extracted from banana peel powder can be used as a dietary supplement for oral nanoemulsion films, as a main component for buccal use due to its good strength, and as a conductive membrane in biosensors [1].

Olive mill waste consists of about 56%liquid waste and 44%solid waste. This waste is characterized by its acidity, very high values of Biological Oxygen Demand (BOD) and Chemical Oxygen Demand (COD), and contains toxic levels of polyphenols. Waste materials pose a challenge in managing olive mill waste [17], Disposing of them is a concern for environmentalists, as solid olive industry waste is usually left to decompose, releasing carbon dioxide into the atmosphere, Previous studies have shown that solid olive mill waste can be mixed with wood and plastic composites, or extruded with polypropylene for use as filler in manufacturing fiberboard. It can also be used as a soil conditioner, or a composting agent, or an energy source, or a bio-adsorbent for heavy metals, or as a treatment to reduce phytotoxicity. However, the challenge remains to find an environmentally friendly and economical way to treat solid and liquid waste [17].

Cellulose extracted from agricultural waste currently has numerous applications as a raw material for producing organic

fertilizer, fuel, alcohol, and paper [15], Cellulose is used as a filler in many products, such as thermoplastics and adhesives[1].

1.4 Methods for Extracting Cellulose from Agricultural Waste

Some agricultural wastes are difficult to decompose due to their complex composition and physical and chemical structure. Therefore, biomass undergoes various processes and methods to overcome this obstacle. Pretreatment processes are processes performed on biomass and its components to increase conversion efficiency and extraction [1].

The main goal of pretreating agricultural waste and lignocellulosic biomass is for the following reasons[1].

1. Reduce energy consumption.
2. Reduce cost.
3. Form sugar from biomass.

Common pretreatment methods or cellulose extraction methods include:

1.4.1 Physical Processes

Physical methods are used to reduce particle size (aiming to increase the accessibility of enzymes and chemicals, conversion efficiency during processing, reduce cellulose crystallization, increase conversion efficiency, and improve product yield). Size reduction contributes to the crystalline nature of cellulose and increases its activity, such as applying high mechanical force to break the rigid structure of cellulose through cutting, grinding, crushing, and pressing methods. Pretreatment with ultrasound and microwaves is also used [1].

1.4.2 Chemical

Chemical methods include the use of acids, alkalis, oxidizing agents, and organic solvents to dissolve the crystalline lignocellulosic structure, to expand the surface of agricultural waste and increase its biodegradability [1].

1.4.3 Physico-chemical

This is done by combining the two previous methods [15].

1.4.4 Biological

This method uses aerobic bacteria, enzymes, fungi, or termites to degrade lignin. This method does not release toxic substances into the environment, consumes little energy, and produces few inhibitory compounds[1].

II. PRACTICAL SECTION

2.1 Apparatus and Tools

Locally made steam pressure vessel equipped with an electronic thermometer and pressure gauge – Mill from the German company SILVER CREST – Beakers – Ordinary glassware – Buchner funnel – Magnetic stirrer – Electric drying oven – Thermometer – Sensitive balance – Filter papers.

2.2 Chemicals Materials

Wheat straw waste and (cobs, stalks, and husks) of green corn plants were collected from scientific research fields in Idlib. They were dried under the sun, then ground, then dried in an oven at 105°C until constant weight, then sieved. - Sodium hydroxide, purity 99.9% from German company ISOLAB - Hydrochloric acid, purity 37% from German company ISOLAB - Hydrogen peroxide, purity from German company ISOLAB - Distilled water - Hexane from Turkish company BEYANLAB - Ethanol, purity 99.9% from German company ISOLAB.

2.2.1 How it works

Cellulose was extracted using three different methods from more than one source (wheat straw, corn cobs, husks, and stalks):

2.2.1.1 First Method (Pressure Cooking Method)



Figure (3): Locally made pressure cooking vessel

40- grams of ground corn cob were weighed and placed in the pressure vessel with one liter of sodium hydroxide solution at a concentration of 4%, and heated for two and a half hours at a temperature of 110°C and a pressure of 1.5 atm (1050 mmHg), in order to get rid of lignin. Then filtration (preferably hot filtration) using large-pore filter paper (filtration takes a very long time), then washing with distilled water until alkalinity is removed (until the filtrate becomes neutral). Then drying at 110°C for 24 hours (until constant weight). Then bleaching with hydrogen peroxide with slight

heating and stirring. Then filtration and washing with distilled water. Then treatment with 500 ml of hydrochloric acid at a concentration of 2.5% with continuous stirring for one hour to break down the cellulose chains as much as possible and obtain microcrystalline cellulose. Then filtration using small-pore filter paper on a Büchner funnel, and washing with distilled water until acidity is removed. Then drying at 105°C until constant weight. The final product was fine white granules weighing 16 grams.

