

THE ALKALI-AGGREGATE REACTION - CONCRETE MICROSTRUCTURAL EVOLUTION

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SYNOPSIS

The alkali-aggregate reaction has been studied by scanning electron microscopy and energy dispersive X-ray analysis, electron probe microanalysis, and X-ray diffraction in concretes containing glass aggregates or hornfels and greywacke aggregates. The surface reaction of the natural aggregates in alkaline solutions has been analysed by X-ray photo-electron spectrometry.

The study of concretes with glass aggregates stored at 20 $^{\circ}$ C and 100 per cent relative humidity has revealed, irrespective of alkali content and type of cement, the formation of a gel containing SiO_z, Na_zO, CaO, MgO and Al_zO₃. Under heat and pressure (210 $^{\circ}$ C at 2 MPa for 48 hours), the gel crystallizes and yields silicates not very different from tobermorite found in autoclaved normal concretes but containing Na and K in solid solutions.

The alkali reaction in two natural aggregate concretes, is also shown by the formation of gels and silicate crystals. The progressive structuring of the gels in silicate crystals is promoted by an increase in temperature. Ettringite and $Ca(OH)_2$ reinforce the alkali-aggregate reaction which may be looked upon as a hydration reaction, partially of the pozzolanic type.

SAMEVATTING

Die alkali-aggregaatreaksie is deur middel van skandeerelektronmikroskopie en energiedispersiewe X-straalontleding, mikrosonde-ontleding en X-straaldiffraksie in beton met glasaggregate of hornfels en grouwak aggregate bestudeer. Die oppervlakreaksie van natuurlike aggregate in alkali-oplossings is met behulp van X-straalfotoelektronspektrometrie ontleed.

Die ondersoek van beton met glasaggregate wat by 20 $^{\circ}$ C en 100 persent relatiewe humiditeit gestoor is het aan die lig gebring dat, ongeag die alkali-inhoud en sementtipe, 'n jel gevorm het wat SiO₂, Na₂O, CaO, MgO en Al₂O₃ bevat. Die jel het met hitte en onder druk (210 $^{\circ}$ C by 2 MPa vir 48 uur) gekristalliseer en silikate voortgebring wat nie veel van tobermoriet verskil wat in geoutoklaafde gewone beton gevind word nie, maar Na en K in soliede oplossings bevat.

Die alkalireaksie in twee betonstukke van natuurlike aggregaat word ook deur die vorming van jel en silikaatkristalle aangetoon. Die progressiewe strukturering van die jel in silkaatkristalle word deur 'n verhoging in temperatuur bevorder. Ettringiet en $Ca(OH)_2$ versterk die alkali-aggregaatreaksie wat beskou kan word as 'n hidreringreaksie, gedeeltelik van die pozzolaantipe.

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FIGURE 1 : Gel (1) penetration in the expanded glass pores (2) wrapped in cement paste (3).



FIGURE 2a : SEM. After one year, gel (6) in contact with the glass aggregate.



FIGURE 2b : SEM. Gel evolution after 18 months. Its texture resembles that of the C-S-H of the cement paste.

1. INTRODUCTION

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In cases where the alkali-aggregate reaction has been detected in degraded concrete containing amorphous silica sands (opal, chalcedony), silicate aggregates (gneiss, schist, sandstones, basalt) or clay dolomitic limestone, investigation has revealed the presence of expansive alkaline silicate gel¹. Studies carried out over the last forty years have come to the conclusion that it is caused by the simultaneous influence of factors such as the alkali content in the cement, the content and type of unstable minerals in the natural aggregates, the humidity of the surrounding environment and the temperature. Recent research has emphasised the role of hydrates such as ettringite² and $Ca(OH)_2^3$ in the cement paste.

In our research we have tried to follow, at a microstructural level, the development of the alkali-aggregate reaction under favourable conditions, that is in concrete containing industrial glass-aggregate and concrete containing natural unstable aggregate, treated at 40 $^{\rm O}$ C.

