White Poplar Microwave Pyrolysis: Heating Rate and Optimization of Biochar Yield

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White poplar is an important biomass resource because of its high yield and fast-growing characteristics. Experiments were conducted to study the effects of microwave power, moisture content, and particle size on the heating rate and biochar yield. A Central Composite Design (CCD) was used to optimize the biochar yield. The CCD results showed that a maximum temperature-increasing rate of 2.71 °C/s was obtained with a microwave power of 2 kW and a small particle size of 100-mesh. High power, small size, and high moisture content would benefit the increase of the heating rate. An optimum biochar yield of 0.905 kg per kg poplar was obtained with a microwave power of 3 kW, moisture content of 1%, and temperature of 500 °C.

Keywords: White poplar; Microwave pyrolysis; Heating rate; Biochar yield; Response surface

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INTRODUCTION

The plantation forest preservation area in China ranks first in the world and covers more than 62 million hectares. Woody biomass is a main resource for sustainable energy (Kim *et al.* 2011). White poplar is a fast-growing plantation forest species that is widely planted along the middle and lower reaches of the Yellow River and the Yangtzi River. In addition to using it as an industrial material (Chen and Cheng 2014), white poplar wood and its residues could be used for bioenergy through thermochemical conversion processes such as gasification and pyrolysis (Gu *et al.* 2013; Makibar *et al.* 2015).

Woody biomass can be converted into syngas, biochar, and bio-oil by using thermochemical conversion processes. Bioenergy is a clean and sustainable energy source. Therefore, the conversion of woody biomass to bioenergy has attracted considerable attention of many researchers (Halim and Swithenbank 2016).

Microwave pyrolysis has gained more attention in recent years (Huang *et al.* 2010; Motasemi and Afzal 2013; Zhao *et al.* 2014). Compared to traditional pyrolysis, microwave pyrolysis does not need external heat and it heats the biomass from inside out, generates rapid and uniform heating, and saves energy (Mašek *et al.* 2013). As a microwave absorber, water molecules can be caused to oscillate, thus generating heat. Therefore, microwaves can pyrolyze humid woody biomass with bulk size; while most other biomass material should be ground and dried before traditional pyrolysis processes (Gu *et al.* 2014; Chen *et al.* 2016).

Microwave power affects the yields of gas, liquid, and products of pyrolysis. Compared with conventional heating, it is possible to control microwave power to obtain high yields of gas and biochar that have high heating values and specific surface areas (Huang *et al.* 2016). The bio-oil yield of microwave pyrolysis is much lower than that from fluidized bed pyrolysis (Ren *et al.* 2012). The yield and characteristics of bio-oil and biochar can be affected by the microwave power, temperature range, and residence time. A biochar yield of 8.0% to 28.9% wood mass basis and bio-oil (a complex mix of more than 20 chemical components), respectively, can be obtained from microwave pyrolysis (Kim *et al.* 2011).

Activated biochars, an ordered mesoporous and microporous structure with a high surface area and a high pore volume (Thue *et al.* 2016), were prepared from wood chips using a microwave-induced chemical activation process. Such material has been used to improve soil conditions (Li *et al.* 2016). For the industrial application of the white poplar microwave pyrolysis, the yield of biochar must be considered.

Although microwave pyrolysis has been applied for several biomass materials, there is a lack of information on the microwave pyrolysis of white poplar (Wang *et al.* 2014; Quyang *et al.* 2016). The objective of this study was to study the heating characteristics of white poplar during microwave pyrolysis and to determine the effect of microwave power, particle size, and moisture content of white poplar on the heating rates and biochar yield during microwave pyrolysis. A central composite design (CCD) that was implemented in Design-Expert 8.0 software, was used to optimize the biochar yield from microwave pyrolysis. Results of this study are important to design full-scale microwave pyrolysis processes for white poplar and other biomass materials.

EXPERIMENTAL

Materials

White poplar planted in Huaian, in the Jiangsu province, China, was used in the experiments. Prior to the pyrolysis experiments, the poplar was prepared as cubes with the dimensions of 10 mm × 10 mm × 10 mm, 20 mm × 20 mm × 20 mm, chopped into sizes of 5 mm, and ground into 100-mesh sized particles. The cubes were prepared by using a cutting machine, and the particles were prepared by using a pulverizer (Daxiang Instruments co. LTD, DX-15). The white poplar samples were dried for 6 h at a temperature of 100 °C in an electrothermal constant-temperature dry box (Subo Instruments co. LTD, DHG101-00). Moisture contents were adjusted by adding deionized water. The moisture contents are shown in Table 1.

Experimental conditions

The microwave system used in this study was designed by the Biomass & Bioenergy Lab of the Nanjing Agricultural University and Nanjing Jinhaifeng Microwave Technology Ltd., China. The experimental system can provide variable power from 1 kW to 3 kW. The system was controlled by the Programmable Logic Controller (PLC; Model: SIEMENS CPU224XP, Siemens, Erfurt, Germany). The system was equipped with three microwave generators that can create the same frequency of 2.45 GHz (Samsung OM75P-31, Korea). They were installed separately on the rear, left, and right wall. The system was designed with the capability to control pyrolysis temperature. A K-thermocouple (Deke Machinery Technology co. LTD, WRNK-131) with a length of 40 cm was used to measure the temperature of the central point of the reaction chamber. The precision of the K-thermocouple was ±0.5 °C. The reaction chamber is 35 cm high and the diameter of its bottom is 15 cm. A data logger was used to save the temperature data in real time. A Programmable Logic Controller (PLC) was selected as a host controller. By using the RS458 communication interface, the temperature data could be transmitted to a PC at real-time and stored. The data collection frequency was 10 Hz. Figure 1 shows a schematic diagram of the experimental system.

