

Preparation of activated carbon from cotton stalk and its application in supercapacitor

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Abstract High specific capacitance and low cost are the critical requirements for a practical supercapacitor. In this paper, a new activated carbon with high specific capacitance and low cost was prepared, employing cotton stalk as the raw material, by using the phosphoric acid (H_3PO_4) chemical activation method. The optimized conditions were as follows: the cotton stalk and activating agent with a mass ratio of 1:4 at an activation temperature of 800 °C for 2 h. The samples were characterized by nitrogen adsorption isotherms at 77 K. The specific surface area and pore volume of activated carbon were calculated by Brunauer–Emmett–Teller (BET) and *t*-plot methods. With these experimental conditions, an activated carbon with a BET surface area of 1,481 cm^2g^{-1} and micropore volume of 0.0377 cm^3g^{-1} was obtained. The capacitance of the prepared activated carbon was as high as 114 Fg^{-1} . The results indicate that cotton stalk can produce activated carbon electrode materials with low cost and high performance for electric double-layer capacitor.

Keywords Supercapacitor · Activated carbon · Cotton stalk · Specific area · H_3PO_4 activation

Introduction

It is well known that fossil fuel-based energy causes lots of economic and environment problems in the world. Growing

energy demand, depleting resources of fossil fuel, and increasing environmental concerns, including the emission of greenhouse gases and local air pollutants, have driven the search for developing alternative energy storage with high power and energy density. Supercapacitor is the most promising electrochemical energy storage device because of its higher power density and longer cycle life compared with the secondary battery. It has been widely used in the information technology industry, such as electronic devices, electric vehicles, military equipment, and high power energy storage devices with an ever decreasing size [1–4].

One of the most important components of a supercapacitor is the electrode. For electric double-layer capacitor (EDLC) electrodes, the common materials are metal oxides, polymers, and porous materials such as activated carbon and carbon aerogel. Moreover, new carbon materials, such as carbon nanotube, have been developed as electrode material, but there are difficulties in practical application due to their complicated preparation and high cost [5]. Compared with other carbon materials, activated carbon is more suitable as supercapacitor electrode material because of its high specific surface area and low cost. In addition, using biomass waste materials to prepare activated carbons is becoming a trend in the preparation of electrode materials for EDLC. Many carbon electrodes of EDLC obtained from biomass materials, such as nutshell [6], banana fiber [7], firewood [8, 9], corn grain [10], bamboo [11], rice husk [12], sunflower seed shell [13], cherry stone [14], and fluff of chinar [15] have been reported.

Cotton is known as one of the most important agricultural commodities in China, which is used to make a number of textile products. As the largest cotton-planting nation in the world, China has an average of 533,300 ha^2 of cotton. Cotton stalks are discarded by cotton planters after picking the cotton, weighing 3,375 to 3,750 kg ha^{-2} and amounting to 1.53 million tons in China every year [16, 17]. As a kind

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of agricultural wastes, they are directly burned as fuels in the countryside. The rest is dumped in the field, which causes great waste. The discarded cotton stalks are rich in lignin so that it can be made into activated carbon that has a wide range of use and high additional value. This is an efficient way to utilize discarded cotton stalk resources.

Activated carbons with a wide variety of pore size distribution are obtained by modifying the preparation conditions of either a physical or chemical activation process. In both methods, there is a reaction of the precursor with the activating agent (H_3PO_4 , ZnCl_2 , KOH , etc.) to develop porosity, but they differ not only in practical procedure but also in the mechanism by which the activating agent develops such porosity [18, 19]. Previous investigations proved its effectiveness in producing excellent activated carbon from cotton stalks [20–23]. As is well known, the chemical constitution of the H_3PO_4 solution used with preparative purposes is a very sensitive property to the increase in its concentration [24]. The activation temperature is one of the factors that affect the performance of active carbon. Figure 1 illustrates the changes in weight as a function of heat for both the cotton stalk precursor and its H_3PO_4 -treated sample. A comparison between the two tracks indicates that the impregnation of phosphoric acid delayed the main thermal degradation of the raw material. The whole pyrolysis process could be divided into three stages. The first stage occurs between ambient temperature and 600 °C, and a minor weight loss observed is mainly due to H_2O release by evaporation and the sample decomposed to uncondensable gas (CO , CO_2 , CH_4 , H_2) [25]. The second stage is in the temperature range of 600–800 °C and the strongest weight loss observed is attributed to the thermal decomposition of the raw material; these reactions are accompanied by further chemical transformations that include dehydration, degradation, and condensation with a loss of aliphatic character and

