

# An Experimental Clarification of the Association of Delayed Ettringite Formation with Alkali–Aggregate Reaction

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## Abstract

*The issue of determining the primary cause of damage to concrete elements in the presence of a combination of delayed ettringite formation (DEF) and alkali–aggregate reaction (AAR) is very controversial. Some authors believe that DEF has been the cause of serious damage (cracking) to concrete elements cured at elevated temperatures, and some others attribute the damage to AAR, with a minor role for DEF. To assist in clarifying the issue, experimental mortar specimens were devised in which either of these chemical processes could occur separately or in combination. The specimens were subjected to either continuous curing at 40°C or initial steam curing at 75°C for 8 h followed by storage at 40°C, 100% RH. The results of length change measurements on these specimens favour the conclusion that AAR is the primary cause of damage, but under certain conditions DEF may contribute to it. However, in the absence of AAR cracking no DEF was observed in the systems examined here, emphasising the primary role of the AAR in causing damage in the form of cracking in concrete. © 1996 Published by Elsevier Science Limited.*

## INTRODUCTION

Premature deterioration of numerous prestressed precast concrete railway sleepers in the past decade has triggered extensive studies on the effects of heat curing on concrete behaviour, and investigation of mechanisms of

deterioration. Earlier reports, largely from Germany, attributed the deterioration to delayed ettringite formation (DEF) in heat-cured concrete.<sup>1,2</sup> This term means that the primary ettringite that normally forms early in the hydration of Portland cement is destabilised at high temperature (above 70°C) steam curing, causing the sulfate ions from ettringite decomposition to remain in the liquid phase of the hardened concrete, and precipitate as ettringite at a later age. Some sulfate ions are retained in the solid phase in the form of monosulfate and some are incorporated in the C–S–H phase. This delayed formation of ettringite was alleged to be an expansive process and the cause of the deterioration. Tepponen and Eriksson<sup>3</sup> reported that deterioration of concrete sleepers from Finland was due to DEF, and that removal of the heat treatment from the manufacture of new sleepers stopped the subsequent cracking. However, Shayan and Quick<sup>4</sup> reported that a sample of a deteriorated concrete sleeper from Finland exhibited considerable alkali–aggregate reaction (AAR) and argued that AAR may have been a major contributor to the damage to the Finnish sleepers. It appears that the improvement noted for the newer Finnish sleepers was not merely due to removal of heat treatment, but probably largely because they had a much reduced alkali content.

Shayan and Quick<sup>5</sup> investigated the cracking of some Australian, prestressed, precast concrete sleepers and concluded that the cracking was primarily due to AAR, with DEF having a minor role, if any. Ettringite filling cracks at the

cement mortar/aggregate boundaries or in the mortar matrix was only observed in a sleeper which was badly cracked and contained considerable signs of AAR. Shayan<sup>6</sup> demonstrated that the aggregate used in the manufacture of the Australian sleepers was reactive towards alkali and capable of causing considerable cracking in laboratory concrete specimens without steam curing, i.e. without DEF. However, the observation of recrystallised ettringite in damaged concrete has prompted some authors to consider it the culprit. The conclusion<sup>7</sup> that DEF was entirely responsible for damage to a bridge in France needs careful re-examination.

The association of AAR and ettringite formation in damaged concrete (both precast and *in situ* cast) has been observed by several authors,<sup>8-13</sup> but the contribution of each mechanism to the deterioration of steam-cured concrete has remained unclear. The conclusion that carbonation of monosulfate and its conversion to ettringite (and ultimately to carboaluminates and gypsum) was responsible for damage to heat-treated concrete<sup>14,15</sup> is not convincing and needs further examination. No such carboaluminate conversion products were detected in the badly damaged Australian or Finnish sleepers.

Although formation of ettringite is used as the driving force for certain cementitious systems deliberately designed to cause expansion (e.g. expansive cements), it is generally agreed that the mere observation of recrystallised ettringite in voids and cracks in concrete does not mean that ettringite in this form was the primary reason for expansion and deterioration,<sup>16-20</sup> Formation of ettringite in voids and preexisting spaces (cracks) cannot cause expansion, although its growth in confined spaces in the cement matrix can produce expansive forces. Expansion of specimens deliberately enriched in sulfate and/or aluminate<sup>21</sup> or cured under unrealistic conditions<sup>23</sup> may not relate to the behaviour of conventional steam-cured concrete. Siedel *et al.*<sup>24</sup> reported a lack of expansion in steam-cured concrete specimens, although recrystallised ettringite was observed for curing temperatures of 60°C and 90°C. This is in agreement with the observations of Shayan and Quick<sup>16</sup> that unless reactive aggregate was present to cause expansion and cracking, noticeable amounts of ettringite were not seen and that recrystallisation of ettringite *per se* did not cause any significant expansion. Diamond<sup>25</sup> con-

cluded that continued expansion of steam-cured mortars containing reactive aggregate was due to precipitation of ettringite in cracks induced by AAR.

