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EXTRACTION AND CHARACTERIZATION OF CELLULOSE FIBERS FROM LOTUS RECEPTACLE RESIDUES: A POTENTIAL SUSTAINABLE FEEDSTOCK FOR BIOPOLYMER REINFORCEMENT

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Abstract

The increasing generation of lotus receptacle residues from lotus processing highlighted the need for effective valorization strategies to produce value-added biobased materials. In this study, cellulose fibers were extracted from lotus receptacles through alkali treatment followed by hydrogen peroxide bleaching. The effects of H_2O_2 concentration, treatment temperature, and treatment time on the cellulose fiber extraction yield were systematically investigated to determine the suitable extraction conditions. The highest extraction yield of 13.64% was achieved using 6 wt% H_2O_2 at 80 ℃ for 1 h. X-ray diffraction analysis confirmed that the extracted cellulose fibers retained the cellulose I crystalline structure, while the crystallinity index increased from 34.39% to 47.67% after chemical treatment. Fourier transform infrared spectroscopy confirmed the presence of the characteristic functional groups of cellulose and revealed a marked reduction in the characteristic absorption bands associated with hemicellulose and lignin following chemical treatment. Scanning electron microscopy demonstrated that the extracted cellulose fibers exhibited a cleaner and smoother surface morphology than the raw lotus receptacles. Thermogravimetric analysis showed that the maximum degradation temperature increased from 298 to 333 °C after chemical treatment, indicating improved thermal stability of the extracted cellulose fibers. These findings demonstrated that lotus receptacles represented a promising renewable source of cellulose for reinforcing biopolymer-based materials, thereby enhancing the value of agricultural residues and supporting the development of the circular bioeconomy in Vietnam.

Keywords: *Cellulose fibers, lotus receptacle, yield optimization*.

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TRÍCH LY VÀ ĐÁNH GIÁ ĐẶC TÍNH CỦA SỢI CELLULOSE TỪ PHỤ PHẨM ĐÀI SEN: NGUỒN NGUYÊN LIỆU TIỀM NĂNG CHO ỨNG DỤNG GIA CƯỜNG VẬT LIỆU POLYMER SINH HỌC

Võ Văn Chương[1], Phạm Thị Kim Hiên[1], Nguyễn Đỗ Thùy Ngân[2], Nguyễn Thanh Huy[1], Bùi Phương Đông[1], Phạm Nguyễn Hồng Như[1] và Nguyễn Chí Thanh[1]*

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Tóm tắt

Sự gia tăng lượng phụ phẩm đài sen từ quá trình chế biến sen đặt ra yêu cầu phát triển các giải pháp tận dụng hiệu quả nhằm tạo ra các vật liệu sinh học có giá trị gia tăng. Trong nghiên cứu này, sợi cellulose được trích ly từ đài sen bằng phương pháp xử lý kiềm kết hợp tẩy trắng bằng hydro peroxide. Ảnh hưởng của nồng độ H_2O_2, nhiệt độ và thời gian xử lý đến hiệu suất trích ly sợi cellulose được nghiên cứu nhằm xác định các điều kiện trích ly thích hợp. Kết quả cho thấy hiệu suất cao nhất đạt 13,64% khi xử lý với H_2O_2 6% ở 80 °C trong 1 giờ. Phân tích nhiễu xạ tia X cho thấy sự hiện diện của cấu trúc tinh thể cellulose loại I với chỉ số độ kết tinh tăng từ 34,39% lên 47,67%. Kết quả phân tích quang phổ hồng ngoại biến đổi Fourier xác nhận sự hiện diện của các nhóm chức hóa học đặc trưng của cellulose, đồng thời cho thấy sự suy giảm của các đỉnh hấp thụ đặc trưng của hemicellulose và lignin sau khi xử lý hóa học. Phân tích nhiệt trọng lượng cho thấy nhiệt độ phân hủy cực đại tăng từ 298 lên 333 °C sau quá trình xử lý hóa học, cho thấy độ bền nhiệt của sợi cellulose thu được được cải thiện. Kính hiển vi điện tử quét cho thấy bề mặt sợi trở nên sạch hơn so với nguyên liệu thô. Những kết quả đạt được chỉ ra tiềm năng của đài sen như nguồn nguyên liệu cellulose có khả năng tái tạo, ứng dụng làm vật liệu gia cường trong vật liệu polymer sinh học, góp phần nâng cao giá trị phụ phẩm nông nghiệp theo định hướng kinh tế tuần hoàn tại Việt Nam.

