DOES SILICA FUME MERELY POSTPONE EXPANSION DUE TO ALKALI-AGGREGATE REACTIVITY?

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Condensed silica fume (CSF) is considered effective in suppressing concrete expansion due to ASR, provided it is used in sufficient amounts. Various mechanisms can be proposed to explain this: higher strength, lower permeability, alkali dilution, portlandite consumption in the cement paste and alkali depletion in the pore solution due to pozzolanic reaction. The most critical mechanism appears to be alkali depletion in the pore solution and consequent pH decrease. However, the long term effectiveness of CSF against ASR is presently questioned by a number of workers. The proposed explanation is the recycling of alkalies which were entrapped early in low Ca/Si and high-alkali pozzolanic CSH. This hypothesis is based on expansion tests on concrete containing CSF and reactive aggregates, and on chemistry of pore solution extracted at different time intervals from equivalent cement-admixture pastes.

INTRODUCTION

Several experimental studies performed during the last 10 to 20 years suggest that condensed silica fume (CSF) can be successfully used to prevent excessive expansion due to AAR, while being of great interest for other purposes, for instance for making high-strength and/or low-permeability concretes. As a result, CSF is frequently used in the field with reactive aggregates. In Iceland, where the cements produced from rhyolitic raw materials contain about 1.5% alkalies (Na_2O eq.) and glassy basalt aggregates are currently used, CSF has been used since the early 70's. The State Cement Works now produces only blended cements with 7-8% CSF (Asgeirsson (1)). In Norway, silica fumes have been commonly used, since first experimented in 1950 (Sellevold & Nielsen (2)). CSF is also frequently used in the province of Québec (Canada), where reactive aggregates are very common, and other types of mineral additives (pulverized fly ash, ground granulated blast furnace slag, natural pozzolans,...) are not available. Blended CSF cements are currently produced in this province by the two principal cement suppliers.

Results of experimental and field studies involving CSF and reactive aggregates, (including opal, pyrex and fused silica), are reviewed by Bérubé and Duchesne (3). These results suggest that the effectiveness of CSF in suppressing AAR expansion depends greatly on numerous parameters, in particular their composition (silica and alkali contents), the % used as cement replacement, the reactive aggregates and the type of alkali-reactivity involved (alkali-silica or alkali-carbonate), the cement used (type, fineness, alkali content,...), etc. For instance, condensed silica fume is not effective against alkali-carbonate reactivity. With alkali-silica reactive aggregates, CSF must be used in sufficient amounts which vary from one reactive aggregate to another, and its composition is critical. In presence of very reactive aggregates, varieties with relatively low alkali and high silica contents usually call for 10 to 15% of cement replacement, depending on the total alkali content of the concrete and on other parameters related to mix design (water/cement,...) and environment (deicing salt, sea water,...) (3). Lesser amounts, say about 5%, are usually not enough, and may even result in higher expansion than control specimens without silica fume ("pessimum effect"). On the other hand, percentages in excess of 10 to 15%

may affect the concrete durability with respect to scaling and freezing/thawing. According to a number of experiments, a lower water/cement, at least between 0.45 and 0.60, normally results in higher expansion, due to the lower amount of residual water and the consequent higher alkali concentration in the pore solution; therefore a lower w/c should call for a higher CSF content to counteract AAR problems. However, the effect on concrete containing reactive aggregates and made with w/c lower than 0.45, like those used in high strength CSF concretes, is unknown.

The long term effectiveness of CSF is presently questioned by a number of workers. Long term experiments and the mechanisms proposed to explain the effectiveness of mineral admixtures in general and of CSF in particular in reducing expansion due to ASR will be reviewed. Conclusions from these studies will be compared to results obtained in expansion tests performed in our laboratory on concrete containing CSF and reactive aggregates, and chemical analysis of pore solution extracted from corresponding cement-admixture pastes at different time intervals. The high pressure extraction method used is the one first proposed by Longuet et al. (4). A complete description of these tests and analyses is found in Duchesne and Bérubé (5).

