

Section 1. Materials Science

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OBTAINING CELLULOSE NANOCRYSTALS AND THEIR USE IN PAPER PRODUCTION

Abstract. Cellulose nanocrystals were obtained from waste paper by hydrolysis. The result of Fourier transform infrared spectroscopy (FTIR) for nanocellulose showed that no new chemical bonds were formed during the production of nanocellulose. The results of X-ray diffraction analysis showed that the crystallinity of the treated and untreated spots was 66% and 73.8%, respectively. The resulting nanocellulose particles were identified. improving the physical properties of paper sheets, in particular, tensile strength, stretch and tear performance. The maximum increase in mechanical properties was achieved with the addition of 6.0% nanocellulose.

Keywords: Paper, waste paper, composite material, cellulose, degree of polymerization, color intensity, tear strength, breaking length, rupture index, nanocellulose, nanocrystal.

Introduction. One of the problems of the paper industry today is the source of raw materials. From this point of view, the reduction of non-renewable resources has led to a trend towards more efficient use of waste paper generated in the paper industry as a raw material for various products. Waste paper as a biomass material Waste paper generated by the paper industry typically makes up about 7% of municipal solid waste and contains inexpensive cellulosic material. In industrialized countries, millions of tons of paper boxes are produced and used, resulting in a huge amount of waste paper. Due to the cellulose content of paper boxes, the production of valuable cellulose-based materials from them can become an alternative recycling method. With the advent of nanotechnology, more and more attention is paid to the extrac-

tion of nanocellulose from various renewable sources and its application in technical fields. The production of nanocellulose from waste paper today is of interest to researchers. For example, we have successfully recovered nanocellulose from waste paper by treating the waste paper with alkali and acid. Nanocellulose showed the following characteristics: diameter from 3 to 10 nm, length from 100 to 300 nm, crystallinity index 75.9%. A review of the literature showed that foreign scientists produce high-quality nanocellulose directly from old newspapers and recycled newsprint. Several studies have been published in recent years using nanocellulose as a reinforcing material for biocomposite materials; however, little is known about its use in paper and pulp production. There are several studies in which nanocellulose is made from old newspapers

or newsprint; therefore, deeper knowledge in this area is of interest. Nanocellulose has received considerable attention due to its use as an additive in papermaking to improve mechanical properties such as tear, tear and tensile strength. In addition, nanocellulose has several advantages: large surface area, biodegradability, high tensile strength and high. Thus, the addition of nanocellulose to paper pulp shows a high potential for increasing hydrogen bonds between cellulose fibers, resulting in stronger formed paper.

Results and discussion

Chemical composition analysis. The chemical composition of refined and unprocessed flax plant cellulose has been studied. Compared to untreated pulps, the content of extractives, lignin and pentosan in purified pulps decreased by 0.34%, 2.83% and 1.47%, respectively. The initial extractivity of crude pulp was about 0.85% and decreased to 0.55% after chemical pretreatment. This decrease may be caused by the removal of ink during ink cleaning. The lignin content decreased from 19.45% to 16.10% due to the decomposition of lignin during the bleaching stage. After bleaching, the chemical composition of the pulp has completely changed, indicating a highly effective removal of components such as lignin, extractives and hemicellulose in the alkaline and bleaching stages.

X-ray diffraction analysis.

The XRD spectra of raw and purified cellulose are presented in (Fig.1. on fig. 2) shows the diffraction peaks of dyed pulps at $2\theta = 16.10^\circ$ and 22.95° , which mostly correspond to untreated pulps. In addition to the main diffraction peaks at $2\theta = 16.10^\circ$ and 22.95° , the raw pulp showed two minor peaks at 12.55° and 29.85° , indicating that the presence of kaolin and calcium carbonate may be associated with the peak for kaolin it is usually at 12.35° and for CaCO_3 the most intense peak is usually at $2\theta = 29.85^\circ$. Refined pulp accounted for 66%, which is 7.8% lower than unprocessed pulp; During the reaction, it was found that the cellulose crystallization zone is destroyed by NaOH . The crystallinity index of nanocellulose was 70.25%. The nanocellulose crystallinity index was higher than that of purified cellulose, but lower than that of untreated cellulose, which is explained by the higher proportion of cellulose crystallites present in untreated cellulose. During hydrolysis, the amorphous region of cellulose was removed, which contributed to the low yield (35.55%) and high crystallinity of nanocellulose. Thus, nanocellulose had a high degree of crystallinity, and this property could be important for paper production.

