Microcrystalline Cellulose Generation from Laboratory Cotton Waste and Conventional Characterizations

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Abstract

Microcrystalline cellulose (MCC) is a green material that has widespread applications in pharmaceuticals, food, cosmetics, and other industries owing to its biocompatibility, biodegradability, hydrophilicity, and acid-insolubility. Therefore, this study presented a simple, fast, and cost-effective approach for preparing MCC from laboratory cotton via alkaline treatment and sulfuric acid hydrolysis. Further, the synthesized cotton-based MCC was characterized using FTIR, XPS, and EDX. Based on these results, the major components were identified as carbon and oxygen. This finding was evidenced by the FTIR analysis, which displayed peak wavelength at 3446.94 cm^{-1} , 2891.11 cm^{-1} , 1649.50 cm^{-1} , 1380.1 cm^{-1} , 1061.19 cm^{-1} , and $1050\text{--}1150\text{ cm}^{-1}$. The surface morphology was also examined by FESEM and FETEM, which showed that the prepared MCC has a smooth surface and a consistent, rod-like shape. In addition, the MCC exhibited the typical diffraction peaks of a crystalline structure of cellulose II at 12.2° , 20° , and 22.03° , which correspond to the diffraction planes of 1–10, 110, and 020, respectively, and had a crystallinity index of 78.7%. Moreover, the prepared MCC had a diameter of $37.80\text{ }\mu\text{m}$ and exhibited good stability with a peak at -76.51 mV . Further, the cotton-based MCC exhibited high thermal stability, as revealed by the TGA.

Introduction

Cellulose is a widely available biopolymer and has received enhancing attention because of its biosynthesis, chemistry, and ultrastructure (Fattahi et al. 2014; Suk et al. 2019; Gobalu et al. 2021; Lim et al. 2021; Vasudevan et al. 2021). As a natural polymer, cellulose is renewable, eco-friendly, inexpensive, non-toxic, biodegradable, and biocompatible (Olivera et al. 2016; Gunny et al. 2021). It is an organic carbohydrate component, composed of repeating β -D-glucopyranose units that have 3-hydroxyl groups/anhydroglucose unit, yielding a high degree of functionality for cellulose molecules (Gopinath et al. 2016; Kusumattaqiin et al. 2015; Yuen et al. 2019). As shown in Fig. 1a, the basic structure of cellulose exhibits a crystalline region depending on the hydrogen bonding, and can also exist in an amorphous state. Moreover, cellulose is usually insoluble in most solvents and has hydrophilic properties owing to the presence of hydrogen bonding. Recently, it has become a popular research topic as a green biomaterial owing to its excellent mechanical and chemical characteristics (Song et al. 2016). Further, it can be easily obtained from plants (e.g., cotton, flax, abaca, coir, jute, hemp, kenaf, and ramie), wood, bacteria, algae, and marine fauna (e.g., tunicates) (Li et al. 2015). Currently, plants are the main source of cellulose—which is present in plant cell walls.

Microcrystalline cellulose (MCC) is applied widely in cosmetics, plastic, food, pharmaceuticals, and other industries because of its biocompatibility, hydrophilicity, biodegradability, and acid-insolubility (Widiarto et al. 2017). MCC is a cellulosic byproduct consisting of monomer D-glucose monomer linked with 1,4- β -glycosidic linkages. “Microcrystalline” refers cellulose chains in natural polymers; a region with a high order degree is named microcrystalline, whereas a region with a low order degree is named amorphous (Haque et al. 2015). Various studies have extracted MCC on a large scale from cotton and wood cellulose using dilute mineral acids (Adel et al. 2010).

MCC can be prepared by different methods depending on the type of cellulosic material, including acid hydrolysis, enzyme-mediated methods, explosive autohydrolysis, and alkaline oxidation (Kushnir et al. 2015; Kusumattaqiin et al. 2015). MCC is normally prepared via the acid hydrolysis method, which uses a strong acid, such as HCl and sulfuric acid (H_2SO_4), as an acid catalyst to eliminate the amorphous part of the cellulose to yield particles consisting of microcrystals (Kusumattaqiin et al. 2015; Suryadi et al. 2017). Acid hydrolysis has many advantages, including low cost, short duration, continuous process, limited quantity of consumed acid, and fine particles of fine products (Chauhan et al. 2009; El-Sakhawy et al. 2007).

