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Biosynthesis of Nanocellulose and Study of Its Properties

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Nanocellulose is an important material with many applications. This work presents an easy and environmentally friendly way to prepare nanocellulose. The method is based on anaerobic fermentation of agricultural waste. The resulting cellulose has nanoscale with a size of about 18 nm and crystals with a size of 15 nm. Compared to the acid hydrolysis method, this method gave better results. As found, the dimensions and crystallization rate are decreased with increasing fermentation time. 37°C is the optimum temperature for the fermentation preparation process. The yield of nanocellulose is 23% of the amount of cellulose-rich residue added at the beginning of the process.

Наноцелюлоза є важливим матеріялом з багатьма застосуваннями. У даній роботі представлено простий і екологічно чистий спосіб приготування наноцелюлози. Метод заснований на анаеробному бродінні відходів сільського господарства. Одержана целюлоза має наномасштаб величиною близько 18 нм і кристали розміром у 15 нм. У порівнянні з методом кислотної гідролізи цей метод дав ліпші результати. Встановлено, що розміри та швидкість кристалізації зменшуються зі збільшенням часу бродіння. 37°C — оптимальна температура для процесу приготування бродінням. Вихід наноцелюлози становить 23% від кількости багатого целюлозою залишку, доданого на початку процесу.

Key words: nanocellulose, fermentation, XRD, SEM, wheat straw.

Ключові слова: наноцелюлоза, ферментація, рентґенівська дифракція, сканівна електронна мікроскопія, пшенична солома.

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1. INTRODUCTION

Cellulose is an important material with many good properties [1]. It is an organic substance of natural origin, which forms the sheaths

of plant cells [2]. Cellulose is found in most plant wastes. It is widely used in many fields such as textiles, textiles, medicine and other fields [4, 5]. Nanocellulose is more important than cellulose because it forms a crystalline nanostructure with special properties [6, 7]. Nanocellulose and nanomaterials have many applications in fields such as medicine, pharmacy, agriculture, analytical chemistry and water purification [8–13]. Nanocellulose was synthesized from the acid hydrolysis of cellulosic materials (wastes such as paper, tissues, cotton waste, *etc.*) [14]. Many researchers synthesized nanocellulose by applying concentrated sulphuric acid to cellulose at a temperature (such as 50°C) for several hours [15]. The acid hydrolysis methods are simple for preparation and possibility of use with various starters [16]. However, getting rid of acid after nipping is difficult and time consuming. Traces of the acid remain even after various treatments [17]. Many researchers have developed better methods for preparing nanocellulose, such as hydrolysis using bacteria or enzyme [18]. The developed methods are more efficient in the synthesis of nanocellulose with good properties, but they are more expensive and more complex [19]. Fermentation of organic waste is an efficient way to dispose of organic waste in a safe manner and to benefit from it as much as possible [20]. During anaerobic fermentation, bacteria work to decompose organic matter. Cellulose is hydrolysed dynamically, giving chains that are shorter and shorter until they end in sugar units that bacteria feed on [21]. In this paper, we will try to isolate nanocellulose formed during the anaerobic fermentation process of wheat straw waste, characterize it and adjust its conditions.

2. EXPERIMENTAL

2.1. Materials and Apparatus

H_2SO_4 98% acid Sigma; IR spectrometer of JASCO model M4100; SEM of TESCAN model MIRA3; x-ray diffraction by Philips model PW1370, Cu (0.154056 nm), step size 0.05 deg.

2.2. Biosynthesis of Nanocellulose

We take the starters (wheat straw) and mix them with a live mixture that contains anaerobic fermentation bacteria and nitrogen-rich substances to help the self-growth of the bacteria and leave the mixture for 30 days to complete the fermentation while maintaining the temperature around 38°C (warning: methane gas is formed during fermentation and is a suffocating and flammable gas; the pres-

sure of the experiment vessel must be relieved under the tug or in an open place every once in a while). The mixture is transferred to a beaker and boiled in order to sterilize it from bacteria and get rid of residual ammonia and dissolved gases. The mixture was filtered with plain filter paper to separate large particles and undissolved residues. The filtrate is concentrated to half and then rested for 24 hours. The clear part is separated and the remaining cellulose is washed several times.

