Optimization of Process Conditions for Microwave-assisted Flax Water Retting by Response Surface Methodology and Evaluation of its Fiber Properties

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Microwave-assistance was used to increase the degumming efficiency in flax water retting. Different pre-soaking times, microwave times, and microwave power were investigated in this study. The relationships between degumming rate and process parameters were established *via* response surface methodology (RSM). The optimum process parameters were a pre-soaking time of 25.5 h, a microwave time of 18.5 min, and a microwave power setting of 570 W. Under these optimal conditions, the degumming rate was 83.85% ± 1.13%, which was 1.33 times higher than that of natural hot water retting ($P < 0.05$). Moreover, the tensile properties and color of the resulting fibers showed that they had tensile properties similar to those of the natural hot water retting fibers. However, the color values for the natural hot water retting fibers were higher than those of the fibers treated with microwave-assisted flax water retting.

Keywords: Microwave; Flax; Retting; Optimization; Fiber properties

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INTRODUCTION

The flax (*Linum usitatissimum* L.) plant stem can be processed to produce flax fibers from its phloem, and it is known as the "fiber queen" (Ruan *et al.* 2015). Flax fiber is frequently used in textile, medical, and physical material fields due to its excellent properties, such as moisture retention, antimicrobial qualities, and air permeability (Zhao *et al.* 2016). Fibers attached to the stems with a close matrix contain hemicellulose, lignin, and pectin (Nair *et al.* 2016), and the process of acquiring pure fiber is called retting/degumming. In the retting process, relaxing the fibers from the plant phloem is performed using either mechanical or chemical methods (Nair *et al.* 2016). Various retting methods include enzyme retting, chemical retting, water retting or microbial retting, and mechanical retting, *etc.* (Nair *et al.* 2016). Enzyme retting demands a lot of water and chemicals that can contaminate the environment (Guo and Zhao 2010). Therefore, it is important to find a method that can decrease pollution, reduce cost, and dramatically enhance the fineness and cleanliness of fiber. Microwave-assisted retting has gained increased interest as a degumming method in industrial retting due to its low energy consumption and eco-friendly qualities. Microwave processing of flax could also lead to elasticity improvement, which is of exceptional interest for both composites and geotextiles applications (Li *et al.* 2020). Application of microwave is feasible for flax retting, as it not only can save energy and reduce pollution but also shorten retting time.

Many factors influence the retting efficiency of the microwave-assisted flax water retting process. Response surface parameter (RSM) has been used to investigate the role of singular process parameters and the impact of their interactions when carrying out the responses (Hasni *et al.* 2017). Du *et al.* (2018) attempted to develop a mathematical model to increase the degumming efficiency, and it related the process parameters. This study aimed to explore the microwave-assisted retting process and improve the degumming efficiency of flax. Further, the fiber properties were also examined.

EXPERIMENTAL

Materials

Flax seeds were purchased from the Heilongjiang Academy of Agriculture Sciences (Heilongjiang, China) and grown in fields. After 110 d, the flax stems were harvested and dried in preparation for retting.

Methods

Microwave-assisted water degumming

Flax stems were prepared by cutting them to equal lengths of 80 mm. Non-retted flax stems weighing 3 g each were soaked in separate 100-mL test tubes with flax bundle and tap water at the ratio of 1:20. The retting experiment lasted for 120 h at 30 °C (Zhao *et al.* 2016). The microwave-assisted/natural hot water degumming (retting) (Nair *et al.* 2015) was performed immediately after the pre-soaking for all samples (Zhao *et al.* 2018). The pre-soaked flax stems were subjected to microwave treatment in a microwave generator (Midea M1-L213C; Midea Group, Beijing, China). After degumming, the flax stems were placed in a ventilated place at room temperature until constant weight, and the fibers were obtained by manually peeling and removing impurities.

Single factor experiment

To evaluate the optimal degumming conditions for flax, the pre-soaking time, microwave time, and microwave power were studied using the 'one factor at a time approach' (keeping the rest factor constant) in order to estimate degumming rate in microwave-assisted water degumming system. To assess the optimum pre-soaking time, flax stems were water-soaked for times ranging from 0 h to 24 h. The optimal microwave time for degumming rate was determined ranging from 7 min to 21 min, keeping all other parameters at their optimum level. To evaluate the effect of microwave power on the degumming rate, experiments were conducted at 100, 200, 400, 550, and 700 W, while keeping other parameters at optimum level. The rated power of the household microwave ovens is 700 W. So one can choose different working positions during work, such as 20%, 40%, 60%, 80%, and 100%. Therefore, the actual working power was estimated according to the rated power.

