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微波法制备电化学电容器用花生壳基活性炭

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摘要: 以花生壳为原料, 氢氧化钾为活化剂, 采用微波加热制备出活性炭, 所制活性炭用于制备电化学电容器用电极材料。通过氮气吸附、恒流充放电及循环伏安对所制活性炭的孔结构及电化学性能进行研究。结果表明, 活性炭的比表面积、总孔容、比电容以及能量密度在炭化时间(6-10 min)以及 KOH 与花生壳的质量比(0.6-2.0)的范围内存在最大值。当 KOH/花生壳的质量比为 1.0, 微波功率为 600 W, 活化时间 8 min, 所制活性炭($AC_{1-600,8}$)比表面积达 1277 m²/g, 并且经 1000 次循环后, 其能量密度高达 8.38 Wh/kg。因此采用微波加热、KOH 活化是一种快速制备电化学电容器用活性炭的低成本方法。

关键词: 花生壳; 微波加热; 活性炭; 电化学电容器

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Microwave-assisted preparation of peanut

shell-based activated carbons and their use in electrochemical capacitors

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Abstract: Activated carbons (ACs) were prepared from peanut shells by KOH activation under microwave heating and were used as electrode materials for electrochemical capacitors (ECs). The pore structure of the ACs was characterized by nitrogen adsorption and the electrochemical performance by galvanostatic charge-discharge and cyclic voltammetry. Results show that the ACs' specific surface area, total pore volume, specific capacitance, as well as energy density are maximized using an activation time from 6 to 10 min or KOH/peanut shell mass ratio from 0.6 to 2.0 under otherwise identical conditions. When the KOH/peanut shell mass ratio was 1.0, microwave power was 600 W and activation time was 8 min, the specific surface area of the resulting AC was 1277 m²/g and its energy density was 8.38 Wh/kg after 1000 cycles. The KOH activation of peanut shells with microwave heating is an efficient approach for the rapid preparation of low cost ACs for ECs.

Keywords: Peanut shell; Microwave heating; Activated carbon; Electrochemical capacitor

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1 Introduction

China is the world's largest peanut producer with a capacity of 5 million metric tons of peanut shells each year. As a byproduct of peanut production, peanut shell is normally used as animal feed or fuel without any value and technology-added applica-

tions. On the other hand, activated carbons (ACs) with sufficient pores and good adsorptivity have been widely used in various fields^[1], e. g. industrial purification, chemical recovery and electrode materials for electrochemical capacitors (ECs). The fact that peanut shell is cost-competitive, readily available in such large quantity, leads us to believe that converting pea-

nut shell into useful technology-added ACs should be highly beneficial and meaningful^[2]. Traditionally, ACs are usually prepared by physical or chemical activations via conventional heating. Among the chemical activation methods, KOH activation is a rather efficient activation agent for preparation of microporous carbons^[3-7]. However, the biggest economic barrier to convert peanut shell to ACs is the high cost of activation, which usually requires extended activation time, high energy consumption and excessive activation agent. This being the case, it would be imperative to invent a rapid and efficient approach to make low-cost ACs.

In terms of heating means, microwave heating has lots of advantages over conventional heating means, e. g. high heating efficiency, easy control of the heating process, rapid temperature rise at low energy consumption^[8,9]. For these reasons, the microwave heating has received extensive attention over the past few years for AC preparation^[10-15]. To date, little has been reported on an efficient and straight-forward preparation of ACs from renewable peanut shell for use as ECs electrode materials.

Herein, the prospect in deriving ACs from peanut shell using microwave heating for ECs was investigated at lowered KOH/peanut shell mass ratios with shortened activation times. The effects of the key determining factors, such as KOH/peanut shell mass ratio and activation time, on the pore structure and electrochemical performance of ACs in ECs were studied.

2 Experimental

2.1 Preparation and characterization of ACs

Peanut shell was obtained from Huaian in Jiangsu, China. Peanut shell particles with a size of about $3 \times 10 \text{ mm}^2$ were cleaned by water scrubbing and then dried at 383 K for 24 h. The analysis results of peanut shell on air dry basis by mass fraction are as follows: 9.52% of moisture, 1.30% of ash, 68.34% of volatile matter and 20.84% of fixed carbon.

