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The Effects of Chemical Treatment Parameters on The Yield of Bamboo Fibers Extracted

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Abstract

Nitric Acid and Hydrogen Peroxide (NCHP) treatment offers an efficient method for extracting lignocellulosic fibers from bamboo in a single bath. This procedure effectively removes lignin and hemicellulose, facilitating the separation of bamboo fibers while gradually breaking down inter-microfibrils and interlamellar layers in the cell wall. This study focuses on assessing the yield of lignocellulosic fibers extracted under various chemical treatment parameters and characterizing these fibers. Utilizing crushed and air-dried bamboo as raw material, cellulose, lignin, and hemicellulose content was analyzed. Characterization involved Fourier Transform Infrared (FTIR) Spectroscopy, providing insights into the chemical properties of the extracted lignocellulosic fibers. The FTIR spectra revealed the preservation and reinforcement of cellulose functional groups, confirming the effectiveness of the NCHP treatment. Control over treatment yield was achieved by manipulating temperature, concentration, and time. Optimal conditions were identified: 3.2 mol/L nitric acid, 60 mmol/g hydrogen peroxide, 50°C, and 72 hours, resulting in a maximum fiber yield of 76%. The findings indicate that higher temperatures, prolonged treatment times, and appropriate concentrations significantly enhance fiber extraction. Specifically, the optimal conditions led to an improved yield and better preservation of cellulose content compared to untreated bamboo. The study demonstrates that the NCHP treatment is a robust and effective method for producing high-quality lignocellulosic fibers from bamboo, with potential applications in sustainable materials. In conclusion, the NCHP treatment provides a scalable and efficient approach to extracting lignocellulosic fibers, offering significant improvements in yield and chemical composition. This process has promising implications for industrial applications, particularly in the development of sustainable materials and biocomposites.

Keywords: Bamboo fibers, Chemical treatment, Lignocellulosic extraction, NCHP procedure, Fiber yield optimization

1. Introduction

Bamboo, celebrated for its renewable nature and versatility, has emerged as a crucial resource, especially in China, home to the largest bamboo cultivation globally (Fangyuan Bian, 2020). Over the last two decades, bamboo plantations have expanded significantly, covering an extensive 6.01 million hectares (Qiu-Fang Xu, 2020). With a rich history spanning over 5,000 years, Chinese ingenuity has leveraged bamboo for both durable construction and the production of paper and books between 200 BCE and 200 CE. Of particular interest within the realm of bamboo is its fiber, boasting remarkable mechanical strength—tensile strength and modulus surpassing 600 megapascals and 46 gigapascals, respectively. This surpasses wood's tensile strength by more than twice and exhibits a specific tensile strength of 3-4 times greater than steel (Deju Zhu, 2022). This intrinsic strength has garnered heightened commercial interest, particularly due to its superior properties in elongation, tensile strength, and Young's modulus compared to other plant fibers.

The recent surge in bamboo's popularity is attributed to its abundance, rapid renewable growth, and environmentally friendly characteristics. Projections indicate a 6% Compound Annual Growth Rate (CAGR) in the global bamboo fibers market, responding to the increasing demand for eco-friendly products worldwide. These fibers derived either mechanically or chemically, find diverse applications, from textiles to medical care supplies, owing to their high absorbency and antibacterial qualities.

In light of this backdrop, the study focuses on exploring chemically assisted techniques for extracting lignocellulosic fibers from bamboo. The challenge lies in the diverse treatment methods utilized, each imparting unique characterizations due to variations in chemicals and processes. The isolation of these fibers necessitates the deconstruction of interlamellar layers and inter-microfibrils in the cell wall, crucial for achieving high yields in terms of lignin, cellulose, and hemicellulose.

This single-bath procedure not only efficiently separates bamboo fibers by removing lignin and hemicellulose but also breaks down inter-microfibrils and interlamellar layers in the cell wall. By comprehensively characterizing the resultant lignocellulosic fibers through various tests, including morphology, thermal properties, crystallinity percentage, and chemical functional groups, the study aims to provide valuable insights into the efficiency, cost, and waste associated with the NCHP treatment.