2.3. Chemical Synthesis of Nanocellulose [15]

Nanocellulose crystals were prepared by acid hydrolysis of cotton. 40 g of cotton (cut with scissors into small pieces) and weighed accurately; then, add to it with stirring 400 ml of sulphuric acid 40%, the reaction mixture was stirred at 50°C for 48 hours. The mixture was transferred to a 6-litre Becher, and the cotton residue was washed well several times and excluded, fill the human volume with distilled water and boil the solution for 5 minutes, then leave for the next day and separate the clear liquid by clearing. Repeat washing until the pH value has risen to medium moderate. The resulting mixture was dried at 110°C for 48 hours. The resulting mixture was preserved for later use and study.

3. RESULTS AND DISCUSSION

During this work, a study was conducted on samples of wheat straw as an organic starter for the anaerobic fermentation process. Nanocellulose was prepared, and we obtained nanocellulose with a yield of approximately 17%. The resulting nanocellulose is a dirty white to yellowish powder. Figure 1 shows pictures of different samples

a *b*

Fig. 1. Pictures of different samples of prepared nanocellulose.

of prepared nanocellulose. Table presents synthesis conditions of the studied nanocellulose.

3.1. FTIR of Nanocellulose

Figure 2 shows FTIR spectra of the synthesized nanocellulose. The broad peaks after 3500 cm^{-1} are due to the OH group in the cellulose structure. In addition, the peaks around 2900 cm^{-1} belong to

TABLE. Properties of the studied nanocellulose and synthesis conditions.

No.	Particles' size nm	Crystal size, nm	Crystallization percentage, %	Synthesis method	Temperature, °C	Time, day	Yield, %
a	350	85.1	63.0%	Hydrolysis H_2SO_4 40%	50	2	58
b	35	23.6	32.0%	Fermentation 4% solid	37	25	15
c	25	18.0	52.5%		37	30	19
d	18	14.7	12.8%		37	40	23
e	110	72.4	33.2%		35	30	11
f	180	110.5	15.8%		40	30	10

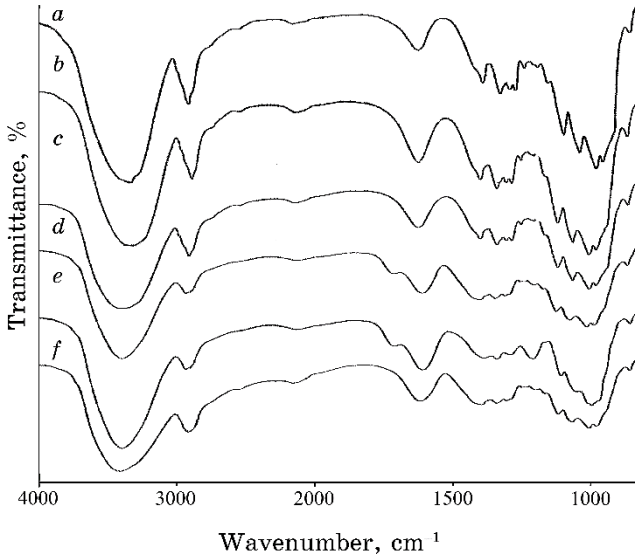

Fig. 2. FTIR spectra of the synthesized nanocellulose.

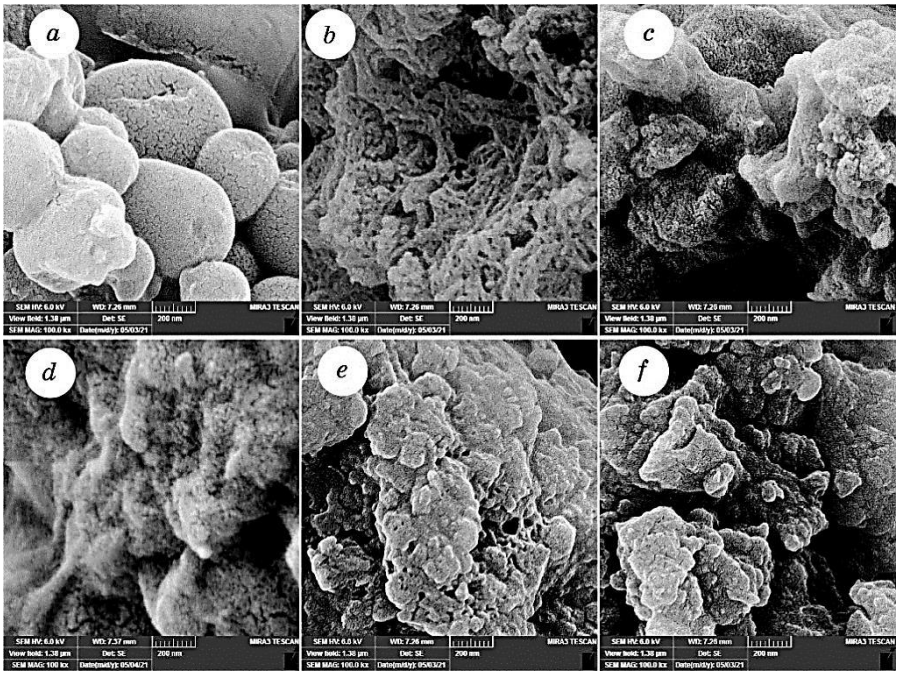

Fig. 3. Nanocellulose prepared by hydrolysis.

