

Article

# Biochar from Microwave Pyrolysis of *Artemisia Selengensis*: Characterization and Methylene Blue Adsorption Capacity

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**Featured Application:** This research provided a new treatment method for *artemisia selengensis*. Thus, it can produce biochar with high adsorbability instead of just wasted.

**Abstract:** In this research, *artemisia selengensis* was used to produce biochar via microwave pyrolysis. The influence of pyrolysis temperature, heating rates, temperature holding time and additive on the biochar yield and adsorbability were all investigated. The results suggest that the biochar yield decreased with the increase of pyrolysis temperature while the adsorbability of the biochar increased with an increase of the pyrolysis temperature; the biochar yield and its adsorbability could achieve the desired value when the heating rate and temperature holding time were in a specific scope; the biochar yield decreased when an additive was added; the adsorbability of the biochar could be increased by adding ZnCl<sub>2</sub> (metal chloride) and Na<sub>2</sub>CO<sub>3</sub> (metal carbonate). According to the orthogonal experiments, the optimal conditions for biochar production were: pyrolysis temperature 550 °C, heating rate 2 °C/s, temperature holding time 15 min, without additive.

**Keywords:** *artemisia selengensis*; microwave pyrolysis; biochar; adsorbability

## 1. Introduction

Activated carbon is an excellent adsorbent that has been applied in many applications [1]. Due to its stable chemical properties, high mechanical strength, acid and alkali resistance, heat resistance, insolubility in water and organic solvents, and renewability, activated carbon has been widely used in the chemical industry, environmental protection, food processing, refined, metallurgy, medicine, military chemical protection, and other fields [2–4]. Biomass is the main raw material of activated carbon. Several reviews report a great deal of work done on applications for the removal of specific pollutants from an aqueous phase [5]. Under the condition of high temperature and low oxygen, biomass could turn to biochar. However, the biochar often has poor quality, ash and other impurities are in the microporous, thus the adsorption performance is not good. Further improvement of the structure is needed to standardize biochar into activated carbon [6]. In recent years, microwave pyrolysis has gained increased attention from many researchers because of its unique heating mode and higher application prospect. Biochar made by microwave pyrolysis has a better structure than those made by traditional electric pyrolysis [7–11].

*Artemisia selengensis*, as compositae sagebrush and perennial herbaceous plant, was planted in shoals and moist areas. Due to its developmental nutrition, the cultivated area of *artemisia selengensis*

in Nanjing, Jiangsu province has reached  $2.33 \times 10^9$  hectares, and the annual production is more than 500,000 tons [12]. After two seasons, the aged stems of artemisia selengensis have no edible value, and they become a big problem that needs to be solved. High humidity and high fiber content made those aging stems difficult to mush. There has been little research focusing on the processing of aged stems. Traditional thermochemical conversion is not suitable for this material because it is highly humid and fibrous. However, microwave pyrolysis can effectively be used for the process because microwaves can affect biomass itself directly [13], and there are not many limitations on its size [14,15]. Water is a good kind of microwave absorber, so high moisture content also brings a high heating rate. Thus, the microwave pyrolysis technique is suitable to convert aged stems into biochar.

In this research, artemisia selengensis was used to produce biochar by using microwave pyrolysis technology. The influence of pyrolysis temperature, heating rates, temperature holding time and additive on the biochar yield and adsorbability were investigated. An orthogonal experiment was designed to find the optimum condition to produce biochar for the maximum adsorbability.

## 2. Materials and Methods

### 2.1. Materials

The artemisia selengensis straws used in this study were collected from Baguazhou, Nanjing (30°10' N, 118°49' E). And the picture of artemisia selengensis straw is shown in Figure 1. The collected straws had an average moisture content of 92.16% and an average diameter of 6 mm. Then, the straws were cut into 3 cm long pieces and stored for the pyrolysis experiments. The contents of artemisia selengensis straws were measured using the Van Soest. The content of hemicellulose, cellulose, lignin and ash is shown in Table 1. As can be concluded from Table 1, artemisia selengensis straws have more cellulose than rice straws, therefore, they are hard to mush. According to Luo and Wang's researches, the main product of cellulose pyrolysis is volatile, and volatile could make the biochar produced have more potential with more micropores [16,17]. Thus, the biochar produced by artemisia selengensis has porous properties. Furthermore, artemisia selengensis straws have little ash, which means there are little non-organic impurities in the biochar, which is a great condition for producing good adsorptive biochar.



