

PRODUCTION OF HIGH DENSITY ACTIVATED CARBON FIBER BY USE OF HOT BRIQUETTING METHOD

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Introduction

Activated carbon fiber (ACF) has high surface area and high adsorption-desorption rate, and its adsorption performance is superior to conventional activated carbon (AC) on weight basis. On volume basis, however, the adsorption performance of ACF is not superior to that of AC, because the bulk density of ACF (~0.1g/cc) is much smaller than that of AC (~0.5g/cc). To show the merit of ACF effectively, it is necessary to increase the bulk density of ACF by some means. Many attempts have been made to do so, including the preparation of formed ACFs with some binders. However, binders are likely to plug the micropores of ACFs, and decrease the merit of ACF significantly.

In this study, we presented a method to produce ACF of high bulk density (HD-ACF) without any binder. A stabilized carbon fiber was heated under the load of mechanical pressure to prepare successfully formed carbon fibers (CF) of different bulk densities. The formed CFs were carbonized and activated to prepare HD-ACFs. The adsorption performance of the HD-ACF was examined in detail, and the possibility of using the HD-ACF as electrode of electrical double layer capacitor (EDLC) was also examined.

Experimental

A fiber made of isotropic pitch was used as a raw material. It was oxidized in an air stream for stabilization. Two kinds of stabilized fibers, OF-A and OF-B were prepared by changing the heating rate and final temperature of stabilization. The ultimate analyses of the pitch fiber and stabilized fibers are listed in Table 1.

Figure 1 shows a schematic of apparatus used for preparing formed carbon fibers. Stabilized fibers were placed between the molds and heated by a so-called infrared-image furnace (Shinku-Riko, RHL-P610P) at the rate of 20K/min to a final temperature T_f under the load of mechanical pressure P . Formed carbon fibers were carbonized at 950 °C in a quartz tube reactor, then it was activated in a stream of 50% volume of H_2O at 900 °C to prepare HD-ACFs. The pore volume distributions of HD-ACFs were characterized by the nitrogen adsorption method

and mercury porosimetry. Figure 2 shows the assembly of the EDLC cell used. A separator made of glass wool was sandwiched by HD-ACFs (electrodes), SUS meshes (collector), and phenol-formaldehyde resin plates. The EDLC cell was charged under a constant potential (0.8V) between SUS meshes. After 10 min of charge, the EDLC was discharged for 10min. The currents of charge and discharge processes were recorded continuously, and discharge capacities were obtained by integrating discharge currents by time.

Results & Discussion

1) Production of HD-ACF

Figure 3 shows SEM photographs of formed OF-A and OF-B at $P=200\text{Kg/cm}^2$ and $T_f=400^\circ\text{C}$. Fibers of OF-B are highly packed without breaking or melting, whereas some parts of OF-A fibers were broken and melted. This is because the degree of stabilization of OF-A was lower than that of OF-B. The formed OF-B was a very rigid tablet of 17mm in diameter and 2 mm in thickness. The bulk density was about 0.9g/cc.

The bulk density of formed fiber could be changed by changing the loading pressure. It was also changed by changing the amount of fiber embedded between the molds but keeping the position of the molds fixed during the heating up to T_f . The latter method did not require the load of high pressure, and was suitable to change the density by keeping the dimension of the formed fiber constant. Using the latter method for forming the fiber, we could change the density of the HD-ACF from 0.2 to 0.86 g/cc. The HD-ACF produced had neither cracks nor carbon fines on it.

2) Pore distribution of the HD-ACF

Figure 4 compares the micropore distributions of the HD-ACF and the ACF prepared under same carbonization and activation conditions. Both distributions are almost coincident, indicating that the forming process does not affect the development of micropore. Only the macropore portions, spaces between fibers, were changed through the forming process as shown in Figure 5. The size and the volume of the interspace decreased with the

increase of bulk density. The void fraction of the interspace changed from 0.025 to 0.83 when the bulk density was changed from 0.86 to 0.2 g/cc. Thus we could change only the bulk density of HD-ACF without changing the adsorption capacity.