Therefore, in the present study, MCC was prepared from laboratory cotton waste using two steps: alkaline treatment and acid hydrolysis. The cotton was treated with an alkaline solvent sodium hydroxide to remove impurities, such as hemicellulose and lignin, before acid hydrolysis. Then, the treated cotton was hydrolysed with acid to decrease the size of the crystalline cellulose. When the cellulose polymer treats with the acid solution, the β -1,4-glycosidic bond is broken; subsequently, the chain is hydrolyzed, as shown in Fig. 1b. This process reduces the degree of cellulose polymerization. In addition, the prepared MCC was characterized using morphological and structural analyses.

Experimental

The cotton was originally obtained from a local market (Perlis, Malaysia) for use in a laboratory as the source material for MCC production. H_2SO_4 (95%) and sodium hydroxide (NaOH) pellets were purchased from Sigma-Aldrich (USA). Carbon-coated copper grids were purchased for the TEM analysis from Electron Microscopy Sciences (Hatfield, UK). Morphological analysis was performed to characterize the MCC using FESEM and FETEM, and structural analyses were performed using FTIR, EDX, XPS, XRD, TGA, zeta potential, and a particle size analyser.

Production of MCC Powder: Alkaline and Acid treatments

The laboratory cotton waste was cut into small pieces, which were turned MCC powder after treated with NaOH (treatment with alkaline) and H_2SO_4 (acid hydrolysis). Specifically, the small pieces of cotton were added with NaOH (5%) and stirred constantly for 4 h at room temperature to obtain a homogenous mixture. Then, the alkali treated MCC powder was filtered and then washed by distilled water many times to reach the neutral pH. This was done to eliminate lignin and hemicellulose impurities in the laboratory cotton. Next, the filtrate was dried at 80°C in an oven for 24 h (Theivasanthi et al. 2017).

The alkali-treated dried cotton samples were further treated by H_2SO_4 . Specifically, the alkali treated sample was mixed with 35.5 M of a concentrated H_2SO_4 solution and stirred constantly at 40°C for 60 min to obtain a well-mixed solution. After mixing, cold water was added to stop the hydrolysis reaction and then suspended and washed by distilled water until the neutral pH. The suspended (bottom) layer was centrifuged for 15 min with the speed of 10,000 $\times g$ for 15 min and then the suspended MCC was dried at 80°C in an oven for 24 h to get the powder form of MCC (Theivasanthi et al. 2017).

Surface Characterization of MCC: Morphology analysis

The morphology of the MCC samples was characterized using FESEM (Hitachi, S-4300SE, Japan), which scanned the sample at high-energy beams (15 kV). The morphologies of the samples were examined using FETEM (JEM 2100F, Jeol, Japan), in which the samples were prepared by dropping the MCC solution on a carbon-coated copper grid and dried.

Surface Characterization of MCC: Surface chemistry analysis

The synthesized MCC was further characterized using structural and functional analyses. An FTIR analysis (Vertex 80V, Bruker, Germany) was employed to find the functional groups, record the emission spectrum of the MCC sample, and a KBr pellet was added to the sample to study the FTIR spectra in the range of $500\text{--}4000\text{ cm}^{-1}$. Absorption was identified with various stretching modes of functional groups between 1500 cm^{-1} and 4000 cm^{-1} . Meanwhile, the peaks below 1500 cm^{-1} often denote bending, ring vibrations and deformation are commonly used as the fingerprint region of the spectrum. Moreover, FESEM equipped with EDX analysis (EDAX, USA) was done to confirm the elements in the synthesized MCC at an accelerating voltage of 15 kV.

XPS (Thermo Scientific, K-Alpha, UK) with monochromatic Al K α micro-focused was utilized to identify the elemental composition on the surface of the MCC. XPS was also used to analyse the chemical composition of the samples from the outer 10 nm of the surface. The synthesized MCC was subjected to XRD (Rigaku, DMAX-2500, Japan) with a Cu-K α wavelength of $1.5406\text{--}1.5444\text{ \AA}$, and a voltage of 40 kV and a current of 100 mA. The sample was scanned at a rate of $2^\circ\text{C}/\text{min}$ in a Cu radiation source at $2\theta = 5^\circ - 50^\circ$. The crystallinity index (Crl) was calculated with SmartLab Studio II software (Rigaku Corporation, Japan).

The thermal stability of the MCC was verified by a progressive enhancement in temperature in the TGA (TG 209 F3 Tarsus, Netzsch, Germany). About 6.30 mg of each synthesized MCC was placed in an alumina pan and heated from 30°C to 600°C at a rate of $10^\circ\text{C min}^{-1}$. The size distributions and particle dispersion stability were analysed by using particle size analyser (ELS2-2000, Otsuka Electronics, Japan) and dynamic light scattering (DLS) with a zeta potential.