**Figure 1.** Artemisia selengensis from Baguazhou.

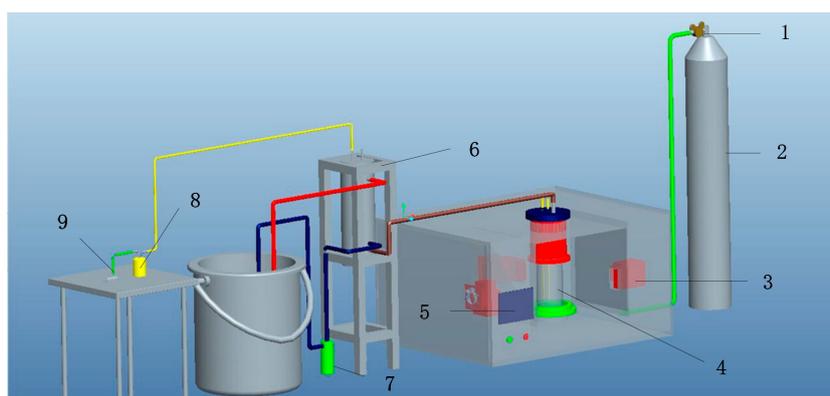
**Table 1.** Artemisia selengensis cellulose content.

Composition	Artemisia Selengensis	Rice Straw
Hemicellulose	4.056%	31.589%
Cellulose	55.335%	36.242%
Lignin	8.621%	7.128%
Ash	0.240%	1.032%

### 2.2. Experimental Conditions

The microwave system used in this study was designed by the Biomass and Bioenergy Lab of the Nanjing Agricultural University and Nanjing Jinhafeng Microwave Technology Ltd. (Nanjing, China).

The experimental system can provide variable power from 1 kW to 3 kW, which means that the heat rate is adjustable by changing the power consumption. The system was controlled by a programmable logic controller (PLC; Model: SIEMENS CPU224XP, Siemens, Erfurt, Germany). The system was equipped with three identical microwave generators that can create a frequency of 2.45 GHz (Samsung OM75P-31, Daegu, Korea). They were installed separately on the rear, left, and right wall. The system was designed capable of controlling the pyrolysis temperature. A thermocouple was used to measure the temperature of the central point of the reaction chamber. A data logger was used to save the temperature data in real time. Figure 2 shows a schematic diagram of the experimental system. The model of the ultraviolet spectrophotometer is UNICO UV-2800 (Unico, Shanghai, China). The model of the scanning electron microscope (SEM) is Phenom ProX (Phenom Scientific, Hillsboro, OR USA).



**Figure 2.** 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-collection bag).

### 2.3. Experimental Method

#### 2.3.1. Biochar Production Method

High purity  $N_2$  (99.99%) was purged for over 10 min to completely expel the air in the microwave pyrolysis oven. The pyrolysis temperature was measured by a thermocouple. Related parameters like pyrolysis temperature and microwave power were established according to the experimental design. To eliminate the influence of the quality of material,  $45 \text{ g} \pm 0.1 \text{ g}$  artemisia selengensis straw was added in every group. The biochar was collected and prepared for acid pickling after cooled.

#### 2.3.2. Acid Pickling

All the biochar samples were mixed with 100 mL hydrochloric acid ( $\text{pH} = 1$ ,  $0.1 \text{ mol/L}$ ). To remove the ash, oxygen-containing functional groups and other impurities, the mixture was boiled for 3 min. After acid pickling, samples were filtered. After the biochar dried, all groups were weighted and prepared for the adsorption experiments.

The biochar yield was calculated using the formula below:

$$\text{Biochar yield} = \frac{m_1}{m_2} \times 100\% \quad (1)$$

where  $m_1$  is the mass of biochar after acid pickling (g) and  $m_2$  is the mass of biomass material (g).

