PREPARATION AND EVALUATION OF DEODORANT ACTIVATED CARBON FIBER

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INTRODUCTION

Activated carbon fibers (ACFs) are unique in their extraodinary large specific surface area and enormous adsorptivity. In semiconductor industry, the removal of trace amounts of gas such as H_2S and NH_3 in the clean room for high integration of semiconductors has been recently required. An ACF, in which Mn is attached to only the surface, has been already commercialized by Nippon Chemical Industrial Co., Ltd., and its deodorant ability for H_2S has been improved in comparison with the conventional ACFs. However, the improvement is insufficient for the requirement mentioned above.

Oya et al. [1] have already been successful in preparing ACF introduced silver into the inside of its fiber. In order to improve the deodorant ability, we have tried to introduce Mn to not only the surface but also the inside of ACF. The purposes of the present work are to prepare phenolic resin-based deodorant ACF and to examine its structure and deodorant ability for H_2S. In addition, deodorant mechanism will be presented.

ERXPERIMENTAL

Spinnable phenolic resin (type: novolac) was supplied by Gunei Chemical Industry Co., Ltd. Manganese acetate ($Mn(CH_3COO)_2 \cdot 4H_2O$) was used as a manganese source. Both of the novolac and manganese acetate were thoroughly dissolved in methanol and Mn-containing novolac was obtained by removing methanol. The Mn-containing novolac was spun and the resulting Mn-containing phenolic resin fiber was cured by soaking it in an acidic formaldehyde solution supplied by Gunei Chemical Industry Co., Ltd. Infusible Mn-containig phenolic resin fiber (Mn-PF) was consequently obtained. Mn-containing carbon fiber (Mn-CF) was obtained by heating the Mn-PF to 900 °C and holding it for 30 min in a stream of N_2 gas. Finally , in order to prepare Mn-containing activated carbon fiber (Mn-ACF), the Mn-CF was activated in the stream of N_2 gas with a steam at 900 °C for 1 h. A commercial deodorant ACF (A-10S) fablicated by Nippon Chemical Industrial Co., Ltd. was employed for comparison of H_2S deodorant ability. The novolac based-ACF without Mn (Mn-free-ACF) was also prepared under the same condition as described above in order to examine the effect of Mn on the deodorant ability.

Mn content was determined by atomic absorption spectrometry. A H_2S deodorant test was performed with detector tubes for H_2S gas. After charging the samples prepared into columns, H_2S gas of 30 ppm diluted with air was made to flow continuously through the columns. The ratio of H_2S removed by the samples prepared was determined by measuring the H_2S concentration in column outlet at 15, 30, 60, 120, 180, and 240 min, respectively, after the injection of the H_2S gas. The dispersion state and crystal structure of Mn particle was examined with a transmission electron microscope (TEM), and X-ray and electron diffraction (XRD and ED) patterns. The distribution of Mn particles on the Mn-ACF surface was examined by energy dispersive X-ray analyzer (EDX). In order to clarify the effect of Mn content on the deodorant ability , the N_2-BET specific surface areas and the pore size distributions of the fibers prepared were analyzed.

RESULTS AND DISCUSSION

Mn contents and N_2-BET specific surface areas of the fibers prepared are

summarized in Table 1. The Mn content of the Mn-ACF is much higher than that of A-10S. Afterword, we have been successful in preparing an ACF in which Mn content is more than 10 wt%. The preparation method in this study facilitates the introduction of relatively large amounts of Mn to the phenolic resin-based ACF. The N_2-BET specific surface area of the Mn-CF increases markedly after the activation process. The specific surface area of the Mn-ACF is comparable to those of conventional ACFs.

H_2S deodorant characteristics of the ACFs and Mn-CF are given in Figure 1. The Mn-ACF maintains the H_2S removal ratio of about 60% even at 240 min and is proved to be superior to A-10S in terms of the H_2S deodorant ability. The ACF is inferior to the Mn-CF, which has a low specific surface area, in terms of H_2S removal ratio. This indicates that Mn introduced is important in improving the deodorant ability for H_2S. However, the deodorant ability of the Mn-CF is not so good as that of A-10S, which has relatively low Mn content and high specific surface area. This demonstrates that large specific surface area is also important in improving H_2S deodorant ability. Both high Mn content and large specific surface area are thus expected to be required for the achievement of the utmost H_2S deodorant ability.

Any definitive diffraction peaks concerning Mn were not recognized in the Mn-CF and Mn-ACF XRD profiles. It can be assumed that the peaks could not be detected because of the lowness of Mn content. Actually well-defined XRD peaks in Mn high-content ACF (Mn content: 11.54 wt%) were observed around 35, 40, and 59°, which corresponds to (111), (200), and (220) diffraction peaks of ß-MnO, respectively. The ED pattern of the Mn-ACF showed (111) and (200) diffraction patterns of ß-MnO. For the Mn-CF, particles with about 100 nm diameter appeared only rarely. On the other hand, particles with about 200 nm diameter were observed for the Mn-ACF. This suggests that the particles are formed through the carbonization process and aggregate to become larger particles through the activation process. Moreover, EDX demonstrated that the particles were also present on the Mn-ACF surface and almost homogeneously distributed on the surface.

CONCLUSIONS

Mn-ACF prepared in this study has been found to show an excellent deodorant ability for H_2S in comparison with conventional ACFs. MnO particles are formed in the fiber and play an important role in improving the deodorant ability.

REFERNCES

1. A.Oya, T.Wakahara, and S.Yoshida, *Carbon*, 31, 1243(1993).

Table 1 - Properties of the fibers prepared

	Mn content[a] (wt%)	S_{BET}[b] (m^2g^{-1}))
Mn-free-ACF	0	1500
Mn-CF	0.52	270
Mn-ACF	0.84	1380
A-10S	0.005	1100

a) From atomic absorption spectrometry.
b) N_2-BET specific surface area.

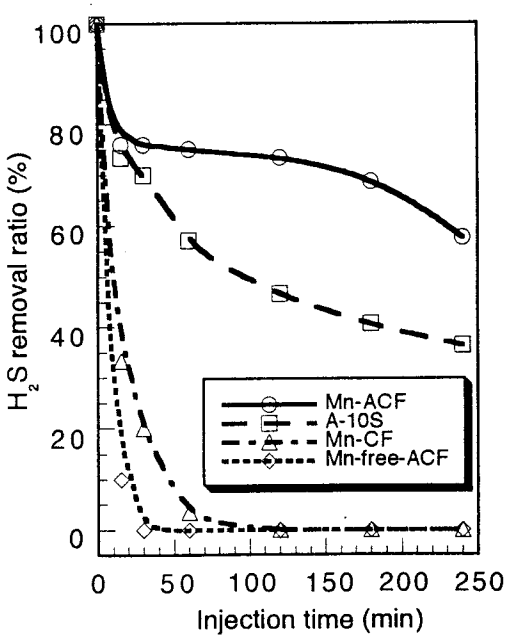

Figure 1 - H_2S removal abilities of the fibers prepared.