Từ khóa: *Đài sen, sợi cellulose, tối ưu hóa hiệu suất.*

1. Introduction

Cellulose is the most abundant renewable natural polymer on Earth, being recognized as one of the most promising biopolymers for a wide range of applications in polymeric materials (Magalhães et al., 2023; Marinho, 2025). For its high mechanical strength, low density, biodegradability, biocompatibility, and the presence of abundant reactive hydroxyl groups on its surface, cellulose has been extensively used as a reinforcing filler in polymer composites and biopolymer matrices (Chandel et al., 2023; Huber et al., 2026; Kargarzadeh et al., 2017). These hydroxyl groups can form intermolecular hydrogen bonds and enhance interfacial interactions with the polymer matrix, thereby improving stress transfer efficiency and enhancing the tensile strength, elastic modulus, thermal stability, and barrier properties of the resulting materials.

In recent years, cellulose extraction from lignocellulosic biomass derived from agricultural residues has attracted increasing attention owing to the abundant availability and low cost of these feedstocks (Mujtaba et al., 2023). Various agricultural by-products, including sugarcane bagasse, rice straw, rice husks, corn cobs, banana pseudostems, and spent coffee grounds, have been extensively investigated as promising cellulose sources to produce biobased materials (Pickering et al., 2016; Venkatarajan et al., 2021). In addition to these commonly investigated agricultural residues, the lotus plant (*Nelumbo nucifer)* was recognized as a promising biomass resource that remained largely underutilized. Lotus is a high-value aquatic crop widely cultivated throughout many Asian countries, including Vietnam, for the production of flowers, rhizomes, and seeds used in the food and pharmaceutical industries (Nguyen & Phan, 2023). However, after harvesting, large quantities of agricultural residues, including leaves, stems, and particularly lotus receptacles, were generally discarded or utilized only for low-value applications. According to a recent report, lotus receptacles contained 88.25% carbohydrates and 22.90% crude fiber on a dry weight basis (Hu et al., 2015). In addition, a previous study on lotus peduncles reported that their major chemical constituents consisted of 41.40 ± 0.29% cellulose, 25.87 ± 0.64% hemicellulose, and 19.56 ± 0.32% lignin (Pan et al., 2011). For comparison, sugarcane bagasse and rice straw typically contain 32-45% and 36.2-47% cellulose, respectively (Alokika et al., 2021; Ning et al., 2021). The comparable cellulose content reported for lotus biomass highlights the potential of lotus receptacles as an alternative lignocellulosic feedstock for cellulose-based materials.

To date, research on lotus receptacles had primarily focused on the extraction of bioactive compounds, including polyphenols, flavonoids, and polysaccharides. For example, one study extracted polyphenols from lotus receptacles using subcritical water extraction to obtain extracts with high antioxidant activity (Yan et al., 2020). Another study optimized flavonoids extraction using macroporous adsorption resins (Limwachiranon et al., 2019), while a separate study isolated polysaccharides from lotus receptacles and evaluated their structural characteristics and antioxidant activities (Yuan et al., 2023). Recently, a study reported cellulose extraction from lotus receptacles by optimizing the alkali treatment process using response surface methodology (RSM) (Duc et al., 2026). The optimization considered three variables, namely NaOH concentration, treatment temperature, and reaction time, and achieved a cellulose extraction yield of 44.17% (Duc et al., 2026). However, the study primarily focused on the alkali treatment stage, whereas the effects of H_2O_2 bleaching conditions on the cellulose recovery yield and the physicochemical properties of the extracted cellulose were not systematically investigated.

