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Ramachandran, V. S.; Sereda, P. J.; Feldman, R. F.

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MECHANISM OF HYDRATION OF CALCIUM OXIDE

BY

V. S. RAMACHANDRAN, P. J. SEREDA AND R. F. FELDMAN

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Mechanism of Hydration of Calcium Oxide

THE mechanism of hydration of calcium oxide to form calcium hydroxide has not been satisfactorily established, despite extensive investigation by many workers¹⁻⁶ utilizing techniques of X-ray diffraction, sorption, radio-active tracers and electron microscopy.

The present investigation follows the hydration of calcium oxide by measurements of dimensional and weight changes and determination of pore-size distribution. Hydration was effected by water in liquid as well as vapour phase at a temperature of $73^{\circ} \pm 1^{\circ}$ F.

Calcium oxide was obtained by heating precipitated calcium carbonate in a muffle furnace for 3 h at various temperatures in the range 800° – $1,200^{\circ}$ C. Compacts were formed at loads varying from 10,000 to 100,000 lb. in a mould of diameter 1.25 in. (ref. 7) in a dry box to protect them from exposure to moisture. Dimensional changes were determined by placing the compacts over a perforated stand in a glass cell equipped with an optical window. After evacuation the cell was connected to a source of water kept at a constant temperature. Any desired humidity could be obtained by adjusting the temperature of water. The dimensional changes in the compact, expressed as percentage of linear expansion, were noted by a travelling microscope to within 0.001 cm. Pore-distribution values were determined by a mercury porosimeter down to a pore diameter of 0.015μ . Sorption balance was used for investigating the weight changes occurring in calcium oxide exposed to water vapour. Typical results obtained with calcium oxide, formed at $1,000^{\circ}$ C, compacted under a load of 20,000 lb. and treated with distilled water or exposed to a constant relative pressure of 0.5 are presented and discussed.

The total porosity of calcium oxide compact was 46 per cent. The pore distribution values show that 79 per cent of the porosity is due to pores of diameter 1– 2μ . There are practically no pores of diameter less than 0.1μ . The pore distribution in 1 c.c. of the compact is shown in Fig. 1.

Contrary to expectation, the compact did not exhibit any disintegration after immersion in water for 48 h. Linear expansion was 8 per cent. Calcium hydroxide content in the water-treated sample was estimated at 99.8 per cent, indicating thereby that calcium oxide had almost completely reacted to form the hydroxide. The total porosity of the resulting calcium hydroxide disk was as low as 17.5 per cent, compared with a porosity of 46 per cent in the untreated calcium oxide compact. Pore-size

Fig. 1. Histograms of pore-size distribution in 1 c.c. of calcium oxide compact before and after liquid phase hydration. A, calcium oxide compact; B, liquid-hydrated disk

distribution values for calcium oxide compact before and after hydration show that most of the pores of diameter 1–2 μ have decreased to less than 0.1 μ during the formation of calcium hydroxide and that 78 per cent of the total pore volume is due to pores of diameter less than 0.1 μ . In terms of volumetric changes, 100 c.c. of calcium oxide compact constitutes a pore volume of 46 c.c. and a calcium oxide volume of 54 c.c. On immersion in water it expands to 123 c.c., in which calcium hydroxide occupies 107 c.c. and the pores 16 c.c. Formation of calcium hydroxide results in a decrease in porosity of 66 per cent.

After exposure to relative water vapour pressures of 0.25 and 0.5, calcium oxide compacts show enormous expansion. In Fig. 2 the percentage of linear expansion ($\Delta L/L \times 100$) is plotted against time in hours for a compact exposed to a relative pressure of 0.5. The rate of expansion is comparatively slow for a few hours and is followed by an almost linear increase in length up to 55 h, after which the curve continues to rise at a slower rate. After 200 h the expansion may be as high as 100 per cent; beyond 200 h

Fig. 2. Dimensional and weight changes of calcium oxide compact exposed to water vapour. A, Linear dimensional change; B, change in weight

the compact gradually disintegrates into a fine powder. Results of changes in weight in calcium oxide compact subjected to water vapour show a slow rate of increase in weight for a few hours followed by a linear increase, after which the weight remains constant.

After a period of 50 h, calcium oxide takes up in chemical combination a stoichiometric quantity of water to form calcium hydroxide; the linear expansion for this water content is about 60 per cent. Expansion beyond 60 per cent may be the result of physical adsorption of water and agglomeration of calcium hydroxide particles due to an 'ageing effect'.

The total pore volume of the expanded compact, corresponding to 60 per cent expansion, can be as high as 70.7 per cent. The pore-size distribution could not be determined for the expanded sample; it was too fluffy and crumbled into a fine powder on application of even a slight pressure during handling. The pore-size distribution values for the powder, however, showed that more than 80 per cent of the pores are larger than 2μ in diameter and that 60 per cent have diameters in the range 2μ to 6μ . In terms of the apparent volume of the compact itself the former value would be much higher. In terms of volumetric changes, 100 c.c. of calcium oxide containing a pore volume of 46 c.c. and calcium oxide volume of 54.0 c.c. increase on exposure to water vapour to 408 c.c., in which calcium hydroxide occupies 107 c.c. and pores 301 c.c. Expansion brings about 555 per cent increase in pore volume.

On the basis of the foregoing results, the hydration

mechanism in the presence of liquid water may be visualized as follows: calcium oxide, subjected to treatment with excess water, undergoes hydration predominantly through solution and crystallization as envisaged by Briss and Thorvaldson⁵. Owing to the presence of large pore volume, crystallization through solution facilitates the formation and growth of crystals in the pores as well as in the spaces previously occupied by calcium oxide. Hence there is a large decrease in the pore volume as well as in the pore size (Fig. 1). Some stress, resulting in a linear expansion of 8 per cent, could develop during crystallization in narrower pores.

If hydration occurs at the solid-vapour interface as a topochemical or solid-state reaction, the mechanism is as follows: the product is formed at the original sites of the reactant. As the specific volume of the product is much greater (1.98 times) (ref. 8) the products are pushed from these sites and there is a large over-all expansion. This is in contrast to the diffusion mechanism operative in the hydration in liquid phase.

The fact that liquid phase hydration, compared with vapour phase hydration, gives such a low expansion explains the effectiveness of 'docking' (immersion in water) in the prevention of 'lime blowing' in bricks⁹. Time-expansion investigation of the vapour phase could provide a new method of determining the reactivity of limes. The technique is being applied to an investigation of the hydration of cements and plaster at the Division of Building Research.

V. S. RAMACHANDRAN
P. J. SEREDA
R. F. FELDMAN

Division of Building Research,
National Research Council,
Ottawa.

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