



High Carboxyl Content Cellulose Nanofibers from Banana Peel via One-Pot Nitro-Oxidative Fabrication

Dung Van Nguyen^{1,2*}, Ha Thi Ngan Nguyen^{1,2}

¹ Faculty of Chemical Engineering, Ho Chi Minh City University of Technology (HCMUT), Ho Chi Minh City 700000, Vietnam

² Vietnam National University Ho Chi Minh City, Linh Trung Ward, Thu Duc City, Ho Chi Minh City 700000, Vietnam

Corresponding Author Email: nvdung@hcmut.edu.vn

<https://doi.org/10.18280/rcma.330208>

ABSTRACT

Received: 4 February 2023

Accepted: 10 April 2023

Keywords:

banana peel, biomass valorization, carboxyl cellulose, ion-exchange, one-pot strategy, nanofiber, nitro-oxidation

Banana peels (BP) are the outer covering of a banana fruit and are usually discarded as abundant and underutilized waste from banana production and consumption. This work investigated the bioresource for one-pot preparation of carboxyl cellulose nanofibers (CCNF) using the nitro-oxidation method. Briefly, BP was added to a mixture of HNO₃ and NaNO₂ at 30–50°C for 6 h. To characterize the obtained material, different methods, including X-ray diffraction, TEM images, FTIR spectroscopy, EDX spectroscopy, and acid-base titration, were used. TEM images reveal that the nanofibers were mainly formed with a diameter of roughly 20–50 nm. Moreover, the results of FTIR, EDX, and titration proved the formation of carboxylic acid (-COOH) groups in CCNF with an amount of 4.9 mmol/g, or 22 wt%. This high carboxyl content makes CCNF an ion-exchange material, thereby enabling the development of new applications. Compared with traditional multistep processes for the preparation of cellulose nanomaterials, the current approach is simpler, uses fewer chemicals, and consumes less energy. Overall, these successful results not only valorize potential banana peel waste but also introduce an advanced material for further use.

1. INTRODUCTION

Presently, agricultural production worldwide creates a tremendous amount of solid waste [1]. However, inappropriate management of the waste source has consequences for the environment, economy, and society [2]. Improper disposal of agricultural waste, such as animal manure or crop residues, can contaminate nearby water sources with nutrients and pathogens [3]. Burning agricultural waste can also discharge harmful air pollutants, causing respiratory issues for adjacent residents [4]. Additionally, the expense of addressing environmental damage due to agricultural waste can be considerable. Consequently, there is an urgent need to convert the lignocellulosic-based waste into value-added products [5], such as carbon-based materials [6, 7], biogas [8, 9], biofuels [10, 11], engineering plastics [12, 13], biocomposites [14-16]. By implementing waste-to-product strategies, it is possible to create a more efficient, resilient, and sustainable economy while simultaneously reducing waste and its environmental impact.

Banana (*Musa sapientum*) is a popular herbaceous plant native to tropical countries [17]. As estimated by FAOSTAT [18] for 2021, the plant is widely farmed for its edible fruit, with a global harvested area of 5.34 million ha and an annual production of 125 million tons. Banana peel, a major byproduct of the banana processing industry, accounts for 30–40 wt% of the fruit [19]. As a result, around 38–50 million tons of banana peel can be released annually. This matter is commonly regarded as agricultural waste and disposed of in

municipal landfills, potentially causing environmental problems [20]. Thus, banana peel becomes an available, abundant, and underutilized residue, and its valorization is necessary.

According to Tiwari et al. [21], banana peel is rich in cellulose (35.3 wt%), moderate in hemicellulose (19.5 wt%), and poor in lignin (6.7 wt%). This renewable resource can, therefore, be beneficial for nanocellulose extraction. In comparison with carbonization processes, the fabrication of cellulose nanomaterials, which could reduce the release of greenhouse gas CO₂ into the atmosphere, has recently been an interesting topic [22]. The nanomaterials not only retain the basic structure and properties of cellulose but also offer the advantages of the nanoscale, such as superior mechanical strength, reproducibility, biodegradability, and facile surface modification [22-24]. These properties make nanocellulose attractive for a wide range of potential applications, like packaging, textiles, cosmetics, biomedical, electronics, and water treatment [25, 26].

Typical methods for the preparation of cellulose nanostructures from biomass are alkaline pretreatment with NaOH, hydrolysis with strong acids like HCl or H₂SO₄, biological treatment with cellulase, or oxidation with NaOCl, NaBr, and TEMPO [27]. Nevertheless, these methods are generally multistep, complex, time-consuming, energy-intensive, and use toxic chemicals [22]. Recently, Sharma et al. [28] proposed a novel strategy for one-pot preparation of carboxyl cellulose nanofibers (CCNF) from several biomass resources using a mixture of HNO₃ and NaNO₂. This nitro-