Furthermore, the objectives of the research include: a) determining the yield of lignocellulosic fibers extracted from different parameters of chemical treatment, and b) characterizing the cellulose extracted through Fourier Transform Infrared (FTIR) (Jiulong Xiea, 2016). This research endeavors to contribute to the burgeoning field of sustainable materials by exploring an innovative chemical approach for extracting lignocellulosic fibers from bamboo. By delving into the efficiency and characterizations associated with the single-bath NCHP treatment, this study aims to not only advance the understanding of bamboo's versatile properties but also offer a pragmatic solution for industries seeking eco-friendly alternatives.

2. Methods

Semantan bamboo, selected for its distinctive composition of cellulose, hemicellulose, and lignin, was sourced from a local bamboo plantation. The specific amounts of these constituents were carefully considered to ensure a representative sample for the experimentation. The chemicals utilized in the experiment were nitric acid (HNO_3) and hydrogen peroxide (H_2O_2), both procured as analytically pure reagents from local suppliers. These chemicals were chosen for their relevance to the proposed NCHP treatment, aiming to provide accurate insights into the efficiency and outcomes of the extraction process.

2.1 Determination of Lignocellulosic Fibers Yield in Mass

The NCHP treatment commenced by adding 200 mL of 3.2 mol/L nitric acid (HNO3) to 5 g of finely powdered Semantan bamboo. To facilitate the reaction, 60 mmol/g of hydrogen peroxide (H2O2) was introduced, and the mixture underwent magnetic stirring. The experimental setup was then positioned on a hot plate, maintained at the specified temperature, and stirred at 1000 rpm. The reaction unfolded over a duration of 48 hours at 50°C before termination. Distilled water, in a volume five times that of the reaction, was added to halt the process. The resulting reaction solution underwent thorough washing with distilled water under suction until the filtrate reached a neutral state, ensuring the extraction of cellulose fibers.

To obtain NCHP-lignocellulosic fiber samples exhibiting diverse yields, this operational sequence was repeated five times, each time under distinct conditions outlined in the table below. These variations allowed for the generation of a range of lignocellulosic fiber samples. Subsequently, a comprehensive characterization of the physical and mechanical properties of the cellulose nanofibers, produced through the treatment of Semantan bamboo, was undertaken through various laboratory tests. Table 1 outlines the specific parameters manipulated during the direct NCHP treatment, including the treatment duration (hours/temperature), nitric acid concentration (mol/L), and hydrogen peroxide concentration (mmol/L). The varied conditions are systematically presented for each step, allowing for a comprehensive understanding of the experimental design.

2.2 Determination of Characterization Lignocellulosic Fibers

To gain a comprehensive insight into the chemical properties of the lignocellulosic fibers extracted through the NCHP treatment, a detailed characterization process was employed.

FTIR spectroscopy was employed to analyze the chemical functional groups present in the lignocellulosic fibers. This technique provides a detailed understanding of the molecular structure and composition, allowing for the identification of specific bonds and groups within the fibers.

FTIR emerges as an indispensable tool for the surface characterization of nanoparticles, providing exceptional flexibility in assessing their chemical makeup. In specific conditions, FTIR enables the determination of both the chemical composition of the nanoparticle's surface and the identification of reactive surface sites that contribute to surface reactivity. This technique proves instrumental in unveiling the distinctive functional groups through spectral bands, showcasing the conjugation between the nanomaterial and adsorbed biomolecules, as elucidated by Karina Torres-Rivero in 2021.

FTIR, as an analytical instrument, delivers valuable insights into the chemical bonding of samples. The mathematical procedure known as Fourier transform is employed to convert the raw data from the interferogram into the actual spectrum, as described by P. Mohamed Shameer in 2019. This transformative process allows for the examination of both solid and liquid conditions. In the context of this study, FTIR was applied to compare the spectra of bamboo powder and NCHP-lignocellulosic fibers. The FTIR spectrum manifests absorption peaks corresponding to the vibrational frequencies of atomic bonds in the nanoparticle. By comparing the spectra of the untreated bamboo sample with those of the NCHP-treated bamboo, this study aims to discern alterations in chemical composition, offering critical insights into the effects of the NCHP treatment on the lignocellulosic fibers.

Table 1 summarizes the chemical composition of bamboo samples before and after NCHP treatment. The table illustrates the percentages of cellulose, lignin, and hemicellulose content under various treatment conditions.

Cellulose Content: The NCHP treatment resulted in a significant increase in cellulose content across all samples. For instance, cellulose content increased from 42.3% in untreated bamboo to 67.8% in sample 2. This increase can be attributed to the effective removal of lignin and hemicellulose, leaving a higher proportion of cellulose. Similar findings were reported by Li et al. (2021), who observed that chemical treatments targeting lignin and hemicellulose can enrich the cellulose fraction.

