PORE SIZE CHARACTERIZATION OF ACTIVATED CARBONS BY USING IMAGE PROCESSING OF TRANSMISSION ELECTRON MICROSCOPE IMAGES

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Introduction

Activated carbons are highly expected to be the bases of new electrical materials, especially the energy devices as an electric double-layer capacitors for use in electronics and electric vehicles, because of their very large specific surface areas (SSA). The microstructure of the pore of active carbons has affects the adsorption work greatly. In order to analyze the details of the properties, numerous research have been focused on the characterization of the pore size of activated carbons. In general, pore analysis techniques based on the gas, such as nitrogen, argon and carbon dioxide, adsorption method has been well developed and applied successfully to characterize the practical activated carbons[1]. But the detailed analysis of the microstructure of the pores, especially regarding the shape or morphology of the pores, has not yet been adequately clarified.
In this study, we report the pore size characterization (PSD) of activated carbons from transmission electron microscopy (TEM) images. TEM provides a very useful technique for analyzing porous materials, because TEM gives visual and detailed information about the pores[2]. As an image-processing object, we used TEM images of the carbons activated with KOH. First we extracted the pore boundaries of the activated carbons by using fuzzy template method obtained from notion of a fuzzy topology, which was established as set with an ambiguous limit by the others[3]. From these results, we obtained the pore size distributions and the pore shape distributions.

Experimental

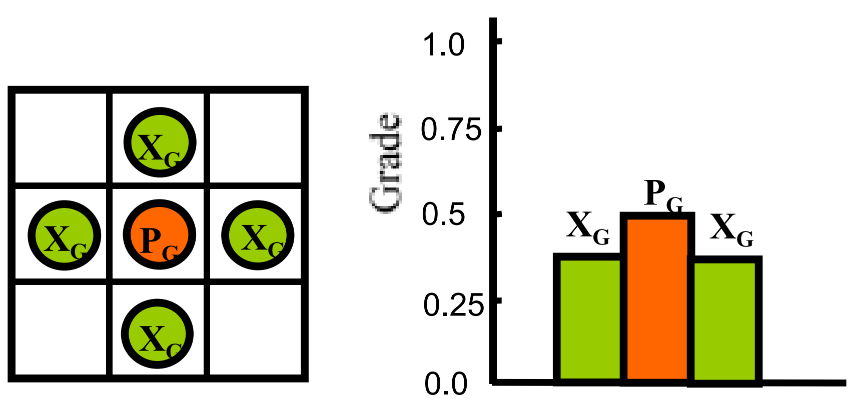
We used the samples, were prepared by chemical activation with KOH. The adsorption properties were measured on a Micrometrics (ASAP 2010, Micrometrics, USA), instrument, with nitrogen and argon at 77.35K and 87.29K. These data were reported in another paper. We observed these samples by HRTEM (JEM 2010 FEF, JEOL Co, Ltd., acceleration voltage of 200kV). The magnification of the observation was x200, 000 and the defocus value is 255 nm. These TEM images were converted to 512x512 pixel and 256 gray scale level images. The dark area in the image is carbon in the materials and the area that looks bright is considered to be the pore in the materials. The picture which include ambiguous light and shade image was considered to be a fuzzy set, and the boundary extraction was performed from the idea of the 'interior' and the 'exterior' obtained on the basis of the fuzzy template drawn from the fuzzy theory. The region of the boundary is obtained by the following equation.

$$A^b = (A^i \cup A^e)^c = X - A^i \cup A^e) \quad (1)$$

Where A^f, A^i, and A^e are boundary, interior, and exterior of pore, and where $(\ )^c$ is inversion operation and X_G is the maximum gray level in a TEM image. The fuzzy template used for this boundary extraction is shown in Figure 1. P_G and X_G denote the grade of the normalized gray level of the TEM image. The boundary A^f corresponds to the area where is not the interior or the exterior of pore. After both the interiors and the exteriors of the pores in TEM images were extracted using fuzzy templates, the boundaries of the pore were determined. From this result, the girth and the area of the each pore were calculated respectively. We characterized the textures of each sample based on these data.

