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NANOCRYSTALLINE CELLULOSE EXTRACTED FROM DISCARDED CIGARETTE BUTTS

(Selulosa Nanokristal yang Diekstrak daripada Sisa Puntung Rokok)

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Abstract

The increase in cigarette consumption has led to significant accumulation of non-degradable cigarette butt (CB) waste worldwide. CBs, though small, contain both cellulose acetate and toxic tobacco substances, posing environmental risks. This includes water pollution and harm to aquatic life by releasing toxins from leachate into waterways. This study addresses the problem by exploring the extraction and hydrolysis of cellulose acetate from discarded CBs, converting it into nanocrystalline cellulose (NCC). Several conditions were examined during this study such as the ratio of water and ethanol for the extraction (1:1, 1:2, 1:3 v/v%) and the efficiency of acid hydrolysis. Analysis techniques including FTIR, TEM, ICP-MS, and XRD were employed. TEM results showed that sample C has the smallest diameter of nanocrystal cellulose with needle-like shape that corresponded to the XRD results that show the highest Crystallinity Index (CI) of 64.8% with 3.66 nm crystallite size. The treatments successfully removed toxic chemicals from the CBs that were not detected in the retained NCC using ICP-MS. The preliminary results obtained open an avenue to reuse the CB to produce environmentally friendly NCC that can be used widely in many applications fields.

Keywords: cigarette butts, nanocrystal, cellulose pulp, extraction, cellulose acetate

Abstrak

Peningkatan penggunaan rokok telah membawa kepada pengumpulan sisa pepejal rokok yang sukar untuk terurai di seluruh dunia. Walaupun saiz puntung rokok (CB) kecil, tetapi ia mengandungi asetat selulosa dan toksin tembakau yang menyebabkan masalah pencemaran. Ini termasuk pencemaran air yang mengancam kehidupan akuatik kesan daripada pelepasan toksin ke dalam salur air. Kajian ini mengatasi masalah tersebut dengan mengkaji kesan pengekstrakan dan hidrolisis selulosa asetat daripada CB, mengubahnya menjadi selulosa nanokristal (NCC). Beberapa keadaan telah dikaji seperti nisbah air dan etanol untuk pengekstrakan (1:1, 1:2, 1:3 v/v%) dan efisiensi hidrolisis asid. Teknik analisis termasuk FTIR, TEM, ICP-MS, dan XRD digunakan. Hasil TEM menunjukkan sampel C mempunyai diameter selulosa nanokristal terkecil dengan bentuk jarum yang sepadan dengan hasil XRD yang menunjukkan Indeks Kristaliniti (CI) tertinggi iaitu 64.8% dengan saiz kristal 3.66 nm. Rawatan yang dilakukan berjaya mengeluarkan bahan kimia beracun daripada CB yang dikesan menggunakan NCC yang dihasilkan menggunakan ICP-MS. Keputusan awal ini membuka peluang untuk mengguna semula CB untuk menghasilkan NCC mesra alam yang boleh digunakan secara meluas dalam pelbagai bidang aplikasi.

Kata kunci: puntung rokok, nanokristal, pulpa selulosa, pengekstrakan, selulosa asetat

Introduction

A cigarette butt (CB) acts as a filter to a cigarette. It filters the toxic substances contained in cigarettes before going through humans. CB is made up of cellulose acetate and rolled with paper. Cellulose acetate is extensively used in textiles, cigarette filters for absorbing vapors and collecting particulate smoke components, surface coatings and inks as an additive, and as a carrier in photographic negatives, motion picture film (celluloid), microfilm, microfiche, and audio tape [1]. The global market for cellulose acetate is expected to reach 1.05 million metric tons by 2017, with an anticipated price of \$1.80 per pound, according to a new analysis by Global Industry Analysis [2].

CB littering is ubiquitous in urban life and can be found abundantly in most public places especially streets, beaches, and food stalls. Studies have consistently found that a substantial majority of smokers litter CB (76.7%-84%) [3]. Even though it is small, the accumulation of CB may lead to environmental problems. Apart from being not easily degraded, the discarded CB may contain harmful chemicals such as nicotine as the major toxic substance. A smoker gets between 1.4 and 2.2 mg of the chemicals in each cigarette, with polycyclic aromatic hydrocarbons, N-nitrosamines, and aromatic amines being the most effective carcinogens [4]. The biodegradability of cellulose acetate is still questionable as some researchers reported that natural organisms could not degrade cellulose acetate with a degree of substitution of greater than 1.5. Therefore, it is not degraded by itself thus creating a big problem to dispose of the CB and recycle them.