oxidation method offers high yield, control over particle size and morphology, high purity, versatility, and scalability. It is also simple, saving chemicals, time, and energy. The wastewater might potentially be neutralized to manufacture nitrogen-rich fertilizers. Synthetically, lignin is reduced to benzoquinone compounds, which are soluble in nitrous oxide species. Nitric acid can break down the hemicellulose components into xylose and other byproducts [29]. Furthermore, the hydroxymethyl ($-\text{CH}_2\text{OH}$) groups at the C6 position of cellulose are oxidized into carboxylic ($-\text{COOH}$) groups, which are not present in most traditional cellulose nanomaterials [30]. Depending on the pH of the environment, the carboxyl groups in nanofibers can shift between acid and salt. In neutral and basic environments, carboxylate ($-\text{COO}^-$) ions could repel other ones, resulting in nanofiber dispersion in aqueous media [28]. Moreover, carboxylate groups in CCNF play a key role in ion exchange that can remove hazardous ions like UO_2^{2+} [31], Pb^{2+} [32], and Hg^{2+} [33]. In addition to heavy metal adsorbents, CCNF can be used as drug delivery materials [34], hemostatic agents [35], reinforced composites [36].

Although certain biomass resources, such as jute fiber, spinifex grass, bamboo cellulose [28], moringa plant [33], and sorghum stalk [37], were used for the preparation of CCNF, considerable research on banana peel has never been reported before. As a result, the current work explored this abundant waste for one-pot preparation of carboxyl cellulose nanofibers using the nitro-oxidation method. The goal of the investigation was to create carboxyl groups with high content for future ion-exchange applications.

2. MATERIALS AND METHODS

2.1 Materials

Banana peel was collected from Thu Duc Market, Ho Chi Minh City, Vietnam. The waste was washed with tap water and distilled water to remove any dust and cut to 3–5 cm. It was put in an oven at 70°C until its mass remained unchanged. The dried material was then crushed, sieved, and stored in a plastic bag for future use. HNO_3 (65.0–68.0%), NaHCO_3 ($\geq 99.5\%$), and NaOH ($\geq 96.0\%$) were purchased from Xilong Scientific Co., Ltd. NaNO_2 ($\geq 99.0\%$), $\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$ ($\geq 98.0\%$), and ethanol ($\geq 99.5\%$) were procured from Guangdong Guanghua Sci-Tech Co., Ltd., Shanghai Zhanyun Chemical Co., Ltd., and VN-Chemsol Co., Ltd., respectively. All chemicals were used as received without any additional purification.



Figure 1. (a) Dried banana peel and (b) CCNF-40 product

2.2 Preparation of carboxyl cellulose nanofibers

The nitro-oxidation method for the preparation of CCNF was referenced in such reports [28, 38]. The detailed procedure was adjusted for banana peel. Briefly, 1.00 g of dried BP (Figure 1a), 1.00 g of NaNO_2 , and 15.0 mL of HNO_3 (65 wt%) were added to a 250-mL round-bottom flask. The mixture was continuously stirred by a hotplate stirrer. The reactions were carried out at 30, 40, and 50°C . After 6 h of reaction, the mixture was primarily added to 250 mL of distilled water and centrifuged to decolorize. Next, the colloidal suspension was cleaned with a mixture of ethanol and water (volume ratio of 1:2) and centrifuged to remove the waste solution. This step was repeated many times until the pH of the suspension reached ~ 3.5 . Carboxylic acid groups were converted into carboxylate groups by adding a 40 g/L NaHCO_3 solution to the suspension of CCNF until its pH reached ~ 7.5 . To eliminate excess NaHCO_3 from CCNF, washing with distilled water and centrifuging were repeated. Finally, the cleaned suspensions were freeze-dried to obtain solid samples (Figure 1b). These product samples are denoted by CCNF-X, where X ($^\circ\text{C}$) represents the synthesis temperature.

2.3 Characterization of carboxyl cellulose nanofibers

The crystalline structures of CCNF samples were characterized by X-ray powder diffraction (XRD) using a Bruker AXS D8 diffractometer over the 2θ range of $5\text{--}50^\circ$. The $\text{Cu-K}\alpha$ radiation ($\lambda=1.5418 \text{ \AA}$) was used as the target. The crystallinity (x) was calculated by the Segal equation [39], using the heights of the peak 200 (I_{total} , $2\theta=22.6^\circ$) and the peak I_{am} ($2\theta=18.0^\circ$). I_{total} represents both crystalline and amorphous structures, while I_{am} indicates the amorphous structure only [40].

$$x = \frac{I_{\text{total}} - I_{\text{am}}}{I_{\text{total}}} \quad (1)$$

Transmission electron microscopy (TEM) images of CCNF were observed with JEOL JEM-1400 equipment. Fourier-transform infrared (FTIR) spectra of BP and CCNF were analyzed using a Tensor 27 spectrometer (Bruker Optics, Germany). Element composition was evaluated using a JSM-7401F microscope incorporating a JED-2300 energy dispersive X-ray (EDX) spectrometer.

