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## Preparation of biomass-based mesoporous carbon with higher nitrogen/oxygen-chelating adsorption for Cu(II) through microwave pre-pyrolysis --Manuscript Draft--

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**TITLE:**

Preparation of Biomass-based Mesoporous Carbon with Higher Nitrogen-/Oxygen-chelating Adsorption for Cu(II) Through Microwave Pre-Pyrolysis

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**KEYWORDS:**

Mesoporous carbon, biomass, microwave pyrolysis, electric furnace, copper adsorption, Fourier transform infrared (FTIR) spectroscopy, modification, nitrogen, adsorption isotherm, adsorption kinetics, functional group

**SHORT ABSTRACT:**

Here, we present a protocol to synthesize nitrogen/oxygen dual-doped mesoporous carbon from biomass by chemical activation in different pyrolysis modes followed by modification. We demonstrate that the microwave pyrolysis benefits the subsequent modification process to simultaneously introduce more nitrogen and oxygen functional groups on the carbon.

**LONG ABSTRACT:**

An environment-friendly technique for synthesizing biomass-based mesoporous activated carbon with high nitrogen-/oxygen-chelating adsorption for Cu(II) is proposed. Bagasse impregnated with phosphoric acid is utilized as the precursor. To pyrolyze the precursor, two separate heating modes are used: microwave pyrolysis and conventional electric-heating pyrolysis. The resulting bagasse-derived carbon samples are modified with nitrification and reduction modification. Nitrogen (N)/oxygen (O) functional groups are simultaneously introduced to the surface of activated carbon, enhancing its adsorption of Cu(II) by complexing and ion-exchange. Characterization and copper adsorption experiments are performed to investigate the physicochemical properties of four prepared carbon samples and determine which heating method favors the subsequent modification for doping of N/O functional groups. In this technique, based on analyzing data of nitrogen adsorption, Fourier transform infrared spectroscopy, and batch adsorption experiments, it is proven that microwave-pyrolyzed carbon has more defect sites and, therefore, time-saving effective microwave pyrolysis contributes more N/O species to the carbon, although it leads to a lower specific surface area. This technique offers a promising route to synthesis adsorbents with higher nitrogen and oxygen content and a higher adsorption capacity of heavy-metal ions in wastewater remediation applications.

## INTRODUCTION:

Activated carbon has unique adsorption properties, such as a developed porous structure, a high specific surface area, and various surface functional groups; therefore, it is employed as an adsorbent in water treatment or purification<sup>1-4</sup>. Besides its physical advantages, activated carbon is cost-effective and harmless to the environment, and its raw material (*e.g.*, biomass) is abundant and easily obtained<sup>5,6</sup>. The physicochemical properties of activated carbon depend on the precursors that are used in its preparation and on the experimental conditions of the activation process<sup>7</sup>.

Two methods are typically used to prepare activated carbon: a one-step and a two-step approach<sup>8</sup>. The term one-step approach refers to precursors being carbonized and activated simultaneously while the two-step approach refers to that sequentially. In view of energy conservation and environmental protection, the one-step approach is more preferred for its lower temperature and pressure demanding.

Besides, chemical and physical activation are utilized to improve the textural properties of activated carbon. Chemical activation possesses apparent advantages over physical activation because of its lower activation temperature, shorter activation time, higher carbon yield, and more developed and controllable pore structure in a certain degree<sup>9</sup>. It has been tested that chemical activation can be performed by impregnating biomass used as feedstock with  $\text{H}_3\text{PO}_4$ ,  $\text{ZnCl}_2$ , or other specific chemicals, followed by pyrolysis to increase the porosity of the activated carbon, because lignocellulosic components of biomass can be easily removed by a subsequent heating treatment, owing to the dehydrogenation capability of these chemicals<sup>10,11</sup>. Hence, chemical activation greatly enhances the formation of activated carbon's pores or improves the adsorptive performance to contaminants<sup>12</sup>. An acidic activator is preferred to  $\text{H}_3\text{PO}_4$ , due to its relatively lower energy demand, higher yield, and less impact on the environment<sup>13</sup>.

Microwave pyrolysis has the superiority in time savings, uniform interior heating, energy-efficiency, and selective heating, making it an alternative heating method to synthesis-activated carbon<sup>14,15</sup>. Compared with conventional electric heating, microwave pyrolysis can enhance thermo-chemical processes and promote certain chemical reactions<sup>16</sup>. Recently, extensive studies have focused on preparing activated carbon by chemical activation from biomass using one-step microwave pyrolysis<sup>9,17-19</sup>. So, it is considerably informative and environment-friendly to synthesis biomass-based activated carbon by microwave-assisted  $\text{H}_3\text{PO}_4$  activation.

In addition, to improve the adsorption affinities of activated carbon toward specific heavy-metal ions, modification by heteroatom [N, O, sulfur (S), *etc.*] doping into carbon structures has been proposed, and this has proven to be a desirable method<sup>20-26</sup>. Defective sites in or at the edges of a graphite layer can be replaced by heteroatoms to generate functional groups<sup>27</sup>. Hence, nitrification and reduction modification are used to modify resultant carbon samples to dope N/O functional groups which play a crucial role in efficiently coordinating with heavy metal to form complexing and ion-exchange<sup>28</sup>.

Based on the findings above, we present a protocol to synthesize N/O dual-doped mesoporous

carbon from biomass by chemical activation and two different pyrolysis methods followed up by modification. This protocol also determines which heating method favors the ensuing modification for doping of the N/O functional groups and, thus, enhancing the adsorption performance.

## **PROTOCOL:**

### **1. Preparation of Bagasse-based Activated Carbon**

#### **1.1. Preparation of the precursor for bagasse-based activated carbon**

1.1.1. Rinse the bagasse (obtained from a farm in Jiangsu, China) with deionized water and put the samples in a drying oven at 100 °C for 10 h.

1.1.2. Crush the dried bagasse with a grinder and sieve the powder through a 50-mesh sieve.

1.1.3. Place 30 g of fine bagasse powder into a 15 wt% phosphoric acid ( $\text{H}_3\text{PO}_4$ ) solution in a 1:1 weight ratio for 24 h. Dry the mixture in an oven at 105 °C for 6 h. Collect the resulting product as the precursor for bagasse-based activated carbon (BAC).

#### **1.2. Conventional electric-heating pyrolysis of the precursor**

1.2.1. Put 15 g of the precursor into a quartz boat and then insert the quartz boat into a quartz glass tube of an electric furnace.

1.2.2. Set the heating rate of the furnace at 5 °C min<sup>-1</sup> to carbonize the sample. When the temperature reaches 500 °C, keep the temperature for 90 min and then allow the resulting activated-carbon sample to cool to room temperature in nitrogen. Ensure a nitrogen flow of 80 mL min<sup>-1</sup> with a rotor flowmeter during the overall process.

1.2.3. Triturate and collect the electrical-furnace-pyrolyzed bagasse-based activated carbon (EBAC) in a beaker and then heat it in a vacuum drying oven at 105 °C for 24 h.

#### **1.3. Microwave pyrolysis of the precursor**

1.3.1. Put 15 g of the precursor in a microwave oven (with a 2.45 GHz frequency).

1.3.2. Set the power of the microwave oven at 900 W to pyrolyze the sample for 22 min, and ensure the nitrogen flow rate at 20 mL min<sup>-1</sup> with a rotor flowmeter. The air inlet of the rotor flowmeter is connected to a nitrogen cylinder using a hose, while the outlet is connected to the air inlet of the microwave oven.

1.3.3. Allow the resultant carbon to cool to room temperature in nitrogen. Triturate and collect the carbon sample in a beaker and then add 300 mL of hydrochloric acid (0.1 M). Stir the mixture

using a magnetic stirrer (at 200 rpm) for more than 12 h at room temperature.

1.3.4. Filter the carbon by filter paper with vacuum filtration and rinse the sample with deionized water until the pH value of the wash water is  $> 6$ . Dry the microwave-pyrolyzed bagasse-based activated carbon (MBAC) in a vacuum drying oven at  $105\text{ }^{\circ}\text{C}$  for 24 h.

## **2. Modification of Electrical-furnace-pyrolyzed Bagasse-based Activated Carbon and Microwave-pyrolyzed Bagasse-based Activated Carbon**

Note: The modification of the two samples was conducted according to the literature<sup>29</sup>.

### **2.1. Nitrification**

2.1.1. Mix 50 mL of concentrated sulfuric and 50 mL of concentrated nitric acids in a beaker at  $0\text{ }^{\circ}\text{C}$  (in an ice bath).

CAUTION: When the mixture of concentrated sulfuric acid and concentrated nitric acid is mixed, the concentrated sulfuric acid should be slowly added to the concentrated nitric acid and stirred with a glass rod and cooled in time.

2.1.2. Add 10 g of EBAC/MBAC to the mixed solution. Use a magnetic stirrer to stir the mixture for 120 min (at 200 rpm).

2.1.3. Filter the nitrified EBAC/MBAC by filter paper with vacuum filtration. Wash the carbon with deionized water until the wash water reaches pH 6, and then dry it in a drying oven at  $90\text{ }^{\circ}\text{C}$  for 24 h.

### **2.2. Reductive modification**

2.2.1. In a three-necked flask, add the 5.05 g of resulting product, 50 mL of deionized water, and 20 mL of ammonia solution (15 M). Stir this mixture for 15 min with a magnetic stirrer (at 200 rpm), then add 28 g of  $\text{Na}_2\text{S}_2\text{O}_4$ , and leave the mixture stirring at room temperature for 20 h.

