



## Preparation of Nanocellulose from waste of Cotton Lint

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### Abstract

The aim of the current study is to prepare nanocellulose (NC) from agricultural wastes: cotton lint rather than throwing it into waste which increases pollution of the environment and thereby reduces environmental pollution to some extent. In this study, the nanocellulose was prepared by acid hydrolysis using sulfuric acid at a concentration (30) % for cotton lint to dissolve the raw materials by sulfuric acid and extract cellulose. It also converts it to nanocellulose by using renewable and advanced methods by the ultrasonic processor for different times at (30-180) minutes. And the outcome was a fine powder with good quality that could have been used in other processes and methods. By completely removing lignin and hemicellulose, Atomic force microscope (AFM) photos demonstrated the smooth surface and small nanoparticles of cotton lint-derived Nano cellulose, while Fourier transform infrared spectroscopy (FTIR) measurements demonstrated the purity of cellulose. 3.303 nm is reported to be the average roughness of cotton lint-derived nanocellulose. There are numerous uses for the manufactured cellulose nanoparticles. Because of this, we think that in the near future, cotton lint with a high cellulose content will be a great source of inexpensive raw materials.

**Keywords:** cotton lint, nanoparticles, and nanocellulose.



## تحضير النانو سيليلوز من مخلفات وبر القطن

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### الخلاصة

تهدف الدراسة الحالية الى تحضير مادة النانو سيليلوز من مخلفات النبات (وبر او زغب القطن). بدلا من رميها في النفايات وزيادة تلوث البيئة وبالتالي تقليل التلوث البيئي الى حد ما. في هذه الدراسة تم تحضير النانو سيليلوز عن طريق التحلل الحامضي باستخدام حامض الكبريتيك بتركيز (30%) (وبر القطن) لإذابة المواد الخام بواسطة حامض الكبريتيك. واستخلاص السيليلوز وتحويله إلى نانو سيليلوز باستخدام طريقة متقدمة ومتجددة بواسطة جهاز الموجات فوق الصوتية لأوقات مختلفة (30-180)min وكانت النتيجة مسحوقا ناعما بصفات جيدة. وقد يتم استخدامه في اجراءات وطرق أخرى اظهر التحليل الطيفي بالأشعة تحت الحمراء (FTIR) نقاء السيليلوز عن طريق إزالة اللجنين والهيم سليولوز تماما من خلال ملاحظة الاهتزازات الخاصة بالسيليلوز في صور طيف FTIR بينما أظهرت صور مجهر القوة الذرية (AFM) ان النانو سيليلوز المشتق من القطن يحتوي على جزيئات نانوية صغيرة و سطح املس ومعدل الخشونة (3.303nm) واظهرت صور المجهر الالكتروني لمسح المجال (FESEM) مناطق بلورية منتظمة ومناطق غير متبلورة واصغر قطر نانوية هو (42)nm توجد استخدامات عديدة لجسيمات السيليلوز النانوية المصنعة لذلك نعتقد في المستقبل القريب ان وبر القطن الذي يحتوي على نسبة كبيرة من السيليلوز وهو مصدر واعد للغاية للمواد الخام بأسعار معقولة .

**الكلمات المفتاحية:** نانو سيليلوز ، وبر القطن، جزيئات نانوية.

### Introduction

Cellulose nanoparticles have received a lot of interest among the many organic nanoparticles for a variety of reasons. Due to its abundance, biodegradability, affordability, and exceptional mechanical strength, cellulose has a variety of significant practical applications (1). Cellulose is the most common polymer and the main component of the bulk of plant biomass (2). A range of leftover plants, such as maize cobs, rice straws, banana stems, sugar beets, soy hulls, and many others, are used to make cellulose nanoparticles (3). The term "nano cellulose" and its derivatives are used to describe a novel type of nanotechnology. This type appears to have numerous applications in the world of materials where it is possible to considerably enhance the physical attributes of strength, flexibility, weight, and optical capabilities (4). Additionally,



the cellulose nano crystal (CNC), is one of the strongest and stiffest organic molecules. CNCs have high surface areas (approximately 250 m<sup>2</sup>/g) and are hydrophilic. Surface stimulation is particularly prone to affect them. (5). Nanocellulose can be categorized using a variety of factors, such as shape, size, and structure. Sheet, spherical, and whiskers are the three main categories of shape features. Micro fibrillated nanocellulose (MFC) has a sheet-like structure in contrast to the two shapes that microcrystalline cellulose (MCC) can take. It should be noted that each nano cellulose structure contains a range of lengths, widths, and crystallinity percentages. These structures can take many different shapes, including those of particles, rods, webs, and fibers. MFC has both web-like and fiber-like forms, whereas NFC exclusively has the fiber-like form due to structural changes (6, 7, 8).