EXPERIMENTS SUGGESTING POOR LONG TERM EFFECTIVENESS OF CSF

Field performance

Unfortunately, only a limited number of field cases involving CSF and reactive aggregates are reported in the literature. In Iceland, expansion problems due to AAR disappeared following the use of about 7.5% of CSF as cement replacement (Asgeirsson (6)). In the Sherbrooke area, in Québec (Canada), sections of a sidewalk were made with a potentially reactive aggregate, a high alkali cement (1.15% Na_2O eq.), and various cement (140-405 kg/m^3) and CSF contents (15 to 40% as cement replacement) (Aitcin & Regourd (7)). This sidewalk is exposed to deicing salts during winter. After 4 years, it presented no external signs of AAR, although some freeze-thaw deterioration (scaling) was observed on the two sections containing 20 and 40% CSF. However, silica gels were observed under the SEM in the section containing the highest cement content (4.7 kg/m^3) and 15% CSF. Since then, the section containing 140 kg/m^3 of cement and 40% CSF has been rebuilt due to severe scaling, and recent inspection of the other remaining sections indicate that the concrete is still in good condition after 10 years of service (Lessard et al. (8)).

Experimental studies

In our laboratory, two CSF's, one high-silica/low-alkali (A: 94% SiO_2, 0.8% Na_2O eq.) and one low-silica/high-alkali (B: 75% SiO_2, 3.6% Na_2O eq.), were tested in presence of two very alkali-silica reactive aggregates from Canada: a siliceous limestone from the Spratt Quarry, near Ottawa (Ontario), and a rhyolitic tuff from Beauceville (Québec). The composition of concrete samples, admixtures and cements used are reported elsewhere (5, Bérubé and Duchesne (9)). The results for a period of 1.5 years are presented in Figure 1. They show that: 1) the high-alkali CSF produces higher expansions; 2) concrete specimens containing 5% CSF and rhyolitic tuff expanded more than control prisms with no CSF ("pessimum effect"); 3) 10% CSF-B is insufficient in the long term; 4) the greater the amount of CSF, the longer the delay before expansion; 5), the rate of expansion of CSF concretes significantly increased in the long run (say after 6 months), while the control leveled off, even with 10% CSF-B. These results suggest that CSF may merely postpone expansion due to AAR. To our knowledge, this is the first mention of a pessimum effect for concrete containing CSF and natural aggregates. This effect cannot be simply explained by the higher permeability of the control samples compared to CSF concrete, e.g. to greater alkali leaching and consequent lower long term expansion (Rogers and Hooton (10)). Indeed, no pessimum effect was observed with the very reactive siliceous limestone tested. In general, the above results are in good agreement with those obtained by Durand (11) who tested a high-silica/low-alkali CSF with three reactive aggregates: 1) the Spratt limestone; 2) a very reactive limestone from Trois-Rivières (Québec), and 3) a chloritic schist responsible for AAR problems in the Sherbrooke area (Québec). The results obtained for a period of 4 years appear in Figure 2. Again, 5% CSF is not sufficient for all three reactive aggregates. Moreover, the rate of

expansion of the concrete prisms made with chloritic schist and Spratt limestone rose significantly during the last two years, even with 10 to 15% CSF, again suggesting that CSF may just postpone AAR expansion. Grattan-Bellew (12) tested concrete specimens made with Spratt limestone and 11% CSF. After showing very low expansion up until to 1 year, the specimens started to expand rapidly, exceeding the usual criterion of 0.04% at 2 years (Figure 3). The author questioned the long term effectiveness of CSF in suppressing expansion due to ASR. The results obtained by Oberholster (13) for concrete cubes exposed outdoor and made with reactive hornfel aggregates from South Africa, two different alkali contents (≈ 3.9 and ≈ 5.1 kg/m^3) and various amounts of silica fumes (0, 5 and 10%) are presented in Figure 4. These results also suggest that CSF may only refer expansion due to AAR. Moreover, the higher the CSF content and the lower the alkali content (e.g. the cement content), the longer the delay before expansion.