2.2.2. Fit a reflux condenser to the flask and warm the mixture up to  $100\text{ }^{\circ}\text{C}$  using an oil bath. Add 120 mL of  $\text{CH}_3\text{COOH}$  (2.9 M) to the flask and allow the mixture to stir for 5 h with a magnetic stirrer (at 200 rpm) under reflux.

2.2.3. Remove the oil bath to allow the solution to cool down to room temperature. Filter the carbon sample and wash it with deionized water until the solution pH  $> 6$ . Dry the modified EBAC/MBAC at  $90\text{ }^{\circ}\text{C}$  and denote it as "EBAC-N/MBAC-N".

## **3. Adsorbent Characterization**

### **3.1. Structural characterization—Nitrogen adsorption/desorption isotherms**

3.1.1. Weigh an empty sample tube. Add a carbon sample (~0.15 g) to the sample tube.

3.1.2. Degas the sample at 110 °C for 5 h in a vacuum. Weigh the sample tube containing carbon. Calculate the weight of the carbon sample.

3.1.3. Install the sample tube into the test area of the surface-area and porosimetry analyzer using liquid nitrogen to measure it at -196 °C<sup>30</sup>.

## 3.2. Chemical characterization—Fourier transform infrared spectroscopy

3.2.1. Check the temperature and hygrometer and observe whether the environment meets the requirements: the temperature should be 16 - 25 °C and the relative humidity 20% - 50%.

3.2.2. Remove the desiccant and dust cover in the sample storehouse.

3.2.3. Dry the carbon sample and potassium bromide at 110 °C for 4 h to avoid the effect of water on the spectrum. Mix the carbon sample with potassium bromide and then use a press mechanism to prepare the test sample.

3.2.4. Put the sample in the test area and set the parameters of the software.

3.2.5. Save the spectra and take out the sample. Perform a required data processing for the spectra<sup>31</sup>.

## 4. Cu(II)-adsorption Experiments

### 4.1. Adsorption isotherm

4.1.1. Place 0.05 g of adsorbent in each of the conical flasks, which contain 25 mL of a CuSO<sub>4</sub> solution (pH 5) with a selected initial concentration (10, 20, 30, 40, 50, 60, 80, and 100 mg L<sup>-1</sup>). Use a 0.1 M HNO<sub>3</sub> and 0.1 M NaOH solution to adjust the pH of each copper solution.

Note: A solution with the selected initial concentration is diluted by a 1 g L<sup>-1</sup> CuSO<sub>4</sub> solution, which is made up of a dissolved 3.90625 g of blue vitriol solid using the vase with a 1,000 mL volume.

4.1.2. Fit lids on the conical flasks and put them in a thermostatic orbital shaker (with a stirring rate of 150 rpm) at 5 °C/25 °C/45 °C for 240 min.

4.1.3. Use 0.22 µm membrane filters to separate the adsorbents from the solution.

4.1.4. Use a flame atomic absorption spectrophotometry to determine the copper concentration of the filtrate.

Note: All experiments were carried out in triplicate and the data were averaged. The adsorption capacity for Cu(II),  $q_e$ , was calculated as follows:

$$q_e = \frac{V(C_0 - C_e)}{m} \quad (1)$$

Here,

$C_0$  = the initial copper concentration ( $\text{mg L}^{-1}$ ),

$C_e$  = the final concentration ( $\text{mg L}^{-1}$ ),

$V$  = the solution volume, and

$m$  = the weight of each adsorbent (g).

## 4.2. Influence of pH

4.2.1. Place 0.05 g of adsorbent in each of conical flasks, which contain 25 mL of a  $\text{CuSO}_4$  solution ( $40 \text{ mg L}^{-1}$ ) with a selected initial pH (2, 3, 4, 5, 6, and 7).

4.2.2. Fit lids on the conical flasks and put them in a thermostatic orbital shaker (with a stirring rate of 150 rpm) at  $25^\circ\text{C}$  for 24 h to reach adsorption equilibrium conditions.

4.2.3. Repeat step 4.1.3-4.1.4.

## 4.3. Adsorption kinetics

4.3.1. Place 0.25 g of adsorbent in a beaker which contains 125 mL of a  $\text{CuSO}_4$  solution ( $30 \text{ mg L}^{-1}$  or  $100 \text{ mg L}^{-1}$ , pH 5) in a  $25^\circ\text{C}$  water bath with magnetic stirring (at 200 rpm).

4.3.2. Use pipettes to draw 5 mL of the solution when the contact time reaches 0.5, 1, 2.5, 5, 10, 30, 60, 120, and 180 min.

4.3.3. Repeat step 4.1.3-4.1.4.

## REPRESENTATIVE RESULTS:

Nitrogen adsorption/desorption isotherms of four samples are presented in **Figure 1**. All adsorption isotherms show a rapid increase in low  $P/P_0$  region and these isotherms belong to type IV (IUPAC classification) demonstrating their pore structure that consists of micropores and dominant mesopores<sup>32</sup>.

The surface physical parameters for all samples obtained from the nitrogen adsorption isotherms are shown in **Table 1**. Microwave pyrolysis and modification both contribute to a smaller Brunauer–Emmett–Teller (BET) surface area and total pore volume, changing the physical morphology of the samples.

Fourier transform infrared (FTIR) spectra of the four samples are given in **Figure 2**. Bands of MBAC at 1167 cm<sup>-1</sup> [carbon (C) - O stretching vibration], 1620 cm<sup>-1</sup> (C = O stretching vibration), 2852 cm<sup>-1</sup> [N - hydrogen (H) stretching vibration], 2922 cm<sup>-1</sup> (C - H stretching vibration), and 3442 cm<sup>-1</sup> (O - H stretching vibration) are more intense than EBAC. These may be attributed to the microwave pyrolysis contributing more oxygen functional groups to the BAC surface. For EBAC-N and MBAC-N, bands around 1573 cm<sup>-1</sup> and 1400 cm<sup>-1</sup> likely represent C = N and N - H groups, respectively. It can be found that the modified carbon materials have obtained distinct nitrogen/oxygen functional groups, and the microwave-pyrolyzed carbon gets more, which is in accordance with the elemental analysis as shown in **Table 1**. It can be speculated that microwave pyrolysis is more adequate to activate the precursor and lay root for further modifications than conventional electric-heating pyrolysis. MBAC-N possesses mainly hydroxyl, carboxyl, amino, and imine functional groups.

**Figure 3** shows the adsorption capacity of the four samples under different pH conditions. The four adsorbents reach the optimal adsorption capacity at pH 5, so the following adsorption experiments are all carried out at pH 5. The samples prepared by microwave pyrolysis exhibited better Cu(II) adsorption capacity before and after the modification, although they had a lower specific surface area and pore volume. In general, the adsorbability of adsorbents depends on the pore structure and surface functional groups. Therefore, the high adsorption capacity of MBAC-N is attributed to more abundant N/O surface groups. The results confirm that the microwave pyrolysis benefits the follow-up introduction of surface functional groups to improve the adsorption capacity more than electric-heating pyrolysis.

The adsorption isotherms of MBAC-N on Cu(II) at 5 °C, 25 °C, and 45 °C are shown in **Figure 4a**. The adsorption properties of samples for Cu(II) become better when the temperature increases. By comparing the isotherm parameters in **Table 2**, it is clear that the Langmuir isotherm model indicates a higher linear correlation coefficient ( $R^2$ ) which is over 0.99 (the fitting line in **Figure 4b**), and the measured adsorption capacity ( $q_{mea}^0$ ) is identical with the calculated one ( $q_{cal}^0$ ). Therefore, the model is more suitable than the Freundlich and Temkin isotherm models, which indicates that the absorption of Cu(II) is a chemical adsorption process<sup>33</sup>.

As shown in **Figure 4c**, MBAC-N can reach about 75% of Cu(II) equilibrium adsorption capacity within 15 min, and it can almost reach the adsorption equilibrium of Cu(II) in about 50 min at different initial concentrations. These prove that MBAC-N has excellent adsorption properties. As can be seen from **Table 3**, the pseudo-second-order model is better than the Lagergren and Elovich models with  $R^2 = 0.999$  (the fitting line in **Figure 4d**). The above results confirm that the adsorption of Cu(II) on MBAC-N is chemisorption. Hence, the chemical interaction mechanism of Cu(II) by the modified carbon is proposed in **Figure 5**. **Table 4** compares the adsorption capacity of Cu(II) by biomass-based activated carbon reported in recent references<sup>34-38</sup>. It is found that MBAC-N has a higher adsorption capacity than other adsorbents reported in the literature, demonstrating it as a promising adsorbent for removing Cu(II).

**FIGURE & TABLE LEGENDS:**



**Figure 1: Nitrogen adsorption/desorption isotherms of carbons.** The inset graph in **Figure 1** shows the nitrogen adsorption/desorption isotherm of MBAC-N in a smaller ordinate range. The data were obtained from the supporting software of the surface-area and porosimetry analyzer. This figure has been modified from Wan and Li<sup>27</sup>.

**Figure 2: FTIR spectra of EBAC, EBAC-N, MBAC, and MBAC-N.** The spectra can confirm the chemical compositions and surface functional groups of the samples. This figure has been modified from Wan and Li<sup>27</sup>.

