

## HYSTERESIS IN SORPTION

### XII. Influence of the Temperature of Activation of Silica Gel on the Hysteresis Effect

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#### INTRODUCTION

It has been shown in a previous paper<sup>3</sup> that the porosity of gels of hydrous oxides depends upon the mode of preparation of the gel. Gels are activated by heating them to a suitable temperature in a current of dry air. The effect of the temperature of activation of silica gel on the hysteresis phenomenon was studied using water vapour as adsorbate.

#### EXPERIMENTAL

*Silica gel.*—Silica gel was precipitated at 25° C. from water glass by adding acetic acid as described in the previous paper.<sup>3</sup>

*Activation of the gel.*—The gel precipitated at 25° C. and air dried was activated by heating to different temperatures in a current of dry air. The following activation temperatures were employed, 35° C., 70° C., 140° C., 300° C., 500° C., 750° C. and 1000° C. A hot water-bath was employed for activation at 35° C. and 70° C. and an electric furnace was used to obtain the other temperatures. A mercury thermometer was used in measurement of temperatures up to 500° C. and a thermo-couple was used for higher temperatures.

*Sorption and desorption of water vapour.*—Sorption and desorption measurements were carried out by employing the quartz fibre spring technique.<sup>1, 2</sup> Equilibrium was attained within 4 hours but 6 hours were actually allowed before each measurement. For the completion of each cycle of sorption and desorption, 6 to 7 days were required. At the end of each cycle the gel was found to retain some water. This bound water in each case is given in Table I.

#### DISCUSSION

All the gels activated at different temperatures, gave permanent and reproducible hysteresis loops (Figs. 1 to 7). When the temperature of

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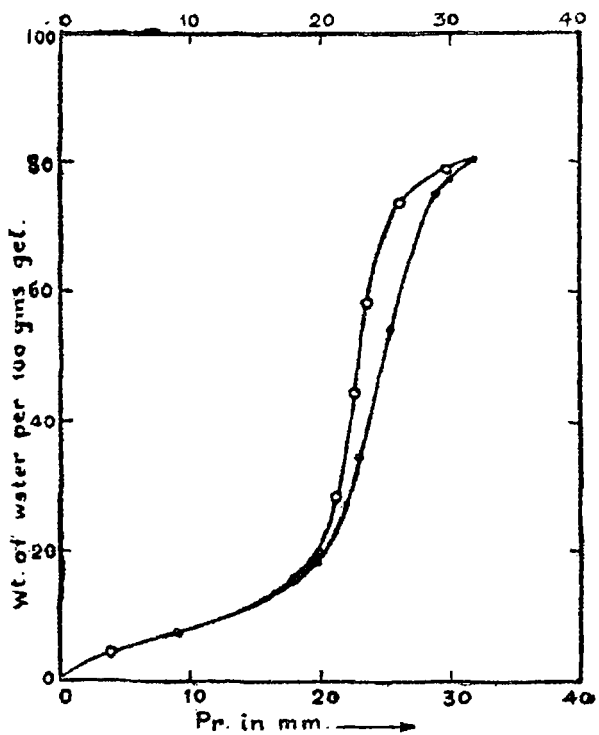


FIG. 1. Silica gel activated at 35° C. (3rd cycle)

