Study on recycling process of waste ion exchange resins to specialty carbon materials

Yusaku SAKATA, Akinori MUTO, M. A. Uddin and N. Lingaih
Department of Applied Chemistry, Faculty of Engineering, Okayama University

Summery

Introduction Ion exchange resins and membranes contains hetero atoms such as S and N. The disposal of these wastes needs high cost and leads to land filling. In this study, the conversion of these wastes to specialty carbon materials was tried with the aim of resource recycling. Here, we report mainly on the size control of nano fine particle of the metal compounds dispersed in the carbon material prepared from metal ion exchange resins. In addition, the research on fine structure of the porous carbonaceous material, which showed the peculiar elasticity behavior, observed at micro Vickers hardness measurement is also reported.

Experimental The commercial chelate resin with iminodiacetic acid group was mainly used. The conventional ion exchange procedure was used to incorporate metal ions such as Ni^{2+}, Fe^{2+} and Fe^{3+} into the resin. The metal ion-exchanged resin was washed by deionized water and dried at room temperature. The resin was placed into the glass tubular reactor kept in electric furnace. The temperature of the reactor was raised to 120℃ and kept for 1 hour in a N_2 stream to dry the resin. The low temperature heat treatment was carried out for 1 hour at 180～220℃ under air or N_2 stream. Then, the resin sample was carbonized for 3 hours at 400 - 700℃ in N_2 stream. Crystalline metal compound in the carbon matrix was identified by the X-ray powder diffraction method. The content of the metal was obtained by the thermogravimetry. The pore structure of the carbon material was analyzed by the N_2 gas adsorption method at 77K. Shape of the carbonaceous material and distribution of the metal compounds were observed by SEM and TEM. The elasticity behavior of carbonaceous materials was observed by Vickers hardness measurement.

Conclusions The following results were obtained as the effective factor for controlling nano particle size of metals in the carbon matrix. *1)* The diameter of metal ultra fine particle increased, as the increase in carbonization temperature. (Ni; 6.4 - 48nm at 400-700℃, Fe; 19-37nm at 400-500℃) *2)* The particle size was affected by the low temperature (180 - 220 ℃) heat treatment prior to the carbonization. *3)* The particle size was also affected by a kind of gas used at the low temperature heat treatment. Smaller metal particles observed when the N_2 gas was used in pretreatment compared to air. *4)* The particle size tended to decrease with increase in the metal content of the composite. The disappearance of the trace at Vickers hardness measurement was observed for the carbonaceous materials whose Ni content was over 15wt% and in witch Ni particle diameter was about 10 nm. *5)* The carbonaceous material made from the waste ion exchange membranes showed the similar physical properties compared to carbonaceous materials obtained from ion exchange resins.