To determine the carboxyl content (molar mass = 45.0 g/mol), cleaned CCNF samples without adding NaHCO_3 (acid form) were freeze-dried. After that, 0.500 g CCNF samples were mixed with $\text{Ca}(\text{CH}_3\text{COO})_2$ (2.00 wt%). The suspensions were continuously stirred for 30 min to reach equilibrium. The mixture was finally titrated with V (mL) of NaOH solution (0.100 M) using phenolphthalein as an indicator. The titration was performed in triplicate, and the mean values of carboxyl contents are presented in Table 1. They were calculated using the following equations:

$$\text{Carboxyl content (mmol/g)} = \frac{0.100 \times V}{0.500} \quad (2)$$

$$\text{Carboxyl content (wt\%)} = \frac{0.100 \times V \times 45.0 \times 100\%}{0.500 \times 1000} \quad (3)$$

3. RESULTS AND DISCUSSION

3.1 XRD patterns of banana peel and CCNF

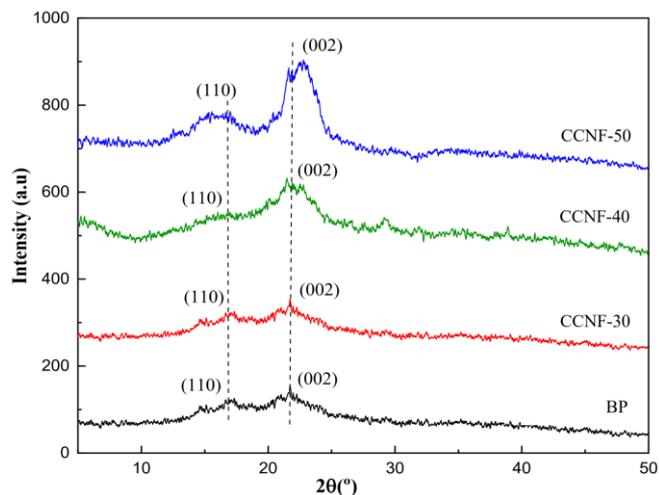


Figure 2. XRD patterns of banana peel and CCNF samples prepared at different temperatures

Figure 2 shows the X-ray diffraction patterns of BP and CCNF samples. All materials possess characteristic peaks at $2\theta=16.2$ and 22.6° , corresponding to the cellulose crystal planes of (110) and (200) [40, 41]. However, the peak intensities of CCNF samples were much higher than those of BP, leading to higher crystallinity (Table 1). In lignocellulosic materials, part of cellulose is a crystal phase, while hemicellulose and lignin are amorphous phases [21, 38]. Therefore, the combination of these three compositions causes

low crystallinity (21%). As mentioned before, HNO_3 and NaNO_2 can convert hemicellulose and lignin into soluble components [29]. Hence, the amorphous phases of hemicellulose and lignin could be removed from BP, leaving solid cellulose fibers with higher crystallinity. When the temperature was raised from 30 to 50°C , the crystallinity of CCNF increased from 40% to 63%. This demonstrates that higher temperatures could speed up the extraction of CCNF from BP.

3.2 TEM images of CCNF

Figure 3 shows TEM images of CCNF prepared at 40 and 50°C . Almost all nanofibers were dispersed well rather than agglomerated. The presence of carboxylate groups could repel each other to aid in the dispersion of CCNF. Although the bundles were not entirely separated, nanostructures were created at 40°C . The fibers are roughly $0.5\text{--}2.0\ \mu\text{m}$ in length and around 50 nm in diameter. This result indicates that the synthesis process removed the amorphous phases of lignin and hemicellulose present in banana peel, resulting in the formation of cellulose nanofibers. When the temperature increased from 40 to 50°C , the diameter of the CCNF samples decreased to around 20–40 nm. The morphology of the nanofibers became obvious when the bundles were separated. Sharma et al. [28] reported that CCNF synthesized from jute fibers at 50°C for 12 h has a length of 190–370 nm and a width of 4–5 nm. Although the as-prepared CCNF was larger in size, the synthesis time and/or temperature were reduced. It could therefore save time and energy. Herein, the carboxyl content rather than the size of CCNF was the major goal. Therefore, reducing the size of CCNF is not essential. Carboxyl content in CCNF is presented in the next part.