## **The Aim of the study**

This study aims at Preparation of Nanocellulose from cotton lint and pear peels instead of throwing it as waste and causing damage to the environment

## **Experimental**

### **Materials**

Agricultural waste is the: cotton lint collected from Diyala, Iraq. Linter is an important by product of the textile industry.

### **Chemical solutions preparation**

The following options were used in this study:

1. Sulfuric acid in concentration (30 %) of cotton lint.
2. Sodium hydroxide with a concentration of (1%) for naturalize the sample to PH7 by adding NaoH (1 %).
3. Sodium hypochlorite (Naocl) is used with a concentration of (4%) for the bleaching process and to remove most of the remaining phenols such as lignin and polyphenols.
4. HydroaSchloric acid with a concentration of (0.1) M was used to change the pH of dye solution.



## Chemical Methods

This technique deals with purifying the raw materials cotton lint and turning these wastes into nanocellulose materials through a number of chemical studies.

Nano cellulose preparation from agricultural waste (cotton lint) cotton lint is extracted of its cellulose in the following steps:

### A. Step of purification:

This process involved repeatedly washing the cotton lint in distilled water (exactly five times). Following that, cotton fiber was dyed and allowed to dry for one full day.

### B. Bleaching technique

In this phase, the sample (15 gm) was introduced to sodium hypochlorite (4%) for 3 hours while being vigorously mixed. The sample was then filtered and repeatedly rinsed with distilled water before being dried at (50 °C) for 3 hours.

### C. Preparation Nanocellulose product:

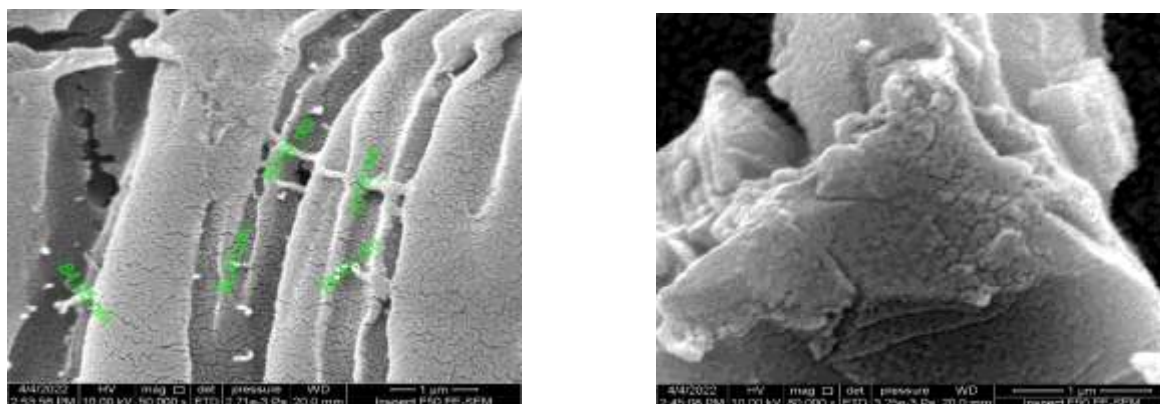
Acid hydrolysis has been used to prepare the final product (9). When the product from the bleaching step has been hydrolyzed in 100 ml of 30% sulfuric acid, the sample must be thoroughly dissolved at 35°C with vigorous stirring before being added to each concentration of H<sub>2</sub>SO<sub>4</sub>. Finally, 150 ml of deionized water must be added to the solution to complete the reaction. The solution then cooled to 8 °C., the produced solution contains (1% of NaOH). Following neutralizing, the prepared suspended solution was filtered after being spun at 10,000 g for 15 minutes (10). 200ml of deionized water was stirred into the sample to produce nanocellulose (NC). After addition, the mixture was sonicated three times for a total of 180 minutes using an ultrasonic processor (11). The solution was chilled to 8 °C for a day after the sonication was finished. Following that, the sample was filtered and maintained at 8 °C. Finally, the sample obtained a perfect powder after drying at 60°C for 12 hours. the fine powder Placed in a polypropylene tube and prepared for measurement.