LABORATORY TESTING

Based on these experiments, it is firmly recommended to use conservative limits when conducting laboratory tests to determine the effectiveness of silica fume in the presence of alkali-reactive aggregates. As discussed elsewhere (3), with a given reactive aggregate and for a given mix design, one cannot use any variety of silica fume, neither in any proportion; such conditions usually call for laboratory testing. The proposed tests are reviewed and discussed elsewhere (3, 9, Duchesne and Bérubé (14)). In this respect, the Pyrex Mortar Bar Method ASTM C 441 is not appropriate since the amount of CSF used (25% as cement replacement) is unrealistic, while pyrex does not behave as a real aggregate. The Concrete Prism Method CSA CAN3-A23.2-14A is the most highly recommended test. The alkali content of the mixture must always be increased to 1.25% of the cement mass (Na$_2$O eq.) by adding NaOH to the mixture water. Otherwise the test is not sufficiently accelerated and low expansion could be observed irrespective of the CSF content. Using a higher cement content (e.g. 420 kg/m^3 rather than 310) and controlling the water/cement between 0.42 and 0.45 is also recommended. With a given reactive aggregate, the condensed silica fume under study should be used in sufficient amounts to produce long term expansions not higher than 0.04% (2 years or more), and precautions should be taken in order to minimize alkali leaching from the test specimens during all the testing period. A more conservative criterion is to achieve expansion similar or lower than control specimens made with a low-alkali cement (<0.6% Na$_2$O equiv.) (3, 9, 14)). The Accelerated Mortar Bar Method (NBRI or ASTM C-9 Proposal P 214), which involves immersion of mortar bars in NaOH 1N at 80°C for two weeks, leads to results which are in good agreement with those from the CSA Concrete Prism Method for pulverized fly ash and granulated blast furnace slag, using a criterion of 0.1% expansion at 14 days (3, 9, 14). However, using the same limit, the method is apparently not severe enough for CSF (14). On the other hand, when used for evaluating the effectiveness of mineral admixtures in suppressing ASR expansion, the autoclave mortar bar method recently proposed by Fournier et al. (15) and Bérubé et al. (16) appeared appropriate for condensed silica fume and blast furnace slag, but a little too severe for pulverized fly ash, using a 5-hour, 0.1% expansion limit criterion (14).

REACTION MECHANISMS

Various mechanisms are proposed to explain the effectiveness of mineral admixtures and CSF against AAR. This subject is overviewed by Turriziani (17) and Bérubé and Duchesne (3). The principal mechanisms suggested are: 1) lower permeability and consequent lower ion mobility; 2) strength improvement and higher resistance to expansive forces developped by AAR; 3) alkali dilution resulting from cement replacement by admixture with lower available alkali content; 4) pozzolanic reaction producing secondary (pozzolanic) CSH which entrap alkali ions and deplete portlandite in the cement paste, thus reducing the alkali ions and the pH in the pore solution.

Effect on porosity and permeability

It is well known that CSF concretes are much less permeable than normal concretes. CSF refines the porosity in the 5 to 50 µm range and produces amorphous CSH which tend to completely fill the transition zone at the aggregate-paste interface (3, Regourd (18)). However, the

bulk porosity remains quite unchanged (3). Consequently, it is suggested that the mobility of the agressive alkali and hydroxyl ions is significantly reduced, which in turn reduces the rate of AAR (11). However, results by Bérubé and Duchesne (3) suggest that the permeability decrease has little effect on AAR expansion, and that ionic diffusion is still relatively easy inside CSF concrete.

Strength improvement

CSF concrete usually develops higher strengths than normal concrete with the same water/cement, and one may suggest in turn that stronger concrete resists more to expansive stresses developed by AAR. As reviewed in (3), the gain in strength may be attributed to various factors such as filler effect, porosity refinement, substitution of discontinuous pores by continuous capillary pores, decrease in portlandite, stronger aggregate-paste interface. However, this gain cannot explain why CSF are effective in reducing expansion in the presence of reactive aggregates. Indeed, experiments conducted in our laboratory on normal concrete indicated that the lower the water/cement (at least between 0.6 and 0.45), the higher the expansion due to AAR, despite a corresponding higher strength (Bérubé and Fournier (19), Bérubé et al. (20)).

Alkali dilution

Some authors attribute the beneficial effect of pulverized fly ash and blast furnace slag to alkali dilution when adding materials (e.g. admixtures) that supply less alkali to the pore solution than the cement. According to Hobbs (21), PFA's and GGBFS's release on an average about 1/6 and 1/2 of their total alkali content in the pore solution, respectively. However, other experiments reported in (3) and (5) suggest that mineral admixtures release almost all their alkalies in the long term. Oberholster (13) clearly demonstrated that blast furnace slag was still very effective after 7 years of outdoor exposure in suppressing expansion due to reactive aggregates, despite the fact that active alkali content of the mix was readjusted in order to obtain the same amount as in the control with no slag. A number of experiments reported in (3) clearly demonstrated that the main beneficial effect of CSF against AAR cannot be attributed to a simple dilution effect. This is not surprizing considering the relatively low CSF amounts normally used in concrete. Moreover, some CSF's contain more than 2% alkali (Na_2O eq.), therefore supply more alkalies than the cement, while remaining effective against AAR, provided a sufficient amount is used (3, 5).