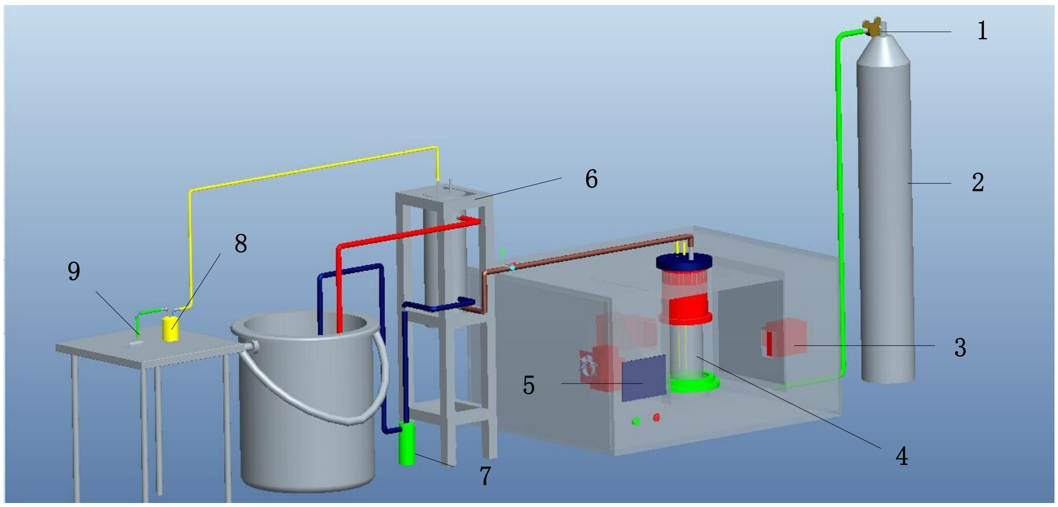

Fig. 1. The microwave pyrolysis system: (1- Relief valve; 2- Nitrogen; 3- Microwave generator; 4- Reaction chamber; 5- Control unit; 6- Condenser; 7-Circulating pump; 8- Gas filter; 9- Gas-collected bag)

Methods

The pyrolysis system was operated batchwise. Prior to each test, the microwave pyrolysis furnace was purged with pure nitrogen (99.999%) for 5 min to remove air from the reaction tank. Nitrogen was purchased from Nanjing Special Gas Factory Co., Ltd., China. To eliminate the influence of material loading amount on the heating rate, the amount of the poplar wood used in each run was 58 g ± 0.1 g.

The temperature of the material was increased over the pyrolysis time until reaching the desired value, then the microwave pyrolysis system controlled it automatically at the desired temperature for 3 min. The pyrolysis time was selected based on the authors' previous research on the pyrolysis of phoenix tree residue (Li *et al.* 2015). When the temperature was at 550 °C the pyrolysis was completed. The desired temperatures were also selected based on the previous research on the phoenix tree residues. Moreover, based on preliminary experiments on white poplar (data not shown), the highest temperature that could be reached with poplar about 850 °C; thus a maximum temperature of 800°C was applied. In this research, the microwave pyrolysis heating process was considered as a three-stage process: drying stage (before 120 °C), rapid heating stage (120 °C to 650 °C), and pyrolysis end stage (over 650 °C). The heating rate in this research was the rate of rapid heating stage. The temperature increase (from 120 °C to 650 °C) over the pyrolysis time was used to fit a linear equation. The heating rate was the slope of the driven linear equation.

Once the desired temperature was reached, the system stopped and the nitrogen was plowed into the pyrolysis chamber to cool the biochar. After the biochar was cooled to room temperature, it was weighted on a scale with two digits accuracy. The yield (M) of the produced biochar was calculated as a percentage of the wet mass of the poplar wood initially loaded in the reactor,

$$M = \frac{m_1}{m_2} \times 100\% \quad (1)$$

where m_1 is the mass of biochar (kg) and m_2 is the mass of biomass material (kg).

Response surface methodology (RSM)

Response surface methodology was applied to optimize the biochar yield using CCD that was implemented in Design-Expert 8.0 software (StatEase, USA) as described by Fan *et al.* (2014). The effects of the final reaction temperature, poplar moisture content, and microwave power on pyrolysis products yield (M) were determined. All the samples were ground into particle sizes of 100-mesh. The test parameters are listed in Table 1, and Table 2 shows the experimental design and results of the CCD test.