#### 2.3.3. Biochar Adsorption Method

The carbon decolorization ability to methylene blue solution can reflect the adsorption ability of 2 nm mesoporous in carbon. Thus the adsorption value of biochar to methylene blue solution is used as an indicator its adsorption performance in this research. The operations are as follows:

CuSO<sub>4</sub> solution (0.025 mol/L, blank group) and methylene blue solution (0.025 g/L, 0.05 g/L, 0.2 g/L and 0.5 g/L) were prepared as standard samples. The wavelength of the ultraviolet spectrophotometer was set at 655 nm. The calibration curve was drawn by measuring the absorbance of standard samples.

Take 0.1 g acid pickled biochar from every test group and put it into a 50 mL conical flask. Twenty milliliter of 0.5 g/L methylene blue solution was added in the conical flask, and then put the conical flask onto constant temperature shaker for 20 min, at temperature 20 °C and speed 120 r/min. If the solution decolorizes after shaking, 5 mL more methylene blue solution (0.5 g/L) should be added and then shake the conical flask until the solution stays blue. The adsorption values of biochar are calculated by the calibration curve and absorbance.

#### 2.4. Experimental Design

The effect of pyrolysis temperature, heating rates, temperature holding time and additive (additive amount 5%) on the biochar adsorbability were investigated. The experiment conditions are shown in Tables 2 and 3.

**Table 2.** The single factor experiment conditions.

No.	Factor		
	Factor	Level	Condition
1–4	A (pyrolysis temperature °C)	450, 500, 550, 600	B = 0.5 °C/s, C = 10 min, D = None
5–8	B (heating rate °C/s)	0.5, 1, 1.5, 2	A = 450 °C, C = 10 min, D = None
9–12	C (temperature holding time min)	10, 15, 20, 25	A = 450 °C, B = 0.5 °C/s, D = None
13–16	D (additive)	None, Char, ZnCl <sub>2</sub> , Na <sub>2</sub> CO <sub>3</sub>	A = 450 °C, B = 0.5 °C/s, C = 10 min

**Table 3.** The orthogonal experiment schedule and result.

No.	A: Pyrolysis Temperature (°C)	B: Heating Rates (°C/s)	C: Temperature Holding Time	D: Additive	Biochar Yield	Adsorbing Capacity (mg/g)
1	450	0.5	10	None	5.00%	6.989
2	450	1	15	Char	3.66%	7.507
3	450	1.5	20	ZnCl <sub>2</sub>	3.19%	17.603
4	450	2	25	Na <sub>2</sub> CO <sub>3</sub>	2.86%	13.979
5	500	0.5	15	ZnCl <sub>2</sub>	2.55%	27.958
6	500	1	10	Na <sub>2</sub> CO <sub>3</sub>	2.39%	41.160
7	500	1.5	25	None	2.72%	41.936
8	500	2	20	Char	2.68%	15.791
9	550	0.5	20	Na <sub>2</sub> CO <sub>3</sub>	2.88%	45.158
10	550	1	25	ZnCl <sub>2</sub>	2.97%	39.348
11	550	1.5	10	Char	-1.42%	10.096
12	550	2	15	None	2.82%	140.564
13	600	0.5	25	Char	1.72%	16.567
14	600	1	20	None	5.00%	62.128
15	600	1.5	15	Na <sub>2</sub> CO <sub>3</sub>	3.66%	9.060
16	600	2	10	ZnCl <sub>2</sub>	3.19%	67.305

### 3. Results

#### 3.1. The Effect of Pyrolysis Temperature

The effect of different pyrolysis temperatures (450 °C, 500 °C, 550 °C, 600 °C) on the biochar adsorption is shown in Figure 3. These experiments were conducted at a heating rate of 0.5 °C/s, a temperature holding time of 10 min and without additive.

