Silver loading effect for the activated carbon fibers pre-treated with acid

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1. Introduction

Functional groups on the surface of microporous carbons are most likely subjected to a wide variety of inter- and intra-molecular interactions. This can be used in the preparation of carbon supported metal catalysis by exchange with cationic metal complexes. In the related literatures [1,2], the influence of surface functional groups on the activity of carbon-supported catalysts has been clearly shown. The modification of surface chemistry resulted in a significant change of the loading capacity and of the catalytic properties [3]. Metal treated activated carbon is a material having adsorption and catalysis; these are of interest in several areas including medical applications, or water and air treatment for the catalytic removal of organic and inorganic pollutants and antibacterials. Especially, silver treated activated carbon by impregnation method is well known for its bacterial effects [4,5]. In this paper, which is the first part of a study on the Ag-activated carbon fiber system pretreated with various sulfuric acids, the purpose is to investigate the effects of physical and textural change of activated carbon fiber, and chemical treatment sequence.

2. Experimental

Self-made activated carbon fiber used as a non-treated carbon fiber material was prepared from PAN based carbon fiber. The molar concentration of from 0.01 to 0.1M diluted sulfuric acid at boiling temperature was used in the oxidation treatment to increase the formation of functional groups without the damage of the activated carbon fiber surface. We prepared series of solutions, mole concentrations of 0.1 M of $AgNO_3$,

for the metal loading effects. Nitrogen adsorption isotherms were obtained by using BET surface area apparatus (ASAP 2010, Micrometrics) at 77K. Scanning electron microscopy (SEM, JSM-5200 JOEL, Japan) was used to observe the pore structure of silver treated activated carbon fiber and the treated silver particles state on the carbon fiber surfaces. As one of the analysis of functional groups, FT-IR spectroscopy (FTS 3000MX, Biored Co.) was used to characterize of silver loading effect of Ag-activated carbon fibers. We used to Boehm titration method [6] for the identification of oxygenated surface group on the carbon fiber surfaces.

3. Results and discussion

The S_{BET}s and porous structure of the Ag-activated carbon fibers are summarized in Table 1. All of the Ag-ACFs gave Type I isotherms characterized by plateau that is nearly horizontal to the p/p_0 axis. The pore size distributions (PSD) calculated for Ag-ACFs using the H-J method are shown one major peak 0-20 Å, which is located in the micropore ranges. Increasing amount of acid treatment leads to a decrease in S_{BET}s and external surface area. But, micropore volume and average pore diameter are presented in constant regular values with increasing amount of sulfuric acid treatment. The FT-IR spectra of a Ag-activated carbon fiber pre-treated with sulfuric acid treatment are shown in Fig. 1. Observation of the absorption bands shows that the changes between the oxidized (acid treatment) and non-oxidized (non treatment) carbon fiber samples are mainly due to the formation functional groups. The most characteristics changes are observed at 1380 and 1719 cm^{-1} of the presence of C-O- and N-O- containing structures. The band centered at 1719 cm^{-1} is ascribed to the stretching vibrations of carboxyl groups on the edges of layer planes or to conjugated carbonyl groups. The weak band appearing at 1380 cm^{-1} is ascribed to the formation of oxygen function groups like a highly conjugated C=O stretching in carboxylic groups, and carboxylate. The band observed at 2360 cm^{-1} is usually ascribed to the presence of aliphatic compounds. The type and quality of oxygen groups are determined with Boehm titration method. The results obtained from the method proposed by Boehm are listed in Table 2. It can be observed that the total acidity and the distributions of groups of various strengths have very different values.

4. Conclusion

The adsorption isotherms of N_2 at 77K onto the metallic silver treated activated carbon fiber samples pre-treated with sulfuric acid are Type I. The IR spectra of Ag-activated carbon fiber samples show that the acid treatment is consequently associated with the

homogeneous distribution of metal with the increased surface acidity of the activated carbon fibers. A positive influence of the acidic groups on the carbon fiber surface by acid treatment is also demonstrated by an increase in the contents of metallic silver with increasing of acidic groups calculated from Boehm titration.