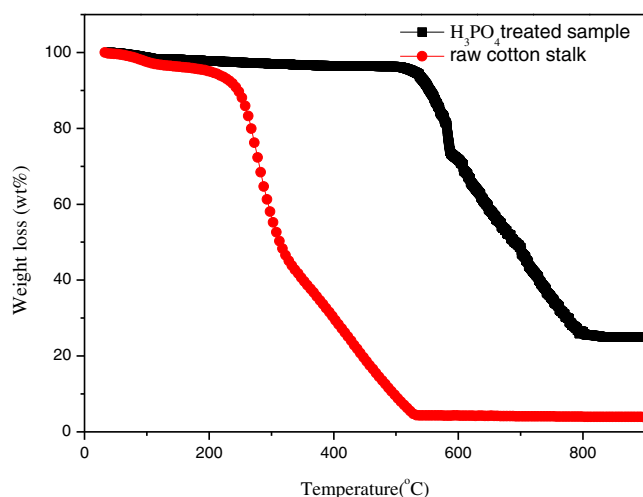


Fig. 1 Thermal gravimetric analyses of cotton stalk in N_2

a corresponding increase in aromaticity and simultaneous release of gases [20]. The third stage appears over a wide temperature range from 800 °C to the final temperature and a slight weight loss. It is indicated that the active sites on the carbons had reacted completely and H_3PO_4 on the botanical structure through the penetration and dissolving of some components by breaking chemical bonds; it is followed by recombination to form more thermally stable new polymeric and aromatic structures. So, the activation temperature of 800 °C is more perfect than others for the preparation of activated carbon from cotton stalk [21–23].

In this paper, cotton stalks were used as the raw material for preparing the supercapacitor's electrode material. The cotton stalk activated carbons (ACs) were modified by H_3PO_4 to improve their surface chemistry performances and pore structures. The influence of chemical activation on the capacitance of activated carbon electrode was observed, and the relationship between the surface chemistry characteristics, pore structures of modified ACs, and the capacitance were researched in order to obtain the highest capacitance.

Experiments

Preparation of activated carbon-based cotton stalk

The dried cotton stalk was pulverized to 200 μm after the powder samples were treated with an aqueous solution of H_3PO_4 (50 wt.%) for 12 h at a mass ratio of cotton stalk to activating agent (H_3PO_4) ranging from 1:2 to 1:5. Then, the mixture was dried, heated up to 800 °C under nitrogen atmosphere with a heating rate of 5 °C min^{-1} , and allowed to dwell in that temperature for 2 h, followed by cooling down to room temperature. The activated carbon products were washed sequentially with 0.5 molL^{-1} $\text{NH}_3\cdot\text{H}_2\text{O}$ to neutralize the excess H_3PO_4 compounds. The samples were washed repeatedly with distilled water until the pH of the washing solution reached 7. The final activated carbon samples were dried at 90 °C in a vacuum oven for 12 h and stored in desiccators. The samples were labeled as AC- x , (AC1, AC2, AC3, and AC4 are H_3PO_4 -to-carbon mass impregnation ratios 1:2, 1:3, 1:4, and 1:5, respectively). All chemicals were of analytical grade and used as received.

