

SURFACE PROPERTIES AND MICROSTRUCTURE OF SOME AUTOCLAVED BUILDING PRODUCTS

A THESIS

Presented to
Faculty of Science
Ain Shams University
Cairo

By
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M. Sc.

For the Degree of
DOCTOR OF PHILOSOPHY
in
CHEMISTRY

1988



قالوا

يُجَبِّحُ بِكَ لَا إِلَهَ إِلَّا اللَّهُ الْحَمْدُ
إِنَّكَ أَنْتَ الْعَالِمُ الْحَكِيمُ

صَدَقَ اللَّهُ الْعَظِيمُ

سورة البقرة (الآية ٢٢٢)



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OF SOME BUILDING PRODUCTS

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ACKNOWLEDGEMENT

I wish to express my deepest gratitude to Dr. S.A.ABO-EL-ENEIN, Professor of Physical Chemistry, Faculty of Science, Ain Shams University, Cairo, for suggesting the subject of this thesis and for his continual interest valuable and helpful guiding throughout the course of this work.

I am also greatly indebted to Dr.M.A.EL-KHALIK Professor of Physical Chemistry, Faculty of Science, Ain Shams University, for his valuable advice during the course of this work.

I wish also to express my deepest gratitude to Dr. E.E.HEKAL, Lecturer of Physical Chemistry, Faculty of Science, Ain Shams University, for his effective, helpful and valuable sharing throughout the course of this work.

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CHAPTER I

INTRODUCTION AND OBJECT OF INVESTIGATION

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CHAPTER I

INTRODUCTION AND OBJECT OF INVESTIGATION

I.A. Introduction:

Several publications have been reported on the hydrothermal reaction of lime-silica, lime-cement, lime pozzolan and cement silica mixtures. In such publications, the effect of lime/silica ratio and autoclaving conditions as well as the effect of the addition of alkalis and some metal oxides have been studied.

Suzuki et al⁽¹⁾ investigated a mixture of quartz and hydrated lime autoclaved at a steam pressure of 10Kg/cm² for 24-96 hrs. Maximum bending strength occurred after autoclaving for 24 hrs, and the maximum compressive strength, after 48 hrs. Morphological examination revealed fibrous or wrinkled foils of semi-crystalline tobermorite at 12-24 hrs and platy crystalline tobermorite at 48 hrs. These products may be C-S-H (II) and C-S-H (I), respectively. The mechanical and morphological examination of these products were also investigated by heating them up to 800°C. The products formed in Ca (OH)₂-SiO₂ gel autoclaved at 140-300°C showed C-S-H below 180°C and xonotlite or gyrolite below 250°C⁽²⁾. The compressive strengths of the products depended on the C/S ratio, maximum values occurring at C/S ratios of 2/3 and 1/3. The work of Kubo and others⁽³⁾ on the Ca(OH)₂-SiO₂ system at about 191°C and 12 kg/cm² showed the formation of spherical

tobermorite, some of it transforming to xonotlite; temperatures above 191°C favoured the formation of xonotlite.

Work has also been done using CaO , SiO_2 and other additives. The effect of $\gamma\text{-Al}_2\text{O}_3$ or NaOH on the formation of tobermorite was investigated⁽⁴⁾. Using starting materials such as CaO with colloidal silica, CaO with aluminosilica gels, or CaO with clinoptilolites at a temperature of 90° or 120°C . The mixtures had $\text{C}/(\text{S}+\text{A}) = 0.8$ or 1.0 or $\text{Ca}/(\text{Si}+\text{Al}) = 0.5, 0.10$ or 0.15 . The products were examined by XRD, SEM, DTA and X-ray fluorescence. The reaction proceeded through the formation of C-S-H gels to $11 \text{ \AA}$ tobermorite. Tobermorite was found to crystallize more rapidly at $\text{C}/\text{S} = 1.0$ than at $\text{C}/\text{S} = 0.8$ in the mixtures containing no aluminium. When aluminium was present tobermorite crystallized more rapidly at $\text{Ca}/(\text{Si}+\text{Al}) = 0.8$ than at $\text{Ca}/(\text{Si}+\text{Al}) = 1.0$. Tobermorite, showing anomalous thermal behavior was produced in the presence of both Al and alkali and its formation was favoured when the ratio $\text{Ca}/(\text{Si}+\text{Al})$ was 0.8 or less.