Among the chemical methods used for cellulose isolation from lignocellulosic biomass, alkaline hydrogen peroxide (AHP) treatment has been recognized as an environmentally friendly approach because of its ability to effectively remove hemicellulose and lignin while

minimizing the formation of environmentally hazardous by-products compared with conventional methods (Ho et al., 2019). However, the efficiency of the AHP process has been reported to depend strongly on the treatment conditions, particularly the H_2O_2 concentration, treatment temperature, and reaction time. Therefore, optimizing these parameters has been considered essential for improving both the cellulose recovery yield and the quality of the extracted cellulose.

Accordingly, the effects of H_2O_2 concentration, treatment temperature, and reaction time on the cellulose recovery yield from lotus receptacles were investigated in this study to determine the suitable extraction conditions. The cellulose obtained under the suitable conditions was characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) to evaluate its chemical structure, crystallinity index, and surface morphology. These findings provided a basis for the effective utilization of lotus receptacles as a sustainable cellulose feedstock for future research and value-added applications.

2. Materials and methods

2.1. Materials

Lotus receptacles (LR) were collected from Chau Thanh Commune, Tay Ninh Province, Vietnam. Sodium hydroxide (NaOH) and hydrogen peroxide (H_2O_2) were purchased from Sigma-Aldrich. All chemicals were used as received without further purification.

2.2. Preparation of cellulose fibers

After collection, the lotus receptacles were washed several times with distilled water to remove surface impurities. The cleaned lotus receptacles were then cut into small pieces and dried at 70 ℃ for 36 h. The dried samples were ground into powder and stored in sealed zip-lock bags until use. For cellulose fiber extraction (CFE), 5 g of lotus receptacle powder was treated with 100 mL of 6 wt% NaOH solution at 80 ℃ under continuous stirring. After 1 h, the suspension was filtered and washed with distilled water until the pH reached 7. The alkali-treated solid residue was subsequently bleached with 100 mL of 6% H_2O_2 solution at 80 ℃ for 1 h, and the pH was adjusted to 10-11 using 5 wt% NaOH solution. After the bleaching treatment, the suspension was filtered and washed with distilled water until the pH reached 7. Finally, the extracted cellulose fibers were dried at 50 ℃ for 24 h. The H_2O_2 bleaching conditions were summarized in Tables 1, 2, and 3, and the overall procedure for CFE from lotus receptacles was illustrated in Figure 1.

Table 1. Effect of H_2O_2 concentration on CFE

No.	H_2O_2 concentration (%)	Temperature (℃)	Time (h)
1	6	80	2
2	8	80	2
3	10	80	2

Table 2. Effect of H_2O_2 treatment temperature on CFE

No.	H_2O_2 concentration (%)	Temperature (℃)	Time (h)
1	6	70	2
2	6	80	2
3	6	90	2

Table 3. Effect of H_2O_2 treatment time on CFE

No.	H_2O_2 concentration (%)	Temperature (℃)	Time (h)
1	6	80	1
2	6	80	2
3	6	80	3



Figure 1. Schematic diagram of the CFE **process from lotus receptacles**

2.3. Characterization

2.3.1. Cellulose fiber extraction yield

CFE yield was calculated using Eq. (1) (Alifa et al., 2024):

$$Y\ (\%) = \frac{m_1}{m_0} \times 100 \qquad (1)$$

where m_0 (g) and m_1 (g) represent the masses of the initial raw material and the extracted cellulose fibers, respectively.

2.3.2. X-ray diffraction (XRD)

The crystalline structure of the cellulose fiber and lotus receptacle samples was characterized by X-ray diffraction (XRD) using an Bruker D8 Advance diffractometer (Germany) equipped with Cu Kα radiation (λ = 1.54056 Å). The diffraction patterns were recorded over a 2θ range of 5-60° at an operating voltage of 40 kV and a current of 45 mA. The crystallinity index was determined according to Eq. (2) (Nuruddin et al., 2021):

$$CrI\ (\%) = \frac{(I_{200} - I_{am})}{I_{200}} \times 100 \qquad (2)$$

where I_{200} represents the maximum diffraction intensity of the (200) plane at 2θ = 22°, while I_{am} corresponds to the minimum diffraction intensity of the amorphous region located between the (110) and (200) planes.

