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Recent advances in the field of cement hydration and microstructure analysis

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ABSTRACT

This paper is a bibliographic tool reviewing experimental and theoretical studies related to cement hydration and microstructure development that have been published within the four years of the interim period between the 12th and 13th International Congress on the Chemistry of Cement.

Contents

1. Introduction

As a contribution to the 13th International Congress on the Chemistry of Cement, the present paper is intended to review theoretical and experimental studies related to hydration of cement and microstructure of hardened cement paste, mortar and concrete. However, other review papers have discussed these subjects recently [224–227,359]. In order to avoid reiteration, it is attempted to provide a more general overview on new data.

The present manuscript is based on relevant studies published in Cement and Concrete Research, Cement and Concrete Composites, Materials and Structures, Journal of the American Ceramic Society, and Advances in Cement Research between 2007 and 2010. A few other publications have been included that were found to be of primary interest. In order to reduce the data to a manageable amount, almost bibliographic completeness has only been preserved in the core fields of this article. Other aspects have been passed in a less detailed manner and will be discussed thoroughly in other reviews during this congress. Most important are links to thermodynamic and simulation studies, characterisation of binders, rheology and early age properties, diffusion and transport, advances in instrumental analytics, and microstructural modifications induced by the interaction of hardened cement paste with aggressive species upon long term exposition.

Hydration of cement is the combination of all chemical and physical processes taking place after contact of the anhydrous solid with water. Chemical reactions of clinker minerals and mineral admixtures play a major role, but other aspects such as agglomeration, adsorption, evaporation, and release of thermal energy need to be considered as well. Most information on this processes stem from experimental investigations being complemented by simulation studies using theoretical approaches to provide a better understanding of the underlying principles driving the interactions. Section 2 is intended to review such fundamental approaches to cement hydration and microstructure development whereas the focus of Section 3 is set on experimental data on the chemical reactions. Recent experimental results related to properties of the hydration products are included in Section 4. The development of the microstructure is a combination of all physico-chemical interactions described in paragraphs 2 to 4 and Section 5 is devoted to this part of the

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field. Most of the results obtained on pure systems are modified by the interaction with mineral additions as well as with organic and inorganic admixtures. Recent advances in studying these systems are considered in Section 6.

2. Fundamental studies including simulation and modelling

Simulation and modelling tools are a powerful extension of traditional experimental investigations. Uncertainties and weak spots in experimental studies can be discussed and quantitative predictions can be made.

Most of the theoretical models and computational facilities that are increasingly applied to cement science have originally been developed in other disciplines. Progress is based on fundamental studies in physics, chemistry, and mineralogy. Better computational tools, theoretical models, improved instrumentation and analytic data developed in these disciplines are increasingly used by the cement science community. Traditionally, thermodynamic calculation also including the computation of supersaturation and phase diagrams play an important role in theoretical studies related to cement chemistry. The kinetic description of chemical reactions is often based on empirical equations whereas the implementation of theories from physical chemistry is still limited to a few working groups.

Improved computational facilities for the investigation of solid phase properties have only been used in a few cases in the reviewed period. Ab initio methods were applied to study the structure of crystalline and semi-crystalline calcium-silicate-hydrates [1–3,88,381], the structure of water at fluid–liquid interfaces [4,5], and the hydration of geopolymers [6].

Chemical reactions can be predicted by thermodynamic calculation. However, the results obtained by computation heavily rely on the accuracy of the data which is employed in these calculations. Thermodynamic properties of solid phases have been derived by solubility measurements [7,8,279,286] and computational methods [9,292]. Calculations based on these and other data have been used to quantify enthalpy changes [341] and to develop phase diagrams related to cement hydration [7,10–15,42,92,268,279,285,316]. The formation of AFm and AFt phases in pure systems and Portland cement has been within the focus of most of these studies also discussing miscibility gaps and the effect of temperature.

Kinetic descriptions of the hydration process have used different approaches ranging from semi-empirical equations to fundamental approaches [15–20,277,460,505]. There is a strong focus on nucleation and crystal growth during hydration of C_3S. It can be expected that a better understanding of such processes will strengthen the application of finite element calculations and related techniques to predict mechanical and other properties of cementitious materials as has been attempted in a few studies [18,21–23]. The quality of the results crucially depends on the theoretical framework and availability of phase properties. Single systems have been investigated to derive information on cohesive forces between crystals which appear to be the strength determining factor in concrete [24–26].

3. Experimental results related to chemical processes

Research related to the chemical reactions during cement hydration has been attracted by the interest in the fundamental principles underlying these reactions as well as in the rate of reaction expressed as evolution of degree of hydration over time. A broad spectrum of experimental facilities has been applied for studying pure systems, Portland cement and blended cements.