Preparation of electrode and supercapacitor

The electrochemical performances of all carbon samples were investigated by using CR2025 coin cell without a reference electrode. The electrodes were composed of activated carbon, carbon black as an electronic conductor, and polyvinylidene fluoride as a binder in a weight ratio of 80:15:5. They were blended in distilled water to form a

slurry. The mixed slurry was pressed onto aluminum foil current collectors (Φ 1.2 cm) and dried at 120 °C for 8 h to fabricate electrodes in vacuum oven. The cells (CR2025) were assembled in an argon-filled glove box. Two electrodes with identical or very close masses were selected and then assembled as supercapacitor. All of the measurements were carried out in 1.0 molL⁻¹ Et₄NBF₄ electrolyte. The supercapacitor was charged and discharged from 0 to 3.5 V on the charge/discharge apparatus (BTS-51, Neware, China). Cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) were tested using a CHI660 electrochemical working station.

Results and discussion

Raw material analysis

The proximate analysis of cotton stalk indicated that this raw material contained 44.47 % of fixed carbon, 5.60 % of hydrogen, 0.53 % of nitrogen, and 49.47 % of oxygen [16, 23]. The proximate analysis result shows that the carbon content of cotton stalk is higher than the other biomass resources. As comparison, the fixed carbon content of

several raw materials for activated carbon production is as follows: cassava peel waste—28.7 % [26], bamboo—16.60 % [27], rubber wood sawdust—23.38 % [28], and palm shell—18.70 % [29].

Compared with other materials, cotton stalk has a higher density of carbon and a larger weight ratio of active material to the capacitor with a certain volume. From the view of practical application, it is a potential raw material for the preparation of activated carbon.

Physical characterization

The physical characterization of activated carbon was conducted by using nitrogen adsorption method and scanning electron microscopy (SEM). Nitrogen desorption was carried out to determine the pore structure of the carbon and SEM analysis was conducted to observe the surface morphology of activated carbon samples. The pore characteristics of the obtained carbon were measured by a Micromeritics (ASAP2010) instrument in nitrogen at 77 K. Mesopore size distributions were determined by Brunauer–Emmett–Teller (BET) method. The micropore volume (V_{mic}) and the total pore volume (V_{tot}) were calculated from the amount of N₂ adsorbed at a relative pressure (p/p^0) of

Fig. 2 SEM image of the activated carbons: **a** AC1, **b** AC2, **c** AC3, **d** AC4