2.3.3. *Fourier transform infrared (FT-IR) spectroscopy*

The chemical functional groups of the cellulose fiber and lotus receptacle samples were analyzed by FT-IR spectroscopy using a Nicolet 6700 instrument (Thermo Scientific). Measurements were performed over the wavenumber range of 4000-700 cm^{-1} at a resolution of 4 cm^{-1}, with 20 scans recorded for each sample.

2.3.4. Scanning electron microscopy (SEM)

The surface morphology of the lotus receptacle and the extracted cellulose fibers was examined using a scanning electron microscope (SEM, S-4800, Hitachi, Japan) operated at an accelerating voltage of 10 kV. Prior to SEM analysis, the samples were mounted on metal stub using carbon tape and coated with a thin layer of platinum (Pt).

3. Results and discussion

3.1. Effect of H_2O_2 treatment conditions on the CFE yield from lotus receptacle

To determine the appropriate conditions for CFE process, the effects of H_2O_2 concentration, treatment temperature, and treatment time on the cellulose fiber yield were investigated, as shown in Figure 2. The results indicated that the cellulose fiber yield decreased with increasing H_2O_2 concentration, treatment temperature, and treatment time.

As the H_2O_2 concentration increased from 6% to 10%, the cellulose fiber yield decreased markedly from 12.23% to 8.28% (Figure 2a). In addition, the cellulose fiber yield decreased slightly from 12.19% to 11.10% as the H_2O_2 treatment temperature increased from 70 to 90 ℃ (Figure 2b). As shown in Figure 2c, the H_2O_2 treatment time exerted a pronounced effect on the cellulose fiber extraction yield. When the treatment time was extended from 1 h to 3 h, the cellulose fiber yield decreased from 13.64% to 9.53%. This decrease was attributed to the fact that, under alkaline conditions, H_2O_2 dissociated to form HOO^-, while the subsequent decomposition of H_2O_2 generated highly reactive species such as hydroxyl radicals (·OH) and superoxide-related species ($O_2^{\bullet -}$) (Gould, 1985). These reactive species oxidized lignin structures, promoted the cleavage of lignin-related linkages, and facilitated lignin removal. The oxidative disruption of the lignin-carbohydrate matrix also facilitated the removal of a portion of the hemicellulose fraction and other amorphous components from the lotus receptacle, thereby increasing the purity of the cellulose fibers (Kuznetsov et al., 2020; Loor, 2022).

Moreover, H_2O_2 treatment under alkaline conditions proceeded most effectively within the temperature range of 80-100 ℃ (Loor, 2022). However, further increases in the H_2O_2 concentration, treatment temperature, and treatment time generated larger amounts of reactive free radicals, which attacked the cellulose chains and caused partial cellulose degradation (Gan et al., 2026; Kuznetsov et al., 2018). Consequently, CFE yield decreased, and the physicochemical properties of the extracted cellulose fibers were adversely affected. These results indicated that the suitable reaction conditions for extracting cellulose fibers with high yield were 6% H_2O_2, 80 ℃, and 1 h. CFE yield obtained in the present study was lower than that reported in a previous study on cellulose fiber extraction from rice straw, in which the highest extraction yield of 46.2% was achieved under the optimum conditions of 4% H_2O_2 for 1 h at pH 12 after supercritical water pretreatment (Freitas et al., 2024). In addition, another study on cellulose extraction from Robusta coffee husks reported cellulose recovery yields ranging from 8.58% to 10.33%, with an average yield of 9.25%, when bleaching was performed using 3% H_2O_2 at 90 ℃ for 90 min (Clara Dione et al., 2025). Comparison with these previously reported results indicated that the cellulose extraction yield depended strongly on both the raw material source and the treatment conditions.

