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# Effect of Oxygen Bias Pressure in Furnace on Structure and Adsorption Properties of Bamboo Charcoal

Wang Wenhao<sup>1</sup>, Wu Zhiwei<sup>2</sup>

<sup>1</sup>Anhui Technical College of Mechanical and Electrical Engineering

<sup>2</sup>College of Mechanical and Electronic Engineering, Nanjing Forestry University, Nanjing 210037

\* Corresponding author: 0124000067@ahcme.edu.cn

**Abstract:** In this paper, the effect of oxygen bias on the structure and adsorption properties of bamboo charcoal was studied using oxygen sensor by controlling of oxygen partial pressure in the furnace. The chemical structure and microstructure of bamboo charcoal were analyzed by infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The adsorption test for bamboo charcoal showed that the adsorption capacity of nitrophenol first increased and then decreased with the decrease of oxygen bias pressure, and the maximum adsorption capacity (222.542 mg/g) was obtained for bamboo charcoal that been prepared at a oxygen bias pressure  $3.910 \times 10^{-4}$  Pa . Therefore, oxygen content must be considered in bamboo carbonization process.

## 1. Introduction

Because of the special porous structure of bamboo charcoal, it has potential and broad application prospects in the fields of environmental protection, health care, medicine, high-tech and so on [1]. Carbonization temperature and carbonization time are two important process parameters [2]. However, it is also necessary to control the oxygen content in the furnace, to form an anoxic high temperature pyrolysis environment to prevent bamboo charcoal from spontaneous combustion, and to carbonize bamboo in a balanced oxygen partial pressure (neutral) or reducing high temperature atmosphere [3]. The effect of oxygen partial pressure on the carbon yield and structure in the furnace was studied in [4-6]. Bamboo charcoal has developed pore formations structure and large surface area with strong adsorption properties, which are often used to purify sewage and harmful gases [7,8]. However, the effect of oxygen content on adsorption properties of bamboo charcoal is rarely reported. In this paper, by adjusting the nitrogen and air flow into the furnace, and using the sensor to measure and control the partial pressure of oxygen in the furnace in real time, the influence of oxygen content in the furnace on the structure and adsorption properties of bamboo after carbonization is studied, which provides a theoretical basis for the carbonization and application of bamboo.

## 2. Materials, equipment and methods

### 2.1 Materials

Bamboo materials selected in Liyang City, Jiangsu Province, Hengjian Township, Nanshan bamboo sea bamboo age of 4 years, the sampling site is about 1 meter from the root, the sample is made into



100mm×35mm cuboid shape. The samples were dried at 120°C for 2 hours, and each group of samples was taken about 300 grams of bamboo.

## 2.2 Equipment and test methods

### (1) Thermogravimetric analysis

The carbonization process of bamboo was analyzed using a thermogravimetric analyzer (netzschstat 409 pc/pg). the heating rate was 10 k/min and the end temperature was 1300oc.

### (2) Carbonization equipment for bamboo

Equipment consists of a crucible resistance furnace for the heating system ( $w = 7.5$  kW,  $t = 1000^\circ\text{C}$ ), a temperature control system (ksj-12) and an oxygen content control system. according to the carbonization process of figure 1(b), the final carbonization temperature is  $900^\circ\text{C}$  and the heating and holding time is 90 min. by adjusting the ratio of nitrogen and air, the oxygen partial pressure  $p_{O_2}$  in the furnace of different furnaces is  $2.938 \times 10^3$ ,  $3.910 \times 10^{-4}$ ,  $1.428 \times 10^{-7}$ ,  $4.851 \times 10^{-12}$  and  $7.314 \times 10^{-12}$  respectively.