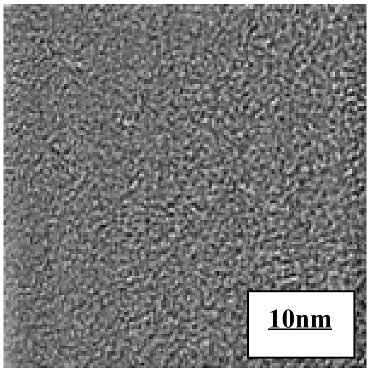
Results and Discussion

Figure 2 shows the TEM images of the samples that we analyzed. SAM1 is activated by using 100[wt%] KOH (SAM2: 200[wt%], SAM3: 300[wt%]. The dark region in the images is the bulk part of the materials, and the area that looks bright is considered to be the pore. Figure 3 shows the result of extracting boundary of the pores in Figure 2 by using the fuzzy template. We calculated the complexity of the pore shape from the result of Figure 3. The complexity of the pore shape is represented from the relation between the pore girth and the roundness of the pore. Each graph in Figure 4 indicates the complexity of the pore shape of each sample. The pore shape is seemed to become gradually smoothness in proportion to the density of KOH. Each graph in Figure 5 shows the distribution of the pore area. The range of the most different size of the pore area is from 0.1[nm] to 0.2[nm]. The size of the pore area is seen to become larger with the increase of the density of KOH-activation.

