

**Experiment title:****Characterization of the transient phases during the hydration of C3A and C4AF****Experiment number:**

CH-1249

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Report:

The chemical reactions occurring when clinker Aluminate phases come in contact with water are of great interest, because they occur during the first stages of the process of setting of cements.

The Afm type [1] phases formed during the hydration of C3A ($\text{Ca}_3\text{Al}_2\text{O}_6$) and C4AF ($\text{Ca}_2\text{AlFeO}_5$) have a layered structure, and water or anionic groups could be allocated between layers.

The stability of the highest hydrated phases is restricted to low temperature ($< 30^\circ\text{C}$) and high humidity.

Experiments were performed to characterize these phases by in-situ X-ray powder diffraction (XRPD). C3A and C4AF were contained in a 1 mm glass capillary; in-situ hydration was achieved by injection of water by a peristaltic pump after the first X-ray pattern was collected. Several patterns were collected during the first 2-3 hours, to follow the kinetics of the reactions.

Fig. 1 shows some XRPD patterns related to the hydration of C3A. The higher hydrate Afm phase (the series C2AH8-C4AH19) decompose to a lower hydrate Afm phase releasing water to the system and finally the more stable phase hydrogarnet (C2AH6) develops.

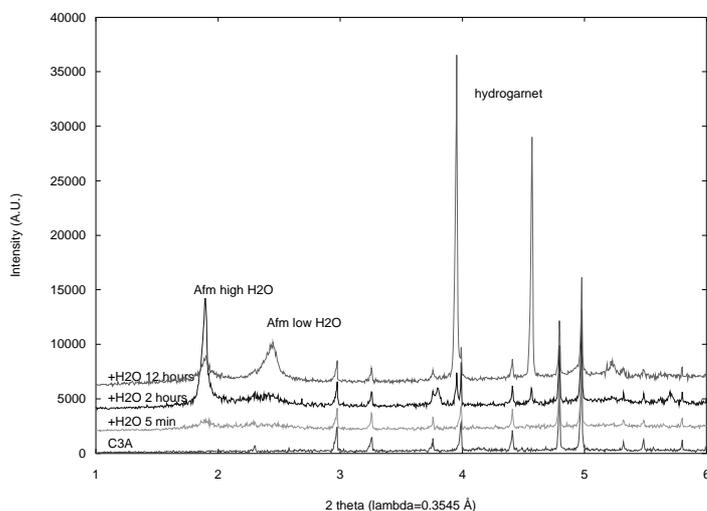


Fig. 1 – Some XRPD patterns related to the kinetic study of the hydration of the C3A phase.

The presence of sulphate ions inhibits the formation of Afm phases and Aft type phases – mainly ettringite – develop [1-2].

Other experiments were performed to follow the kinetics of ettringite formation in systems composed by C3A and bassanite (Ca-sulphate hemihydrate), C4AF and bassanite and in mixture of Portland, Al-cements and bassanite. The XRPD patterns were analyzed by Rietveld method. Fig.2-3 show a plot of the calculated weight fractions of the phases present in the system C3A-CaSO4-H2O and in a more complex system.

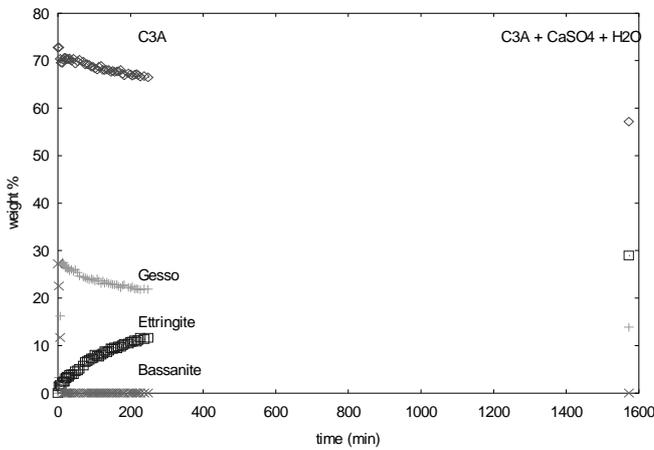


Fig.2 – Plot of the calculated weight fraction of the phases present in the system C3A-CaSO4-H2O