The filter used for a cigarette is composed of cellulose acetate which is not easily degradable and may lead to the accumulation of CB on the ground and the toxins leaching out into the groundwater [5]. Recently, there have been several initiatives taken to overcome this problem such as recycling CBs to produce supercapacitors, bricks, insecticides, inhibitors, sound absorbing material [6], or cellulose pulp [5]. Toxic chemicals trapped in the CB filters that leaching out and dissolving into the water can cause considerable damage to the environment and various organisms [3]. Incinerating or landfilling are not the best

methods to dispose of cigarette butts. It incurs more costs and is challenging to dispose waste that contains a lot of organic and toxic materials in landfills. Since many toxic compounds are released into the air when the waste is burned, incinerating CBs is also an unsustainable approach [4]. Recycling CB problematic due to the toxic substances contained in the filter. Any mechanism to dispose of the filter needs to consider its impact on the environment and the feasibility of the methods. The current method has initiated recycling CB to produce nano crystalline cellulose (NCC) by using pure ethanol in the extraction. However, the maximum yield of cellulose acetate and NCC produced was not high [7]. Therefore, to increase the yield, the extraction solvent needs to be studied. It is known that ethanol can play a role in particle size and morphology, where a decrease in the aqueous solution's dielectric constant due to the addition of ethanol causes supersaturation, which in turn leads to the development of many homogenous nuclei and reduced average particle sizes [8]. Furthermore, instead of pure ethanol, aqueous ethanol can be an eco-friendly method for the extraction of cellulose [9].

Thus, this study aimed to use aqueous ethanol for the extraction of cellulose acetate from discarded CBs and convert it into nano-crystalline cellulose to reduce the environmental impact of discarded CBs. Apart from that, the heavy metals content in the recovered cellulose pulp was investigated to ensure safe usage when reused as other materials such as in biomaterial, catalysis, or polymer coating.

Materials and Methods

Materials

CBs were collected in public areas in Kuantan, Pahang. Ethanol (99.8%), sodium hydroxide pellets (NaOH), sulfuric acid (H₂SO₄), and sodium hypochlorite (NaOCl) were obtained from Merck (Malaysia). All chemicals were of analytical grade. Double distilled water was used for all experiments.

Sample preparation

Excess paper and tobacco were manually removed from the collected CB to ensure the toxic substance from the tobacco does not leach out during the extraction. Then about 10 g of cleaned CBs were weighted prior to soaking and later, they were washed with water for 24 h. Following that, the water was drained, and the cleaned CBs were gathered.

Cellulose acetate extraction and isolation of nanocrystalline cellulose

The extraction method followed previous research [7] with a slight modification. Approximately 10 g of the washed CBs (1:10 w/v%) were extracted using water and ethanol at the ratios of 1:1, 1:2, and 1:3 (v/v%), respectively, for 24 h at room temperature. After the

extraction, the solid materials were collected as cellulose acetate (CA-CB). The obtained CA-CB was bleached with 1.25 w/v% sodium hypochlorite for 6 h at room temperature. Following the bleaching procedure, the samples were dried and deacetylated for 24 hours at room temperature using 1 w/v% ethanolic NaOH. After mixing the samples, nano-crystalline cellulose (NCC-CB) was then isolated using 55 wt% of sulfuric acid at 45 °C for 45 minutes to remove the amorphous areas from the cellulose that had been produced. The percentage yield of the extracted cellulose from CB (Cel-CB) was determined using Equation (1).

% Yield of Cel – CB =
$$\frac{\text{Weight of extracted Cel-CB}(g)}{\text{Weight of CB}(g)} \times 100\%$$
 (1)

Subsequently, NCC-CB suspension was diluted 20 times its original volume at room temperature using double-distilled water. This process was repeated 5 times. Then, the NCC-CB suspension was freeze dried at -60° C for

72 h. A pestle and mortar were used to grind the resulting film (NCC-CB) to a powder and stored in sample vial for further analysis. The yield of NCC-CB was calculated using Equation (2).

