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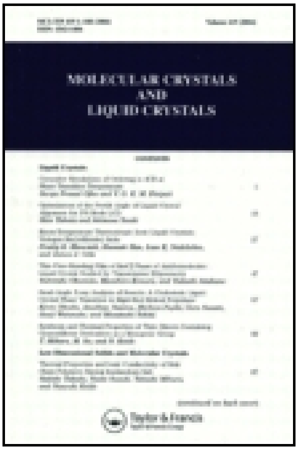

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T. Miyazaki [a], T. Oshida [a], T. Nakatsuka [b], H. Yamamoto [b], M. Okamoto [b] & M. Endo [b]

[a] Nagano National College of Technology, Nagano, 381-8550, Japan

[b] Shinshu University, Nagano, 380-8553

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ANALYSIS OF PORE STRUCTURE OF ACTIVATED CARBON BY USING IMAGE PROCESSING OF TEM IMAGES

T. Miyazaki and T. Oshida
Nagano National College of Technology, Nagano 381-8550, Japan

T. Nakatsuka, H. Yamamoto, M. Okamoto, and M. Endo
Shinshu University, Nagano 380-8553

We report an attempt to extract the pore structure of activated carbon from transmission electron microscopy (TEM) images. We have tried to use TEM of coconut-shell-based activated carbons, combined with image analysis, in order to make TEM a quantitative technique. It is important that the pore boundaries are clarified to analyze the pore structure, but extraction of the edges of pores is difficult by conventional techniques. In this work, we attempted to extract of the pore boundaries of the activated carbons by using fuzzy template obtained from a notion of a fuzzy topology.

Keywords: activated carbon; pore; TEM; image analysis; fuzzy theory

INTRODUCTION

Activated carbons have a feature of high adsorption, because of their high specific surface areas (SSA) and porous structure. It is known that the adsorption characteristic of the activated carbon is closely related to the pore size in them and the chemical properties on the surface of the pores. The gas-adsorption method is generally used for measurement of the SSA, but we can not know the SSA of the pores smaller than gaseous molecule by this technique [1,2]. In order to analyze in detail the structure of the microscopic pores, we used transmission electron microscopy (TEM) combined with image analysis. Since the brightness variation of the boundary of pores that appear in the TEM image is very ambiguous, it is difficult to distinguish the boundary of pores. However, we have already developed a technique that extracts the ambiguous boundary of the object. The technique uses a fuzzy template on the basis of fuzzy topology [3].

In this study, we used this technique to extract pore boundaries of TEM images of coconut-shell based activated carbons with different SSAs, and analyzed the distribution of the areas and the distribution of the volumes.

EXPERIMENTAL DETAILS

The technique of the boundary extraction allows Eq. (1).

$$A^f = (A^i \cup A^e)^c = X - (A^i \cup A^e) \tag{1}$$

where A^f, A^i, and A^e are boundary, interior, and exterior of pore, and where $()^c$ is inversion operation and X 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 [3].

TEM images of the coconut-shell activated carbon used for the experiment are shown in Figure 2, respective to the TEM images of specimens. The each content fraction of NaOH was the following, t21 was 100 wt%, t23 was 200 wt%, and t28 was 300 wt%. The heat treatment was performed at 700°C for 1h under an inert atmosphere. The activation temperature and time are the same. These specimens are observed using a 200 kV accelerating voltage TEM (JEOL 2010), and the observation magnification is ×200,000.

The TEM pictures are digitized by an image scanner with 1200 dpi, and the data is transferred to an array of 512 × 512 pixels. Each pixel has 256 gray levels, and the size of the pixel is 0.104 nm. Figure 3 shows the

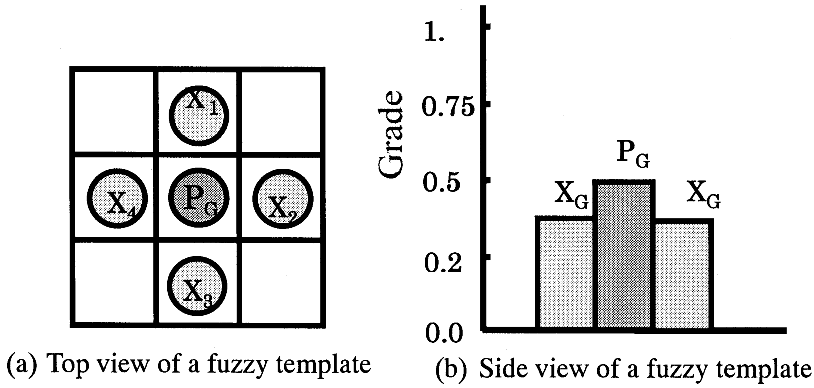

FIGURE 1 A fuzzy template model.