**Figure 3: Effect of solution pH on Cu(II) adsorption.** The concentration of copper in the solutions is 40 mg L<sup>-1</sup>. The test is conducted at 25 °C and at 150 rpm for 24 h, to reach adsorption equilibrium. This figure has been modified from Wan and Li<sup>27</sup>.

**Figure 4: Representative adsorption property analysis of MBAC-N.** (a) This panel shows the adsorption isotherms of Cu(II) on MBAC-N at 5 °C, 25 °C, and 45 °C. (b) This panel shows the fitting result for copper adsorption by using the Langmuir isotherm. (c) This panel shows the kinetics of Cu(II) on MBAC-N at the initial concentrations of 30 mg L<sup>-1</sup> and 100 mg L<sup>-1</sup>. (d) This panel shows the fitting result for copper adsorption at 25 °C by using the Pseudo-second-order model. This figure has been modified from Wan and Li<sup>27</sup>.

**Figure 5: Proposed mechanism for Cu(II) adsorption by modified carbon.** In this reaction process, the chemical adsorption mainly involves ion exchange and complexing.

**Table 1: Structural characteristics and elemental compositions of EBAC, EBAC-N, MBAC, and MBAC-N.** The textural data are analyzed using the BET method. The relative weight percentage of the elements is calculated based on the dry ash-free basis. This table has been modified from Wan and Li<sup>27</sup>.

**Table 2: Isotherm parameters of Cu(II) on MBAC-N at different temperatures.** The fitted parameters are from linearized Langmuir, Freundlich, and Temkin adsorption models. This table has been modified from Wan and Li<sup>27</sup>.

**Table 3: Kinetic parameters of Cu(II) on MBAC-N at different initial concentrations.** The fitted parameters are from linearized Lagergren, Pseudo-second-order, and Elovich models. This table has been modified from Wan and Li<sup>27</sup>.

**Table 4: Comparison of the adsorption capacity of Cu(II) on different adsorbents.** The ability of activated carbon to remove Cu(II) is significantly affected by the pH of the solution, so the adsorption capacity of the contrast biomass-based carbon materials should be obtained close to pH 5.

## DISCUSSION:

In this protocol, one of the critical steps is the successful preparation of mesoporous carbon with better physicochemical properties by the one-step approach, where optimal experimental

conditions need to be determined. So, in a previous study<sup>28</sup>, we have carried out orthogonal array microwave pyrolysis experiments, considering the effect of the impregnation ratio of bagasse and phosphoric acid, pyrolysis time, microwave oven power, and drying time. Besides, great care must be taken in tedious Cu(II)-adsorption experiments, especially when the pH value of the solution is adjusted, because the pH value has a great influence on Cu(II) removal by activated carbon (**Figure 3**). It is imperative to test the actual copper concentration of the CuSO<sub>4</sub> solution with a defined initial concentration and use this value as C<sub>0</sub> in Equation (1).

A larger specific surface area and higher pore volume of biomass-based activated carbon can be obtained by chemical activation. However, the specific surface area and total pore volume both decrease during the subsequent pyrolysis and modification process, which is likely due to the collapse and blockage of the pores<sup>27</sup>, resulting in a reduction of the adsorption capacity. Therefore, further work is required to prepare biomass-based mesoporous carbon with both a high surface area and abundant functional groups.

Microwave pyrolysis is verified to more adequately synthesize biomass-based mesoporous carbon with a higher nitrogen/oxygen-chelating adsorption for Cu(II), which has many advantages over widely used conventional heating methods. However, it is not possible to control the instantaneous temperature accurately during the microwave pyrolysis process. Biomass is a good microwave absorption material, whose temperature can rapidly increase under the effect of a microwave. Clearly, future work needs to examine how the pyrolysis temperature affects the physicochemical properties of the biomass-based carbon.

A detailed description of the modification mechanism is beyond the scope of this article, but it can be found in earlier published literature<sup>27</sup>. The potential importance of nitrification and reduction modification which can effectively introduce more N/O functional groups concurrently on the surface of carbon samples is worth appreciating. However, the modification process contains numerous experimental steps and the utilization of dangerous concentrated strong acid. A simpler and more effective nitrogen/oxygen modification method may be tested and adopted in further studies.

We have demonstrated an environment-friendly energy-efficient method for preparing biomass-based mesoporous carbon by microwave pyrolysis and dope N/O groups simultaneously on the carbon using a nitrification and reduction route. Such N/O dual-doped activated carbon owns a higher adsorption capacity of heavy-metal ions in an aqueous solution, which is applicable for wastewater remediation. We expect that this protocol will provide ideas for the rapid preparation of high-adsorptive carbon from biomass by time-saving, effective microwave pyrolysis and will be optimized in the future.

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#### **DISCLOSURES:**

The authors have nothing to disclose.

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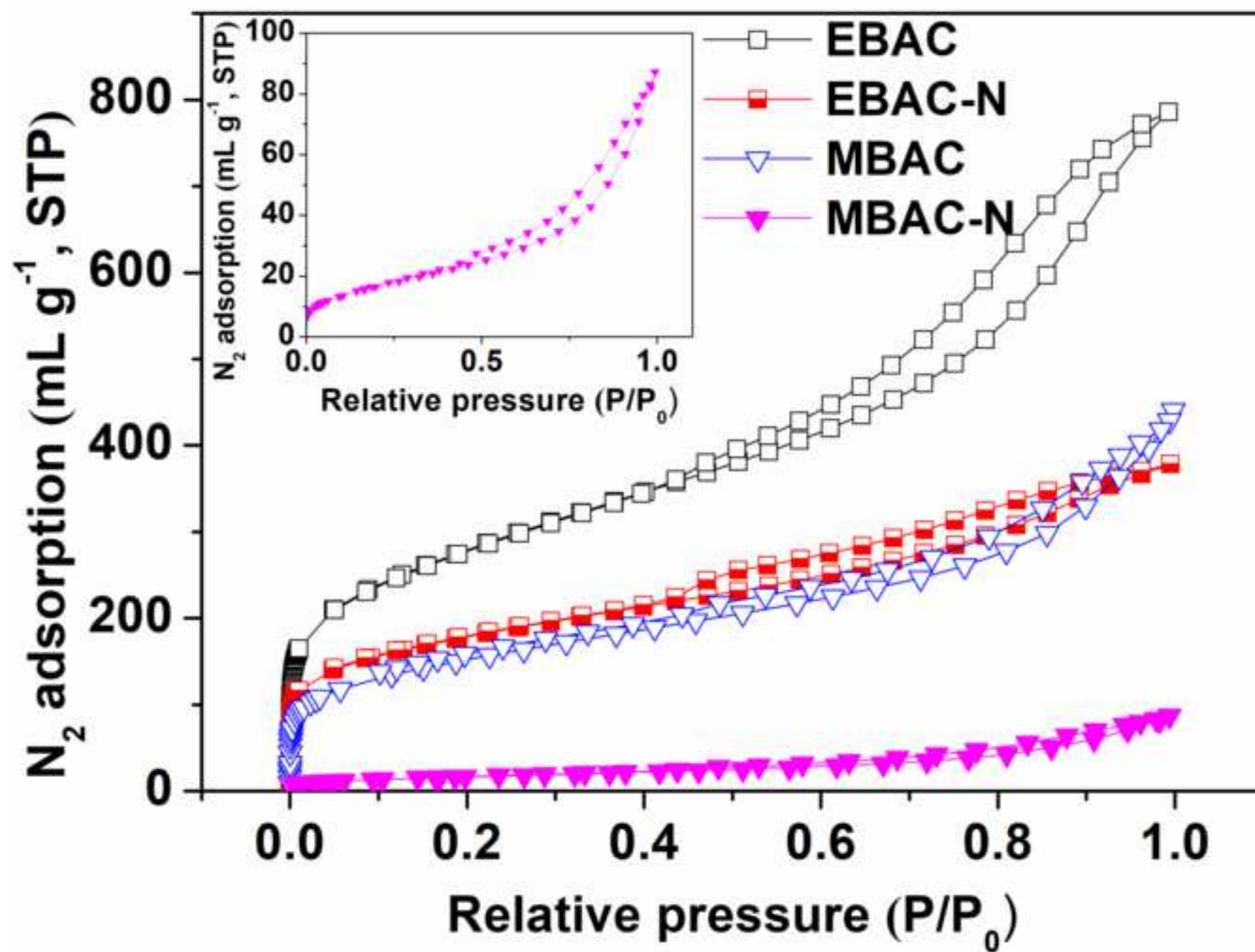
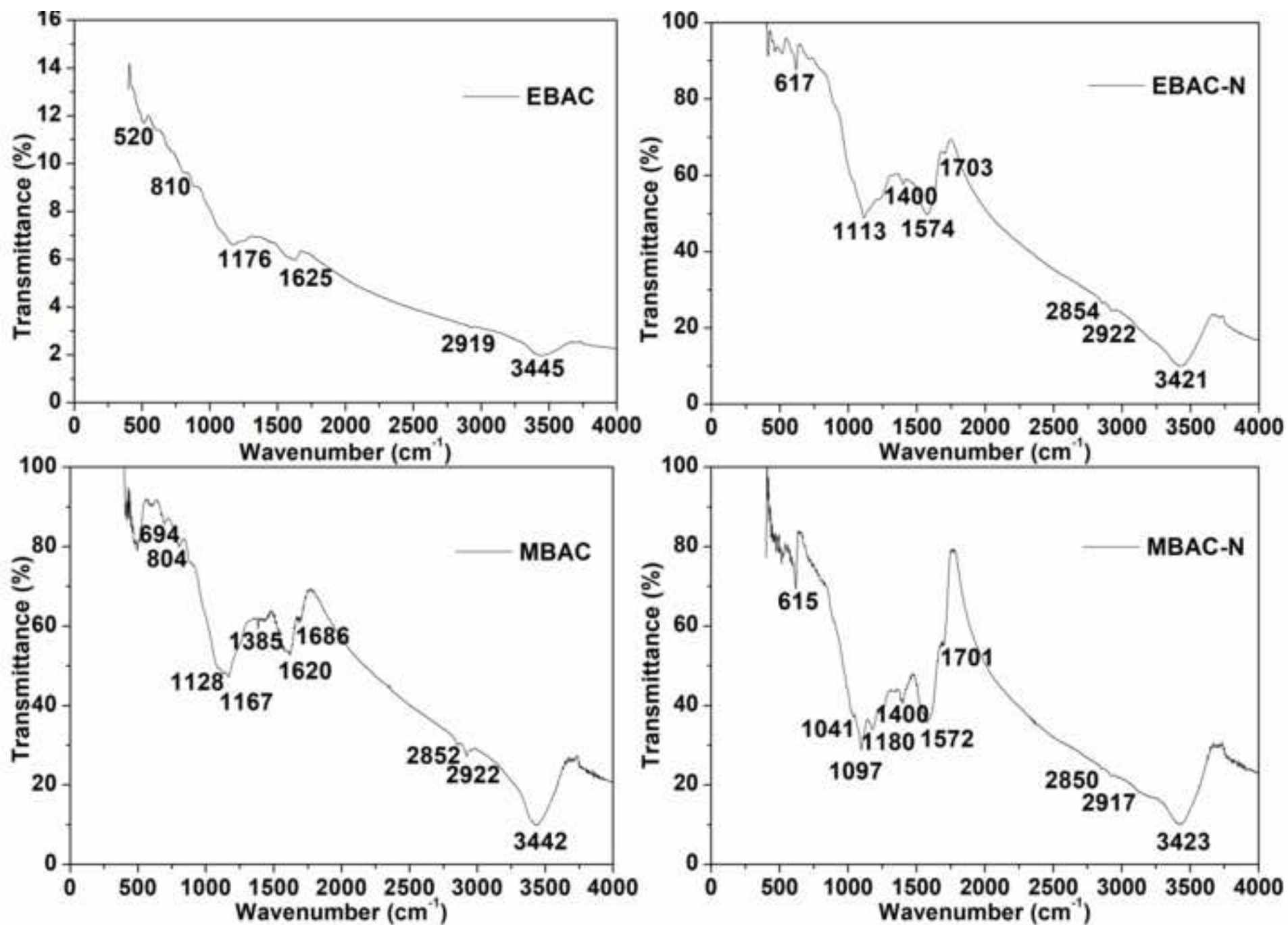