This paper reports the results of experimental work on specimens in which it was attempted to enhance the formation of AAR products and ettringite either alone or in combination. The purpose was to assess the contribution of each phase to the measured expansion or deterioration of the specimens.

## EXPERIMENTAL WORK

Mortar bars having a cement:sand:water ratio of 1:2:0.4 were chosen as the model specimens. Four parameters were selected for creating specimens with different behaviours, viz. aggregate type ( $\times 2$ ), sulfate content ( $\times 2$ ) cement alkali level of mortar (4), and temperature of curing ( $\times 2$ ).

### Aggregate

The aggregate types were either a non-reactive sand or the same sand in which 7.5% by mass of particles was replaced by opal.

### Sulfate levels

The Portland cement used had the composition: SiO<sub>2</sub> = 22.0, Al<sub>2</sub>O<sub>3</sub> = 4.4, Fe<sub>2</sub>O<sub>3</sub> = 3.64, CaO = 62.9, MgO = 1.48, K<sub>2</sub>O = 0.46, Na<sub>2</sub>O = 0.60, TiO<sub>2</sub> = 0.16, MnO = 0.06, P<sub>2</sub>O<sub>5</sub> = 0.08, SO<sub>3</sub> = 2.74, and ignition loss = 1.30%, i.e. the sulfate level of the cement was 2.74% SO<sub>3</sub> (corresponding to 5.9% gypsum). In order to determine a realistic content of additional gypsum, separate portions of the cement were blended with this material so that the blends contained 2.5, 5.0, 7.5 and 10% added gypsum. The blended portions were then made into slurries using a water:cement ratio of 0.4. These were placed in a fog room with protection from dripping water and dried briefly in air after 18 h of hydration. The material was then pulverised gently and subjected to X-ray diffraction (XRD) analysis. The maximum addition level for which no residual gypsum was detected in the hydrated cement was chosen, this being the 2.5% corresponding to 1.16% SO<sub>3</sub>. Therefore the enhanced sulfate level was 3.9%.

### Alkali levels

In the original cement the level of alkali was as given in the cement composition (i.e. 0.90% Na<sub>2</sub>O equivalent). The enhanced level of alkali was obtained by using 1 M NaOH as the mixing solution compared with water for the original level. This was to enhance the occurrence of AAR in the presence of reactive aggregate.

### Curing temperature

One set of specimens was stored at 40°C, 100% RH, after being demoulded and the initial length reading taken. Another set was initially steam cured at 75°C for 8 h (rise time of 4 h from ambient temperature ≈20°C, and 6 h of cooling) and the initial length reading taken, then stored at 40°C, 100% RH. A third set, which was made for confirmation of an observation, was demoulded 5 h after casting and placing in a fog room, then initial length taken, steam cured as above and stored at 23°C in fog.

### Specimens

Two mortar bars (25 × 25 × 285 mm) and four cubes (25 mm) were cast from each combination of the main variables for measurement of length changes and determination of compressive strength, respectively, at various ages. In addition, one set of mortar specimens was made to contain a mixture of either NaOH + Na<sub>2</sub>SO<sub>4</sub> or KOH + K<sub>2</sub>SO<sub>4</sub> in such a way as to produce amounts of SO<sub>3</sub> equivalent to those containing the additional 2.5% gypsum as well as being 1 M in NaOH or KOH in mixing solution. This was to compare the effects of added Na and K on the expansion behaviour.

Other sets of mortar bars, without any added gypsum or opal, were made (for measurement of length changes after steam curing at 75°C for 8 h) in which the mixing solution was either water, 0.5 M NaOH or 1 M NaOH, with or without 10% silica fume replacing cement on a mass basis. Companion cylindrical specimens 50 mm diameter by 100 mm long were made for expression and analysis of the pore solution, after steam curing at 75°C for 8 h and storage under sealed conditions for 28 days.

### Examinations

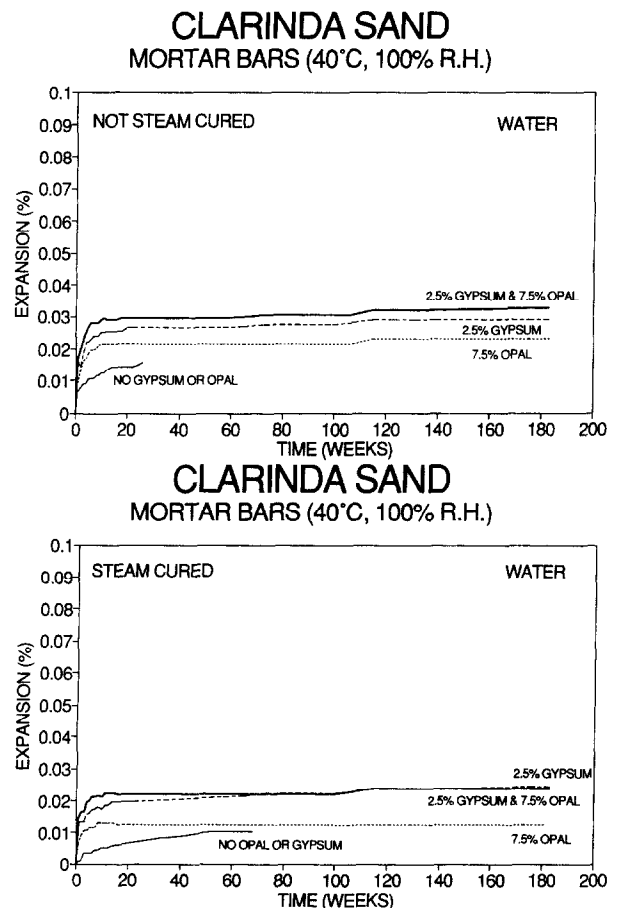
Representative specimens were removed from the cubes made from those mixes related to the