## Results and Discussion

Using a scanning electron microscope (SEM), an atomic force microscope (AFM), an X-ray diffraction (XRD), and a Fourier transform infrared spectroscope, the product of nanocellulose (NC) generated from agricultural materials was detected (FT- IR).

### **Field emission scanning electron microscopy (FESEM).**

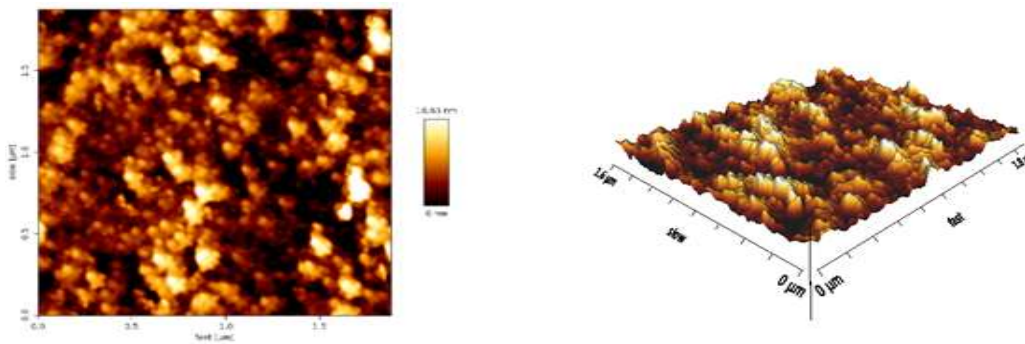
Important visual data about the shape of cellulose is provided by field emission scanning electron microscopy (FESEM). The morphology of the sample nanoparticles was examined using FESEM (12). After isolation techniques, the external side's morphology is visible using a scanning electron microscope (FESEM). The size and shape of the samples may be seen in the FESEM photos. In addition to displaying non-amorphous regions, it also demonstrated the presence of regular crystalline regions. Distribution of spherical shape like nanoparticles with irregular distribution. From FESEM images it is confirmed in figure 1, the surface morphology of the NC made from cotton lint shows layers that are separated from one another by a few nanotubes. Demonstrate an irregular distribution of spherical-shaped nanoparticles with a homogeneous distribution. The modest size of the particles is corroborated by the FESEM photos. Cotton lint has a nanometer size of (42) nm.



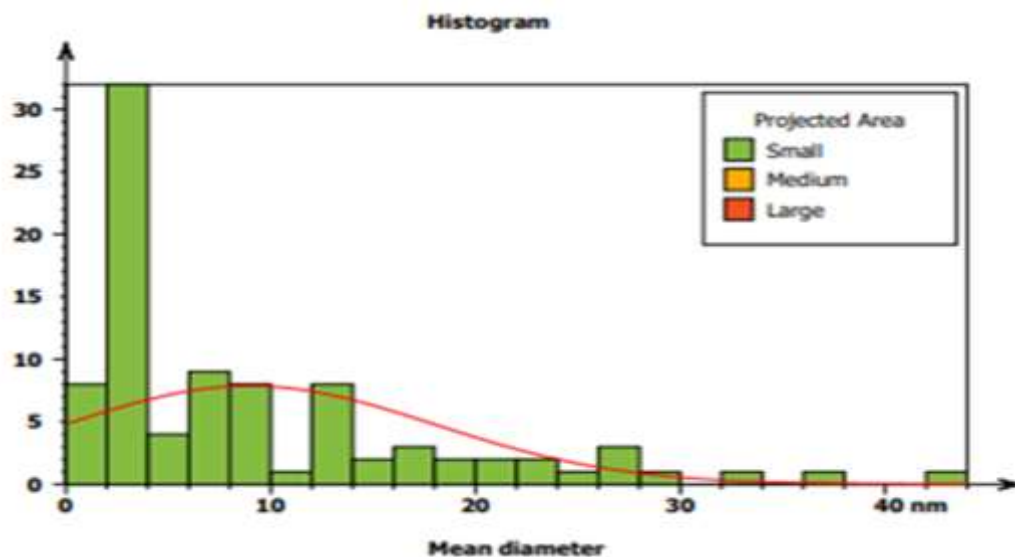
**Figure 1:** The Fe-SEM images of (NC) produced from cotton lint

## Atomic forces microscopes (AFM)

The average granule size of nanocellulose and its morphological properties were determined using the Atomic forces microscope (AFM) (13). Figure (2) depicts typical AFM images of nanocellulose made from cotton lint and pear peels in two- and three-dimensions. The images exhibited spherical forms and a very homogeneous distribution. The granular distribution patterns of the nanocellulose made from cotton lint nanoparticles are shown in figure (3). It is discovered that cotton lint-derived nanocellulose has an average roughness of 3.303 nm and an average diameter of 49.67 nm.



