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Fuat Güzel[a]
[a] DEPARTMENT OF CHEMISTRY FACULTY OF SCIENCES, DICLE UNIVERSITY, DIYARBAKIR, TURKEY

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Determination of Surface Areas of Active Carbons by Retention of Ethylene Glycol and Ethylene Glycol Monoethyl Ether

FUAT GÜZEL
DEPARTMENT OF CHEMISTRY
FACULTY OF SCIENCES
DICLE UNIVERSITY
DIYARBAKIR, TURKEY

ABSTRACT

Experiments of ethylene glycol and ethylene glycol monoethyl ether retention on some active carbons were carried out to measure the surface areas of the carbons. Some differences were observed between nitrogen-specific surface area values and those obtained from ethylene glycol retention. However, since the differences decreased through an increase of surface oxygen content as a result of treatment of the carbons with H_2O_2, it was evident that ethylene glycol retention depended upon the surface oxygen content. Also, the surface area values obtained from retention of ethylene glycol monoethyl ether did not depend upon the surface oxygen content. Measurements of the ethylene glycol monoethyl ether retention process could be performed in a shorter time than the equivalent ethylene glycol retention measurements.

Key Words. Adsorption; Activated carbon; Determination of surface area

INTRODUCTION

The value of the surface area of solid matter is one of the most important properties in surface chemistry, adsorption, and catalysis studies. For the determination of surface area, such methods as gas adsorption, adsorption of solute from solution, heat of wetting, etc. are used. Of these, the com-

monest is the gas adsorption method using nitrogen gas and evaluating the surface area with B.E.T. (Brunauer–Emmett–Teller) analysis at 77 K. This method has been recommended as a standard method by IUPAC for all solids (1).

In the present study, besides the ethylene glycol retention method (2, 3) used by many researchers for the determination of surface area, an ethylene glycol monoethyl ether retention method was investigated on commercial active carbons as well as on active carbons produced from almond shells prepared under various conditions.

Assessment of the method was made by comparing the results with those obtained from conventional gas adsorption methods.

EXPERIMENTAL

In this study, almond shells, ground to 2 mm, were treated with sulfuric acid solution at a ratio of 1:1 (w/w) for 6 hours and then subjected to the following procedures:

a. Carbonization for 1 hour in the CO_2 gas medium (or activation) (B-7)
b. Carbonization for 4 hours in the CO_2 gas medium (or activation) (B-8)

and along with these newly prepared active carbons, Merck-2514 (M-2514) and Merck-2184 (M-2184) active carbons were also studied (4). The specific surface areas of these active carbons were determined (4) by nitrogen gas (77 K) adsorption, and experiments retention of ethylene glycol (EG) and ethylene glycol monoethyl ether (EGME) were conducted using a gravimetric system. In both experiments, active carbon samples which had been dried at 373 K for 16 hours, were made into 0.3 g samples with

TABLE 1
The Values of the Nitrogen Specific Surface Area and the Amounts of Ethylene Glycol and Ethylene Glycol Monoethyl Ether Retained

Sample	S_{N_2} ($m^2 \cdot g^{-1}$)	m_{EG} ($g \cdot g^{-1}$)	S_{EG} ($m^2 \cdot g^{-1}$)	m^*_{EG} ($g \cdot g^{-1}$)	S^*_{EG} ($m^2 \cdot g^{-1}$)	m_{EGME} ($g \cdot g^{-1}$)	S_{EGME} ($m^2 \cdot g^{-1}$)	m^*_{EGME} ($g \cdot g^{-1}$)	S^*_{EGME} ($m^2 \cdot g^{-1}$)
B-7	630.0	0.1831	586.0	0.1931	618.2	0.1715	595.6	0.1695	588.6
B-8	869.0	0.2261	723.7	0.2496	798.8	0.2227	773.5	0.2246	780.8
M-2184	974.4	0.2432	778.3	0.2559	819.0	0.2427	843.0	0.2450	851.2
M-2514	1160.3	0.1769	566.2	0.2651	851.6	0.2436	846.1	0.2404	834.9