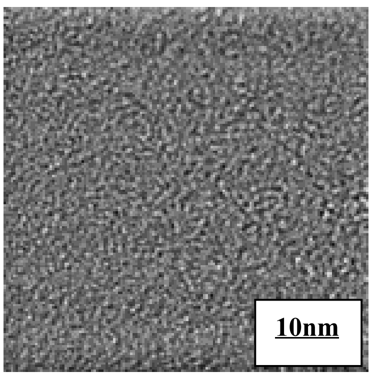


(a)Top view of a fuzzy template (b)Side view of a fuzzy template

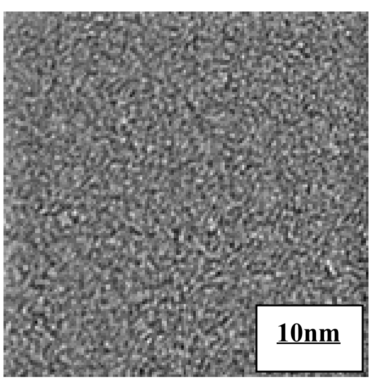
Figure 1. A model of the fuzzy template



(a) SAM1

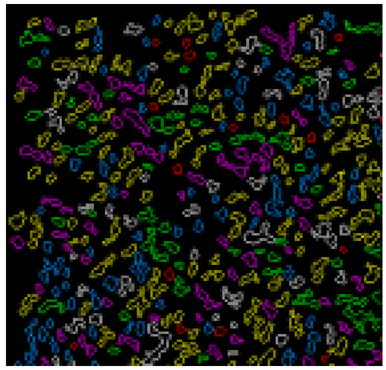


(b) SAM2

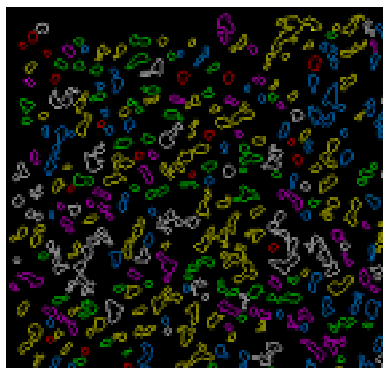


(c) SAM3

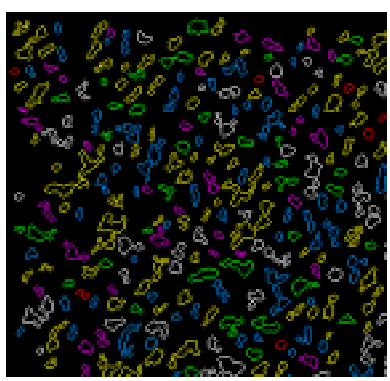
Figure 2. TEM images of the samples



(a) SAM1

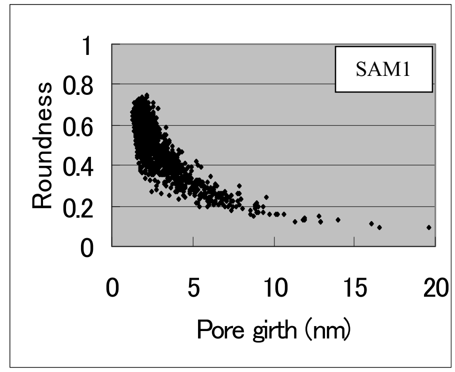


(b) SAM2

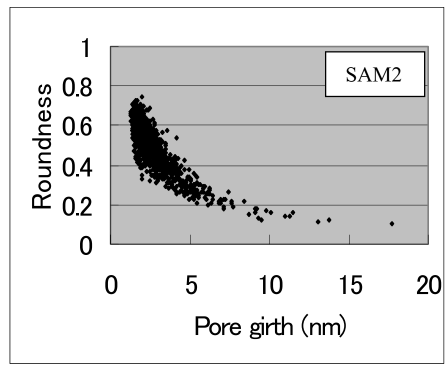


(c) SAM3

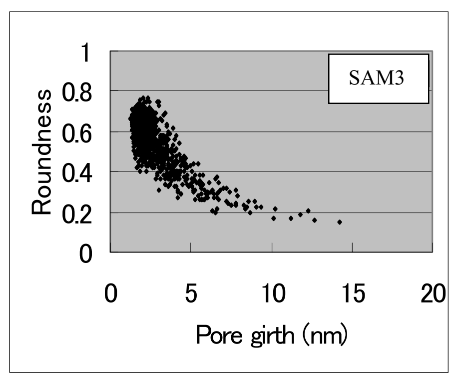
Figure 3. The extracted boundary from TEM images



(a) SAM1



(b) SAM2



(c) SAM3

Figure4. The roundness of the pore versus the pore girth

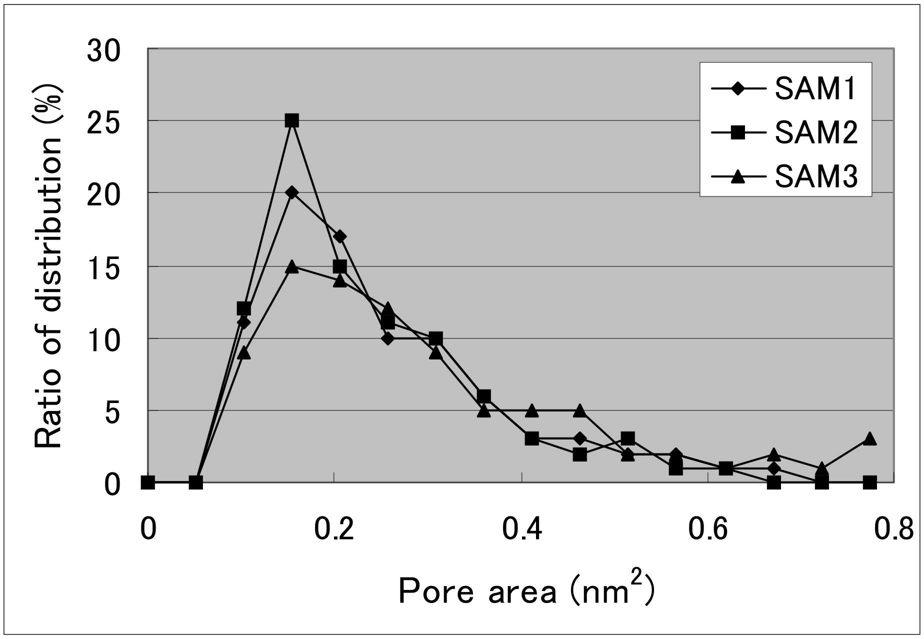


Figure 5. Pore area size distributions

However, the number of the pore in SAM3 decreases during that range. This is the reason why each pore is seen to combine with a neighboring pore, becoming a larger pore.

Conclusions

We were able to estimate the pore area and the complexity of the pore from the boundary of the pores that were extracted without the effects of difference of the brightness gradient of the image. From these results, we obtained the fundamental data to know the relation between the micro- structure of the activated carbon and the density of KOH-activation in detail.

References

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