

# Activation of mesocarbon microbeads with different textures and their application for supercapacitor

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## ABSTRACT

Three kinds of mesocarbon microbeads (MCMBs) with different textures were activated by potassium hydroxide at 900 °C and used as electrode materials for supercapacitor. The effects of textures of precursors on electrochemical performances of activated MCMBs were investigated. Nitrogen sorption measurements (at 77 K) showed that three kinds of activated MCMBs possess high specific surface areas (>2000 m<sup>2</sup>/g) and different porosity characteristics. MCMB prepared by emulsion method from bulk mesophase pitch (MCMB-e) has an irregular and distorted lamellar structure of oriented aromatic hydrocarbons. The unique texture of MCMB-e leads to the largest specific surface area (2542.8 m<sup>2</sup>/g) and the highest micropore volume (0.8236 cm<sup>3</sup>/g) after activation. Galvanostatic charge–discharge results showed that the activated MCMB-e has the highest specific capacitance of 326 F/g at the current density of 20 mA/g and better rate capability in 6 M KOH electrolyte. The good capacitive behavior of the activated MCMB-e may be attributed to the high-surface area, abundant micropores, closed-packed mesopores and macropores, as well as moderate crystal structures.

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## 1. Introduction

Supercapacitor, as one of new energy storage elements, has been extensively developed for the application in portable electronic systems, hybrid electric vehicles and industrial power managements, due to its high power density and long cycle life [1,2]. In recent years, efforts in searching better electrode materials of supercapacitor have attracted considerable attention. Among various candidates' materials, activated carbons are mostly investigated due to their large surface areas, good electric conductivity, excellent chemical stability, and relatively low cost [3–8]. The charge storage mechanism of activated carbon electrode is that electrolyte ions are accumulated at the electrode/electrolyte interface to form an electrochemical double layer. Therefore, the high-surface area, large pore volumes, and suitable surface functional groups have been considered to be the key factors for high specific capacitance of activated carbon [9,10]. With deepening of the relevant research, it is recognized that the appropriate pore structure is indispensable for ideal supercapacitor electrode materials, besides the high-surface area and large pore volume [11]. Several earlier studies have shown that high-surface-area carbon materials containing mesopores (>2 nm), which facilitate the electrolyte ion diffusion in the material, are highly desirable as supercapacitor electrode [3,12–14]. However, some researchers have recently found the important role of micropores in the enhancement of capacitance. These works lead to a better understanding of ionic

transport in narrow micropores [15–17]. In addition, Xia et al. have reported that hierarchical porous carbons with controlled micropores and mesopores displayed great potential for supercapacitor applications [11]. Hence, appropriate pore structure (pore size distribution) is necessary for carbon electrode to obtain high specific capacitance.

The pore structures of activated carbon depend not only on the activating agent and activation conditions, but also on the nature of precursors [18]. We have reported that mesocarbon microbead (MCMBs) was used as precursor to prepare the activated carbon with different pore size distributions [19,20]. Recently, activated mesocarbon microbeads (AMCMBs) have been used as electrode materials of supercapacitor, which show much higher energy densities per both weight and volume than those of the conventional activated carbon with large power densities [21,22]. The good electrochemical performances are attributed to the high packing density and unique microstructures of precursor. MCMBs have various textures due to their different starting materials and preparation conditions, such as Brooks/Taylor type (latitude-like) [23], Honda type (longitude-like) [24], Kovac/Lewis type (similar longitude-like) [25], and Huttinger type (onion-like) structure [26], etc. Therefore, it is necessary to deeply investigate the relationships between the textures of MCMBs and pore structures of AMCMBs, and find an effective way to further improve their capacitive performance by controlling pore structures of activated carbon. In addition, the MCMB prepared by emulsion method (MCMB-e) is emphasized especially here because it has a relatively narrow particle size distribution, a high stability against chemical reagents, and unique microstructure which is different from the conventional MCMBs. MCMB-e with high ion-exchange capacity has been used as filler for a

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**Table 1**  
Properties of FCC-decant oil and bulk mesophase pitch.

Sample	Element analyses (wt.%)		SP <sup>a</sup> (°C)	AC <sup>b</sup> (vol%)	Solubility (wt.%)				Ash (wt.%)
	H	C			HS <sup>c</sup>	HI-TS <sup>d</sup>	TI-PS <sup>e</sup>	PI <sup>f</sup>	
FCC-decant oil	14.12	82.5	–	–	39.5	47.8	12.6	0.1	<0.5
Mesophase pitch	4.28	94.11	300	100	3.6	17.1	15.6	63.7	<0.5

<sup>a</sup> SP: softening point.

<sup>b</sup> AC: anisotropic content.

<sup>c</sup> HS: heptane soluble.

<sup>d</sup> HI-TS: heptane insoluble–toluene soluble.

<sup>e</sup> TI-PS: toluene insoluble–pyridine soluble.

<sup>f</sup> PI: pyridine insoluble.

liquid chromatograph [27–29]. However, there is no study on the activation of MCMB-e and using it as electrode materials in supercapacitor. Hence, it is particularly interesting to apply activated MCMB-e as electrode materials for supercapacitor.

