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*A LA MEMOIRE D'ATATÜRK AU CENTENAIRE DE SA NAISSANCE*

# Effect of Outgassing Temperature on Surface Area of Sepiolite

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4

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## DEDICATION TO ATATÜRK'S CENTENNIAL

Holding the torch that was lit by Atatürk in the hope of advancing our Country to a modern level of civilization, we celebrate the one hundredth anniversary of his birth. We know that we can only achieve this level in the fields of science and technology that are the wealth of humanity by being productive and creative. As we thus proceed, we are conscious that, in the words of Atatürk, "the truest guide" is knowledge and science.

As members of the Faculty of Science at the University of Ankara, we are making every effort to carry out scientific research, as well as to educate and train technicians, scientists, and graduates at every level. As long as we keep in our minds what Atatürk created for his Country, we can never be satisfied with what we have been able to achieve. Yet, the longing for truth, beauty, and a sense of responsibility toward our fellow human beings that he kindled within us gives us strength to strive for even more basic and meaningful service in the future.

From this year forward, we wish and aspire toward surpassing our past efforts, and with each coming year, to serve in greater measure the field of universal science and our own nation.

# Effect of Outgassing Temperature on Surface Area of Sepiolite

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**ABSTRACT**

In order to prepare a catalyst support, the adsorptive properties of sepiolite from Eskişehir /Turkey have been examined. Change in surface area and micropore size distribution have been determined from the nitrogen adsorption-desorption isotherms at -196°C after outgassing of powder samples at various temperatures and $10^{-4}$ torr. It was observed that the surface area increases to 400 $m^2/g$ at a temperature of 100°C, and then decreases to lower values rapidly.

## INTRODUCTION

Generally in the chemical technology many catalytic processes are carried out in presence of supported catalyst(1). Supported catalysts are very useful in chemical technology, hence their many important properties such as activity, selectivity, durability, and mechanical strength are very much dependent on the pore structure of support (2,3). Also, surface area of the support is very important because a catalyst with larger surface area causes greater catalytic activity (4). Therefore we attempted to determine the surface area and micropore-size distribution of sepiolite, which is to be used as a catalyst support. The surface area of support and active sites in the catalyst can be determined from physical adsorption (5) and chemical adsorption (6) measurements respectively. On the other hand, micropore-size distribution (7) of support can be evaluated from the capillary condensation region of the desorption isotherm (8).

## EXPERIMENTAL

The sepiolite samples in powder form (—200 mesh) were outgassed in vacuum of the value $10^{-4}$ torr at the temperatures 30°C, 50°C, 100°C, 150°C, 200°C, 250°C, and 300°C for 2 hours. Nitrogen adsorption-desorption experiments were carried out in a pyrex adsorption apparatus (9) at liquid nitrogen temperature. The volumes (v) of the adsorbed nitrogen on per gram sepiolite were calculated in $cm^3$ at standart temperature and pressure. Than the adsorption-desorption isotherms have been obtained by plotting the volume of gas at STP versus the relative pressure $(p/p_o)$ and are shown in Fig. 1. Here, $p_o$ is the liquefaction pressure of nitrogen and p is the equilibrium pressure in adsorption cell at the liquid nitrogen temperature.

## RESULTS

The spesific surface area (A) of the sepiolite samples have been calculated using B.E.T. method (10). The limited adsorption volumes $(V_p)$ were taken as liquid volume of nitrogen at the relative pressure $p/p_o = 1$ and $-196°C$. Also, the mean pore radius have been calculated with the equation $\bar{r} = 2V_p/A$. The limited adsorption volume of the samples are almost the same with the value $V_p = 0.7\ cm^3/g$. The effect of outgassing temperature on the spesific surface area and the mean pore radius are shown in Fig. 2. Upon outgassing above 100°C a rapid decrease in the surface area takes place.

The cumulative pore volume (V) was taken as liquid nitrogen volume at the chosen value of relative pressure $p/p_o$ and calculated with the equation.

$$V = (v/22400)\ V^1 \qquad (1)$$

where, v is the adsorbed gas valume at STP and $V^1$ is the molal volume of liquid nitrogen with the value 34.65 $cm^3/g$. The radius of a pore, r, was calculated with the corrected Kelvin relationship (11), which is given for nitrogen at the boiling point depending on the relative pressure as follows:

$$r = \frac{9.52}{\ln(p_o/p)} + \frac{7.34}{\ln(p_o/p)^{1/3}} \qquad (2)$$

After obtaining a series of values of V and r, dV/dr values have been calculated and plotted versus r. The micropore size distributions of samples, which are outgassed at 30°C and 50°C, are shown in Fig. 3. The Figure 3 shows the effect of outgassing temperature on the micropore size distributions of sepiolite.