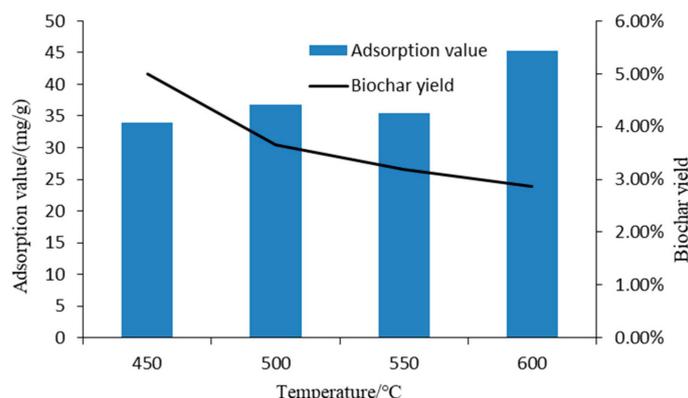


Figure 3. Effect of temperature on the biochar yield and adsorbability.

As shown in Figure 3, with the increasing pyrolysis temperature, the biochar yield decreases, but the adsorbability of the biochar increases. The adsorbability of biochar reached a peak at a pyrolysis temperature of 600 °C, and the highest adsorbing capacity was 45.302 mg/g. It is worth mentioning that the moisture content of the sample was about 98.16%. After pyrolysis, the maximum biochar yield was 5%, and the actual conversion rate was 63.8%, which was relatively high. High temperature can promote the secondary reaction of biochar with other products and vapor in the reactor, thus the biochar yield decreases as the pyrolysis temperature increases. The influence of the pyrolysis temperature on the adsorbability of biochar was not obvious, but from 550 °C to 600 °C, the adsorbability of biochar increased significantly. It may suggest that a high pyrolysis temperature can promote a secondary reaction of biochar with the gas and liquid products [13,18], thus the biochar structure was optimized and the adsorbability of the biochar rose significantly. Combined with the result of carbon yield, the variation trend of the biochar yield reduced after 550 °C, which means that the secondary reaction was roughly completed and this also proved that a secondary reaction could improve the adsorbability of biochar to some extent.

### 3.2. The Effect of Heating Rate

The effect of different heating rates (0.5 °C/s, 1 °C/s, 1.5 °C/s, 2 °C/s) on the biochar adsorption is shown in Figure 4. These experiments were conducted at a pyrolysis temperature of 450 °C, a holding time of 10 min and without additive.

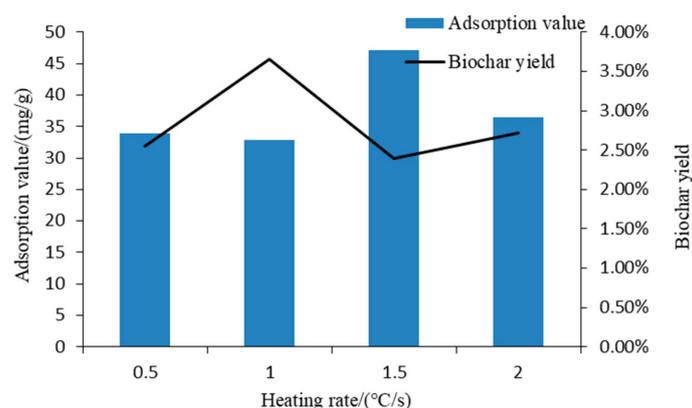


Figure 4. Effect of heating rate on the biochar yield and adsorbability.

As shown in Figure 4, the biochar yield reached a peak of 3.66% when the heating rate was 1 °C/s. The adsorbability of the biochar reached its highest value of 47.114 mg/g when the heating rate was 1.5 °C/s. However, at other heating rates, the biochar yields were close to 2.50%, and adsorbing capacities of biochar were close to 35 mg/g. It may suggest that when the heating rate was low,

the reaction speed and the gas production yield were also low. The gas production could not release in time, thus the probability of a secondary reaction increased, and the biochar yield was low. On the contrary, when the heating rate was high, the reaction speed was also high. The gas could not release in time, which increased the probability of the initiation of a secondary reaction. Though the secondary reaction could improve the adsorbability of the biochar, the low heating rate leads to a low gas production yield, thus the quality of the biochar was poor [19]. However, when the heating rate was high, the reaction speed was also high, and the gas products might release in a short time, thus the biochar structure was irregular, and thus resulted in poor adsorption.