In the present study, three kinds of MCMBs prepared by the different methods from the same starting materials, fluidized catalytic cracking (FCC) decant oil, are activated by KOH at the same conditions. The effects of textures of MCMBs on pore structures of activated MCMBs are investigated. The AMCMBs used as electrode materials for supercapacitor are studied to evaluate the relationships between their electrochemical performances and textures of their precursors.

## 2. Experimental

### 2.1. Preparation and activation of MCMBs

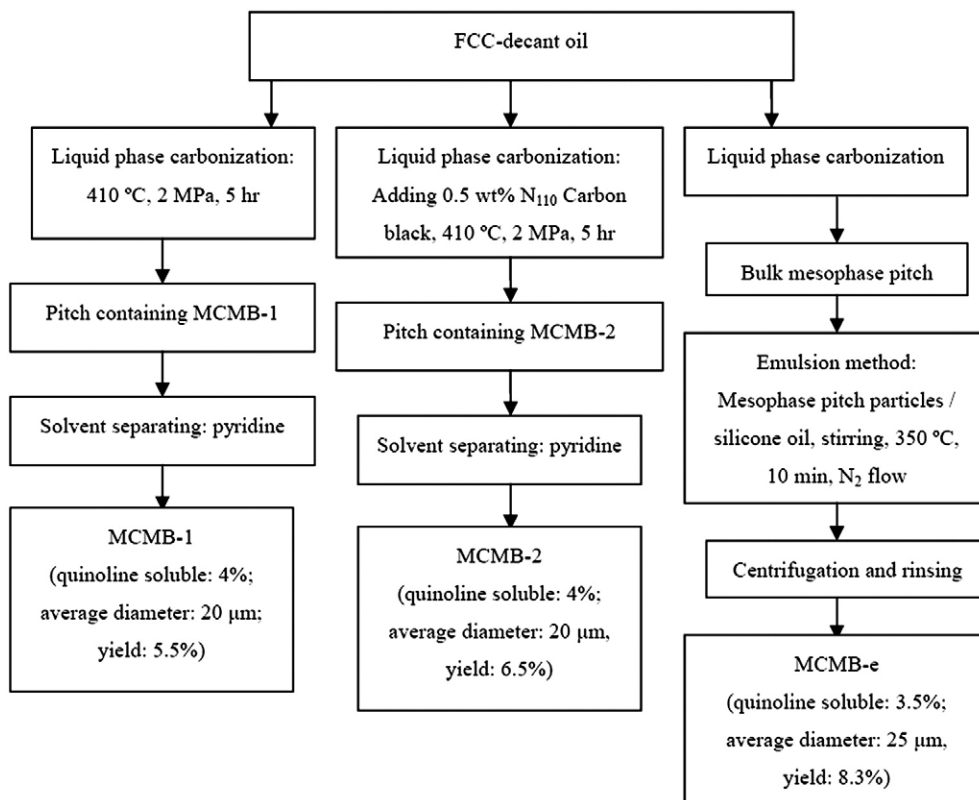
Three kinds of MCMBs were used as the precursors of the activated materials. MCMB-1 was formed during the liquid phase carbonization

of FCC-decant oil and was extracted by pyridine. MCMB-2 was from the same FCC-decant oil with adding 0.5 wt.% N<sub>110</sub> carbon black as nucleating agent. MCMB-e was prepared by emulsion method from bulk mesophase pitch (derived from the same FCC-decant oil). The properties of FCC-decant oil and bulk mesophase pitch are listed in Table 1. The schematic of preparation of MCMBs is shown in Fig. 1.

The chemical activation of MCMBs used KOH as the activating agent. The mass ratio of KOH to MCMBs was 5:1 [19,20]. After impregnated 12 h, the mixtures were heated at 110 °C in an oven to remove the water. Then, the dried materials were activated at 900 °C for 1 h under nitrogen flow. After cooled down, the resultant samples were impregnated in enough 2 M HCl solution for a few hours and later washed with a great deal of de-ion water to remove K and Cl ions. The washed samples were dried at 110 °C for 12 h to obtain the finally three activated products (AMCMB-1, AMCMB-2, and AMCMB-e).

### 2.2. Characterizations

To observe their texture features, MCMBs were mounted with epoxy resin, polished, and observed with the use of a Leitz polarizing microscope. Scanning electron microscope (SEM, HITACHI S-4700) was employed to analyze the morphology of those MCMBs and their activated products. Powder X-ray diffraction (XRD) patterns were recorded on a Rigaku B/max-2400 diffractometer using Cu K<sub>α</sub> radiation (40 kV and 200 mA, λ = 1.54 Å). Raman spectra were taken at room temperature using JY LabRAM HR800. The excitation source had a wavelength of 633 nm. The optical power at the sample position was maintained at 0.3 mW. Surface areas and pore structures of AMCMBs were obtained from N<sub>2</sub> gas adsorption isotherm measured at 77 K by using ASAP200M gas adsorption surface area analyzer according to Brunauer–Emmett–Teller (BET) and density functional theory method, respectively.



**Fig. 1.** Schematic of preparation of MCMBs by different methods.

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