2. THE ALKALI-AGGREGATE REACTION IN GLASS-AGGREGATE CONCRETE

The glass-alkali reaction involving sodium-calcium glasses has already been studied at the Research centre for the hydraulic binders industry (CERILH) by G Durniak⁴. The industrial glass used was shown to be reactive by the ASTM 289-71 test. Concrete specimens prepared from different types of cement with variable alkali content and stored at 100 per cent relative humidity or immersed in water at 20 °C developed gels containing essentially Na, K, Si, Ca and to a lesser extent, Mg and Al. This gel, in the case of massive glass aggregates induces expansion and rapid destruction of the specimens. On the other hand where expanded glass aggregates of the same composition were employed, there was neither swelling nor loss of mechanical strength after two years in water at 20 °C. The gel can be seen in the glass pores i.e. the gel which causes the swelling in the former instance now collects in the pockets (Figure 1).

The concrete interstitial solution (extracted by compression and analysed by chemical means) contains, from an early age, a high sodium content which originates from the dissolution of the glass. By one year, the pH of the aqueous phase is in the region of 13 and good correlation exists between the OH⁻ ion concentration and those of the alkalis Na⁺ and K⁺. Calcium and magnesium were not detected.

Examination by scanning electron microscopy (SEM) shows that the gel noted at one year (Figure 2a) develops a texture which when observed at 18 months resembles that of the C-S-H of the cement paste (Figure 2b).

After two years storage in water at 20 °C, the samples were autoclaved for 48 hours at 210 °C under 2,2 MPa pressure. The gel inside the expanded glass aggregates was transformed into foliated crystals of which the X-ray diffraction pattern was similar to that of tobermorite

alkali oxide content of the cement used is not known. The aggregates are of a similar mineralogical nature to those making up Concrete A, but the crystallisation is slightly coarser. They are formed of large quartz crystals and feldspars (orthoclase, albite) and a brown pleochroic ground-mass comprising mainly phyllitic minerals (chlorites or vermiculite, biotite) mixed with small quartz and feldspar crystals. The micas and feldspars show localised signs of degradation. In both concretes, the alkali-aggregate reaction manifests itself, in an outer dark zone and an inner white zone around the majority of aggregates fractured along a cleavage plane. The texture and composition of these two zones are clearly shown by scanning electron microscopy and by electron probe microanalysis.

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(a) Analysis of Concrete A. In Concrete A stored at 40 °C, the internal white zone consists of foliated crystals more or less dispersed containing Si, K and Ca (Figures 4a and 4b). The dark zone in contact with the aggregate is made up of a cracked gel rich in SiO₂ but containing also Ca and K (Figures 5a and 5b).



FIGURE 5a : SEM. Concrete A, 40 °C, Gel in contact with the aggregate.



FIGURE 5b : EDS. Gel analysis.

This gel seems to achieve a pseudomorphosis of the aggregate without solution continuity between the gel and the rock (Figure 6). Between the white deposit and the dark edge often exists a zone with a texture intermediate between that of a gel and that of a polycrystalline phase (Figures 7a and 7b). Furthermore, a gel of molar ratio CaO/SiO_{2} (C/S = 1,3) higher than that of the gel in contact with the aggregate (C/S = 0.25) may be observed in the cement paste, notably in the larger pores (Figure 8). This composition is close to that of the more usual C-S-H (C/S = 1,5). Some ettringite C₃ A.3CaSO₄.32H₂O rich in SiO₂ and some thaumasite CaCO₃.CaSO₄.CaSiO₃.15H₂O in large crystals surround the aggregates and are seen present in the cement paste (Figure 9). The calcium hydroxide, Ca(OH)₂ also in large crystals, is likewise to be found in contact with the aggregates. The hydrated cement paste still encloses several grains of anhydrous cement which contain a noticeable amount of potassium either in the form of sulphates in inclusion in C_3 S or in solid solution in C_3 A (Figure 10).

Although their porous texture and their water content could lead to error in the CaO/SiO, ratios, the electron probe microanalysis shows that the gel in the cement paste contains more calcium but less potassium than that which surrounds the aggregates (Table 1). The values are to be compared with those obtained by N Thaulow and T Knudsen⁶ for gels in the case of the opal/cement paste reaction.

The crystals of the white zone sampled by scraping are characterized by 10 X-ray diffraction peaks. The two most intense are at 12,3 and 8,6 Å. The width of these peaks is comparable to those obtained from clay minerals. The peak at 8,6 Å has already been measured by A Buck and K Mather⁷, in samples of concrete degraded by alkaliaggregate reaction; it has been attributed to a phase analog of hydrated tricalcium silicate $Ca_6 (Si_2O_7)OH_6$.