3) Application of the HD-ACF to the electrode of EDLC

The electrodes having same dimensions (15 mm ϕ and 0.7 mm thick) were prepared from the HD-ACFs having different densities, the ACF, and the ACF formed with a binder. The carbonization and activation conditions were same for these ACFs. Figure 6 compares the discharge currents of the EDLCs with the electrodes. The discharge currents of EDLCs with the HD-ACF electrodes were all higher than those with the ACF and ACF-binder electrodes. Using the HD-ACF ($d=0.86 \text{ g/cm}^3$) as the EDLC electrode, we can keep the discharge current at high level for more than 20 s. The total discharge capacity of the EDLC was 5 times larger than that of the EDLC with the ACF electrode. This means that we can make the size of EDLC significantly smaller using the HD-ACF as the electrode, indicating that the HD-ACF is suitable material to the EDLC electrode.

Table 1 Ultimate analyses of pitch and stabilized fibers [wt%]

	C	H	N	O
PF	92.2	5.6	1.7	0.5
OF-A	83.9	3.8	2.1	10.2
OF-B	71.6	2.2	2.1	24.0

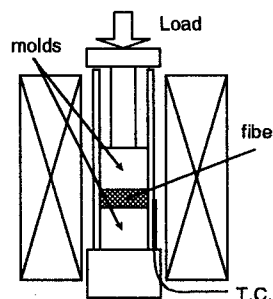


Fig.1 Schematic of apparatus used for hot briquetting

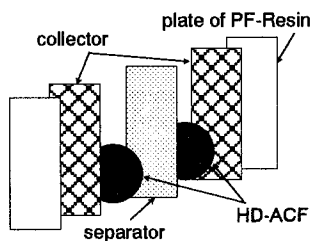


Fig.2 The assembly of EDLC used



OF-A



OF-B

Fig.3 SEM photographs of formed OF-A and OF-B

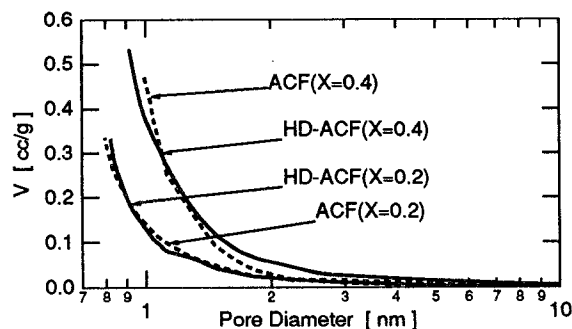


Fig.4 Micropore distributions of HD-ACFs

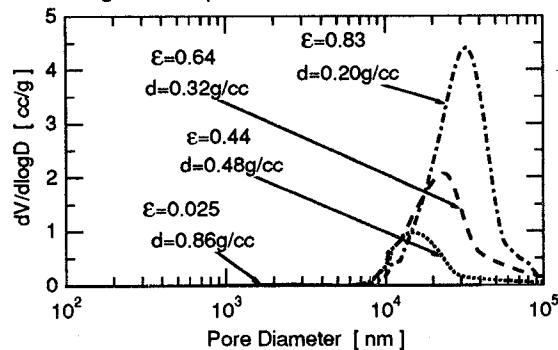


Fig.5 Macropore distributions of HD-ACFs

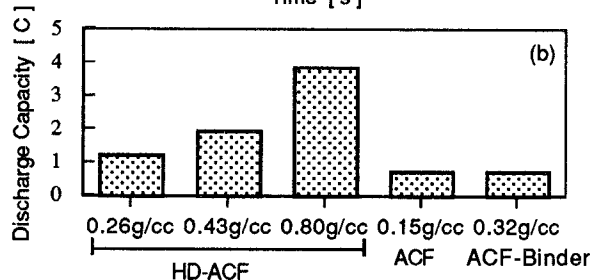
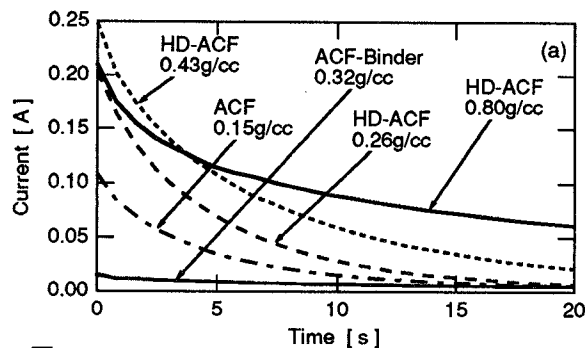


Fig.6 Discharge currents (a) and capacities (b) of HD-ACFs

Acknowledgements

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