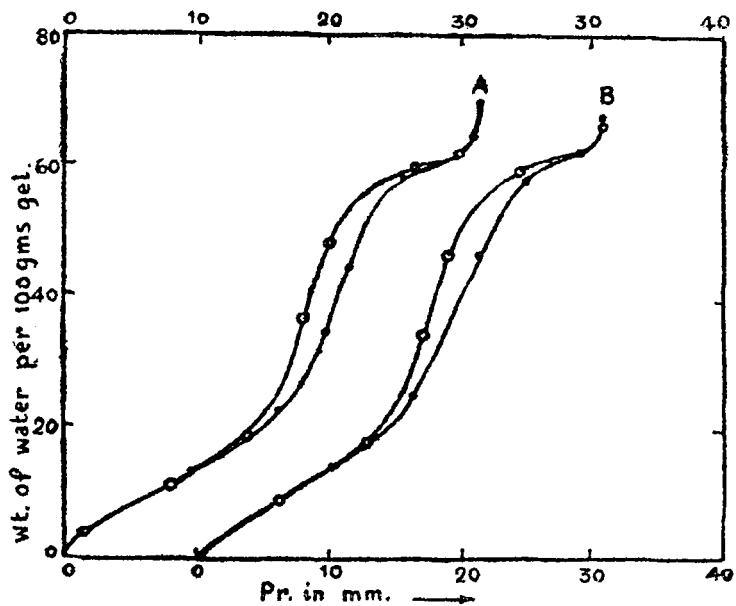


FIG. 2. Silica gel activated at 70° C. (A 2nd cycle, B 3rd cycle)

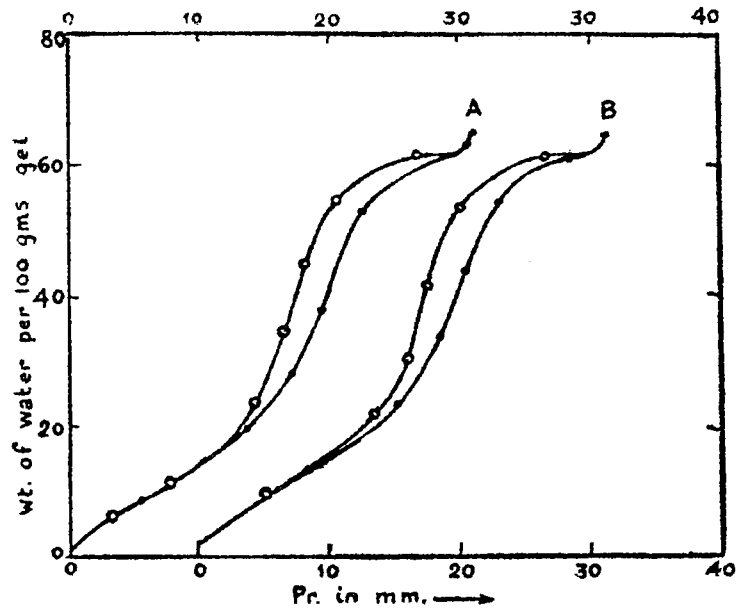


FIG. 3. Silica gel activated at 140° C. (A 1st cycle, B 2nd cycle)

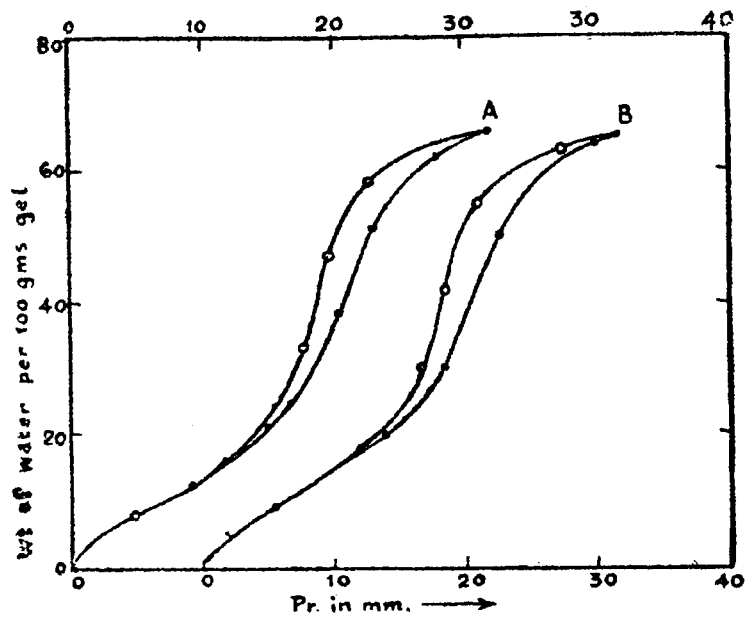


FIG. 4. Silica gel activated at 300° C. (A 2nd cycle, B 3rd cycle)