(b) Analysis of Concrete B. The alkali-aggregate reaction which was produced under natural environmental conditions in Concrete B is of the same type as that noted in Concrete A which was accelerated in the laboratory. Nevertheless the gels, around the aggregates and at the edges of the cement paste were richer in calcium than those in Concrete A (Table 2).

Otherwise, the crystallized calcium carbonate in plates or needles (Figure 11), foliated silicate crystals (Figures 12a and 12b), massive ettringite (Figure 13) and calcium hydroxide are also observed in contact with the aggregates.

(c) Behaviour of natural aggregates in KOH and Ca(OH)₂ solutions. Sound aggregates taken from samples A and B were polished and then immersed in solutions of KOH(N); Ca(OH)₂ (saturated) and a mixed KOH(N) and saturated Ca(OH), solution all at 80 °C. The treatment time was from three to five days.

After 3 days in the KOH(N) solution, the aggregates of Concretes A and B showed evidence of slight attack which $Ca_{5}Si_{5}H_{4}O_{19}$, or crystals approaching foshagite $Ca_5 Si_3 H_6 O_{1.4}$ (Figures 3a and 3b). These two silicates which can be detected in ordinary autoclaved concrete contain in this case Na and K alkalis.

The same treatment applied to 28-day-old samples lead to the appearance of the same crystalline hydrated silicates (essentially sodium rich). This gel form has not been detected inside the expanded glass aggregates prior to autoclave treatment. Temperature acceleration of the alkali-aggregate reaction may thus be used for accelerated testing.

3. ALKALI-AGGREGATE REACTION IN NATURAL AGGREGATE CONCRETES

Two concretes from South Africa referred to as Concrete A and Concrete B form the subject of this study.

Concrete A, was prepared at the NBRI laboratories from cement containing 0,97 per cent equivalent Na₂O. This



FIGURE 3a : SEM. Autoclaved glass aggregate concrete after 2 years in water at 20 °C. Crystals resembling tobermorite C₅ Si₅ H₄ O₅ .



FIGURE 3b : SEM. Autoclaved glass aggregate concrete after 2 years in water at 20 °C. Crystals resembling foshagite $Ca_5 Si_3 H_6 O_{14}$.

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sample was a piece of concrete cube which had been stored at 40 °C for one year under 100 per cent relative humidity. The aggregate is a mixture of dense hornfels and greywacke⁵. These aggregates show a tendency to cleave which is demonstrated by their rupture along distinct planes when subjected to the concrete bresilian test. The mineral content is mainly quartz, feldspars (albite, anorthite, orthoclase), phyllosilicates (biotite mica, chlorites and/or vermiculite) with which various minerals such as magnetite and pyrite may be associated. The quartz is localised in veins or in small cryptocrystalline overlapping elements of poorly defined shape. The undulatory extinction of most of the crystalline minerals in polarised transmitted light indicates that they are in a state of stress. Some sections parallel to the obvious cleavage planes appear to be highly enriched in mica whose oriented crystals are closely bound with cryptocrystalline quartz.

Concrete B was sampled by core drilling from a retaining wall of a bridge built 20 years ago near Cape Town. The



FIGURE 4a : SEM. Concrete A, 40 °C. White deposit on the aggregate. Crystalline silicate of K and Ca.



FIGURE 4b : EDS. Analysis of crystals.



Absorbed electron image



Calcium

5



Silicon



Potassium

FIGURE 8 : EPMA. Concrete A, 40 °C. Gel deposit (1) in a pore of the cement paste (2), near an aggregate (3)



FIGURE 9 : SEM. Concrete A, 40 °C. Heaped ettringite.

was shown by the silhouetting of the micaceous phases. After 5 days aggregate A had formed a thin layer with a cellular or smooth foliated structure (Figure 14). The smooth deposit may be due to the partial exfoliation of the micaceous elements. The large quartz crystals were, on the scanning electron microscope scale, free of reaction product. In the Ca(OH)₂ solution, no sign of attack was observed after 3 days.

In the KOH and Ca(OH)₂ mixed solution, a very large reaction zone was evident after 3 days on both the A and B aggregates. The foliated structure was more or less cellular. On aggregate B, the texture of the deposit resembled that of tangled K and Ca silicate crystals forming the white zone observed in Concretes A and B (Figure 15).