The C_3S-water system has the highest significance of all pure systems and has subsequently been studied in a number of reports. The pre-hydration of the anhydrous solid upon contact with gaseous water during storage has been described by a surface sensitive technique [27]. Hydration studies have relied on experimental as well as on theoretical and computational efforts [16,17,19,20,28,70,225,244,296,362,374,415].

It is widely accepted that the hydration of C_3S proceeds in different stages. The initial reaction after contact with water is terminated within a few minutes. A period of very low chemical activity follows, lasting for 1 to 6 h, which has been designated as dormant or induction period. A number of hypotheses have been presented in the past to explain the presence of the induction period, most prominent being the formation of a metastable hydrate layer and dissolution-growth models. Both ideas have been supported by recent works [16,17,28,225,229,230] and the dissolution of C_3S has been studied by experimental and theoretical means [16,17,20,28,70,159,228,244]. Despite its origin being still a matter of debate, the length of the induction period was found to be manipulable by the addition of calcium hydroxide, calcium carbonate [229], artificial C–S–H [29], and nano-sized SiO_2 [69]. The end of the induction period marks the onset of the main hydration period that is associated with the formation of massive amounts of C–S–H and calcium hydroxide [32] as well as with the release of thermal energy.

It has been suggested to replace C_3S and other clinker minerals by crystalline C–S–H produced in an autoclave process and rendered more reactive by co-grinding with quartz [30].

The reaction of C_3A has been investigated in pure and cementitious systems [33–37,238,353,426]. The early hydration of C_3A plays an important role in concrete rheology and is much affected by the dissolution rate of calcium sulphate which itself depends on a number of factors such as presence of organic admixtures, anhydrite/gypsum relation, and size of C_3A particles. Most of these factors have a direct impact on the amounts of AFm and AFt being formed.

The hydration of Portland cement has been addressed in a high number of studies focussing on special properties or the performance of specific admixtures and mineral additions. However, a few fundamental studies providing new data on degrees of hydration of individual clinker minerals, amount of hydrates formed, pore solution chemistry and heat release have been published [15,61,66,71,83, 85,231,423]. It has been derived that temperature, the addition of limestone to Portland cement, and the availability of water may induce significant modifications to the AFm–AFt balance. The consumption of clinker minerals has been monitored by quantitative X-ray diffraction based on the Rietveld method. As better laboratory equipment and software become available, it is expected that new data will be added in the near future. The results obtained in pure systems can be compared to blended systems and the performance of admixtures can be evaluated by this method, too. Still difficult is the assessment of the amorphous phase in hydrated systems and a few approaches are currently tested for this purpose. Pore solution chemistry has been studied for a long time and improved evaluation software for the calculation of activity coefficients at high ionic strengths enables the calculation of supersaturations for relevant mineral phases from this data. It is expected that this method will provide new insights into the chemical processes taking place during hydration. It will also support investigations on the binding of minor elements such as iron, magnesium, manganese, and alkalis.

Supplementary information was provided by other studies targeting on new cementitious products, mineral additions, admixtures, early age properties, and novel techniques [62–65,67,68,72,73,76,80,277,279,299, 301,319,340,355,360,374,399]. The results obtained for the reference materials or conditions used in these investigations and the ones in Section 6 can be compared to the data in the fundamental studies mentioned above.

The early stage of cement hydration plays a decisive role on workability. Many efforts have been spent to describe the rheology of cement paste, mortar, and concrete. References related to this topic are included in Section 5.

The hydration of cementitious systems under autoclave conditions has been investigated for a wide range of chemical compositions [12,38–43,75,209,218,252,366,378,383,385,388,406]. Fundamental studies have focused on the hydration of C_3S at elevated temperature and pressure as well as on phase diagrams in the C–A–S–H system. Applied research has primarily been driven by the need to find appropriate sealing materials for oil well drilling.

New cementitious systems have been investigated, most prominent being geopolymers [6,31,44–48,83,85,129,144,175,176,187,200,207,236,245,270,290,291,295,306,316,364,368–370,372,375,382,387,392,396,398,405,407–409,411,419,433,462,479,488,503,509]. A broad spectrum of methods ranging from ab initio calculation to compressive strength testing has added new data related to the performance of this material. Other systems have attracted much less attention such as:

- magnesium oxychloride cement [49,329,401,468,525]
- belite cement [50,68,76,414]
- supersulphated slag cement [51]
- aluminous cement [52]
- calcium sulfoaluminate cements [74,77,251,278,285,320,333,342,345,471,506]
- calcium aluminate cement [56,275,300,403]
- calcium phosphate cements [343,386,413,418,420]
- portlandite/brucite binders [78,81,327,401,404].