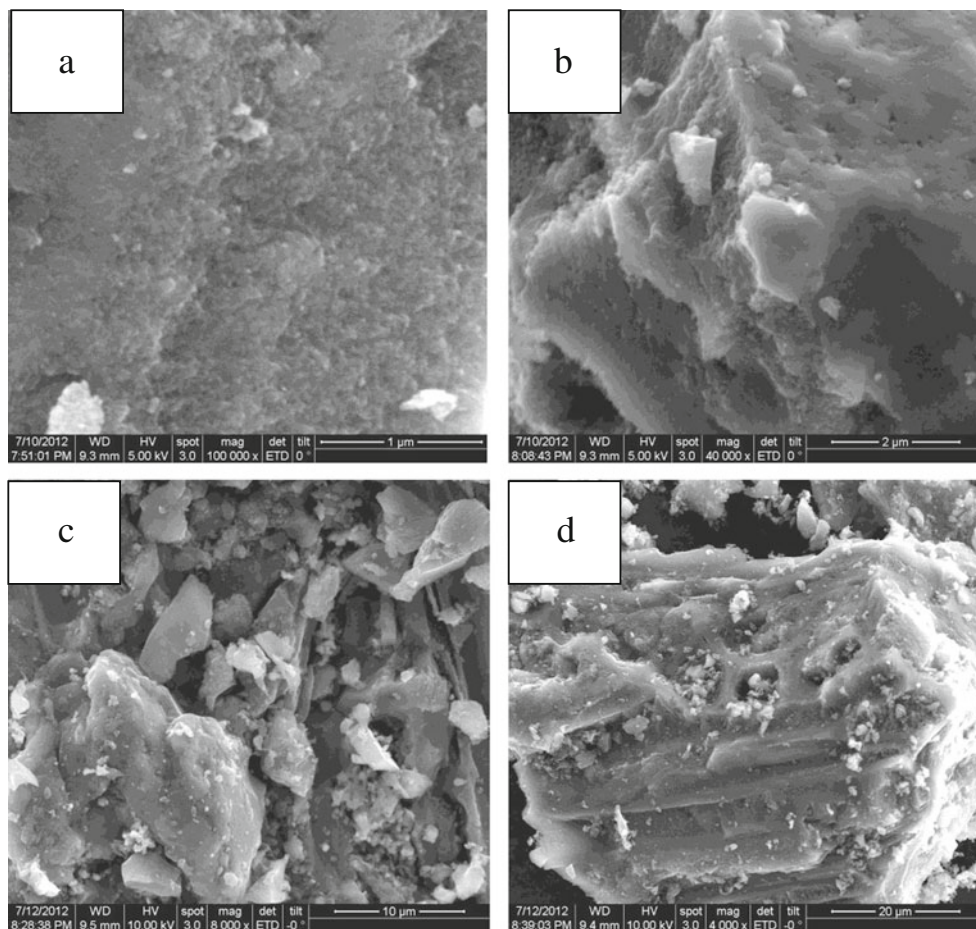


Table 1 Pore characteristic of modified activated carbons derived from cotton stalk

Sample	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	S_{mic} ($\text{m}^2 \text{g}^{-1}$)	V_{tot} ($\text{cm}^3 \text{g}^{-1}$)	V_{mic} ($\text{cm}^3 \text{g}^{-1}$)	$V_{\text{mic}}/V_{\text{tot}}$ (%)
AC1	1,392	86.70	0.904	0.0496	5.487
AC2	1,389	74.90	1.120	0.0584	5.214
AC3	1,481	44.54	1.210	0.0377	3.115
AC4	1,412	76.70	0.945	0.0518	5.481

10^{-5} and 0.95, respectively. Carbon was degassed at 473 K in a vacuum condition for a period of at least 24 h.

The SEM image of the samples demonstrated in Fig. 2 depicts the surface morphology of the activated carbons. It can be seen that a different surface morphology was observed for all activated carbons studied. They were changed by the addition of H_3PO_4 as a chemical activation agent. The surface modifications have a significant effect on the surface area and pore distribution of the activated carbons.

The effect of activation on pore characteristic

As known, the specific capacitance of electrode materials is related to the BET surface area and the pore structure. The pore characteristics of these samples are given in Table 1. It can be seen that the BET surface area of activated carbon derived from cotton stalk was higher than peanut shell-based microporous carbon ($726 \text{ m}^2 \text{g}^{-1}$) [30] and commercial activated carbons available in Indonesia ($500\text{--}700 \text{ m}^2 \text{g}^{-1}$) [26]. These pore structures are the combination of micropore and mesopore volumes, which is supported by the pore distribution of activated carbons determined by BET and t -plot method. Figure 3a shows the nitrogen adsorption isotherms of these samples at 77 K. This result indicates that H_3PO_4 activation greatly enhanced the pore development during the chemistry activation process. Compared with other samples, the nitrogen adsorption isotherms of AC3 have an evident type IV isotherm; a hysteresis loop can be observed in the adsorption/desorption isotherms, indicating the existence of mesopore size in modified activated carbons after H_3PO_4 activation. For comparison, the pore size of AC3 is shown in Fig. 3b, and it is clearly indicated that the pore volume of AC3 is the larger among all of the samples.

The electrochemical capacitance has a close correlation with the pore diameter distribution based on the “double electric layer” energy storage mechanism [30, 31]. We can draw the conclusion that the pores with their diameter distribution from 2 to 3 nm have the most important contribution to the electrochemical characteristic of samples in our system.

AC3 has the highest total pore volume and mesopore volume among all the ACs investigated. The peak pore size of AC3 centered at 3 nm. Comparing with other sample contents of mesopores, AC3 has a larger specific surface

area and micropore volume, which are the main factors for higher specific capacitance.