The polished sections of aggregate A were analysed by Xray photoelectron spectrometry (XPS) after the 3-day treatment. The tracing of peak profiles Ca_{2p} and Si_{2p} showed a significant enlargement of the silicon peak (width at mid-



Absorbed electron image



Silicon

FIGURE 6 : EPMA. Concrete A, 40 °C. Pseudomorphosis of aggregate (1) by the gel (2)



FIGURE 7a : SEM. Concrete A, 40 °C. Microcrystalline gel.



Calcium



Potassium







4



Absorbed electron image



Aluminium



Potassium



Sulphur

C/S

FIGURE 10 : EPMA. Concrete A, 40 °C. Anhydrous clinker grain containing K in the form of alkali sulphates included in C_3 S (1) and in solid solution in C_3 A (2). Cement paste (3)



Concrete B. Calcium carbonate FIGURE 11 : SEM. crystals.

height 2,6 \pm 0,1 eV) in the case of the aggregate stored in the KOH(N) solution and in that immersed in the KOH and $Ca(OH)_2$ solution. The width of this peak was 2,2 \pm 0,1 eV in the non-treated aggregate diagramme and in that of the section stored in the lime solution. In an XPS study on the properties of Beltane opal, W A Gutteridge and D W Hobbs⁸ found two species of silicon Si_{2D} with different binding energies which led them to suggest the simultaneous presence of a sodium silicate and of a sodium calcium silicate.

The value of the CaO/SiO₂ molar ratios obtained via a calibration curve of Ca_{2p}/Si_{2p} against the Ca/Si atomic ratio⁹ was as follows:

C/S	=	0,11 in the standard sample (non
		treated aggregate),
C/S	=	0,12 in the KOH treated aggregate,

= 0,61 in the KOH and Ca(OH)₂ treated aggregate.

	CaO	SiO2	K ₂ O	C/S	(C + K)/
Gel near	17,8 ± 0,4	63,5 ± 0,5	2,84 ± 0,11	0,30	0,33
aggregates	15,5	54,1	0,84 ± 0,03	0,31	0,32
	13,0	63,6	4,20 ± 0,10	0,22	0,26
	13,0	59,5	5,10	0,23	0,29
	13,6	58,8	5,10	0,25	0,30
	13,2	65,3	6,50	0,22	0,28
Gel in	41,4 ± 0,6	34,7 ± 0,4	0,41 ± 0,03	1,3	1,3
the	39,3	34,1	0,57	1,2	1,2
cement	40,5	36,5	0,59	1,2	1,2
paste	41,5	35,3	0,36	1,3	1,3
Crystals	11,2 ± 0,5	52,4 ± 0,5	8,9 ± 0,2	0,23	0,33
	12,3	53,4	9,4	0,24	0,36
	12,1	51,7	10,5	0,25	0,38
	11.3	56.8	8,4	0,21	0,30

	CaO	SiO₂	K₂0	Na ₂ O	C/S	(C + K)/S
Gel near aggregates	23,7 ± 0,4 21,5	53,8 ± 0,5 51,1	1,7 ± 0,1 4,7 ± 0,1	∿ 0,40 ∿ 0,40	0,47 0,45	0,49 0,51
Gel in the cement paste	35,2 ± 0,6	27,9 ± 0,4	0,67 ± 0,03	n.d.	1,4	1,4
Crystals	9,1 ± 0,3	43,1 ± 0,5	6,6 ± 0,2	2,1 ± 0,3	0,23	0,32

TABLE 1 - Concrete A. Electron probe microanalysis of gels and crystals (12 kV, 50 nA) Composition at various points of the sample, (confidence interval with a 5 per cent threshold).

TABLE 2 - Concrete B. Electron probe microanalysis of gels and crystals. Same conditions as in Table 1.

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FIGURE 14 : SEM. Concrete A. Polished section after 5 days in a KOH(N) solution at 80 °C. Cellular (1) and foliated (2) deposit. Quartz (3) with no deposit.



Concrete B. Polished section after FIGURE 15 3 days in a KOH + Ca(OH), solution at 80 °C.

