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Research Article

Novel Chemo-Mechanical Production of Nanocellulose from Coconut Pith

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ABSTRACT

Nanocellulose has been receiving tremendous attention due to its unique properties such as high strength, flexibility low density, biocompatibility, and natural hydrophilicity with numerous hydroxyl groups effortlessly modified for different purposes. Lignocellulosic biomass, especially coconut residues like coconut shells, coconut fibers, coconut husks, etc., has been effectively utilized for cellulose production in various sizes and morphologies such as microcrystalline cellulose, nanocellulose crystals, nanocellulose fibrils. In this study, novel chemo-mechanical production of nanocellulose from coconut pith (CP) is developed by implementing two-stage alkaline pretreatment, ball milling, acid hydrolysis, centrifugation, and dialysis. Nanocellulose obtained from CP is evaluated for its particle size distribution, zeta potential, chemical composition, crystalline and chemical structure. All parameters of ball-milling time, solid-to-liquid ratio, and hydrolysis duration greatly affect the formation of cellulose nanoparticles from CP as well as the stability of the particle system. Based on the experimental results, the cumulative intensity of particle size below 300 nm and in the range of 300 – 600 nm is respectively 40.93% and 59.08%, indicating the evident effectiveness of the developed chemo-mechanical approach in nanocellulose synthesis from cellulose-rich CP with an initial high holocellulose content of 49.07%. Remarkably, the crystallinity index of our nanocellulose from CP is as high as 72.08% compared to that from coconut husks. The present work demonstrates the potency of combining ball milling and acid hydrolysis in stable nanocellulose synthesis from CP and lays the foundation for further investigation to apply the formulated technique in industrial production.

Key words: Coconut Pith, Nanocellulose, Chemo-mechanical Process, Ball Milling, Acid Hydrolysis

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INTRODUCTION

Cellulose, which is commonly found in plants and microorganisms, is the most abundant polymer on Earth. The chemical structure of cellulose consists of repeating β-D-glucose monomers linked by β-(1,4) glycosidic bonds. Natural cellulose chains are typically bundled together to produce a fibrous morphology with highly ordered crystal and amorphous regions that are consequently isolated as cellulose-based nanoparticles for useful applications in futuristic materials[1]. Recent efforts have focused on extracting cellulose from lignocellulosic biomass and converting it to nanoscale materials having different morphologies of crystals, fibers, rod-like shapes, and spheres. Nanocellulose has emerged as a prominent and outstanding material due to its outstanding properties such as extremely high crystallinity, low density, excellent mechanical strength, high surface area-to-volume ratio, biocompatibility, hydrophilicity, and ease of chemical modification depending on the application orientation[1]. As a result, nanocellulose has been extensively studied in varied advanced materials such as biomedical devices, food and fruit packaging films, textiles, Pickering emulsifiers, energy storage, paper transistors, solar cells, and even wastewater treatment sorbents[1,2].

Agriculture plays a crucial role in developing countries like Vietnam, India, Indonesia, and Malaysia. Since the worldwide population continues to grow, the demand for food production increases, and subsequently, the capacity for agricultural waste also substantially rises. These residues contribute to various environmental problems as the current solution involves burning them on-site or landfilling them, leading to greenhouse gas production of methane, carbon monoxide and dioxide (COx), and polycyclic aromatic hydrocarbons, to name a few[3]. These by-products must be recycled and processed into renewable high-performance materials not only to minimize the emission of environmental pollutants but also to increase the value of agriculture. Nowadays, coconut palm is planted in more than 90 countries with a cultivation area of up to 12.25 million hectares and consumption of fresh water, coconut oil, coconut milk-based foods, nuts, soap, cosmetics, and margarine[4]. In Vietnam, coconut ranks fourth among

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perennial industrial crops, following rubber, pepper, and cashew.

Coconut pith (CP) is a valuable by-product derived from post-harvest coconut processing. It is obtained during the separation of coconut fibers from coconut shells and constitutes approx. 70% of the weight of the shells. CP is presently utilized to nourish plants and enrich soil, making it a popular choice in modern farming, landscaping, and greenhouse cultivation. The major composition of lignin (35-50%), hemicellulose (2-6%), and cellulose (24-30%) in CP varies among different species and cultivated regions[5]. The presence of hydroxyl, carboxyl, ether, phosphate, and amino groups within its constituents[6] makes CP a potential source of raw materials to recover value-added products, synthesize nanocellulose, and be functionalized for a variety of applications.