Pozzolanic reaction

CSF can be regarded as a super-reactive "aggregate" due to its high amorphous silica content and extreme fineness. CSF particles react rapidly with the basic and alkaline concrete pore solution and develop pozzolanic reaction with portlandite. The reaction mechanisms involved are:
1) Dissolution or gelification of CSF particles, which depletes some OH^- and alkali ions.
2) Reaction of silica ions or silica gel with portlandite to produce (pozzolanic) CSH with a low CaO/SiO_2 ratio and which entrap alkali ions. This reaction progressively depletes portlandite, alkali and hydroxyl ions.
3) The pH is lowered as a result of alkali depletion in the pore solution. Indeed, portlandite supplies just enough OH^- ions to reach equilibrium with alkali ions. Provided CSF is present in sufficient amounts, the concentration of alkali and hydroxyl ions falls to a critical level under which the alkali aggregate reactions are considerably reduced, if not totally stopped. High CSF contents (e.g. 20% or more) are normally sufficient to deplete all the portlandite; in such a case, the pH decreases under 12.45 (which is the saturation pH with respect to portlandite, in absence of alkali ions), irrespective of the alkali concentration.

Chemistry of pore solution

Several researchers mentioned elsewhere (3) performed chemical analyses of pore solution extracted under high pressure from hydraulic mixtures. In general, the tests were performed on pure cement pastes (in absence of reactive aggregates), or on mortar specimens with or without reactive aggregates. To our knowledge, only one attempt was made on concrete specimens (11). A

comprehensive overview of the subject is found in Nixon and Page (22) and other experiments are described in (3). These experiments confirmed that: 1), in the presence of CSF, significant decreases of Na$^+$, K$^+$ and OH$^-$ ion concentrations occur in the pore solution, even for relatively low CSF contents (e.g. 5 to 10%); 2), the amount of residual OH$^-$ and alkali ions in the pore solution is inversely proportional to the CSF content; 3), the pH is lower than 12.45 with high CSF contents, (30% in a particular study); 4) K$^+$ ions are sligthly more depleted than Na$^+$ ions in the pore solution, in other words more integrated into the pozzolanic CSH, and 5), all concentrations (K$^+$, Na$^+$ and OH$^-$) are higher in the pore solution when the water/cement is lower, which agrees with the fact that a fixed amount of alkalies is dissolved in a smaller volume of pore solution.

Reaction products

According to many authors (3), the CSH produced by pozzolanic reaction between portlandite and silica fume present a low CaO/SiO$_2$ ratio ($\approx$1). On the other hand, low Ca/Si are favourable in order to entrap more alkalies in their structure (3). These observations are in good agreement with the fact that alkalies are depleted in the pore solution. Moreover, as observed on many occasions with the electron microprobe or the scanning electron microscope (3), CSH formed by pozzolanic reaction between portlandite and silica fume present textures and chemical compositions similar to the classical reaction products of alkali-silica reactions, and effectively contain significant amounts of alkalies, much more than normal CSH.

Role of portlandite

As previously mentioned, portlandite is depleted in the cement paste as a result of pozzolanic reaction and production of additional CSH. The higher the amount of CSF, the higher the depletion in portlandite. This has been clearly demonstrated by a number of workers (3). Moreover, 20 to 24% CSF is normally sufficient to deplete all the portlandite, which allows the pH to decrease under 12.45. Under such conditions, AAR should not take place, eventhough the alkali concentration is high in the pore solution. For the two silica fumes reported in (5) (see Figure 1), the reduction in portlandite increased with the amount of CSF and its SiO$_2$ content. However, in the same study (5), the mineral admixture which consumed the highest amount of portlandite was a very high alkali fly ash, which in turn was the worst among all admixtures tested in reducing AAR expansion in concrete. In fact, the most critical factor is the alkali concentration, which controls the pH (portlandite must release OH$^-$ ions to obtain solution equilibrium, not more). In our mind, the depletion in portlandite is just a consequence of the pozzolanic reaction and is not beneficial per se, unless completely depleted. Indeed, just a few percents of this phase in the cement paste is sufficient to supply OH$^-$ ions in equilibrium amounts to the pore solution.