Results And Discussion

Production of MCC from laboratory waste cotton

The naturally obtained cellulose material is composed with glucose units connected by β -1,4 glycosidic bonds (Figs. 1a and b). To prepare MCC from the laboratory cotton waste, the samples were first treated with NaOH for alkaline treatment and then H_2SO_4 for acid hydrolysis (Figs. 1ci-iii). Next, the treated samples were dried at 80°C (24 h) to obtain the MCC powder (Figs. 1iv and v). The synthesized MCC was further characterized by morphological analyses to examine the size, surface, and surface structure to

confirm the chemical composition, functional group, thermal stability, particle size distribution, and stability of the MCC.

Characterization of MCC: Morphology analysis

A morphological analysis of the MCC sample was done utilizing FESEM and TEM. The FESEM results revealed a uniform particle size distribution and rod-like structure particles (Figs. 2a and b). Further, the shape of the particles increased the surface area and increased the reactivity of the fiber. Natural MCC was used to determine the surface morphology of the prepared MCC to get the uniform size of particle, and a slightly rough formation was observed because of the chemical treatment during the delignification process. The surface morphology of the prepared MCC was further examined using FESEM with accelerated electrons under 15 kV of energy. Note that MCC is produced by breaking the fibers via acid hydrolysis (Kale et al. 2017). The results showed a particle size range of 22.1 μm to 37.2 μm with an average size of 29.4 μm . FETEM was also used to study morphology at different magnification scales. The results revealed irregularly shaped particles with individual molecules in different ranges (Figs. 2c and d). Moreover, at a higher magnification, the cotton-based MCC shape was different from the FESEM image

Surface chemical bonds analysis: FTIR

The FTIR spectra of the MCC sample were examined in the range of 500–4000 cm^{-1} (Fig. 3a). According to the infrared spectra of the MCC, O – H stretching was observed at 3446.94 cm^{-1} and C = O carbonyl ring stretching occurred at 1649.50 cm^{-1} . Thus, the alkali treatment decreased the hydrogen bonding of cellulose by removing hydroxyl groups (–OH) when reacting with NaOH, whereas the C – H and C – O bonds of cellulose exist in the polysaccharide aromatic rings (Theivasanthi et al. 2017). C – H stretching in the cellulose structure was noticed at 2891.11 cm^{-1} , whereas C – O stretching was noted in the range of 1050–1150 cm^{-1} . Moreover, a C – O–C pyranose ring in cellulose occurred at 1061.19 cm^{-1} . Further, the peak at 1381.10 cm^{-1} confirms the presence of cellulose in the MCC sample. Moreover, the peak at 900 cm^{-1} occurred because of the glycosidic linkage between the sugar units (Trilokesh and Uppuluri 2019).

Elemental analysis: EDX

EDX equipped with FESEM was employed to find the chemical composition of the MCC. The EDX spectra peaks of MCC correspond to the energy levels of C and O, as shown in Fig. 3b, wherein C and O are major peaks at 0.3 KeV and 0.6 KeV, respectively, indicating the adequate synthesis of MCC from the laboratory cotton waste. The percentages of O and C were 57.47% and 42.53%, respectively, in the MCC.

Surface Elemental Composition and Chemical State: XPS

XPS analysis was used to find the chemical composition of the MCC surface (Figs. 4a-c). The results clearly show that O and C are the dominant peaks at binding energies of 531.74 eV and 284.68 eV,

respectively. This confirms that the synthesized MCC mainly consisted of O and C, which are the major elements in cellulose.

Crystallographic Structure Analysis: XRD

XRD was used to elucidate the crystallinity of the MCC. Generally, the polymer material of MCC is semi-crystalline because it still contains an amorphous part in addition to the dominant crystalline parts (Alavudeen et al. 2017). Crystalline polymers produce sharp peaks, whereas amorphous polymers tend to produce blunt or widened peaks (Fig. 5a). The MCC created from laboratory cotton exhibited the typical diffraction peaks of a crystalline cellulose II structure at 12.2° , 20° , and 22.03° , which represent the diffraction planes of 1–10, 110, and 020, respectively (French 2014). After alkaline treatment and acid hydrolysis on cotton, the cellulose I peak 22.03° (020) was split into two peaks 20° (110) and 22.03° (020) indicating the formation of cellulose II (French 2014; Trilakesh and Uppuluri 2019). The CrI of MCC was reported as the percentage of crystallinity calculated according to the diffraction patterns. Based on the diffraction patterns, the CrI of the prepared MCC was decreased as 78.7% after alkaline treatment and acid hydrolysis, as compared with that of cotton (81.2%). This decrease in CrI is caused by the conversion of the structure from cellulose I to cellulose II, which is associated with the destruction of the cellulose I structure in cotton by molecular chain cleavage and the subsequent reformation of the crystalline structure to cellulose II (Yue et al. 2013).