The previous method was used but replacing ground corn cob with ground green corn leaves. Starting from 40 grams yielded 12.06 grams of cellulose, but the shape of the cellulose was different from that extracted from the cob; it was needle-like and the particle size was larger, but its color was bright white.

The same method was also used for wheat straw. Starting from 40 grams, 13.5 grams were obtained.

2.2.1.2 Second Method (Soaking Method without Heating)

30 grams of ground corn cob were soaked in 0.5 liters of sodium hydroxide solution at a concentration of 1.5% for 22 hours at a temperature of 15°C. Then filtration and washing to remove alkalinity. Then drying in the oven at 60°C. Then bleaching with hydrogen peroxide in a basic medium of sodium hydroxide at 60°C until the color becomes light. The cellulose was yellowish white. Then filtration and washing. Then drying at 60°C until constant weight. The final product weighed 13.2 grams and was yellowish white.

The previous method was used using 40 grams of wheat straw. The weight of the cellulose produced was 18 grams.

The previous method was also used for 40 grams of corn stalk. The weight of the cellulose produced was 7.98 grams.

2.2.1.3 Third Method (Organic Solvent Method)

25 -grams of ground corn cob were weighed, then washed with hot distilled water to remove aqueous extracts, waxy materials, etc. Initially, the filtrate was brown, then turned pale yellow. Then the sample was placed in the oven to dry at 100°C until constant weight. Then it was soaked in a sodium hydroxide solution at a concentration of 2% (solid/liquid ratio 1:40) for 2 hours at 15°C. Then washed free of alkali. Then bleached with hydrogen peroxide, but a dense foam formed preventing reaching 60°C. It was left for two hours. Then washing with an acetic acid solution (pH 4-5). Then washing with distilled water. Then treatment with hexane. Then washing with ethanol to remove hexane residues. Then bleaching with hydrogen peroxide at 60°C prevented foam formation, but the cellulose color remained yellowish. Then

filtration and washing with distilled water. Then drying at 105°C until constant weight. The final weight of cellulose was 7.4 grams.

III. RESULTS AND DISCUSSION

- The yield for the first method was: 40%, 30.12%, and 33.75% for corn cob, its leaves, and wheat straw respectively, and it was white in color.



- The yield for the second method was: 44%, 26.6%, and 45% for corn cob, its stalk, and wheat straw respectively, and it was yellowish white.



- The yield for the third method was: 29.6% and its color was yellowish white.



- The insolubility of the resulting cellulose in toluene, hexane, acetone, ethanol, as well as in diluted

hydrochloric acid and diluted sodium hydroxide, was confirmed.

FT-IR spectra were also taken for each sample and compared with a standard cellulose spectrum. The match was good in most cases:

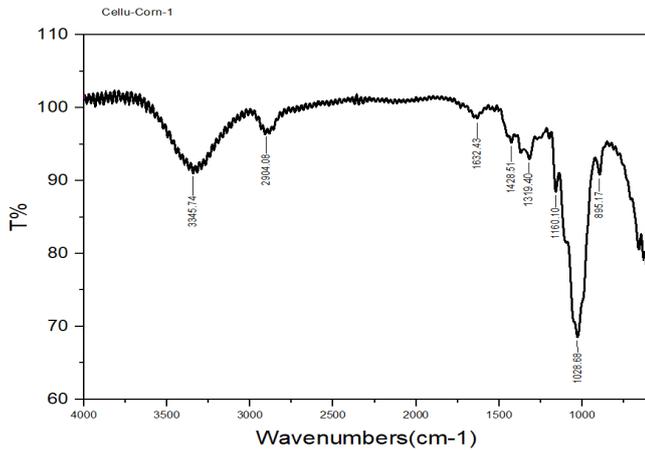


Figure (3): Represents the spectrum of corn cob (Pressure method)

