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NATIONAL RESEARCH COUNCIL OF CANADA DIVISION OF BUILDING RESEARCH

MOISTURE SORPTION MEASUREMENTS ON PLASTER

bу

Peter M. Jones

Report No. 167

of the

Division of Building Research

Ottawa

February 1959

PREFACE

Since water is the most important agent commonly involved in problems of dimensional change and deterioration in building materials, information on the equilibrium moisture contents of materials is of decided interest in building science. The Division has constructed, as reported earlier in DBR Internal Report 133, a sorption balance for determination of sorption isotherms. The additional use of a small optical strain gauge which can be read through the glass walls of the apparatus has permitted simultaneous measurement of length change at various equilibrium moisture contents. The anomalous behaviour of gypsum plaster in some early experiments led to the studies which are now reported. These in turn have drawn attention to the possibility that gypsum plaster may lose water of hydration under indoor climate conditions of low relative humidity, and further studies are now in progress.

Ottawa February 1959 N.B. Hutcheon Assistant Director

MOISTURE SORPTION MEASUREMENTS ON PLASTER

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Peter M. Jones

The nature of the adsorption of water on plaster as previously determined and reported in DBR Internal Report No. 133 (1), was unusual and unexpected. The B.E.T. plot was not linear and, in addition, the surface area calculated from the test results was unusually large. Both the isotherm and the corresponding dimensional change indicated that large changes occurred in the narrow range of values of p/p_0 between 0 and 0.1, and again when p/p_0 of 0.8 was exceeded. These results prompted speculation regarding possible mechanisms which would account for this behaviour of plaster, and consequently further work was undertaken.

The study described here was designed to confirm the previous results and to reveal the possible mechanism. Samples were prepared under normal conditions of hydration, as well as under conditions which, it was thought, would produce coarse crystals of low surface area.

Apparatus and Samples

The apparatus used was the same as that previously used, the description and manner of operation of which have been reported (1). Samples were prepared as described in the following paragraphs. It should be noted that the original constituents and manner of mixing were the same in all cases, the differences being in the conditions of setting and subsequent treatment.

(a) Unfired "Hot" Sample

This sample was prepared by adding 530 grams of pottery plaster to 318 grams of distilled water. Five seconds after addition, the mixture was stirred for 35 seconds, placed in moulds, and allowed to set at 95°C. The resulting substance was then removed and stored in a desiccator at 50 per cent relative humidity for 1 month.

(b) Unfired "Cold" Sample

This sample was prepared by adding 530 grams of pottery plaster to 318 grams of distilled water. Five seconds after addition the mixture was stirred for 35 seconds. This mixture was placed in moulds and allowed to set at room temperature. It was then removed and stored in a desiccator at 50 per cent relative humidity for 1 month.

(c) and (d) Fired Samples

Portions of the "hot" and "cold" samples were fired at 850 to 900°C for 16 hours, allowed to cool, and stored in a desiccator over calcium chloride until used.

Results and Discussion

(a) Unfired Samples

Evacuation of adsorbed gases from the system required a long time. During this evacuation the "hot" sample rapidly lost 1 per cent of its weight, but then remained unaltered, while the "cold" sample showed a continuous loss over a period of three weeks until it had lost almost 20 per cent of its weight. These rates of loss are shown in Fig. 1.

Results of the water vapour adsorption measurement are plotted in Fig. 2. B.E.T. calculations were made on these samples and plots are shown in Figs. 3 and 4. The surface areas calculated from these plots are 166.1 m²/g and 16.73 m²/g for the "cold" and "hot" samples respectively.

In the case of the "cold" sample, the adsorption isotherm shows two portions where a large increase in weights occurs, between 0 and 0.1 values of p/p_0 and when p/p_0 of 0.8 is exceeded. The B.E.T. plot does not give a straight line, but nevertheless on calculating the surface area of the sample a very large value is found, $166.1 \, \text{m}^2/\text{g}$. The surface area of concrete has previously been found to be $43 \, \text{m}^2/\text{g}$. Plaster is thought to be in a crystalline form when solid, whereas concrete is thought to be a gel, and it would hardly be expected that a crystalline structure would have a larger surface area than a gel.