A few previous studies reported the ability to fabricate nanocellulose from CP. The common method used in nanocellulose fabrication from CP is acid hydrolysis to dissolve the amorphous regions of the cellulose chains and retain the acid-resistant crystalline regions. Subha et al. utilized a mixture of HCl and H_2SO_4 at a solid-to-liquid ratio of 1:40 g/mL for 8 h to synthesize and purify nanocellulose from CP. The resulting nanocellulose exhibited the total crystallinity index of cellulose I and cellulose II up to 83.7% and an average size of 144.22 nm based on scanning electron microscopy[7]. Later, Kumar et al. synthesized cellulose nanocrystals from CP by applying absolute H_2SO_4 hydrolysis on pretreated CP-derived pulp. The crystallinity index of the as-fabricated nanocellulose was around 75.17%. The morphology of the nanocellulose showed its rod-like structure with an average width and length of 12-20 nm and 112-308 nm, respectively[8].

The typical methods for producing nanocellulose from lignocellulosic biomass involve chemical, mechanical, and enzymatic treatments, either individually or in combination[9]. To the best of our knowledge, there has been no research work in developing a green chemo-mechanical procedure for fabricating nanocellulose from CP without the consumption of harsh ingredients and investigating the influence of synthesis parameters on the production efficiency and characteristics of the nanosystems. In this work, the combination of ball milling and acid hydrolysis at a low concentration of 64.0% has been studied for the first time to synthesize a stable nanosystem from pre-treated pulp of CP. Different aspects, including particle size distribution, zeta potential, crystal and chemical structure, and holocellulose content of the intermediate products as well as final nanocellulose are comprehensively evaluated by advanced analysis methods.

MATERIALS AND METHOD

Materials

Coconut pith (CP) was collected from Ben Tre province, Vietnam. Sodium hydroxide (NaOH, 98%), hydrogen peroxide (H_2O_2, 30%), and sulfuric acid (H_2SO_4, 98%) were purchased from Xilong, China. A dialysis membrane with a molecular weight cut-off (MWCO) of 14 kDa was used to dialyze acidic nanocellulose suspension. All solutions were prepared in distilled water (DW).

Cellulose recovery from CP

Raw coconut pith (RCP) was rinsed with DW, dried under sunlight, and ground into powder by a blender in 5 min. Cellulose recovery from CP was conducted via a two-step procedure of alkali treatment and bleaching. For the first period, CP was immersed in 4.0% NaOH at 80 °C with a solid-to-liquid ratio (SLR) of 1:20 g/mL and the mixture was continuously stirred for 2 h. After that, the mixture was separated by using a filter cloth to obtain the alkali-treated coconut pith (ACP). The second stage was conducted by using a mixture of 10.0% H_2O_2 and 1.0% NaOH. The treatment condition was SLR of 1:30 g/mL, temperature of 80 °C, and time of 1 h. The mixture was then separated by filter cloth, washed with DW until neutral pH, and dried to obtain the bleached coconut pith (BCP).

Nanocellulose synthesis from BCP

Nanocellulose synthesis from BCP was conducted by chemo-mechanical procedure of ball milling and acid hydrolysis. Firstly, an aqueous suspension of BCP was ball-milled at different points of time from 4 to 8 h to mechanically reduce the cellulose length. The suspension was then hydrolyzed with 64.0% H_2SO_4 at 50 °C. The SLR and hydrolysis time were investigated in the range of 1:10-1:50 g/mL and 30-90 min, respectively. After the appropriate time, the suspension was diluted with DW to stop the hydrolysis. Finally, the suspension was centrifuged and dialyzed with a dialysis membrane to remove excessive acid and yield neutral nanocellulose. Table 1 tabulates the experimental design for studying the effects of synthesis conditions on the characteristics of CP-based nanocellulose. The images of raw CP, intermediate products and nanocellulose obtained are illustrated in Figure 1.

