Optimizing the Isolation of Microfibrillated Bamboo in High Pressure Enzymatic Hydrolysis

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Bleached bamboo fiber was treated with a high pressure enzymatic hydrolysis (HPEH) process in order to produce microfibrillated bamboo fiber (MBF). Mixture design of experiments was utilized to determine the optimal constituents of fiber, enzymes, and water for the HPEH process on the isolation yield of the MBF. Results showed the optimal combination for the maximal yield isolation of the MBF was 1 g fiber, 1 g enzyme, and 1 L water at 90 MPa and 70 °C. The influence of the reaction time of the HPEH process (6 to 48 h) was also evaluated in this study. Morphological and thermal property analyses of untreated and treated bamboo fibers revealed that the HPEH process was effective for removing non-cellulosic components from the fibers. Thus, the HPEH process is an effective method for the isolation of the MBF, with the benefits of elevated crystallinity and thermal stability.

Keywords: Bamboo fiber; High pressure; High pressure subcritical enzymatic hydrolysis; Morphological characterization; Thermal properties

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INTRODUCTION

Bamboo is recognized as a superior source of cellulose fiber because of its rapid growth rate, high cellulose content, and excellent mechanical properties (Abdul Khalil *et al*. 2012). There are more than 50 bamboo species available in Malaysia; among these, almost 14 are commercially utilized (Wong 1989). The *Gigantochloa* genus is one of the most utilized bamboo species in Malaysia because of its uniform size, thick culm wall, and ease of cultivation (Mustafa *et al*. 2011).

The utilization of bamboo fibers in composite science and technology have attracted interest because of properties such as biodegradability and clean emissions (Abdul Khalil *et al*. 2007; Alwani *et al*. 2014). The application of bamboo fiber for use in composites may improve certain properties of the polymer composite matrix (*i.e*., the fracture toughness and impact behavior); however, the flexural strength of the composites were found to be hardly affected (Shih 2007; Abdul Khalil *et al*. 2010, 2013; Jawaid *et al*. 2014). Numerous researchers have investigated how to improve the mechanical and barrier properties of bamboo fiber-reinforced composites, including pre-treatment of the fiber by means of physical, chemical, or enzymatic catalysts (Abdul Khalil *et al*. 2013; Alwani *et al*. 2014; Dungani *et al*. 2014). In the author's previous study, subcritical enzymatic hydrolysis at high pressure was found to be an effective and promising treatment for bamboo fiber in order to remove non-cellulosic materials from the fiber. In such treatments

the bamboo fiber was treated at a fiber to enzyme ratio of 2:1 in 500 mL of water, with a pressure of 90 MPa and a temperature of 70 °C (Abdul Khalil *et al*. 2014). However, further studies on the development of a high pressure subcritical enzymatic hydrolysis (HPEH) process are needed in order to scale-up the technology to the industrial level.

In an enzymatic hydrolysis process, the mixture of the fiber, enzymes, and water initiate the isolation of microfibrillated bamboo fiber (MBF). The yield of the isolated MFB and its quality potentially depend on the relative proportions of fiber, enzymes, and water. However, there has been a lack of studies to determine the optimal proportions of these components. Therefore, the present study was conducted to determine the correct formulation of fiber, enzymes, and water for the isolation yield of the MBF using a statistical technique, namely the mixture design of experiment. Moreover, the influence of the reaction time for the HPEH process was also evaluated.

EXPERIMENTAL

Materials

Dried bamboo chips (*Gigantochloa scortechinii*) (1.5 cm^2), with an overall moisture content of approximately 5%, were obtained from the Forest Research Institute of Malaysia (FRIM), in Kepong, Selangor, Malaysia. Pulping and bleaching of the raw bamboo fiber was conducted as described by Abdul Khalil *et al*. (2014). In detail, the pulping of raw bamboo was performed in an "Ibsutek Zat 92" (RB Supply Enterprise, Penang, Malaysia) 20-L stainless steel rotary digester with 25% NaOH and 0.1% anthraquinone (AQ) as cooking liquor. The cooking temperature and time were 170 °C and 3 h, respectively, and the ratio of cooking liquor to bamboo fiber was 7:1. After pulping, the bamboo fibers were washed with distilled water to eliminate chemicals (NaOH and/or AQ), acquired during the cooking process.