Figure2





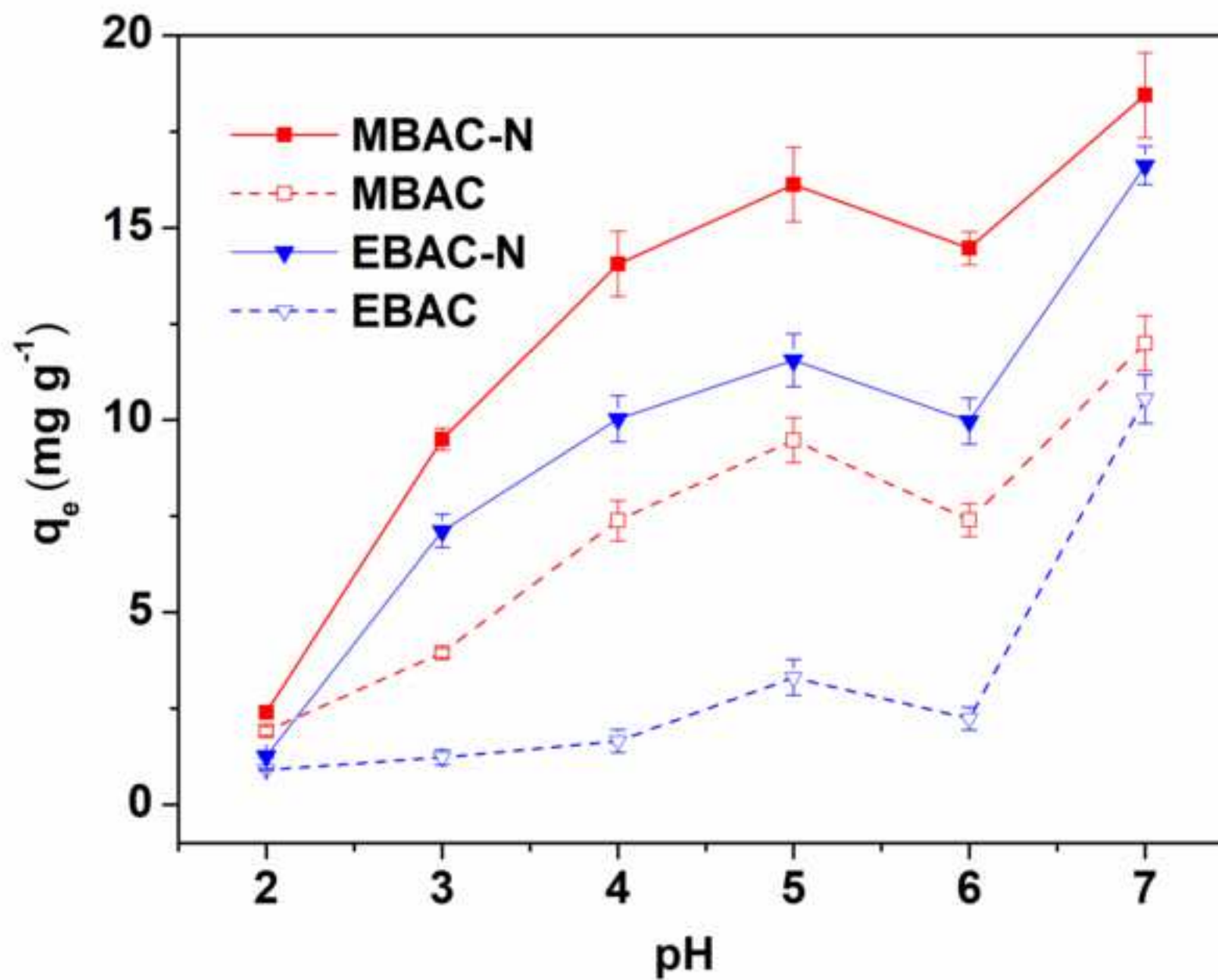
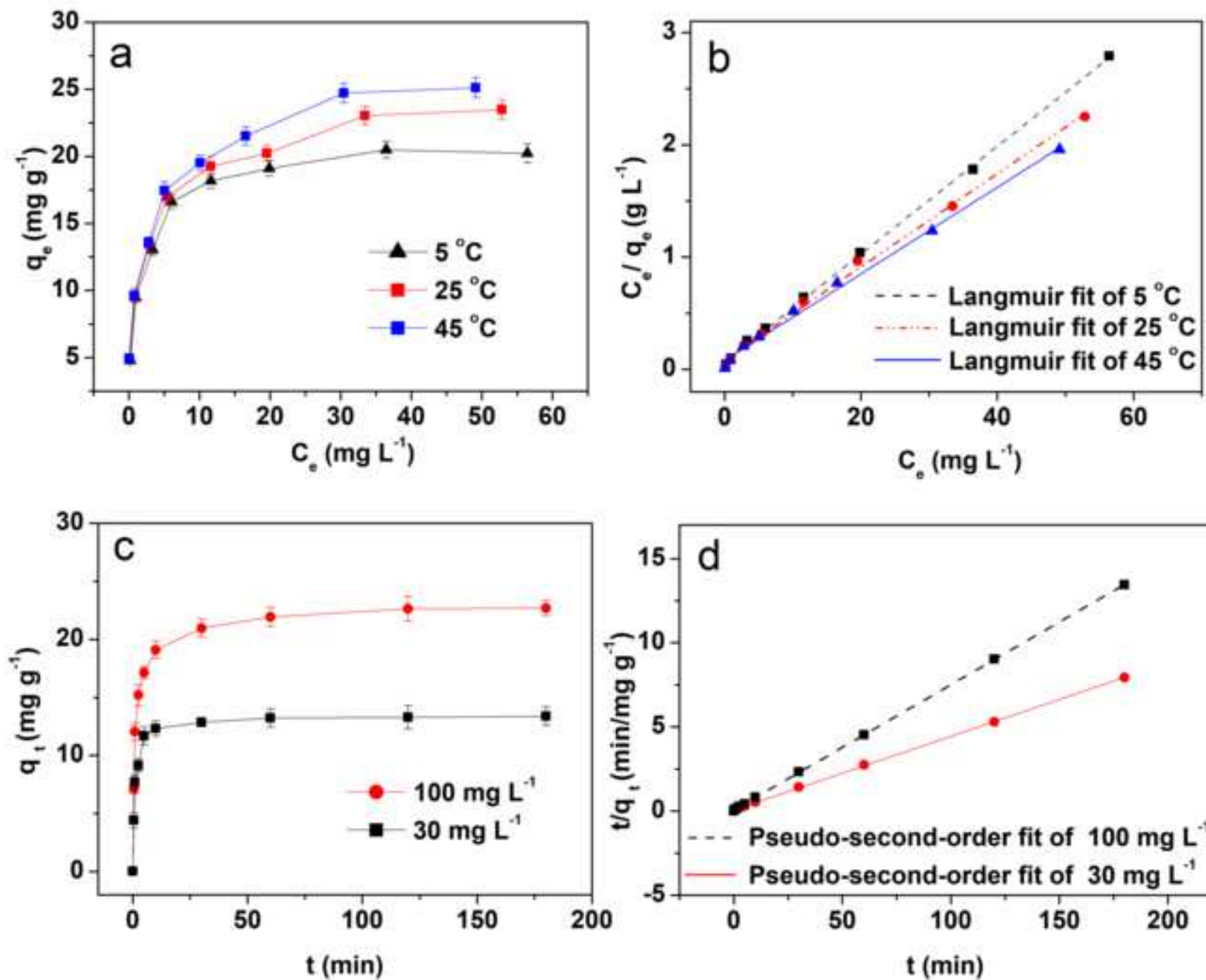
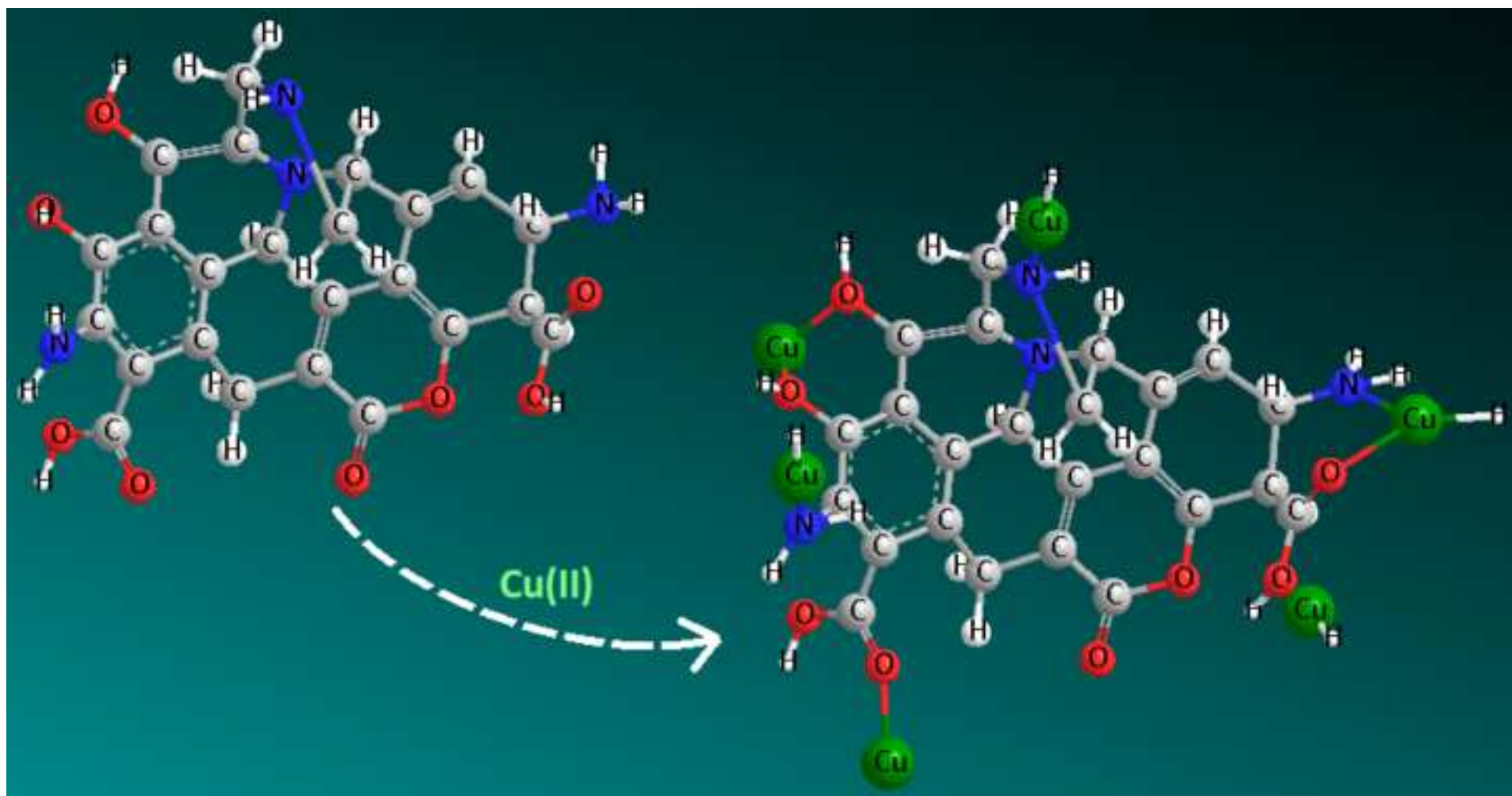