The activation of blast furnace slag has been studied in low alkaline solutions [58].

4. Properties of hydration products and hardened cement paste

Most properties of concrete depend much on the quality of the hardened cement paste and the interface between hardened cement paste and aggregate. The hardened cement paste produced by the interaction of Portland or blended cement with water is composed of anhydrous phases (unreacted clinker minerals, mineral additions), hydrate phases modified by foreign ions incorporation and adsorption (C–S–H, ettringite, portlandite, AFm), minor phases such as hydrotalkite, pore solution, and pores.

The hydrate phase most abundant in hardened cement paste is calcium silicate hydrate, denoted C–S–H. It has remained difficult to study this phase due to a variable chemical composition, the lack of a true long range order, a very high surface favouring the absorption of foreign ions, and the sensitivity to vacuum, elevated temperature, and carbon dioxide. A number of studies have supplied new data and used existing information for the application in improved models or computational facilities [4,8,9,14,24,83–91,93,94,113,235,236,246,276,291,303,304,310,338,347,369,371,381,395,397,400,410,412,421,422,425,429,474,480,499,536]. Progress has been made in the characterization of this phase with respect to its nanostructure, thermodynamic properties, solubility (including the impact of aluminium incorporation), electron binding energies, morphology, adsorption, intercalation of organic molecules, and mechanical properties.

Other phases have been studied including:

- ettringite [7,10,14,95,96,121,252]
- calcium hydroxide [97,121,311]
- crystalline calcium silicate hydrates produced at high temperature and pressure [1,2,9,42,87,373,377,385,389]
- AFm-phases [7,121,279,334,379]
- alkali silicate gel [272].

Foreign ions can be immobilised by various mechanisms such as adsorption on the surface of hydrates, direct precipitation, complex formation, and incorporation in the hydrate structure via ionic substitution. These mechanisms have been studied for a wide range of elements [107–113,118,129,148,223,232,236,258,265,287,302,305,310,321,335,508]. Some of these elements are intrinsically present such as alkalis, whereas others can be added in order to manipulate specific properties of the hardened concrete. Scientific interest in foreign ion immobilisation is to large extent associated with attempts to use cement for waste stabilisation, also including radioactive waste. Supplementary information is available from studies on the performance of inorganic admixtures (Section 6).

Hydration may result in self-desiccation and cause shrinkage of concrete during hardening. Water can be adsorbed and desorbed during hydration and service life contributing to volume changes [59,60,116,117,248,253,273,278,280,304,326,328,341,347,383,395,432,437,443,453,461,467,470,480,513,516,529,534,537].

5. Microstructure of hardened cement paste, mortar, and concrete

Concrete is material that undergoes a change from plastic to elastic state due to chemical reactions taking place in the first few days after mixing. While being in the plastic state, the properties have extensively been described by rheology [35,36,53–57,82,120,130–136,212,217,221,237,239,249,254,262,271,297,298,322,346,442,448,449,459,464,472,481,493,494,504,518,523,535]. Apart from the publication of new data on specific systems and methods, review papers and theoretical studies applying fluid mechanics and finite element techniques have been published. New data has underlined that the beginning of the main hydration period is directly connected to setting time and the onset of rapid gain in yield stress. Studies related to admixtures such as superplastizisers and their effect on rheology are summarised in Section 6.

The amount of water added during concrete production has a serious impact on its workability. Later on, much of the mixing water is consumed in chemical reactions with the cement; the remainder still being in the liquid state is enclosed in the microstructure. The remaining water is directly responsible for the porosity of the hardened cement paste affecting mechanical and transport properties of concrete [63]. The amount and distribution of pore sizes has been characterised by mercury intrusion porosimetry (MIP), a method with its own features and limitations. Other methods such as ultrasound attenuation, adsorption measurements, small angle neutron scattering, X-ray tomography, ^{1}H NMR spectroscopy, and thermoporosity have added supplementary information, often in combination with MIP [15,98–100,122–128,137,138,152,156,167,240,250,266,273,278,284,289,293,303,309,331,351,390,393,408,409,423,431,437,473,495,507].

The classical approach to mechanical properties relies on compressive strength evolution over time. This data is increasingly complemented by:

- numerical simulation based on finite element approaches and other methods [18,21,22,101,242,260,281,444,452,469,496,515,530]
- wave emission and propagation [136,155,168–170,233,237,259,273,283,331,514,528,533]
- nanoidentation methods [102,104–106,235,371,416,477,483].