Dried peanut shell (9 g) was mixed with KOH solution at different KOH/peanut shell mass ratios. The mixture was dried at 383 K for 24 h after being impregnated for 12 h at room temperature, and then transferred to a crucible. The crucible was heated in a LWMC-205-type microwave oven with a microwave power of 600 W from 6 to 10 min of activation to prepare the ACs. The temperature of reactants in the crucible was measured by an armor-type thermocouple during microwave heating.

ACs were successively washed with 0.5 mol/L HCl solution and distilled water, until pH = 7.0 was

reached. ACs were then dried at 383 K for 24 h. The resultant AC is designated as AC_{x-y-z} , where x refers to the KOH/peanut shell mass ratio, y the microwave power, and z the activation time. For example, $\text{AC}_{1-600-8}$ refers to the AC prepared with a KOH/peanut shell mass ratio of 1, a microwave power of 600 W and an activation time of 8 min. Characterization of the pore structure of ACs was performed on the basis of nitrogen adsorption-desorption isotherms measured on a sorptometer ASAP2010 at liquid nitrogen temperature.

2.2 Preparation and electrochemical measurements of AC electrodes

The electrode slurry was made by mixing AC, carbon black (CB) and poly(tetrafluoroethylene) (PTFE) in a mass ratio of 87 : 5 : 8. The slurry was coated onto nickel foam with a diameter of 12 mm. Prior to packaging and test, two disk electrodes with an active mass of about 40 mg were dried at 393 K for 2 h under vacuum. One cell was composed of two similar electrodes separated by polypropylene membrane. The cell was tested in 6 mol/L KOH solution using a symmetrical button cell configuration by cyclic voltammetry on an electrochemical workstation (CHI-760C, Chenghua, Shanghai). The electrochemical performance of AC electrodes in the cell was investigated on a land cell tester (Land, CT-2001A).

3 Results and discussion

3.1 Pore structure of ACs

The N_2 adsorption-desorption isotherms of the ACs are shown in Fig. 1.

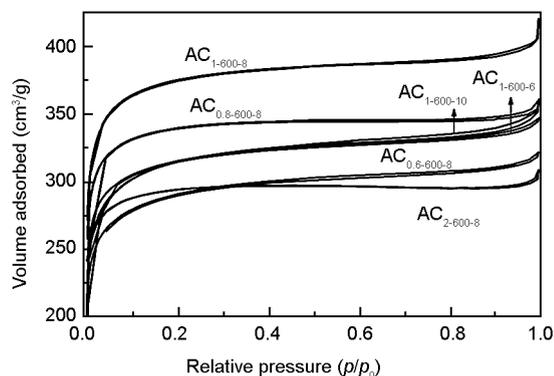


Fig. 1 N_2 adsorption-desorption isotherms of ACs.

It is found that all the ACs are microporous as evidenced by the Type I isotherm. The pore structure parameters of the ACs are shown in Table 1. It can be seen that the specific surface area, total pore volume, micropore volume of the AC all exhibit maxima with activation time for 8 min or with KOH/peanut shell mass ratio for 0.8 under otherwise identical conditions

investigated. At a KOH/peanut shell mass ratio of 1.0 with a microwave power of 600 W and an activation time of 8 min, the S_{BET} of $\text{AC}_{1-600-8}$ reach $1277 \text{ m}^2/\text{g}$. The S_{BET} of ACs produced by microwave heating is larger than that from biomass using conventional heating methods even at a longer activation time^[16], which is ascribed to the efficiency of microwave heating at molecular level. The yields of $\text{AC}_{1-600-6}$, $\text{AC}_{1-600-8}$ and $\text{AC}_{1-600-10}$ are 24.4%, 21.8% and 18.0%, respectively, showing the same trend in the yields of ACs with activation temperature^[17]. The final activation temperature of $\text{AC}_{1-600-6}$, $\text{AC}_{1-600-8}$ and

$\text{AC}_{1-600-10}$ are 1083, 1113 and 1133 K, respectively, and the average heating rate in the preparation of ACs is rather high, ranging from 84 to 132 K/min. Elevated activation temperatures for $\text{AC}_{1-600-6}$, $\text{AC}_{1-600-8}$ and $\text{AC}_{1-600-10}$ caused by the increasing activation time are favorable for releasing more gaseous products and thus are responsible for the decreasing AC yields.