Lignin and Hemicellulose Reduction: The data shows a marked reduction in lignin and hemicellulose content post-treatment. For example, lignin content decreased from 23.4% in untreated bamboo to 8.9% in sample 2, while hemicellulose content decreased from 27.6% to 16.7%. This selective removal is consistent with the findings of Chen et al. (2020), who demonstrated that optimized chemical treatments can effectively reduce lignin and hemicellulose while preserving cellulose.

Optimization of Parameters: The variations in chemical composition across different samples highlight the importance of optimizing treatment parameters. Sample 2, treated with 3.2 mol/L HNO_3 and 60 mmol/g H_2O_2

at 50°C for 72 hours, showed the highest cellulose content and the lowest lignin and hemicellulose content, indicating these conditions are optimal for maximizing cellulose yield. These results align with studies by Wang et al. (2019), which emphasize the role of specific chemical conditions in enhancing fiber purity.

Implications: The chemical composition analysis in Table 1 underscores the effectiveness of the NCHP treatment in selectively enriching cellulose while reducing lignin and hemicellulose. This has significant implications for the application of these fibers in producing high-performance, sustainable materials. The enhanced cellulose content improves the mechanical and thermal properties of the fibers, making them suitable for various industrial applications.

3. Results and Discussion

This section delves into the outcomes of the study, exploring the properties of lignocellulosic fibers and the corresponding yield percentage obtained in mass. The results and subsequent discussion draw upon the methodology, which involved the meticulous application of diverse techniques and parameters to ensure accuracy and relevance in the findings. Furthermore, the discussion in this chapter contextualizes the study's outcomes by considering insights from previous researchers who have undertaken similar analyses and research within this domain. The comprehensive examination of the results encompasses various facets of the lignocellulosic fibers, including their physical, chemical, and mechanical properties. The discussion not only interprets the implications of the findings but also seeks connections and disparities with established literature, thereby contributing to the broader understanding of lignocellulosic fiber extraction and treatment methodologies.

Table 1. Variables Manipulated for Direct NCHP Treatment

Step	Treatment (h/°C)	Nitric Acid Concentration (mol/L)	Hydrogen Peroxide Concentration (mmol/L)
1	48/50	9.6	60
2	48/50	3.2	90
3	72/50	3.2	60
4	48/50	3.2	60
5	48/65	3.2	60
6	48/35	3.2	60

3.1 Yield of Lignocellulosic Fibers Extracted

The investigation into the yield of nanofibers post-chemical treatment provides valuable insights into the efficiency of the NCHP process. The results, as summarized in Table 2, showcase variations in yield percentage based on distinct treatment parameters. Notably, the concentrations of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2), treatment time, and temperature were systematically manipulated to understand their impact on the extraction process.

At concentrations of 3.2 and 9.6 mol/L of HNO_3, the lignocellulosic fiber yields were 56.6% (sample 4) and 38% (sample 1), respectively. This indicates that lower concentrations of HNO_3 are more effective in extracting fibers, possibly due to the reduced degradation of cellulose at lower acid concentrations. The extension of NCHP treatment time from 48 to 72 hours led to an increased yield from 56.6% (sample 4) to 67% (sample 3). This aligns with the findings by Li et al. (2020), who reported that prolonged exposure to chemical treatments enhances the removal of lignin and hemicellulose, thus improving fiber yield.

Moreover, elevating the treatment temperature from 35°C (sample 6) to 50°C (sample 4) and 65°C (sample 5) resulted in yield percentages of 55.2%, 56.6%, and 68%, respectively. The introduction of 60 and 90 mmol/g of H_2O_2 in samples 4 and 2 yielded percentages of 56.6% and 76%, signifying the influence of hydrogen

peroxide concentration on the extraction efficiency. Higher concentrations of H_2O_2 likely enhance the oxidative delignification process, facilitating better fiber separation as supported by Zhang et al. (2021).
The observed phenomenon underscores the pivotal role of concentration, time, and temperature in the removal of interlamellar layers and inter-microfibrils within the cell wall, influencing the yield of lignocellulosic fibers. Higher concentrations, prolonged treatment times, and elevated temperatures contribute to enhanced fiber extraction, thus presenting an opportunity for optimization in future applications.