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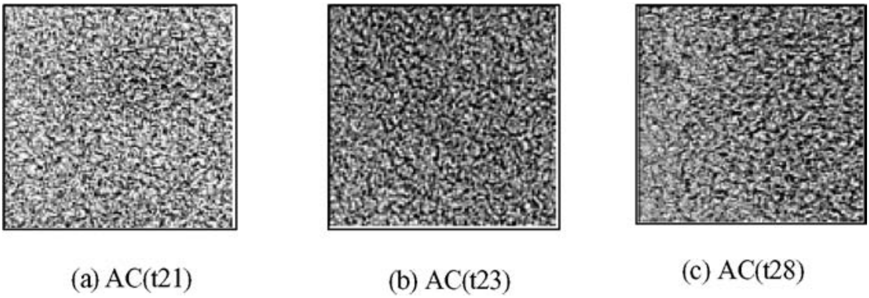

FIGURE 2 TEM images of activated carbon.

extraction process of the boundary from the part of the image of Figure 2(a). The dark area in the image is the bulk part of the coconut-shell activated carbon, and the area that looks bright is considered to be the pore. In this image processing, we used the fuzzy template with 3×3 in size, and 0.5 for the both grade value P_G and X_G.

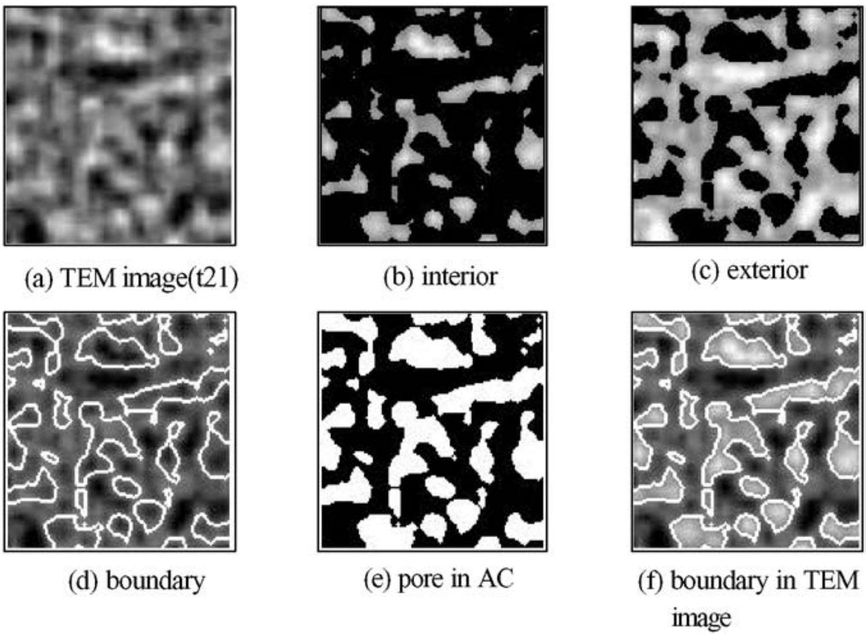

FIGURE 3 The boundary extraction process by this technique.

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DISCUSSION

Figure 3(a) is the enlarged part of Figure 2(a). Figures 3(b) and (c) are the extraction results of the pore area, and the bulk part of the activated carbon, respectively. Figure 3(d) is the boundary area obtained from both of Figures 3(b) and (c). Figure 3(e) is the binary transformed image, to clarify the pore boundary. The white area in it indicates the interior of pore,

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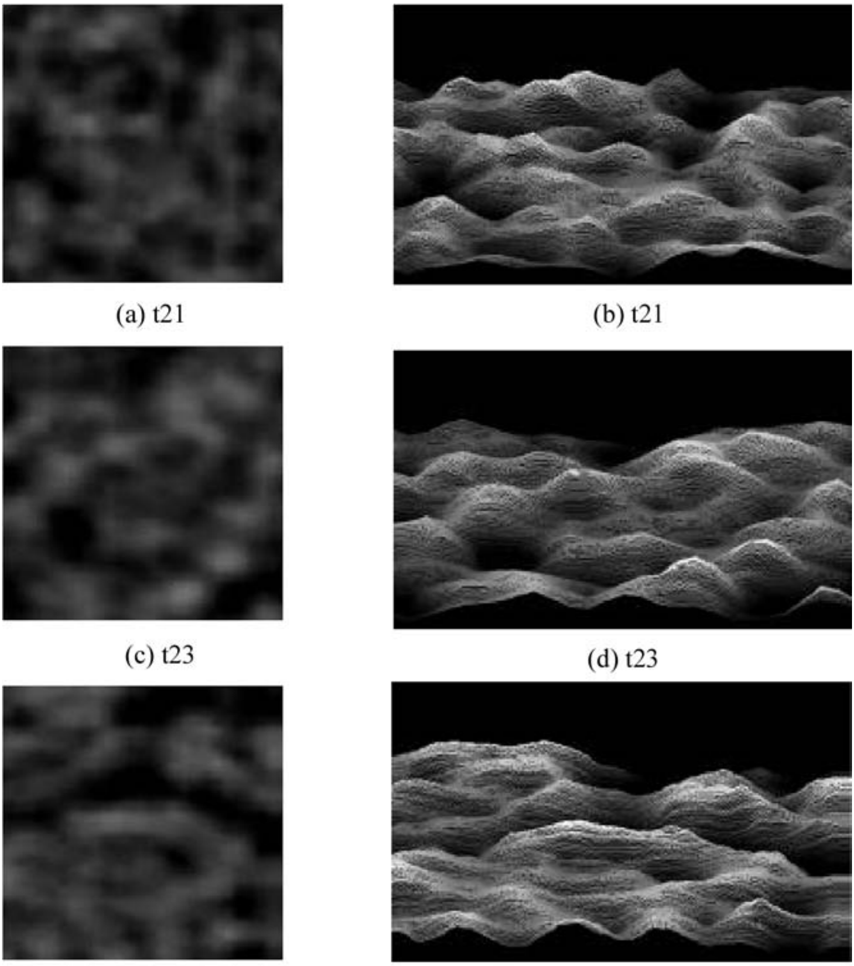