Electrochemical measurements

The charge–discharge curves of ACs between 0 and 3.5 V at the current density of 0.5 A g^{-1} are shown in Fig. 4. It can be seen that no obvious voltage drop can be observed at the beginning of the discharge process for the electrode, and the charge–discharge curves at the current density of 0.5 A g^{-1} were regularly of triangular shapes, showing better capacitor

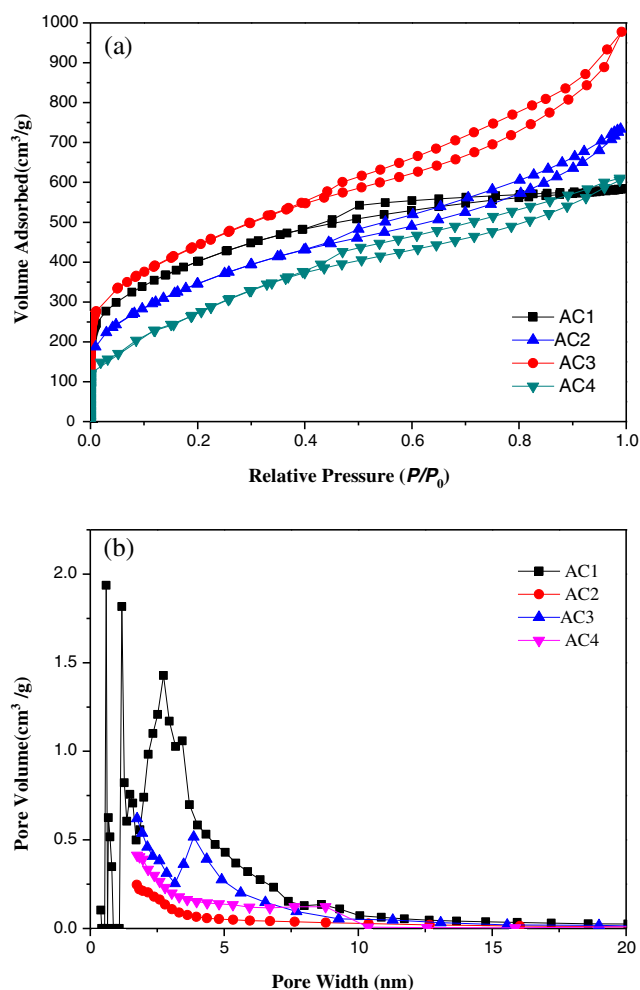


Fig. 3 **a** Nitrogen adsorption isotherms of modified activated carbons derived from cotton stalk. **b** Pore size distribution of modified activated carbons derived from cotton stalk

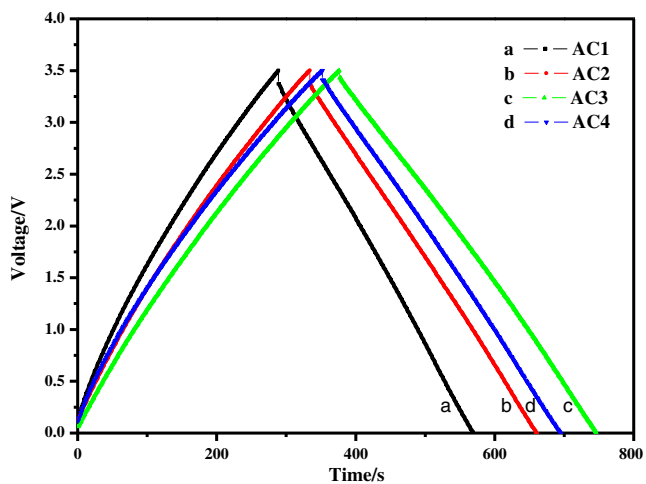


Fig. 4 The charge/discharge curves of the samples AC1, AC2, AC3, and AC4 at 0.5 Ag^{-1}

behavior for this material. The initial voltage drop observed in the charge–discharge profiles indicated that the electrochemical impedance is relatively small. It was also found that the specific capacitance of AC3 is significantly larger than those of other samples at the same current densities, and it is the best electrode material in the samples. Besides, it can be seen that a drastic change in the capacitance value occurred when the current density increased from 0.5 to 2 Ag^{-1} (shown in Fig. 5). This can be easily understood as follows [32]: at a low current density, the ion can be transported and diffused into the pores easily. Hence, the capacitance is higher. However, when the current density increases, the ion cannot be easily diffused into the pores, which results in such a way that the effective ion attachment occurred only at the surface of the electrode. As a result, the capacitance decreases. Based on the evaluation in the entire experimental result, the highest capacitance value that can be obtained was AC3. Its capacitance value is as high