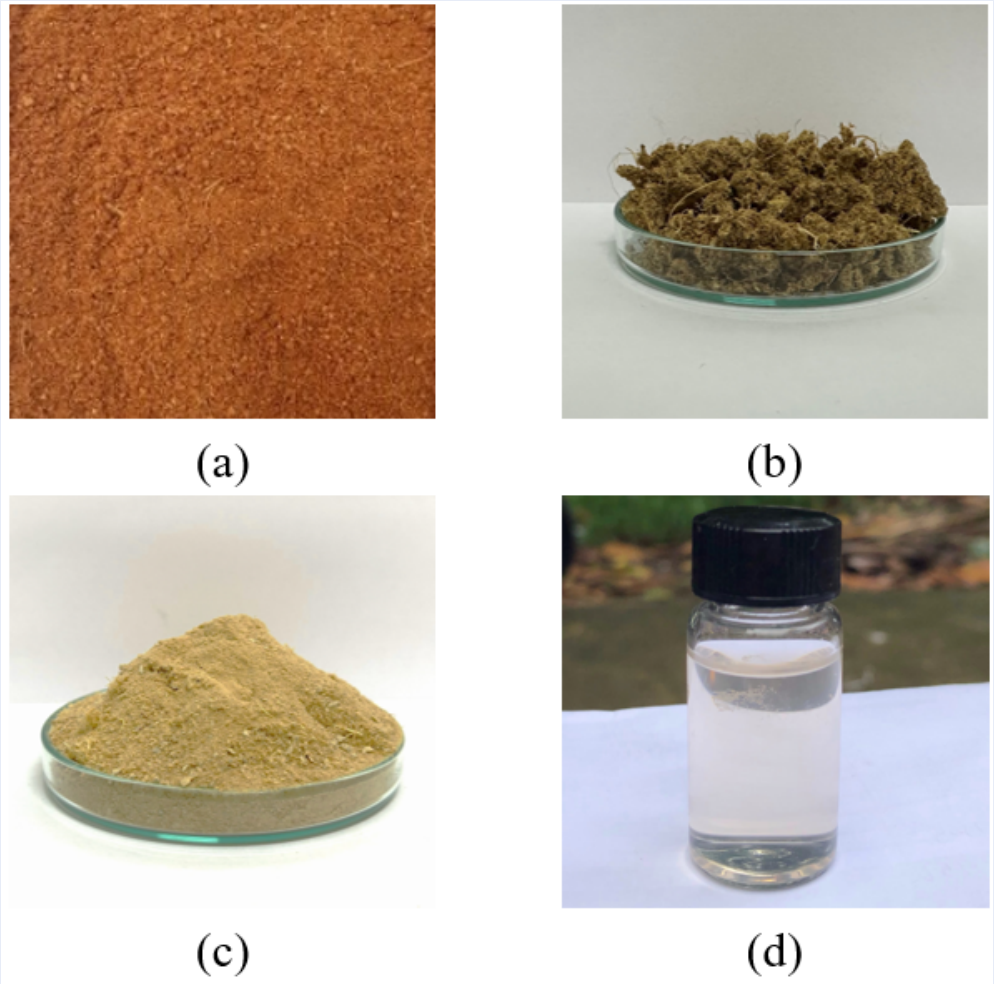

Figure 1: Images of raw CP (a), ACP (b), BCP (c), and nanocellulose (d).

Table 1: Experimental design for nanocellulose synthesis by ball milling-acid hydrolysis.

Ball-milling time (h)	SLR (g/mL)	Hydrolysis time (min)
4; 5; 6; 7; 8	1:30	60
6	1:10; 1:20; 1:30; 1:40; 1:50	60
6	1:30	30; 45; 60; 75;90

Characterization of cellulose and nanocellulose derived from CP

The holocellulose content of samples was determined by following the National Renewable Energy Laboratory procedure (NREL/TP-510-426) as described in our previous work[10]. The chemical structure of CP-derived specimens was analyzed by Fourier Transform Infrared spectroscopy (FTIR, Bruker Alpha II) over the wavenumber range of 4000-500 cm^{-1} at a resolution of 4 cm^{-1}. Particle size distribution and zeta potential of nanocellulose synthesized under different conditions were obtained by utilizing Dynamic Light Scattering (DLS, Zetasizer Nano ZS90). The crystallinity profile of RCP, BCP, ACP, and nanocellulose obtained from coconut pith was collected by X-ray Diffraction analysis (XRD, D8 Advance Bruker) with Cu-Ka radiation (λ = 1.5418 Å), scanning range 2θ of 5-80° and a scan rate of 0.02°. The crystallinity index (CrI) of specimens was also calculated by Eq.