Evaluation of degumming efficiency

The degumming efficiency was measured with the Fried test. Fried test scores reflect the degree of degumming between plant phloem fiber and wood core fiber (Rognes *et al.* 2000). The degree of flax retting was calculated according to the average score (Zhang *et al.* 2000). Because Fried test scores can be subjective, the percentage change in weight loss of flax stem from non-retted to microwave-assisted water-retted flax phloem, *i.e.*, the degumming rate, was also detected.

Experimental design

The system of microwave-assisted water retting involves interactions among several variables; thus, traditional methods were inefficient for optimizing the process (Ruan *et al.* 2015). The central composite design (CCD), which is the standard RSM, was employed for microwave-assisted water retting. Table S1 presents the experimental range of each variable and the levels of the independent variables. To obtain the optimum combination and influence of parameters on the microwave-assisted water retting, 20 experiments were performed in this reaction research, as considered necessary by the CCD (Table S2). The fit of the model was evaluated by means of two diagnostic residuals (Hasni *et al.* 2017). The test of statistical significance was performed on the total error criteria with a confidence level of 95%. The fitted polynomial equation was as follows (Eq. 1),

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i x_i + \sum_{i<j} \sum \beta_{ij} x_i x_j \quad (1)$$

where Y is the response, β_0, β_i, β_{ii}, β_{ij} are constant coefficients, and x_i, x_j are the coded independent variables or factors.

Fiber properties

According to the GB/T 17345-31 (2008) standard, the weight loss, strength, long fiber ratio (Hassan *et al.* 2004), and ratio variation of strength were determined. The color of the flax fibers was evaluated by the CIE 1976 L^*, a^*, b^* color space *via* the method of Saltzman (1981); L^*, a^*, and b^* represent the whiteness/darkness, redness/greenness, and yellowness/blueness of the color of fibers, respectively. A fiber sample of approximately 20 mm in width and 10 mm thick was taken and put on a blank sheet of paper. The 5 to 6 points of each sample were chosen and determined *via* a tristimulus colorimeter, and the average values were recorded (Minolta Co., Ltd., Tokyo, Japan) (Ruan *et al.* 2015). Micrographic observation was performed by microscope (Olympus BX43; Olympus Corporation, Tokyo, Japan).

Statistical analysis

The data in this study were each obtained from three independent experiments, and the mean value ± standard deviation (SD) is presented. The experimental design was performed with the statistical software Design Expert (version 7.0.0, Stat-Ease, Minneapolis, MN, USA). In addition, JMP 9.0.2 (SAS Institute Inc., Cary, NC, USA) software was used to analyze statistical data.

RESULTS AND DISCSUSSION

Effect of One Factor at a Time Experiments for Microwave-assisted Water Retting

Effect of pre-soaking time

Pre-soaking time is a key factor that directly influences the retting efficiency of flax. Figure 1 depicts the pre-soaking time for the Fried test and degumming rate of flax.

The result of the Fried test showed that the degree of degumming improved significantly ($P < 0.05$) as pre-soaking time increased. Figure 1b revealed that the degumming rate increased significantly as the pre-soaking duration increased ($P < 0.05$). The degumming rate increased 10.92% ± 0.23% when the pre-soaking time was increased from 4 h to 12 h. However, changing the pre-soaking time from 12 h to 20 h produced an increase of only 3.03% ± 0.07%, and the degumming rate did not change after 20 h. Similar results on pre-soaking time and flax degumming were found in previous research (Nair *et al.* 2016). However, Nair *et al.* (2013) reported that the maximum degumming efficiency was reached when flax stems were pre-soaked for 24 h, which suggests a longer duration than that identified in this study.