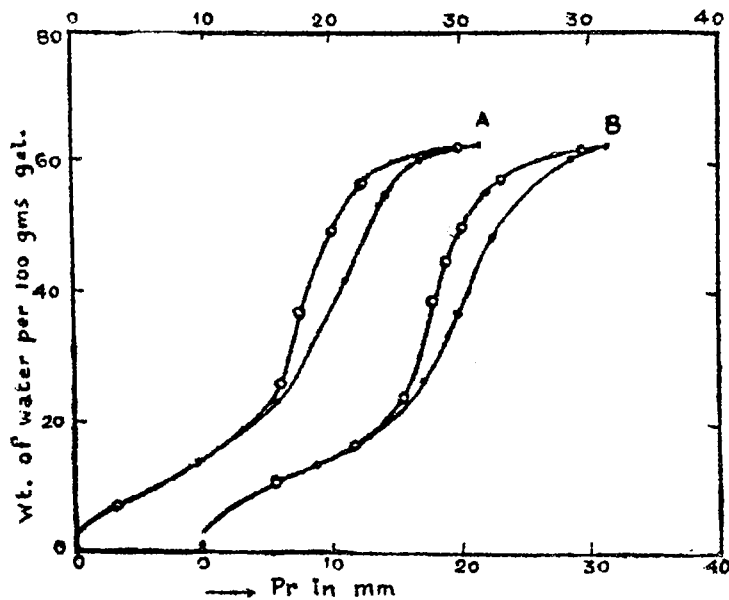


FIG. 5. Silica gel activated at 500° C. (A 3rd cycle, B 5th cycle)

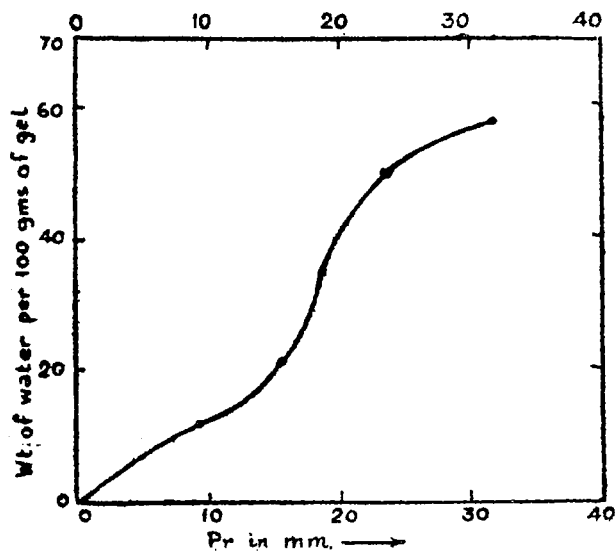


FIG. 6. Silica gel activated at 750° C.

activation of the gel is raised from 35° C. to 140° C., there is a decrease in the total sorptive capacity of the gel for water at the saturation pressure, indicating a corresponding decrease in the total capillary volume (Fig. 8). There

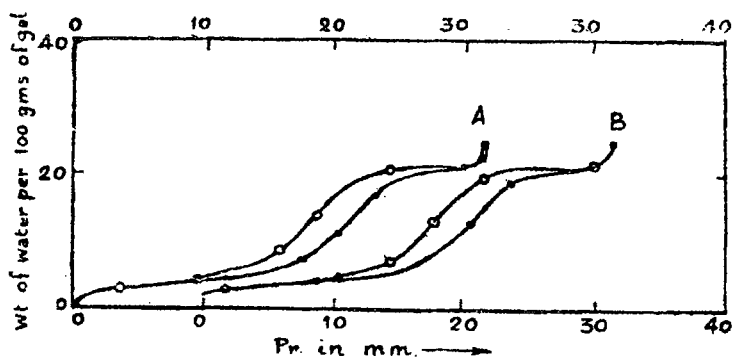


FIG. 7. Silica gel activated at 1000° C. (A 3rd cycle, B 4th cycle)