Transport properties are especially relevant for the long-term performance of concrete exposed to water and aggressive species including carbon dioxide. New studies have addressed gas, ion, and moisture transport in concrete for a broad range of conditions [103,114,115,126–128,139–143,153,154,162,164–166,216,267,310,314,339,455,490,510,511,519]. Diffusion coefficients were obtained by direct measurement using standard methods and NMR techniques, as well as by calculations based on a combination of Nernst–Planck equation with a finite element approach. It has been suggested to decelerate the transport of harmful ionic species by the addition of viscosity modifiers.

The evolution from plastic to elastic state is due to chemical reactions taking place on the surface of cement particles. The C–S–H growing from the cement grains are responsible for setting of the cement and strength development. Electron microscopy has been proven very helpful for the interpretation of spectroscopic data and especially powerful is the combination of both methods giving access to space-resolved spectroscopy. The microstructure of cementitious systems has been studied by electron microscopy and numerous other methods [145–151,157,234,276,288,313,329,384,411,419,458,475,491,501,522]. High resolution scanning electron microscopy is now able to observe features as small as a few nanometers. The formation of pores with a width between 4 and 20 nm has been observed during the induction period of Portland cement accompanying the formation of the first C–S–H nuclei. There is an ongoing discussion about hollow shell formation (Hadley grains) around cement particles during hydration.

Despite hardened cement paste is relatively dense, the interface to aggregate, reinforcement and other inclusions is known to have a higher porosity [106,119,149,158,160,161,281,348,484].

6. Blended systems

Traditionally, concrete has been produced by mixing Portland cement, aggregate, and water. This recipe has been modified by the addition of organic and inorganic materials. The use of blended cements and admixtures has allowed to reduce the carbon dioxide emission per ton of produced cement and made the way for innovations such as high performance concrete and self compacting concrete. On the other hand, the higher number of ingredients in the mix has rendered the concrete system more complicated and there is currently a high interest in understanding the interactions between clinker phases, mineral additions, and admixtures.

Admixtures are mostly organic compounds that modify early age and final properties of concrete. A broad range of materials has been investigated including

- grinding aids [255,274,349,524]
- superplasticizers [34,53,54,56,57,121,134,135,147,171–175,219,220,238,248,263,264,330,344,353,361,365,379,424,434–436,466,502,527]
- shrinkage reducing admixtures [64,176–178,248,471,476,529]
- air entraining agents [99,179–181,208,454,531]
- viscosity modifying agents [182,218,244,250,294,435]
- retarder [489].

Additional information is available from rheological studies in Section 5.

Inorganic admixtures are used to improve the performance of concrete. The addition of $C_{12}A_7$, Al_2O_3/SO_3-based accelerator, $LiNO_3$, $Ba(OH)_2$, MgO, $CaCl_2$, C_3A, and TiO_2 has been investigated experimentally [183–186,211,214,257,261,274,350,356,362,417,458].

A wide range of mineral additions is employed. Depending on their reactivity, these have been classified as hydraulic, pozzolanic or filler materials. Some new cementitious systems such as geopolymers are included in Section 3. Other studies have focused on blends of Portland cement clinker with

- blast furnace slag [41,75,80,155,198,215,247,274,276,277,287,309,314,360,365,391,406,456,465,484,492,497,498,500,505,532,534]
- metakaolin [54,65,75,85,199,200,204,215,243,282,324,354,402,430,441,485,501,526]
- coal fly ash [41,65,80,179,180,188–191,210,215,241,247,254–256,273,277,287,288,291,301,309,314,352,357,391,407,409,411,432,439,445,458,484,485,494,500,510,531]
- other pozzolanic materials [38,40,69,79,80,163,192–198,204,215,222,256,269,274,288,308,309,312,313,315,317,318,323,325,336,355,357,358,361,363,367,376,380,394,428,432,435,446,450,463,482,485,501,511,512,520]
- calcium carbonate [13,135,163,201,202,249,268,273,274,299,337,427,431,443,445,478,484,500,517,521]
- other materials [163,203,205,206,213,254,256,307,332,438,440,447,451,457,486,487].

7. Conclusions

A high number of studies connected to cement hydration and microstructure analysis has been published within the four years of the interim period between the past and the present ICCC. Most of these were purely empirical investigations often related to specific materials and properties. The number of studies advancing the state-of-the-art of our fundamental understanding of cementitious systems has been much lower. However, we have witnessed new efforts to implement new methods, instruments, and models that have been developed in other scientific disciplines. These activities have been driven by the need to reduce the carbon dioxide emissions related to the production of cement. This stimulus is expected to be working in the future and will support efforts to improve the scientific basis for understanding chemical reactions and microstructure development during cement hydration.

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