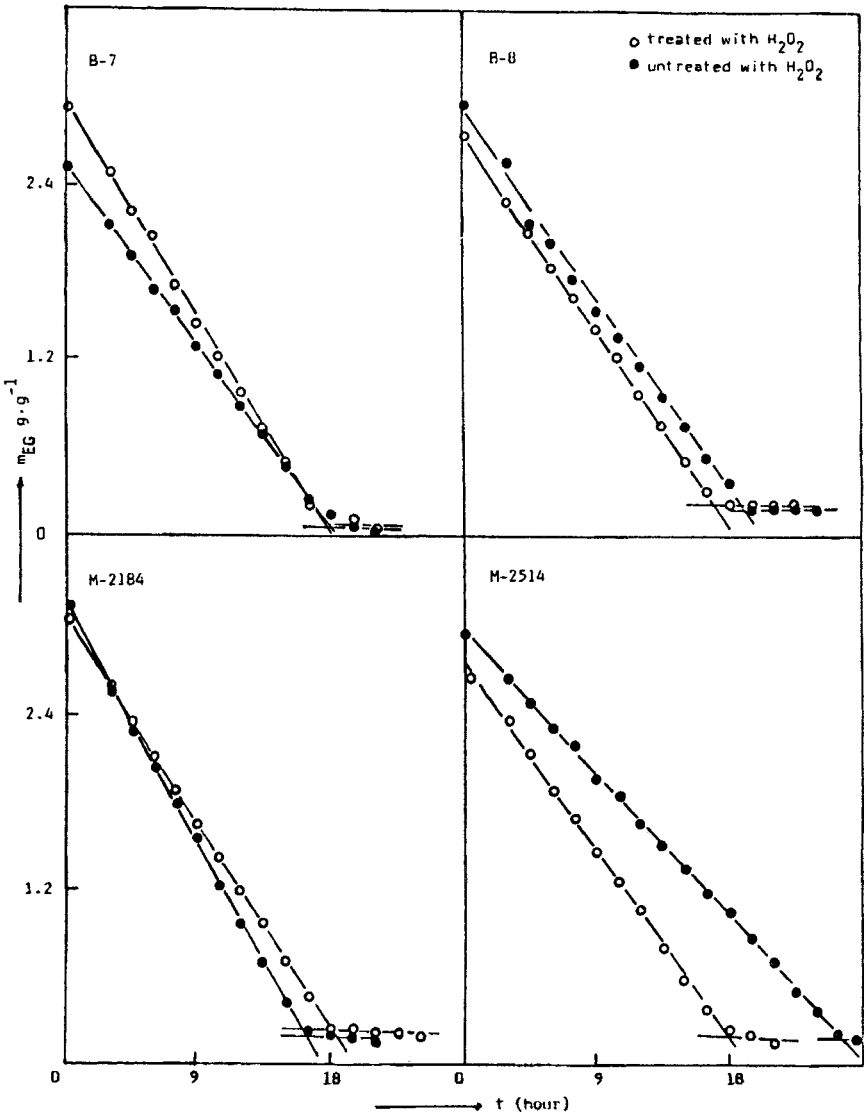

FIG. 1 Plots of time-dependent ethylene glycol retention related to various active carbons.