References

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Table 1. Comparison of physical parameters of metallic silver supported activated carbon fibers after sulfuric acid treatment

Sample	Parameter			
	S_{BET}(m^2/g)	Micropore Volume (cm^3/g)	External Surface Area (m^2/g)	Average Pore Diameter (Å)
Ag $_{0.1}$-ACF	1923	0.554	527.7	17.39
0.01H_2SO_4-$Ag_{0.1}$-ACF	1924	0.568	411.6	17.13
0.03H_2SO_4-$Ag_{0.1}$-ACF	1881	0.574	475.9	17.17
0.05H_2SO_4-$Ag_{0.1}$-ACF	1845	0.570	442.3	17.17
0.07H_2SO_4-$Ag_{0.1}$-ACF	1730	0.544	355.8	16.95
0.1H_2SO_4-$Ag_{0.1}$-ACF	1686	0.511	396.9	17.27

Table 2. Number of Surface Species (meq/g) Obtained from Boehm Titration

Sample	Functional Group (meg/g)				
	Carboxylic	Lactonic	Phenolic	Acidic	Basic
$Ag_{0.1}$-ACF	1.01	0.52	0.90	2.43	1.31
$0.01H_2SO_4$-$Ag_{0.1}$-ACF	1.56	0.73	0.95	3.24	1.12
$0.03H_2SO_4$-$Ag_{0.1}$-ACF	1.66	1.32	2.87	5.85	1.22
$0.05H_2SO_4$-$Ag_{0.1}$-ACF	2.03	1.85	2.33	6.21	1.11
$0.07H_2SO_4$-$Ag_{0.1}$-ACF	1.95	2.01	3.88	7.84	1.13
$0.1H_2SO_4$-$Ag_{0.1}$-ACF	4.56	3.21	3.09	10.86	1.45

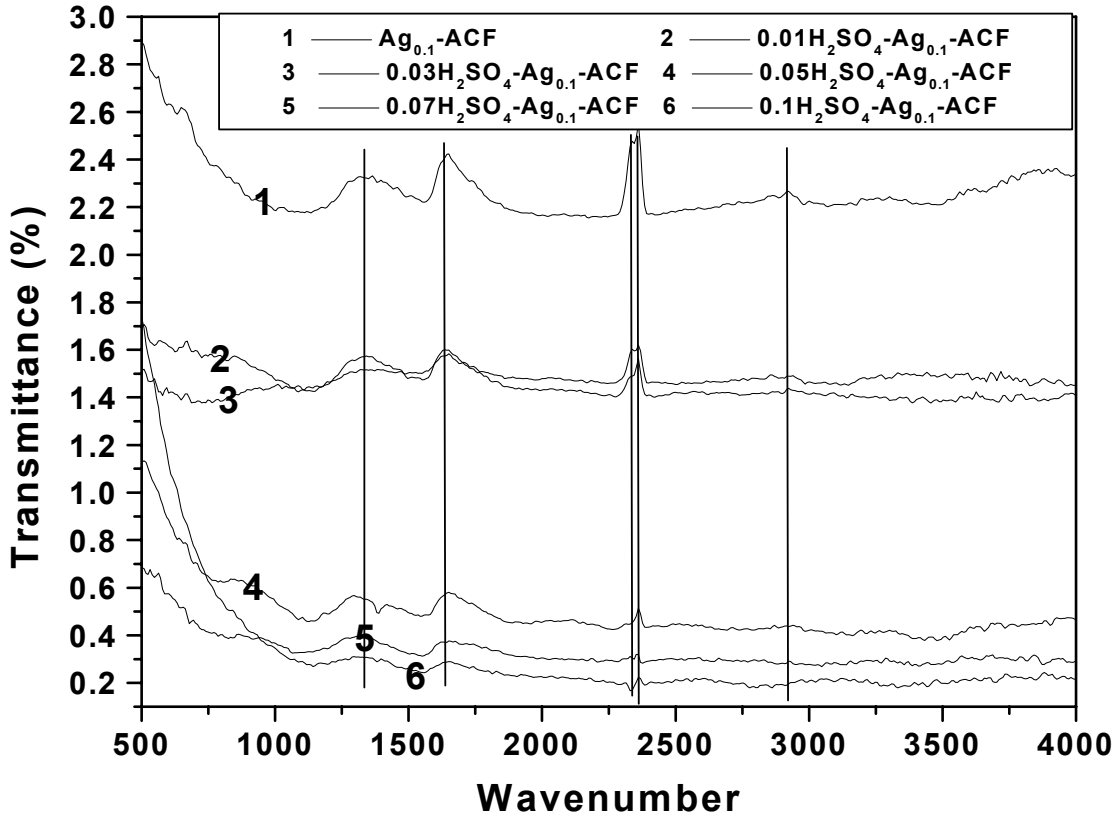

Fig. 1. Infrared spectra recorded from the acid pre-treated activated carbon fibers loaded with silver ; 1. $Ag_{0.1}$-ACF, 2. $0.01H_2SO_4$-$Ag_{0.1}$-ACF, 3. $0.03H_2SO_4$-$Ag_{0.1}$-ACF, 4. $0.05H_2SO_4$-$Ag_{0.1}$-ACF, 5. $0.07H_2SO_4$-$Ag_{0.1}$-ACF and 6. $0.1H_2SO_4$-$Ag_{0.1}$-ACF.