the C–H bond. The peak at 1600 cm^{-1} may belong to the C–O bond. The peaks around 1200 cm^{-1} belong to the C–H bonds.

3.2. SEM of Nanocellulose

Morphology of nanocellulose was studied by scanning electron microscopy (SEM). Figure 3 shows SEM image of nanocellulose. Figure 3, *a* shows nanocellulose prepared by hydrolysis with a size of 350 nm and a distinctive crystalline shape. Figure 2, *b*, *c*, *d*, *e*, *f* shows cellulose prepared by the biological method; all samples are fibres with relatively small dimensions (35, 25, 18, 110, 180 nm, respectively). The method of preparation greatly affects the shape of the sample and the size of its particles. The longer time corresponds to the minutes of smaller size, and the conditions were ideal for bacteria to work at 37°C.

3.3. XRD of Nanocellulose

Figure 4 shows the XRD spectrum of the prepared material. The x-ray diffraction spectrum of the samples is useful in determining the size

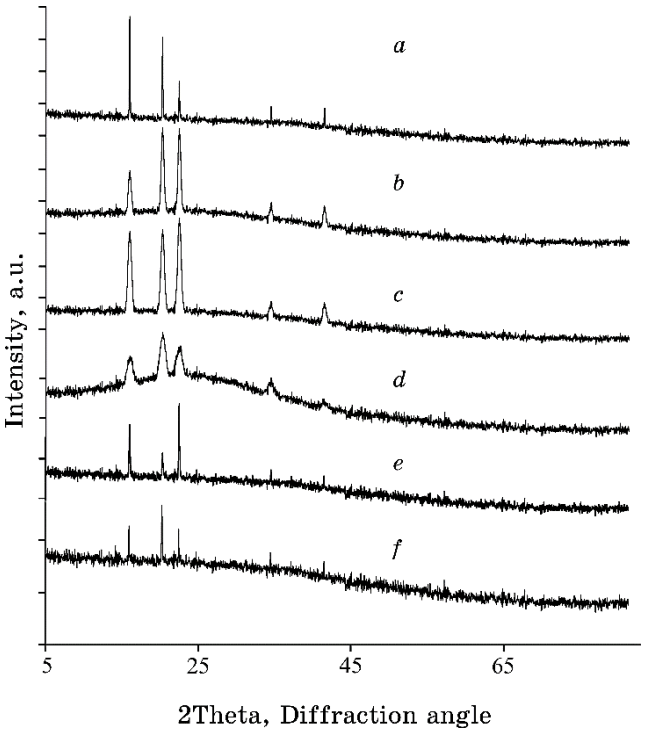

Fig. 4. XRD spectrum of the studied nanocellulose.

of the particles and the percentage of crystallinity. Figure 4 shows the XRD spectrum of the studied nanocellulose. Spectrum (in Fig. 4, *a*) represents the nanocellulose prepared by acid hydrolysis. It is noticed a high crystallinity rate and sharp peaks indicating relatively large crystals, while the samples are prepared by the biological method. Figure 4, *b*, *c*, *d*, *e*, *f* had less sharp peaks and more noise. Its crystal size is relatively small with a low crystallinity rate. Crystallization percentage and crystal size decrease with increasing fermentation time. This corresponds to a decrease in volume in SEM.

4. CONCLUSIONS

The biopreparation method is an effective method that can produce nanocellulose samples easily and with great efficiency. The fermentation method is a green method that depends on bacteria and does not need chemicals. The fermentation preparation method helps to get rid of some agricultural waste. Nanocellulose prepared by the biological method has a smaller size of up to 50 nanometres with crystals up to 33 nanometres and a low crystallinity rate of less

than 40%. The method can be used for industrial preparation in large quantities, and there is no difficulty in isolating the prepared cellulose.

5. HIGHLIGHTS

A green biosynthesis method of nanocellulose from agricultural waste.

Adjusting the preparation conditions of nanocellulose preparation, the effect of heat and time, and comparing them with acid hydrolysis preparation by a green method.

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