Table 1. Parameters and Levels for the CCD Test

Factors	-Alpha	+Alpha	Low	High
Temperature A (°C)	600	800	640.5	759.5
Moisture content B (%)	3	15	5.43	12.57
Microwave power C (kW)	1.5	2.5	1.7	2.3

Table 2. Experimental Design and Results of the CCD Test

Test No.	Independent Variable			Response Value
	A (°C)	B (%)	C (kW)	M
1	600	9	2	79.7%
2	759.5	12.57	1.7	82.0%
3	700	3	2	80.1%
4	759.5	5.43	1.7	80.6%
5	640.5	12.57	2.3	83.7%
6	640.5	5.43	1.7	84.9%
7	700	15	2	81.9%
8	700	9	2.5	81.9%
9	700	9	2	80.0%
10	640.5	5.43	2.3	83.7%
11	700	9	2	81.0%
12	700	9	2	80.1%
13	759.5	12.57	2.3	85.7%
14	700	9	2	80.0%
15	700	9	1.5	74.9%
16	640.5	12.57	1.7	83.7%
17	700	9	2	78.5%
18	800	9	2	80.7%
19	759.5	5.43	2.3	83.3%
20	700	9	2	79.8%

Single Factor Experimental Design

There are many factors that affect the microwave heating rate. In this study, only three factors were investigated: microwave power, moisture content, and size of the material. The experiments were conducted at variable microwave powers (1 kW, 1.5 kW, 2 kW, and 2.5 kW), sizes of materials (10-mm cubes, 20-mm cubes, and 100-mesh, chippings), and moisture contents of 3%, 6%, 9%, and 12% (wet basis). All experiments ran in duplicate. Table 1 shows the single factor experiment design. As shown, for each experimental group, only one factor was changed while keeping the others constant.

Table 3. Single Factor Experimental-design

Experimental Group No.	Moisture Content (%)	Microwave Power (kW)	Material Size
1 to 4	3	1, 1.5, 2, 2.5	100-mesh
5 to 8	3	2	10-mm cube, 20-mm cube, 100-mesh chippings
9 to 12	3, 6, 9, 12	2	100-mesh

RESULTS AND DISCUSSION

Analysis of Heating Rate

Effect of microwave power on heating rate

The temperature of the biomass material inside the pyrolyzer at different powers over the pyrolysis time, for poplar powder of 100-mesh and moisture content of 3%, is shown in Fig. 2.

As shown in Fig. 2, at a microwave power of 1 kW, the heating rate was extremely slow (0.13 °C/s), and the highest temperature was only 335 °C. Under these conditions, no gas was collected. When the microwave power was increased to 1.5 kW, the heating rate was approximately 0.81 °C/s, and the highest temperature was 450 °C (it is possible to achieve a higher temperature, but the microwave generator might overheat). When the microwave power was increased to 2 kW, the heating rate was 2.71 °C/s, and the highest temperature was 814 °C. When the power was further increased to 2.5 kW, the heating rate was 2.67 °C/s, which changed minimally. It is worth mentioning that at this condition there was a drop in the time-temperature curve at 500 s. According to the analysis of the shape of biochar in the reactor, a material collapse might appear at that moment; thus a temperature drop trend formed, and the final temperature was also lower than that of 2 kW.

The results indicated that microwave power had a certain effect on poplar microwave pyrolysis. However, when the power was higher than a special value between 2 and 2.3 kW, the temperature changes of poplar microwave pyrolysis were not significant. The findings suggested that the material had a peak heating rate during microwave pyrolysis. Moreover, the maximum heating rate depended on the material features. Microwave power had an impact on the heating rate before reaching the maximum rate. However, enhancing the microwave power cannot obviously accelerate the increase of temperature after reaching the maximum rate.

Effect of particle size on heating rate

The effect of different particle sizes (ground passed 100-mesh, chipped into cubes with the dimension of 10 mm × 10 mm × 10 mm, and 20 mm × 20 mm × 20 mm) on the heating rates and biomass temperature over the residence time is shown in Fig. 3. These experiments were conducted with the poplar that had a moisture content of 3% and used a microwave power of 2 kW.

