

MIXTURE ACTIVATION OF PAN FIBER BY KOH AND CO₂

Y.Z.ZHANG F. HE M.Z WANG and B.J.ZHANG
Institute of Coal Chemistry, Chinese Academe of Sciences
P.O. Box 165 Taiyuan, 030001, P.R.China

Introduction

Activated carbon fibers (ACF) sometimes are required to contain mesopores when they are used in some applications, such as electrode in secondary cell and double-layer capacitor, support for catalyst. The activation of carbonaceous materials by KOH has received considerable attention since it was proposed by Wennerberg(1). But most ACF made by this method are microporous. In order to improve the mesoporosity in the ACF, the polyacrylonitrile (PAN) fiber was reactivated by CO₂ after heat-treated with KOH. The pore structure and adsorption properties of the resulted ACF were detected. It was found that the pore of the ACF was widened after reactivation and its adsorption of methylene blue was also increased, but the surface area was lightly decreased.

Experimental

The stabilized PAN felt was impregnated by KOH aqueous solution with certain concentration, the proportion of KOH in PAN fiber was determined by the weight gain of the felt after impregnation to the precious one. Then the felt was placed in a horizontal reactor. After raising the reaction temperature to a certain temperature under nitrogen flow, the felt was stayed for 10 minutes, then the CO₂ was passed through the reactor for a desired period of time. The felt was removed after the reactor was cooled to room temperature. The samples without CO₂ activation was also prepared⁽²⁾.

The surface area and the pore structure were investigated with an automatic volumetric sorption analyzer (ASAP2000) using nitrogen as adsorbent at 77K. The surface area and the pore size distribution were determined using the BET equation and the BJH method, respectively. The I₂ and methylene blue adsorption was determined following the method proposed by Chinese National Standards(GB7702.7-87 and GB7702.6-87, respectively).

Results and Discussion

The stabilized PAN fiber with 10.0wt% KOH was subjected to activation by CO₂ for 10min at different temperature. Both of the adsorption of I₂ and methylene blue of the resultant ACF rapidly increase with activation temperature (Fig.1). The similar situation occurs when precursor with 5.0wt% KOH was activated by CO₂ at 850 °C for different periods of time (Fig.2). The adsorption of obtained ACF increase with the activation time or decrease with the yield. When the yield is 5.4%, the I₂ adsorption and methylene blue of ACF is 1311mg/g, 264mg/g respectively. The nitrogen adsorption isotherm of the ACF is shown in Fig. 3 labeled as line B. One of the ACF activated only by KOH (33.9wt%) at 800 °C for 10min is shown in Fig.3 labeled A at the same time. Both lines increase with relative pressure at the begin then reach a platform indicating the existence of many micropores in these samples. But compared to the line A, line B increased slowly at low relative pressure. The pore size distribution size in Fig.4 shows that there are many micropores near 2.0nm in sample B, but almost all the pores in sample A are less than 2.0nm. Sample A and sample B have the similar BET surface areas (1332m²/g and 1295 m²/g, respectively), but the latter has the larger pore volume(0.552ml/g and 0.634ml/g, respectively), which testify the pores in latter are larger than that in former one.

Before activated by CO₂, there are many micropores in PAN-based fiber and some of them may be enlarged or new mesopores may be developed when the fiber is reacted with CO₂, and the alkali and its compound act as catalyst during the part gasification.

Conclusions

The PAN-ACF with mesopores in 2.0nm can be produced by re-activated the PAN-ACF pretreated by KOH.

The adsorption properties of the PAN-ACF can be improved by prolonging the activation time and increasing the activation temperature.

References

1. Wennerberg A.N., US Pat. 3,624,004; 4,082,694

2. Zhang Y.Z., He F., Wang M.Z., Zhang B.J., Material Science and Engineering, (in chinese),1996,(1),1-5

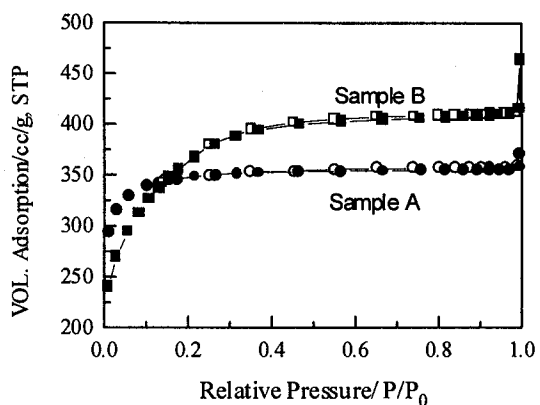


Fig. 1 N₂ Adsorption Isotherms of Sample A and B

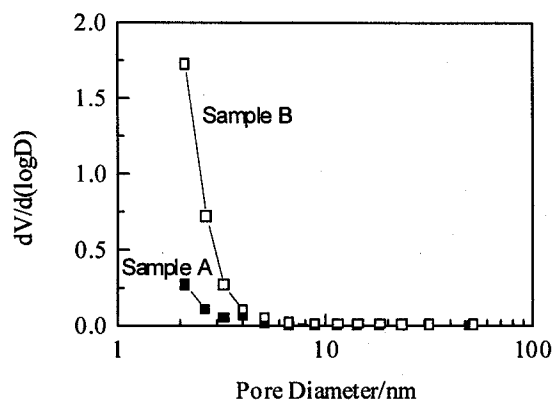


Fig. 2. Pore Size Distribution Curves of Sample A and B

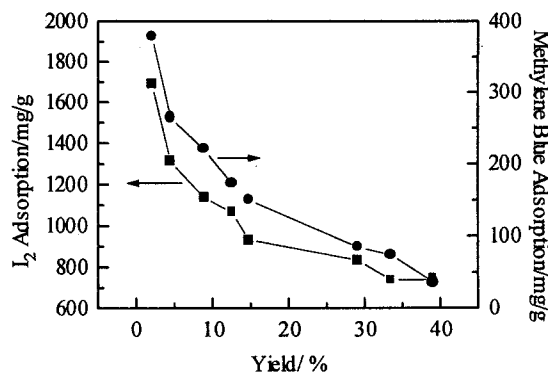


Fig. 3 Adsorption of Samples with Different Yields (KOH 5.0wt%, 850°C)

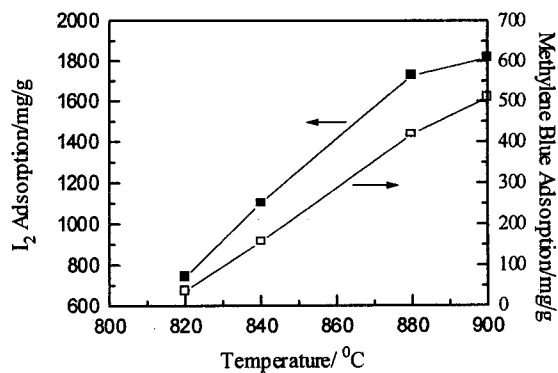


Fig. 4 Adsorption of Samples Activated at Different Temperature (KOH 10.0wt%, 10min)