It is thus postulated that the measured increase in weight is not all physical adsorption, but part chemisorption resulting in hydration. This thought is supported by a closer study of the results. Twenty per cent by weight of water was removed on degassing which corresponds to the theoretical water of hydration of 21 per cent. This difference is probably due to the fact that the sample was not pure CaSO₁. 2H₂O_•

Thus it is postulated that the initial increase of 6 per cent during adsorption corresponds to hydration from the anhydrous CaSO₄ to the hemihydrate CaSO₄.1/2 H₂O. Physical adsorption appears to take place on this material until the second large increase occurs. It is thought that this corresponds to hydration from the hemihydrate to the dihydrate.

Calculations and B.E.T. plots using the linear form of the equation were made on the postulated adsorption portion of the curve, and are given in Fig. 5. The approximate value of the surface area was found to be 28.4 $\rm m^2/g_{\circ}$ which is a more realistic figure when compared to the value for concrete.

The dimensional change was very large and was beyond the range of the extensometer. But it appears that values obtained show the tendencies exhibited in the weight change (Fig. 6).

In the case of the "hot" sample, separate experimentation by firing showed that the original sample contained 2.54 per cent total water content and so was not in the form of the hemihydrate. During the degassing period the sample lost 1 per cent by weight and re-adsorbed this on adsorption, but it showed no tendency to hydrate as did the "cold" sample.

No dimensional change was made on the "hot" sample.

(b) Fired Samples

It was hoped that these samples would have different structures and that they would be suitable for pure adsorption measurements without any hydration. The isotherms are given in Fig. 7 and the dimensional change results in Fig. 8. It is seen that the adsorption is very small indeed. No B.E.T. calculations were made and so no surface area measurements were possible. The dimensional changes are also of a very small order.

Conclusions

It is apparent that the adsorption characteristics of plaster cannot be studied in this apparatus, because of the decomposition that occurs at high vacuum. It is postulated that during vacuum drying and subsequent exposure to increasing humidities in the test apparatus it dehydrates and then rehydrates in three stages:

- (1) hydration to hemihydrate CaSO_h.1/2 H₂O
- (2) physical adsorption on the hemihydrate
- (3) hydration to the dihydrate CaSO1. 2H2O•

The "hot" sample shows no tendency to hydrate, which raises the following questions:

- (1) what is the constitution of this "hot" plaster?
- (2) why does no hydration take place?

The firing of the samples did not produce samples on which the pure adsorption isotherms could be measured but produced samples which had undergone physical changes resulting from either fusing of the samples, which gave low surface area or some other action which rendered the samples inert to water adsorption.

Reference

1. Mossman, C.E. Moisture adsorption measurements. National Research Council, Division of Building Research, DBR Internal Report 133. Ottawa, October 1957.