(a) t21 (b) t21 (c) t23 (d) t23 (e) t28 (f) t28

FIGURE 4 Enlarged part of TEM and contour image.

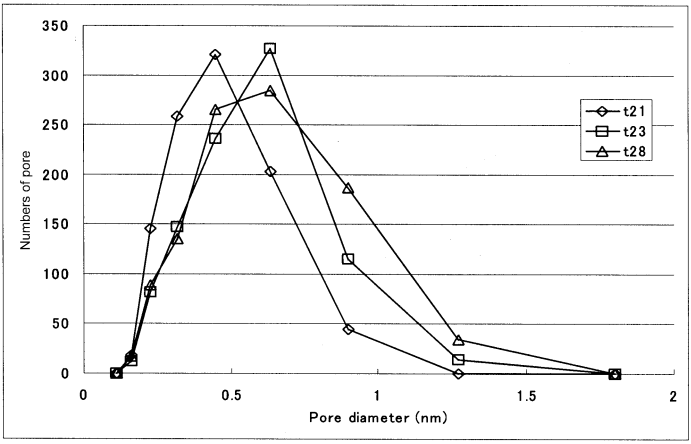

FIGURE 5 Pore diameter size distribution.

and the black area corresponds to the bulk part. Figure 3(f) is the superposed image of the Figures 3(a) and (d).

Figures 4(a), (c) and (e) are each part of original TEM images in Figure 2. Further, Figures 4(b), (d) and (f) show 3-D images of them. These images are obtained by using a gray level of the TEM image. In these figures, we can see the activation effect. The specimen t28 activated with the highest fraction (300 wt%) of NaOH, also has the smoothest face, and the biggest size pore of three specimens.

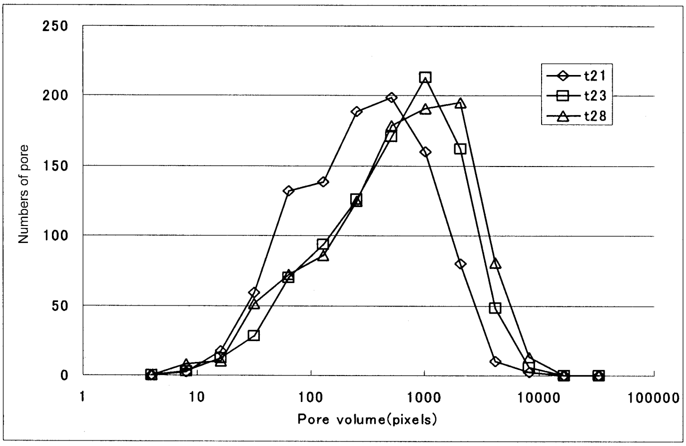

FIGURE 6 Pore volume size distribution.

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Figure 5 shows the distribution of the pore area. The condition of extraction of the pore area is that both the grade P_G and the grade X_G are 0.5, and the size of fuzzy template is 3×3 pixels. At first the size of the pore area, shown in Figure 5, are seen to become larger with an increase of the activation time. Next, each pore is seen to combine with a neighboring pore, becoming a larger pore. The graph demonstrates that the size of the pore area depends on the density of NaOH.

Figure 6 shows the distribution of the pore volume, calculated by using the intensity of the TEM image. Because we can't measure a distance of depth on the specimen, we use a [pixel^3] for the unit of pore volume. But we can compare relatively the size of the pore volume among the specimens. The shift of the pore size distribution was observed, as well as the pore area, with an increase of the fraction (100 wt%–300 wt%) of NaOH.

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CONCLUSIONS

In order to analyze the pore structure from the TEM images of coconut-shell activated carbons, we extracted successfully the boundary of the pore, by using the fuzzy template derived from the finite fuzzy topology. Because the gray level of the boundary changes gradually in the TEM image, it had been difficult to extract the pore boundary by the conventional technique. Conventional meaning, the transformation from a gray scale image to a binary image. On the other hand, by the present technique, the center of the boundary area can be extracted without the effects of difference of the brightness gradient of the image. We applied this technique to the TEM images of three kinds of coconut-shell activated carbons. The distributions of the area and the girth of the pore were characterized respectively and characterized quantitatively.

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