main variables for scanning electron microscopy (SEM) together with energy-dispersive X-ray (EDX) analysis. At the completion of expansion measurements, specimens from mortar bars were also removed for this purpose as well as for XRD analysis. Conventional methods were used for the determination of ionic compositions of the expressed pore solutions.

## RESULTS AND DISCUSSION

### Expansion behaviour

Figure 1 shows expansion curves for mortar bars made with water as the mixing liquid. Figures 1(a) and (b) for the non-steam-cured and the steam-cured specimens, respectively (both subsequently maintained at 40°C, 100% RH), show that very little expansion occurred in these specimens. The alkali level was probably inadequate to produce sufficient amounts of



**Fig. 1.** Expansion curves for mortar bars made with mixes with and without 2.5% of added gypsum or 7.5% of opal, having water as the mixing liquid: (a) stored at 40°C, 100% RH and (b) steam cured at 75°C for 8 h, then stored at 40°C, 100% RH.

expansive AAR gel. However, the expansion of bars made with the control mix was only 0.01% and distinctly less than those for the other mixes. Note that early expansion of steam cured specimens was less than that of non-steam-cured specimens (see later).

For mixes in which 1 M NaOH was used as the mixing solution (Fig. 2), much larger mortar bar expansions were noted in the presence of reactive aggregate. Expansion curves for the non-steam-cured specimens (Fig. 2(a)) show that the addition of 2.5% gypsum had no effect on the expansion under the storage conditions of 40°C, 100% RH. The reactive aggregate was the sole reason for the observed expansion. For the steam-cured specimens (Fig. 2(b)) the additional gypsum had no effect on the expansion of mortar bars with nonreactive sand. This is despite the fact that the potential for DEF is much greater than in the control mix. However, in the presence of reactive aggregate, the addition of 2.5% gypsum had a dramatic effect on the

expansion behaviour of the mortar bars subjected to steam curing. Comparison of Figs 2(a) and (b) shows a number of very interesting points.

In the absence of reactive aggregate, and lack of AAR effects, the potential for DEF is not realised despite the composition being favourable for enhanced amounts of ettringite to form, or if DEF occurs it does not cause any expansion.

The maximum expansion of mortar bars containing opal was much greater under the storage conditions of 40°C 100% RH than with steam curing. Considering that the initial length reading was taken after the steam-curing process, it was thought the above difference arose because some expansion took place as a result of steam curing, and was not measured because of the time the initial reading was taken. However, this latent expansion was measured under the storage conditions of 40°C, 100% RH due to the slower rate of reaction in the first 24 h at room temperature (23°C) before demoulding. Figure 3, showing expansion curves for mortar bars that were demoulded 5 h after casting and initial length measurements taken before steam curing, confirms the above reasoning. It shows that about 0.15% expansion took place as a result of steam curing (intercept on the y-axis) and was missed in subsequent measurements for Fig. 2(b). However, because the specimens were stored at 23°C, the total expansion is still less than that shown in Fig. 2(a) for storage conditions of 40°C, 100% RH.