In KOH activation at over 673 K, the reaction between KOH and carbon occurs based on the following equation^[18].



Table 1 Pore structure parameters of ACs.

AC sample	D_{ap} (nm)	S_{BET} (m^2/g)	V_{t} (cm^3/g)	V_{mic} (cm^3/g)	S_{mic} (m^2/g)	S_{ext} (m^2/g)	Yield (%)
$\text{AC}_{1-600-6}$	1.99	1065	0.53	0.38	839	226	24.40
$\text{AC}_{1-600-8}$	1.98	1277	0.63	0.47	1028	249	21.80
$\text{AC}_{1-600-10}$	2.03	1082	0.55	0.36	786	296	18.00
$\text{AC}_{0.6-600-8}$	2.00	990	0.50	0.34	733	256	23.10
$\text{AC}_{0.8-600-8}$	1.92	1145	0.55	0.45	967	178	24.50
$\text{AC}_{2-600-8}$	1.90	995	0.47	0.39	850	145	14.20

* D_{ap} , average pore diameter; S_{BET} , specific surface area; S_{mic} , micropore surface area (<2 nm); S_{ext} , external surface area (>2 nm); V_{t} , total pore volume; V_{mic} , micropore volume (<2 nm).

The formed metallic potassium intercalates to the carbon matrix, resulting in a widening of the spaces between carbon atomic layers and an increase of pore volume. At over 923 K, the surface metal complexes are responsible for a further gasification, which leads to the widening of micropores. In particular, the D_{ap} of $\text{AC}_{1-600-10}$ is 2.03 nm, the largest among all the ACs.

The yields of ACs are related to both on the KOH/peanut shell mass ratio and the activation time. The yield of $\text{AC}_{0.8-600-8}$ is 24.5%, the highest in this work. Table 1 shows that all the ACs are microporous with the S_{BET} , V_{t} and V_{mic} of $\text{AC}_{1-600-8}$ being the largest among all ACs. These micropores less than 1 nm would be a positive contributor to the improved capacitance of ACs in ECs^[19].

3.2 Electrochemical performance of ACs

Cyclic voltammetry is usually used to characterize the capacitive behaviors of electrode materials in ECs. Fig. 2 shows the cyclic voltammetry curves of $\text{AC}_{1-600-8}$ electrodes at different scan rates. The cyclic voltammetry curves of $\text{AC}_{1-600-8}$ electrodes retain a symmetric rectangular shape with increasing the scan rate from 2 to 50 mV/s, which suggests a quick charge propagation in $\text{AC}_{1-600-8}$ electrodes.

The specific capacitance of a AC electrode in ECs was calculated from the slope of the discharge curve^[20]. The variation of specific capacitance of the AC electrodes with discharge current density is presented in Fig. 3a, b. The inset in Fig. 3a is the

1000th charge-discharge curve of AC electrodes at a current density of 0.05 A/g. A slight decrease in specific capacitance is observed with increasing discharge current density in Fig. 3a, b.

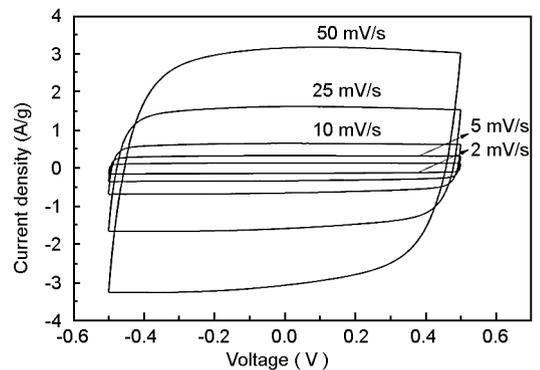


Fig. 2 Cyclic voltammetry curves of $\text{AC}_{1-600-8}$ electrode at different scan rates.

