Available online at
https://journal.uitm.edu.my/ojs/index.php/MJCET

e-ISSN: 2682-8588

Malaysian Journal of Chemical Engineering & Technology 7(2) 2024, 187–194

Malaysian Journal of Chemical Engineering and Technology

Optimisation of chemical treatment parameters for enhanced yield of cellulose nanofibres from Semantan Bamboo (*Gigantochloa scortechinii*)

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ARTICLE INFO

Article history:
Received 8 January 2024
Revised 7 October 2024
Accepted 7 October 2024
Online first
Published 31 October 2024

Keywords:
Semantan bamboo
Cellulose nanofibre
Chemical treatment
Yield percentage

DOI:
10.24191/mjcet.v7i2.575

ABSTRACT

Semantan bamboo (*Gigantochloa scortechinii*) is one of available bamboo in Malaysia and an ideal source for cellulose based nanofibres. However, research on this bamboo has been moderately studied. Therefore, this study conducted a direct and simple chemical treatment using nitric acid and hydrogen peroxide which motivated by previous studies to extract cellulose nanofibre from Semantan bamboo. The Semantan bamboo was used in powder form with particle size in the range of 250 µm to 425 µm. The parameters of the chemical treatment which are temperature, duration of treatment and concentration of nitric acid and hydrogen peroxide were varied. The yield percentage of extracted Semantan bamboo cellulose nanofibres was determined and its functional groups was characterised using Fourier-transform infrared (FTIR) spectroscopy. The findings found the optimum that 76% of extracted Semantan bamboo cellulose nanofibres was obtained at treatment condition of 50 °C, 48 hours and concentration of HNO_3 and H_2O_2 at 3.2 mol/L and 60 mmol/g, respectively.

1. INTRODUCTION

Bamboo, a versatile and sustainable material, has gained significant attention as an alternative to conventional construction materials due to its excellent mechanical properties and widespread availability in tropical regions. Among various bamboo species, Semantan bamboo (*Gigantochloa scortechinii*), native to Malaysia, stands out due to its advantageous properties, including high tensile strength, durability, and

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https://doi.org/10.24191/mjcet.v7i2.575

rapid growth rate, making it ideal for diverse industrial applications, particularly in fibre extraction for composites (Goh et al., 2020). Despite its potential, research on the full mechanical benefits of Semantan bamboo fibres, especially for nanocellulose extraction, remains limited (Wang et al., 2020).

The extraction of cellulose fibres from bamboo has been the focus of several studies, with various methods employed, including mechanical, chemical, and enzymatic processes (Johar et al., 2012; Abdul-Khalil et al., 2012). However, many of these approaches face challenges such as incomplete removal of non-cellulosic materials like lignin and hemicellulose, which affects the purity and performance of the extracted fibres (Li et al., 2015; Rosa et al., 2010). These limitations underscore the need for more efficient extraction methods to yield high-quality cellulose fibres while maintaining the structural integrity of the bamboo fibres (Peng et al., 2012).

This study adopts a chemical treatment process using a mixture of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2) to enhance the extraction efficiency of cellulose nanofibres from Semantan bamboo. This method, inspired by previous research, has shown promise in achieving higher fibre purity and yield by effectively removing lignin and hemicellulose (Wang et al., 2020; Johar et al., 2012). The treatment parameters such as temperature, duration, and chemical concentrations are optimised to further improve the extraction process and maximise the yield of cellulose nanofibres (Li et al., 2015).

The objective of this study is to investigate the effects of varying chemical treatment parameters specifically, temperature, duration, and concentrations of nitric acid and hydrogen peroxide on the yield of cellulose fibres extracted from Semantan bamboo. By optimizing these parameters, this research aims to provide valuable insights into improving fibre extraction techniques, which could pave the way for enhanced utilisation of Semantan bamboo fibres in composite materials and other high-performance applications (Peng et al., 2012; Rosa et al., 2010).

2. METHODOLOGY

2.1 Materials

Semantan bamboo fibres was purchased from a local supplier. The Semantan bamboo fibres were cut into shorter lengths and dried in the vacuum oven at temperature of 100 °C as shown in Fig 1(a). The initial and final weight of Semantan bamboo fibres were measured until the moisture content was constant and less than 10%. Then, the dried Semantan bamboo fibres were crushed using grinding machine and sieved to obtain bamboo fibres in powder form with a size of 40–60 mesh (particle size between range 250 μm to 425 μm) as shown in Fig 1(b). Finally, the Semantan bamboo nanofibres was stored in a glass desiccator prior to be used in the experiment. Nitric acid (HNO_3) with 60% purity and hydrogen peroxide (H_2O_2) with 30% purity were manufactured by Merck, Germany.