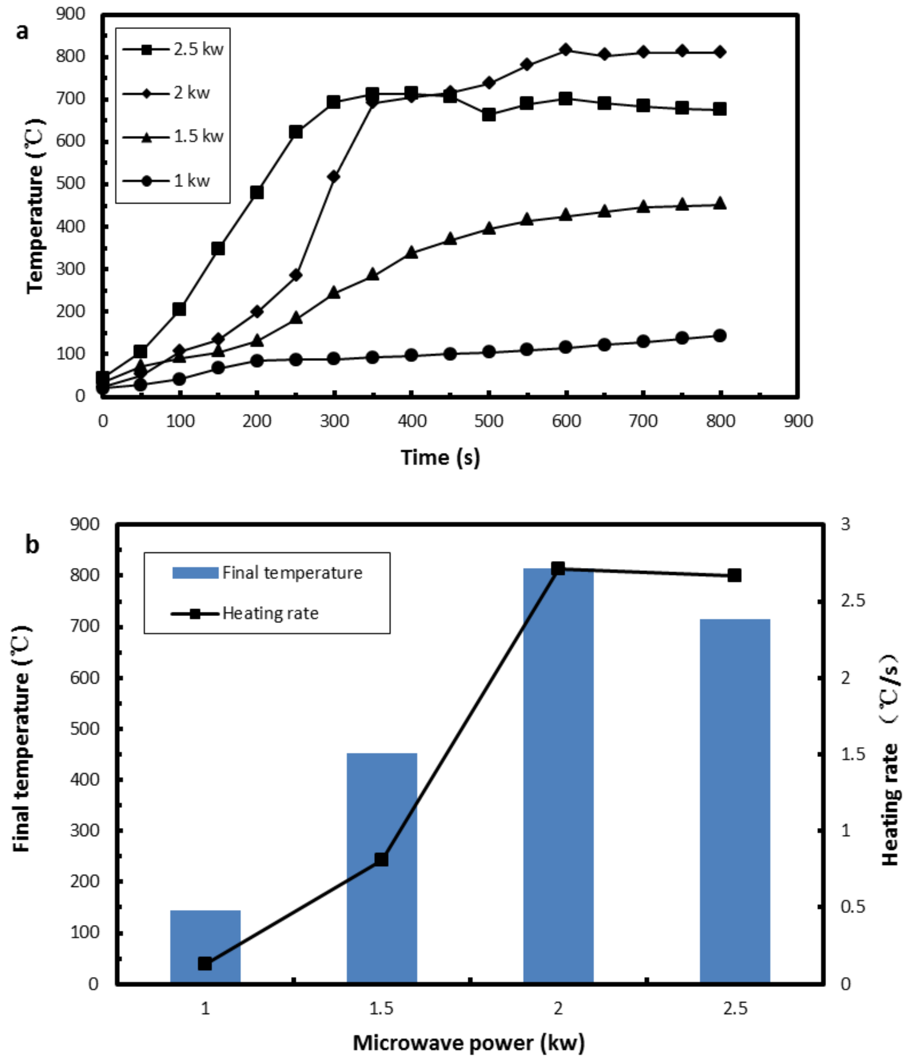

Fig. 2. The effect of diverse microwave power on the temperature curves (a) and heating rate (b) during poplar microwave pyrolysis

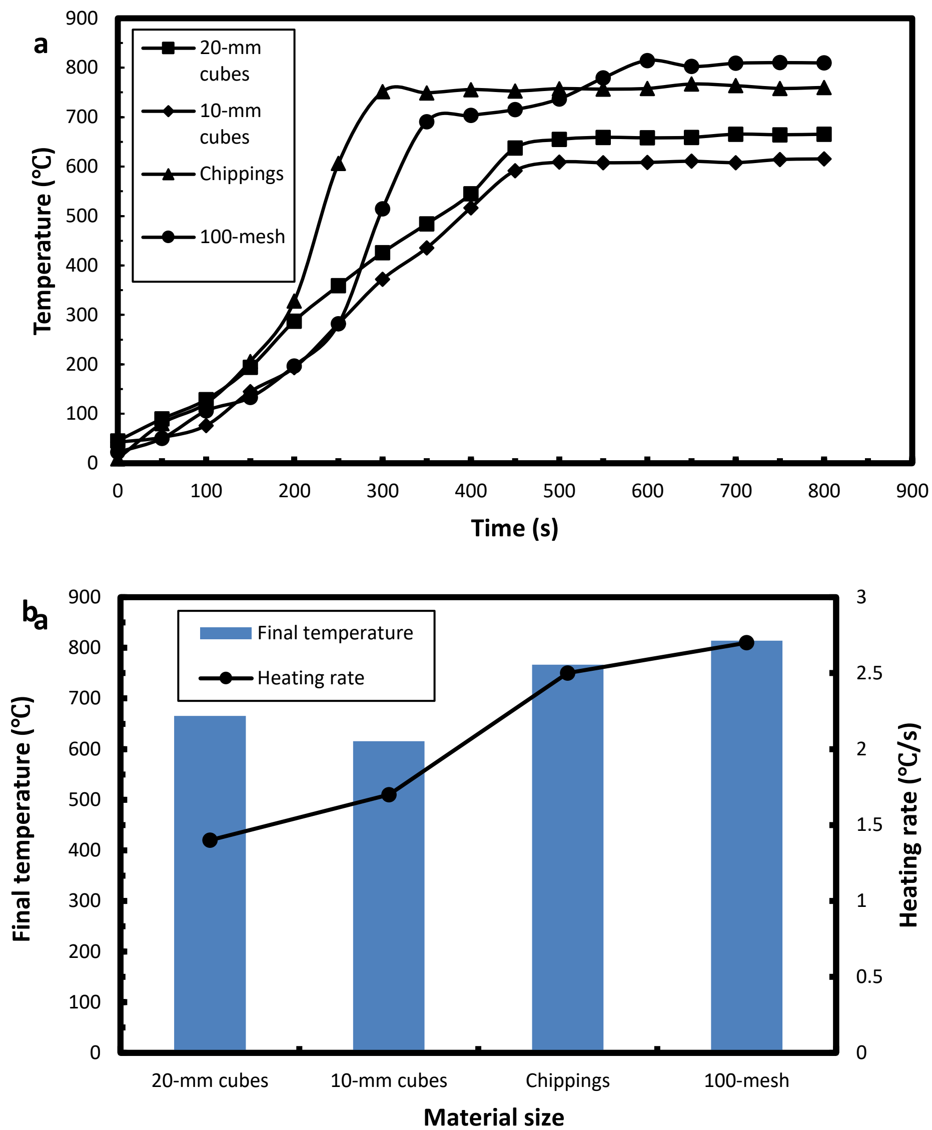

Fig. 3. Effect of poplar size on the temperature curves (a) and heating rate (b) during microwave pyrolysis

As shown in Fig. 3, higher temperatures were obtained for smaller particles than for the large particles. Crushed poplar into powder, both the 100-mesh and chippings samples had similar heating rates, which were approximately 2.7 °C/s. The heating rates for the cubes with different dimensions were similar with an average heating rate of approximately 1.5 °C/s. Particle size had little effect on the heating rates at higher temperatures.