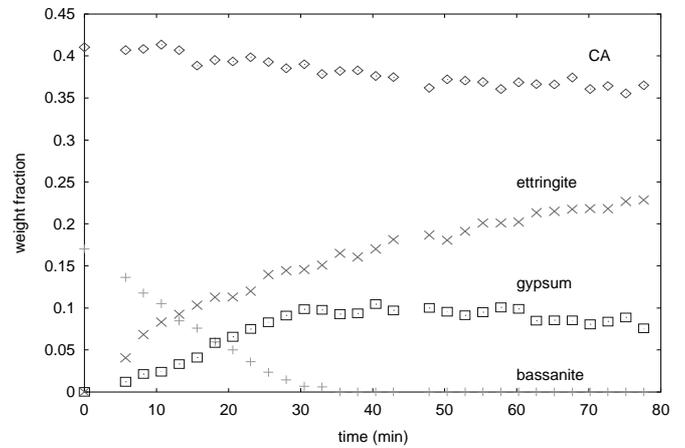


Fig.3 – Plot of the calculated weight fraction of some phases present in a mixture Portland-Al cement-CaSO4

The presence of organic polymers such as superplasticizers, retarding agents... affect the chemistry and the kinetics of the system. Fig. 4 is related to the hydration of C3A-CaSO4 in the presence of a solution of a commercial naphtalene-sulphonate superplasticizer. The apparent increase of C3A content is justified by the presence of an amorphous phase rich in polymers and sulphate ions, as showed by an estimate of the chemical composition of the crystalline phases recalculated assuming a pure composition of the phases.

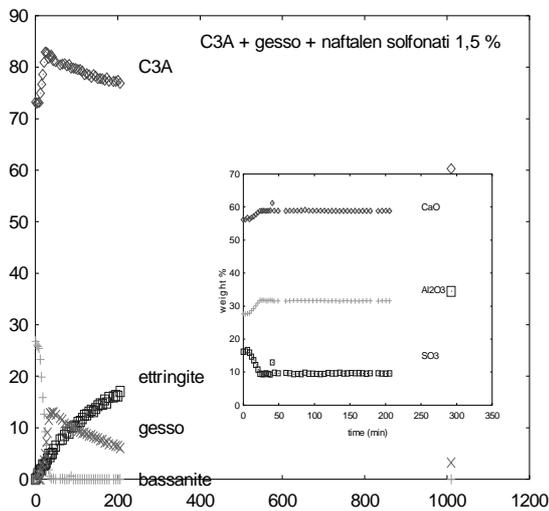


Fig. 4 – Plot of the calculated weight fraction obtained by Rietveld method (using the GSAS software [3]) of the phases in the system C3A-CaSO4 hydrated with a solution 1.5 % of naphtalen-sulphonate based superplasticizer. During hydration an amorphous phase rich in polymer and sulphate ions forms. A recalculation of the chemical composition of the phases reveal a decrease in the content of sulphate in the crystalline part of the system.

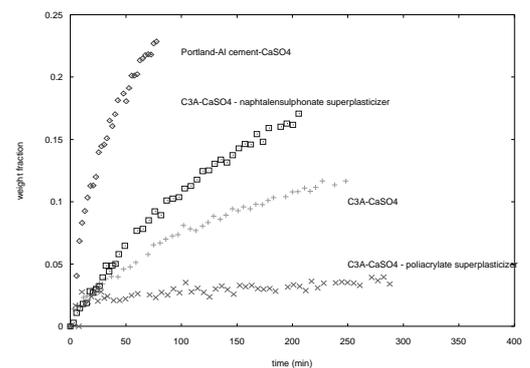


Fig. 5 – Plot of the weight fraction of ettringite formed in different systems. Ettringite nucleates and grows through a gel phase, as revealed by independent ESEM studies. The composition of the gel (i.e. Ca++ content, the presence of organic polymers...) affects the kinetics of the reaction

REFERENCES

- [1] Taylor, H.F.W. (1990). Cement Chemistry, Academic Press, New York
- [2] Bensted J. & Barnes P. Ed.(2001). Structure and Performance of Cements, 2nd Ed., Spon Press, London & New York
- [3] Larson A.C. & Von Dreele R.B. (1988). Los Alamos National Laboratory Report LAUR 86-748