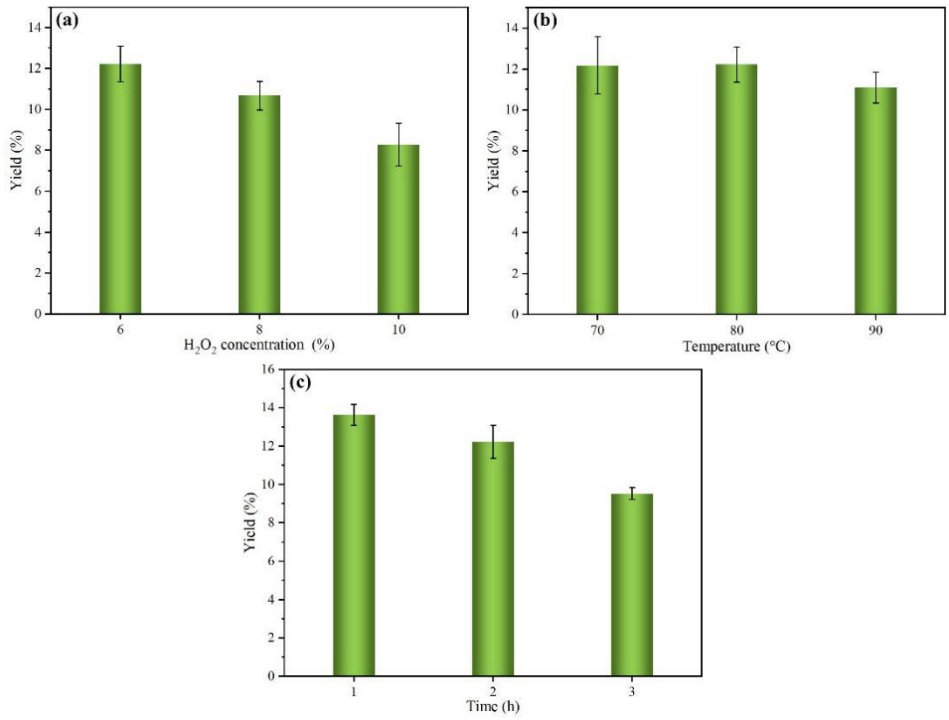

Figure 2. Effects of (a) H_2O_2 concentration, (b) treatment temperature, and (c) treatment time on CFE yield

3.2. XRD analysis

The XRD patterns of the lotus receptacle and the cellulose fibers extracted under the optimal conditions were presented in Figure 3. The results showed that both samples exhibited the characteristic diffraction peaks of cellulose I at 2θ values of approximately 15.67°, 22.56°, and 35.01°, corresponding to the (110), (200), and (004) planes, respectively (Saad et al., 2025).

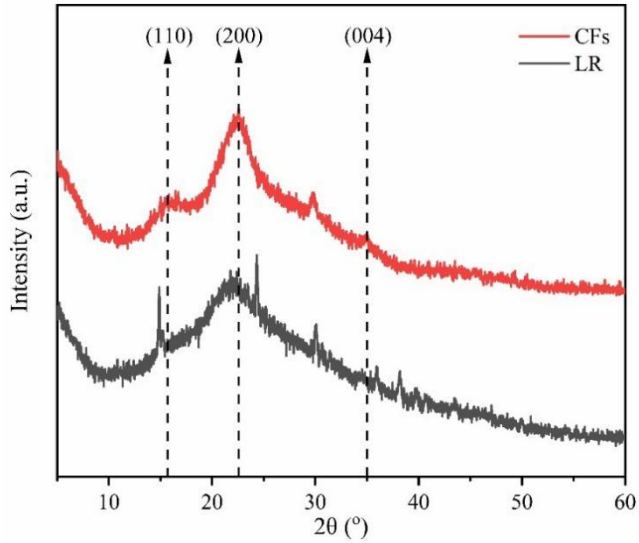

Figure 3. XRD patterns of the lotus receptacle and extracted cellulose fibers

After chemical treatment, the diffraction peak corresponding to the (200) plane of the cellulose fibers became sharper and more intense than that of the raw material (Gupta et al.,

2021). In addition, the crystallinity index of the extracted cellulose fibers reached 47.67%, which was higher than that of the raw lotus receptacle (34.39%). This increase was attributed to the chemical treatment, which effectively removed amorphous constituents, including hemicellulose, lignin, and other non-cellulosic components that originally surrounded the crystalline cellulose domains within the raw material (Dominic C.D et al., 2020; Sadare et al., 2022). These findings were consistent with those reported in a previous study (Saad et al., 2025).