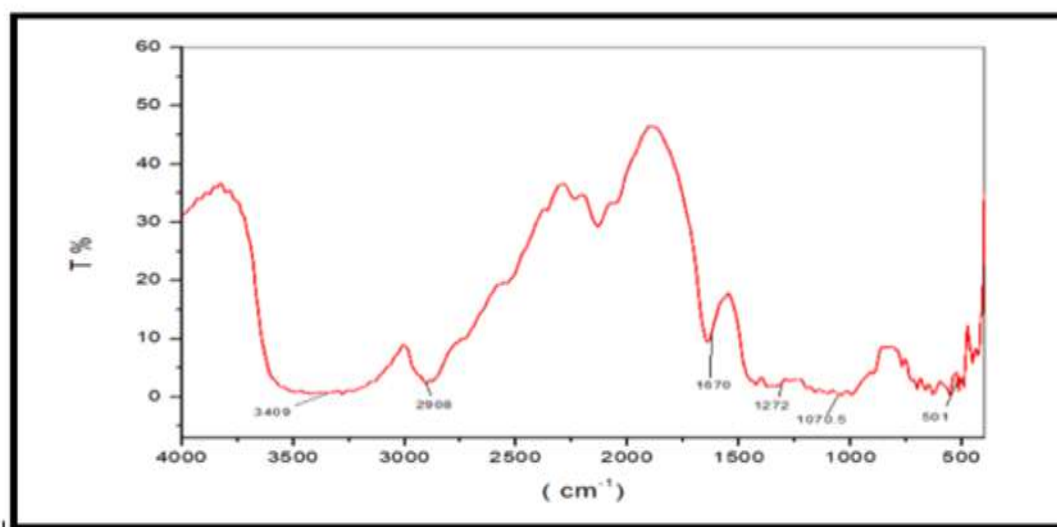
**Figure 2:** Shows AFM image of (NC) prepared from cotton lint.



**Figure 3:** Granularity cumulating distribution of NC prepared from cotton lint

## Fourier transforms infrared spectroscopy (FT-IR)

The chemical structure of the lignocellulosic components found in the samples was determined using FTIR analysis. The spectrum were captured between 500 and 4000  $\text{cm}^{-1}$ . It illustrates how the chemical makeup of the fibers changes over time. Changes in the hydroxyl and carboxyl groups are used to track differences related to the transition from macro to nanomaterials (14). Figure (4) displays the FTIR spectra of NC made from cotton lint and 30% sulfuric acid. The peaks at 3400 and 2900  $\text{cm}^{-1}$ , which were observed in all the samples, were caused by the stretching vibrations of cellulose's hydroxyl group and its aliphatic saturated (C-H) ring, respectively. the vibration caused by the (O-H) bending peak at 1670  $\text{cm}^{-1}$  (14,15). The range between 1100 and 1500  $\text{cm}^{-1}$ , where it is impossible to attach signatures for specific vibrations because complicated overlap effects may occur in this region, showed smoothed peaks. These tiny peaks may also be caused by the proteins and hemicelluloses found in the cellulose fiber walls (16). The spectra of all samples are typical of cellulose. A ccording to thr literature , Presenting peaks at 1160 and 496  $\text{cm}^{-1}$  which indicates, the purity of the crystalline band of cellulose, with characteristics of C-O stretching vibration and elongation of cellulose typical pulp  $\beta$ -glycoside bonds, respectively, especially in nanocellulose spectra (17).



**Figures 4:** FT-IR spectrum of Nanocellulose produced from cotton lint



## Conclusions

It can be concluded from this study that:

1. Cotton lint waste was used as a raw source to create nanocellulose. Without using costly procedures like bleaching, acid hydrolysis, freezing, or drying processes, NC product was made from the cellulose included in the raw materials.
2. Raw materials were sonicated using an ultrasonic device, and the result was a powder with good qualities that might have been used in various procedures and methods. NC product was distinguished by (AFM, FESEM, and FT-IR)
3. Images from an atomic force microscope (AFM) revealed that the nanocellulose made from cotton lint has a smooth surface, tiny nanoparticles, two and three dimensions
4. By entirely eliminating lignin and hemicellulose, Fourier Transform Infrared spectroscopy (FT-IR) demonstrated the purity of cellulose.

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