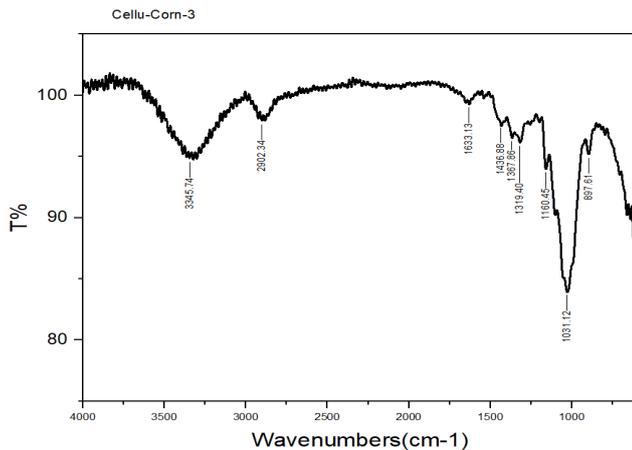


Figure (4): Represents the spectrum of corn husks (Pressure method)

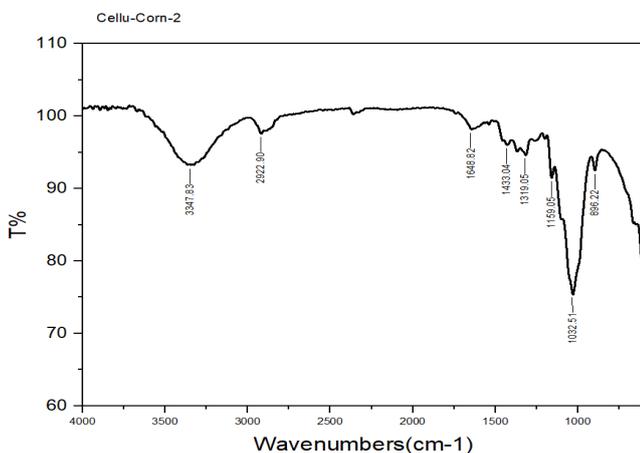


Figure (5): Represents the spectrum of corn stalk (Soaking method)

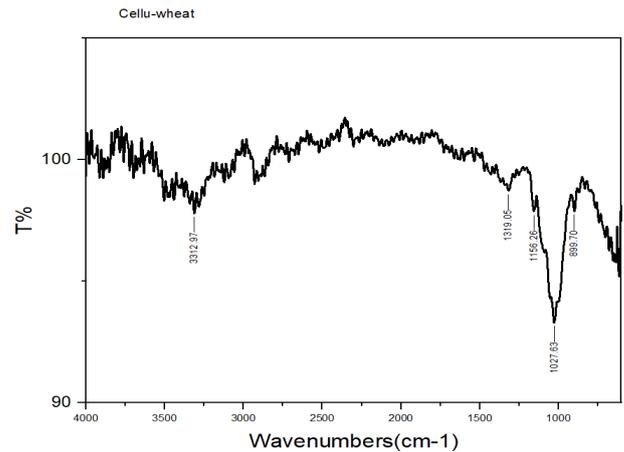


Figure (6): Represents the spectrum of wheat (Soaking method)

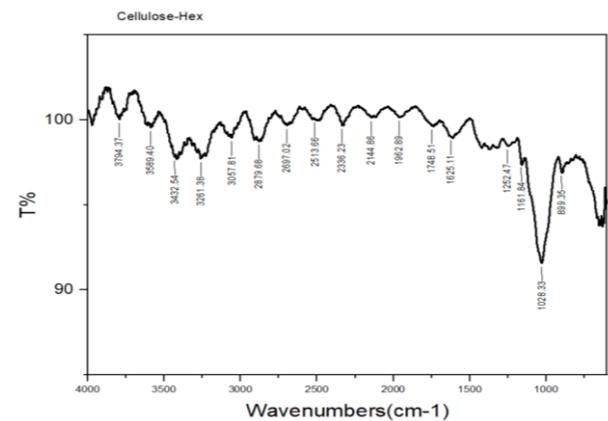


Figure (7): Spectrum of corn cob (Organic Solvent method)

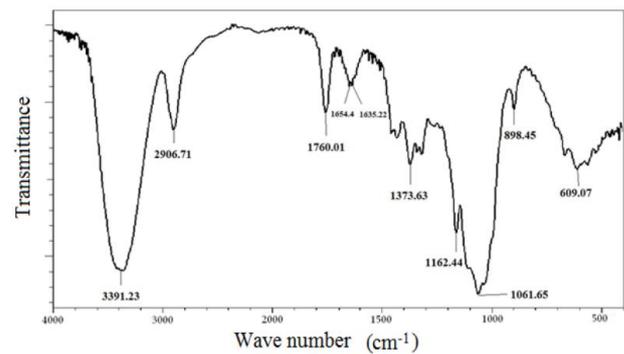


Figure (8): Represents the standard cellulose spectrum

Interpretation of the main characteristic peaks of the standard cellulose spectrum:[18]

1. A broad and large peak at wave number 3391.23 cm^{-1} : Expresses O-H stretching vibration.
2. The peak at 2906.17 cm^{-1} : Expresses C-H stretching vibration.
3. The peak at $\sim 1760 \text{ cm}^{-1}$: Expresses C=O stretching vibration, i.e., indicates carbonyl groups. Carbonyl groups are not present in the cellulose formula; they

result from the degradation of cellulose to sugars (glucose containing aldehyde groups) or the presence of lignin or hemicellulose residues.