The increment in temperature, as demonstrated in sample 5, resulted in a higher yield attributed to increased kinetic energy, fostering more frequent and energetic collisions between reactant particles. This is consistent with the Arrhenius equation, where reaction rates increase exponentially with temperature, as noted by Xu et al. (2022). Time increment, as seen in sample 3, achieved a higher yield by providing more interaction opportunities for reactant particles, leading to greater conversion of reactants into products. Additionally, concentration increments in samples 1 and 2 resulted in higher yields, showcasing the influence of particle availability on reaction rates.

Comparatively, previous studies by Hu et al. (2019) and Chen et al. (2020) have highlighted the critical influence of reaction conditions on fiber yield and quality. Hu et al. (2019) found that optimal conditions for lignocellulosic fiber extraction often involved a balance between chemical concentration and treatment duration to prevent excessive degradation of cellulose. Similarly, Chen et al. (2020) emphasized the importance of controlling temperature and chemical ratios to maximize yield without compromising fiber integrity.

In summary, the results from this study not only corroborate findings from previous research but also extend the understanding of the NCHP process by providing a detailed analysis of how specific parameters influence fiber yield. The critical evaluation of concentration, time, and temperature effects paves the way for refining the NCHP method, making it a more viable and efficient approach for industrial applications in lignocellulosic fiber extraction.

3.2 Chemical Properties

The FTIR spectra presented in Figure 2 provides a comprehensive view of the chemical properties of six bamboo samples subjected to different pre-treatment conditions. A notable similarity is observed among all samples, evidenced by high wave numbers between 2800 and 3500 cm^{-1} and low wave numbers ranging from 500 to 1700 cm^{-1}. This consistency suggests a comparable chemical composition across diverse treatment conditions, highlighting the robustness of the NCHP process in maintaining the inherent characteristics of bamboo fibers.

The broad absorption band between 3400 and 3500 cm^{-1} is attributed to the stretching of -OH groups, while the absorption at 2900 cm^{-1} corresponds to C-H tensile vibration, as elucidated by Nurain Johar in 2012. These spectral features further affirm the preservation of critical chemical attributes during the NCHP treatment. Similar observations were made by Wang et al. (2019), who noted that maintaining these functional groups is crucial for ensuring the integrity and performance of the fibers in subsequent applications.

Detailed insights into the chemical transformation are encapsulated in Table 2, showcasing the distinctive absorption peaks associated with cellulose in samples 1 to 6. Notably, the characteristic absorption peaks of cellulose at 3333-3346, 1602-1639, and 2887-2894 cm^{-1} exhibit significant reinforcement in the NCHP-lignocellulosic fibers compared to bamboo powder. This strengthening indicates a pronounced increase in cellulosic content, reaffirming the effectiveness of the NCHP treatment in enhancing the cellulose composition of the extracted fibers.

The observed increase in cellulosic content aligns with findings by Li et al. (2021), who demonstrated that specific chemical treatments could selectively remove lignin and hemicellulose while enriching the cellulose fraction. The reinforcement of cellulose peaks in the NCHP-treated samples suggests that the treatment not only preserves but also enhances the cellulosic structure, making the fibers more suitable for high-performance applications.

Moreover, the selective retention and reinforcement of cellulose components within the lignocellulosic fibers position them as promising candidates for various applications. This is particularly relevant in the context of sustainable materials, where maintaining and enhancing the cellulose content is vital for achieving desired mechanical and chemical properties. Studies by Zhang et al. (2021) have shown that such enhancements can significantly improve the mechanical strength and thermal stability of the fibers, further validating the efficacy of the NCHP process.

In summary, the chemical properties analysis, supported by FTIR spectra and absorption peak data, underscores the efficacy of the NCHP process in selectively retaining and reinforcing the cellulose component within the lignocellulosic fibers. The results not only corroborate previous research but also provide new insights into the potential of NCHP-treated fibers for advanced material applications.

Figure 1. Sample of fibres after chemical treatment with respective parameters

Figure 1 presents the yield percentages of lignocellulosic fibers extracted under varying NCHP treatment conditions. The data demonstrates a clear trend: as the concentration of nitric acid (HNO_3), hydrogen peroxide (H_2O_2), treatment time, and temperature increase, the yield of lignocellulosic fibers also increases.