% Yield of NCC – CB =
$$\frac{\text{Weigh of NCC-CB isolated}(g)}{\text{Weigh of Cel-CB used}(g)} \times 100\%$$
 (2)

Characterization

The obtained cellulose powder (NCC-CB) was characterized based on the particle size, surface, crystal shape, morphology and elemental analysis using Particle Size Distribution, FTIR, TEM, XRD and ICP-MS. These instruments were used to identify the physical properties, chemical properties, and microstructure of the NCC-CB. XRD with radiation at 45kV and 40mA provides information about crystallinity and phase composition. The samples were scanned with a step size of 0.05° and a scan rate of three seconds per step. The

$$CI(\%) = \frac{I_{crys} - I_{am}}{I_{002}} \times 100\%$$

Where I_{crys} is the maximum intensity of diffraction at the highest peak around 20° to 22° angle and I_{am} is the

Crystallite size
$$(nm) = \frac{0.94\lambda}{\beta \cos \theta}$$

Where λ is the wavelength at 0.154, β is the full width half maximum (FWHM), and θ is the corresponding

crystallinity index of NCC and crystallite size were calculated by using Equation (3) and (4), respectively. TEM provides the photomicrograph of the dimensions and microstructure of NCC-CB using high resolution transmission electron microscopy (HRTEM, JEM-2011/JEOL). ImageJ software was used to analyze the dimension of the NCC from TEM image. The functional groups of NCC-CB were confirmed using Fourier transform Infrared spectroscopy (FTIR) within a range of 4000 – 500 cm⁻¹. The elemental analysis was conducted using ICP-MS Elan 9000 (Perkin-Elmer).

(3)

minimum intensity of amorphous material around 18° angle. The crystallite size of the sample was calculated using Scherrer equation as in Equation (4):

Bragg angle [10].

Results and Discussion

Cellulose and nano-crystalline yield

Nano-crystalline Cellulose-Cigarette Butt (NCC-CB) was isolated from CB after the extraction with ethanolic sodium hydroxide, bleaching, deacetylation, and sulfuric acid hydrolysis. NCC-CB differs to Cel-CB in terms of amorphous region, whereby, in NCC-CB, the amorphous region is lesser when compared to Cel-CB. This is due to acidic hydrolysis. According to Table 1, sample A with the extraction ratio of water to ethanol 1:1 (v/v) obtained 98.76% of Cel-CB yield, sample B with the extraction ratio of 1:2 (v/v) gives 97.76% yield of Cel-CB and sample C with the extraction ratio of 1:3 (v/v) yielded 83.98% of Cel-CB. As a comparison, the yield of Cel-CB obtained by previous research [7] was at 60% with the extraction ratio of water to ethanol of 1:1 (v/v) which was lower than the yield obtained in the current study. On the other hand, for the yield of NCC-CB isolated using 55% wt. of sulfuric acid at 45 °C for 45 minutes, sample C shows the highest yield at 33.7%,

followed by sample B at 24.4% and sample A at 15.1%. The yield of NCC-CB obtained from the previous study [7] was a bit higher than the current study which was at 29.4% when using an extraction ratio of 1:1 (v/v). However, the yield of NCC-CB in this study was 33.7%, was improved with extraction ratio 1:3 (v/v) which is higher than previous studies that yielded 29.4% from discarded cigarette butt with the usage of 99.8% ethanol extraction [7], 23.5% from corncob [11], and 15-20% from olive fiber [12]. It shows that when ethanol in the extraction ratio is increased, the yield of NCC increases. Based on Table 1, it can be concluded that the extraction ratio of water to ethanol at 1:1 (v/v) gives the best yield of Cel-CB at 98.76% and the extraction ratio of 1:3 gives the best yield of NCC-CB at 33.7%. The trend of NCC increased with decreasing of the yield of Cel-CB. The yield from the treatment in Sample B and C was also higher than the yield obtained by other studies using pineapple leaves (32.1%) and sugarcane bagasse (28.6%) as a source of nanocellulose [10].