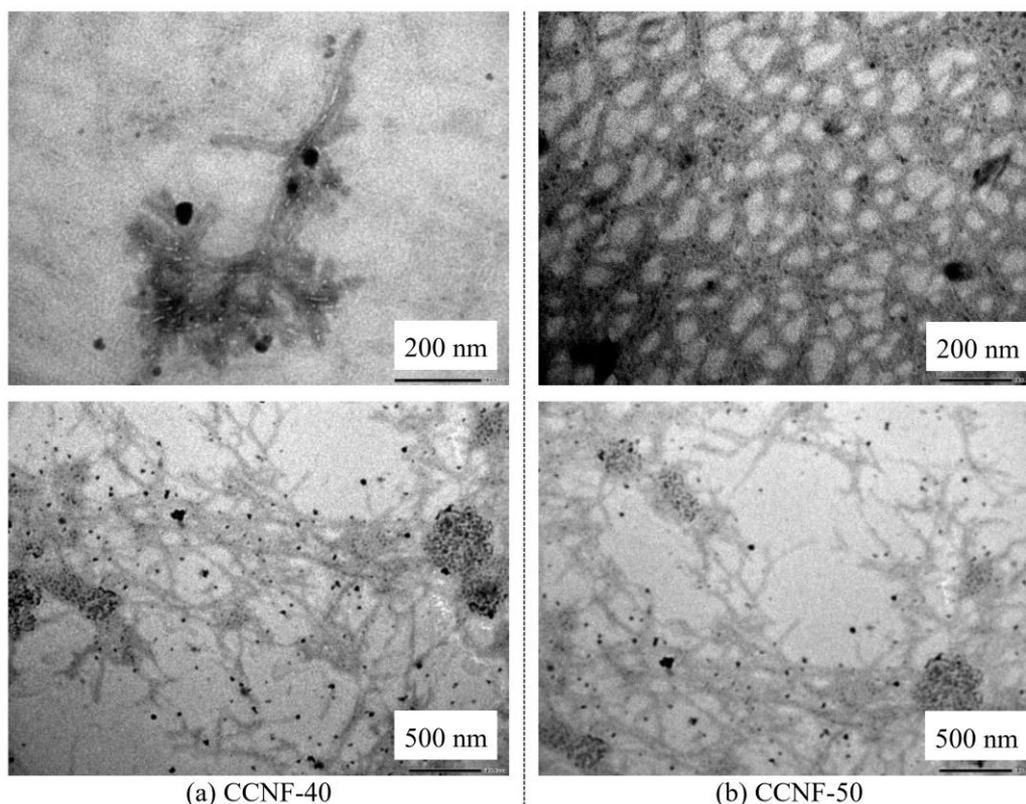


Figure 3. TEM images of CCNF samples prepared at (a) 40°C and (b) 50°C

3.3 Carboxyl content in CCNF

The carboxyl contents of banana peel and CCNF samples are shown in Table 1. BP contained almost no acid groups. Conversely, all CCNF samples exhibited high carboxyl content (3.1–4.6 mmol/g), demonstrating that the mixture of HNO₃ and NaNO₂ oxidized BP effectively. According to Chen et al. [33], the oxidation of hydroxymethyl groups into carboxylic acid groups could be expressed as follows:



Without heating (30°C), HNO₃ and NaNO₂ partially oxidized BP to yield carboxylic acid groups. When the

synthesis temperature was raised from 30 to 40°C, the carboxyl content significantly increased from 3.1 to 4.6 mmol/g (14 to 21 wt%). Higher temperatures could accelerate the process. However, increasing the temperature from 40 to 50°C resulted in a slight improvement in carboxyl content from 4.6 to 4.9 mmol/g (21 to 22 wt%). These results suggest that at 40°C, the conversion of hydroxymethyl groups into carboxylic acid groups was nearly complete. Compared with other reports in the literature (Table 1), the current study provides much higher carboxyl content. Although the synthesis conditions were less severe, it seems that the soft structure of BP might be convenient for the nitro-oxidation process. Thus, biomass resources could play an important role in the formation of carboxylic groups in the CCNF products.

Table 1. Crystallinity and carboxyl content of banana peel and CCNF samples

| Sample | Temperature (°C) | Time (h) | Crystallinity (%) | Carboxylic acid content | | References |
|----------------------|------------------|----------|-------------------|-------------------------|-------|------------|
| | | | | (mmol/g) | (wt%) | |
| Banana peel | - | - | 21 | ~0 | ~0 | |
| CCNF-30 | 30 | 6 | 40 | 3.1 | 14 | This study |
| CCNF-40 | 40 | 6 | 47 | 4.6 | 21 | |
| CCNF-50 | 50 | 6 | 63 | 4.9 | 22 | |
| CCNF (Jute fibers) | 40 | 16 | | 0.9 | 4 | [36] |
| CCNF (Jute fibers) | 50 | 12 | | 1.1 | 5 | [28] |
| CCNF (Spinifex) | 50 | 12 | | 0.2 | 1 | [28] |
| CCNF (Moringa) | 40 | 16 | | 1.0 | 4 | [33] |
| CCNF (Sorghum stalk) | 50 | 12 | | 0.7 | 3 | [37] |