(1), as suggested in the previous work[10]:

$$Crl = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (1)$$

where I_{002} and I_{am} are, respectively, the intensity of 002 lattice diffraction and at 2θ of 18°.

RESULTS AND DISCUSSION

CP purification

To demonstrate the efficiency of cellulose recovery, the chemical composition of raw coconut pith, alkali-treated, and bleached cellulose pulp is analyzed. In particular, the holocellulose content of RCP, ACP, and BCP is 49.07 ± 0.77%, 65.42 ± 0.80%, and 76.31 ± 1.53%, respectively. After two-stage chemical pretreatment with NaOH solution and a mixture of NaOH:H_2O_2, the holocellulose content of CP pulp significantly increases, indicating that the undesired non-cellulosic components like hemicellulose and lignin are effectively eliminated. However, the solid recovery yield is as low as 43.87% due to the mass loss at filtration and rinsing to collect the cellulose-rich pulp.

Nanocellulose synthesis from BCP

After ball milling and acid hydrolysis, the synthesized nanocellulose samples in suspension were measured for their particle size distribution and zeta potential by DLS assay. According to Figure 2, the particle size distribution of CP nanocellulose is greatly influenced by the ball-milling time, but no significant variation is observed in its zeta potential, which ranges from -16.5 to -21.4 mV. Ball-milling time of 4 h and 6 h at the same hydrolysis condition results in nanocellulose having consistent particle size of below 600 nm, in which the respective cumulative intensity for particle size of below 300 nm is 44.37% and 40.93%. Besides, cellulose particles obtained at ball milling periods of 5 h, 7 h, and 8 h are distributed into three particle size regions: below 300 nm, 300-600 nm, and 600-900 nm, especially the intensity of particles having a size of below 300 nm is only about 5.40 to 8.61%.

Sofla et al. proposed that there was a possibility of a fibrous cellulose layer forming around the grinding balls during milling. As a consequence, the impact power of the balls on the particles decreased, leading to an increase in particle size[11]. Therefore, the cumulative intensity for particle size of below 300 nm at 7 and 8 ball-milling time is significantly low, whereas that for 600-900 nm size region is up to 23.55 – 40.60%. Moreover, the particle size distribution of all nanocellulose particles prepared at different ball-milling conditions shows two separate peaks of below

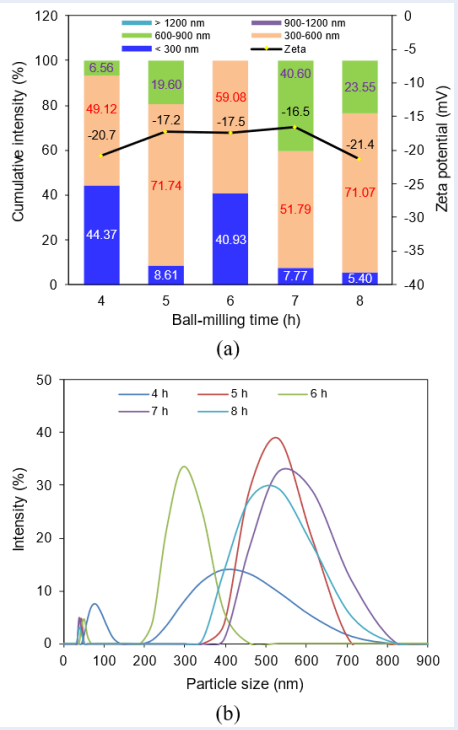

Figure 2: Cumulative intensity, zeta potential (a), and particle size distribution (b) CP-derived nanocellulose with increasing ball-milling time. The SLR and hydrolysis time are respectively fixed at 1:30 g/mL and 60 min.