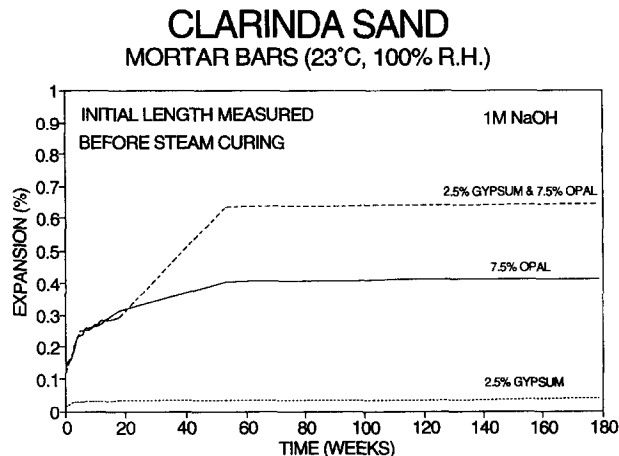
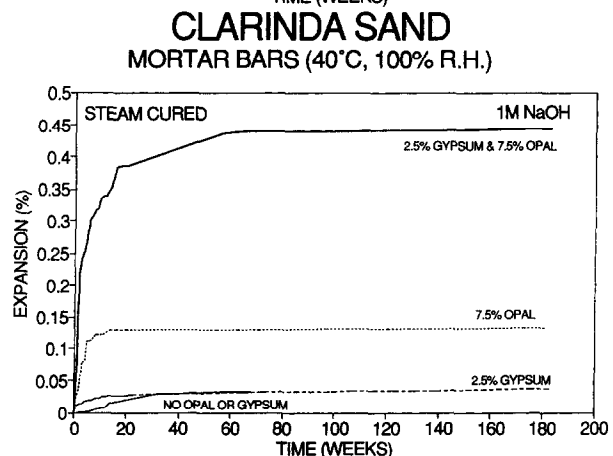
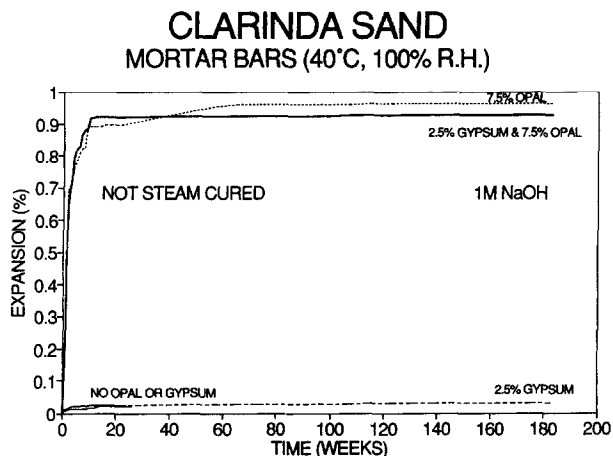


Fig. 2. Expansion curves for mortar bars made with mixes with and without 2.5% of added gypsum or 7.5% of opal, having 1 M NaOH as the mixing liquid. (a) stored at 40°C, 100% RH and (b) steam cured at 75°C for 8 h, then stored at 40°C, 100% RH.

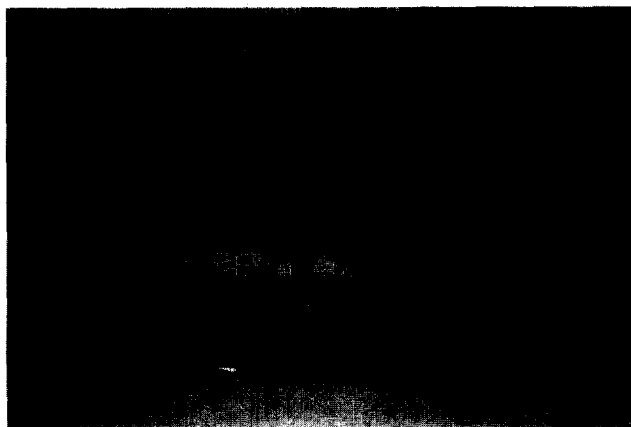
Fig. 3. Expansion curves for mortar bars that were demoulded at 5 h for which the initial length measurement were taken before steam curing. The steam-curing process has caused an expansion of 0.15%, and DEF is probably occurring, during the subsequent storage period.

Whereas 2.5% additional gypsum had no effects on the expansion of specimens at 40°C, 100% RH (Fig. 2(a)), its presence had a dramatic effect on expansion of steam-cured specimens containing reactive aggregate. Both Fig. 2(b) and Fig. 3 show that the presence of gypsum considerably increased the expansion.

### Possible causes of additional expansion

#### *Deposition of secondary ettringite in cracks formed by AAR*

On this hypothesis, the expansion beyond that shown by the specimens without additional gypsum is attributed to the growth of ettringite crystals in cracks developed as a result of AAR. Figure 4 shows the severe warping of both steam-cured (326) and 40°C-cured (342) specimens containing opal and additional gypsum, and both have substantial microcracking. Diamond<sup>25</sup> suggests that growth of ettringite in preexisting cracks could cause expansion, whereas Johansen *et al.*<sup>18</sup>, Glasser *et al.*<sup>19</sup>, and Scrivener and Taylor<sup>17</sup> suggest that it does not. Fu *et al.*<sup>6</sup> suggest that secondary ettringite nucleation occurs at the surfaces of preexisting microcracks and state that steam-cured Portland cement paste subjected to severe drying undergoes large expansions. Shayan<sup>27,28</sup> has clearly demonstrated that expansion and warping of large precast concrete panels made with white cement paste and coarse aggregate only (no sand) was due to the high shrinkage potential of such a concrete. The induced shrinkage cracks were filled with calcium hydroxide under