3.4 FTIR spectra of banana peel and CCNF

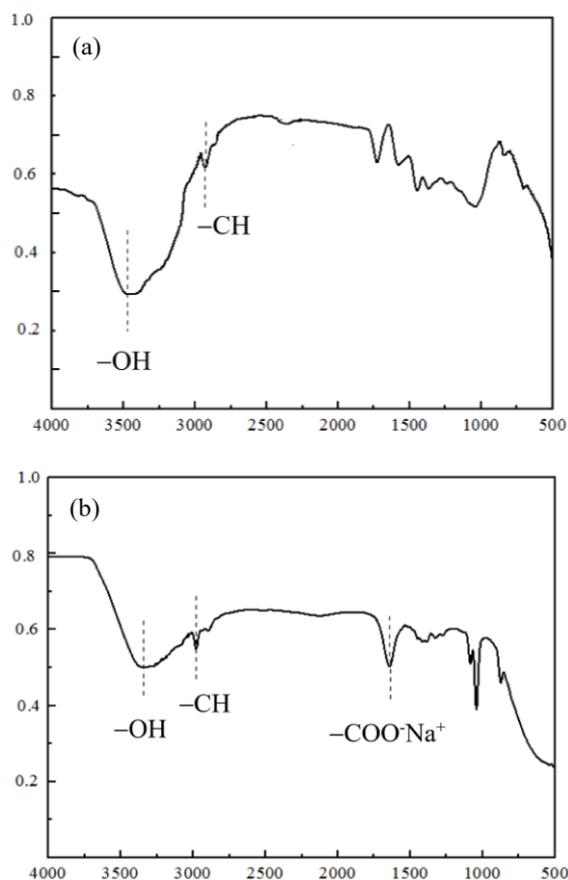


Figure 4. FTIR spectra of (a) banana peel and (b) CCNF-40

Figure 4 shows a similar vibration band at around 3300–3500 cm⁻¹, corresponding to O–H stretching band of cellulose [42]. The peak observed at approximately 2980 cm⁻¹ is due to the aliphatic saturated C–H stretching vibration of cellulose [43, 44]. These functional groups of cellulose are present in both BP and CCNF-40. Additionally, the characteristic peak at 1715 cm⁻¹ for C=O bond in hemicellulose and the minor signature at 1570 cm⁻¹ for C=C bond in lignin were only found in BP but disappeared in CCNF-40 [45]. These results prove that the synthesis process removed most of hemicellulose and lignin from BP to extract cellulose nanofibers. Conversely, the presence of the peak at 1643 cm⁻¹ in CCNF-40 demonstrates that –COO⁻ groups were formed from the oxidation of –CH₂OH groups [28, 33].

3.5 EDX spectra of banana peel and CCNF

The elemental compositions of banana peel and CCNF-40 were determined using EDX analysis (Figure 5 and Table 2). The raw material contained the primary elements C and O, as well as trace elements Mg, K, and Si. These minerals, which might be absorbed from the living environment of bananas, have been reported in the literature [24, 44]. Compared with BP, the synthesis procedure dramatically lowered the C content but significantly increased the O content. The oxidation of –CH₂OH groups into –COO⁻ groups could increase O content. Furthermore, the O content of lignin is generally lower than that of cellulose. As a result, the elimination of lignin during the synthesis process could contribute to the increase in O content. Mg, K, and N did not appear in CCNF-40, indicating that the cleaning process removed these ions as well as perhaps extra NaNO₂ and NaHCO₃. Nonetheless, the high Na content of CCNF-40 could

be attributed to $-\text{COONa}$ groups rather than soluble salts of NaNO_2 and NaHCO_3 . Overall, the results of acid-base titration, FTIR spectra, and EDX spectra confirmed the formation of carboxyl groups during the nitro-oxidation of banana peel.

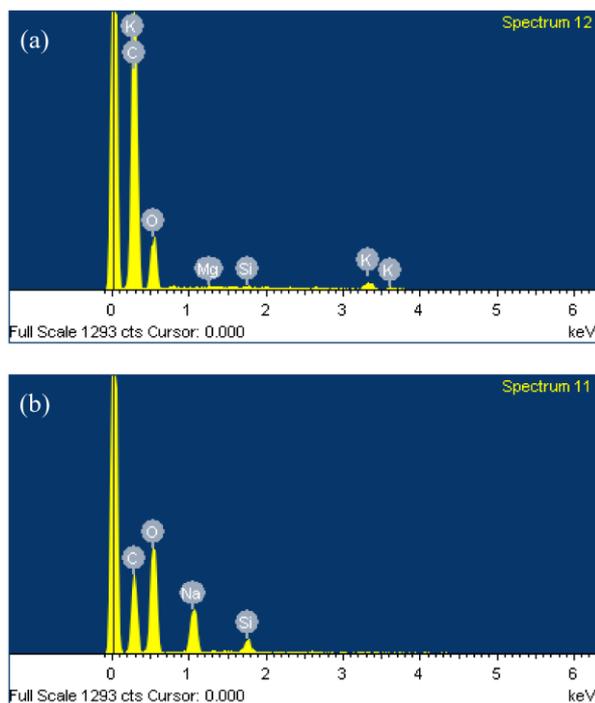


Figure 5. EDX spectra of (a) banana peel and (b) CCNF-40

Table 2. Elemental compositions of banana peel and CCNF-40 from EDX analysis

| Sample | C | O | Mg | Si | K | Na |
|----------------|-------|-------|------|------|------|------|
| BP (%mol) | 78.42 | 21.07 | 0.08 | 0.09 | 0.34 | - |
| CCNF-40 (%mol) | 44.55 | 48.32 | - | 0.92 | - | 6.21 |

4. CONCLUSIONS

In this study, carboxyl cellulose nanofibers were successfully fabricated from banana peel via the nitro-oxidation method using a mixture of HNO_3 and NaNO_2 at 30–50°C for 6 h. The results of XRD, EDX, and FTIR proved that most of the hemicellulose and lignin in BP were removed to obtain CCNF. TEM images show that the diameter of CCNF roughly ranges from 20 to 50 nm. In addition, the nitro-oxidation formed carboxylic acid groups with a content of 4.9 mmol/g (22 wt%), which was better than such previous studies. Compared with traditional cellulose nanomaterials, the as-prepared CCNF with this high carboxyl content can open new applications as an ion-exchange material. The current one-pot method is also simple, saves time, chemicals, and energy, and can be scalable. The wastewater can be used for the production of nitrogen-rich fertilizer. In spite of this, the use of strong acid (HNO_3) and the possible release of nitrogen oxides during the nitro-oxidation process can pose a significant safety hazard to workers if not handled properly. Addressing these limitations will improve the sustainability of the method. Overall, the primary findings valorize the abundant banana peel for the facile one-pot preparation of cellulose nanofibers with high

carboxyl content. The process can be optimized and modeled, and the obtained material can be explored for further use.