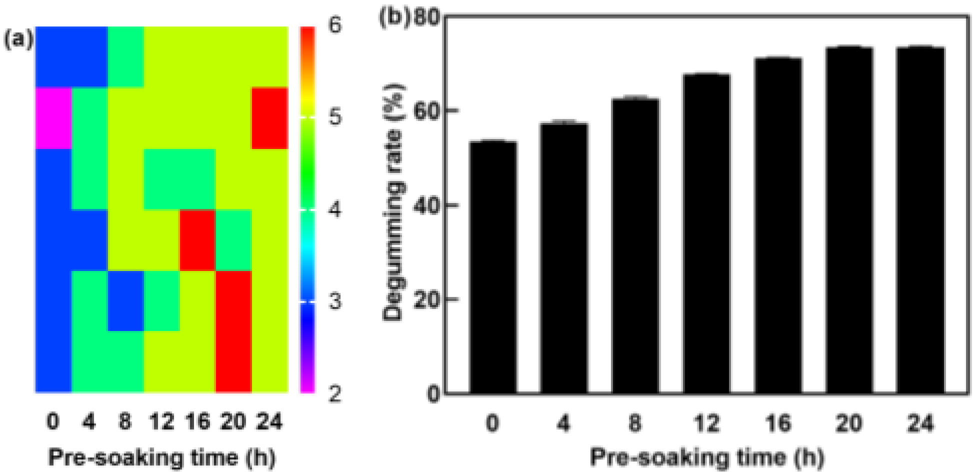

Fig. 1. Degumming efficiency of microwave-assisted water retting with different pre-soaking times: a) Fried test results, b) Degumming rate

Effect of microwave time

Research was also conducted to identify the optimum microwave time, and it was found that as the microwave time increased from 7 min to 15 min, the Fried test scoring increased (Fig. 2a). Over 75% of its scoring on the Fried test was retained after 15 min of microwave.

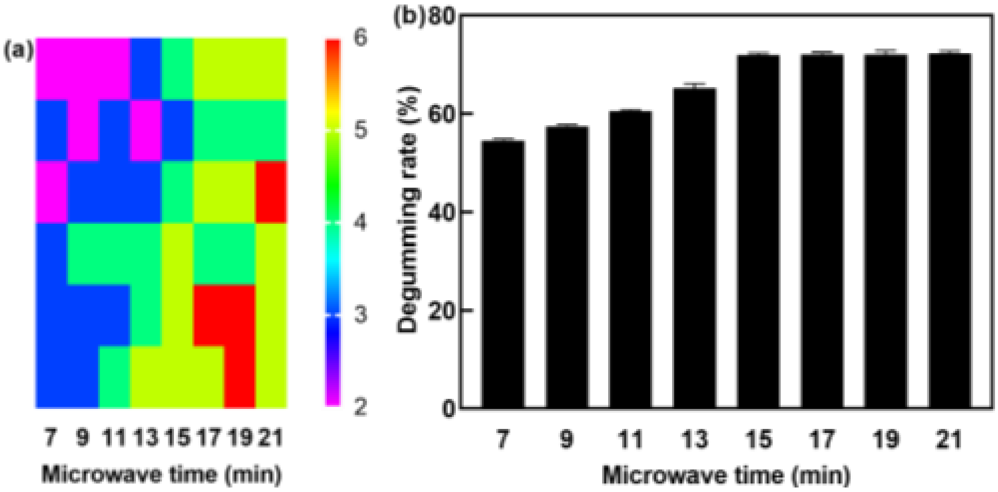

Fig. 2. Degumming efficiency of microwave-assisted water retting with different microwave times: a) Fried test results, b) Degumming rate

Figure 2b shows that the degumming rate significantly ($P < 0.05$) increased as microwave time increased. This was due to the influence of microwave in damaging the strong pectin bonds that adhered to the flax fiber; this influence is called the "non thermal-effect" of microwave processing (De la Hoz *et al.* 2005). However, beyond 15 min, there was marginal additional change, and the change graph shows a plateau. All water was evaporated as microwave time increased and the over-heating of plant stems occurred, which could result in low quality fiber (Nair *et al.* 2016). Therefore, 15 min was selected as the microwave time with maximum degumming rate of 71.15% ± 1.47%. This result was similar to the results of Nair *et al.* (2013).

Effect of microwave power

The experiments were conducted by varying the microwave power. As shown in Fig. 3a, after reaching 550 W of microwave power, the Fried test scores were all above 75%, which was higher ($P < 0.05$) than the scores obtained from 100 W to 400 W. Fried test scores were more stable under high power conditions, and approximately 100% of the scores remained after treatment at 700 W. Figure 3b revealed that the degumming rate significantly increased as microwave power increased ($P < 0.05$). This was because the non-thermal and thermal influence of microwave power facilitated the hydrolysis of pectin (Tsubaki and Azuma 2011). The overall conversion increased to 74.31% ± 2.06% when microwave power increased to 550 W. The percentage increase in the degumming rate was > 70%, which was significantly ($P < 0.05$) greater than the degumming rates achieved with lower microwave power (< 400 W). However, a further increase in microwave power resulted in a decrease of the percentage degumming rate to 73.17% ± 1.24 %.