According to the heating rates of block samples and powdered samples, the material size has little effect on the heating rate. The heating rate difference between powdered samples and block samples may be caused by the heat dissipation. This is because the block samples have larger air contact area; thus higher heat flux led to a lower heating rate.

Effect of moisture content on heating rate

The effect of moisture content on the heating rate is shown in Fig. 4. These experiments were conducted with poplar powder particle sizes of 100-mesh and a microwave power of 2 kW. The different moisture samples were prepared by adding water.

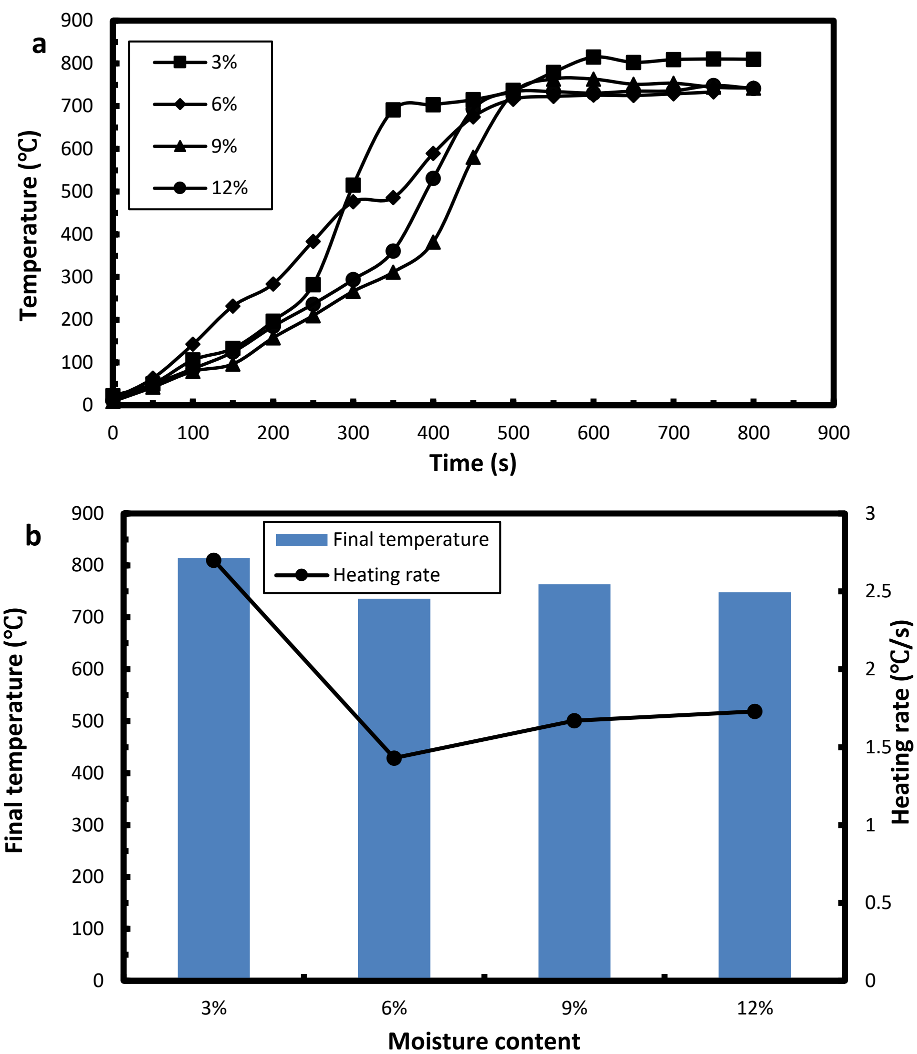

Fig. 4. The effect of moisture content on the temperature curves (a) and heating rate (b) during microwave pyrolysis

As shown in Fig. 4, the moisture content had little effect on the highest temperature. Compared with the heating rates, ignore the 3% moisture content group, the other three heating rates increased with the increase of moisture content; this suggested that water is a good microwave absorber, which was similar to Wang's research (Wang *et al.* 2012). However, the heating rate was extremely high at the moisture content of 3% (2.7 °C/s). This may have been because at the higher moisture content, water in the samples could not exhaust in time after conversion into vapor. After that, the vapor was involved in other endothermic reactions, thus resulting in the highest heating rate at a low moisture content.

Optimization of Biochar Yield

A regression analysis was conducted to determine the relation between biochar yield and pyrolysis temperature, biomass particle size, moisture content, and their interactions. The regression analysis was conducted using the coded values as presented in Eq. 2,

$$M = 3.06 - 4.75 \times 10^{-3}A - 0.04B - 0.45C + 2.94 \times 10^{-5}AB + 5.35 \times 10^{-4}AC + 2.4 \times 10^{-3}BC - 2.4 \times 10^{-6}A^2 + 8.9 \times 10^{-4}B^2 + 0.02C^2 \quad (2)$$

where the P values of A, B, AB, BC, and C^2 were 0.7608, 0.5316, 0.4662, 0.7636, and 0.7297, respectively. The results suggested that these factors were not significant, and thus they were removed. The optimized equation is as follows:

$$M = 0.8 + 0.012C + 9.45 * 10^{-3}AC + 8.33 * 10^{-3}A^2 + 0.011B^2 \quad (3)$$

The Model F-Value of 3.31 implies the model is significant. The P value of the fitting equation was 0.03, which indicated the fitting was good and the model was suitable. The ANOVA for this model are listed in Table 4. The response surface plot and contour plot of the effects of the three studied factors on biochar yield are shown in Figs. 5 through 7.

