



## Synthesis and characterization of cotton fiber-based nanocellulose



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### ABSTRACT

Nanocellulose prepared from the natural material has a promising wide range of opportunities to obtain the superior material properties towards various end-products. In this research, commercially available natural cotton was treated with aqueous sodium hydroxide solution to eliminate the hemicellulose and lignin, then cellulose was collected. The collected cellulose was subjected to acid hydrolysis using sulfuric acid to obtain nanocellulose. The prepared nanocellulose was further characterized with the aid of Fourier transform infrared spectroscopy, X-ray diffraction and Scanning Electron Microscopy to elucidate the chemical structure, crystallinity and the morphology.

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### 1. Introduction

For the past few decades, researchers have paid an attention to nanoscience and nanotechnology, because of its dynamic properties and applications [1–4]. Nanotechnology (“nanotech”) deals the manipulation on an atom and molecular scale. It was subsequently reached a clear level with one dimension size ranged from 1 to 100 nanometers [5–9]. Nanotechnology and Nanoscience deal the ability to observe and to control the individual atom and molecule. Nanomaterials from cellulose play a major role in the nanotechnology field. Currently, researchers are finding different ways to deliberately create materials at the nano-level to be used their advantages, including lighter weight, higher strength, and good chemical reactivity than their large-scale counterparts and enhanced control of light spectrum.

Now-a-days, one of the hottest super materials is nanocellulose (NC) and widely been used. It has several applications, like pharmaceutical, food, electronics, due to its light, transparent and strong property. NC can be obtained from any natural cellulosic source materials, such as wood pulp, which consists of a tightly packed array of a needle-like structure called ‘nanofibril’. The preparation

strategy is systematic and obtained by top-down and bottom-up approaches. It is produced from the plant matter that has been reduced into small pieces and purified followed by homogenized to remove the non-homogenous compounds like lignin [10]. The creation of nanocellulose is completely in a neutral manner. Although NC offers diverse physical and chemical properties, even it makes the salt water to be drinkable. There is increasing interest in the use of cellulose nanofibre. Further, due to their higher aspect ratio, cellulose nanofibers have greater reinforcing capability than the currently used macro/microfibre feedstocks.

Cellulose is the major biodegradable polymer abundant on the earth. It is found in structural components of the cell wall of all green plants [11]. It is a polysaccharide compound and the homopolymer of glucose. A large number of glucose units combines to form a cellulose polymer molecule, which depends on their chain length and rate of polymerization [12]. The structure of cellulose is ( $\beta 1 \rightarrow 4$ ) linkage [13] as shown in Fig. 1a & b. It shows a very low conductivity and high resistivity of electricity. Based on the solubility nature, cellulose is classified into three categories. They are  $\alpha$ ,  $\beta$ , and  $\gamma$ , where  $\alpha$  is insoluble,  $\beta$  is precipitate in nature and  $\gamma$  is completely soluble, cellulose is found to be rich in natural cotton [14].

Cotton is the soft small ball-like fibrous material produced in the protective case. The cotton plant is the shrub and it contains large cellulose. Cotton producing places in the world are located in Amer-

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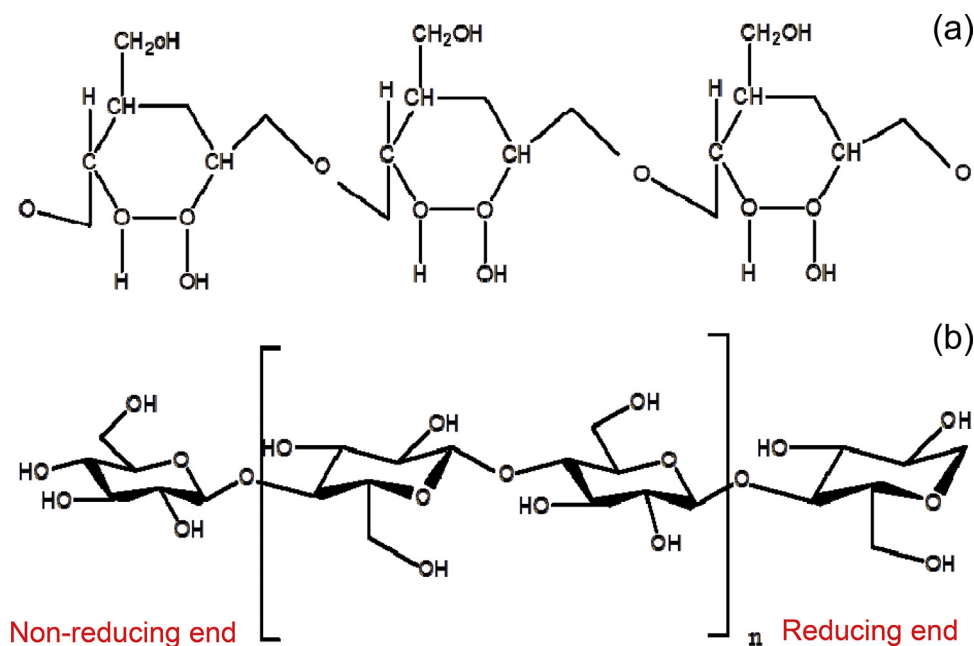