Long term behavior and effect of external alkalies

A number of results presented before (Figures 1 to 4) suggest that the beneficial effect of CSF may not persist for a long period of time. This is also questioned by Bhatty & Greening (23) who observed in some occasions that alkalies may be released in the long term and be available for alkali-aggregate reactions. Chemical analysis performed in our laboratory on pore solution extracted at different time intervals from cement pastes made with 5, 10 and 15% of the two CSF's reported in Figure 1 (5), also suggest that the effectiveness of CSF against ASR is not related to portlandite depletion, but to the amount of alkalies in the pore solution, in other words to the pH. Alkalies entrapped early in pozzolanic CSH may be eventually recycled to some extent in the pore solution (Figure 5A). Indeed, the alkali concentration increased in the pore solution with time, while decreasing in the presence of effective fly ashes (Figure 5B) and ground granulated blast furnace slag (Figure 5C). In this particular study, a reduction of 35% of alkalies in the pore solution with respect to control specimens containing no mineral admixture was found to be sufficient to keep expansion under 0.04% up until to 1.5 years (5).

As also suggested by Bhatty (23), such an alkali recycling could be explained by the fact that the pozzolanic CSH which are formed very rapidly with CSF are in chemical equilibrium with the usual high alkali/low calcium pore solution. However, as the concentration of alkali in the solution drops, Ca^{+2} will replace alkalies in these CSH. The recycled alkalies may rapidly aggress the potentially reactive aggregates since it has been demonstrated before that CSH produced by pozzolanic reaction between silica fume and portlandite tend to fill the transition zone at the cement/aggregate interface (18). Let us recall that the classical products of alkali-silica reaction, which present textures and chemical compositions similar to the CSH produced by pozzolanic reaction between silica fume and portlandite (18), also tend to reorganize in the long term by trapping more and more calcium from the pore solution, and/or by contact with the cement paste (portlandite) (Bérubé and Fournier (25)), while probably releasing alkalies. Provided that portlandite is not entirely depleted by pozzolanic reaction, (which normally requires CSF content in excess of 20%), additional alkali from external sources such as sea water and deicing salt should also produce the same effect as the alkali recycling (3). However, these alkalies will not be deleterious if they are not counterbalanced by OH⁻ ions, e.g. if there is no more portlandite in the cement paste.

CONCLUSION

Among the mechanisms proposed to explain the effectiveness of condensed silica fume (and of mineral admixtures in general) in suppressing expansion due to alkali-aggregate reactivity, the most important appears to be the depletion of alkali ions and the consequent pH decrease in the pore solution, as a result of pozzolanic reactions involving portlandite and condensed silica fume. These reactions result in pozzolanic CSH with a low CaO/SiO_2 and with relatively high alkali contents. The CSF content must be sufficient enough to reduce the alkali in the pore solution and the pH to a safe level, but expansive AAR may take place if the concrete is exposed furtherly to external sources of alkali (sea water, deicing salts,...). Indeed, OH⁻ ions will be released in solution by the residual portlandite to satisfy equilibrium with the alkali ions. The other effects resulting from the pozzolanic reactions (higher strength, lower permeability, portlandite depletion) are thought to play a minor role in the effectiveness of CSF against AAR, (unless the CSF content is sufficient to deplete all the portlandite).

On a long term basis, there is a possibility that the low CaO/SiO_2 and high-alkali CSH produced by early pozzolanic reactions entrap calcium, while recycling alkalies in the pore solution, which then become available for alkali-aggregate reactions. Such a phenomena could explain why hydraulic samples containing 10% condensed silica fume or more sometimes develop significant expansion on a long term basis. Such an alkali increase with time in the pore solution of cement pastes made with CSF has been observed in this study as well as by other workers.

Unfortunately, well documented information concerning long term field performance of CSF concretes made with reactive aggregates is not available. For this reason, it is firmly recommended to use very conservative criteria when conducting expansion tests on concrete, and to perform the tests for at least 2 years.

