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APPENDICE

APPENDIX A

Publication Resulting from This Research Work

PUBLICATIONS

Asian Proceeding

1. **Patompong Maneeprom**, Ukrit Kijsirichareanchai, Adisak Siyasukh, Siriporn Larpkiattaworn, Tawatchai Charinpanitkui, Nattaporn Tonanon. The generation of micropore and/or mesopore on 3D – interconnected macroporous carbon monolith by physical activation. Proceedings of Regional Symposium on Chemical Engineering 13th, pp. 99-100. December 3 – 5, 2006, Singapore, 2006.

The generation of micropore and/or mesopore on 3D – interconnected macroporous carbon monolith by physical activation

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ABSTRACT

The 3D - interconnected macroporous carbon monolith structure, which has a little porosity on its skeleton, has been successful prepared. The CO_2 and steam activation are used to develop this structure in order to increase the porosity. Nitrogen adsorption – desorption and SEM are used for characterization of the monolith is prepared in this work. The activation with steam shows higher micropore development than CO_2 activation. In steam activation, two step activation is more influential in microporosity development than one step activation. On the other hand, two step CO_2 activation is less powerful than one step CO_2 activation in microporosity development. After activation, the macroporoes are still preserved so that the porosity structure of activated RF carbon monolith exhibits the bi – modal (micropore – macropore) behavior.

1. INTRODUCTION

The 3D interconnected macroporous carbon monolith is prepared in the novel method by using ultrasonic technique in gel preparation process. The unique character of this structure allows flow paths through the monolithic column. The interconnected macropore is prepared from the novel method, exhibiting the distribution of size around 1 – 3 μm. However, the mesopore could not be observed and a few micropore is detected on carbon skeleton. The interconnected carbon materials have potential to be candidates for applications such as column for HPLC, catalyst supports and electrode of electric double layer capacitor (EDLC) [1]. However the mentionable applications require porosity in skeleton.

Activation is an essential method for improving surface properties of porous materials. Physical and chemical activation are used for modifying surface as well as increasing porosity of porous materials. CO_2, steam and air are widely used as activating agents in physical activation and activate in range 700 – 1100 °C [2]. One step and two step activations are widely employed as physical activation. In the two step activation, the starting materials are carbonized to produce char and the resulting char is activated with oxidizing agents but in one step activation, the carbonization and activation stages are combined into one step procedure [3].

The aim of this research is to generate and increase porosity in the range of nanoscale on the 3D interconnected macroporous carbon monoliths by using CO_2 and steam activations.

2. EXPERIMENT

2.1 Preparation of 3D interconnected macroporous carbon monoliths

This material is prepared by the sol – gel method which use resorcinol and formaldehyde as the precursors and apply ultrasonic irradiation during gelation state.

2.2 Physical activation

2.2.1 Two step activation

RF gel is carbonized in horizontal furnace to produce RF carbon and the resulting RF carbon is activated with oxidizing agents (CO_2 and steam). In the temperature ramp – up and cool – down stages, the nitrogen flow rate at 200 cc/min is used. The temperature rise with rate 10 °C/min to the desire temperature, the nitrogen is switched to activation agents for activation process.

In CO_2 activation, the CO_2 flow rate is fixed at 100 cc/min. In case of steam activation, steam and nitrogen (200 cc/min) are pre – mixed before feeding into reactor. The partial pressure of steam is fixed at 0.46.

2.2.2 One step activation

RF gel is activated with either CO_2 or steam under the activation conditions which are similar with two step activation.

2.3 Characterization

The porosity of activated RF carbon is determined by nitrogen adsorption – desorption at -196 °C. The scanning electron microscope (SEM) is used to investigate the morphology of RF carbon.

3. RESULTS AND CONCLUSION

The sample surface area (S_{BET}) is calculated by BET equation and micropore volume (V_{mic}) is determined by t – method as shown in Table 1. In activation with CO_2 and steam, it could be observed that microporosity is mainly developed on carbon skeleton. The activation with steam both two step and one step activation gave higher microporosity when compared with CO_2 activation.

Since the micropore volume in two step steam activation is higher than one step steam activation, larger BET surface area in two step steam activation than one step steam activation could be expected and then experimentally confirmed as shown in Table 1 (sample b and d). On the other hand, comparison of sample c and e in Table 1 reveals that two step CO_2 activation gives rise to less micropore volume than one step activation.

From the porosity results show in Table 1 together with the micrograph of interconnected macroporous structure in Fig. 1, it could be implied that micropore appears on the carbon skeleton. Therefore, the structure of interconnected macroporous carbon which passed the activation process exhibits in bi – modal (micropore – mesopore) behavior.

Table 1
The porosity of RF carbon

Sample	S_{BET} (m^2/g)	V_{mic} (cm^3/g)
a	522	0.23
b	1367	0.59
c	583	0.24
d	1065	0.51
e	729	0.34

RF carbon is activated at 850 °C for 1 h. Sample **(a)** RF carbon without activation, **(b)** two step steam activation, **(c)** two step CO_2 activation, **(d)** one step steam activation and **(e)** one step CO_2 activation

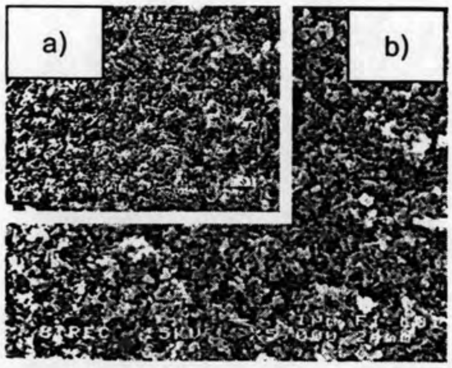

Fig. 1. SEM micrograph (cross section at 5000x) of interconnected macroporous structure **(a)** before activation and **(b)** after two step steam activation

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VITA

Mr. Patompong Maneeprom was born on December 27, 1982 in Songkhla, Thailand. In 2005, I received and got the second class honor in Bachelor's Degree of Science in major field chemical engineering in the faculty of science, Chulalongkorn University. My senior project studied about the adsorption of SO_2 on activation carbon is impregnated with KI. After that, I gained admission to Graduate School of Chulalongkorn University and graduated in 2007 with the thesis entitled "Preparation of micropore or mesopore on 3D interconnected macroporous carbon monoliths by physical and chemical activation".