The comparison of Fig. 2 and Fig. 3 indicated that the increase of the surface is caused by decrease of radii of the micropores. On the other hand, the limited adsorption volume of sepiolite doesn't change with the outgassing temperature, whereas the micropore size distribution changes sharply.

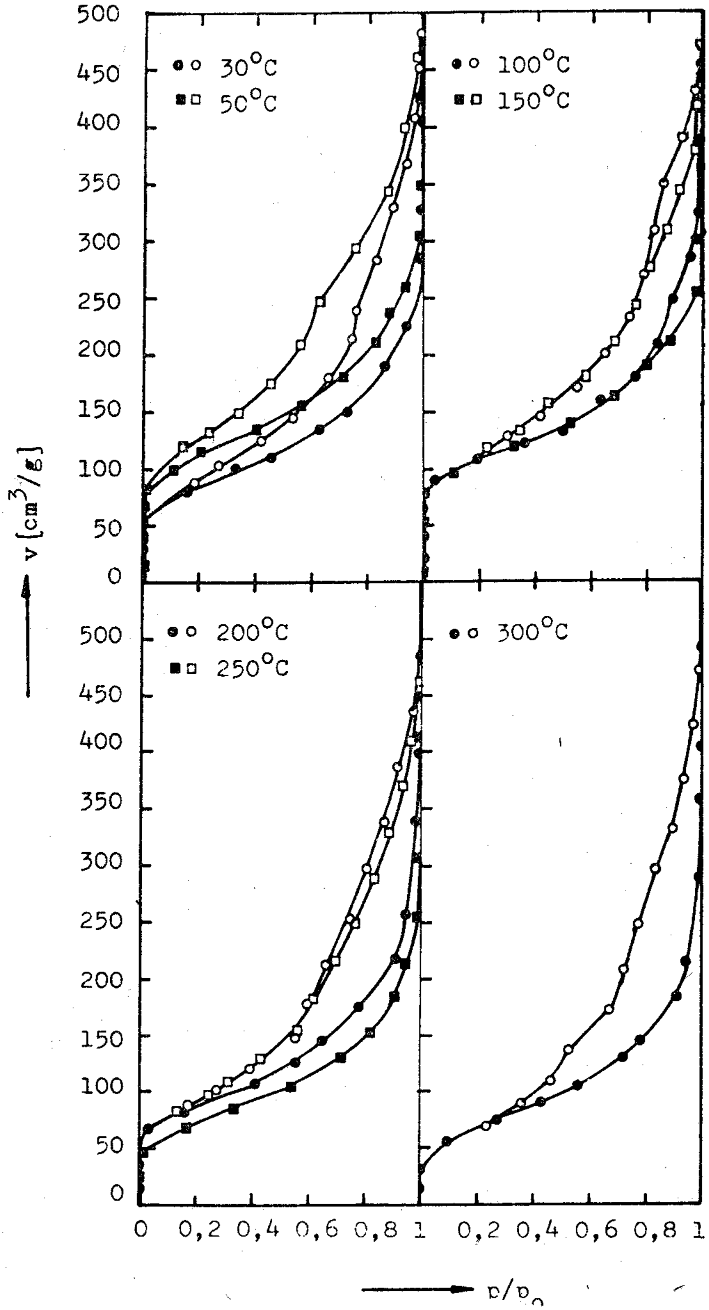


Fig. 1. Nitrogen adsorption ( ● ■ )- desorption ( ○ □ ) isotherms (at -196°C) on sepiolite outgassed at varius temperatures.