ACKNOWLEDGMENT

We acknowledge Ho Chi Minh City University of Technology (HCMUT), VNU-HCM for supporting this study.

REFERENCES

- [1] Babu, S., Singh Rathore, S., Singh, R., Kumar, S., Singh, V.K., Yadav, S.K., Yadav, V., Raj, R., Yadav, D., Shekhawat, K., Ali Wani, O. (2022). Exploring agricultural waste biomass for energy, food and feed production and pollution mitigation: A review. *Bioresource Technology*, 360. <https://doi.org/10.1016/j.biortech.2022.127566>
- [2] Capanoglu, E., Nemli, E., Tomas-Barberan, F. (2022). Novel approaches in the valorization of agricultural wastes and their applications. *Journal of Agricultural and Food Chemistry*, 70(23): 6787-6804. <https://doi.org/10.1021/acs.jafc.1c07104>
- [3] Koul, B., Yakoob, M., Shah, M.P. (2022). Agricultural waste management strategies for environmental sustainability. *Environmental Research*, 206, 112285. <https://doi.org/10.1016/j.envres.2021.112285>
- [4] Kaushal, L.A., Prashar, A. (2021). Agricultural crop residue burning and its environmental impacts and potential causes – case of northwest India. *Journal of Environmental Planning and Management*, 64(3): 464-484. <https://doi.org/10.1080/09640568.2020.1767044>
- [5] Ambaye, T.G., Vaccari, M., Bonilla-Petriciolet, A., Prasad, S., van Hullebusch, E.D., Rtimi, S. (2021). Emerging technologies for biofuel production: A critical review on recent progress, challenges and perspectives. *Journal of Environmental Management*, 290. <https://doi.org/10.1016/j.jenvman.2021.112627>
- [6] Yrjälä, K., Ramakrishnan, M., Salo, E. (2022). Agricultural waste streams as resource in circular economy for biochar production towards carbon neutrality. *Current Opinion in Environmental Science & Health*, 26, 100339. <https://doi.org/10.1016/j.coesh.2022.100339>
- [7] Do, T.V.T., Bui, Q.L.N., Nguyen, H.M., Lam, H.H., Tran-Thuy, T.M., Nguyen, L.Q., Ngo, D.T.H., Nguyen, D.V. (2022). One-pot fabrication of magnetic biochar by FeCl_3 -activation of lotus seedpod and its catalytic activity towards degradation of Orange G. *Materials Research Express*, 9, 105601. <https://doi.org/10.1088/2053-1591/ac9819>
- [8] Livi, M., Trifirò, F. (2022). Pyrogasification to produce biogas and biomethane from wood wastes. *Annales de Chimie - Science des Matériaux*, 46(4): 169-172. <https://doi.org/10.18280/acsm.460401>
- [9] Kapoor, R., Ghosh, P., Kumar, M., Sengupta, S., Gupta, A., Kumar, S.S., Vijay, V., Kumar, V., Kumar Vijay, V., Pant, D. (2020). Valorization of agricultural waste for biogas based circular economy in India: A research outlook. *Bioresource Technology*, 304, 123036. <https://doi.org/10.1016/j.biortech.2020.123036>