3.3. FTIR result

The changes in the chemical functional groups of the extracted cellulose fibers relative to the lotus receptacle were characterized by FTIR spectroscopy, as presented in Figure 4. The FTIR spectrum of the lotus receptacle exhibited a broad absorption band in the wavenumber range of 3700-3000 cm^{-1}, which was attributed to the O-H stretching vibration of hydroxyl groups in the cellulose structure, with a maximum absorption peak at 3383 cm^{-1} (Duc et al., 2026). The symmetric and asymmetric stretching vibrations of the C-H bonds were observed in the wavenumber range of 2920-2855 cm^{-1} (Ho et al., 2024). Furthermore, the absorption bands at 1740 cm^{-1} and 1526 cm^{-1} were assigned to the C=O stretching vibration of carbonyl groups and the C=C stretching vibration of aromatic rings associated with hemicellulose and lignin, respectively (Saad et al., 2025; Song et al., 2019). Furthermore, the absorption band at 1508 cm^{-1} was attributed to the C=C stretching vibration of aromatic rings present in the lignin structure (Başaran Kankılıç & Metin, 2020). In addition, the absorption band at 1317 cm^{-1} was attributed to the C-C and C-O skeletal vibrations. The absorption bands at 1061 cm^{-1} and 897 cm^{-1} were assigned to the C-O-C skeletal vibration of the pyranose ring and the C-O-C stretching vibration of the β-glycosidic linkage in cellulose, respectively (Al-Awa et al., 2023; Elmoudnia et al., 2023).

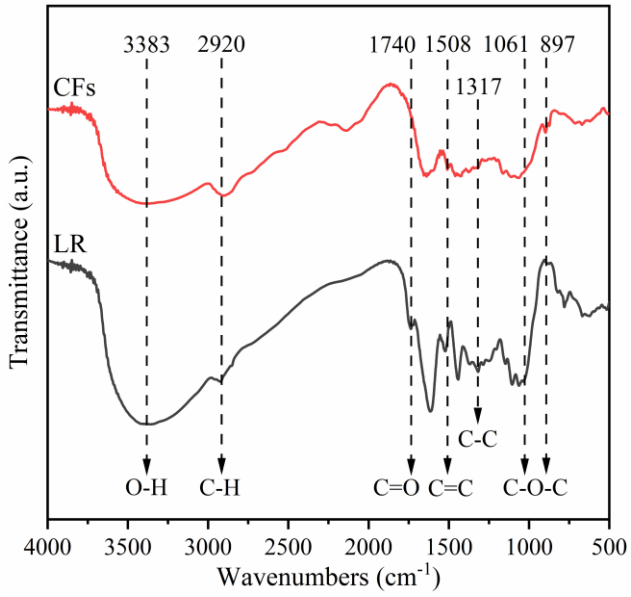

Figure 4. FTIR spectra of the lotus receptacle and extracted cellulose fibers

Compared with the FTIR spectrum of the lotus receptacle, the FTIR spectrum of the extracted cellulose fibers showed the disappearance of the absorption band at 1740 cm^{-1} and a decrease in the intensity of the absorption band at 1508 cm^{-1}, which were associated with hemicellulose and lignin, respectively. These changes indicated that the NaOH treatment followed by H_2O_2 bleaching effectively removed a portion of the hemicellulose and lignin

from the lotus receptacle structure (Han & Geng, 2023). These findings were consistent with the XRD results discussed above.