### 3.3. The Effect of Holding Time

The effect of different temperature holding times (10 min, 15 min, 20 min, 25 min) on the biochar adsorption is shown in Figure 5. These experiments were conducted at a pyrolysis temperature of 450 °C, a heating rate of 0.5 °C/s, and without additive.

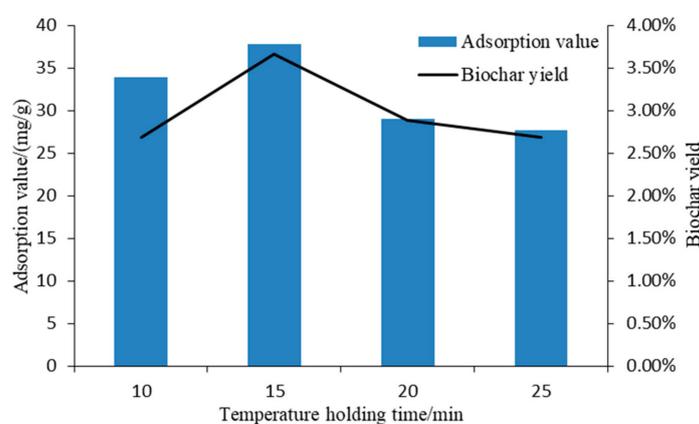
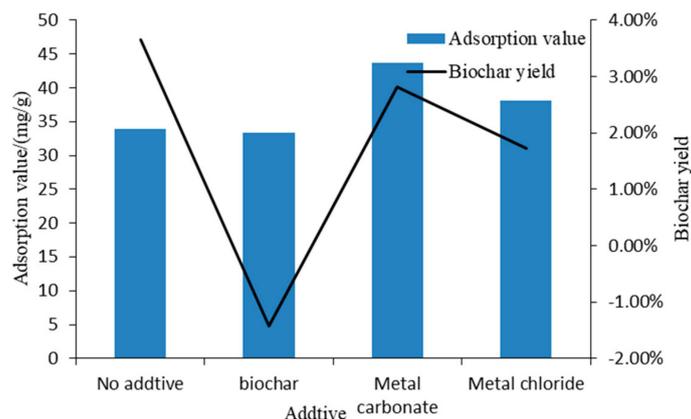


Figure 5. Effect of holding time on the biochar yield and adsorbability.

As shown in Figure 5, with the temperature holding time increasing, the biochar yield and adsorbability of biochar firstly increased at the holding time from 10 to 15 min, then decreased. At a temperature holding time of 15 min, the biochar yield and biochar adsorbability reached a peak value of 3.66% and 37.794 mg/g relatively at the same time. This may suggest during the first 15 min, the remaining material in the reactor continued to react, and the main reaction was coking for the liquid product, thus the biochar yield increased. As the holding time increased, the main reaction became biochar with other products, thus the biochar yield decreased. For the biochar adsorption, because of a short temperature holding time, the biochar structure could not reach an optimum; when the temperature holding time was exceeded, the biochar structure was destroyed by the high temperature [1]. What is more, longer temperature holding times led to more damage to the pore structure. To optimize the adsorbability of biochar, an optimal temperature holding time of approximately 15 min should be selected.

### 3.4. The Effect of Different Additives

The effect of different additives (none, biochar, metal carbonate- $\text{Na}_2\text{CO}_3$ , metal chloride- $\text{ZnCl}_2$ ) on the biochar adsorbability is shown in Figure 6. These experiments were conducted at a pyrolysis temperature of 450 °C, a heating rate of 0.5 °C/s, and a temperature holding time of 10 min.