- [10] Hamzah, N., Tokimatsu, K., Yoshikawa, K. (2019). Solid fuel from oil palm biomass residues and municipal solid waste by hydrothermal treatment for electrical power generation in Malaysia: A review. *Sustainability*, 11(4): 1060. <https://doi.org/10.3390/su11041060>
- [11] Oyebanji, J.A., Onokwai, A.O., Okokpujie, I.P., Ukegbu, C.F., Suyi-Ajayi, A., Nnochiri, E.S., Tartibu, L.K. (2023). Characterization of *Adansonia digitata* (baobab wood) bio-oil and biochar produced using a fixed-bed tubular reactor. *Revue des Composites et des Matériaux Avancés*, 33(1): 39-46. <https://doi.org/10.18280/rcma.330106>
- [12] Abid, A., Bouhtoury, F.C.E., Gabsi, S. (2020). Recovery of residues from olive industry: Characterization of simple and acetylated lignin. *Annales de Chimie - Science des Matériaux*, 44(5): 327-332. <https://doi.org/10.18280/acsm.440504>
- [13] Park, S.A., Im, C., Oh, D.X., Hwang, S.Y., Jegal, J., Kim, J.H., Chang, Y.W., Jeon, H., Park, J. (2019). Study on the synthetic characteristics of biomass-derived isosorbide-based poly(arylene ether ketone)s for sustainable super engineering plastic. *Molecules*, 24(13): 2492. <https://doi.org/10.3390/molecules24132492>
- [14] Bijjam, R.R., Chandanam, S., Nandipati, G., Dhoria, S.H. (2022). Optimization of machining parameters in drilling of glass/hemp/bamboo fibres based hybrid polymer composites. *Annales de Chimie - Science des Matériaux*, 46(3): 127-133. <https://doi.org/10.18280/acsm.460303>
- [15] Djebloun, Y., Hecini, M. (2022). Physical and thermomechanical characteristics of epoxy biocomposites laminates reinforced by mat of date palm tree fibers. *Revue des Composites et des Matériaux Avancés*, 32(6): 277-283. <https://doi.org/10.18280/rcma.320603>
- [16] Babty, F., Fekkar, S., Derouiche, A., Mordane, S., El Assyry, A., Hachim, A. (2023). Experiments on mechanical properties of bio-composite produced from the shell of argan nuts. *Revue des Composites et des Matériaux Avancés*, 33(1): 59-64. <https://doi.org/10.18280/rcma.330109>
- [17] Barreto, A.C.H., Costa, M.M., Sombra, A.S.B., Rosa, D. S., Nascimento, R.F., Mazzetto, S.E., Fachine, P.B.A. (2010). Chemically modified banana fiber: Structure, dielectrical properties and biodegradability. *Journal of Polymers and the Environment*, 18(4): 523-531. <https://doi.org/10.1007/s10924-010-0216-x>
- [18] FAOSTAD. (n.d.). World production bananas. <https://www.fao.org/faostat/en/#data/QCL/visualize>
- [19] Sial, T.A., Khan, M.N., Lan, Z., Kumbhar, F., Ying, Z., Zhang, J., Sun, D., Li, X. (2019). Contrasting effects of banana peels waste and its biochar on greenhouse gas emissions and soil biochemical properties. *Process Safety and Environmental Protection*, 122: 366-377. <https://doi.org/10.1016/j.psep.2018.10.030>
- [20] Mohd Zaini, H., Roslan, J., Saallah, S., Munsu, E., Sulaiman, N.S., Pindi, W. (2022). Banana peels as a bioactive ingredient and its potential application in the food industry. *Journal of Functional Foods*, 92, 105054. <https://doi.org/10.1016/j.jff.2022.105054>
- [21] Tiwari, G., Sharma, A., Kumar, A., Sharma, S. (2019). Assessment of microwave-assisted alkali pretreatment for the production of sugars from banana fruit peel waste. *Biofuels*, 10(1): 3-10. <https://doi.org/10.1080/17597269.2018.1442665>
- [22] Reshmy, R., Philip, E., Paul, S.A., Madhavan, A., Sindhu, R., Binod, P., Pandey, A., Sirohi, R. (2020). Nanocellulose-based products for sustainable applications-recent trends and possibilities. *Reviews in Environmental Science and Bio/Technology*, 19(4): 779-806. <https://doi.org/10.1007/s11157-020-09551-z>
- [23] Naeem, M. A., Siddiqui, Q., Khan, M. R., Mushtaq, M., Wasim, M., Farooq, A., Naveed, T., Wei, Q. (2020). Bacterial cellulose-natural fiber composites produced by fibers extracted from banana peel waste. *Journal of Industrial Textiles*, 51(1): 990S-1006S. <https://doi.org/10.1177/1528083720925848>
- [24] Khawas, P., Deka, S.C. (2016). Isolation and characterization of cellulose nanofibers from culinary banana peel using high-intensity ultrasonication combined with chemical treatment. *Carbohydrate Polymers*, 137: 608-616. <https://doi.org/10.1016/j.carbpol.2015.11.020>
- [25] Rai, R., Dhar, P. (2022). Biomedical engineering aspects of nanocellulose: a review. *Nanotechnology*, 33(36): 362001. <https://dx.doi.org/10.1088/1361-6528/ac6fef>
- [26] Aoudi, B., Boluk, Y., Gamal El-Din, M. (2022). Recent advances and future perspective on nanocellulose-based materials in diverse water treatment applications. *Science of The Total Environment*, 843, 156903. <https://doi.org/10.1016/j.scitotenv.2022.156903>
- [27] Phanthong, P., Reubroycharoen, P., Hao, X., Xu, G., Abudula, A., Guan, G. (2018). Nanocellulose: Extraction and application. *Carbon Resources Conversion*, 1(1): 32-43. <https://doi.org/10.1016/j.crcon.2018.05.004>
- [28] Sharma, P.R., Joshi, R., Sharma, S.K., Hsiao, B.S. (2017). A simple approach to prepare carboxycellulose nanofibers from untreated biomass. *Biomacromolecules*, 18(8): 2333-2342. <https://doi.org/10.1021/acs.biomac.7b00544>
- [29] Zhan, C.B., Sharma, P.R., Geng, L., Sharma, S.K., Wang, R., Joshi, R., Hsiao, B.S. (2019). Structural characterization of carboxyl cellulose nanofibers extracted from underutilized sources. *Science China Technological Sciences*, 62(6): 971-981. <https://doi.org/10.1007/s11431-018-9441-1>
- [30] Xu, Y., Liu, X., Liu, X., Tan, J., Zhu, H. (2014). Influence of HNO₃/H₃PO₄-NANO₂ mediated oxidation on the structure and properties of cellulose fibers. *Carbohydrate Polymers*, 111: 955-963. <https://doi.org/10.1016/j.carbpol.2014.05.029>
- [31] Sharma, P.R., Chattopadhyay, A., Sharma, S.K., Hsiao, B.S. (2017). Efficient removal of UO₂²⁺ from water using carboxycellulose nanofibers prepared by the nitro-oxidation method. *Industrial & Engineering Chemistry Research*, 56(46): 13885-13893. <https://doi.org/10.1021/acs.iecr.7b03659>
- [32] Sharma, P.R., Chattopadhyay, A., Zhan, C., Sharma, S.K., Geng, L., Hsiao, B.S. (2018). Lead removal from water using carboxycellulose nanofibers prepared by nitro-oxidation method. *Cellulose*, 25(3): 1961-1973. <https://doi.org/10.1007/s10570-018-1659-9>
- [33] Chen, H., Sharma, S.K., Sharma, P.R., Chi, K., Fung, E., Aubrecht, K., Keroletswe, N., Chigome, S., Hsiao, B.S. (2021). Nitro-oxidized carboxycellulose nanofibers from moringa plant: effective bioadsorbent for mercury removal. *Cellulose*, 28(13): 8611-8628. <https://doi.org/10.1007/s10570-021-04057-5>