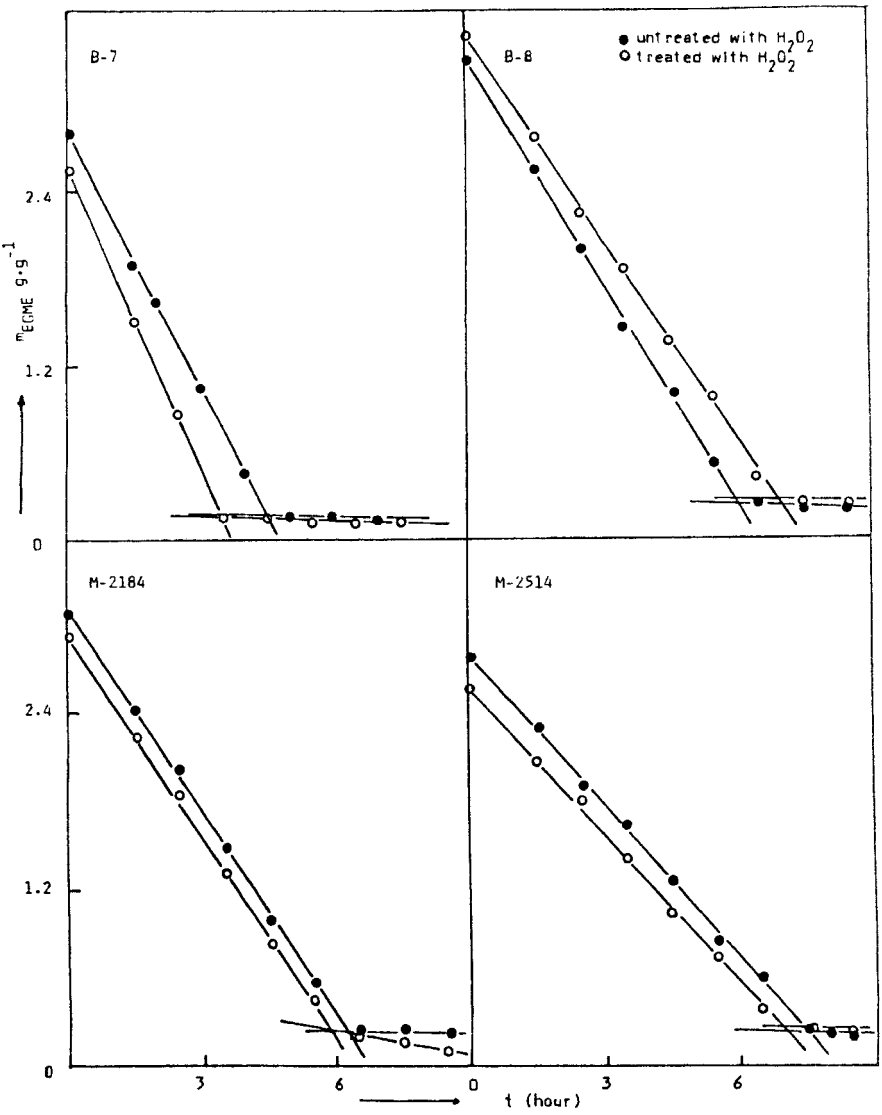

FIG. 2 Plots of time-dependent ethylene glycol monoethyl ether retention related to various active carbons.

EG or EGME. To wet the carbons completely with the adsorbed liquids, the samples were kept in a vacuum desiccator containing $CaCl_2$ at 308 K and then placed in a water bath for 16 hours. Later, a vacuum pump with a discharge power of 10^{-2} mmHg was used to remove any excess liquid. When the weights became stable, the amounts of retained EG and EGME (m_{EG} and m_{EGME}) were determined.

RESULTS

Table 1 shows the values of specific surface areas (S_{N_2}) as calculated from nitrogen adsorption (77 K) results for the active carbons used (4).

Figure 1 shows the amount of EG retained in the solid as a function of time and of EG treated with 9.8 N H_2O_2 to observe the effect of surface oxygen content on the retention of EG. Figure 2 plots the same information for EGME. The m_{EG}, m_{EGME}, m^*_{EG}, and m^*_{EGME} values for the samples were obtained from the point when the curves break and become horizontal (Table 1), where m^*_{EG} and m^*_{EGME} are the amounts of EG and EGME retained by active carbons treated with H_2O_2. The surface areas (S_{EG}, S_{EGME}, S^*_{EG}, and S^*_{EGME}) of the samples were determined by using Eq. (1) and the amounts of liquid retained (m_{EG}, m_{EGME}, m^*_{EG}, and m^*_{EGME}). The cross-sectional areas covered by a single molecule of EG or EGME at 308 K were assumed to be 0.330 and 0.520 nm^2, respectively (3, 5) (Table 1).

$$S\ (m^2 \cdot g^{-1}) = n\ (mol \cdot g^{-1}) \cdot \sigma\ (m^2) \cdot N_A\ (mol^{-1}) \qquad (1)$$

where n is the numbers of moles retained of EG or EGME per gram of carbon, σ is the cross-sectional area of the EG or EGME molecules retained, and N_A is Avogadro's number.