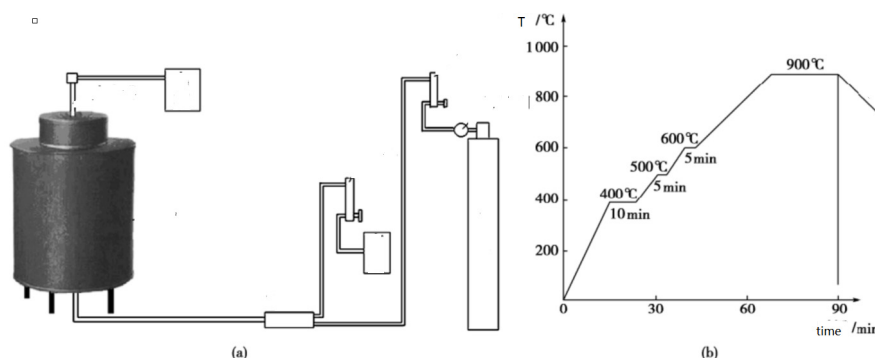


Fig.1 Carbonization test equipment (a) and heating process curve of carbonization of bamboo (b)[6]

(3) Fourier transform infrared spectrometer (FTIR, Nicolet 380) analysis, using potassium bromide pressure plate method to prepare the sample. about 1~2 mg of trace bamboo charcoal powder was put into the mortar, and then about 200 mg of spectral pure (kbr) was mixed and ground to the powder shape. the particle size was as small as possible as the infrared light wavelength to avoid the dispersion. After the grinding is finished, the sample is dried, and the sample is pressed into 0.5 mm sheet by the sheet press.

(4) Adsorption characteristics of p-nitrophenol from bamboo charcoal. Bamboo charcoal grinding particle size 200 mesh, nitrophenol solution concentration can be well expressed by absorbance. the maximum absorption wavelength \955; of p-nitrophenol was measured by uv-vis spectrophotometer (uv-2450) at 316 nm. the adsorbent concentration was 600 mg/l. at room temperature, the conical bottle containing the sample was put into a water bath constant temperature oscillator (shz-a) with a 138 r/min oscillation for 3 h. Take a sample every half hour to measure its absorbance to find out the adsorption amount of bamboo charcoal until the adsorption equilibrium is reached. According to the equation of adsorption capacity, the absorbance is determined and calculated Value of adsorption capacity:

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

Where,  $q$  is the adsorption capacity,  $v$  is the volume of the adsorbent,  $c_0$  and  $c_e$  are the initial and equilibrium concentrations of the adsorbent, respectively, and  $m$  is the bamboo carbon weight (g).

## 3. Results and Analysis

From the thermogravimetric curve (figure 2), it can be seen that there is a relatively gentle weightlessness step at 25-225°C and a very steep weightlessness at 225-380°C, indicating that a violent decomposition reaction occurs at this time, releasing a large amount of gas; there is a gentle

weightlessness process at 380-450°C, and the mass at 450-1300°C is close to stationary. The overall quality change is -74.66%. on the dsc curve, there are two obvious endothermic peaks near 380°C and 450°C with an area of 31.13 J/g and 103.1 J/g, respectively. It can be seen from the thermogravimetric curve The carbonization process goes through four stages, namely, drying stage, precarbonization stage, carbonization stage and calcination stage. Before 300°C, it was the drying stage of bamboo, mainly because the adsorbed water in bamboo was heated and evaporated, and the weight loss in this stage was relatively gentle, and there was no chemical change. 300-350°C is the precarbonization stage of bamboo charcoal. the weight loss rate of bamboo in this stage becomes faster, and there is an endothermic peak in the dsc curve, indicating that an endothermic decomposition reaction occurs at this time. At this time, the unstable cellulose in bamboo and the cyclic and chain structure in hemicellulose are destroyed, resulting in a large amount of carbon monoxide, carbon dioxide and a small amount of vinegar Acid. at 350-450°C, the weight loss rate is large, and there is also an endothermic peak in the dsc curve of this stage, and the area is larger, indicating that the decomposition reaction is more intense and enters the carbonization stage. at this time, cellulose and hemicellulose continue to decompose, and lignin also begins to decompose, producing a large number of gases and decomposition products, mainly carbon monoxide, carbon dioxide, acetic acid, bamboo coke oil, methanol and so on. After entering the calcination stage, the thermogravimetric curve of this stage is stable, basically no chemical reaction occurs, but the volatile matter remaining in bamboo charcoal is excreted. Analysis of the thermogravimetric curves of bamboo charcoal can understand the occurrence of bamboo charcoal in various stages of carbonization changes and reactions to achieve the purpose of improving production efficiency and product quality. according to the thermogravimetric analysis, it is determined that the final carbonization temperature of this paper is 900°C.