Figure4

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**Table 1.**

Adsorbents	EBAC	EBAC-N	MBAC
Pore structure parameters			
BET surface area (m <sup>2</sup> g <sup>-1</sup> )	978	609	543
Total pore volume (cm <sup>3</sup> g <sup>-1</sup> )	1.22	0.59	0.68
Mesoporous volume (cm <sup>3</sup> g <sup>-1</sup> )	1.09	0.47	0.58
Mean pore size D <sub>p</sub> (nm)	4.97	3.84	5.01
Mesoporous rate (%)	89.52	80.24	85.32
Elemental content (wt%)			
C	92.23	79.31	87.28
H	1.76	1.26	1.65
N	0.08	4.01	0.58
O	5.82	15.15	10.33
S	0.11	0.27	0.16
Yield (%)	53.35	/	57.23

---

MBAC-N

---

61

0.13

0.11

8.89

84.61

72.44

1.12

5.52

20.54

0.38

/

---

Table 2.

Isotherm models	Parameters	MBAC-N		
		5 °C	25 °C	45 °C
Langmuir	$q_{cal}^0$ (mg g <sup>-1</sup> )	20.82	24.09	25.97
	$q_{mea}^0$ (mg g <sup>-1</sup> )	20.23	23.47	25.12
	b (L mg <sup>-1</sup> )	0.73	0.51	0.49
	R <sup>2</sup>	0.999	0.996	0.995
Freundlich	K <sub>F</sub> (L mg <sup>-1</sup> )	8.802	9.65	10.56
	n	3.937	3.902	4.032
	R <sup>2</sup>	0.907	0.967	0.987
Temkin	A <sub>T</sub> (L mg <sup>-1</sup> )	29.57	32.3	49.8
	B (L mg <sup>-1</sup> )	2.94	3.19	3.16
	R <sup>2</sup>	0.969	0.985	0.955

Table 3.

Kinetic models	Parameters	MBAC-N	
		30 mg L <sup>-1</sup>	100 mg L <sup>-1</sup>
Lagergren	k <sub>1</sub> (min <sup>-1</sup> )	0.037	0.045
	R <sup>2</sup>	0.714	0.934
	q <sub>e,mea</sub> (mg g <sup>-1</sup> )	13.39	22.69
Pseudo-second-order	q <sub>e,cal</sub> (mg g <sup>-1</sup> )	13.44	23.25
	k <sub>2</sub> (g (mg min) <sup>-1</sup> )	0.08676	0.03031
	R <sup>2</sup>	0.999	0.999
	q <sub>e,mea</sub> (mg g <sup>-1</sup> )	13.39	22.69
Elovich	α <sub>E</sub> (g (mg min) <sup>-1</sup> )	379.73	312.25
	β <sub>E</sub> (mg g <sup>-1</sup> )	0.738	0.411
	R <sup>2</sup>	0.799	0.901

**Table 4.**

Adsorbents	pH	q <sub>e</sub> (mg g <sup>-1</sup> )	References
Wood-based granular activated carbon	5.5	6.016	34
Baobab fruit shell-derived activated carbon	6	3.0833	35
Olive stone AC (COSAC)	5	17.08	36
Activated carbonfrom date stones	5.5	18.68	37
Walnut shell based activated carbon	5	9.3	38
Plasma modified activated carbon		21.4	
MBAC-N	5	25.12	This study



Name of Material/ Equipment	Company
All chemicals and reagents (phosphoric acid, Nanjing Chemical Reagent Co., Ltd	
Electric furnace	Luoyang Bolaimaite Experiment Electric Furnace C
Microwave oven	Nanjing Yudian Automation Technology Co., Ltd
Surface-area and porosimetry analyzer	Beijing Gold APP Instrument Co., Ltd
Fourier transform infrared (FTIR) spectrome	Nicolet
Flame atomic absorption spectrophotometry	Beijing Purkinje General Instrument Corporation
Element Analyzer	Germany Heraeus Co.