DISCUSSION

As seen in Table 1, for some samples the S_{N_2} and S_{EG} values are significantly different. In order to increase the surface oxygen contents of the samples with a view to explain the reason for this, the samples were processed with 9.8 N H_2O_2 for 6 hours, and the EG and EGME surface areas (S^*_{EG} and S^*_{EGME}) were determined again. In this case the relationship between the sample and EG increases, the S^*_{EG}'s are larger than the S_{EG}'s, and the difference between S^*_{EG} and S_{N_2} is less than the difference between the S_{EG} and S_{N_2}. A similar relationship was also observed by Molina-Sabio et al. (3) in the M-2514 sample. They found the ethylene glycol surface area of M-2514 to be 526 $m^2 \cdot g^{-1}$ before it was treated with H_2O_2, and as 810 $m^2 \cdot g^{-1}$ after treatment. Nevertheless, in a study carried

TABLE 2
Properties, Names, and Formulas of Ethylene Glycol and Ethylene Glycol Monoethyl Ether (5)

	Ethylene glycol	Ethylene glycol monoethyl ether
Formula	$HOCH_2CH_2OH$	$C_2H_5OCH_2CH_2OH$
Other names	1,2-Ethanediol	Ethanol, 2-ethoxy
Molecular weight ($g \cdot mol^{-1}$)	62.07	90.12
Density at 20°C (g/cm^3)	1.1155	0.9311
Boiling point (°C)	197.2	135.1

out by Puri et al. (2) on carbon black for samples treated and untreated with H_2O_2, there was not a significant difference between the treated and untreated samples, and the retention of EG did not depend upon the oxygen content of the carbon surface. On the basis of our observations in this study, we can say, like Molina-Sabio et al., that the retention of EG depends not only on the pore structure of the sample but also on the surface oxygen content needed for the surface to become thoroughly wet with EG.

Also, when the values of S_{EGME} and S^*_{EGME} were examined, insignificant differences were observed. It follows that the retention of EGME does not depend upon the surface oxygen content of the samples. The reason for these results probably originates from the polar group at each end of EG and the polar group at one end and a nonpolar group at the other end of EGME, as indicated by the formulas of EG and EGME given in Table 2.

When the EG and EGME retention methods are compared from the viewpoint of application, the EGME method is better because it requires less time (Figs. 1 and 2). This is probably because EGME has a higher vapor pressure at the experimental temperature and the surplus liquid on the surface evaporates more quickly.

As a result, existing conventional methods used to identify the surface area of active carbons have such disadvantages as high cost, requiring more time, and a dependence on the surface characteristics. The suggested method based upon the retention of EGME was found to be a useful and low cost method for determining the surface areas of active carbons. The results obtained by this method are in agreement with those of the nitrogen gas adsorption (77 K) method.

REFERENCES

1. IUPAC, *Manual of Symbols and Terminology for Physicochemical Quantities and Units*, Appendix II, Part I–II, Butterworths, London, 1975.
2. B. R. Puri, D. D. Singh, and U. Gupta, *Carbon*, *17*, 121–123 (1979).
3. M. Molina-Sabio, C. Salinas-Martinez De Lecea, F. Rodriguez-Reinoso, C. Puente-Ruiz, and A. Linares-Solano, *Ibid.*, *23*(1), 91–96 (1985).
4. F. Güzel and Z. Tez., *Sep. Sci. Technol.*, *28*(8), 1609–1627 (1993).
5. D. L. Carter, M. D. Heilman, and C. L. Gonzalez, *Soil Sci.*, *100*, 356–360 (1965).

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