Table 4. ANOVA for Response Surface Reduced Quadratic Model

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob>F
Model	5.433E-003	4	1.358E-003	3.31	0.0391
C	2.133E-003	1	2.133E-003	5.20	0.0376
AC	7.145E-004	1	7.145E-004	1.74	0.2065
A^2	1.011E-003	1	1.001E-003	2.47	0.1372
B^2	1.798E-003	1	1.798E-003	4.39	0.0536
Residual	6.148E-003	15	4.098E-004		

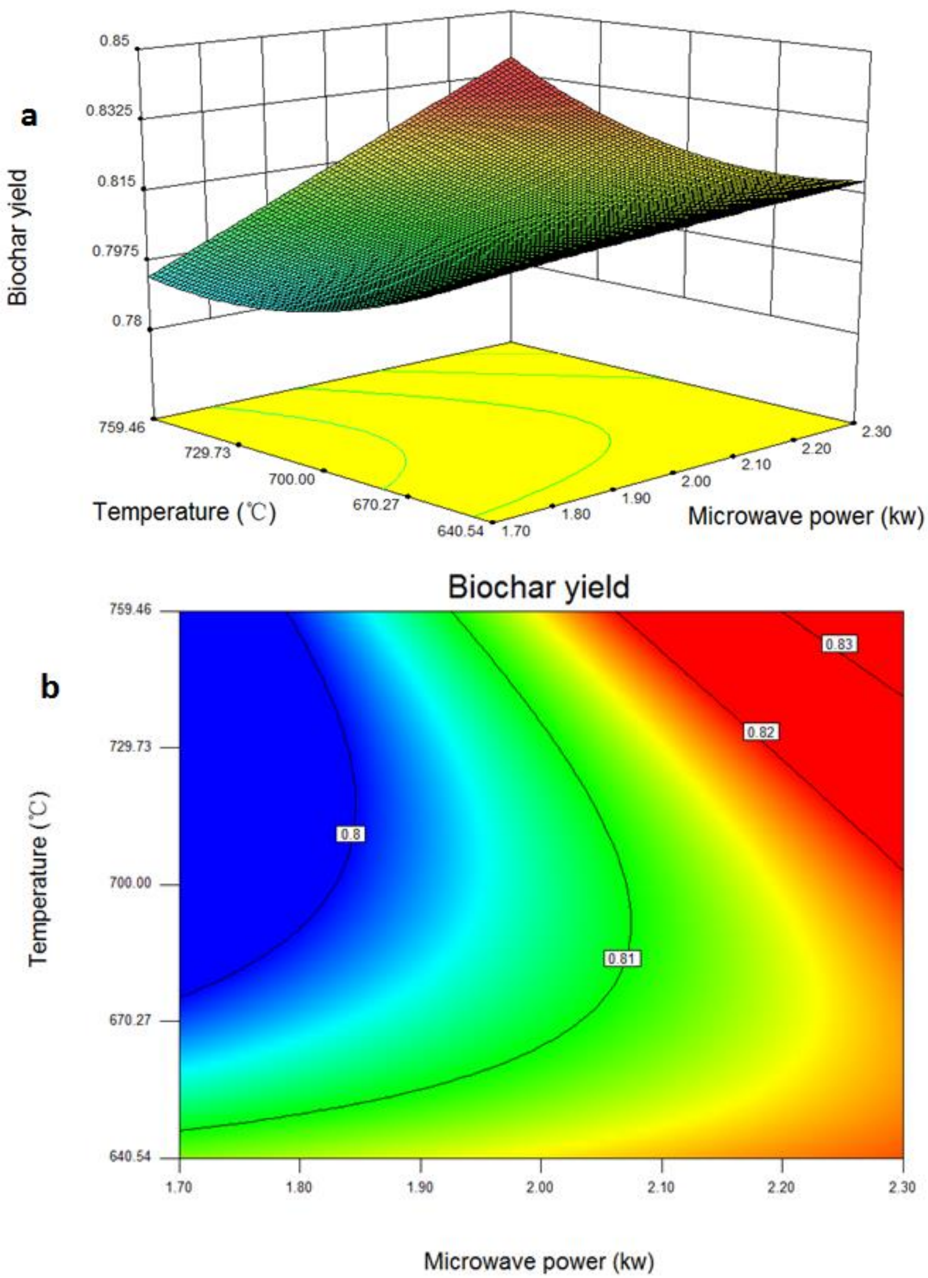

Fig. 5. The response curves (a) and contour lines (b) of biochar production under different temperatures and microwave power

