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Discussion

Reply to the discussion by P.E. Grattan-Bellew of the paper "Delayed ettringite formation in heat-cured Portland cement mortars"☆

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We wish to thank Dr Grattan-Bellew for his interesting comments on our paper [1]. Dr. Grattan-Bellew has expressed concern that the delayed ettringite formation (DEF) expansion observed after laboratory curing at 100°C [1] may not indicate the performance of steam-cured concrete in the field and therefore may not be suitable for the evaluation of the DEF potential of the cement, because the curing temperature is too high. His suspicion is based on the following: (1) he considers that the cement that we used would fall into the ASTM Type I category, while he believes that only Type III cement is susceptible to DEF; (2) our expansion data cannot be fitted to the curve of the rate of expansion vs. the C_3A content of cement that was obtained on the basis of his experimental conditions as presented elsewhere [2].

We wish to emphasise that the principal objective of our paper [1] was to investigate the mechanism of DEF expansion by observing the microstructural evolution of the heat-cured mortars during the period of DEF expansion. The high curing temperature (100°C) was selected to ensure that DEF expansion occurred relatively quickly in the mortars. The effect of variation in the temperature of curing on the occurrence of DEF expansion has been thoroughly investigated by Lawrence [3], who has shown that there is little evidence that the mechanism that is followed after curing at 100°C is different from that operative at 85°C. Some other experimentalists have also used a curing temperature of 100°C for DEF investigations, for example Heinz and Ludwig [4].

The cement that we used was a UK rapid hardening Portland cement (RHPC), probably approximating to an old-fashioned RHPC. Its specific surface area was 488 m^2/kg, which unfortunately was not quoted in our paper [1]. We do not understand how Dr Grattan-Bellew reached the conclusion that our cement falls into ASTM Category Type I. It is generally considered that the important factor separating Type I from Type III cement is the specific surface area (greater than 450 m^2/kg for Type III), and the chemical analyses are similar or identical in the two classes. The SO_3 contents of Type I and Type III cements can differ slightly (according to ASTM C150-94) since Type I has maximum percent of SO_3 of 3.0% while Type III can have between 3.5 and 4.5%, depending on the C_3A content; in this case 4.5% is appropriate since our percent of C_3A was 8.3, and our percent of SO_3 was in fact 3.2. Mortars made from this cement not only exhibited DEF expansions after heat curing at 100°C, but also exhibited a slower expansion (starting after about 90 days and continuing beyond 400 days) after curing at 90°C, as reported by Shao et al. [5].

It is not at all surprising that our expansion data cannot be fitted to the curve of expansion rate vs. C_3A content of cement obtained by Grattan-Bellew et al. [2] in view of the very different experimental conditions used in the two studies. It is well known that DEF expansion is very sensitive to the experimental conditions. The induction period, the ultimate magnitude of expansion, and so forth can be largely influenced by factors such as curing temperature, heating regime, size of specimen, water:cement ratio, type of aggregate, grade of aggregate, etc. [6,7].

Grattan-Bellew et al. [2] used a most unusual heating program to bring about DEF expansion in which the specimens were first heated at 95°C for 12 h, followed by three thermal cycles. Each cycle included heating at 85°C for 24 h and subsequent storage at ambient temperature for 24 h. The heating rate used was very fast, reaching the ultimate temperature in 1 h. In contrast, we used a common heating regime in which the specimens were heated at a rate of 20°C/h to 100°C and held for 12 h. The size of our samples was larger than those of Grattan-Bellew. Cement mortars treated under such different experimental conditions inevitably exhibited different behavior in terms of DEF expansion.

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The potential for DEF is certainly raised by increasing the curing temperature, as indicated in the literature [1,6] and by our recent microstructural and mineralogical investigations [8,9]. The stability of the sulfoaluminate products at elevated temperatures is dependent on the temperature of the heat cure, and can be roughly divided into three regions. At temperatures below 70°C, ettringite is stable and therefore the potential for DEF is low. At temperatures between 70 and 90°C, ettringite is partially or fully destroyed, but monosulfate is stable and the potential for DEF may be raised above the critical level for expansion to be observed. The potential for DEF is further increased by raising the heat curing temperature to the range between 90 and 100°C, because in this range not only is ettringite destroyed but monosulfate is also partially or fully destroyed. The formation of hydrogarnet and drastic retardation in the hydration of the interstitial clinker phases (especially the ferrite phase) in heat-cured cement products during subsequent hydration at ambient temperatures are also factors that enhance the DEF potential, which are discussed more fully elsewhere [8,9]. The curing temperatures chosen in our work (100°C) and in Grattan-Bellew's (95°C) both fall into the same range of high curing temperature (90–100°C). Hence we believe that they do not result in any significant difference in terms of the potential for DEF.

In our opinion, curing at 100°C may provide the basis for an accelerated method to evaluate the DEF potential of a cement used in steam-cured concrete in the field, even though field concrete may never be heated to such a high temperature. The lower the curing temperature, the longer the induction period for DEF expansion. The results reported by Lawrence [3] showed that the mortar cured at 100°C for 3 h started to expand after storage for about 90 days, while the same mortar cured at 70°C expanded only after storage for 400 days. We realize that some cements, which after curing at 100°C show expansion, may not expand when they are cured at a lower elevated temperature. Misleading results might be obtained from all the currently available methods for the assessment of DEF potential. A new accelerated test is required that is soundly based on a thorough understanding of the mechanism of delayed ettringite formation.

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