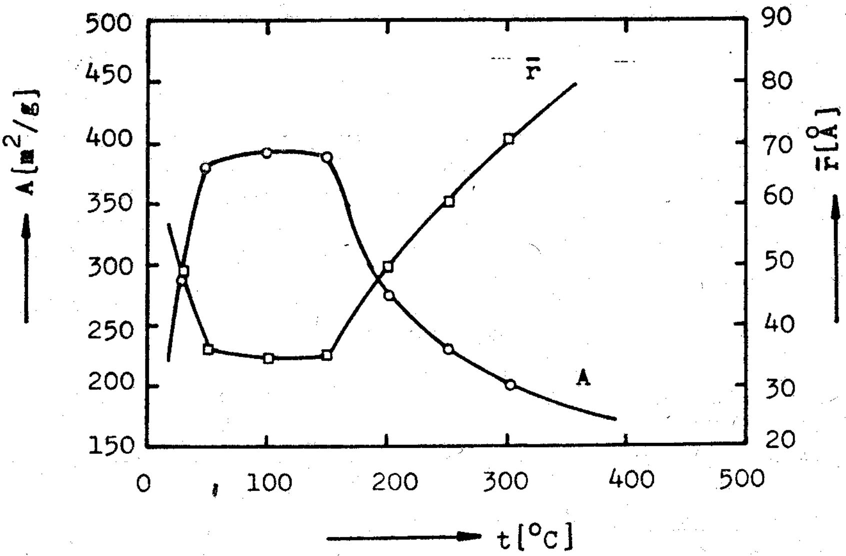


**Fig. 2. Effect of outgassing temperature on surface area and mean pore radius of sepiolite.**

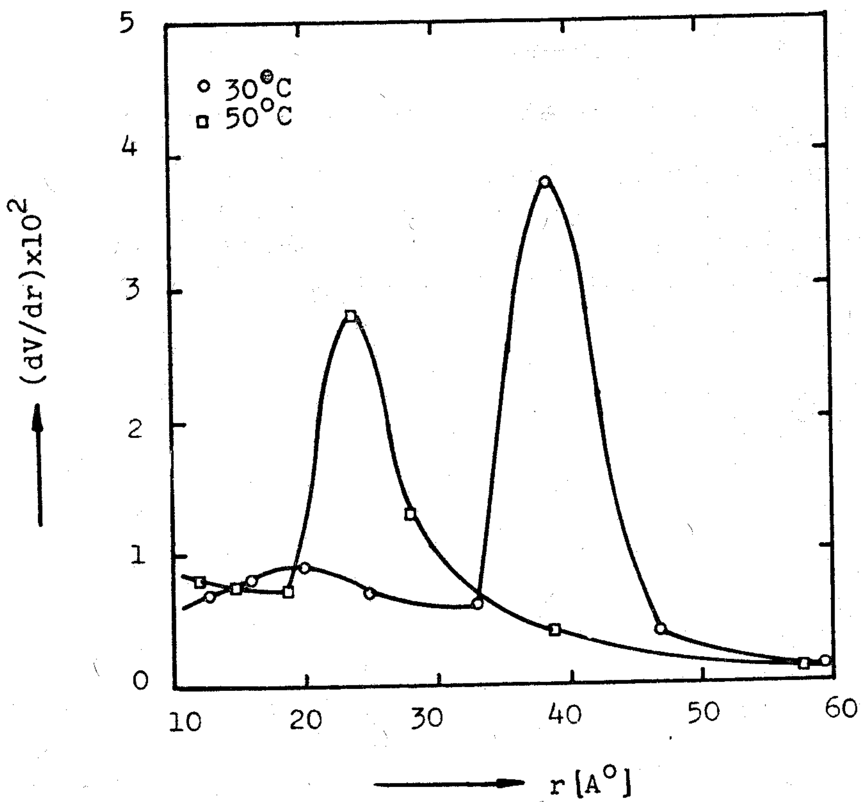


**Fig. 3. Effect of outgassing temperature on micropore-size distribution of sepiolite**

## ÖZET

Katalizör yatağı hazırlamak amacıyla Eskişehir taşı adıyla bilinen sepiolitin adsorplama özellikleri incelenmiştir. Farklı sıcaklıklarda ve $10^{-4}$ torr'da ısıtılan toz halindeki örneklerin üzerinde azotun -196°C daki adsorpsiyon ve desorpsiyon izotermlerinden sepiolitin yüzey alanı ve mikrogözenek boyut dağılımındaki değişmeler tayin edilmiştir. Sonuç olarak, yüzey alanının vakumdaki ön ısıtma ile önce artarak 100°C da 400 $m^2/g$ değerine ulaştığı ve sonra küçük değerlere hızla düştüğü saptanmıştır.

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