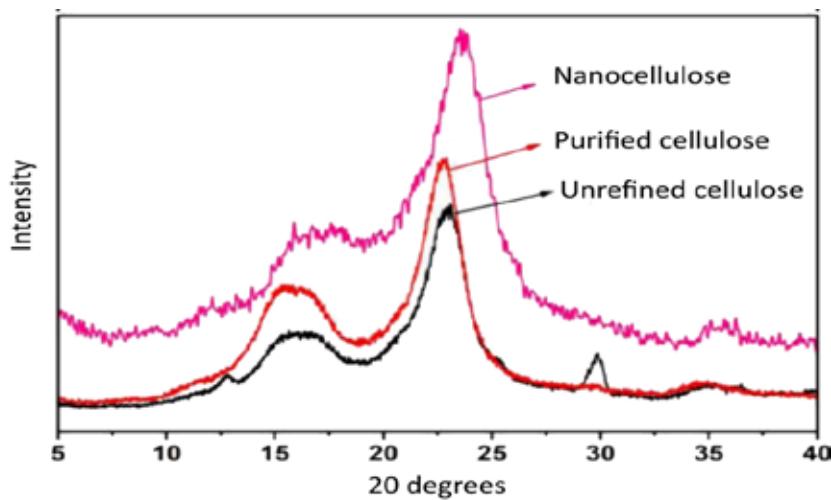


Figure 1. X-ray diffraction patterns of untreated (a) and purified (b) celluloses

Fourier transform infrared spectroscopy (FTIR) analysis. The infrared spectra of untreated

cellulose (a), purified cellulose (b), nanocellulose (c) and microcrystalline cellulose (d) are presented

in Figure 2. The absorption peak at 1165 cm^{-1} was consistent with the stretching of the glycosidic bond (C-O-C) of cellulose. The FTIR spectra in Figure 2 showed an extended vibrational absorption peak of -OH from about 3200 cm^{-1} to 3600 cm^{-1} , which was significantly stronger than that of the untreated celluloses (Figure 2 a). reported that the ink had an absorption band at 3380 cm^{-1} , which reduced the concentration of hydroxyl groups from untreated cellulose. In the spectrum of untreated (a) cellulose, the characteristic peak at 1733 cm^{-1} corresponds to the C = O stretching frequency of carboxyl groups in their acidic forms. In addition, the absorption peak at 1733 cm^{-1} can be attributed to the C = O stretching of hemicelluloses. The peak intensity at 1733 cm^{-1} of purified cellulose (spectrum b) was lower than that

of untreated cellulose C (spectrum a), which may be caused by the degradation of hemicelluloses by NaOH pretreatment. Figure 2 a (unrefined cellulose) showed the presence of lignin in areas at 1505 cm^{-1} due to aromatic vibrations in the C plane. The peak of cellulose at 1505 cm^{-1} (Figure 2 a) was significantly weaker than that of untreated celluloses (Figure 2 b), indicating the effectiveness of using chemical treatment to remove lignin. The 1263 cm^{-1} peak presented in the cellulose spectra was also a weak intensity attributed to lignin. Compared to the starting material, changes in the peaks at 1733 , 1505 and 1263 cm^{-1} indicated that the chemical treatment removed hemicellulose and lignin from the surface of the fibers. These results were consistent with the results of the chemical composition analysis.