The role of water in a cement paste is still not completely understood. Chatterji⁽⁵⁾ prepared $11.3 \text{ \AA}$ tobermorite by autoclaving a lime-silica mixture and stored the material in water for 14 days. The wet, as well as

the material dried at 130°C , was investigated by XDR. Weight and length changes were obtained on cut samples exposed to conditions as follows: (a) Soaking in water for 3 days, (b) drying at 105°C , (c) again soaking in water for 3 days, (d) equilibrating at 43% RH, (g) re-soaking in water for 3 days, (h) drying at 105°C , and (i) drying at 130°C . He concluded that soaking in water does not increase the interlayer spacing and drying does not decrease it. In a discussion of the above paper Feldman⁽⁶⁾ argued that the results were not applicable to the hydrated portland cement. The system studied by Chatterji was radically different from the poorly-crystallized hydrated portland cement and the shrinkage values could be explained by the normal Bangham type length change. In addition, poorly-crystalline materials such as C-S-H (I) and C-S-H(II) may have been present in samples studied by Chatterji, and water loss could be due to these phases. Alternatively, $11 \text{ \AA}$ tobermorite may have decomposed at a slow rate by the static heating between 100 and 300°C , but this may not have been detected by XRD.

In the system $\text{Na}_2\text{O}-\text{CaO}-\text{SiO}_2-\text{H}_2\text{O}$, studied at 150°C , an increase in the concentration of NaOH led to a decrease in the reactivity of quartz and suppression of crystallization of tobermorite from C-S-H^(7a). At longer times

(4 to 128 days) formation of pectolite ($\text{NaCa}_2\text{Si}_3\text{O}_8\text{OH}$) was promoted. Chuichi and Kawaguchi^(7b) prepared mixtures containing 0.83 mole CaO : 1 mole SiO_2 and 2 to 20 mole percent Cr_2O_3 by Co-precipitation, and autoclaved them between 150 and 530°C for 2 to 156 hrs. Chromium oxide substitutes to an extent of 12 mole percent in C-S-H (I); tobermorite, truscottite, gyrolite and xonotlite are other products. The basal spacing of C-S-H (I) decreased from 11.5 Å to 10.85 Å when it contained chromium. Autoclaving of mixtures containing CH and quartz (C/S ratio= 1.3 to 2.0) at 350°C, produced kilchoanite at C/S= 1.5 under a rapid heating regime. This product was accompanied by xonotlite at C/S = 1.3 and by calciochondrodite at C/S= 2.0. At slower heating rates the products were xonotlite, foshagite and dellaite.

Collepari et al⁽⁸⁾ investigated low pressure steam curing at 50-90°C of compacted lime-pozzolan mixtures. The reaction products were composed mainly of amorphous C-S-H gel similar to that obtained at room temperature and a poorly-crystallized tobermorite which is normally obtained in the autoclave hydration of lime-quartz and cement-quartz mixtures. In the low pressure steam treatment the percentage of combined lime does not increase but even decreases if the temperature is brought above 70°C. The influence

of the lime/Pozzolan ratio on the amount of combined lime changes with the type of pozzolan. The pore structure of the material obtained after the steam treatment consists of pores having diameters in the range 0.02-0.2 μm belonging to the reaction product and of pores in the 0.2-1 μm range initially present in the pozzolan. It appears that the use of pozzolans as siliceous materials would make possible the fabrication of construction materials having good strength through a simple, low pressure steam treatment instead of the more expansive treatment in the autoclave.

Sauman⁽⁹⁾ investigated reactions between power station fly ash and calcium oxide under hydrothermal conditions. Power station fly ash (2900 cm^2/g specific surface) was mixed with lime in a weight ratio 3:1. The mixtures were autoclaved at 175°C and 0.9 MPa for varying times, subsequently cooled and dried in N_2 at 105°C, and examined by DTA, XRD and electron microscopy. After hydrothermal treatment for 2-4 hours C-S-H (I), 11 Å tobermorite and hydrogarnet (with approximate composition C_3ASH_4) had formed. After 4 hrs C-S-H (I) changes into 11 Å tobermorite. Mixtures of fly ash and lime in various weight ratios (1:1-10) were autoclaved and the largest relative amount of tobermorite phase was found in 2:1 and 3:1 mixtures.

Sauman⁽¹⁰⁾ in another investigation explored the conditions for the formation of and the character of binding phases in autoclaved building materials. Imperfectly developed crystalline $11\text{\AA}$ tobermorite and CSH_n phases are the major components in compact silicate concretes. Tobermorite is formed in cellular concretes produced from quartz sand or fly ash and is recognized by the well developed tabular and leafy crystals which are mostly found in pores and microfissures of the substance. In asbestos cement products, tobermorite with a different lattice arrangements is the main contributor to mechanical strength. In fibrous insulating and structural materials xonotlite is the dominant binding phase providing that amorphous silica has been used. Highest compressive strengths are obtained when the products include tobermorite and imperfectly developed crystals. The C-S-H_n component is characterized by a significantly lower binding ability, while the hydrogarnet phase has none and can even destroy the structure of the products.

The characteristics of autoclaved lime-silica products attract continuing interest. Sauman⁽¹¹⁾ studied the morphology of the binding components (amorphous gels, and several varieties of $11\text{\AA}$ tobermorite) as functions of the type and grains sizes of the filter used. An earlier paper⁽¹²⁾ studied the character of products formed