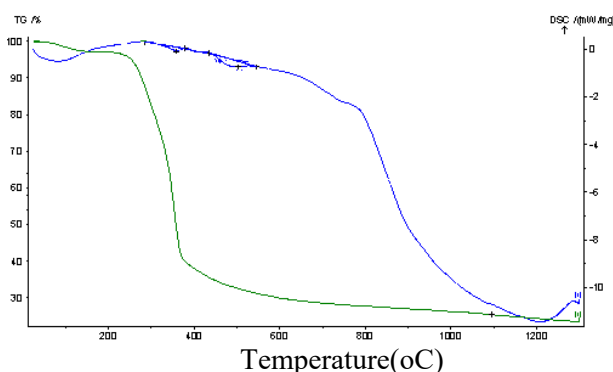


Fig.2 Thermogravimetric analysis of bamboo

### 3.1 Fourier transform infrared spectroscopy

The main components of bamboo charcoal are C, H and O elements, in addition to a small amount of N and metal elements. So the infrared spectrum of bamboo charcoal mainly reflects the vibration of C, H and O elements [9]. as shown in the spectral diagram (figure 3), the specimens all have vibrations at 3400–3500  $\text{cm}^{-1}$ , which is the intermolecular hydrogen bond o-h stretching vibration. except for the oxygen partial pressure of  $4.851 \times 10^{-12}$  Pa, the other specimens have a wide peak between 1000 and 1200  $\text{cm}^{-1}$ , which is the c-o stretching vibration. In addition, except for the oxygen partial pressure of  $4.851 \times 10^{-12}$  Pa, the C = C stretching vibration indicates the presence of olefins; the specimens with oxygen pressure of  $3.91 \times 10^{-4}$  Pa and  $7.314 \times 10^{-14}$  Pa have C = C skeleton vibrations, indicating the presence of aromatic hydrocarbons. the oxygen partial pressure is  $7.314 \times 10^{-14}$  Pa with vibration at 2922  $\text{cm}^{-1}$ , indicating a saturated c-h stretching vibration absorption. The partial pressure of oxygen in the furnace is  $2.938 \times 10^3$  Pa, and the bamboo charcoal has the structure of alcohol and olefin, the structure of alcohol, olefin and aromatics, and the partial pressure of oxygen is  $1.428 \times 10^{-7}$  Pa the prepared bamboo charcoal has alcohol, olefin structure; the bamboo charcoal with oxygen partial pressure of  $4.851 \times 10^{-12}$  Pa in the furnace has only the structure of intermolecular hydrogen bond; the bamboo charcoal with oxygen partial pressure of  $7.314 \times 10^{-14}$  Pa in the furnace has the structure of

olefin, aromatics, methyl. When the oxygen content decreases, the vibration peak of O-H becomes wider and smaller, indicating that the oxygen content decreases and the more O-H decomposes. The C-O vibration peak is also increasing, and the C-H vibration peak gradually appears, and the C=C vibration peak becomes weaker, indicating that when the oxygen content decreases, C=C and O-H decompose into C-H and C-O. The chemical composition of bamboo charcoal has a certain effect on its adsorption ability and conductivity. The O-H hydrogen bond in bamboo charcoal adsorbs water molecules and many ions. The results showed that the influence of oxygen partial pressure on the composition of the chemical structure functional groups of bamboo charcoal was not significant, mainly affects the degree of decomposition of C = C bonds. This is because the oxidation reaction occurs when the oxygen content is high, with a small amount of C = C bonds oxidized to unsaturated C-O and C-C bonds.

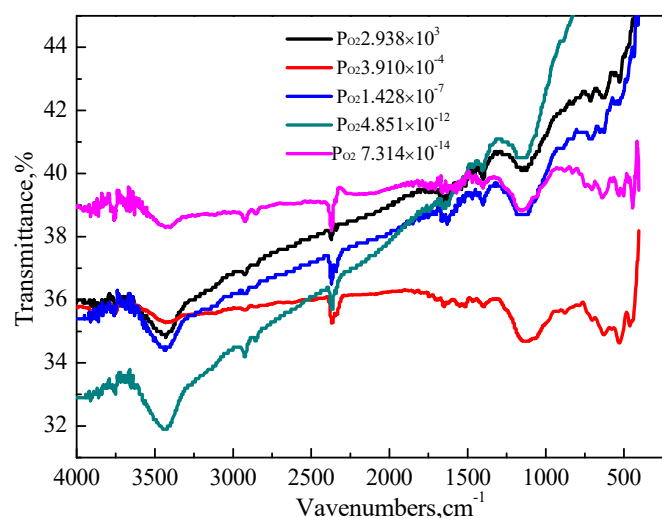


Fig.3 Infrared map of bamboo charcoal prepared under different oxygen content in furnace