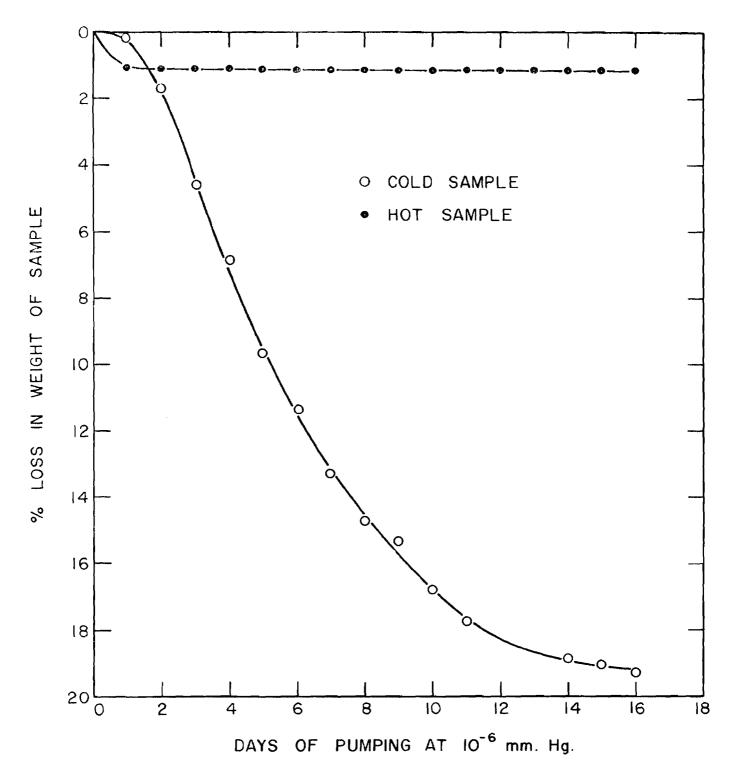


FIGURE I

REMOVAL OF H₂O FROM PLASTER SAMPLE BY

EVACUATION AT 10⁻⁶ mm. Hg

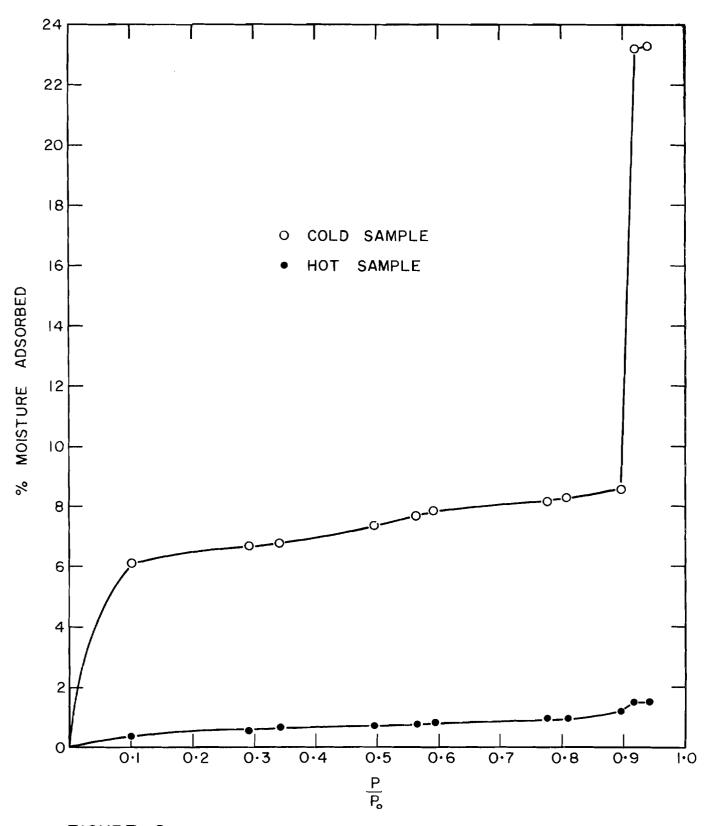


FIGURE 2
MOISTURE SORPTION ON PLASTER SAMPLES

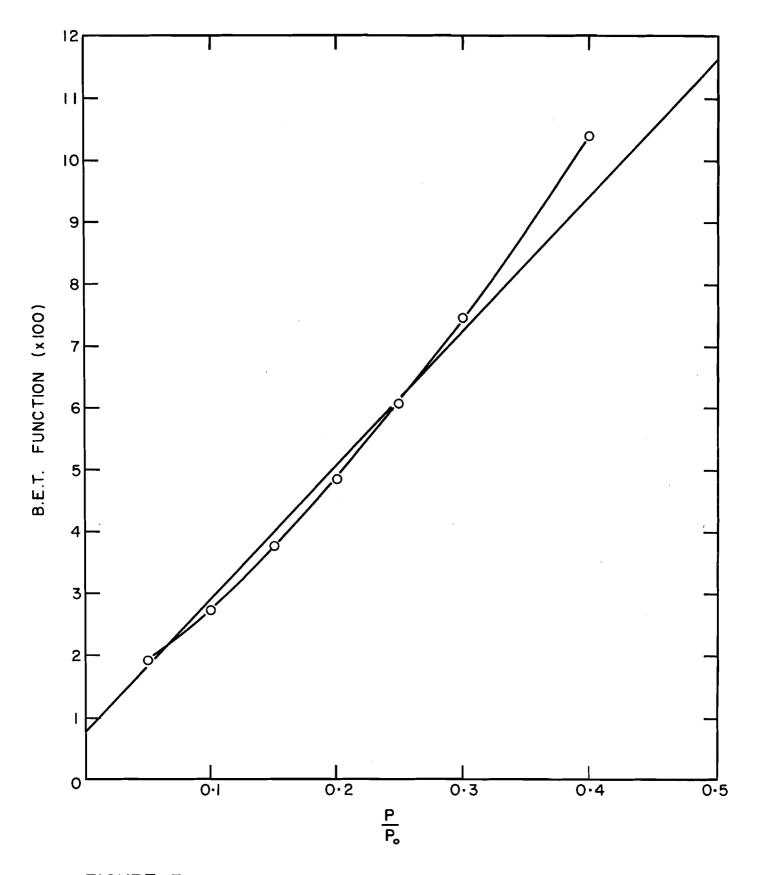


FIGURE 3

B.E.T. PLOT OF "HOT" PLASTER USING ADSORPTION RESULTS. CALCULATED SURFACE AREA = $16.73 \text{ m}^2/g$