Catalog Number	Comments/Description
o., Ltd	Analytical grade
	2.45 GHz frequency
	Vc-Sorb 2800TP
	6700
	A3
	CHN-O-RAPID



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Institution: Nanjing Agricultural University  
Article Title: Preparation of biomass-based mesoporous carbon with higher nitrogen/oxygen-debating adsorption for Cu(II) through microwave pre-pyrolysis  
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Dear editor and reviewers:

Thank you very much for the comments on the manuscript “Preparation of biomass-based mesoporous carbon with higher nitrogen/oxygen-chelating adsorption for Cu(II) through microwave pre-pyrolysis”. We find the reviewers’ comments most helpful and genuinely appreciate your time and effort. We have made serious revision and explanation as recommended by the reviewers. Modified places are marked in red. We also deleted or corrected some words. We hope the revised paper could be in accordance with the instructions of JoVE.

### **Response beneath each comment**

#### **Editorial comments:**

1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues. The JoVE editor will not copy-edit your manuscript and any errors in the submitted revision may be present in the published version.

**Response: The manuscript has been proofed carefully.**

2. Unfortunately, there are a few sections of the manuscript that show overlap with previously published work. Though there may be a limited number of ways to describe a technique, please use original language throughout the manuscript. Please see lines: 37-41, 52-61, 68-71, 166-167, 189-191, 218-220.

**Response: The statement on Line 52-61 (in original manuscript) has been deleted and replaced with other content according to the suggestion of reviewer 3#. Lines 37-41, 68-71, 166-167, 189-191, 218-220 (in original manuscript) have been revised to original language.**

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**Response: Each Table has been uploaded individually as an .xls or .xlsx file.**

6. Figure 1: Please explain the inset in its figure legend.

**Response: Revised as the editor's advice.**

7. Please provide an institutional email address for each author if possible.

**Response: We have changed the original email address to an institutional email address for each author in the revised manuscript.**

8. Please rephrase the Short Abstract to clearly describe the protocol and its applications in complete sentences between 10-50 words: "Here, we present a protocol to ..."

**Response: Revised as the editor's advice.**

9. Please revise the Long Abstract to focus on the method being presented rather than the results of a specific experiment. Include a statement about the purpose of the method. A more detailed overview of the method and a summary of its advantages, limitations, and applications is appropriate. Please focus on the general types of results acquired.

**Response: Revised as the editor's advice.**

10. Please revise the Introduction to include all of the following:

- a) A clear statement of the overall goal of this method
- b) The rationale behind the development and/or use of this technique
- c) The advantages over alternative techniques with applicable references to previous studies
- d) A description of the context of the technique in the wider body of literature



e) Information to help readers to determine whether the method is appropriate for their application

**Response: Revised as the editor's advice.**

11. Please add more details to your protocol steps. Please ensure you answer the “how” question, i.e., how is the step performed? Alternatively, add references to published material specifying how to perform the protocol action.

**Response: More details have been added to protocol steps.**

12. 1.2.2: Is 90 min the holding time at 500 °C, or the total time that the precursor stays in the furnace? Please specify.

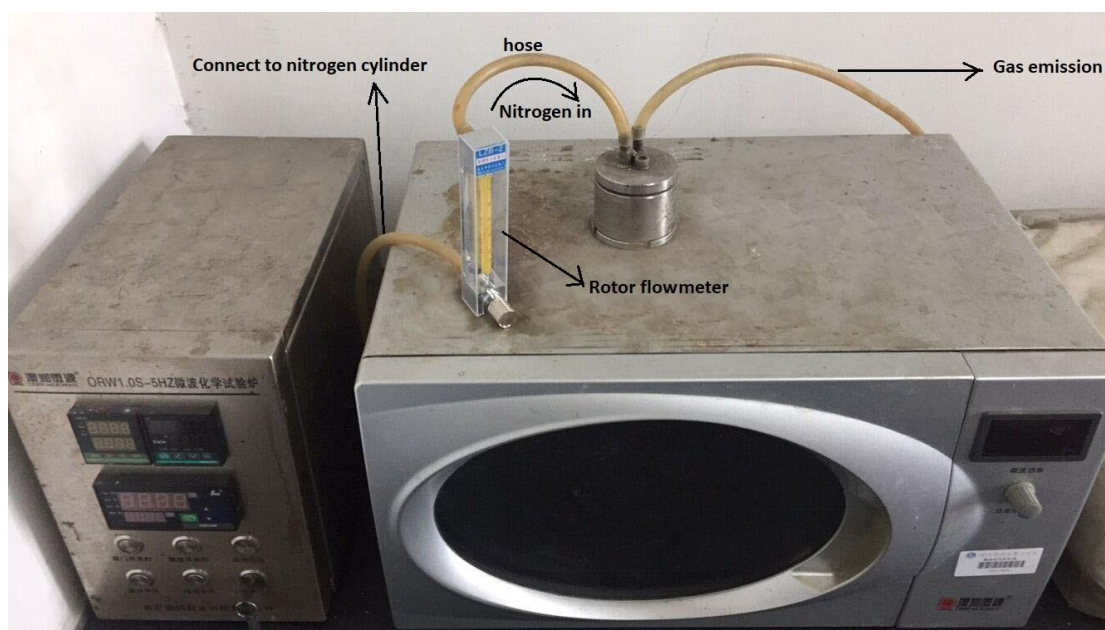
**Response: 90 min is the holding time. Relative protocol steps have been revised.**

13. 1.2.3: Please change “store” to “heat” as the temperature in the oven is relatively high (105 °C).

**Response: Revised as the editor's advice.**

14. 1.3.2: Please describe how the nitrogen line is connected to the microwave and how the flow rate is controlled.

**Response: The N<sub>2</sub> flow rate is controlled with a rotor flowmeter. The air inlet of the rotor flowmeter is connected to a nitrogen cylinder using a hose while the outlet is connected to the air inlet of microwave oven.**



15. 1.3.3: Please describe how phosphates are removed, presumably by decanting the



water? Is a filter or centrifuge used to separate the solid from the liquid?

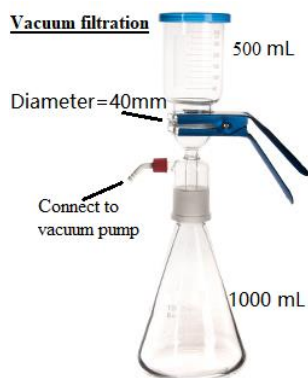
**Response:** Actually, phosphates are removed in step 1.3.4 “Filter the carbon by filter paper with vacuum filtration and rinse the sample with deionized water until...”. In order to disambiguate, the statement in step 1.3.3 “to remove phosphates (byproducts of  $\text{H}_3\text{PO}_4$  with bagasse)” has been removed.

16. Please include a space between all numbers and their corresponding units: 150 rpm; etc.

**Response:** Revised as the editor’s advice.

17. 1.3.4: Please describe how this is actually done. What is the filter size? Drying in the oven for how long?

**Response:** Step 1.3.4 has been revised. Drying in the oven for 12 h. Filter the carbon by filter paper with vacuum filtration (as shown in the picture). In fact, the purpose of filtration is to separate the carbon from the liquid, so the size of



the filter is not a factor.

18. 2.1.1: Is this done in a hood? If so, please specify.

**Response:** Without a hood.

19. 2.1.2/2.2.1/2.2.2: What is the stirring speed?

**Response:** All steps related to stirring have been revised.

20. 2.1.3: What is the filter size?

**Response:** Filter the carbon by filter paper with vacuum filtration (See picture in comment 17) In fact, the purpose of filtration is to separate the carbon from the liquid, so the size of the filter is not a factor.

21. 2.1.4: What is considered as a neutral pH, over 6 or 7?

**Response:** The statement “a neutral pH” has been changed to “pH 6”.

22. 2.2.3: Please specify the solution pH.

**Response: The statement “pH was constant” has been changed to “pH over 6”.**

23. 3.1.3: Please add more details here or include a reference.

**Response: We add a reference to step 3.1.3.**

24. 3.2.3: At what temperature and for how long are the carbon samples and potassium bromide dried?

**Response: The statement “fully” has been changed to “at 110 °C for 4 h”.**

25. 3.2.4: Please specify the parameters if applicable.

**Response: The parameters are not applicable. Besides, we add a reference to step 3.2.4.**

26. As we are a methods journal, please revise the Discussion to explicitly cover the following in detail in 3-6 paragraphs with citations:

- a) Critical steps within the protocol
- b) Any modifications and troubleshooting of the technique
- c) Any limitations of the technique
- d) The significance with respect to existing methods
- e) Any future applications of the technique

**Response: Revised as the editor’s advice.**

27. Please do not abbreviate journal titles. Please include volume and issue numbers for all references.

**Response: Revised as the editor’s advice.**

## Reviewer 1

Comments: The manuscript describes a GOOD work AND is well presented. Authors need following points to be included before reconsideration.

**Comment 1:** Abstract should contain some quantitative information also.

**Response:** Thank you very much for your suggestion. According to the editor's advices, the long abstract should focus on the method and its purpose *etc.*, as JoVE is a methods journal. The abstract has been revised as editor's advices, so it may not contain some quantitative information.

**Comment 2:** English must be improved.

**Response:** Thank you very much for your suggestion. The spelling and grammar issues have been proofed.

**Comment 3:** Novelty of the work be established.

**Response:** We have revised the ABSTRACT and INTRODUCTION seriously. It can be found that the novelty of the work is to present a protocol to synthesis nitrogen/oxygen dual-doped mesoporous carbon from biomass by chemical activation and two different pyrolysis methods followed up by modification. We demonstrate that the microwave pyrolysis benefits the subsequent modification process to simultaneously introduce more nitrogen/oxygen functional groups on the carbon.