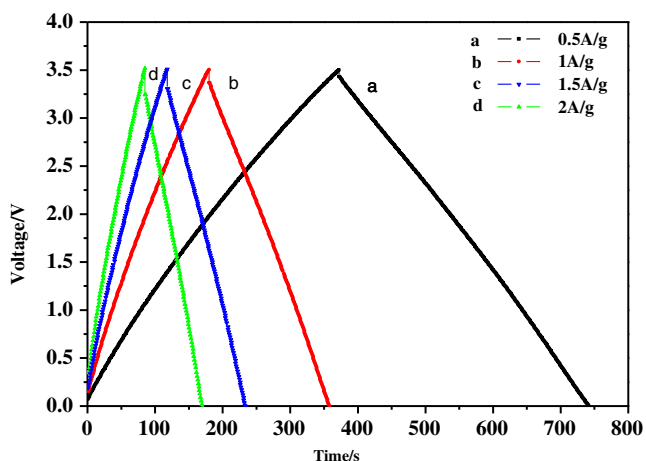


Fig. 5 The charge/discharge curves of the samples AC3 at different current densities

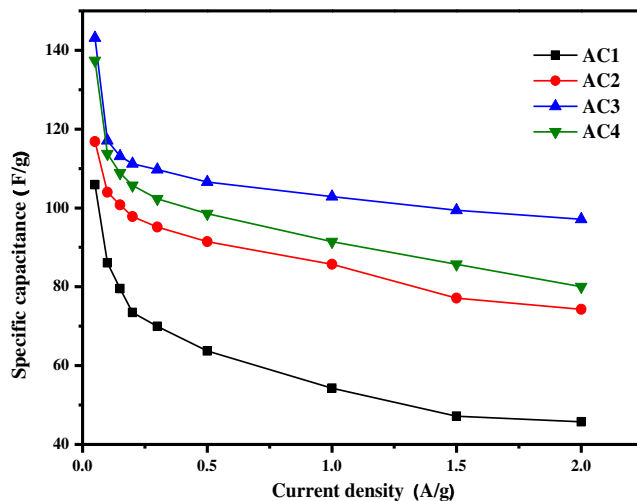


Fig. 6 Discharge-specific capacitance of supercapacitors at different current densities

as 114 and 98 Fg^{-1} at a current density of 0.5 and 2 Ag^{-1} from Fig. 6, respectively. Actually, this capacitance value is relatively higher than those of other electrode materials for supercapacitor application from a biomass precursor [33, 34]. Owing to its simple preparation process and the availability of the simple source in a large scale, activated carbon from cotton stalk exhibits excellent electrochemical performance especially at high current density.

The specific capacitance C of the electrode can be calculated by the following equation [35–37]:

$$C = (2 \times I \times \Delta t) / (m \times \Delta V) \tag{1}$$

where Δt is the discharge time, ΔV is the voltage change during discharge, I is the discharge current, and m is the mass of active material on one electrode.