**Figure 6.** Effect of additive on the biochar yield and adsorbability.

As shown in Figure 6, the additives have a significant influence on biochar yield. The biochar yield reached the highest value without additive. For the biochar adsorbability, the addition of biochar exerted no significant influence on the adsorbability. The values of the adsorption were both around 33.455 mg/g with or without additive. However, the addition of metal carbonate or metal chloride improved the adsorbability of the biochar. The highest adsorbing capacity was 43.748 mg/g when Na<sub>2</sub>CO<sub>3</sub> (metal carbonate) was used as an additive. This may suggest that the additive improves the ability of the sample to absorb microwaves, and this could cause hotspots in the samples, thus the adsorbability of the biochar could be improved. Moreover, metal carbonate releases CO<sub>2</sub> when heated, which can improve the biochar pore forming efficiency. It is worth mentioning that when adding biochar, the biochar yield was negative. This is because the additives were not considered as reactants, thus the mass was subtracted during the calculation of the biochar yield. However, in fact, biochar added as an additive indeed reacted, thus the subtraction of reacted additive caused the biochar yield to be negative. For the adsorbability, when the biochar acts as an additive, it could improve the adsorbability of the biochar, but since the moisture content of the sample was 92.16% and the additive content was 5%, the final biochar obtained was mainly the additive biochar. In addition, for the additive biochar, it was re-heated, and the structure was damaged, and this caused the poor adsorbability.

### 3.5. Orthogonal Experiment Analysis

Due to the high moisture content of the material, all groups of the biochar yield were low, so there is no need to discuss the optimal conditions for biochar yield in this research. Thus, in this part, only biochar adsorbability was analyzed.

ANOVA (analysis of variance) was used in this paper to analyze biochar adsorbability. The results are shown in Tables 4 and 5.

**Table 4.** The orthogonal experiment results calculation table.

Factor	A	B	C	D
k <sub>1</sub>	11.5195	24.1680	31.3875	63.6543
k <sub>2</sub>	31.7113	38.2858	46.2723	12.4903
k <sub>3</sub>	58.7915	19.6738	35.9200	38.0535
k <sub>4</sub>	39.5150	59.4098	27.9575	27.3393

**Table 5.** The orthogonal experiment results analysis of variance table.

Variance	Sum Of Squares	DOF (Degree Of Freedom)	MS (Mean Square)	F-Value	Significance
A	4591.9187	3	1530.64	1.599	Insignificant
B	3833.0697	3	1277.69	1.335	Insignificant
C	759.8648	3	253.288	0.265	Extremely significant
D	5580.7002	3	1860.23	1.943	Insignificant
Deviation	2871.7577	3	957.253		

Note:  $F_{0.01}(3,3) = 29.46$ ,  $F_{0.05}(3,3) = 9.28$

As shown in Table 5, all four factors have an insignificant influence on the adsorbability of the biochar following the order of  $D > A > B > C$ , which is: additive > pyrolysis temperature > heating rate > temperature holding time. To improve the biochar adsorbability, the optimal conditions were  $A_3B_4C_2D_1$ , that means the pyrolysis temperature is 550 °C, the heating rate is 2 °C/s, the temperature holding time is 15 min and without additive.

Two more repeated experiments were done according to the optimal conditions, and the results are shown in Table 6.

**Table 6.** The verification test results.

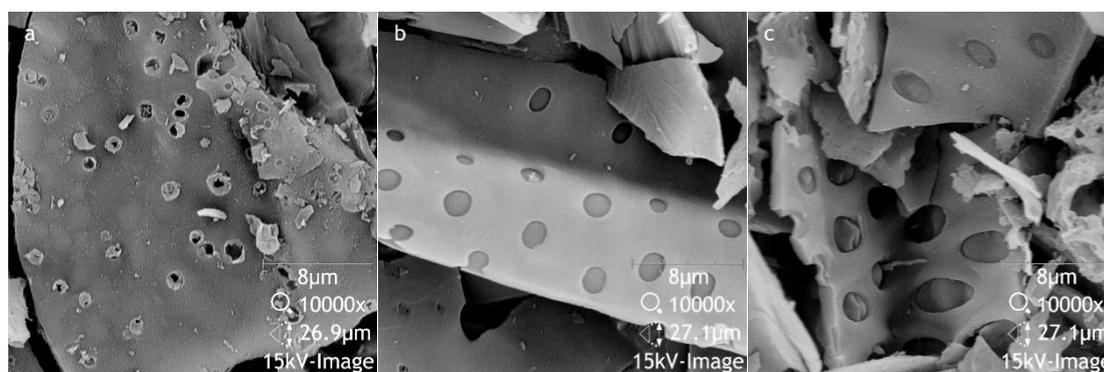
Test No.	Adsorbing Capacity mg/g	AVG (Average) mg/g	SD (Standard Deviation) mg/g
1*	140.564		
2	142.172	140.038	2.440
3	137.378		

\*: The result was picked from the orthogonal experiment.