### 3.2 Effect of partial pressure of oxygen on adsorption properties of bamboo charcoal in furnace

Figure 4 depicts the adsorption equilibrium time and concentration adsorption curve of bamboo charcoal p-nitrophenol solution. The adsorption amount reached equilibrium after 3 hours of adsorption, but after 5 hours, the decrease occurred, which was mainly caused by desorption, and the adsorption equilibrium time of bamboo charcoal for p-nitrophenol was determined to be 3 hours. However, with the increase of nitrophenol concentration, the adsorption capacity showed an increasing trend. theoretically, the maximum concentration of adsorption capacity should be used. in this paper, mainly to study the effect of oxygen partial pressure on the adsorption performance of bamboo charcoal under the same conditions, the adsorbent of 600 mg/l should be used as the experimental agent. The effect of oxygen partial pressure on adsorption properties of bamboo charcoal is described in Table 1. when the oxygen partial pressure is higher and lower, the adsorption capacity is less than that of  $3.910 \times 10^{-4}$  when the oxygen partial pressure is  $3.910 \times 10^{-4}$  (222.542 mg/g). usually, the larger the specific surface area of bamboo charcoal, the stronger the adsorption capacity. According to the cross-section morphology of bamboo charcoal (Fig.5), the typical honeycomb-like structure of bamboo charcoal can be seen. however, the specimens corresponding to the high oxygen partial pressure (figure 5(a), figure 5(b)) had relatively loose vascular bundles forming pores. the vascular bundles of other specimens (figure 5(c), figure 5(d), figure 5(e)) were relatively dense with fewer pores. Therefore, it can also be seen from the morphology of the section that the prepared under low oxygen partial pressure bamboo charcoal has a small surface area, mainly due to the lower oxygen concentration inhibiting the formation of micropores. whereas the higher oxygen partial pressure causes excessive carbonization, which causes the layers of the striated pores to be gradually destroyed



to form large pores (figure 5(a)), thus causing a decrease in the specific surface area. It can be seen that it is necessary to control the oxygen content in the carbonization process.

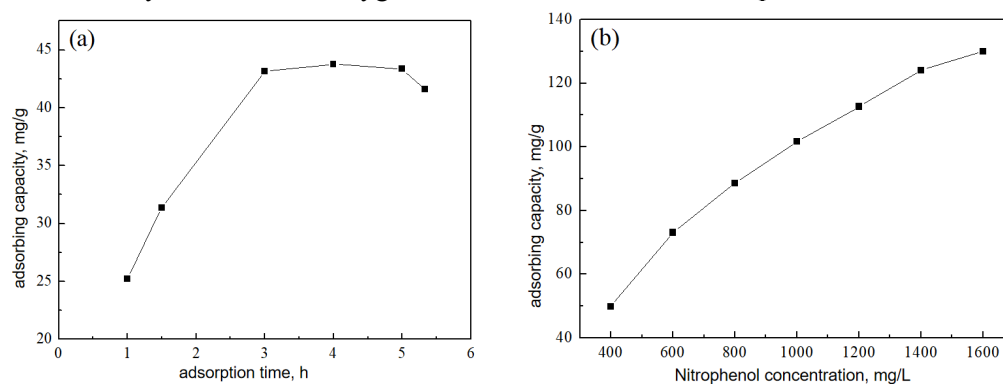


Fig.4 Adsorption equilibrium time (a) and concentration adsorption curve of p-nitrophenol solution (b)

Table 1 Effect of partial pressure of oxygen on adsorption properties of bamboo charcoal

