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Study of Nucleation and Growth Processes of Ettringite in Supersaturated Diluted Solutions

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Abstract : Ettringite $Ca_6Al_2(SO_4)_3(OH)_{12}26H_2O$ is one of the major hydrates formed during cement hydration. Ettringite forms in Portland cement from the reaction between tricalcium aluminate $Ca_3Al_2O_6$ and calcium sulfate. Ettringite is also present in calcium sulfoaluminate cement in which it is the major hydrate, formed by the reaction between yeelimite $Ca_4(AlO_2)_6SO_4$ and calcium sulfate. About the formation of ettringite, numerous results are available in the literature even if some issues are still under discussion. However, almost all published work about ettringite was done on cementitious systems. Yet in cement, hydration reactions are very complex, the result of dissolution-precipitation processes and are submitted to various interactions. Understanding the formation process of a phase alone, here ettringite, is the first step to later understand the much more complex reactions happening in cement. This study is crucial for the comprehension of early cement hydration and physical behavior. Indeed formation of hydrates, in particular, ettringite, will have an influence on the rheological properties of the cement paste and on the need for admixtures. To make progress toward the understanding of existing phenomena, a specific study of nucleation and growth processes of ettringite was conducted. First ettringite nucleation was studied in ionic aqueous solutions, with controlled but different experimental conditions, as different supersaturation degrees (β), different pH or presence of exogenous ions. Through induction time measurements, interfacial ettringite crystals solution energies (γ) were determined. Growth of ettringite in supersaturated solutions was also studied through chain crystallization reactions. Specific BET surface area measurements and Scanning Electron Microscopy observations seemed to prove that growth process is favored over the nucleation process when ettringite crystals are initially present in a solution with a low supersaturation degree. The influence of stirring on ettringite formation was also investigated. Observation was made that intensity and nature of stirring have a high influence on the size of ettringite needles formed. Needle sizes vary from less than 10µm long depending on the stirring to almost 100µm long without any stirring. During all previously mentioned experiments, initially present ions are consumed to form ettringite in such a way that the supersaturation degree with regard to ettringite is decreasing over time. To avoid this phenomenon a device compensating the drop of ion concentrations by adding some more solutions, and therefore always have constant ionic concentrations, was used. This constant β recreates the conditions of the beginning of cement paste hydration, when the dissolution of solid reagents compensates the consumption of ions to form hydrates. This device allowed the determination of the ettringite precipitation rate as a function of the supersaturation degree β. Taking samples at different time during ettringite precipitation and doing BET measurements allowed the determination of the interfacial growth rate of ettringite in m^2/s. This work will lead to a better understanding and control of ettringite formation alone and thus during cements hydration. This study will also ultimately define the impact of ettringite formation process on the rheology of cement pastes at early age, which is a crucial parameter from a practical point of view.

Keywords : cement hydration, ettringite, morphology of crystals, nucleation-growth process
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