**Fig. 4.** Photograph of two sets of mortar bars showing the warping of the bars as a result of AAR expansion. Both contained opal and additional gypsum and were mixed with NaOH. One (326) was steam cured and the other (342) was not, and both were subsequently stored at 40°C, 100% RH, for the entire period of measurement.

moist conditions, and new shrinkage cracks developed in the next drying phase which were filled again in a subsequent moist cycle. This caused continued expansion and significant warping of the panels, which had to be taken out of service. In this case calcium hydroxide rather than ettringite was filling the cracks. However, in steam-cured specimens of higher sulfate content ettringite may also form in such cracks, but this may or may not cause expansion depending on the environment as well as the composition of the pore solution. If cracks are fine and filled with a solution supersaturated with respect to ettringite, then crystallisation of ettringite could cause expansive forces.<sup>20,29</sup>

It is proposed that if ettringite merely fills the cracks it would not cause expansion. However, under drying conditions, the filling material itself may develop fine microcracks, which would be filled with the liquid phase in moist conditions. If this liquid is supersaturated with respect to the solid, then crystallisation pressure could develop on precipitation of further ettringite. Although this is possible under field conditions, it is not likely under constantly humid test conditions. Therefore, there may be another reason for the observed expansions in the presence of reactive aggregate and additional gypsum (see below).

#### *Simultaneous AAR and precipitation of ettringite*

On this hypothesis, the occurrence of AAR is considered to be accompanied by the formation of ettringite within the paste as well as in cracks. Glasser *et al.*<sup>19</sup> have reasoned that precipitation of ettringite from the pore fluid of steam-cured concrete, on cooling, increases the alkalinity of the fluid, which becomes still more concentrated with increasing withdrawal of more liquid for formation of more ettringite. They suggested that this increased alkalinity could enhance AAR if a reactive aggregate is present.

However, it appears more likely that this process takes place in the reverse direction, particularly in the system used in these experiments where the mixing solution was already 1 M in NaOH concentration. It is known that the addition of alkali dissolved in the mixing solution<sup>30,31</sup> increases the sulfate concentration. Steam curing also increases the sulfate concentration of the pore solution due to decomposition of ettringite at high temperature. Therefore, the mortar bars used here would

have three important features soon after being made, viz. reactive aggregate, and high alkali and high sulfate concentrations in the pore solution. The rapid expansion of 0.15% during the steam-curing process is obviously due to AAR, because sulfate ions remain in the pore solution in this period and ettringite does not precipitate. AAR continues during subsequent storage period. Shayan *et al.*<sup>32</sup> have shown that reactive silica (silica fume agglomerates) in steam-cured concrete rapidly removes the alkalis from the pore solution to form alkali-silica gel, and their XRD data indicate that this enhances the formation of ettringite. The reason is that by reducing the alkali hydroxide concentration in the pore solution, it enhances the sulfate and calcium ion concentrations, which in turn induce precipitation of ettringite at the same time. Therefore, the sulfate ion concentration is also reduced in the presence of silica fume. Figure 5 shows changes in the ionic concentrations of the pore solutions of mortar

specimens, steam cured at 75°C for 8 h, as a function of NaOH concentration of the mixing solution. The effects on the ionic concentrations of adding silica fume (SF) are also shown.

The fine opal aggregate used here has effects on the concentrations similar to those of silica fume.<sup>31</sup> Therefore, precipitation of ettringite could occur simultaneously or soon after AAR has taken place. This had happened in a steam-cured concrete, as indicated by XRD analysis, at the age of one day,<sup>32</sup> but was much more pronounced at 28 days and particularly one year. The coarser texture of steam-cured specimens<sup>33,34</sup> probably means that ettringite precipitation may occur in the paste. The exact mechanism of expansion, i.e. whether the precipitation occurs directly from the pore solution present in the paste or at surfaces of the aluminate phase in contact with the sulfate-rich pore solution, or both, is not clear from these data. Both are capable of causing expansion in the confined spaces in the paste. In any case, the larger expansion of steam-cured mortar bars containing opal and 2.5% additional gypsum, compared to those with only opal, is attributed to crystallisation pressure of secondary ettringite, either in the microcracks or in the paste.

It must be noted that experiments such as those reported by Fu *et al.*<sup>35</sup> are irrelevant to secondary or delayed ettringite formation in steam-cured concrete, and are more related to external sulfate attack. This is because they removed the initial highly alkaline liquid phase of cement hydration from which secondary ettringite is supposed to form. It must also be noted that the behaviour of commercial reactive aggregates and their influence on DEF may be different from that observed for the highly reactive opal.