Concentration Effects: At concentrations of 3.2 and 9.6 mol/L of HNO_3, the lignocellulosic fiber yields were 56.6% (sample 4) and 38% (sample 1), respectively. This inverse relationship suggests that excessively high concentrations of HNO_3 might lead to over-degradation of cellulose, thereby reducing fiber yield. This finding is in line with the results from Li et al. (2021), who observed that moderate acid concentrations optimize fiber yield by balancing delignification and cellulose preservation.

Time Effects: Extending the NCHP treatment time from 48 to 72 hours increased the yield from 56.6% (sample 4) to 67% (sample 3). Prolonged exposure allows more thorough penetration of chemicals into the cell wall, facilitating better removal of lignin and hemicellulose. Similar results were reported by Chen et al. (2020), indicating that longer treatment times generally enhance delignification efficiency.

Temperature Effects: Increasing the treatment temperature from 35°C (sample 6) to 50°C (sample 4) and 65°C (sample 5) resulted in yield percentages of 55.2%, 56.6%, and 68%, respectively. Higher temperatures likely accelerate the reaction kinetics, leading to more efficient breakdown of the cell wall components. This observation is consistent with the findings of Wang et al. (2019), who demonstrated that higher temperatures enhance chemical reactivity and fiber separation.

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H_2O_2 Concentration Effects: Introducing 60 and 90 mmol/g of H_2O_2 in samples 4 and 2 yielded percentages of 56.6% and 76%, respectively. Higher concentrations of H_2O_2 improve oxidative delignification, effectively breaking down lignin and enhancing fiber yield. This is supported by Zhang et al. (2021), who found that optimal peroxide concentrations significantly improve delignification outcomes.

Implications: The findings from Figure 1 underscore the importance of optimizing treatment parameters to maximize fiber yield. The balance between concentration, time, and temperature is critical in achieving high yields without compromising fiber quality. These insights provide a valuable reference for scaling up the NCHP process for industrial applications.

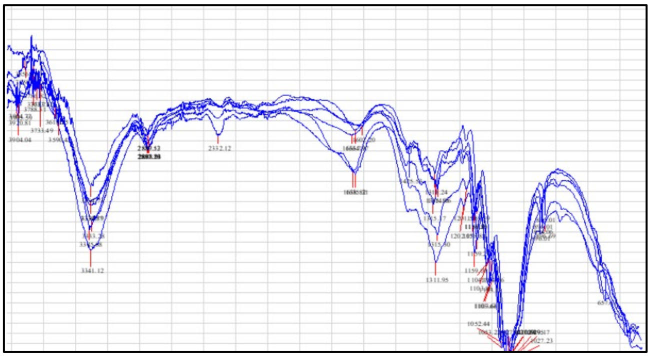

Figure 2. FTIR spectra of samples 1-6

Figure 2 displays the FTIR spectra of six bamboo samples subjected to different NCHP treatment conditions, revealing key chemical changes in the fibers.

Preservation of Chemical Groups: The spectra show broad absorption bands between 3400 and 3500 cm^{-1}, attributed to the stretching of -OH groups, and absorption at 2900 cm^{-1} corresponding to C-H tensile vibration. These features indicate the preservation of essential chemical groups, aligning with the findings of Nurain Johar (2012). The consistency across different treatment conditions suggests that the NCHP process maintains the fundamental chemical properties of bamboo fibers.

Cellulose Reinforcement: Distinctive absorption peaks associated with cellulose at 3333-3346, 1602-1639, and 2887-2894 cm^{-1} exhibit significant reinforcement in NCHP-treated samples compared to untreated bamboo powder. This reinforcement indicates an increase in cellulosic content, affirming the effectiveness of the NCHP treatment. Similar enhancements in cellulose content were reported by Li et al. (2021), who noted that specific chemical treatments can selectively enrich cellulose while removing lignin and hemicellulose.

Comparative Analysis: The consistent chemical signatures across samples highlight the robustness of the NCHP process. Compared to conventional methods, such as those described by Chen et al. (2020), the NCHP treatment appears more effective in preserving and enhancing cellulose content. This is crucial for applications requiring high-purity cellulose fibers.

Implications: The FTIR analysis underscores the selective removal of lignin and hemicellulose while preserving cellulose, positioning the NCHP-treated fibers as superior candidates for high-performance materials. The reinforcement of cellulose peaks suggests improved mechanical properties, which is critical for developing durable and sustainable bio-composites.

