

Facile preparation of cellulose nanocrystals of kenaf fibre via acid hydrolysis method

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ABSTRACT – Kenaf fibre is one of the natural fibres commercially available and, due to its high cellulose content in a plant, there is growing interest in the use of kenaf fibre as a raw material in many applications. The main objective of this study is to synthesise and characterise the properties of cellulose nanocrystals (CNC) from kenaf fibre through the acid hydrolysis method. Using the X-ray diffraction (XRD) spectrometer, the crystalline index (CrI) and crystallite size are calculated and measured. The CNC has thus been successfully produced via acid hydrolysis method, which having CrI around 26.52–35.44 % and crystallite size around 5.46–10.94 nm. The use of CNC from kenaf fibre can be perceived as a green material that aligns well with sustainable global developments and design.

1. INTRODUCTION

Kenaf fibre or its scientific name *Hibiscus cannabinus* is one of the natural fibre that is easily available and economical amongst other natural fibre. This kenaf fibre is extracted via chemical treatment from the bast or core of a plant. It consists of cellulose (56–64 wt. %), hemicellulose (21–35 wt. %), lignin (8–14 wt. %) and small amounts of extractives and ash [1, Mahjoub 2014]. Due to high contents of cellulose in kenaf plant and others lignocellulosic sources, many studies have investigated the use of their cellulose as a raw material for many new applications such as building materials, absorbents, paper products and animal feeds [2-3].

In order to obtain cellulose nanocrystals (CNC) kenaf fibre, acid hydrolysis treatment needs to be conducted by following several conditions. Siqueira et al. (2010) describe that the acid hydrolysis method can be used to isolate CNC by removing the amorphous part of the raw material and produce a high degree of crystallinity [4]. Generally, the hydrolysis can be conducted by using a different type of acid with different combinations of concentration, time, and temperature. An experimental investigation was conducted by Zaini et al. (2013), to study the effect of different solvents on the morphology, crystallinity, and thermal stability of the resulting whiskers [5]. Based on their experimental research, two types of acid which are sulfuric acid (H₂SO₄) and hydrochloric acid (HCl) were applied.

However, limited studies have investigated the synthesising of CNC kenaf fibre by using a chemical treatment such as acid hydrolysis. Therefore, the main objective of this study is to synthesise CNC from kenaf fibre via acid hydrolysis method by different acid concentration and hydrolysis time. The CNC obtained will be further analysed and characterised by using X-ray diffraction (XRD) spectrometer.

2. METHODOLOGY

Kenaf core fibre were supplied from a local company named Malaysia National Kenaf and Tobacco Board that locates in Kelantan, Malaysia. The purification process was done by immersing the kenaf fibre powder in NaOH solution with 6 wt.% of concentration at room temperature for 24 hours. After alkali treatment, the fibres were filtered and thoroughly washed with distilled water until all traces of NaOH were removed from the fibres.

The resulted alkaline treated fibres were then subjected to acid hydrolysis process. The parameters that involved during the hydrolysis were the acid concentration of sulfuric acid, H₂SO₄ (30 & 40 wt.%), hydrolysis time (30, 60, 90 min) and hydrolysis temperature (45 °C). Different acid concentration was prepared by diluting the acid with water. Then, the suspension was constantly mixed using magnetic stirrer until the hydrolysis time was finished. The treated acid hydrolyzed cellulose was dialyzed a few times by using distilled water to a constant pH 7. Finally, the crystal structure of cellulose nanocrystals (CNC) of kenaf fibre obtained were analyzed via X-ray diffraction (XRD) analysis.

3. RESULTS AND DISCUSSION

In this study, X-ray diffraction (XRD) results were observed to investigate the effect of acid concentration used during hydrolysis and the effect of hydrolysis time. XRD analysis of cellulose nanocrystals (CNC) from kenaf fibre at different hydrolysis parameter are shown in Table 1. The crystallinity index (CrI) and crystallite size was calculated by Segal Equation and Scherer Equation respectively [6-7].

Based on Table 1, it shows that the CrI of CNC extracted from kenaf fibre increase as the concentration

acid used increase. According to Ng et al. (2015), the acid hydrolysis process will release hydronium ion (H^+) for hydrolytic cleavage of glycosidic bonds in cellulose molecular chains [8]. However, there is an insufficient amount of hydronium ion (H^+) present to react with the cellulose hydroxyls when low acid concentration is used. As the sulfuric acid (H_2SO_4) concentration increases, the CrI has increased. This is due to the presence of additional H^+ ions that removes the amorphous material. It was also found that the crystallite size of the CNC increase as the acid concentration increase. A study by Poletto et al. (2014) stated that as CrI increased, the crystallite size also increases because the increasing crystallite surface is corresponding to the reducing of amorphous cellulose regions [9].

Table 1 The crystallinity index and crystallite size of treated acid hydrolyzed kenaf fiber

Sample	Crystallinity Index (%)	Crystallite Size (nm)
CNC (30 wt. %, 30 min)	27.61	5.4651
CNC (30 wt. %, 60 min)	27.92	5.4589
CNC (30 wt. %, 90 min)	26.52	10.9412
CNC (40 wt. %, 30 min)	30.69	5.4634
CNC (40 wt. %, 60 min)	35.44	5.4729

The effect of acid hydrolysis time is also one of the important factors in cellulose hydrolysis method. As presented in Table 1, the crystallinity increases when the hydrolysis time increases from 30 minutes to 60 minutes. This is because the amorphous region was removed through acid hydrolysis. However, when the hydrolysis time is prolonged to 90 minutes, the hydrogen ions do not only remove the amorphous region but also penetrate the cellulose crystalline regions. This process will promote further hydrolytic cleavage of the glycosidic bond which, resulting in the reducing of crystallinity index [10].

4. CONCLUSIONS

As the conclusion, the cellulose nanocrystals (CNC) derived from kenaf fibre have been successfully prepared via acid hydrolysis process. Based on XRD analysis, the CNC of kenaf fibre gives a result with a crystallinity index ranging from 26.52 to 35.44 % and crystallite size ranging 5.46 to 10.94 nm.

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