The bleaching of the bamboo pulps was performed accordingly by using H_2O_2 and NaOH. The pulps were treated with 3% H_2O_2 (35% v/v), 3% NaOH (0.33 M), and 0.5% $MgSO_4$ (0.039 M) at 80 °C for 2 h. Upon completion of the bleaching process, the samples were rinsed with water. Subsequently, oven-drying was done for 24 h at 60 °C. Subsequently, the bleached bamboo fiber was stored at 4 °C for further studies. The enzyme, "Marugoto C" (cellulase, vegetable origin), was obtained from the Supercritical Technology Research Corporation (Nishi-ku, Hiroshima, Japan). All of the chemicals were analytical grade and used as received from Bumificient Sdn. Bhd., Lumpur, Malaysia, without further purification.

High Pressure Subcritical Enzymatic Hydrolysis

The experiments were conducted following a similar procedure as described in Abdul Khalil *et al*. (2014). One gram of bamboo fiber, 1 g enzyme, and 1 L water were taken into a high density polyethylene bag. The details of the enzyme are presented in Table 1. Subsequently, the bag was sealed and placed into a supercritical water fermenter vessel (Toyokoatsu Co., Ltd, Hiroshima, Japan). After closing the lid of the vessel, the temperature and pressure of the vessel were to set to 70 °C and 90 MPa, respectively. When the temperature reached 70 °C, water was introduced to the vessel in order to raise the pressure to 90 MPa. The treatment was conducted for time periods ranging from 6 h to 48 h. After the treatment, the treated bamboo fiber was filtered and washed with water.

Subsequently, the bamboo fiber was oven-dried and kept in a desiccator for further analysis. The percentage yield of the MBF was determined from Eq. 1, as follows:

$$Y(\%) = \frac{Wt}{W_0} \times 100 \quad (1)$$

where W_0 is the weight of untreated bamboo fiber and W_t is treated bamboo fiber at time t.

Table 1. Details of Enzyme Used for Fiber Resolution in HPEH (source: Supercritical Technology Research Corporation, Ltd., Hiroshima, Japan)

Product's name	Enzyme "Marugoto D"
Main Component	Cellulase
Purpose	Break and rearrange fiber matrix
Objects	Fodders, vegetables, fruits, etc.
pH	4.0 to 5.0
Temperature	50 to 70 °C

Mixture Design of Experiments

A mixture design was imposed to determine the optimum combination of bamboo fiber, enzyme, and water for the highest isolation yield of MBF. The level of the variable was selected, as shown in Table 2. The quadratic model used for selecting the mixture design was as follows (Eq. 2),

$$Y = b_1A + b_2B + b_3C + b_{12}AB + b_{13}AC + b_{23}BC \quad (2)$$

where Y is the predicted yield; b_1, b_2, and b_3 are the constant coefficient of the linear terms; and b_{12}, b_{13}, and b_{23} are the constant coefficients of the interaction terms. Minitab statistical software (version 16.1, USA) was used to analyze the data and fit the model equation. The fit of the model equation was checked using the adjusted coefficient of determination.

Table 2. Level of the Variables in the Mixture Design

Sample	Low level	Upper level
Fiber (g)	1.0	2.0
Enzyme (g)	1.0	2.0
Water (L)	0.5	1.0

Characterization

The characterizations of the isolated MBF treated using the HPEH technology were conducted following methods similar to Abdul Khalil *et al*. (2014). The morphological analysis of the treated and untreated fibers was conducted by field emission scanning electron microscopy (FE-SEM) (EVO MA10, Germany). The analysis of the functional groups of the fiber was determined using Fourier transform infrared spectroscopy (FT-IR) (Thermo Scientific, USA), the crystallinity of the fiber was determined using X-ray diffraction (XRD), and thermal properties of the fiber were determined using a thermogravimetric analyzer (TGA) (SDTA 851; Mettler Toledo, Switzerland).

Determination of the Chemical Compositions

The contents of chemical components of bamboo fibers, such as extractives, holocellulose, α-cellulose, lignin, and ash, were determined following the standards outlined by TAPPI test methods, as described by Wahab *et al*. (2013). The extractives content in the bamboo fiber was determined according to the T204 cm-88 and T264 cm-88 methods. Holocellulose contents in treated and untreated bamboo fiber were determined according to the method provided by Wise *et al*. (1946). The α-cellulose contents were determined following the T203 cm-74 method. The lignin content in treated and untreated bamboo fiber was investigated by the T222 cm-88 method. The ash content in treated and untreated bamboo fiber was determined by following the T211 cm-93 method.