Fig. 3a indicates that specific capacitance of AC electrodes increases with heating time from 6 to 10 min, and eventually reaches a plateau at 8 min. Fig. 3b shows that specific capacitance of AC electrodes is associated with the KOH/peanut shell mass ratio. The specific capacitance of AC electrodes prepared with a KOH/peanut shell mass ratio of 1.0 and an activation time of 8 min is the highest among those AC electrodes with a KOH/peanut shell mass ratio from 0.6 to 2.0 and an activation time of 8 min, which is ascribed to the largest S_{BET} , V_{t} and V_{mic} of $\text{AC}_{1-600-8}$. In contrast, the specific capacitance of

AC₁₋₆₀₀₋₁₀ electrode in Fig. 3a is smallest among AC electrodes investigated, which is likely due to that some carbonylic functional groups in AC₁₋₆₀₀₋₁₀ were removed and/or some micropores in AC₁₋₆₀₀₋₁₀ were widened caused by an extended heating or elevated temperature^[21-23]. Apparently, Table 1 shows that the D_{ap} and S_{ext} of AC₁₋₆₀₀₋₁₀ are the biggest among all ACs. The specific capacitance of AC₁₋₆₀₀₋₈ decreases from 242.8 to 228.4 F/g with current density from 0.05 to 1.20 A/g, and the capacitance retention of AC₁₋₆₀₀₋₈

electrode eventually reaches as high as 94.0%. The capacitance retention of other AC electrodes is also found rather high, ranging from 91.5% to 92.6%. It is noteworthy that the specific capacitances of all AC electrodes derived from peanut shells via KOH activation by microwave heating in 6 mol/L KOH electrolyte are significantly improved over those of AC electrodes from dehydrogenated chars by conventional phosphoric acid activation for 30 min^[24].

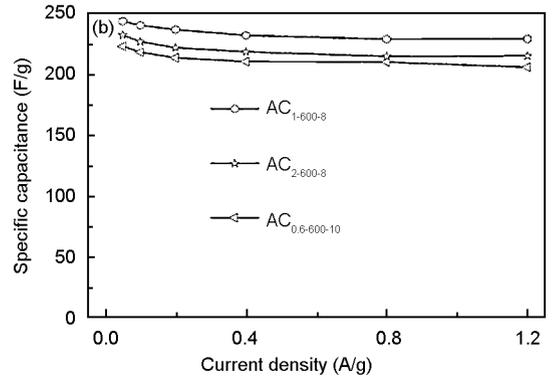
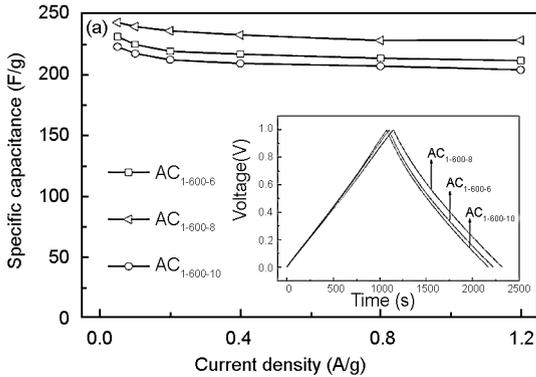


Fig. 3 Specific capacitance of AC electrodes vs. current density: (a) ACs made at different activation times, (b) ACs made at different KOH/peanut shell mass ratios.

For ECs made of AC electrodes, energy density of ECs (E , in Wh/kg) is usually calculated on the basis of Eq. (1)^[25].

$$E = 1/2CV^2 \quad (1)$$

Where C is the capacitance of the two-electrode capacitor (F/g), V the usable voltage (V) excluding the IR drop occurring at the discharge.

Average power density of ECs (P , in W/kg) is calculated according to Eq. (2)^[7].

$$P = E/\Delta_{td} \quad (2)$$

Where Δ_{td} denotes the time spent in discharge.

The variation of energy density of AC capacitors with power density is presented in Fig. 4.