100 nm and greater than 200 nm, suggesting the diameter and length of CP nanocellulose in that order. Figure 3 shows that the SLR between BCP and H_2SO_4 has a strong influence on the formation of nanocellulose particles. Only SLRs of 1:30, 1:40, and 1:50 g/mL yield a particle size of below 300 nm. Unexpectedly, the synthesized cellulose nanoparticles at the SLR of 1:40 and 1:50 g/mL tend to aggregate into larger ones with size of over 300 nm. This phenomenon can be attributed to hydrogen bonds on the cellulose surface, which cause the nanoparticles to aggregate and subsequently impact their size[12]. The results show that an SLR of 1:50 g/mL results in a diverse distribution of size regions below 300 nm, 600-900 nm, 900-1200 nm, and above 1200 nm along with the lowest zeta potential of -12.6 mV. Because of the highest cumulative intensity at the particle size of below 300 nm (69.04%) and the second lowest zeta potential of -17.5 mV, the SLR of 1:30 g/mL is chosen as an appropriate condition for nanocellulose synthesis from CP.

Hydrolysis time is also a significant parameter affecting the production of nanocellulose in terms of particle size and zeta potential. As seen in Figure 4, the

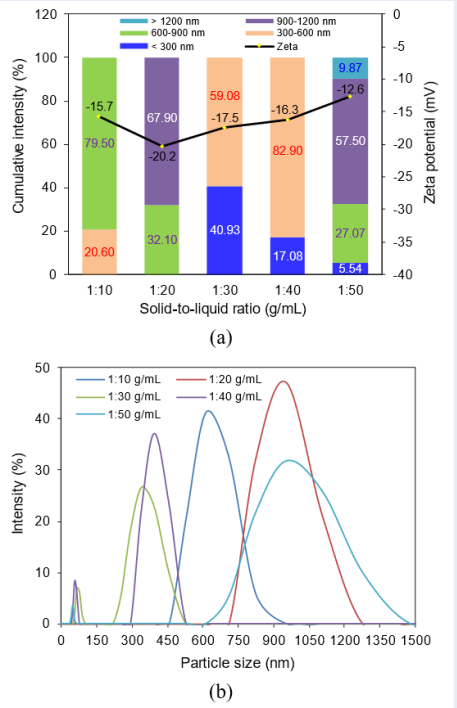

Figure 3: Cumulative intensity, zeta potential (a), and particle size distribution (b) CP-derived nanocellulose with varied SLR. The ball-milling and hydrolysis time is respectively fixed at 6 h and 60 min.

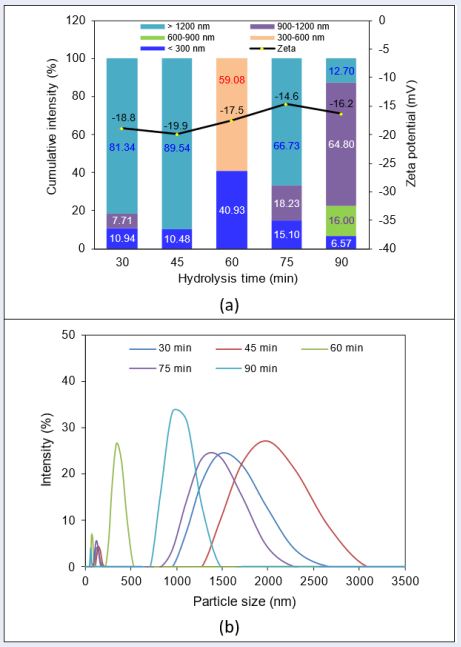

Figure 4: Cumulative intensity, zeta potential (a), and particle size distribution (b) CP-derived nanocellulose with increasing hydrolysis time. The ball-milling time and SLR are kept at 6 h and 1:30 g/mL, respectively.

short hydrolysis duration of 30-45 min inefficiently yields nanocellulose suspension based on the cumulative intensity for size above 900 nm up to 89%. Previous studies on other categories of lignocellulosic biomass reported that the appropriate hydrolysis time was about 45-55 min at H_2SO_4 concentration above 60% and SLR of 1:20 g/mL[12]. Indeed, when the hydrolysis time is longer from 60 to 90 min, more nanocellulose particles below 900 nm are produced. In particular, the hydrolysis duration of 60 min only creates nanocellulose particles with a size below 600 nm, it is chosen as the hydrolysis time to fabricate nanocellulose from CP in this study.