Table 1. Yield of Cellulose (Cel-CB) and Nano-crystalline cellulose (NCC-CB) from Cigarette Butt

Sample Ethanol to Water Ratio (v/v)		Yield of Cel-CB (%)	Yield of NCC-CB (%)	
A	1:1	98.76	15.1	
В	1:2	94.76	24.4	
C	1:3	83.98	33.7	

Aqueous dispersion and morphology of nanocrystalline cellulose: Correlogram of particle size

Dynamic Light Scattering (DLS) determines how randomly particles move when they undergo Brownian motion, and it is not applicable when a particle's motion is not random. Figure 1 shows the DLS measurement. From Figure 1, sample A has increased correlation at higher delay times between 100000-1000000µs which indicates sedimentation, whereas for Sample B, there is no sudden increase which indicates that there is a lower level of sedimentation happening in Sample B. In Sample C, there is an increase in correlation that happened earlier than in Sample A.

Particle Size Distribution (PSD)

The particle size distribution of nanocrystalline

cellulose (NCC) from cigarette butt is shown in Figure 2. The size distribution in Sample A shows the largest size distribution in a range 100–1000 nm with 655.4 nm indicated microcrystalline size (MCC). While in Sample B and Sample C, the largest size distribution was in the range of 10-100 nm with 41.75 nm and 30.47 nm, respectively, indicating nano cellulose crystal (NCC). The formation of size differs by the extraction ratio of water to ethanol which is conducted in Sample A, B, and C. It shows that the treatments had improved the cellulose crystal size. The Polydispersity index (PdI) for Sample A, B and C were 0.859, 0.400 and 0.410, respectively. High reading of PdI may result from a sample containing large particles reflected in Sample A. Samples B and C have the lowest PdI which corresponds to Sadeghifar [13], their NCC extracted with HBr of 1M gives PdI of 0.41 reflected small particles. In conclusion,

the extraction ratio of 1:2 and 1:3 (v/v) used for samples B and C improved the size distribution of NCC and this

corresponds to the yield of NCC in Table 1.

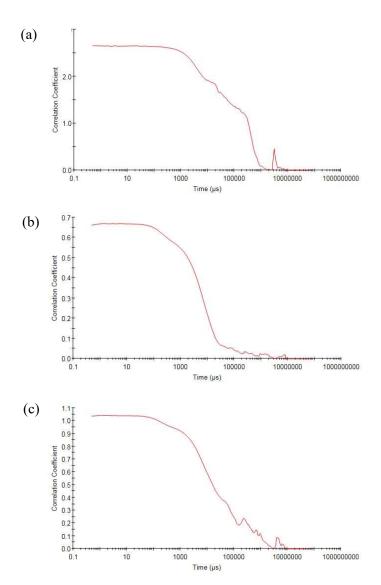


Figure 1. Dynamic light scattering (DLS) measurement of a) Sample A, b) Sample B and c) Sample C of NCC-DCB

Transmission electron microscope (TEM)

The crystallinity and morphology of the NCC-CB produced from this study were analyzed using TEM to analyze the nanometer size samples. Figure 3 shows the TEM micrograph for Sample A, B and C that varied the extraction ratio of water to ethanol 1:1, 1:2, and 1:3 (v/v), respectively. The TEM shows obtained nanocrystals cellulose resulting from sulfuric acid hydrolysis. The amorphous portion of cellulose microfibrils was

predicted to be cleaved transversely by the acid hydrolysis treatment under regulated conditions, preserving the straight crystalline domains. Eventually, the treatment should cause the fibers' size to decrease from the micron to the nanometer scale [14]. Here it can be seen that the nanocrystals cellulose is rod in shape for Sample A and needle-like in shape for Sample B and C with a reduction in size due to the treatments, respectively. The observed behavior may be explained

by the fact that ethanol creates a layer and adsorbs more strongly than water on the surface, preventing agglomeration during crystallization [8]. The length and diameter range of Sample A is found to be in a micro size which corresponded to 50% of ethanol extraction, contrary to Sample B and C, where the size of the cellulose crystals is found to be in the nanometric range which corresponded to 66% and 75% of ethanol extraction. From Figure 3(b) and 3(c), the formation of nanocrystalline requires further extraction ratio to have complete shape and size of NCC as in other studies. However, other studies that used other sources of

cellulose such as sugar palm fiber [15], pine wood and corncob [11] obtained smaller size than that was obtained in this study that due to higher exposure to more concentrated sulfuric acid (>60%) on the cellulose fiber. According to Yu [14], their nano-crystals diameter is in the range of 15–20 nm with different method of preparing NCC which only involves alkali treatment with NaOH, using aqueous chlorite to bleach, and acid hydrolysis with 10 molL⁻¹ of sulfuric acid. The size 170 nm in diameter nanocrystals were isolated from sugarcane bagasse which was extracted with 17.5% w/v of sodium hydroxide solution [16].