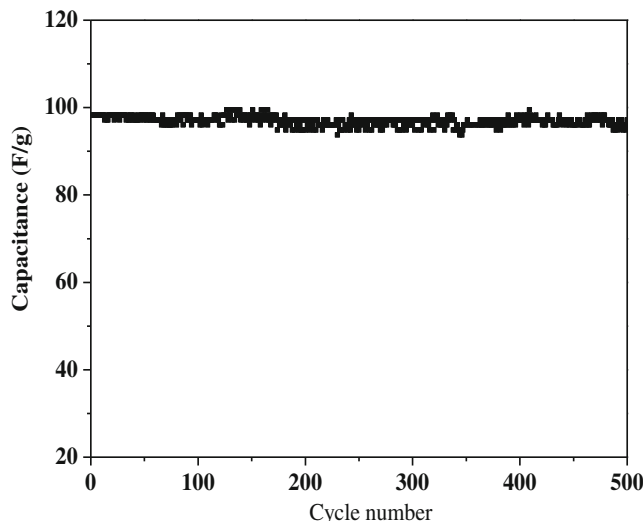


Fig. 7 Charge–discharge cycling stability of AC3 at a current density of 2 Ag^{-1} in $1 \text{ molL}^{-1} \text{ Et}_4\text{NBF}_4$ electrolytes

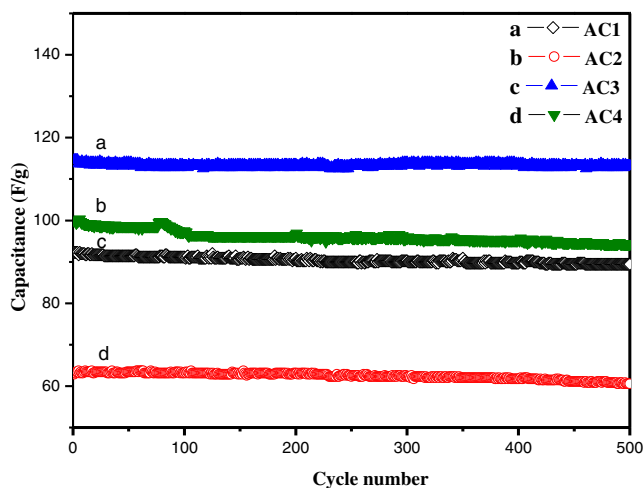


Fig. 8 Charge–discharge cycling stability of the AC electrode materials for 500 cycles at a current density of 0.5 A g^{-1} within the potential window range from 0 to 3.5 V

For further understanding of the electrochemical performances, the cyclic charge–discharge tests were performed at a fair current density of 2 A g^{-1} in 1 mol L^{-1} Et_4NBF_4 electrolytes, and the corresponding results are shown in Fig. 7. As shown in Fig. 7, the specific capacitance of AC3 in 1 mol L^{-1} Et_4NBF_4 electrolyte is up to 98 F g^{-1} at 2 A g^{-1} and keeps remarkably stable over 500 cycles, which shows that the high capacitance retention ratio is 95.3 %. These results demonstrate that the activated carbons derived from cotton stalk exhibit better cycle stability and a very high degree of reversibility during repetitive charge/discharge cycles in organic electrolytes. Figure 8 demonstrates that the AC materials have long-term electrochemical stability, but the AC3 electrode has higher capacitance and better electrochemical stability than the other materials,