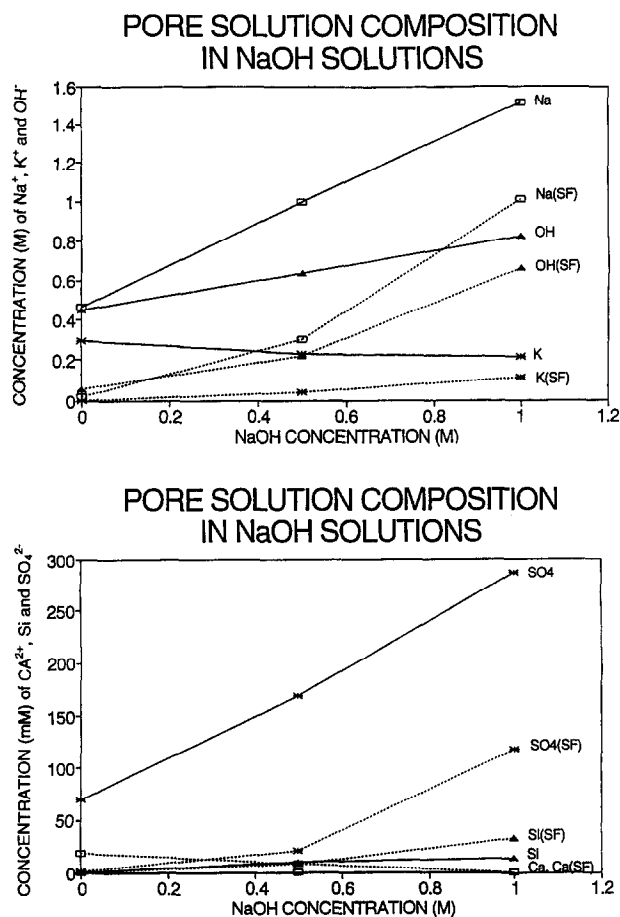


Fig. 5. Compositions of pore solution of steam-cured mortar cylinders made with solutions of varying NaOH concentrations as the mixing solution. SF indicates companion mixes in which silica fume (10%) replaced cement on a mass basis.

### Effects of Na and K on expansion

Expansion curves for mortar bars in which the additional sulfate and alkalinity were provided by either  $\text{Na}_2\text{SO}_4 + \text{NaOH}$  or  $\text{K}_2\text{SO}_4 + \text{KOH}$  in the mixing solution are given in Fig. 6. In the case of the Na system, significant expansion was obtained only when reactive aggregate was present, although some expansion was noted in its absence. This may well be due to susceptibility of the sand used in this system because the added  $\text{Na}_2\text{SO}_4$  will produce more alkalinity according to  $\text{Na}_2\text{SO}_4 + \text{Ca}(\text{OH})_2 \rightarrow \text{CaSO}_4 +$

2NaOH, the  $\text{CaSO}_4$  being removed in the formation of ettringite. The higher expansions of mortar bars containing opal in this system compared to those in which gypsum was added is also probably due to greater reactivity of opal under the more alkaline conditions. Formation of secondary ettringite may also have contributed to the expansion as with added gypsum. The compressive strength of the mix at one day was 50.3 MPa without opal and 30.4 MPa in the presence of opal.

The same argument applies to the K system except that a much larger expansion was noted

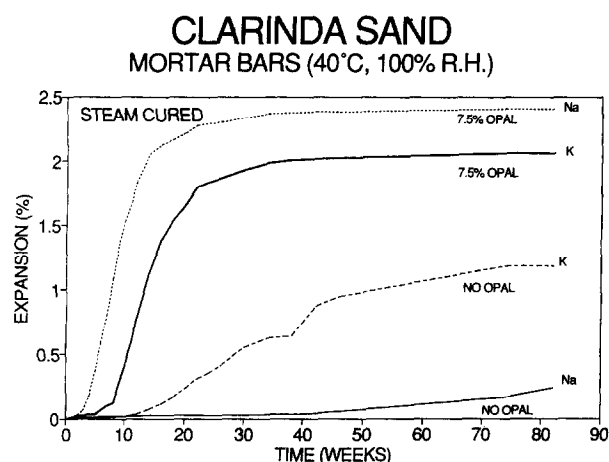
in the absence of opal. Although the solid phases were not analysed by XRD, it is likely that syngenite formed as a secondary phase and contributed to the expansion. The compressive strength of the mix at one day was 36.8 MPa without opal and 31.3 MPa in the presence of opal. These results may have some implications for the type of salts present in chemical admixtures such as superplasticisers.

### Compressive strengths and X-ray patterns of mortars

The compressive strengths of the various mixes are given in Table 1. The most obvious point to note is the reduction in strength of the mixes containing NaOH compared to water, which was also observed previously<sup>36</sup> and was attributed to incorporation of Na in the C-S-H and development of a more dispersed system. Mixes containing additional gypsum developed higher strengths, probably due to additional ettringite formation. XRD traces of the fine fractions separated from some mixes cured at 40°C, 100% RH, or steam cured, confirmed that mixes containing gypsum had larger amounts of ettringite, reflected by stronger peaks at about 9.7 Å and 5.6 Å.