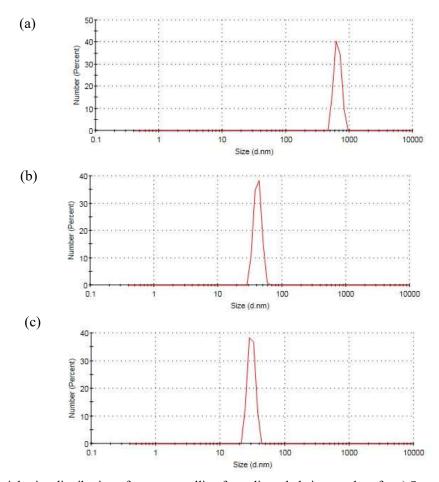


Figure 2. Particle size distribution of nano-crystalline from discarded cigarette butt for a) Sample A, b) Sample B and c) Sample C

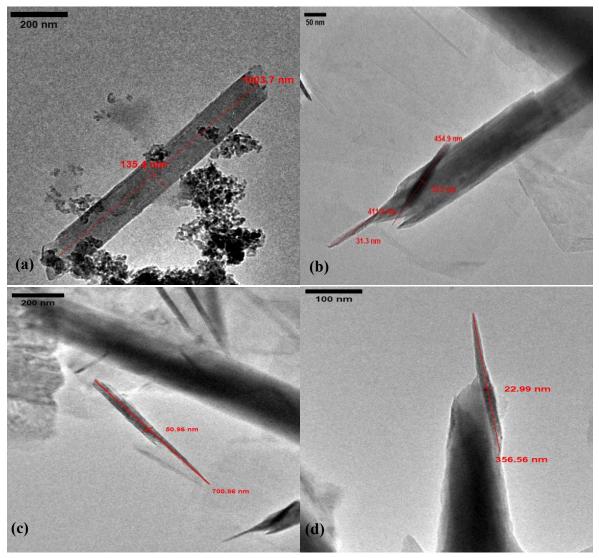


Figure 3. TEM image of nanocrystalline cellulose from a) Sample A; b and c) Sample B; and d) Sample C

The aspect ratio shown in Table 2 for this study indicated that the treatments did not interfere with the formation of the crystal. The aspect ratio is increasing as the ratio is increasing. The aspect ratio for treatment in Sample B and C exhibited more than ten (Table 2) which indicated that the isolated NCC has a mechanical stability for polymer composites synthesis corresponding to the minimal aspect ratio of more than ten to achieve optimal stress transmission during the fiber-matrix interaction[17].

Fourier-transform Infrared Spectroscopy (FTIR)

The successful isolation of the freeze-dried NCC-CB

from cigarette butt was confirmed by FTIR. All three samples show a broad band at 3320 cm⁻¹ in the absorbance regions of O–H with increasing intensities as the NCC yield increased with the treatment extraction ratio (Figure 4). The O-H stretching vibration of the O-H groups in cellulose molecules is shown by a broad band in the range of 3100 to 3600 cm⁻¹ that is visible in the FTIR spectra of every sample. The spectra were compared with the cigarette butt extract without treatment that showed the two main absorbance regions at 3338 cm⁻¹ and 1634 cm⁻¹, respectively, that indicate band for O–H and –NH group [18]. After the treatments for Sample A, B, and C there were no sign of C=O at

1743 cm⁻¹ and –NH group as in untreated cigarette butt in the orange line in Figure 4. This shows that the treatments in this study can remove ketone and amine [7], [18]. The spectra of sample A, B and C show a C–H stretching band at 2890, 2880, 2894 cm⁻¹, respectively. Furthermore, a peak in band detected at 1363, 1368,1370 cm⁻¹ is related to the bending vibration of the

C–H and C–O bonds in the polysaccharide aromatic rings [19]. In addition, the absorption peak was linked to aromatic C–C in the plane symmetrical stretching vibration of aromatic rings seen in lignin at 1604 cm⁻¹ in the untreated sample. These bands were no longer present in the FTIR spectra of the current NCC.