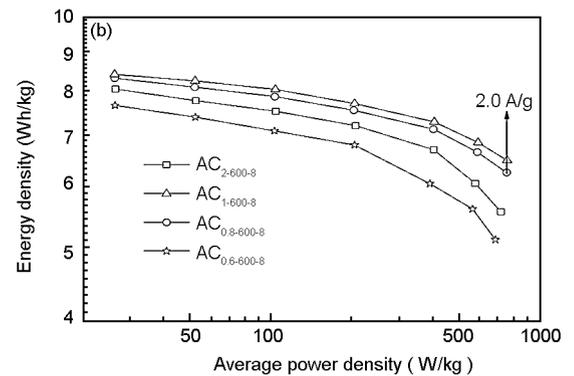
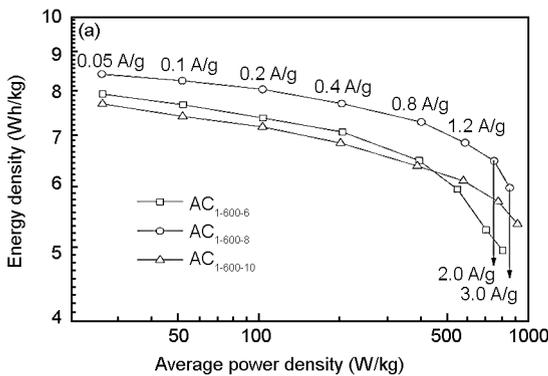


Fig. 4 Energy density of AC capacitors vs. average power density: (a) ACs made at different activation times, (b) ACs made at different KOH/peanut shell mass ratios.

Fig. 4a, b demonstrate that energy density of AC capacitors decreases with increasing the power density for all AC capacitors, which suggest that less energy are released at higher power output. At lower discharge current density of 0.05, 0.1, 0.2, 0.4 and 0.8 A/g in Fig. 4a, the energy density of AC₁₋₆₀₀₋₁₀ capacitor is nearly equal to that of AC₁₋₆₀₀₋₆ capacitor

under the same power density. However, the energy density of AC₁₋₆₀₀₋₁₀ capacitor at higher discharge current density of 1.2, 2.0 and 3.0 A/g is bigger than that of AC₁₋₆₀₀₋₆ capacitor under the same power density. The bigger D_{ap} of AC₁₋₆₀₀₋₁₀ electrode is responsible for the bigger energy density of AC₁₋₆₀₀₋₁₀ capacitor at higher discharge current density due to the faster ion

transport. Fig. 4a shows that energy density of AC capacitors reach a maximum with increasing the activation time from 6 to 10 min at the same current density. Fig. 4b also shows that energy density of AC capacitors reaches a maximum with increasing KOH/peanut shell mass ratio from 0.6 to 2.0 at the same current density from 0.05 to 2.00 A/g. Specifically, the energy density of AC₁₋₆₀₀₋₈ capacitor is the largest among all AC capacitors, as shown in Fig. 4a, b. The energy density of AC₁₋₆₀₀₋₈ capacitor decreases only from 8.41 to 5.98 Wh/kg with increasing the discharge current density from 0.05 to 3.00 A/g, indicating a retention rate of the energy density of 71.1% at the highest current density. The energy density retention rate for other AC capacitors at the highest current density ranges from 62.6% to 69.8%.

4 Conclusions

ACs were derived from peanut shell by KOH activation by microwave heating for only 6-10 min. The prepared ACs were used to prepare electrodes for ECs. The S_{BET} and V_t of ACs, specific capacitance of AC electrodes as well as energy density of AC capacitors all exhibit maxima with activation time from 6 to 10 min or with KOH/peanut shell mass ratio from 0.6 to 2.0. The S_{BET} of AC₁₋₆₀₀₋₈ reaches 1277 m²/g and AC₁₋₆₀₀₋₈ capacitor demonstrates a high cycle stability with an energy density of 8.38 Wh/kg even after 1000 cycles. KOH activation of peanut shell by microwave heating is found to be an efficient and straightforward approach to the production of low-cost ACs for ECs.

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7. Manuscript Submission; Online submission; <http://xxtcl.sxicc.ac.cn/EN/volumn/home.shtml>