### Microstructural features of mortar bars

Scanning electron microscope (SEM) examination of the 90-day-old mortar cubes used for



**Fig. 6.** Expansion curves for steam-cured mortar bars made with mixing solutions in which additional sulfate and alkali were added either as  $\text{Na}_2\text{SO}_4 + \text{NaOH}$  or by  $\text{K}_2\text{SO}_4 + \text{KOH}$  to be equivalent to the system 2.5% additional gypsum + NaOH, with respect to  $\text{SO}_3$  content and hydroxyl ion concentration.

**Table 1.** Compressive strengths (MPa) of cubes made from various mixes\*

Mix	Mixing solution	Steam curing†	Age (days)			
			1	7	28	90
Control mix cement + sand + water 1:2:0.4	Water	Yes	51.8	56.8	65.6	75.3
		No	21.8	36.6	51.4	61.5
7.5% opal included in the mix	1 M NaOH	Yes	15.0	19.7	35.6	46.2
		No	12.8	22.7	35.6	42.9
		Yes	45.1	53.2	71.2	78.5
		No	21.0	55.2	69.4	78.6
2.5% additional gypsum in the mix	1 M NaOH	Yes	18.2	27.0	43.3	50.6
		No	13.4	26.2	35.7‡	44.8‡
		Yes	2.4	56.2	78.7	90.6
		No	10.3	48.4	71.6	80.3
7.5% opal and 2.5% additional gypsum in the mix	1 M NaOH	Yes	24.3	29.9	46.5	52.0
		No	11.7	36.5	52.6	58.5
		Yes	45.0	55.9	73.0	88.3
		No	13.1	40.7	61.4	76.3
	1 M NaOH	Yes	24.1	38.6	47.6‡	54.6‡
		No	11.3	28.3	38.3	46.5‡
		No	11.3	28.3	38.3	46.5‡

\* Each value is the average of four determinations.

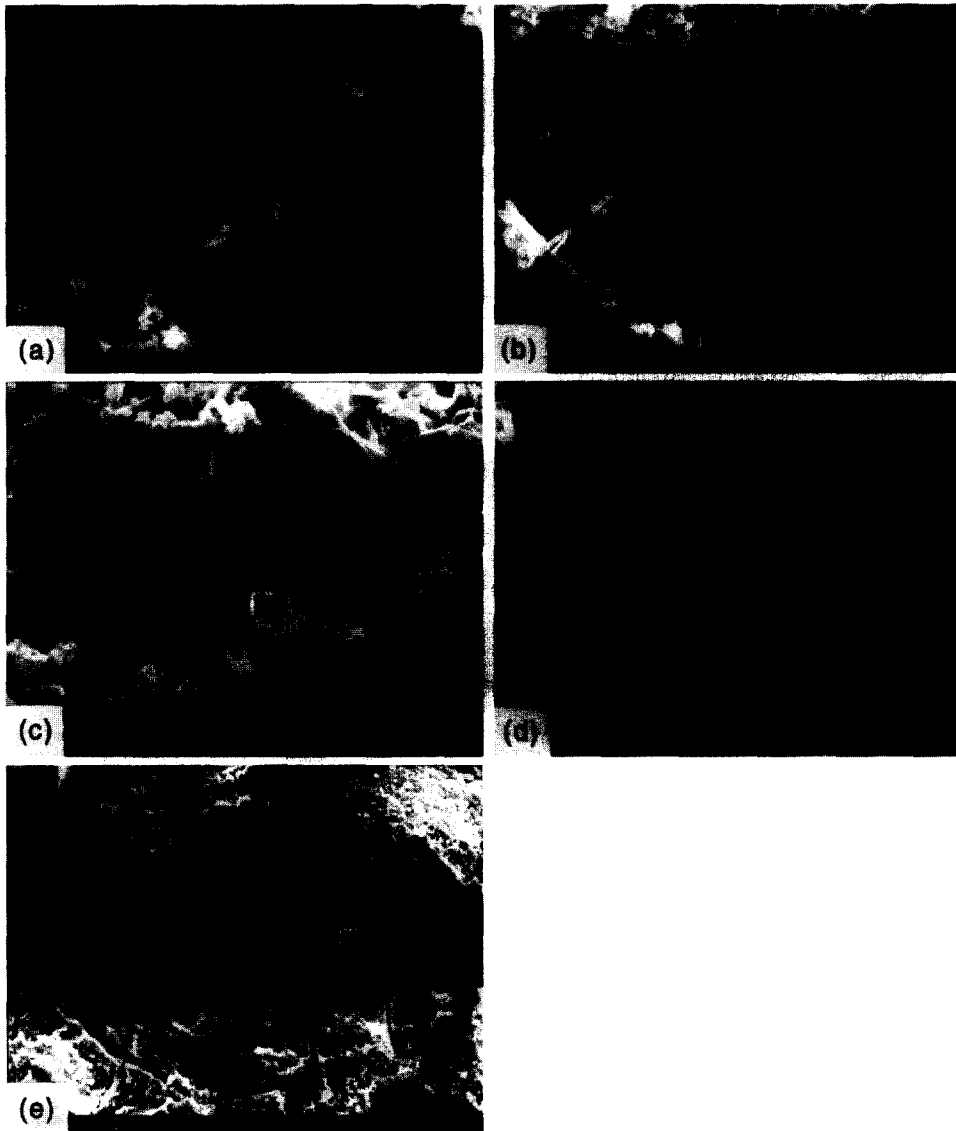
† Steam cured at 75°C for 8 h; after cooling and demoulding stored at 23°C, 100% RH. Those not steam cured were stored at 23°C, 100% RH, after demoulding at 24 h.