2.2 Chemical treatment

Five gram (5 g) of Semantan bamboo powder was mixed into a flask containing 3.2 mol/L of HNO_3 and 60 mmol/g of H_2O_2. The solution was stirred using magnetic stirring for 48 hours at 50 ℃ on a hot plate at a speed of 1000 rpm (Wang et al., 2020). The treatment was conducted in a fume hood. The mixture was then washed with distilled water until the filtrate was neutral and filtered using a vacuum pump to get the Semantan bamboo cellulose nanofibres. The filtered Semantan bamboo cellulose nanofibres were dried in the oven at 50 ℃, overnight. Then the mass of dried Semantan bamboo cellulose nanofibres was weighted and recorded. The treatment was repeated at varied parameters and namely as Treatment No. 1 until Treatment No. 6 as reported in Table 1.

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(a)

(b)

Fig. 1. (a) Semantan bamboo fibres (b) Semantan bamboo nanofibres in powder form

Source: Author's own collection

2.3 Determination of Semantan bamboo cellulose nanofibres yield percentage

The yield percentage of the extracted Semantan bamboo cellulose nanofibres based on biomass was calculated using Eq. (1) (Rasheed et al., 2020).

$$\text{Yield (\%)}=\frac{\text{Weight after treatment (gram)}}{\text{Weight before treatment (gram)}} \times 100 \tag{1}$$

The calculation provides a quantitative measure of the efficiency of the chemical treatment process, offering insight into how varying treatment parameters influence fibre extraction. A higher yield percentage indicates a more effective removal of lignin and hemicellulose, which is critical for achieving high-quality cellulose nanofibres suitable for industrial applications (Wang et al., 2020).

2.4 Characterisation of functional group

The functional groups of samples and its changes were determined using Fourier-transform infrared (FTIR) spectroscopy (Spectrum 100 FTIR, Perkin Elmer, United States of America). FTIR spectroscopy was performed on Semantan bamboo nanofibres before and after treatments. The samples were scanned over a wavenumber range from 4000 to 515 cm^{-1} and the FTIR spectra was recorded.

3. RESULTS AND DISCUSSION

This study discussed on the obtained yield percentage of extracted Semantan bamboo cellulose nanofibres at different chemical treatment conditions. Four parameters of chemical treatment were varied which are temperature, duration of treatment and concentration of nitric acid and hydrogen peroxide. Thus, the functional groups of extracted Semantan bamboo cellulose nanofibres were characterised using FTIR.

https://doi.org/10.24191/mjcet.v7i2.575

3.1 Yield percentage of extracted Semantan bamboo cellulose nanofibres

The yield percentage of extracted Semantan bamboo cellulose nanofibres was determined using Eq. (1). Meanwhile, Table 1 shows the calculated yield percentage. The investigation of yield percentage of extracted Semantan bamboo cellulose nanofibres provides valuable insights into the efficiency of the chemical treatment. The summarised results in Table 1, shows variations in yield percentage at varied chemical treatment parameters. For Treatment No. 4, 56.6% of extracted cellulose nanofibres was obtained at treatment condition of 50 °C, 48 hours and concentration of HNO_3 and H_2O_2 at 3.2 mol/L and 60 mmol/g, respectively. However, when increased the concentration of HNO_3 to 9.6 mol/L, the yield percentage was decreased to 38% (Treatment No. 1). Most probably, the crystalline cellulose region was unstable and degraded under high acidic condition. On the other hand, at higher concentration of H_2O_2 at 90 mmol/g the yield percentage was improved to 76% which is the highest yield obtained (Treatment No. 2).

Table 1. Yield percentage of extracted Semantan bamboo cellulose nanofibres

Treatment No	Time (hr)	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

Source: Authors' own data

Meanwhile, by prolonged the duration of treatment from 48 hours to 72 hours slightly increased the yield percentage to 67% (Treatment No. 3). Similar observation found at higher temperature of 65 °C (Treatment No. 5) where the yield percentage was slightly improved to 68%. However, at lower temperature of 35 °C (Treatment No. 6) resulted the lowest yield percentage which is 55.2%. The result of varied yield percentage shows that the varied parameters (concentration of HNO_3 and H_2O_2, the duration of treatment and temperature) promotes lignin removal. However, further investigation on the percentage retained of cellulose, hemicellulose and lignin contents after the chemical treatment is recommended to be determined according to the technique described by the Technical Association of the Pulp and Paper Industry (TAPPI) (Tanpichai et al., 2019). Fig. 2 shows the sample of extracted Semantan bamboo cellulose nanofibres that was obtained from the chemical treatment.

https://doi.org/10.24191/mjcet.v7i2.575

Fig. 2. Sample of extracted Semantan bamboo cellulose nanofibres of treatment no. 1 to 6

Source: Author's own collection

3.2 Functional groups of extracted Semantan bamboo cellulose nanofibres

FTIR analysis was conducted to characterise the functional groups of the extracted Semantan bamboo cellulose nanofibres. Thus, the changes of functional groups after the treatment were investigated by comparing the FTIR spectrums as shown in Fig. 3.