Table 2. The dimension and aspect ratio for sample with different extraction ratio treatment

Samples	L, length (nm)	D, diameter (nm)	Aspect ratio (L/D)
A	1003.7	135.4	7.413
В	454.9	35.2	12.923
	411.4	31.3	13.143
	700.3	50.9	13.731
C	356.6	23.0	15.504

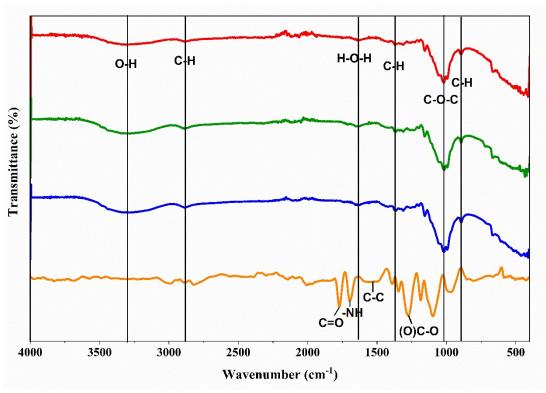


Figure 4. FTIR spectra of Sample A (red line), B (green line), C (blue line), and untreated cigarette butt (orange line)

Finally, the band peaks at 1016, 1018 and 1016 cm⁻¹ for sample A, B and C respectively, due to the stretching vibration of the C–O–C pyranose ring, which is antisymmetric in phase. The most important absorption band is at 800–950 cm⁻¹ (which is associated with the glycosidic linkages between glucose units in cellulose)

[19] when comparing with the FTIR result obtained previously [7], 3320 cm⁻¹ (O–H), 2900 cm⁻¹ (C–H), 1646 cm⁻¹ (H–O–H), 1437 cm⁻¹ CH2, 1371 cm⁻¹ (C–H), 1018 cm⁻¹ (C–O–C) and 898 cm⁻¹ (C–H) with the extraction of ethanol to water of 1:1 (v/v). This shows that the FTIR results for sample A, B and C have the

same absorption band to prove that it is NCC chemical structure, and the ratio of extraction did give little impact on FTIR results.

X-ray powder diffraction (XRD)

The XRD patterns of the NCC-CB in Sample A, B and C show a characteristic of cellulose III structure with a wide peak around $2\theta = 12^{\circ}$ (101), 20° (10 $\overline{1}$), 22° (002) and 25° (111) (Figure 5) similar shape with other NCC obtained from cigarette butt [7]. After acid hydrolysis, the isolated NCC-CB Crystallinity Index (CI) and crystallite size were calculated using Equation 3 and 4, respectively as shown in Table 3 for sample A, B and C. This data is lower than the data obtained previously [7] using CBs as the cellulose source, which is 94.5% for NCC. However, for this study, the CI was increasing as

the treatment increased the extraction ratio. When referring to the untreated cigarette butt, the CI was 28.26% which was due to the amorphous region in the cellulose being removed during the acid hydrolysis [7]. There is no significant difference between all three samples. Sample A had the highest peak is highest at 22° whereas Sample B and C recorded the highest peak at 20°. This is due to the conversion of cellulose I to cellulose III as the extraction ratio increased [20]. Sample C shows the highest crystallinity Index (CI), 64.8% with the smallest crystallite size 3.66 nm. This suggests that the extraction ratio does not have a negative effect on crystallite of cellulose. Further, an increase in the extraction ratio can be extended to obtain more CI.