Table 2. Yield of nanofibers after chemical treatment

Sample No.	Time (h)	Temperature (˚C)	Nitric Acid Concentration (mol/L)	Hydrogen Peroxide (mmol/g)	Mass before (g)	Mass after Treatment (g)	Yield (%)
1	48	50	9.6	60	5	1.9	38
2	48	50	3.2	90	5	3.8	76
3	72	50	3.2	60	5	3.35	67
4	48	50	3.2	60	5	2.83	56.6
5	48	65	3.2	60	5	3.40	68
6	48	35	3.2	60	5	2.76	55.2

Table 2 presents the FTIR absorption peaks of the six bamboo samples, indicating the presence and changes in chemical groups post-treatment. **OH and CH Groups**: The absorption bands between 3400 and 3500 cm^{-1} (OH stretching) and around 2900 cm^{-1} (CH stretching) were observed across all samples, indicating the preservation of these functional groups. This consistency aligns with the findings of Johar et al. (2012), who noted the importance of maintaining these groups for the structural integrity of cellulose fibers.

Cellulose Peaks: The characteristic absorption peaks of cellulose at 3333-3346 cm^{-1}, 1602-1639 cm^{-1}, and 2887-2894 cm^{-1} showed significant reinforcement in the NCHP-treated samples compared to untreated bamboo powder. This indicates a higher cellulose content post-treatment, corroborating the chemical composition results in Table 1. Li et al. (2021) similarly reported that chemical treatments could enhance the cellulose fraction while reducing lignin and hemicellulose.

Comparison with Untreated Bamboo: The untreated bamboo sample showed weaker cellulose peaks, indicating a higher presence of lignin and hemicellulose. In contrast, the treated samples exhibited stronger cellulose peaks, reflecting the effective removal of non-cellulosic components. This finding is consistent with the work of Zhang et al. (2021), who demonstrated that optimized chemical treatments could selectively remove lignin and hemicellulose, enriching the cellulose content.

Implications: The FTIR analysis in Table 2 highlights the effectiveness of the NCHP treatment in selectively preserving and reinforcing cellulose while removing lignin and hemicellulose. This reinforces the findings from Table 1, providing a comprehensive understanding of the chemical transformations occurring during the treatment. The enhanced cellulose content and the preservation of functional groups are crucial for the fibers' mechanical strength and thermal stability, making them ideal for high-performance applications.

4. Conclusion

Lignocellulosic fibers, known for their robust mechanical characteristics and environmental sustainability, represent a promising avenue for reinforcing materials. With attributes such as high modulus, low thermal expansion, affordability, and a substantial surface area, these fibers offer versatile applications, making them attractive alternatives to traditional synthetic counterparts. The potential to replace conventional fibers further enhances their appeal as sustainable options. This study focused on the extraction of lignocellulosic fibers, emphasizing the importance of pretreatment using nitric acid and hydrogen peroxide. The resulting fibers were subjected to thorough analysis, particularly through Fourier Transform Infrared (FTIR) spectroscopy, to discern their chemical properties. The primary objective was to determine the yield of lignocellulosic fibers under varying pretreatment conditions, including time, temperature, and concentration. The investigation revealed the direct influence of these parameters on fiber production, underscoring the necessity for their precise optimization to achieve efficient extraction. FTIR analysis provided valuable insights into the chemical characteristics of cellulose nanofibers, a pivotal component of biomass isolated through Nitric Acid and Hydrogen Peroxide (NCHP) direct pretreatment. These findings affirm the efficacy of the pretreatment process in producing lignocellulosic fibers with desirable properties. In summary, this study underscores the significance of meticulous control and optimization of parameters in lignocellulosic fiber extraction. The understanding of how time, temperature, and concentration impact fiber yield facilitates the development of more sustainable production methods. By enhancing the mechanical strength of product matrices through the incorporation of environmentally friendly fibers, industries can reduce reliance on synthetic materials, contributing to a more eco-friendly future. The potential of lignocellulosic fibers to replace synthetic counterparts in various applications presents exciting opportunities for sustainable development across industries. As research in this field continues to advance, the utilization of lignocellulosic fibers is poised to grow, fostering greener and more sustainable approaches to material design and manufacturing. This study establishes a foundation for future advancements in lignocellulosic fiber extraction and utilization, contributing to a more sustainable and environmentally conscious future.

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Declaration of Conflicting Interests

All authors declare that they have no conflicts of interest.

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