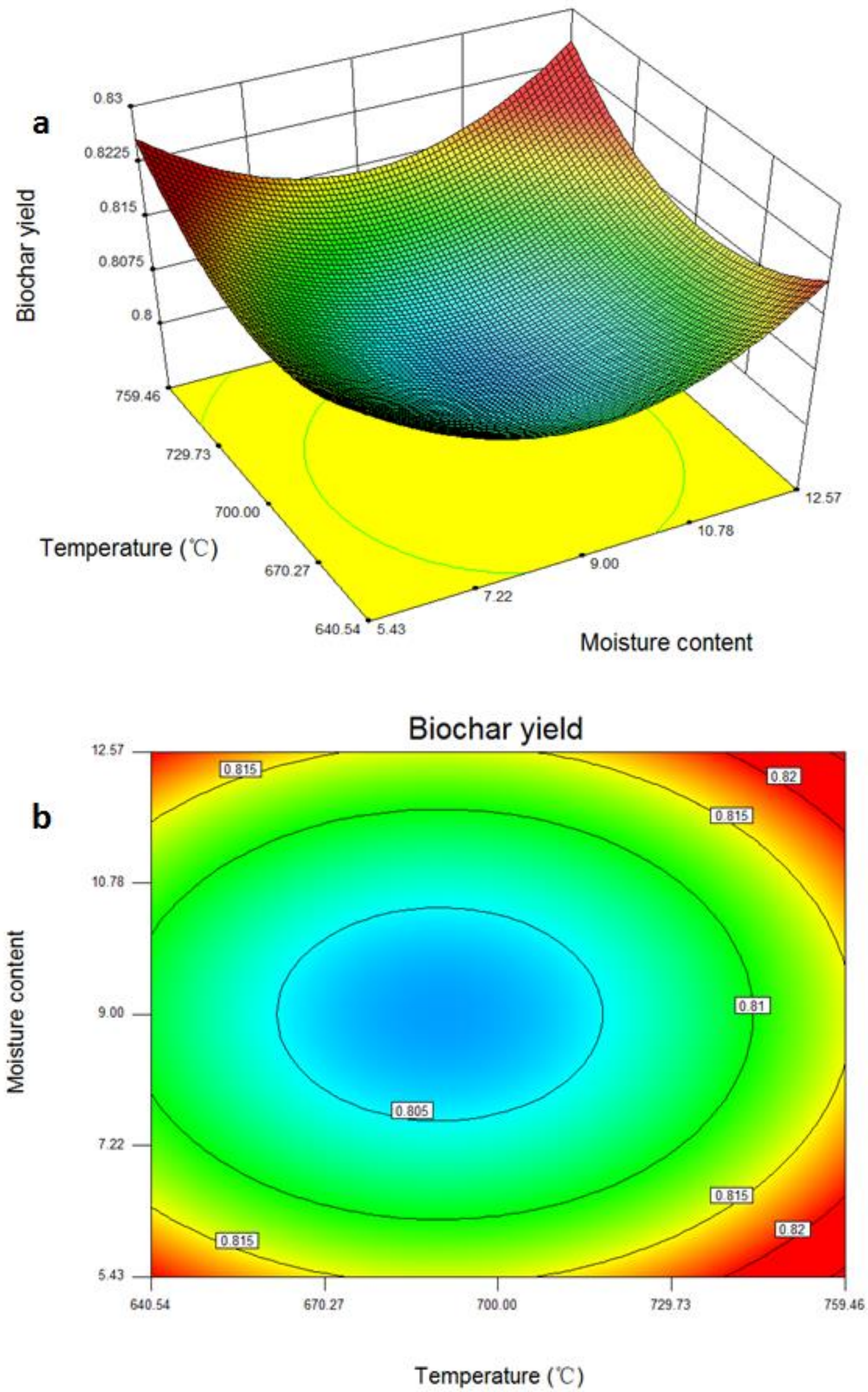

Fig. 6. The response curves (a) and contour lines (b) of biochar production under different temperatures and moisture contents