3.4. Thermogravimetric analysis

The thermal stability of the lotus receptacles and extracted cellulose fibers was presented in Figure 5. The TGA results showed that both samples underwent three main stages of thermal degradation. The first stage was attributed to the evaporation of moisture and low-molecular-weight compounds with relatively low thermal stability, resulting in a mass loss of approximately 7% at temperatures below 140 ℃ (Elango et al., 2025). The second stage represented the main thermal degradation region of the materials, with the highest mass losses of 70.62% for the cellulose fibers and 50.96% for the lotus receptacles occurring over the temperature range of approximately 140-380 ℃. The substantial mass loss observed during this stage was attributed to the thermal degradation of hemicellulose and cellulose present in the chemical structures of the lotus receptacles and extracted cellulose fibers (Nazbakhsh et al., 2025). Notably, the cellulose fibers exhibited a greater mass loss than the lotus receptacles during this stage. This phenomenon was attributed to the chemical treatment, which reduced the fiber dimensions and consequently increased the specific surface area of the cellulose fibers compared with that of the raw material. The increased specific surface area resulted in greater exposure to heat, thereby leading to a more pronounced mass loss within this temperature range (Ho et al., 2024). The final stage occurred over the temperature range of 380-700 ℃, with mass losses of approximately 37.31% and 24.15% for the lotus receptacles and cellulose fibers, respectively. The mass loss during this stage was attributed to the slow thermal degradation of lignin remaining in both samples. Finally, the residual mass after thermal decomposition was lower for the cellulose fibers than for the lotus receptacles, indicating that the chemical treatment effectively removed non-cellulosic components and consequently increased the cellulose purity (Romruen et al., 2022). In addition, Figure 5b showed that the maximum degradation temperature (T_{max}) of the cellulose fibers (333 ℃) was higher than that of the lotus receptacles (298 ℃). This increase in T_{max} was caused by the effective removal of a portion of hemicellulose and lignin from the lotus receptacle structure during the chemical treatment, which consequently enhanced the thermal stability of the extracted cellulose fibers (Romruen et al., 2022).

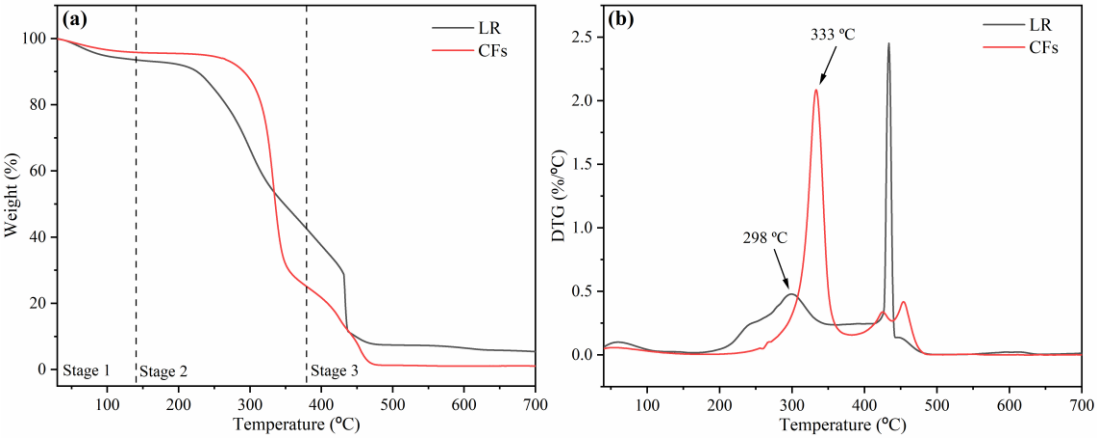

Figure 5. (a) TGA and (b) DTG curves of the lotus receptacles and extracted cellulose fibers

3.5. Surface morphology of lotus receptacles and extracted cellulose fibers

SEM images of the lotus receptacle sample and the extracted cellulose fibers were presented in Figure 6. As seen in Figure 6 (a, b), the lotus receptacle sample showed a rough

surface with fiber bundles tightly bound together (Hossain et al., 2024); in addition, the presence of impurity patches on the surface was also observed. This phenomenon was due to the presence of compounds such as lignin, hemicellulose, and pectin, which acted as binding agents coating the cellulose fibers (Thi Thuy Van et al., 2022). Furthermore, after the chemical treatment process, the cellulose fibers (CFs) showed a smoother surface and displayed a fibrous structure (Figure 6 c, d) (Ravindran et al., 2019). This indicated that the chemical treatment process partially removed amorphous impurities such as hemicellulose and lignin from the structure of the lotus receptacle (Raza et al., 2022). This result was consistent with findings of previously reported study (Malarat et al., 2023).