ACKNOWLEDGEMENTS

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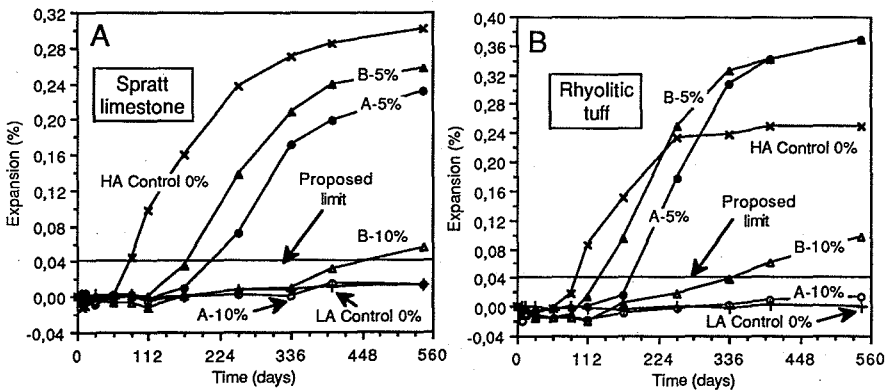25. Bérubé, M.A., and Fournier, B., 1986, Canadian Mineralogist 24, 271.

Figure 1: Expansion of concrete prisms made with a high alkali content (350 kg/m^3 of cement, 1.25% Na$_2$O eq. of the mass of cement), various amounts of two CSF's, one high silica/low alkali (CSF-A) and one low/silica/high alkali (CSF-B), and two very reactive aggregates, the Spratt limestone (A) and a rhyolitic tuff (B). Low alkali controls were also made with a low alkali cement (0.54 Na$_2$O eq.) (From Duchesne and Bérubé (5)).
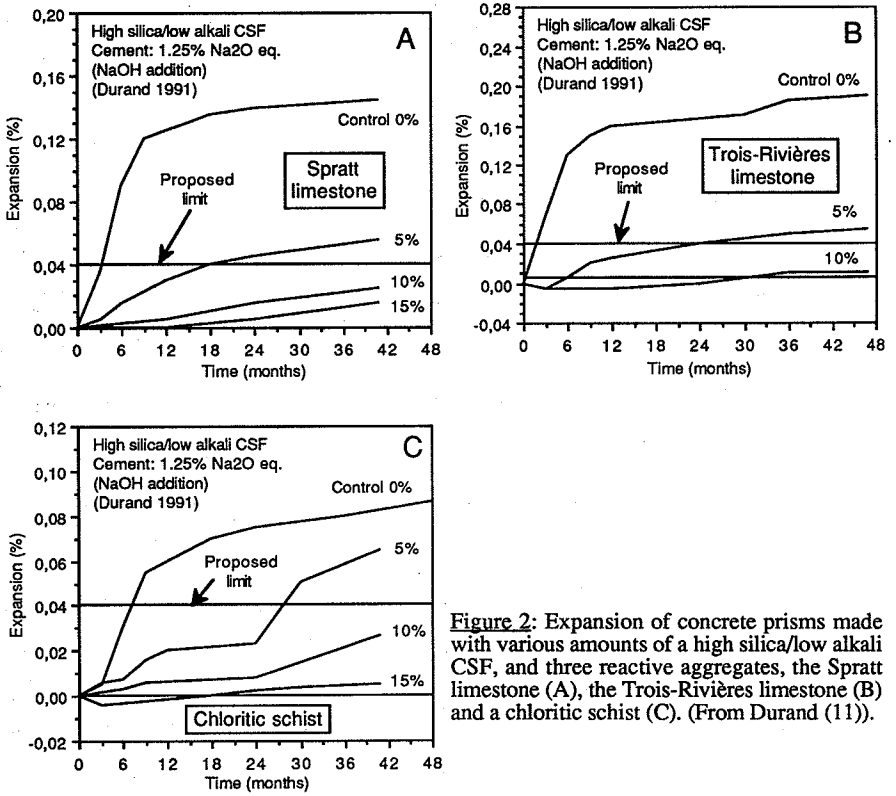
Figure 2: Expansion of concrete prisms made with various amounts of a high silica/low alkali CSF, and three reactive aggregates, the Spratt limestone (A), the Trois-Rivières limestone (B) and a chloritic schist (C). (From Durand (11)).
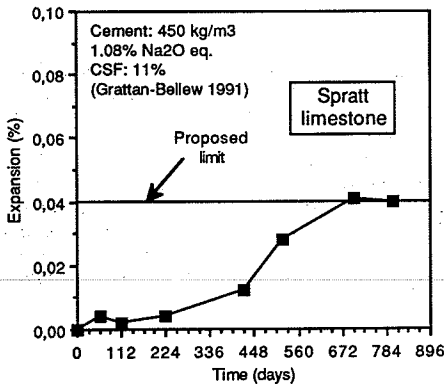