Thermal Stability: TGA

The thermal stability of MCC was confirmed using a TGA. The TGA curves of the synthesized MCCs are shown in Fig. 5b. The initial weight loss of the materials began at 50°C – 100°C because of the evaporation of moisture on the sample surface (Haque et al. 2015; Nasution et al. 2017). Meanwhile, the thermal decomposition of MCC began at 250°C to 370°C , accounting for a total weight loss of 70%. These results indicate that the MCC created from laboratory cotton has high thermal stability.

Stability and Size Distribution: Zeta potential and particle size analysis

A zeta potential analysis was used to elucidate the stability of the particles in the MCC sample by measuring the surface charge of the particles in the solution. The zeta potential for the MCC was -76.51 mV, which indicates the stability of the particles (Fig. 6a). This value is consistent with that reported by Mahajan et al. (2014). Thus, MCC is suitable for the encapsulation of enzymes and other molecules. In addition, a particle size analyser was used to examine the MCC particle size. When MCC is dispersed in a solution, it exhibits Brownian motion, in which smaller particles exhibit faster motions. The scattered light from the particles displays fluctuations corresponding to individual particles when the laser light illuminates the particles under the effect of Brownian motion. This fluctuation is detected using the pinhole-type photon detection method and can determine the particle size and particle size distribution. The particle size of MCC had a diameter of $37.80\text{ }\mu\text{m}$, as shown in Fig. 6b, with a polydispersity index (PDI) of 0.304. The low PDI value of the prepared MCC indicates a narrow particle size distribution,

suggesting an even particle size. Therefore, this study confirmed that laboratory cotton could be used as a potential raw material for isolating MCC for use as a green material in many industrial applications.

Conclusions

Based on the result of this research, laboratory cotton waste is a potential source for the instant preparation of MCC. Specifically, this research revealed that MCC can be prepared from laboratory cotton quickly and easily via alkaline treatment and H_2SO_4 hydrolysis. The synthesized MCC was characterized using morphological and structural analyses, and the major components of the sample were confirmed using FTIR, XPS, and EDX. Moreover, the FTIR peaks of MCC were observed at 3446.94 cm^{-1} , 2891.11 cm^{-1} , 1649.50 cm^{-1} , 1380.1 cm^{-1} , 1061.19 cm^{-1} , and $1050\text{--}1150\text{ cm}^{-1}$, indicating it is a cellulose material. The surface morphology of the prepared MCC was examined using AFM, FESEM, and TEM, which revealed that the prepared MCC has a smooth surface and a consistent, rod-like shape. In addition, the synthesized MCC exhibited the typical diffraction peaks of the crystalline structure of cellulose II at 12.2° , 20° , and 22.03° , which represent the diffraction planes of 1–10, 110, and 020, respectively, and the CrI was 78.7%. Based on the results of zeta potential and particle size analyses, the cotton-based MCC with a diameter of $37.80\text{ }\mu\text{m}$ displayed excellent stability, with a peak at -76.51 mV . Moreover, the synthesized MCC had high thermal stability. Therefore, this study concludes that laboratory cotton is a suitable source for preparing MCC as a green material for various field applications.

Declarations

Acknowledgement

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Conflict of interest None.

Ethical standards This study does not involve human participants, animals, and potential conflicts of interest.

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Figures

Figure 1

(a) The structure of cellulose; (b) Acid hydrolysis of cellulose into microcrystalline cellulose. (c) Synthesis of microcrystalline cellulose from laboratory cotton. (i) Cotton wool; (ii) After alkali treatment; (iii) After acid hydrolysis; (iv) Microcellulose; (v) MCC powder.

Figure 2

Morphological analysis of the MCC. a) FESEM was examine at 100 μm magnification; b) FESEM was examine at 20 μm magnification; c) FETEM – scale bar at 1 μm; d) FETEM – scale bar at 500 nm.

Figure 3

a) FTIR spectrum of MCC, which explain the chemical bond examined in the MCC, b) EDX analysis of MCC shows the presence of elements.

Figure 4

XPS pattern of MCC from laboratory cotton. a) Survey scan, b) Carbon C1s, c) Oxygen O1s.

Figure 5

a) XRD pattern of MCC from laboratory cotton. b) Thermal analysis of MCC. The TGA curve indicate the reduction of weight as the temperature increased from 270 to 600 $^{\circ}\text{C}$.

Figure 6

a) Zeta potential of MCC from laboratory cotton. b) Dynamic light scattering of the MCC.