- [34] Zhu, L., Kumar, V., Banker, G.S. (2001). Examination of oxidized cellulose as a macromolecular prodrug carrier: preparation and characterization of an oxidized cellulose-phenylpropanolamine conjugate. *International Journal of Pharmaceutics*, 223(1): 35-47. [https://doi.org/10.1016/S0378-5173\(01\)00725-6](https://doi.org/10.1016/S0378-5173(01)00725-6)
- [35] Kotatha, D., Rungrodnimitchai, S. (2018). Synthesis and characterization of nanofiber of oxidized cellulose from nata de coco. *International Journal of Chemical Engineering*, 2018, 2787035. <https://doi.org/10.1155/2018/2787035>
- [36] Sharma, S.K., Sharma, P.R., Lin, S., Chen, H., Johnson, K., Wang, R., Borges, W., Zhan, C., Hsiao, B.S. (2020). Reinforcement of natural rubber latex using jute carboxycellulose nanofibers extracted using nitro-oxidation method. *Nanomaterials*, 10(4): 706. <https://doi.org/10.3390/nano10040706>
- [37] Chen, H., Sharma, P.R., Sharma, S.K., Alhamzani, A.G., Hsiao, B.S. (2022). Effective thallium (I) removal by nanocellulose bioadsorbent prepared by nitro-oxidation of sorghum stalks. *Nanomaterials*, 12(23): 4156. <https://doi.org/10.3390/nano12234156>
- [38] Nguyen, D.V., Nguyen, H.N., Tran-Thuy, T.M., Lam, H.H., Nguyen, L.Q., Ngo, D.T.H. (2022). One-step preparation of carboxyl cellulose nanofibers from lotus seedpod residue. *Vietnam Trade and Industry Review*, 10, 372-379. <https://tapchicongthuong.vn/bai-viet/tong-hop-mot-giai-doan-soi-nano-carboxyl-cellulose-tu-phu-pham-dai-sen-89494.htm>
- [39] Segal, L., Creely, J.J., Martin, A.E., Conrad, C.M. (1959). An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer. *Textile Research Journal*, 29(10): 786-794. <https://doi.org/10.1177/004051755902901003>
- [40] Thygesen, A., Oddershede, J., Lilholt, H., Thomsen, A.B., Ståhl, K. (2005). On the determination of crystallinity and cellulose content in plant fibres. *Cellulose*, 12(6): 563. <https://doi.org/10.1007/s10570-005-9001-8>
- [41] Kalita, E., Nath, B.K., Deb, P., Agan, F., Islam, M.R., Saikia, K. (2015). High quality fluorescent cellulose nanofibers from endemic rice husk: Isolation and characterization. *Carbohydrate Polymers*, 122: 308-313. <https://doi.org/10.1016/j.carbpol.2014.12.075>
- [42] Li, Q., Renneckar, S. (2011). Supramolecular structure characterization of molecularly thin cellulose I nanoparticles. *Biomacromolecules*, 12(3): 650-659. <https://doi.org/10.1021/bm101315y>
- [43] Schwanninger, M., Rodrigues, J.C., Pereira, H., Hinterstoisser, B. (2004). Effects of short-time vibratory ball milling on the shape of FT-IR spectra of wood and cellulose. *Vibrational Spectroscopy*, 36(1): 23-40. <https://doi.org/10.1016/j.vibspec.2004.02.003>
- [44] Akpomie, K.G., Conradie, J. (2020). Banana peel as a biosorbent for the decontamination of water pollutants. A review. *Environmental Chemistry Letters*, 18(4): 1085-1112. <https://doi.org/10.1007/s10311-020-00995-x>
- [45] Sharma, P.R., Zheng, B., Sharma, S.K., Zhan, C., Wang, R., Bhatia, S.R., Hsiao, B.S. (2018). High aspect ratio carboxycellulose nanofibers prepared by nitro-oxidation method and their nanopaper properties. *ACS Applied Nano Materials*, 1(8): 3969-3980. <https://doi.org/10.1021/acsnm.8b00744>

NOMENCLATURE

| | |
|------|------------------------------|
| BP | Banana peel |
| CCNF | Carboxyl cellulose nanofiber |