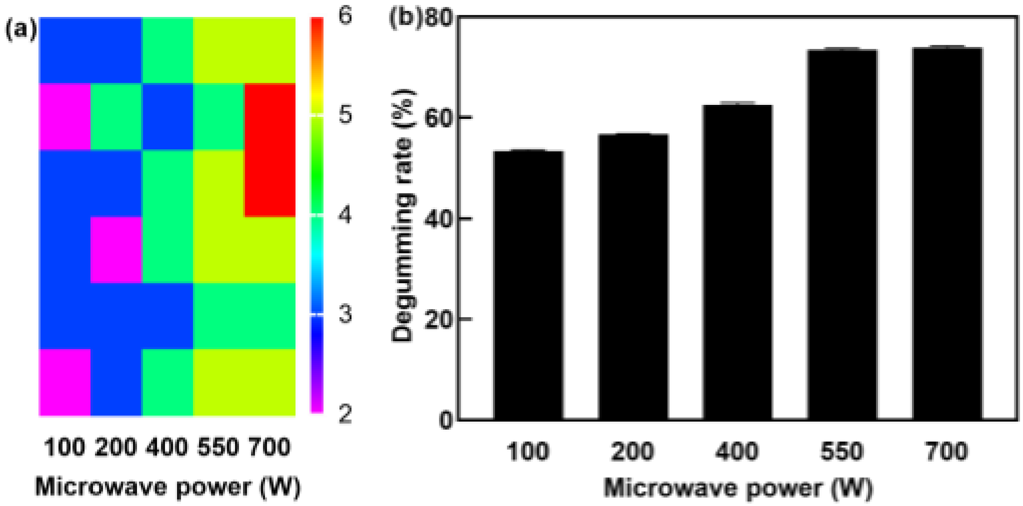

Fig. 3. Degumming efficiency of microwave-assisted water retting with different microwave power levels: a) Fried test scores, b) Degumming rate

Optimization of Retting Process by CCD

Optimization of the microwave-assisted retting process was carried out using 3 factors at three levels, which required a total of twenty runs. Table 2 shows the mean predicted and experimental values according to the quadratic model of pre-soaking time (A), microwave time (B), and microwave power (C) used to predict degumming rate. The analysis of variance (ANOVA) and regression analysis results of the model are shown in

Table 1. Because of the lower P value and higher F value, the results indicated that the model was highly significant at the 95% confidence level. The "Lack of Fit F-value" of 0.1149, implies that the Lack of Fit is not significant relative to the pure error. Non-significant lack of fit is good for the model to fit. (Singh *et al.* 2018). The adjusted coefficient of determination (Adj R^2) and the coefficient of determination (R^2) were 0.8561 and 0.9243, respectively. The R^2 of model was close to unity 1, which indicated that the developed model of the degumming rate was representative of the process (Hasni *et al.* 2017). The value of adequate precision was 8.968, and the adequate precision obtained in this study was greater than 4.0, which indicated that this response had better precision and reliability (Ikrang and Umani 2019; Luo *et al.* 2019). In addition, the model was suitable for experimental relationships between the response and the variables (Anwar *et al.* 2017). The regression equation that describes the effects of the retting process variables on the degumming rate in terms of the actual values of the variable is given in Eq. 2:

$$\text{Degumming rate (\%)} = 77.44 + 8.12 \times A + 5.97 \times B + 8.51 \times C + 6.92 \times AB - 10.77 \times A^2 - 11.19 \times B^2 - 6.38\, C^2 \qquad (2)$$