RESULTS AND DISCUSSION

Isolation of Microfibrillated Bamboo Fiber

A mixture design was examined using Minitab 16.1 software in order to define an optimal formulation of fiber, enzyme, and water for the maximal isolation yield of the MBF using the HPEH treatment process. The design consisted of 10 experimental runs. The mixture design is an efficient tool to optimize the enzyme and water content for the effective bamboo fiber hydrolysis process and to determine the interaction effect of each component in the hydrolysis process (Cornell 2002). For the experimental design, the response variable was considered to be dependent on the propositions of the formulation components (Suwannarangsee *et al*. 2012). The amount of each component was generated using the Minitab software, as presented in Table 3.

Table 3. Mixture Design of Experiments for the Isolation Yield of Cellulosic Bamboo Fiber

Run	Fiber (g)	Enzyme (g)	Water (L)	Yield (%)	
				Experimental	Calculated
1	1.00	1.50	0.50	87.610	87.493
2	1.25	1.25	0.50	91.987	91.932
3	1.33	1.08	0.59	85.496	85.799
4	1.50	1.00	0.50	88.752	88.562
5	1.08	1.08	0.84	87.659	87.827
6	1.00	1.25	0.75	91.678	91.908
7	1.25	1.00	0.75	89.379	89.774
8	1.08	1.33	0.59	88.065	88.302
9	1.00	1.00	1.00	91.091	91.142
10	1.17	1.17	0.66	89.876	88.853

Table 3 displays the percentage yield of isolated MBF at 90 MPa and 70 °C, for each experimental run. It was observed that each experimental value of percentage isolated yield was close to the calculated values, which revealed a good fit to the model equation. Table 4 presents the summery of analysis of the variance (ANOVA) of the mixture design analysis. Estimation of the regression coefficient for the isolation yield of MBF, as well as linear, quadratic, and interaction terms of the model, were calculated using the least squares method. The degree of significance for each factor was determined by considering the *P*-value at the confidence level α= 0.05. The linear, quadratic, and interaction terms between

the enzyme and water had a significant effect on the isolation yield. The adjusted determination coefficient (R^2 adj) value of 0.91 was close to 1, indicating a high degree of correlation between the observed and predicted values.

Table 4. Analysis of Variance of Mixture Design Analysis

Source	DF[1]	Sum of squares	Mean Squares	F-value	p-value
Regression	5	36.5801	7.31602	19.67	0.006
Linear	2	26.8695	3.94115	10.60	0.025
Quadratic	3	9.7106	3.23686	8.70	0.032
Fiber*Enzyme	1	-1.1208	1.07218	2.88	0.165
Fiber*Water	1	1.0939	1.12932	3.04	0.156
Enzyme*Water	1	7.4958	7.49584	20.16	0.011
Residual Error	4	1.4874	0.37185		
Total	9	38.0675			

[1]DF: Degrees of freedom; R^2 = 0.96; R^2 (adj) = 0.91

Figure 1 shows the contour and surface plot for the isolation of MBF using the HPEH process. As shown in Fig. 1, the yield increased with increasing fiber and enzyme content of the mixture. However, the yield decreased when water was more than 1 L of the mixture. To determine the optimum combination of fiber, enzyme, and water in the mixture, Minitab software was used to predict the optimal combination point for maximizing the isolation yield of the MBF. The combination with the highest efficiency was found to be 1 g of fiber, 1 g of enzyme, and 1 L water; this resulted in the highest isolated yield of 92%.