For the activated carbon index, the national primary standard (for water purification) requires the adsorbing capacity for methylene blue to be more than 135 mg/g [1], which means that the biochar produced under these conditions only needs acid pickling to reach the national primary standard. This means that the biochar has a high application value.

### 3.6. SEM Representation

The three SEM tests were from the repeat test in a single factor experiment (pyrolysis temperature 450 °C, heating rate 1 °C/s, temperature holding time 10 min, without additive); metal-carbonate-added test in a single factor experiment (pyrolysis temperature 450 °C, heating rate 1 °C/s, temperature holding time 10 min,  $Na_2CO_3$  additive) and the optimal condition test (pyrolysis temperature 550 °C, heating rate 2 °C/s, temperature holding time 15 min, without additive). For the SEM, the amplification factor is 10,000, the scale length is 8 μm, and images are shown in Figure 7.



**Figure 7.** SEM (scanning electron microscope) images of biochar: (a) repeat test group in single factor experiment; (b) metal-carbonate-added test group; (c) the optimal condition test.

Figure 7a shows the images of the repeat test group in a single factor experiment with an adsorbing capacity of 33.911 mg/g (pyrolysis temperature 450 °C, heating rate 1 °C/s, temperature holding time 10 min, without additive). As can be seen in Figure 7a, the surface pores of the biochar are quite small, the diameters of those pores are less than 1 µm and have a non-uniform distribution.

Figure 7b represents the result of the metal-carbonate-added test group in a single factor experiment with an adsorbing capacity of 43.748 mg/g (pyrolysis temperature 450 °C, heating rate 1 °C/s, temperature holding time 10 min, Na<sub>2</sub>CO<sub>3</sub> additive). As can be seen in Figure 7b, the number of biochar surface pores is larger than that in Figure 7a and the surface pores are more structured and evenly distributed although the surface pores are shallow.

Figure 7c shows an image of the optimal condition test with an adsorbing capacity of 140.564 mg/g (pyrolysis temperature 550 °C, heating rate 2 °C/s, temperature holding time 15 min, without additive). The image shows that in this test, biochar has the largest number of surface pores, the biggest pore diameter, the deepest surface pores, and the most even distribution.

#### 4. Conclusions

As the pyrolysis temperature increased, the biochar yield decreased while the adsorbability of the biochar increased and reached a peak at a pyrolysis temperature of 600 °C.

The biochar yield and adsorbability had a similar trend as the heating rate increased. The biochar yield reached a peak at a heating rate of 1 °C/s, and the adsorbability of the biochar reached a peak at 1.5 °C/s.

The biochar yield and adsorbability both increased when the temperature holding time was increased, reaching the highest value at 15 min. Then they both decreased when the holding time was longer than 15 min.

Additives decreased the biochar yield. There was no significant influence on the adsorbability of the biochar whether carbon (or nothing added) was used as an additive. The addition of Na<sub>2</sub>CO<sub>3</sub> (metal carbonate) or ZnCl<sub>2</sub> (metal chloride) could improve the adsorbability of the biochar. The highest adsorbing capacity was reached when Na<sub>2</sub>CO<sub>3</sub> (metal carbonate) was used as an additive.

For the biochar adsorbability, the order of influence for the four factors is additive > pyrolysis temperature > heating rate > temperature holding time. The optimal conditions were pyrolysis temperature 550 °C, heating rate 2 °C/s, temperature holding time 15 min and without additive. The SEM results also indicate the reasons for the differences of adsorbability from the structures.

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