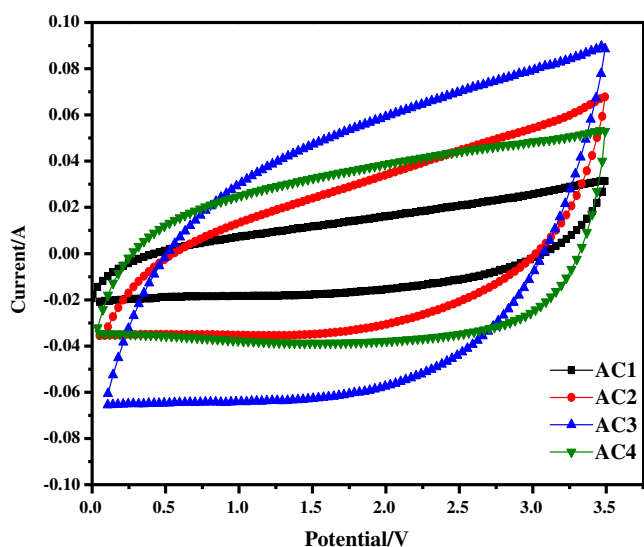


Fig. 9 Cyclic voltammogram of AC electrodes with a scanning rate of 50 mV s^{-1}

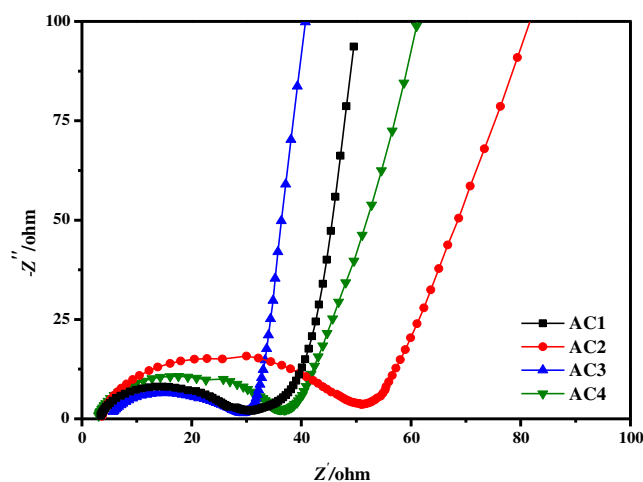


Fig. 10 The impedance spectroscopy of AC electrodes

which proves again that AC3 can be used as electrode material.

The CV measurement was performed from 0 to 3.5 V with a scanning rate at 50 mV s^{-1} . As shown in Fig. 9, all curves have similar and symmetric rectangular shapes between positive and negative scanning rate, indicating the good reversibility characteristic of EDLC during charge and discharge process of carbon electrode. The cyclic voltammogram of AC3 had a broader area compared with other electrode materials, indicating that the capacitance of the sample AC3 is higher than those of others. This evidence supports the result of the galvanostatic charge–discharge experiment.

The EIS analysis has been one of the effective methods in examining the fundamental behavior of electrode materials for supercapacitor. It was performed by Nyquist plots at an applied potential of 3.5 V and frequency range between 10^{-2} and 10^5 Hz . As shown in Fig. 10, all the impedance spectra of these samples are almost similar, which contain a distorted semicircle in the high frequency region due to porosity of the electrode and a linear part at the low frequency region due to diffusion-controlled doping and undoping of anions that result from Warburg behavior. The high-frequency intercept in the semicircle with the real axis gives the internal resistance value of these cell capacitors. It is the sum of the resistance of the electrolyte solution, the intrinsic resistance of the active material, and the contact resistance at the interface active material/current collector. The internal resistance of AC electrode is estimated to be about 3Ω , which is the same for all samples. From the point of intersecting with the real axis in the range of low frequency, the AC3 resistance was estimated to be 30Ω for the activated carbon electrode, which is a little bit lower than those of other electrode materials, and the vertical line exhibits the domination of the capacitance behavior at the electrolyte/carbon interface. It can be deduced that the high electrochemical

performance of the AC3 in our work can be attributed to the predominant production of porosity material with high electrical conductivity.

Conclusions

Activated carbon has been prepared from cotton stalk. The H_3PO_4 activation processes on the samples have improved the physical and electrochemical properties of the active carbon, such as BET surface area, micropore volume, electrical conductivity, and specific capacitance. The sample AC3 has the highest BET surface area and micropore volume at a temperature of 800 °C with its value of $1,481 \text{ m}^2\text{g}^{-1}$ and $0.0377 \text{ cm}^3\text{g}^{-1}$, respectively. The capacitances of the samples were found to cause the electrical conductivity to increase linearly with increased activation mass ratio. The sample AC3 showed a supercapacitive effect with a capacitance value as high as 114 F g^{-1} at 0.5 A g^{-1} and 98 F g^{-1} at 2 A g^{-1} . Besides, it also displays a high-rate long-cycle electrochemical performance even at a current density of 2 A g^{-1} and a capacitance retention ratio of 95.3 %. Owing to the unique characteristic, the simple preparation, and the large availability of material, the cotton stalk will become one kind of potential material for supercapacitor electrode.

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