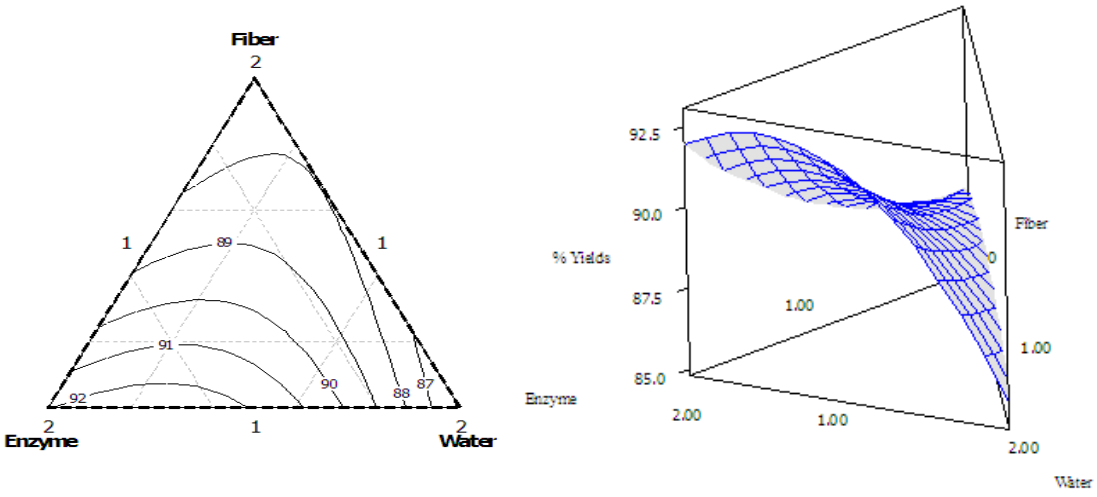

Fig. 1. The triangular contour and surface plot for the isolation of microfibrillated bamboo fiber (MBF) using of the high pressure subcritical enzymatic hydrolysis (HPEH) process

Morphological Analysis

The morphology of the untreated and the HPEH treated bamboo fibers were investigated using SEM analysis, as shown in Fig. 2. The visualization of the untreated bamboo fiber showed an irregular surface, which was probably due to the presence of non-cellulosic materials, such as lignin, hemicelluloses, and waxy materials (Mustafa *et al*.

2011). Conversely, the treated bamboo fiber, at various treatment times, appeared to be uniform and smooth with an average diameter of 2.51 to 4.49 µm. Thus, these results confirm that the HPEH treatment was effective in removing the non-cellulosic components (*i.e.*, lignin, hemicelluloses, and waxy materials) that lead to irregularities on the raw bamboo fiber's surface (Jonobi *et al*. 2011; Hossain *et al*. 2014).