**Comment 4:** All the adsorbents reported be compared in a tabular form to establish the superiority of the work.

**Response:** Revised as the reviewer's advice. The following content is added in the revised manuscript as well as the comparison table (Table 4).

Table 4 compares the adsorption capacity of Cu(II) by the recently reported biomass-based activated carbon. It is found that MBAC-N has a higher adsorption capacity than other adsorbents reported in the literature, demonstrating it a promising adsorbent for removing Cu(II).

**Table 4.**

Adsorbents	pH	$q_e$ (mg g <sup>-1</sup> )	References
Wood-based granular activated carbon	5.5	6.016	Loganathan et al., 2018

Baobab fruit shell-derived activated carbon	6	3.0833	Vunain et al., 2017
Olive stone AC (COSAC)	5	17.08	Bohli et al., 2017
Activated carbon from date stones	5.5	18.68	Bouhamed et al., 2016
Walnut shell based activated carbon	5	9.3	Wu et al., 2018
Plasma modified activated carbon		21.4	
MBAC-N	5	25.12	This study

Bohli T, Ouederni A, Villaescusa I. Simultaneous adsorption behavior of heavy metals onto microporous olive stones activated carbon: analysis of metal interactions[J]. Euro-Mediterranean Journal for Environmental Integration, 2017, 2(1):19.

Bouhamed F, Elouear Z, Bouzid J, et al. Multi-component adsorption of copper, nickel and zinc from aqueous solutions onto activated carbon prepared from date stones[J]. Environmental Science & Pollution Research, 2016, 23(16):1-6.

Loganathan P, Shim W G, Sountharajah D P, et al. Modelling equilibrium adsorption of single, binary, and ternary combinations of Cu, Pb, and Zn onto granular activated carbon[J]. Environmental Science & Pollution Research, 2018(15):1-12.

Vunain E, Kenneth D, Biswick T. Synthesis and characterization of low-cost activated carbon prepared from Malawian baobab fruit shells by H<sub>3</sub>PO<sub>4</sub> activation for removal of Cu(II) ions: equilibrium and kinetics studies[J]. Applied Water Science, 2017, 7(8):4301-4319.

Wu L, Wan W, Shang Z, et al. Surface modification of phosphoric acid activated carbon by using non-thermal plasma for enhancement of Cu(II) adsorption from aqueous solutions[J]. Separation & Purification Technology, 2018, 197.

**Comment 5:** Following refs. must be included in the Introduction part.

Materials Science and Engineering C ;Volume 32, Issue 1, 1 January 2012, Pages 12-17

Environmental Science and Pollution Research ;Volume 19, Issue 4, May 2012, Pages 1224-1228

Waste Management ;Volume 17, Issue 8, 1998, Pages 517-522

Journal of Colloid and Interface Science;Volume 342, Issue 2, 15 February 2010, Pages 518-527

Journal of Colloid and Interface Science ;Volume 337, Issue 2, 15 September 2009, Pages 345-354

Journal of Colloid and Interface Science ;Volume 340, Issue 1, 1 December 2009, Pages 16-26

Journal of Hazardous Materials ;Volume 185, Issue 1, 15 January 2011, Pages 17-23

RSC Advances ;Volume 2, Issue 16, 14 August 2012, Pages 6380-6388

Journal of Colloid and Interface Science ;Volume 344, Issue 2, 15 April 2010, Pages 497-507

Materials Science and Engineering C;Volume 31, Issue 5, 20 July 2011, Pages 1062-1067

Chemical Engineering Journal ; 180, 15 January 2012, Pages 81-90

Journal of Colloid and Interface Science ;Volume 371, Issue 1, 1 April 2012, Pages 101-106

Journal of Hazardous Materials ;Volume 183, Issue 1-3, November 2010, Pages 402-409

J. Mol. Liquids, 173 (2012) 153-163.

2003 Separation Science and Technology 38 (2) , pp. 463-481  
 Environmental Engineering Research, 20(1) (2015) 001-018.  
 Adv. Colloid Interface Sci, 211 (2014) 92-100.  
 Adv. Colloid Interface Sci., 193-194 (2013) 24-34.  
 Journal of Molecular Liquids 221 (2016) 1029-1033.  
 Journal of Molecular Liquids 221 (2016) 930-941.  
 Scientific Reports 6 (2016) 31641.  
 Journal of Saudi Chemical Society 19(5) (2015) 521-527.  
 Journal of Molecular Liquids 209 (2015) 374-380  
 RSC Advances 5 (2015) 34645-34651.  
 Journal of Colloid and Interface Science 452 (2015) 126-133.  
 Journal of the Taiwan Institute of Chemical Engineers 45 (2014) 1910-1917.  
 Journal of Molecular Liquids 198 (2014) 409-412.  
 Journal of Molecular Liquids 193 (2014) 160-165  
 Materials Science and Engineering C 33 (2013) 2235-2244.  
 Journal of Molecular Liquids 181 (2013) 133-141.  
 Materials Science and Engineering C 33 (2013) 4725-4731.  
 Materials Science and Engineering: C 33 (2013) 91-98.  
 Journal of Molecular Liquids 177 (2013) 394-401.  
 Journal of Molecular Liquids 178 (2013) 88-93.  
 Environmental Science and Pollution Research 20 (5), 2828-2843(2013)  
 RSC advances 2 (22), 8381-8389(2012)  
 Journal of Colloid and interface Science 362 (2), 337-344(2011)  
 Encyclopedia of Surface and Colloid Science, 2004 Update Supplement 5, 1, 2004  
 RSC advances 2 (22), 8381-8389, 2012  
 Journal of Colloid and interface Science 362 (2), 337-344 , 2011  
 Materials Science and Engineering: C 33 (1), 91-98, 2013  
 Industrial & Engineering Chemistry Research 50 (24), 13589-13613, 2011  
 Journal of colloid and interface science 362 (2), 457-462 , 2011  
 Advances in colloid and interface science 211, 93-101, 2014  
 Separation and purification technology 89, 245-251, 2012  
 J. Mol. Liquids 173(2012)153-163.  
 International Journal of Environmental Analytical Chemistry 84, 947-964 (2004).  
 Environmental Science and Pollution Research 21, 3218-3229 (2014).  
 Journal of Molecular Liquids 219, 858-864 (2016).  
 Journal of Molecular Liquids 224, 171-176 (2016).  
 Journal of Molecular Liquids 211, 457-465 (2016).  
 Materials Science and Engineering: C, 33( 2013) 2235-2244.  
 J. Colloid Interface Sci., 452 (2015) 126-133.  
 Chem Eng J. 284 (2016) 687-697.  
 RSC Adv., 5 (2015) 34645- 34651.  
 J. Mol. Liq., 177 (2013) 394-401.  
 Chemical Eng. J., 268(2015) 28-37.  
 J Colloids Surface Sci., 417 (2014) 420-430.

Materials Science & Engineering C 33 (2013)4725-4731.18438 - 18450.  
Materials Science and Engineering C Volume 33, Issue 4, 1 May 2013, Pages 2235-2244  
Journal of Colloid and Interface Science Volume 452, August 05, 2015, Pages 126-133  
Chemical Engineering Journal Volume 284, January 15, 2016, Pages 687-697  
Journal of Molecular Liquids Volume 177, January 2013, Pages 394-401  
RSC Adv., 5 (2015) 18438 - 18450.

**Response: Thank you very much for your suggestion. We have learned a lot from the literatures you have provided. Taking into account the length of the paper and the content relevance of these references, twelve most relevant references have been cited in the revised manuscript, as follows:**

**Journal of colloid and interface science 362 (2), 457-462, 2011**

Adsorption process of methyl orange dye onto mesoporous carbon material–kinetic and thermodynamic studies

**Advances in colloid and interface science 211, 93-101, 2014**

Processing methods, characteristics and adsorption behavior of tire derived carbons: A review

**RSC Adv., 5 (2015) 18438 - 18450.**

Removal of basic dye Auramine-O by ZnS:Cu nanoparticles loaded on activated carbon: optimization of parameters using response surface methodology with central composite design

**Environmental Science and Pollution Research 20 (5), 2828-2843(2013)**

Sorption of pollutants by porous carbon, carbon nanotubes and fullerene- An overview

**Environmental Science and Pollution Research; Volume 19, Issue 4, May 2012, Pages 1224-1228**

Column with CNT/magnesium oxide composite for lead(II) removal from water

**Industrial & Engineering Chemistry Research 50 (24), 13589-13613, 2011**

Rice Husk and Its Ash as Low-Cost Adsorbents in Water and Wastewater Treatment

**Journal of Colloid and Interface Science ;Volume 340, Issue 1, 1 December 2009, Pages 16-26**

Adsorptive removal of hazardous anionic dye “Congo red” from wastewater using waste materials and recovery by desorption

**Adv. Colloid Interface Sci., 193-194 (2013) 24-34.**

Adsorptive removal of dyes from aqueous solution onto carbon nanotubes: A review

**Journal of Molecular Liquids 211, 457-465 (2016).**

Green Synthesis of Iron Nano-Impregnated Adsorbent for Fast Removal of Fluoride from Water

**Chemical Eng. J., 268(2015) 28-37.**

Modeling of competitive ultrasonic assisted removal of the dyes – Methylene blue and Safranin-O using Fe<sub>3</sub>O<sub>4</sub> nanoparticles

**Chemical Engineering Journal, Volume 284, January 15, 2016, Pages 687-697**

Removal of hazardous dyes-BR 12 and methyl orange using graphene oxide as an adsorbent from aqueous phase

**Chemical Engineering Journal; 180, 15 January 2012, Pages 81-90**

Cadmium removal and recovery from aqueous solutions by novel adsorbents prepared from orange peel and Fe<sub>2</sub>O<sub>3</sub> nanoparticles



## Reviewer 2

Comments: This paper presents the use of modified activated carbon produced from microwave pyrolysis in the adsorption of copper ion. The protocol provided are descriptive. The results and discussions presented would be useful to the reader. Following are my comments for improvements:

**Comment 1:** Line 87-88, it's confusing on how the 15 wt% of phosphoric acid solution was prepared in 1:1 mass ratio. What is the SI unit for the "mass"?