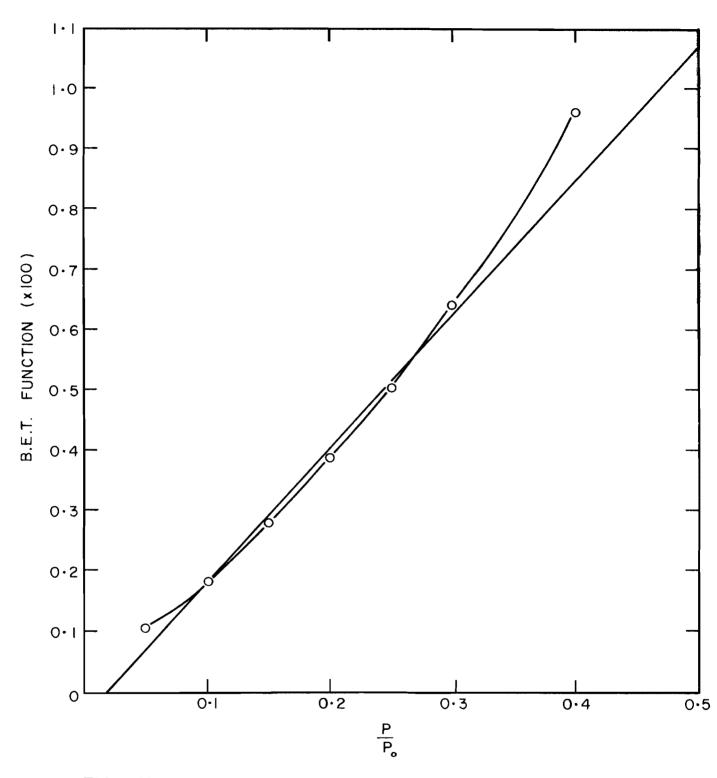


FIGURE 4

B.E.T. PLOT ON "COLD" PLASTER (ASSUMING NO

HYDRATION) USING ADSORPTION CURVE. CALCULATED

SURFACE AREA = $166 \cdot 1 \text{ m}^2/g$

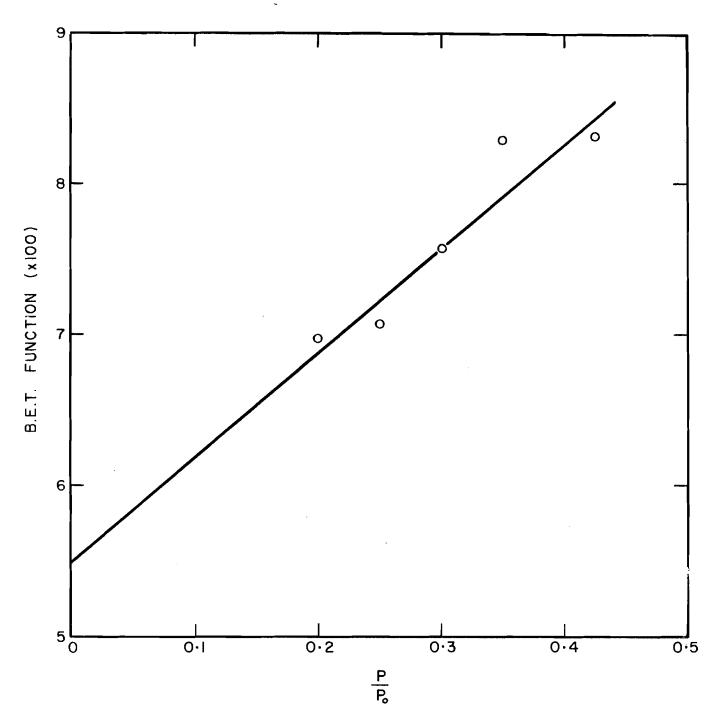
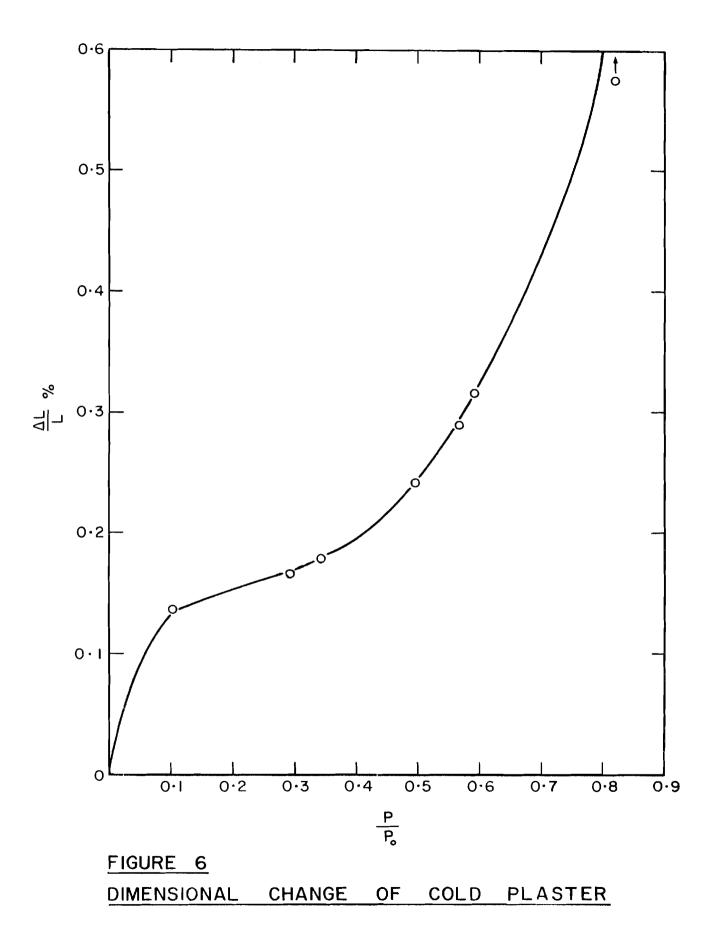