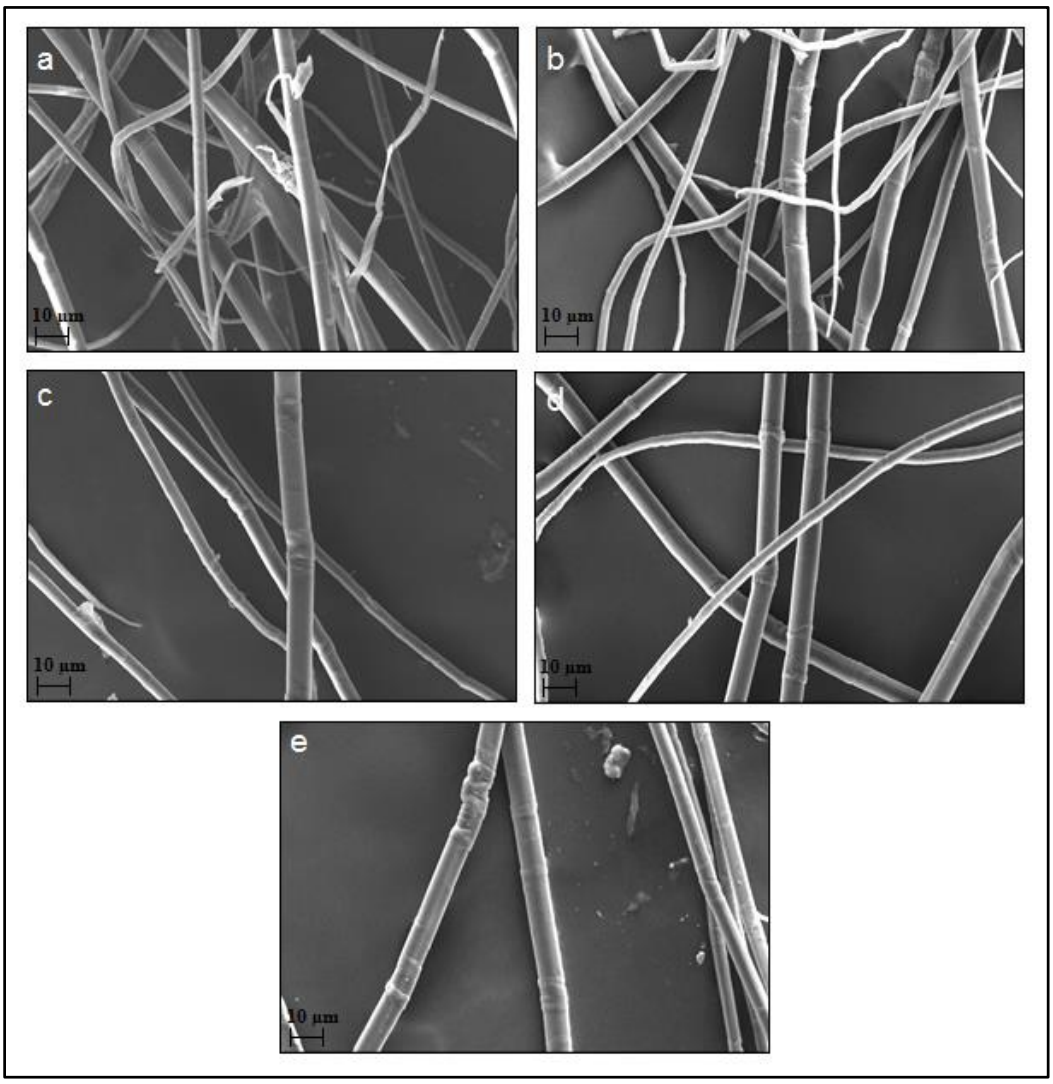

Fig. 2. Scanning electron micrographs of (a) 0 h (untreated), (b) 6 h, (c) 12 h, (d) 24 h, and (e) 48 h high pressure subcritical enzymatic hydrolysis (HPEH) of bamboo fibers. Experimental conditions: 1 g of fiber, 1 g of enzyme, and 1 L of water at 90 MPa and 70 ºC

Influence of Treatment Time

The treatment time may also play a role in the HPEH of bamboo fiber for the isolation of MBF. Therefore, the influence of treatment time for the isolation of MBF was determined, as shown in Fig. 3. It was observed that the percentage isolated yield of MBF increased from 84.87% to 92.30% with increasing treatment time from 6 h to 24 h. However, there was no considerable increase of isolated yield when treatment time was increased beyond 24 h. The increase of the percentage isolated yield of MBF with the hydrolysis time probably, because of providing higher energy and force to fiber suspension

to gradually breakdown the H-bonds, those keep fibers together. The negligible increase of MBF yield with hydrolysis time over 24 h might be due to the saturation of H-bond in the fiber suspension with the higher energy and force generation.

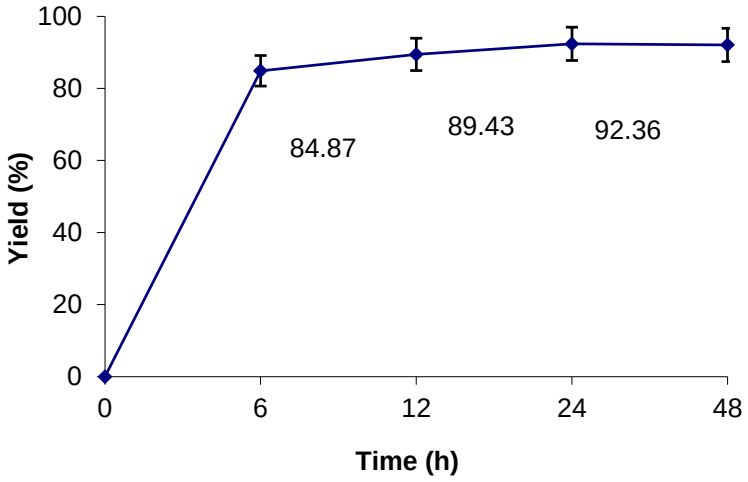

Fig. 3. The influence of treatment time for the isolation of microfibrillated bamboo fiber (MBF) using high pressure subcritical enzymatic hydrolysis (HPEH). Experimental conditions: 1 g of fiber, 1 g of enzyme, and 1 L of water, at 90 MPa and 70 ºC

Fourier Transform Infrared Spectrometry Analysis

Figure 4 shows the FT-IR spectra of the HPEH bamboo fibers after various treatment time periods (6, 12, 24, and 48 h).