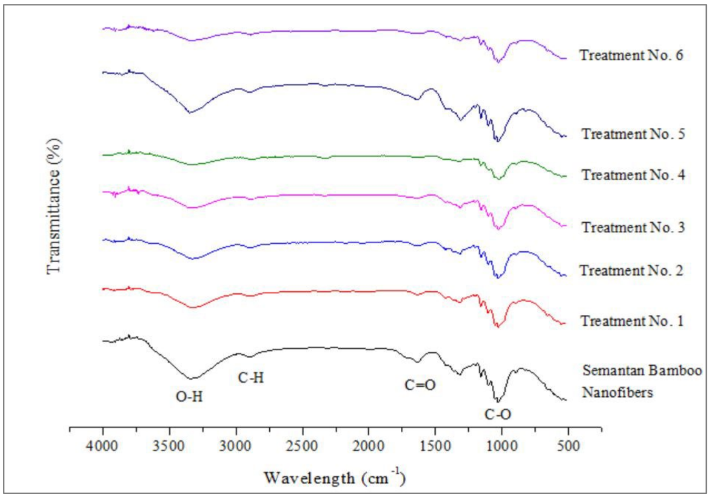

Fig. 3. FTIR spectrum of Semantan bamboo nanofibres and extracted Semantan bamboo cellulose nanofibres from Treatment No. 1 to 6, respectively

Source: Author's own data

https://doi.org/10.24191/mjcet.v7i2.575

The wide absorption bands between 3250–3350 cm^{-1} were corresponded to O–H stretching due to hydrogen bonded hydroxyl (OH) groups of cellulose and absorbed water in the sample (Khan et al., 2024). Besides, the C–H functional group of cellulose was observed at the peaks of 2850–2900 cm^{-1} region (Xia et al., 2024). Furthermore, the C–O group of the cellulose was indicated at the strong absorption peaks of 1025–1035 cm^{-1} region (Asmare et al., 2024). These O–H, C–H and C–O functional groups of cellulose were presented at all spectrums. This finding shows that the cellulose region was not affected by the chemical treatment. Meanwhile, the peak of 1620 cm^{-1} represents the C=O stretching of lignin and hemicellulose (Zhu et al., 2024; Adil et al., 2024). The peak intensity was relatively lower for Treatment No. 1 to Treatment No.6 compared to Semantan bamboo nanofibres which suggests the lignin and a small part of hemicellulose contents was reduced and dissolved during the treatment. Similar observation was found for peak intensity of 1260 cm^{-1} due to removal of lignin content during the treatment (Wang et al., 2023).

4. CONCLUSION

This present study found that cellulose nanofibres was successful extracted from Semantan bamboo at optimum yield of 76% via one bath chemical treatment at mild condition of 3.2 mol/L aqueous HNO_3 and 90.0 mmol/g H_2O_2 at 50 °C for 48 hours. The effect of the concentration of H_2O_2 is more significant in extracting the cellulose nanofibres compared to the temperature and duration of the treatment and concentration of HNO_3. The removal of hemicellulose and lignin in the Semantan bamboo nanofibres during the chemical treatment was supported by the FTIR result.

ACKNOWLEDGEMENTS/FUNDING

The authors would like to thankful to the Universiti Teknologi MARA, Malaysia for the financial support given under SRP Research Grant Scheme 100-RMC 5/3/SRP (068/2021).

CONFLICT OF INTEREST STATEMENT

The authors declare that this research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest. There are no conflicting interests with the funders, or any other parties involved in this study.

AUTHORS' CONTRIBUTIONS

Umar Hazim Bin Muhd Fauzi: Conceptualisation, methodology design, lab work execution, direct chemical treatment on bamboo fibres, formal analysis, and drafting of the original manuscript; **Nur Ilya Farhana Bt Md Noh**: Project administration, overall conceptualisation, and data analysis; **Suffiyana Binti Akhbar**: Data curation, formal analysis, and contributions to manuscript revision; **Zakiah Bte Ahmad**: Validation of results, supervision of lab procedures, and critical review and editing of the manuscript; **Rahida Wati Binti Sharudin**: Data analysis, visualisation, and preparation of graphical representations; **Sajith Thottathil AbdulRahman**: Supervision, verification of experimental procedures, and final validation; **Bijesh Paul**: Manuscript review, editing, and validation of data interpretation.

https://doi.org/10.24191/mjcet.v7i2.575

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