Table 1. Results of the Regression Analysis of the CCD

Factor	Coefficient Estimate	F-value	P > ｜F｜
Intercept		13.56	0.0002**
A	8.11	17.65	0.0018**
B	5.97	9.54	0.0115*
C	8.51	19.36	0.0013**
A*B	6.92	7.50	0.0209*
A*C	1.26	0.25	0.6295
B*C	-1.41	0.31	0.6891
A^2	-10.77	32.71	0.0002**
B^2	-11.19	35.32	0.0001**
C^2	-6.38	11.48	0.0069**
Lack of Fit		3.18	0.1149
R^2 = 0.9243 Adj R^2 = 0.8561 Adeq precision = 8.968			
P < 0.05 indicate model terms are significant*, P < 0.01 indicate model terms are very significant**			

Response surface plots and contour plots for expansion ratio and porosity were generated as a function of two independent variables, while other independent variables were kept at their centre point. Figure 4 shows the estimated response function and the effects of the independent variables (A, B, and C) on the dependent variables values. The convex surface plots indicated that there was a maximum predicted value of response variables. The results showed that the interactions among pre-soaking time and microwave retting time significantly affected the degumming rate (P = 0.0209 < 0.05) (Fig. 4). The degumming rate reached a highest value when the level of pre-soaking time, microwave time, and microwave power were 0.56, 0.40, and 0.69, respectively. The optimum conditions for degumming rate predicted by the model were found as a pre-soaking time of 25.6 h, a microwave time of 28.45 min, and a microwave power of 569 W. However, considering the actual operating conditions, the pre-soaking time, microwave time, and microwave power were adjusted to 25.5 h, 18.5 min, and 570 W, respectively. Nair *et al.* (2013) reported that flax has a better retting efficiency when soaked for 24 h and exposed to microwave retting for 20 min. Based on these results, a maximum degumming rate of 83.85% ± 1.13% was predicted by the software, which was 1.33 times higher than that predicted for natural hot water retting (62.94% ± 2.53%) ($P < 0.05$).