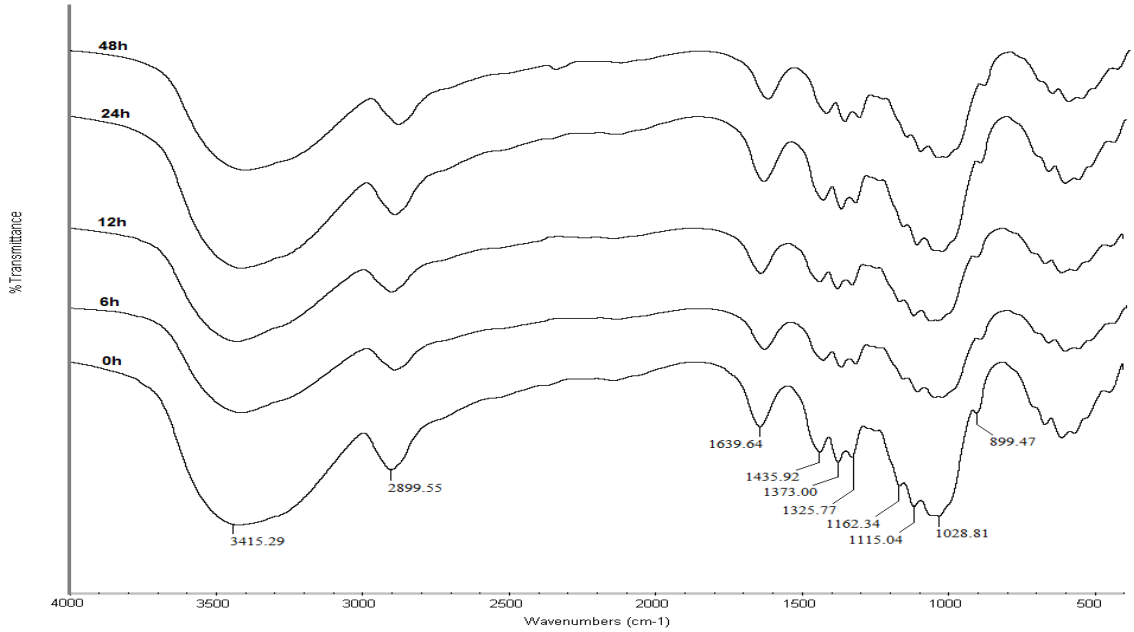

Fig. 4. FT-IR spectra of untreated and high pressure subcritical enzymatic hydrolysis (HPEH) treated bamboo fibers after various treatment time periods of treatment. Experimental conditions: 1 g of fiber, 1 g of enzyme, and 1 L of water, at 90 MPa and 70 ºC

From the spectrum, hydroxyl groups (-OH) were represented by a strong and broad band (3415 cm^{-1}). The band at 2899 cm^{-1} occurred because of the C-H stretching (Abdul Khalil *et al*. 2014). Bands at 1639 cm^{-1}, 1435 cm^{-1}, 1373 cm^{-1}, and 1162 cm^{-1} corresponded to water absorption, bending vibration of the CH_2 groups, C-H groups of the aromatic ring, and the C-O-C linkages in lignin, respectively (Jonoobi *et al*. 2009). Furthermore, the absorption peak at 1028 cm^{-1} was assigned to the stretching of O-H and C-H groups, whereas the vibration peak at 899 cm^{-1} was characteristic of glycoside bond formation (Chan *et al*. 2012). The HPEH treatment caused the reduction of the strong and broad bands of the treated bamboo fiber, especially at the stretching vibration band between 3000 and 3500 cm^{-1}, which revealed the removal of the phenol and alcoholic waxy materials from the fiber.

X-ray Diffraction Analysis

Cellulose contains a partially crystalline and amorphous structure resulting from the placement of the chain, which is held closely by mutual H-bonding in the crystalline region, and the absence of such organized H-bonding in the amorphous region (Abdul Khalil *et al*. 2010a; Jonoobi *et al*. 2011). The native cellulose crystalline structure results from the formation of peaks at 2θ, in the range of 18 °C to 22.5 °C. Figure 5 shows the crystallinity formation of untreated and HPEH treated bamboo fibers. The crystallinity value for the untreated bamboo fiber was 60.89%. Whereas, the crystallinity values of MBF were 67.97%, 68.34%, 73.15%, and 73.21% for the subcritical enzymatic hydrolysis durations of 6, 12, 24, and 48 h, respectively.