‡ Indicates microcracking of specimens.

strength testing, together with EDX analysis, showed that only minor indications of AAR were observed in the opal+water systems, but extensive AAR gel was observed in the opal+NaOH systems. This is in complete agreement with the expansion behaviours discussed earlier. However, the level of alkali in the gel was relatively low, indicating leaching during the storage period. Massive (recrystallised) ettringite was most abundant in what appeared to be cracks in the mixtures containing added gypsum+opal+NaOH (steam cured), whereas little ettringite was found in the mixtures with added gypsum+NaOH but without opal (steam cured). Short needles of ettringite were common in voids in both the

mixtures containing added gypsum+water (40°C) and added gypsum+opal+water (40°C). These, however, are considered as primary ettringite that forms in all Portland cement concretes and are considered harmless.

Examination of mortar bars after the completion of expansion measurements ( $\approx 4$  years) by SEM and EDX showed that although some morphological features of the AAR gels were preserved, the chemical nature of the gels had changed such that they had completely lost the alkali and become enriched in Ca. Neither could any alkali be detected in the paste as a whole, whereas this was possible in the 90-day-old mortar cubes. This is attributed to strong leaching of the alkali out of the mortar bars,



**Fig. 7.** Ettringite crystals (arrowed) in mortars containing: (a) water and added gypsum, (b) NaOH and added gypsum, (c) and (d) NaOH, opal and added gypsum. The mortars in (a)–(c), but not (d), were steam cured. (e) Morphology of AAR gel formed in a mix containing opal and NaOH, preserved even after the composition had been depleted in alkali and much enriched in Ca.



which is probably why expansion ceased a long time before the completion of the measurements, although depletion of reactive silica (opal) could be another reason. If leaching is the reason, then the full potential for DEF would not have been realised either. Examination of 8-months-old autoclaved mortar bars<sup>37</sup> has shown that ettringite had formed on the external surfaces of the bars, and indicates that leaching is possible due to the mobility of chemical constituents making up ettringite.

Figure 7 shows some forms of ettringite in some of the steam-cured mortar bars (except Fig. 7(d), which was cured at 40°C) aged nearly four years. In the mixes made with water and added gypsum, the ettringite was largely present as a very open structure of needles in the air voids (Fig. 7(a)). A similar occurrence was noted for the added gypsum+NaOH mixes, but the ettringite assemblage was more compact (Fig. 7(b)). This is attributed to the much higher concentration of sulfate in the presence of NaOH. Mixes that contained added gypsum+opal+NaOH showed a different morphology of ettringite which was also present in microcracks in the form of shorter, more stubby needles (Fig. 7(c)). This seemed to be associated with additional expansion as shown by the expansion curves in Fig. 2(b). A similar morphology was observed in the added gypsum+opal+NaOH mixes which were stored at 40°C, 100% RH, despite the fact that the additional gypsum did not appear to contribute to the expansion. Ettringite was occasionally found at the cement/aggregate boundary, but this was not common. The question as to whether ettringite formation in the paste or its deposition in microcracks contributed to the expansion could not be resolved from this examination. Ettringite, in the paste could well be poorly crystalline (or colloidal) and not easy to detect by SEM or even XRD. Transmission electron microscopy may prove more useful for this purpose. Figure 7(e) shows an example of the morphology of AAR gel being preserved, whereas the composition, as indicated earlier, had lost its alkali and become richer in Ca.

## CONCLUSIONS

This study has shown that:

Steam curing of Portland cement mortar, even with raised gypsum content, does not lead

to DEF in the absence of reactive aggregates that cause severe expansion and microcracking.

In mixes containing reactive aggregate, steam curing causes further expansion at elevated levels of alkali and gypsum compared to mixes without added gypsum, indicating that DEF plays a role in such cases.

In mixes containing reactive aggregate and alkali and cured at 40°C, 100% RH, additional gypsum does not cause additional expansion, indicating that the increased amount of primary ettringite *per se* does not contribute to expansion, but that heat treatment is perhaps necessary for additional expansion to occur due to DEF. It should be noted that the added NaOH itself enhances the solubility of gypsum, but this by itself does not lead to DEF.

Potassium appears to have an expansive influence on steam-cured mixes even in the absence of reactive aggregate, and this may result from the formation of syngenite in the mortar matrix after the steam-curing period. This may have implications in relation to the compositions of chemical admixtures used in steam-cured concrete.

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