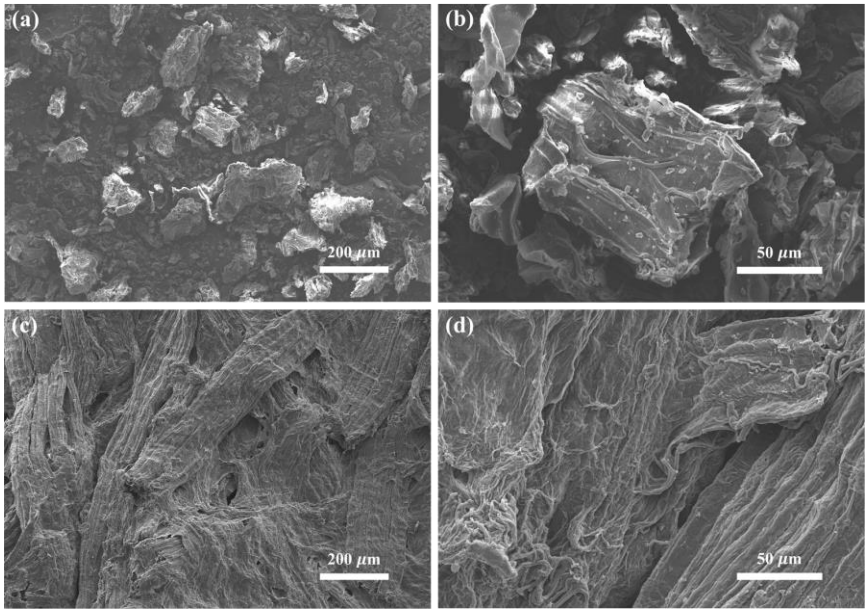

Figure 6. SEM images of (a, b) lotus receptacles and (c, d) extracted cellulose fibers

4. Limitations and future development

Although the present study has demonstrated the scientific and practical significance of cellulose extraction from lotus petioles, several limitations should be considered regarding its practical applicability. First, the cellulose purity was not quantitatively determined, including the contents of α-cellulose, lignin, and residual hemicellulose. In addition, the extraction process was conducted at the laboratory scale and involved alkaline treatment followed by repeated washing steps, which may result in considerable consumption of water and chemicals. Furthermore, the treatment and management of the alkaline wastewater generated during the extraction process have not yet been investigated or evaluated.

Future studies should focus on optimizing the extraction process to improve the quality of the cellulose material while reducing water, chemical, and energy consumption. Quantitative compositional analysis should be conducted to determine the contents of cellulose, hemicellulose, and lignin, thereby providing a more comprehensive assessment of the quality of the obtained material. Further investigations into the treatment and recovery of alkaline wastewater are also needed to evaluate the overall feasibility and sustainability of the process. Finally, further studies should fabricate and evaluate polymer composites containing the obtained cellulose material to assess its potential as a reinforcing agent, with particular emphasis on the effects of its content and structural characteristics on the properties of the resulting composites.

5. Conclusion

In this study, cellulose fiber was successfully extracted from lotus receptacles using alkali treatment followed by hydrogen peroxide bleaching. The highest cellulose fiber extraction yield of 13.64% was obtained under the suitable extraction conditions of 6 wt% H_2O_2, 80 ℃, and a treatment time of 1 h. Structural characterization suggested the effective removal of hemicellulose and lignin while preserving the cellulose I crystalline structure. The crystallinity index increased from 34.39% for the raw lotus receptacle to 47.67% for the extracted cellulose. FTIR analysis confirmed the presence of the characteristic functional groups of cellulose, whereas the TGA results showed that the extracted cellulose fibers exhibited higher thermal stability than the lotus receptacles. Furthermore, SEM observations revealed a cleaner fiber surface after chemical treatment. These findings suggest that lotus receptacles are a promising renewable feedstock for cellulose production. The extracted cellulose fibers have potential application as a reinforcing material for biodegradable polymer systems, contributing to the valorization of lotus-processing residues and supporting the development of the circular economy.

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