The increase in crystallinity values for treated bamboo fiber reveals that the HPEH process is an effective method for removing the non-cellulosic materials from the bamboo fiber (Jonoobi *et al*. 2011).

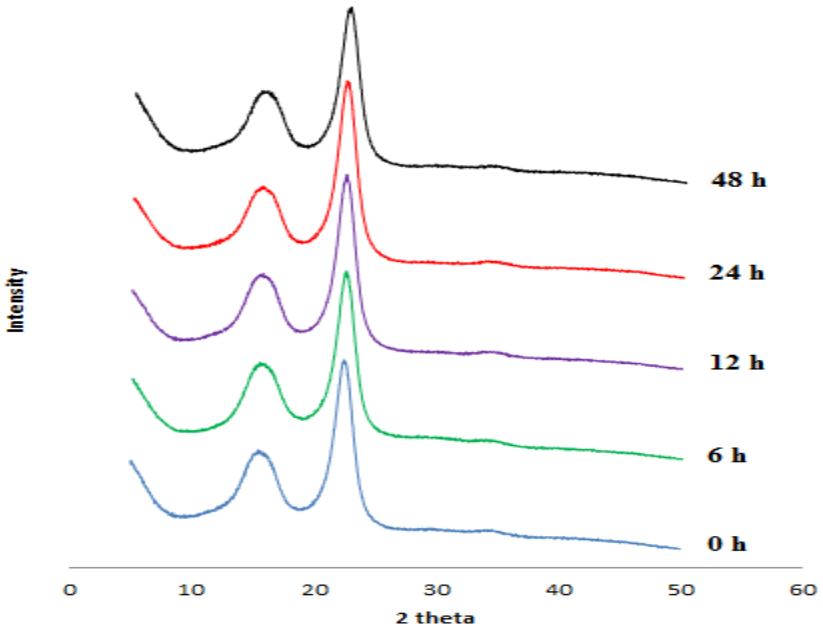

Fig. 5. XRD diffractograms of high pressure subcritical enzymatic hydrolysis (HPEH) treated bamboo fibers. Experimental conditions: 1 g of fiber, 1 g of enzyme, and 1 L of water, at 90 MPa and 70 °C

Thermal Properties Analysis

Thermogravimetric analysis was performed to evaluate the weight loss, the decomposition temperature, and the thermal stability of the untreated and treated bamboo fibers, as presented in Fig. 6. The thermal characteristics of the treated bamboo fibers, including the initial decomposition temperature (T_{onset}), maximum degradation temperature (T_{max}), and char residue at 550 °C, are summarized in Table 5. Results indicate that the HPEH treatment of the cellulosic bamboo fiber possessed an initial decomposition below 150 °C, because of the evaporation of water. It was observed that the T_{onset} temperature increased from 307 to 345 °C when increasing the treatment time from 0 h (untreated) to 48 h. However, the increased of the T_{onset} temperature over the 24 h treatment period was negligible. Similarly, the maximum degradation temperature (T_{max}) for the untreated bamboo fiber was found to be 354 °C. The T_{max} temperature increased with increasing treatment time until the 12 h point (369.35 °C), after which the increase in the T_{max} temperature was negligible. It was observed that the residue present in the untreated bamboo fiber decreased with increasing the treatment time of the HPEH process. The lowest value of the percentage residual was found to be about 6% for over 24 h of treatment.