FIGURE 5

B.E.T. PLOT ON "COLD" SAMPLE (ASSUMING HYDRATION OCCURS). CALCULATED SURFACE AREA = 28.42 m/g



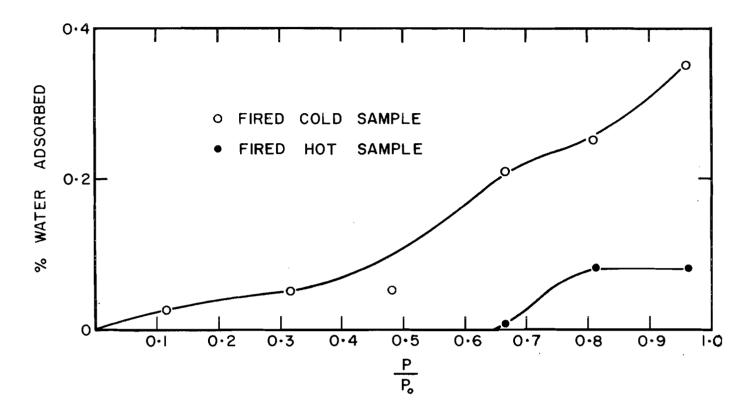


FIGURE 7
ADSORPTION OF WATER VAPOUR ON FIRED PLASTER
SAMPLES

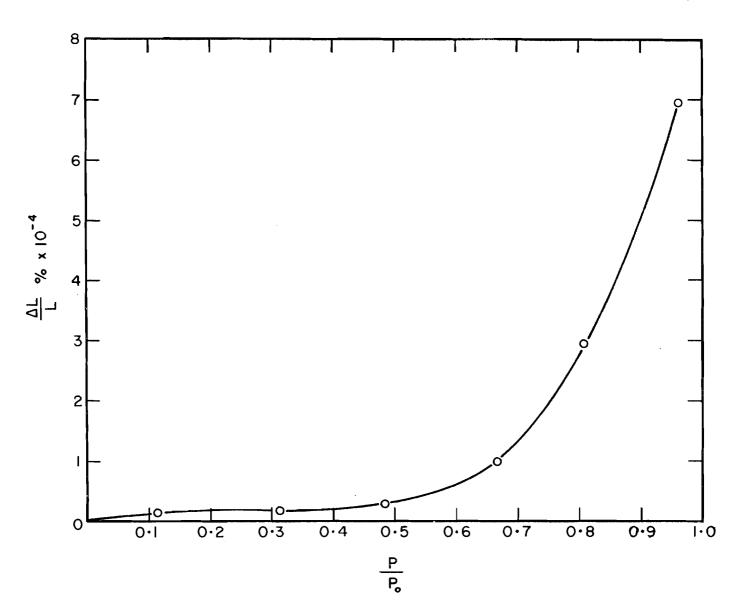


FIGURE 8

DIMENSIONAL CHANGE OF FIRED COLD PLASTER

SAMPLE