Partial pressure of oxygen in furnace (Pa)	Bamboo charcoal (g)	Initial concentration (mg/L)	Dilution multiple	Absorbance (Abs)	filtrate concentration (mg/L)	equilibrium concentration (mg/L)	Adsorbance (mg/g)
$2.938 \times 10^3$	0.1033	600	50	0.500	4.1224	206.1181	190.650
$3.910 \times 10^{-4}$	0.1182	600	50	0.312	1.4782	73.90999	222.542
$1.428 \times 10^{-7}$	0.1113	600	50	0.635	6.0211	301.0549	134.297
$4.851 \times 10^{-12}$	0.1081	600	50	0.719	7.2025	360.1266	110.950
$7.314 \times 10^{-14}$	0.1027	600	50	0.671	6.5274	326.3713	133.218

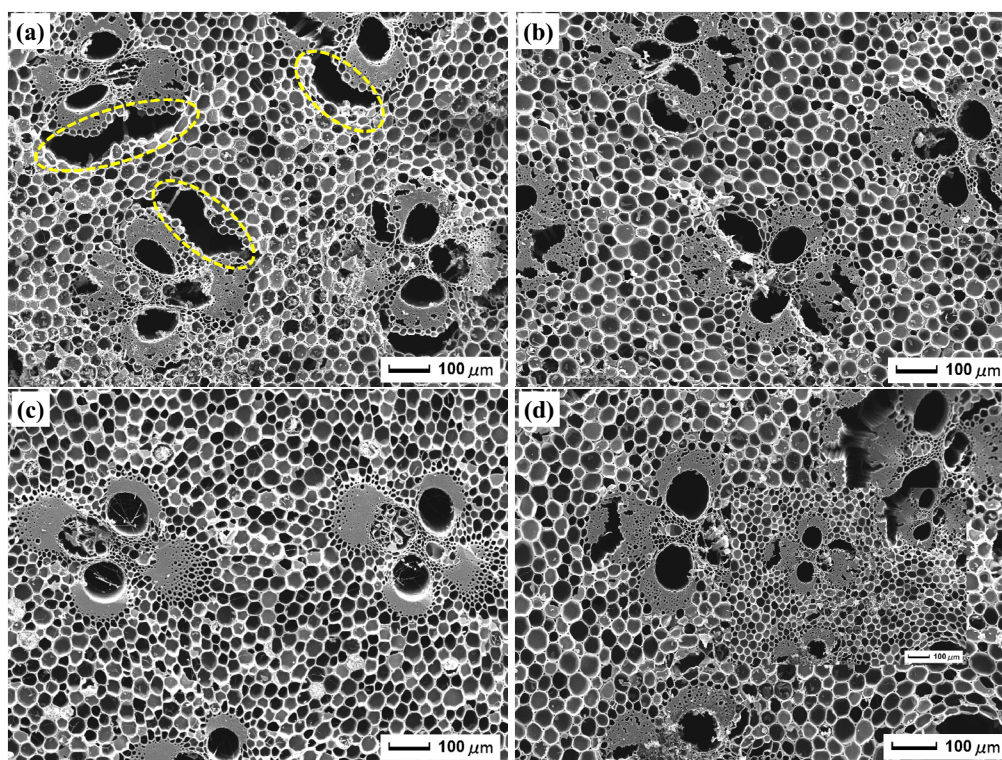


Fig.5 Section morphology of bamboo charcoal under different oxygen pressure: (a)  $2.938 \times 10^3$  Pa, (b)  $3.910 \times 10^{-4}$  Pa, (c)  $1.428 \times 10^{-7}$  Pa, (d)  $4.851 \times 10^{-12}$  Pa, (e)  $7.314 \times 10^{-14}$  Pa.

#### 4. Conclusion

Different structures of bamboo charcoal were prepared by controlling the oxygen partial pressure in the furnace. when the oxygen partial pressure is high (2.938103pa), it will cause excessive carbonization, which will gradually destroy each layer of the grain hole to form a large hole. When the partial pressure of oxygen is low, the vascular bundle has less relatively dense pores. the adsorption results of bamboo charcoal showed that the maximum adsorption capacity of bamboo charcoal was 222.542 mg/g when the oxygen fraction was  $3.910 \times 10^{-4}$  Pa. Therefore, in the process of bamboo carbonization, the oxygen content in the furnace should be controlled according to the purpose of use.

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