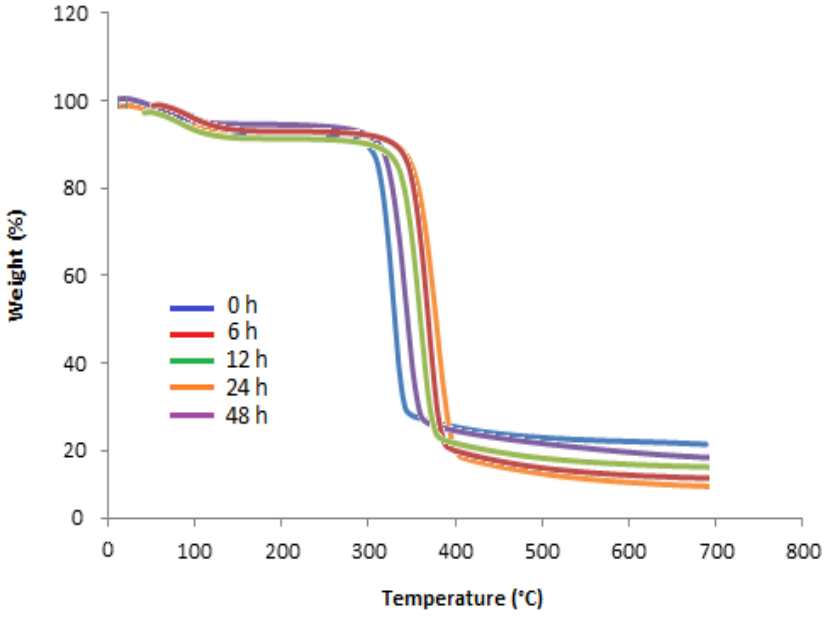

Fig. 6. TGA thermograms of high pressure subcritical enzymatic hydrolysis (HPEH) treated bamboo fibers

Table 5. Thermal Properties of Pressurized Enzymatic Hydrolysed Bamboo Fibers

Run Time (h)	T_{onset} (°C)	T_{max} (°C)	Residue (%)
0	307.09	353.86	22.85
6	342.89	368.07	7.320
12	342.91	369.35	7.060
24	344.53	369.44	6.180
48	344.72	368.47	6.170

Chemical Compositions in Treated and Untreated Bamboo Fiber

The chemical compositions including extractive, holocellulose, α-cellulose, lignin, and ash were determined, as presented in Table 6. It was observed that the percentage of extractive, lignin, and ash contents in bamboo fiber decreased with hydrolysis time, indicating that HPEH process was effectively removing extractive materials, lignin, and ash constituents from the bamboo fiber. On the other hand, holocellulose and α-cellulose content was increased with increasing treatment time. The highest percentage of holocellulose and α-cellulose content were determined to be 92.31% and 96.48%, respectively. Table 6 shows the lowest extractive, lignin, and ash contents, and the highest holocellulose and α-cellulose contents were observed at enzymatic hydrolysis time at 24 h, which is in line with morphological and thermal properties analyses of treated and untreated bamboo fiber.

Table 6. Chemical Compositions of Treated and Untreated Bamboo Fiber using HPEH

Run time (h)	Chemical composition (%)				
	Extractive	Holocellulose	α-cellulose	Lignin	Ash
0	5.33 (±0.32)	77.77 (±0.34)	70.49 (±0.22)	28.63 (±0.18)	2.56(±0.33)
6	5.02 (±0.45)	79.68 (±0.15)	73.63 (±0.33)	27.54 (±0.29)	2.50(±0.11)
12	3.86 (±0.31)	90.85 (±0.47)	86.91 (±0.21)	22.83 (±0.12)	1.71(±0.15)
24	2.78 (±0.11)	92.31 (±0.21)	96.48 (±0.26)	15.52 (±0.23)	0.73(±0.10)
48	2.96 (±0.10)	89.66 (±0.30)	95.54 (±0.42)	19.95 (±0.22)	1.54(±0.25)

(± -standard deviation)

CONCLUSIONS

1. The optimal formulation for maximizing the yield isolation of MBF were 1 g of fiber, 1 g of enzyme, and 1 L of water at 90 MPa and 70 °C.
2. The percentage of isolated yield increased from 84.87% to 92.3% when the treatment time period increased from 6 h to 24 h.
3. Scanning electron microscopy imaging of the treated bamboo fiber at various treatment times resulted in the visualization of uniform and smooth morphological structures, with an average fiber diameter of 2.51 to 4.49 μm.
4. The crystallinity values of MBF were determined to be 67.97%, 68.34%, 73.15%, and 73.21% for the HPEH time periods of 6, 12, 24, and 48 h, respectively.
5. The thermal stability of the bamboo fiber increased with increasing treatment time. The highest T_{oneset} and T_{max} temperature were determined to be 344.72 °C and 368.47 °C, respectively.
6. Based on the results of the present study, it can be concluded that the HPEH process is an effective method for removing the non-cellulosic materials from the bamboo fiber, yielding high crystallinity values and thermal properties.

ACKNOWLEDGMENTS

The authors would like to thank the Ministry of Education for providing the research Grant No. FRGS-203/PTEKIND/6711325.

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Article submitted: March 23, 2015; Peer review completed: June 27, 2015; Revised version received: June 30, 2015; Accepted: July 1, 2015; Published: July 14, 2015.
DOI: 10.15376/biores.10.3.5305-5316