**Response:** The statement “Place 30 g of fine bagasse powder into 15 wt% of phosphoric acid solution in a 1:1 mass ratio for 24 h.” means “Place 30 g of fine bagasse powder into 30 g of phosphoric acid solution (15 wt%) for 24 h”. The ratio of their weight is 1:1. In order to disambiguate the sentences, all “mass” has been replaced by “weight”.

**Comment 2:** Line 200-203, in order to claim the modified activated carbon produced from microwave pyrolysis contained more nitrogen/oxygen functional groups than the one prepared from conventional pyrolysis, the author should present the results of elemental composition (C, H, N, O, S) of both samples to support their claim.

**Response:** Revised as the reviewer’s advice. The results of elemental composition of samples are listed in Table 1.

**Comment 3:** The author should justify why the word "pre-pyrolysis" is used instead of "pyrolysis". Also, they should be consistent to the use of one term.

**Response:** In this study, the pyrolysis process is followed up by modification. And we demonstrated that the microwave pyrolysis benefits the follow-up introduction of surface functional groups to improve the adsorption capacity than electric-heating pyrolysis. So the word “pre-pyrolysis” is used instead of “pyrolysis” in original manuscript. For ease of understanding, we have replaced “pre-pyrolysis” with “pyrolysis” in the text of the revised manuscript.

**Comment 4:** In the introduction, the author should describe why chemical activation was preferred than physical activation. Following articles are suggested as both references and citations to the author:

-Microwave-assisted pyrolysis with chemical activation, an innovative method to

convert orange peel into activated carbon with improved properties as dye adsorbent  
-Production of activated carbon as catalyst support by microwave pyrolysis of palm kernel shell a comparative study of chemical versus physical activation.

**Response: Thank you very much for your suggestion. The INTRODUCTION has been revised as the reviewer's advice. These references have been cited in the revised manuscript.**

**Comment 5:** The author should also describe why one step activation was preferred.

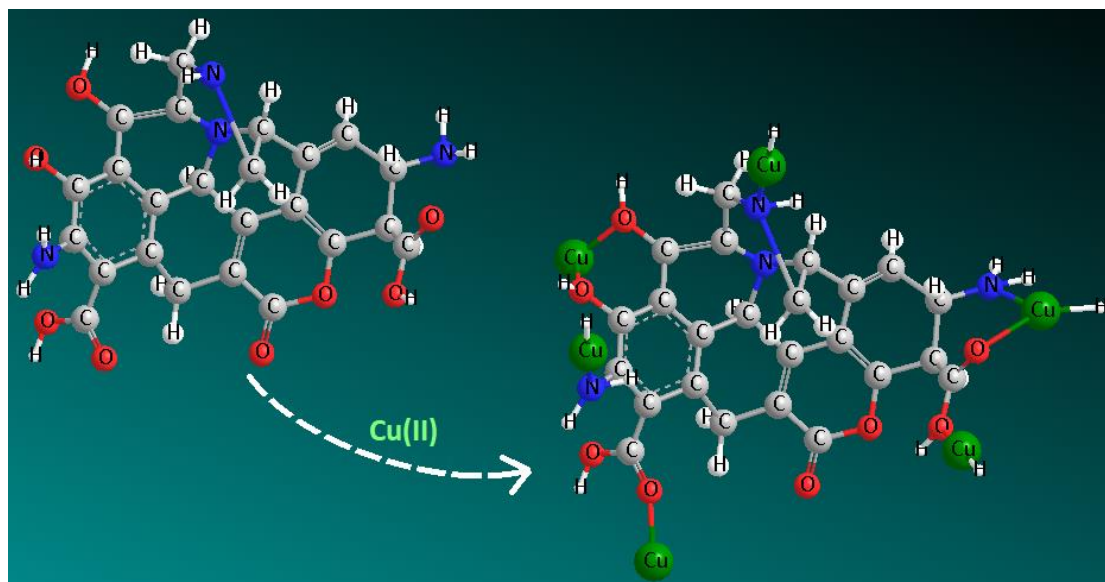
**Response: Thank you very much for your suggestion. Revised as the reviewer's advice.**

### Reviewer 3

Comments: This manuscript "Preparation of biomass-based mesoporous carbon with higher nitrogen/oxygen-chelating adsorption for Cu(II) through microwave pre-pyrolysis" is logical, the organization of sentence is good.

**Comment 1:** While they only describe the adsorption capacity increase after doping in this article, the reasons for the adsorption capacity increase did not explain, which needs to supplement. The manuscript should be improved according to the comments before considering for publications.

**Response:** Thank you very much for your suggestion. Revised as the reviewer's advice. The mechanism analysis is added in the revised manuscript as well as the reaction scheme (Figure 5).



**Comment 2:** The analysis of mechanism needs to be strengthened instead of describe the increase in nitrogen/oxygen-chelating can improve the adsorption capacity of Cu(II) only. The specific adsorption mechanism of Cu(II) need to be supplement.

**Response:** Revised as the reviewer's advice. The mechanism analysis is added in the revised manuscript as well as the reaction scheme (Figure 5).

**Comment 3:** The logical of INTRODUCTION should be organized, the conventional electric-heating doesn't need to contrast with Microwave irradiation too much, and the feasibility analysis of doping or advantage analysis of doping in previous should be used to elicit your own experimental research.

**Response:** Thank you very much for your suggestion. The INTRODUCTION has been revised as the reviewer's advice.

**Comment 4:** The advantages of preparation of biomass-based mesoporous carbon need to be highlighted in comparison with other studies, and the adsorption mechanism of Cu(II) after doping should be appointed in.

**Response:** Revised as the reviewer's advice. The following content is added in the revised manuscript as well as the comparison table (Table 4).

Table 4 compares the adsorption capacity of Cu(II) by the recently reported biomass-based activated carbon. It is found that MBAC-N has a higher adsorption capacity than other adsorbents reported in the literature, demonstrating it a promising adsorbent for removing Cu(II).

**Table 4.**

Adsorbents	pH	q <sub>e</sub> (mg g <sup>-1</sup> )	References
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Bouhamed F, Elouear Z, Bouzid J, et al. Multi-component adsorption of copper, nickel and zinc from aqueous solutions onto activated carbon prepared from date stones[J]. Environmental Science & Pollution Research, 2016, 23(16):1-6.

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Vunain E, Kenneth D, Biswick T. Synthesis and characterization of low-cost activated carbon prepared from Malawian baobab fruit shells by H<sub>3</sub>PO<sub>4</sub> activation for removal of Cu(II) ions: equilibrium and kinetics studies[J]. Applied Water Science, 2017, 7(8):4301-4319.

Wu L, Wan W, Shang Z, et al. Surface modification of phosphoric acid activated carbon by using non-thermal plasma for enhancement of Cu(II) adsorption from aqueous solutions[J]. Separation & Purification Technology, 2018, 197.

**Comment 5:** Line 218'The higher becomes the temperature, the better are adsorption properties.'should be 'The adsorption properties of samples for Cu(II) become better with the temperature increasing.'; Line 228-229'The above results confirm that the adsorption of MBAC-N on copper is chemisorption and it has a promising ability to remove copper ions.'should be'The above results confirm that the adsorption of Cu(II) on MBAC-N is chemisorption and MBAC-N is an promising absorbent to remove Cu(II).';

**Response:** Thank you very much for your suggestion. Revised as the reviewer's advice.

**Comment 6:** Words in this paper should keep consistent, as'Cu(II)'in Line 222 and Line 223 ; 'copper ion'in Line 158 and Line 224.

**Response:** Thank you very much for your suggestion. All terms “copper ion” have been replaced by “Cu(II)” as the reviewer's advice.

**Comment 7:** The references should be quoted as far as possible for nearly three years. For example, the following papers with relative topics about activated carbons prepared or modified by microwave pyrolysis, such as Chemical Engineering and Processing, 2015, 91: 67-77; Journal of Industrial and Engineering Chemistry, 2016, 39:27-36; Korean Journal of Chemical Engineering, 2015, 32(6): 1129-1136, Bioresource Technology, 2016, 201: 41-49, etc. which are valuable for being referred for analysis the results in manuscript.

**Response:** Revised according to the reviewer's advices. Some old references have been replaced by recent relevant papers in the revised manuscript. These references have been cited in the revised manuscript.

**Comment 8:** In Fig. 4, which kind of AC was used to adsorb the Cu(II) ?

**Response:** Figure 4 shows representative adsorption property analysis of MBAC-N.

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