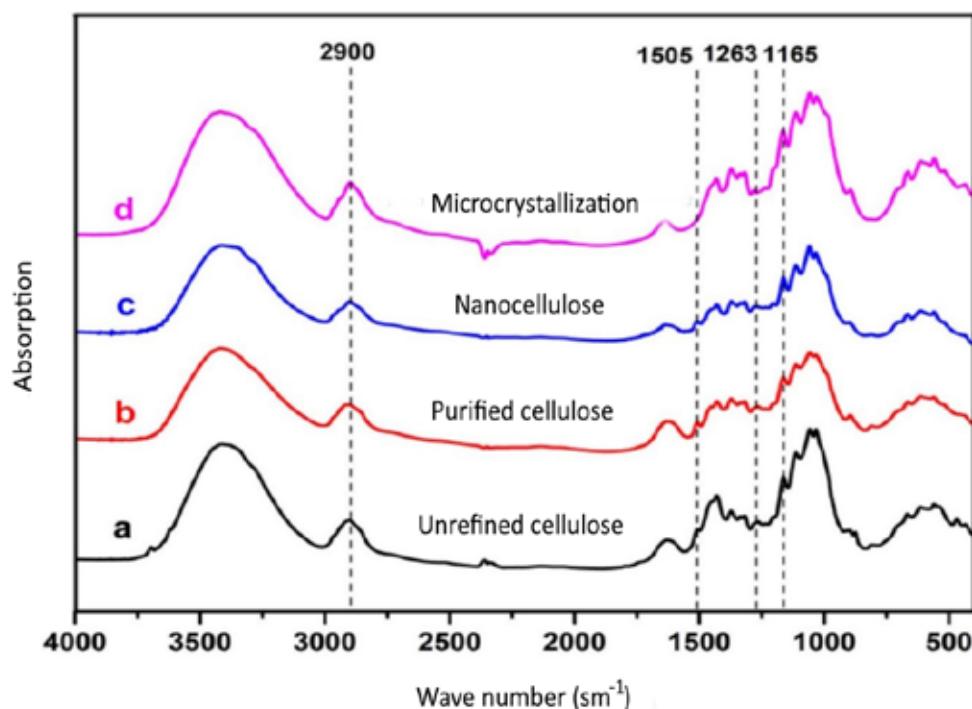


Figure 2. FTIR spectra of untreated cellulose (a), purified cellulose (b), nanocellulose (c), and microcrystalline cellulose (d)

The spectrum of nanocellulose includes areas similar to microcrystalline cellulose. The peak near 1635 cm^{-1} was due to adsorbed water. The characteristic bands of β -(1-4) glycosidic bonds at 896 cm^{-1} as well as the C-O-C stretching vibration of pyranose at 1165 cm^{-1} were also clearly shown in the spectra,

which were the main features. Accordingly, the intensity of the C-H stretching at 2900 cm^{-1} was lower for the treated celluloses than for the untreated celluloses, indicating that the distortion of the hydrogen-bonding structure and crystal shape occurred during the ink purification process. This result was consistent with

the (XRD) result. Overall, these results showed that the molecular structure of cellulose was not destroyed after pretreatment with H_2SO_4 , and the nanocellulose still showed the basic chemical structure of cellulose.

Effect of nanocellulose on physical properties of paper. With an abundance of surface hydroxyl groups, large specific surface area, high aspect ratio, excellent mechanical properties, and high heat resistance, nanocellulose has demonstrated its potential as a reinforcement in papermaking and to increase

the strength of paper sheets. Nanocellulose and carboxymethylcellulose have been used in papermaking to improve the physical performance of paper sheets without structural damage to the fibers. In the current study, flax plant cellulose nanocellulose and carboxymethylcellulose were added to the pulp to make paper. In addition, the effects of additives with different dimensions were evaluated on the tensile strength, tear index, burst index and elongation of the paper sheets with the results shown in Figure 3.