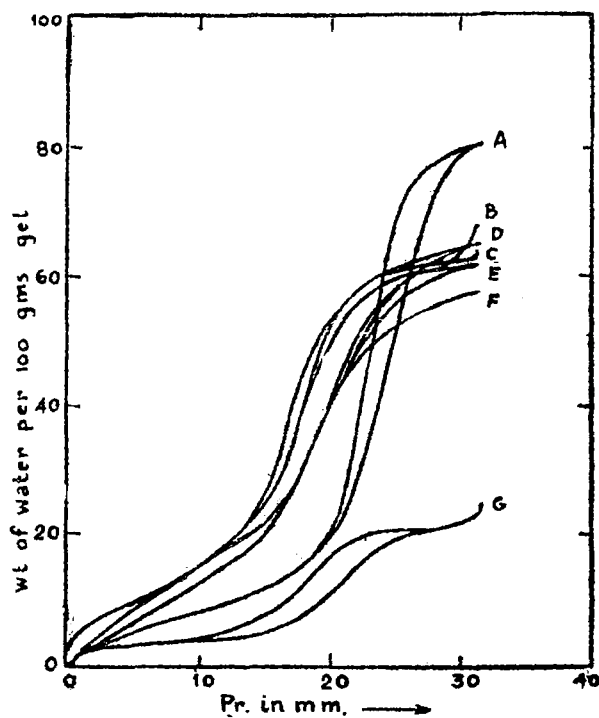


FIG. 8. Silica gel activated at A 35° C., B 70° C., C 140° C., D 300° C., E 500° C., F 750° C., G 1000° C.

is also a small decrease in the area of the loop which indicates a corresponding decrease in the total cavity volume of the gel.

From 140° C. to 500° C., the total sorptive capacities of the gels remain practically the same and the hysteresis loops are nearly coincident. These

TABLE I

Activation temperature °C.	Bound water per 100 gm. of gel	Residual water per 100 gm. of gel	Sorptive capacity for water at saturation pressure per 100 gm. of gel
35	0.05	..	84.2
70	0.93	6.90	68.1
140	0.90	6.13	63.1
300	0.93	6.67	63.6
500	2.23	6.56	63.0
750	2.97	..	58.2
1000	1.90	0.00	23.6

facts indicate that the total capillary volume and also the cavity volume in each gel remain practically unaltered.

Above 500° C., a rise in the temperature of activation has a marked influence. There is a decrease in the sorptive capacity and the area of the loop indicating a decrease in the total capillary volume and in the cavity space.

The above interesting changes lead to the following conclusions. Rise in the temperature of activation from 35° C. to 140° C. brings about a contraction of the gel and collapse of some of the capillaries resulting in a diminution of the total capillary volume. There is also a small decrease in the residual water of the gel.

From 140° C. to 500° C., the residual water in the gel is practically the same. The gel suffers no structural change as indicated by the unaltered character of the hysteresis loops.

Above 500° C., an increase in the activation temperature has a profound influence on the gel structure. The residual water is lost, the gel shrinks and there is a marked collapse of the capillaries. The total capillary volume in the gel activated at 1000° C. decreases nearly to a third of the capillary volume of the gel activated at 500° C.

For producing the maximum capillary volume in silica gel, a high temperature of activation is harmful. In this connection it may be pointed out that by measuring the sorptive capacity for carbon tetrachloride at saturation pressure, employing the dynamic method of measurement of sorption, Rao, K. S., and Rao, B. S.<sup>4</sup> have reported that for gels of alumina and ferric oxide, there is an optimum temperature of activation.

#### SUMMARY

The effect of the activation temperature of silica gel on the hysteresis effect has been studied. Sorption and desorption of water vapour at 30° C.

have been conducted on gels activated at 35° C., 70° C., 140° C., 300° C., 500° C. and 1000° C.

All the gels gave permanent and reproducible hysteresis loops. A marked variation however, was noticed in the total sorptive capacity and the area of the loop.

The results indicate that from 35° C. to 140° C., there is a decrease in the capillary space in the gel but from 140° C. to 500° C., the capillary space remains practically unaltered. Whereas, above 500° C., the gel suffers structural change, the capillaries collapse and there is a marked decrease in the total capillary volume.

#### REFERENCES

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