Fig. 1. Structures of cellulose. (a) Natural cellulose; (b) Nanocellulose.

ica, Asia, and Africa and the largest cotton production is from China [13]. The natural fibre used to produce NC is cotton, which has many advantages like wide availability, low-cost, renewable, abundant, strong, durable and biodegradable. Cotton swells in a high humid environment, water and in certain concentrated solutions [15–17].

Acid hydrolysis is the common method used for the preparation of NC [18]. Acid hydrolysis of cotton produces the hydrocellulose, which is not affected by cold weak acids. In acid hydrolysis, the most important considered point is cellulose acid ratio. The present research uses sulphuric acid-mediated synthesis and characterized the NC after acid hydrolysis method. Their physiochemical, structural and cellulose crystallinity index analyses were carried out by FTIR and XRD analysis.

## 2. Materials and methods

All reagents procured were of analytical grade and used without additional purification steps. Commercially available Cotton was used as the source material. Sodium hydroxide pellets (NaOH) and Sulphuric acid were received from Sigma-Aldrich. All experimental solutions were prepared using de-ionized (DI) water.

### 2.1. Synthesis of nanocellulose

The synthesis of NC follows a systematic procedure. Commercially available cotton was initially made into powder without impurities. The powdered sample was then treated with alkali solution NaOH solution. After this process cellulose was obtained. This sample was further subjected to acid hydrolysis. During the entire process of preparation, DI water was used as the solvent and stirred for ensuring uniform mixing. The pH was checked at every step and maintained as neutral (Fig. 2).

### 2.2. Alkali treatment on cotton

Commercially available Cotton was brought and ground in order to make a powder. 5% NaOH was added to the sample and was subjected to stirring constantly at room temperature for 4 h to get homogenous mixing. Then, it was washed and filtered several times

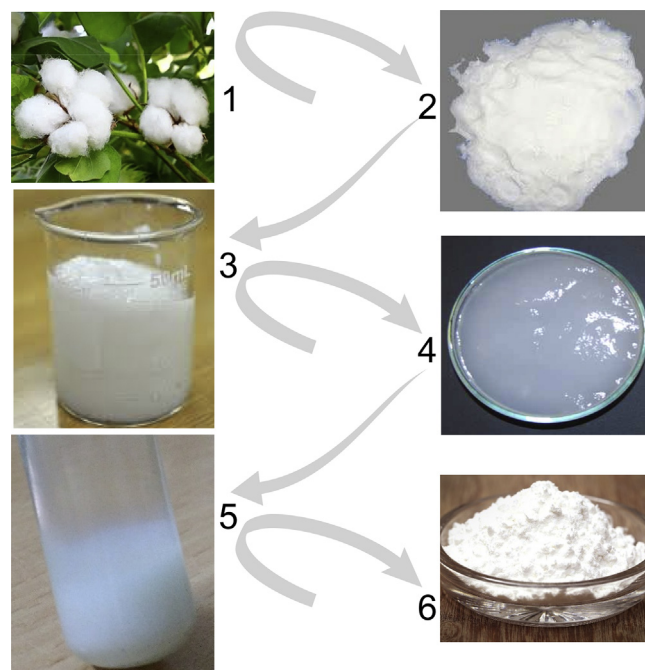


Fig. 2. Synthesis of Nanocellulose. (1) Raw cotton; (2) Cotton powder; (3) after Alkali treatment; (4) after Acid hydrolysis; (5) Nanocellulose; (6) Nanocellulose powder.

with DI water adjusted to a neutral pH for the removal of lignin and hemicelluloses. The filtrate was dried at 80 °C for a day.

### 2.3. Acid hydrolysis

The alkali treated cotton was then added to the 10 ml of concentrated sulphuric acid and 20 ml of DI water, heated at 40 °C with constant stirring for an hour to get a well-mixed solution. It was then washed several times with the water adjusted to a neutral pH. The prepared suspended solution was centrifuged at 10,000 × g for 15 min. The obtained nanocellulose was dried at 80 °C in order to obtain a nanocrystalline powder.

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