4. The two peaks at 1654 cm^{-1} and 1635 cm^{-1} : Express O-H bending vibration.
5. The peak at 1162 cm^{-1} and 1061 cm^{-1} : Express C-O-C stretching vibration.
6. The peak at 1373 cm^{-1} : Expresses C-H bending vibration.
7. The peak at 898 cm^{-1} : Expresses β -glycosidic bond vibration.

* Comparison of the standard spectrum with the other spectra:

By comparing all the spectra obtained by the three methods with the standard cellulose spectrum, we find a good match. We find absorption spectra for all the main group bonds in cellulose, and we notice the absence of the peak at $\sim 1760\text{ cm}^{-1}$ representing the carbonyl group vibration. This is a good indicator as it shows that the resulting cellulose has good purity and indicates no degradation of the resulting cellulose.

As for the spectrum of the cellulose from the third method, it is far from the standard cellulose spectrum, indicating the impurity of the cellulose and the inefficiency of the method used.

* Comparison of the three methods used based on FT-IR spectra:

- 1) First Method - Pressure Cooking: Produces pure, smooth, bright white cellulose. It is environmentally friendly, does not require many harmful solvents, is somewhat fast and economical, and gives a good yield. However, it requires a special reactor, energy, and there are some risks due to handling a pressurized and hot reactor.
- 2) Second Method - Soaking: Produces cellulose with good purity, yellowish-white in color. This method is economical, does not require energy, is environmentally friendly (does not require many solutions and solvents), and gives a good yield. But it requires a long time.
- 3) Third Method - Using Organic Solvents: Produces impure cellulose, yellowish-white in color. This method is uneconomical (costly), not environmentally friendly, and requires a very long time.

IV. CONCLUSIONS

Three different methods were used to extract cellulose from more than one source of plant waste available in Idlib - (wheat straw and green corn stalks, leaves, and cobs). The methods were environmentally friendly, easy to apply, and

several to secure a new source of cellulose from low-cost and available sources, and to dispose of it by burning, which is an environmental pollutant. It provides a suitable alternative source to forest wood and cotton, thus preserving forests, opening the way for establishing new industries based on cellulose, reducing the problem of securing raw materials for these industries, providing new job opportunities, and opening the door for innovation in recycling agricultural waste.

First Method (Pressure Cooking): A fast and environmentally friendly method that produces pure cellulose. It can be applied in food and pharmaceutical industries. However, there is danger in its application due to the simultaneous application of pressure and high temperature. It was used to extract cellulose from corn cobs, its leaves, and wheat straw. The yield was 40%, 30.12%, and 33.75% respectively. The resulting cellulose was white and smooth.

Second Method (Soaking): An easy-to-apply method, especially in industries that do not require high-purity cellulose, such as textiles, paper, and biodegradable plastic materials instead of petroleum-based ones. It does not require high energy, many equipment, or many chemicals. The yield of this method is higher than the previous one, but the cellulose produced by this method has good purity. This method was used to extract cellulose from corn cobs, its stalk, and wheat straw: 44%, 26.6%, and 45% respectively. The resulting cellulose was yellowish white.

Third Method (Organic Solvent): A lengthy and costly method that requires a toxic and expensive solvent. The resulting cellulose was impure, as indicated by the FT-IR spectrum. The yield is low due to the numerous processing and filtration steps, which cause loss of small cellulose molecules through the filter paper pores. This method was used to extract cellulose only from corn cobs. The yield was 29.6%. The resulting cellulose was yellowish white.

V. RECOMMENDATIONS

The water resulting from extraction can be utilized in several industries, such as:

1. Extraction of hemicellulose sugars.
2. Manufacturing bioethanol from fermentation of present sugars.
3. Extraction of lignin phenols and their use in many industries, such as adhesives.
4. Focus on methods that are both environmentally friendly and economical, not just on yield.
5. Head towards high value-added products, such as nanocellulose and cellulose derivatives.

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