Figure 3: Expansion of concrete prisms made with the Spratt limestone and 11% CSF. (From Grattan-Bellew (12)).
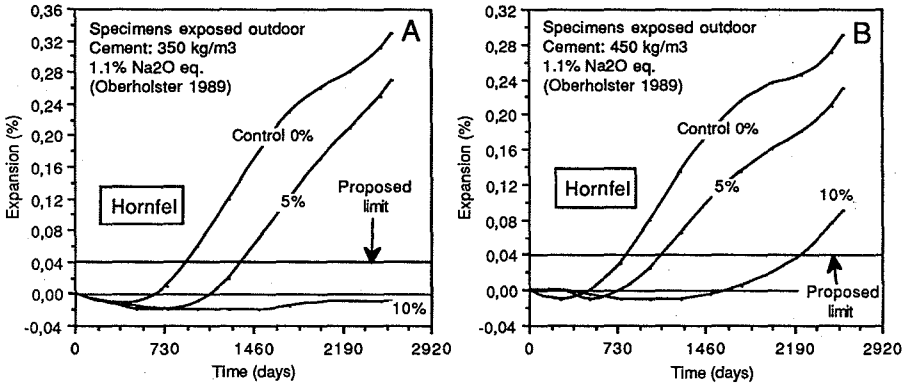
Figure 4: Expansion of concrete cubes exposed outdoor and made with reactive hornfel aggregates from South Africa and various CSF contents (0, 5 and 10% of the mass of cement replaced by a CSF volume equivalent). Two cement contents were used 450 kg/m^3 (A) and 350 kg/m^3 (B). (From Oberholster (13)).
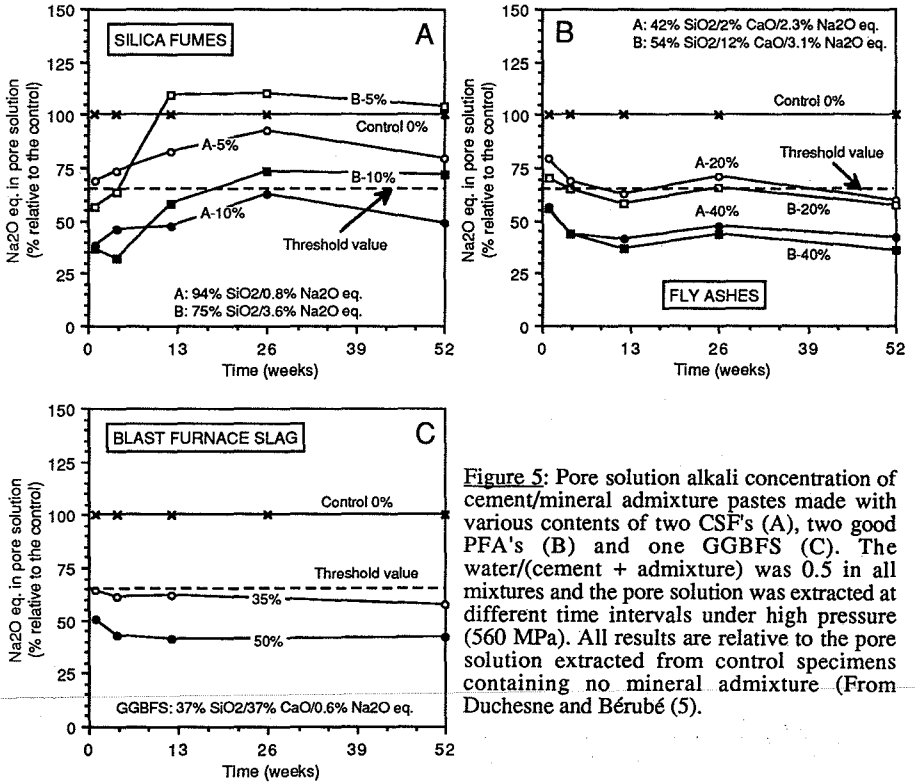

Figure 5: Pore solution alkali concentration of cement/mineral admixture pastes made with various contents of two CSF's (A), two good PFA's (B) and one GGBFS (C). The water/(cement + admixture) was 0.5 in all mixtures and the pore solution was extracted at different time intervals under high pressure (560 MPa). All results are relative to the pore solution extracted from control specimens containing no mineral admixture (From Duchesne and Bérubé (5).

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