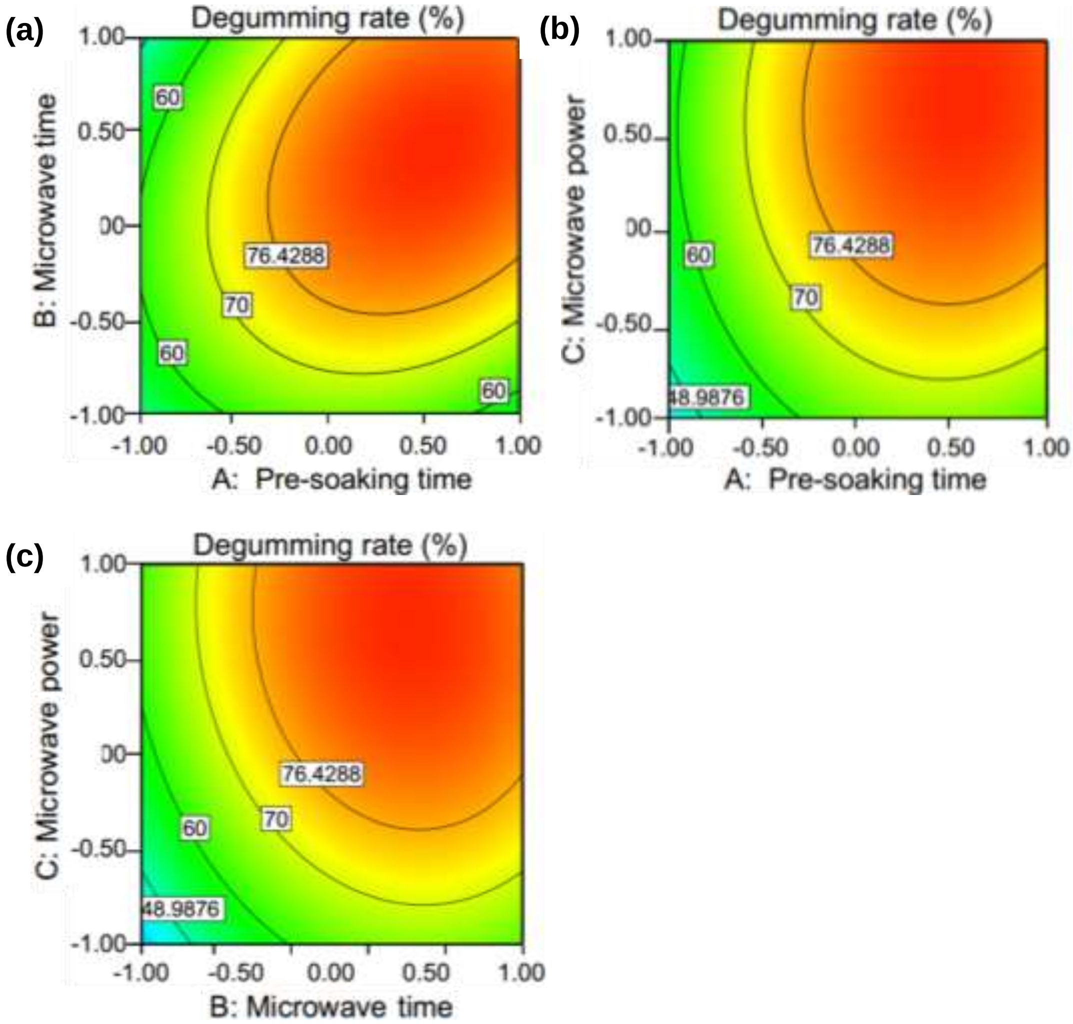

Fig. 4. The contour plots that show the effect of pre-soaking time and microwave time (a); pre-soaking time and microwave power (b); microwave time and microwave power (c) on the microwave-assisted water retting of flax

Bazaria and Kumar (2016) researched microwave-assisted water retting at the optimum retting conditions to test the adequacy of the model equation for predicting the response values. The results showed that the degumming rate of experimental values was 82.41% ± 1.07%, which was close to the predicted values (83.85% ± 1.13%), but higher than those reported by Ruan *et al.* (2015). Therefore, the model was suitable for assessing the behaviour of the retting process conditions of microwave-assisted water retting (Ikrang and Umani 2019).

Properties of Fiber

Weight loss of flax stem is another indicator of the degree of degumming. The results showed that the microwave-assisted water retting (12.74% ± 1.01%) led to a higher percentage of flax loss than natural hot water retting (9.27% ± 0.21%) (Table 2). This data were in line with the degumming rate results. It could be concluded that the application of microwave-assisted water retting resulted in more thorough degradation of pectin substances.

Table 2. Properties of Flax Fiber

Retting Method	Degumming Rate (%)	Strength (N)	Ratio Variation of Strength (%)	Long Fiber Ratio (%)	Color		
					*L**	*a**	*b**
Microwave-assisted Water Retting	82.17 ± 1.14[a]	162.93 ± 2.37[a]	27.28 ± 1.03[a]	14.20 ± 0.65[a]	69.19 ± 1.04[a]	3.65 ± 0.17[a]	24.08 ± 1.09[a]
Natural Hot Water Retting	62.94 ± 2.53[b]	162.78 ± 3.19[a]	26.45 ± 0.61[a]	13.71 ± 0.24[a]	64.18 ± 1.14[b]	4.17 ± 0.23[a]	24.41 ± 1.33[a]
Different letters indicate significant ($P < 0.05$) difference among relative activity within the same column							

The strength, ratio variation of strength, and long fiber ratio of flax showed no significant difference due to treatment by either microwave-assisted water retting or natural hot water retting, as the microwave did not change the coefficients of the property values of the flax. The tensile properties in this work were lower than the values reported by Zhao *et al.* (2018) but were within average ranges (Faruk *et al.* 2012). The properties of fibers can indicate their resistance to deformation under applied loads or stresses (Brindha *et al.* 2017). The whiteness of flax fibers exposed to microwave-assisted water retting was higher than those with natural hot water retting, and there was no significant different in the redness or yellowness of the fibers. The colors of the fibers were different because the dissolved pectin components degraded differently. Moreover, the nature hot water retting system included dissolved and settled contaminants and colored materials, which can influence the whiteness of fibers (Wang and Postle 2004). This demonstrated that the industrial application of microwave-assisted water retting would not be harmful (Nair *et al.* 2013). The microstructure pictures of fibers in Fig. 5 show that the fiber had a rougher, fibrillated fiber surface topography after treatment by natural hot water retting, which was because lignin, pectin, and other components were not completely removed (Xu *et al.* 2015).