Similar to the phenomenon witnessed in the experiment of investigating the influence of SLR on the characteristics of nanocellulose[12], aggregation occurs at the hydrolysis time over 75 min and results in the high cumulative intensity for size above 900 nm (77.50 - 84.96%) even though nanocellulose particles with their size below 600 nm are obtained. Figure 4b clearly shows the excellent nanocellulose synthesis efficiency at an acid hydrolysis time of 60 min because no intensity peak is found at the particle size over 500 nm.

FTIR and XRD spectra of nanocellulose

The nanocellulose suspension resulted from the hydrolysis condition of ball-milling time of 1 h, H_2SO_4 64.0%, SLR of 1:30 g/mL, hydrolysis duration of 60 min was characterized in terms of chemical structure and crystallinity. According to Figure 5, there are characteristic peaks of cellulose at 3340 cm^{-1}, 2900 cm^{-1}, 1030 cm^{-1}, and 897 cm^{-1} in the FTIR spectra of BCP and nanocellulose[13]. The absence of peaks at 1605 cm^{-1} and 1250 cm^{-1} in the FTIR spectrum of nanocellulose indicates the effectiveness of eliminating non-cellulosic compounds like hemicellulose and lignin. In particular, the peak at 1605 cm^{-1} might be assigned to C-O bonds in hemicellulose or aromatic ring vibrations in lignin[14]. The peak at 1250 cm^{-1} is attributed to the Syringyl ring and C–O stretching of lignin and xylan[15]. The high intensity of signals at 1030 cm^{-1} and 897 cm^{-1} demonstrates the evident presence of β-(1,4)-glycosidic linkages in the cellulose chains[13] only when non-cellulosic components are eliminated.

Finally, the crystallinity profile of RCP, ACP, BCP, and nanocellulose is analyzed by using the XRD method. As shown in Figure 6, there are characteristic peaks at 2θ of 15.5°, 22°, and 34.5°, respectively, representing the (110), (101), (200), and (004) planes of

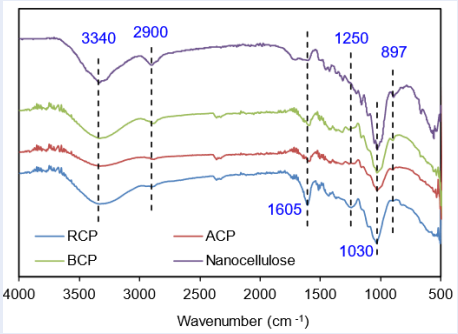

Figure 5: FTIR spectra of raw coconut pith, alkali-treated and bleached CP, and CP-derived nanocellulose.

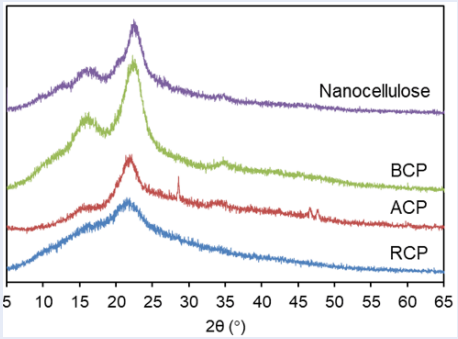

Figure 6: XRD spectra of raw coconut pith, alkali-treated and bleached CP, and CP-derived nanocellulose.

cellulose chains. After chemical pretreatment, the intensities at these peaks are witnessed in the BCP spectra. The crystallinity index of RCP, ACP, and BCP is 32.08%, 42.58%, and 60.35% in that order, indicating that the pretreatment removes the amorphous regions in the CP structure. It should be noted that the crystallinity index of BCP in this work is much higher than that of bleached pulp from coconut shells (51.49%) and coconut husks (56.73%) [16]. Nanocellulose obtained from the chemo-mechanical procedure developed in this study exhibits its significantly increased crystallinity index of 72.08% which is comparable to that of nanocellulose crystals from coconut husk (79.3%) [17], but higher than that of nanocellulose fibrils from coconut shells (57.61%) and coconut husk (49.20%) [16]. Therefore, CP-derived nanocellulose offers a wide range of applications such as excellent oxygen barriers in food packaging, electronic devices, inks for 3D printing, oilfield servicing fluids, and strengthening additives in composites [9].