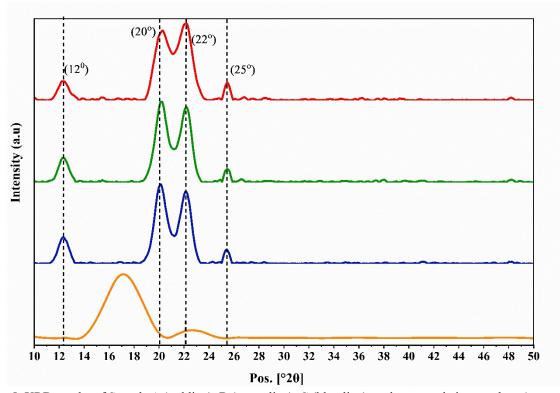


Figure 5. XRD results of Sample A (red line), B (green line), C (blue line), and untreated cigarette butt (orange line)

Table 3. Crystallinity index and crystallite size of nanocrystalline cellulose from cigarette butt

Sample	Crystallinity Index (%)	Crystallite Size (nm)
A	44.9	4.25
В	48.6	3.99
C	64.8	3.66

Inductively coupled plasma mass spectrometry (ICP-MS)

CB can contain toxic chemicals that come from cigarettes as it functions to filter the chemicals from getting into the human body. To reuse the CBs as a source of cellulose for NCC, the contents need to be examined. The metal contents in the powder NCC-CB were analyzed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and compared with the previous study [18] as shown in Table 2. Based on Table 2, Sample A has most metal elements from the discarded CB due to the inappropriate extraction ratio during the isolation of the NCC-CB. Contrary to Sample B and Sample C, the metal contents were reduced and most of them were not traceable. The amount of heavy metal content reduced with increasing extraction ratio. This shows that the heavy metal contents in the cigarette butt

can be removed, and this opens an avenue to recycle the waste in an environmentally safe way.

As the treatment ratio increased it improved the content in the CB and enhanced the CI of the isolated NCC from CB, and it is confirmed by the formation of the NCC-CB shown in Figure 3(b) and 3(c) for Sample B and Sample C. This shows that the samples contain high percentage of nano-crystals cellulose [21]. Apart from that, heavy metal contents in the discarded CB were removed successfully using all the extraction ratios, indicated by lead, nickel, copper, arsenic, cadmium, cobalt, manganese, and titanium were not traceable in all samples. However, further treatment needs to be done to remove or reduce other metals such as aluminum and iron [17].

Table 2. Elements analyzed by ICP-MS for Sample A, B, and C and compared with the metal leached from untreated cigarette butt [18]

	Concentration (ppm)				
Elements	Sample A	Sample B	Sample C	Untreated Cigarette butt [18]	
Na	368	139	60.9	239	
Mg	100	69.4	64.1	620	
Al	1320	720	323	950	
Pb	-	-	-	2.39	
K	162	40.6	20.9	929	
Ca	540	417	559	766	
V	2.53	-	1.52	-	
Cr	21.6	11.1	6.99	85	
Fe	403	194	154	590	
Ni	3.02	-	-	2.90	
Cu	2.12	4.66	-	11	
Zn	27.4	21.8	16	35.3	
As	1.54	-	-	-	
Ag	-	1.94	7.49	0.18	
Cd	-	-	-	0.99	
Co	-	_	-	0.04	
Mn	-	_	-	178	
Ti	-	-	-	106	
Ba	-	-	-	48.7	
Sr	-	-	-	39.6	

Conclusion

In conclusion, reusing CB to isolate NCC can be a solution to the CB littering problem. The NCC can be used further in polymer industries. The NCC isolated in this study using water to ethanol of 1:3 (v/v) gives the best results of crystallinity index with 64.8%,

polydispersity index of 0.40, and crystallite size 3.66 nm with needle-like shape showing the improvement in the treatment. The NCCs yield was considerably great between 24-34% and MCC yield was 15%, by only utilizing more aqueous ethanol (1:3 v/v), proving the preparation method was environmentally friendly. The

chemical structure of NCC in FTIR spectra of sample A, B and C present similar bands, indicating the same chemical structure for all the NCC. ICP-MS was conducted to determine the content of metal found in the samples and aluminum and iron were found to be the highest. Thus, further treatment needs to be done with aqueous ethanol (1:3 v/v) and sulfuric acid treatment to improve the NCC isolated from the CB to use in various application fields especially in polymer and medical. The results obtained here suggest that CB is capable of being one of the sources of cellulose to produce NCC with the advantage of being easily biodegradable and environmentally friendly.

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