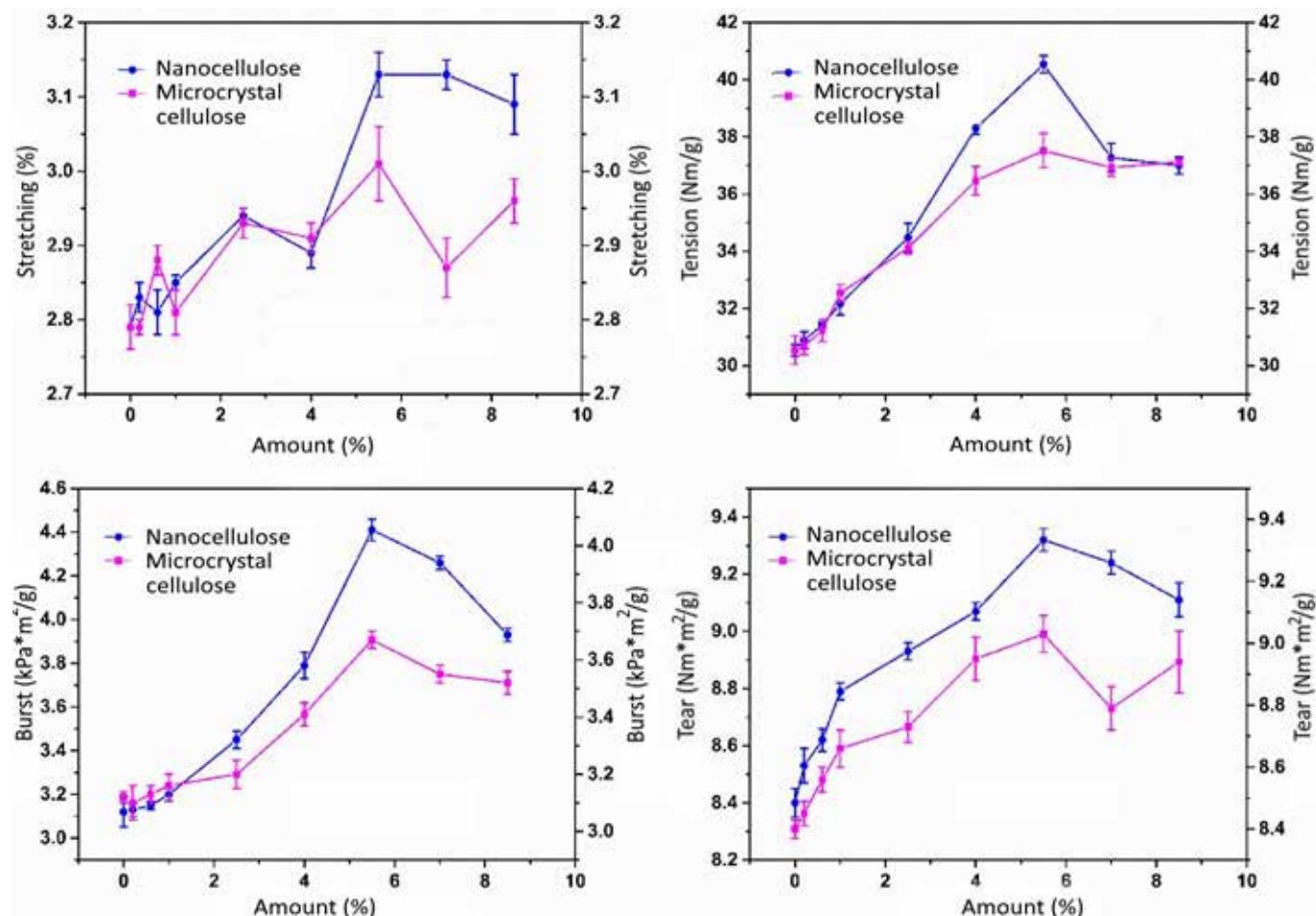


Figure 3. Effect of nanocellulose and carboxymethylcellulose content on elongation, burst and tear indices of paper sheets

The strength of paper sheets was increased by adding nanocellulose, which led to the formation of bonds between the fibers. In particular, the stretch index increased to a maximum of 5.5–6% nanocellulose, which was 32.75% higher than paper sheets without nanocellulose. The maximum values of the

tear index, tear index and elongation of paper sheets were obtained at a nanocellulose content of 5.5–6 wt. These measures were increased by 41.45%, 11.01% and 12.25% respectively. An increase in various physical properties has been achieved with the addition of nanocellulose, depending on the adhesion be-

tween the fibers. In addition, the physical properties of the paper sheets have been improved by the combination of hydrogen bonds. In addition, the relative binding area can be increased by the high specific surface area of nanocellulose to improve its physical properties. The maximum increase in physical properties is achieved by adding 5.5–6% nanocellulose. Despite this, with the addition of nanocellulose of more than 5.5–6%, the physical properties showed a tendency to decrease due to the aggregation of nanofibrous particles. The results lead to the conclusion that nanocellulose has demonstrated the ability to improve the physical properties of paper sheets.

This study also measured the effect of carboxymethyl cellulose as an additive in papermaking. On fig. 3 shows a comparison of the effect of different amounts of additives on physical properties. With the addition of 5.5–6% nanocellulose and 5.5–6% carboxymethylcellulose, the stretch index reached the maximum values of 40.55% and 37.55%, respectively. The gap index first increased and then decreased with increasing carboxymethyl cellulose content. This behavior can be explained by the increased peak pressure caused by the addition of the appropriate carboxymethyl cellulose before the paper breaks. However, adding more creates more negative charges in the system, which increases the electrostatic repulsion between carboxymethylcellulose and the fibers. Therefore, the explosibility index

of the paper sheet is lowered. The explosibility index was gradually increased to 17.65% with the addition of 5.5–6% carboxymethyl cellulose. With the addition of 5.5–6% carboxymethyl cellulose, the tear index and elongation increased by 12.85% and 7.95%, respectively.

This study clearly showed that physical properties were associated with nanocellulose and carboxymethylcellulose additives. The most obvious result of this study is that the highest tensile strength of paper sheets was obtained with the addition of nanocellulose and not with the addition of carboxymethylcellulose. For example, compared to carboxymethylcellulose, nanocellulose as an additive improved the physical properties of paper sheets. Thus, nanocellulose is a good candidate as an industrial reinforcing material to improve the physical properties of paper sheets.

Conclusion. The XRD patterns showed that the crystallinity index of the nanocellulose was higher than that of the purified cellulose, but lower than that of the untreated cellulose. FTIR spectra showed that hemicellulose and lignin were removed after chemical treatment. The thermal stability index of nanocellulose was lower than that of untreated cellulose. TEM has shown that nanocellulose has a length of 300 to 600 nm and a width of 10 to 30 nm. Nanocellulose has improved the physical properties of paper sheets. These physical properties reached their maximum values with the addition of 5.56% nanocellulose.

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