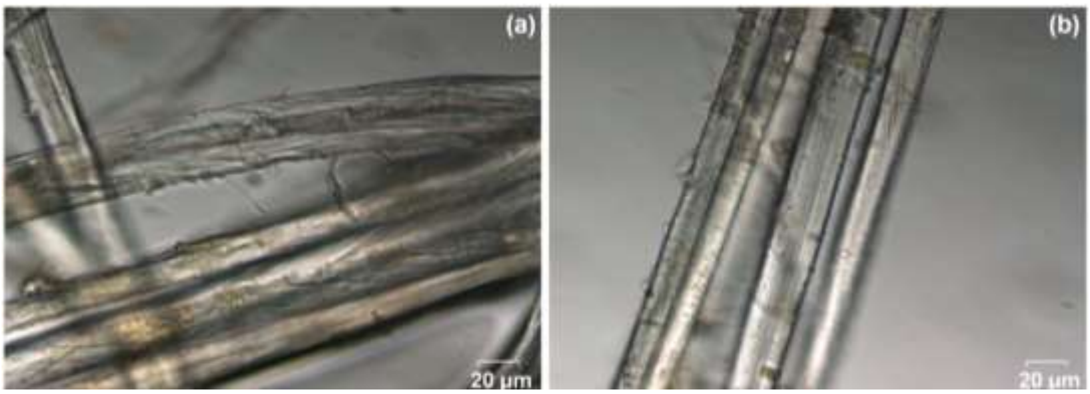

Fig. 5. Surface view of fibers *via* optical microscope of (a) natural hot water retted and (b) microwave-assisted retted

In contrast, the fiber exposed to microwave-assisted water retting had a smooth surface. This result was in accordance with the results of others (Nair *et al.* 2014), which demonstrated the effectiveness of microwave-assisted water retting. This method almost completely removed pectin substances in the flax stem, which resulted in the production of pure, individualized fibres. In addition, microwave-assisted water retting resulted in a shorter retting duration than that of natural hot water retting. However, it may be lead to some water pollution and contamination arising from pre-soaking of the flax material. So, in the further research, the changes in color, total dissolved solids (TDS), pH, conductivity quantity of sludge generated, total organic carbon (TOC), total organic nitrogen (TON), total organic phosphorus (TOP) and chemical oxygen demand (COD) of the pre-soaking water should be measured. Such research can help establish whether this type of retting can be useful for agriculture, industry or landscape development.

CONCLUSIONS

1. The degumming efficiency of flax was affected significantly by pre-soaking time, microwave time, and microwave power ($P < 0.05$).
2. At optimum process parameters of 25.5 h of pre-soaking time, 18.5 min of microwave time, and 570 W of microwave power, the degumming rate for fibers exposed to microwave-assisted treatment was 82.41% ± 1.07%, which was 1.33 times higher than those treated with natural hot water retting ($P < 0.05$).
3. Compared with natural hot water retting samples, the properties of fibers exposed to microwave-assisted treatment did not significantly change ($P > 0.05$), and the color and surface of the fibers were significantly improved ($P < 0.05$).
4. Microwave-assisted water retting resulted in a shorter retting duration than that required by nature hot water retting.

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APPENDIX

Table S1. Range of Different Factors Studied in the CCD

Variable Quantity	Level				
	-1.682	-1	0	1	+1.682
A: Pre-soaking Time (h)	3.2	10	20	30	36.8
B: Microwave Time (min)	6.6	10	15	20	23.4
C: Microwave Power (W)	297	400	500	600	668

Table S2. Experimental Design and Results of the CCD

Trial no.	A	B	C	Degumming Rate (%)	
				Estimated Value	Predicted Value
1	-1	1	1	50.61±0.03	45.97
2	1	-1	-1	32.30±0.11	33.29
3	-1.68	0	0	30.76±0.03	33.38
4	0	-1.68	0	42.46±0.05	35.91
5	0	0	0	74.34±0.13	77.45
6	0	0	0	75.35±0.04	77.45
7	0	0	0	73.36±0.12	77.45
8	0	0	0	74.74±0.11	77.45
9	1	1	1	85.45±0.17	77.45
10	1	1	-1	66.76±0.01	61.63
11	-1	1	-1	41.13±0.02	34.18
12	-1	-1	1	49.47±0.12	50.40
13	-1	-1	-1	30.17±0.06	33.76
14	0	1.68	0	43.66±0.11	56.02
15	0	0	-1.68	42.21±0.13	45.17
16	0	0	0	84.55±0.10	77.45
17	1	-1	1	52.45±0.02	55.50
18	0	0	0	83.26±0.15	77.45
19	0	0	1.68	71.11±0.12	73.76
20	1.68	0	0	57.74±0.14	60.74