CONCLUSIONS

In summary, cellulose nanoparticles are successfully fabricated from coconut pith using the novel two-stage chemo-mechanical procedure of ball milling and acid hydrolysis. The bleached coconut pith should be ball-milled in 6 h and then hydrolyzed by H_2SO_4 64% at SLR of 1:30 g/mL within 60 min to yield a stable suspension of nanocellulose having 100% particle size of below 600 nm and zeta potential of -17.5 mV. The analysis demonstrates that the holocellulose content and crystallinity index of bleached pulp are correspondingly up to 76.31 ± 1.53% and 60.35%. By utilizing a developed chemo-mechanical process, the crystallinity index of CP-derived nanocellulose remarkably increases to 72.08%. Further studies should investigate the feasibility of scaling up this effective chemo-mechanical process to implement it in the industrial manufacturing of nanocellulose from coconut residues.

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ABBREVIATION

ACP: Alkali-treated coconut pith
BCP: Bleached coconut pith
CP: Coconut pith
DLS: Dynamic light scattering
DW: Distilled water
FTIR: Fourier transform spectroscopy
NC: Nanocellulose
RCP: Raw coconut pith
SLR: Solid-to-liquid ratio
XRD: X-ray diffraction

COMPETING OF INTERESTS

The authors declare that they have no competing interests.

AUTHORS' CONTRIBUTIONS

Nga H.N. Do: Investigation, Characterization, Writing – original draft. **Chi T.X. Nguyen**: Methodology, Formal analysis. **Huy T.A. Nguyen**: Data analysis. **Phong T. Mai, Thang V. Le**: Resources, Writing-review & editing. **Phung K. Le**: Conceptualization, Funding acquisition, Project administration, Writing – review & editing.

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Bài nghiên cứu

Phương pháp kết hợp cơ-hóa học mới trong tổng hợp nanocellulose từ mụn dừa

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TÓM TẮT

Nanocellulose đã nhận được sự chú ý rất lớn do các đặc tính độc đáo của nó như độ bền cao, tính linh hoạt, khối lượng riêng thấp, khả năng tương thích sinh học và tính ưa nước tự nhiên với nhiều nhóm hydroxyl có thể dễ dàng biến đổi cho các mục đích khác nhau. Sinh khối ligno-cellulose, đặc biệt là phụ phẩm dừa như vỏ dừa, xơ dừa, mảnh dừa, v.v. đã được tận dụng hiệu quả để sản xuất cellulose với nhiều kích cỡ và hình thái khác nhau như cellulose vi tinh thể, nanocellulose dạng tinh thể và dạng sợi. Trong nghiên cứu này, quá trình sản xuất nanocellulose từ mụn dừa (CP) được thực hiện theo phương pháp hóa-cơ học mới bằng cách thực hiện tiền xử lý kiềm hai giai đoạn, nghiền bi, thủy phân axit, ly tâm và thẩm tách. Nanocellulose từ CP được đánh giá về phân bố kích thước hạt, thế zeta, thành phần hóa học, cấu trúc tinh thể và hóa học. Tất cả các thông số về thời gian nghiền bi, tỷ lệ rắn-lỏng và thời gian thủy phân đều ảnh hưởng lớn đến sự hình thành hạt nanocellulose từ CP cũng như độ ổn định của hệ hạt. Dựa trên kết quả thực nghiệm, cường độ tích lũy của các hạt có kích thước dưới 300 nm và trong khoảng 300 – 600 nm lần lượt là 40,93% và 59,08%, cho thấy hiệu quả rõ rệt của phương pháp cơ-hóa học được phát triển trong tổng hợp nanocellulose từ CP giàu cellulose có hàm lượng holocellulose ban đầu cao đạt 49,07%. Đáng chú ý, chỉ số tinh thể của nanocellulose có nguồn gốc từ CP cao đến 72,08% tương đương với các tinh thể nanocellulose làm từ mảnh dừa. Công trình này chứng minh hiệu quả của việc kết hợp nghiền bi và thủy phân axit trong tổng hợp nanocellulose ổn định từ CP và tạo tiền đề cho việc nghiên cứu sâu hơn để áp dụng kỹ thuật đã xây dựng vào sản xuất công nghiệp.

Từ khoá: Mụn dừa, Nanocellulose, Quá trình cơ-hóa học, Nghiền bi, Thủy phân acid

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