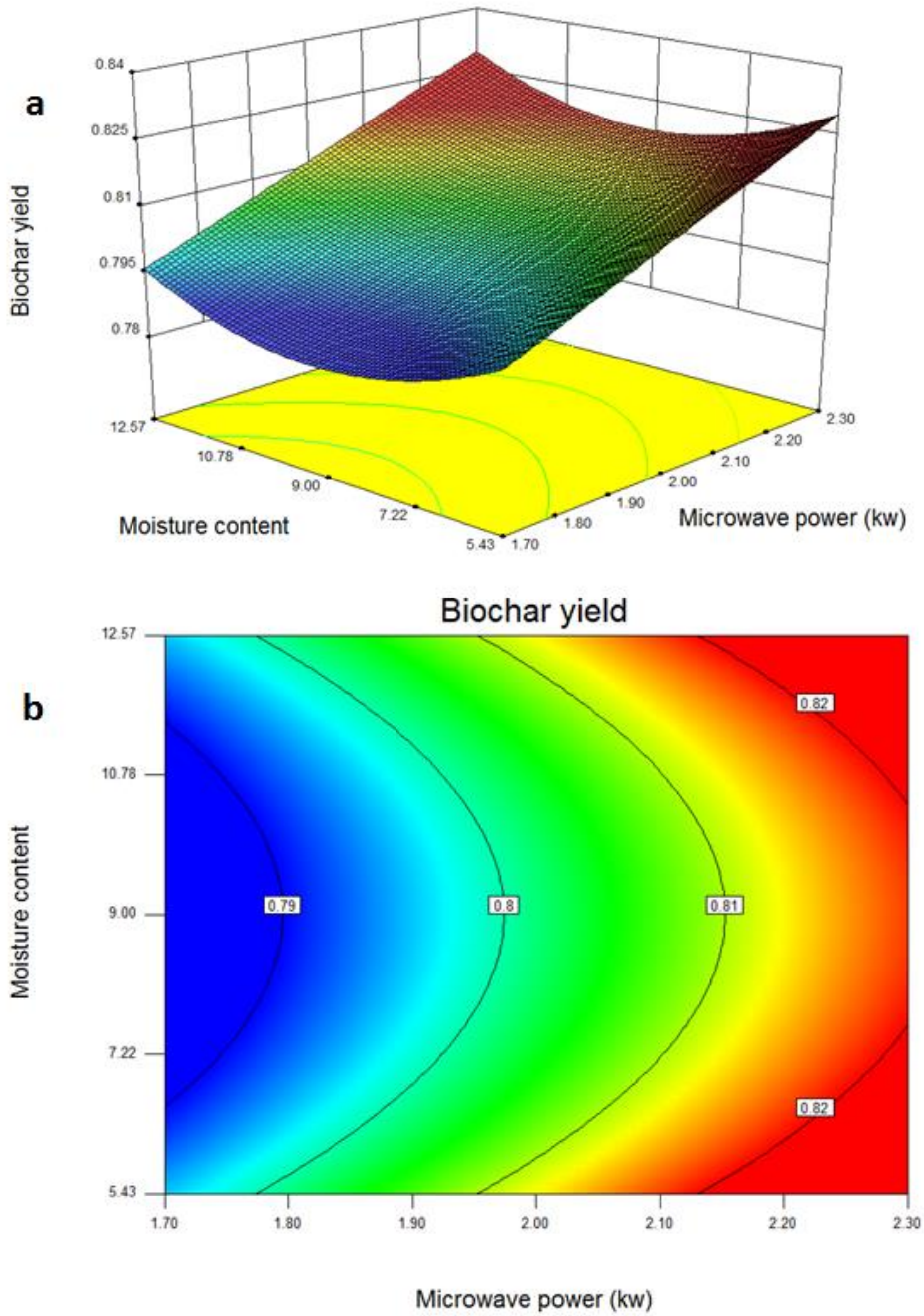

Fig. 7. The response curves (a) and contour lines (b) of biochar production under different moisture contents and microwave power

In Figs. 5 and 6, the temperature showed an inhibition and then promotion effect on the biochar production. This suggests that high temperature improved the pyrolysis. High temperature made the gas and liquid products more active and promoted secondary reaction with solid product. Therefore, the biochar amount decreased. However, when the temperature was higher, the liquid products were evaporated rather than decomposed, which led to an increased biochar amount.

Figures 5 and 7 showed that the biochar yield increased with the increase of microwave power and microwave power input affected the heating rate. Higher power input leads higher heating rate, and according to the research of Huang et.al (Huang et al. 2010), high heating rate decreased the yield of liquid. Therefore, the reaction of liquid production with biochar was suppressed, and the biochar production yield increased.

Figures 6 and 7 showed that the biochar yield first decreased, and then it increased with increased moisture content. Higher moisture content also increased the probability of secondary reactions. This can explain why the biochar yield first decreased. But according to the results of the single factor experiment, water is a good microwave absorber; thus higher moisture content leads higher heating rate. High heating rate decreased the yield of liquid; thus the reaction of liquid production with biochar was suppressed. This can explain why the biochar yield increased. However, because this experiment was limited by its conditions, different moisture content samples were made up of dried powder by adding water, so the mechanism of moisture content on biochar production needs further research.

Model Validation

By using the function of optimization in Design-Expert 8, the limits of three factors were set up, and the goal was to maximize biochar yield. According to the results of the software, the optimization parameters were temperature A 500 °C, moisture content B 1%, and microwave power C 3 kW. According to the parameters, three repeated verification tests were performed, and the experimental results are shown in Table 4.

Table 4. Verification of Test Results

Test No.	Biochar Production per Kilogram Poplar (M/kg)	Avg. (kg)	SD	Predicted Value (kg)
1	0.913	0.905	0.011	0.978
2	0.897			
3	0.906			

As shown in Table 4, the standard deviation (SD) was 0.011, which meant that the pyrolysis process was stable. The average (Avg.) was 0.905 kg, and according to the model, the predicted value was 0.978 kg (error, 8.07%). This result suggested that the fitting was good and the model was suitable.

CONCLUSIONS

1. Microwave power affected the heating rate during microwave pyrolysis of poplar. When the power reached 2kW, the heating rate was limited by the poplar features. Hence, the further increasing of power did not result in an increased heating rate.
2. High heating rates were achieved with the poplar powder than with bulk poplar. However, similar heating rates were observed with the cube samples (1.5 °C/s) or powdered samples (2.7 °C/s).
3. Higher heating rates were achieved with higher moisture contents. However, higher moisture content also brings other endothermic reactions, which decreased the heating rate. Thus, the highest heating rate was 2.7 °C/s when the moisture content was 3%.

4. The higher the pyrolysis power, the higher the biochar yield. The best data fitting for biochar yield as a function of moisture content and microwave power was: $M = 1.93 + 2.36 \times 10^{-6}A^2 + 7.5 \times 10^{-4}B^2 - 3.3 \times 10^{-3}A - 0.013B + 0.04C$.
5. It is feasible to use microwave pyrolysis the poplar for biochar, and the efficiency can be satisfactory.

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