(b) As a result of the dissolution of the cement alkali, the interstitial aqueous phase of the concrete essentially contains the ions K^+ and Na⁺ and the hydroxyl ions OH⁻. (The concentration of Ca^{2+} is very low in comparison to those of the alkalis³.¹³.) The hydroxyl ions of the interstitial alkaline solution with a high pH value (close to 13) attack reactive parts of the aggregate such as cryptocrystalline quartz. The product of the reaction will be a quite low viscosity 'sol' capable of diffusing into the surrounding cement paste¹⁴. The aggressive 'sol' may react directly with those hydrated paste constituents which are rich in calcium (C-S-H and Ca(OH)₂) with the release of Ca²⁺ ions. The calcium ions diffuse in their turn towards the reaction zone in contact with the aggregate, which may explain their presence in the gel and the crystals formed from the alkali-reaction. This process is all the more, easy because Ca(OH)₂ forms in direct contact with aggregates in the first stages of hydration. A supplementary argument in favour of this process is the partial 'gelification' of the cement paste next to the aggregate and the existence of pores more or less full of a gel which is rich in calcium (molar ratio CaO/SiO₂ $\stackrel{\circ}{\rightharpoonup}$ 1,3). The 'gelification' of the cement paste is due to its impregnation by the aggressive 'sol' which attacks the constituents themselves which are rich in calcium, and provokes the formation of a secondary gel to fill the pores.

(c) Ettringite crystallization in abundance may also reinforce the alkali-aggregate reaction in two ways. Firstly it is susceptible to the creation of stress states which lead to the cracking of the concretes and secondly it may change the composition of the paste interstitial solution. The formation of ettringite absorbs calcium and allows a larger dissolution of Ca(OH), which in turn liberates hydroxyl ions which attack the aggregate².

(d) The alkali-aggregate reaction may be looked upon as a hydration reaction as G M Idorn¹⁵ suggested in his general report to the Congress on the Chemistry of Cement (Paris -1980). This reaction would be partially of the pozzolanic type.

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FIGURE 12a: SEM. Concrete B. Crystals of the white deposit on the aggregate.



FIGURE 12b: EDS. Analysis of crystals.



Concrete B. Massive deposit of FIGURE 13: SEM. ettringite.

The depth of the X-ray photoelectron spectrometry investigation, in the case of calcium silicates was of the order of 60Å. Thus, these first studies show that a superficial reaction between the aggregate and the alkaline solution occurs rapidly at 80 °C and that it is greatly encouraged when in the presence of lime.

4. DISCUSSION

The alkali-aggregate reaction in the concretes studied is shown by the formation of gels and Ca, Na and K silicate crystals : gels of high SiO₂ content (molar ratio CaO/SiO₂ $^{\circ}$ 0.2 to 0.4) around the aggregates and richer calcium gels $(CaO/SiO_2 \xrightarrow{1} 1,3 \text{ to } 1,4)$ in the cement paste. The crystals in contact with the aggregates have a higher concentration of sodium in the case of glass aggregates, of potassium in the case of natural aggregates than the gels, but their composition is close to that of the gel surrounding the aggregates (CaO/SiO₂ $\stackrel{\circ}{-}$ 0,23). It is this gel which, by absorbtion of water, is essentially responsible for the expansion and cracking of concrete¹⁰. It appears likely that the formation of crystalline silicates follows that of the gel, of which the progressive structural formation has been observed in the case of glass aggregates. As is shown by the tests with Concrete A at 40 °C, and with the aggregates treated in an alkaline solution at 80 °C, and autoclaved concretes with glass aggregates, the development of the gel structure is promoted by a rise in temperature. The texture and the morphology of these crystals appear close to well crystallized tobermorites.

In both Concrete A and B, the alkali-aggregate reaction is related to the presence of alkalis in the form of sulphates in the cement and in solid solution in the clinker minerals, C. A in particular (0.97 per cent Na₂O equivalent for Concrete A) combined with the fact that the aggregates contained reactive phases such as cryptocrystalline quartz, micas, chlorite, and vermiculite. On the one hand, the state of strain of the small quartz crystals enabled them to react with alkalis¹¹; on the other hand, in the presence of water or alkaline solutions the phyllitic minerals were likely to scale off¹².

The reaction progresses according to the natural cleavage planes in the rock. As a result of crushing, it is possible that certain planes of cleavage were open, allowing penetration and diffusion of the aggressive alkaline solution to the interior of the aggregate.

The results of our analysis tend to show that in the reaction mechanism between alkalis and aggregates four situations will be likely.

(a) The reactive parts of the aggregate will essentially be very finely crystallized quartz whose state of strain probably brings together the more reactive forms of silica (amorphous silica, opal) and the phyllitic minerals (chlorite, vermiculite, and alteration products of feldspars and micas) capable of either reaction or being destroyed in contact with the interstitial cement solution.







Mr D A St John (DSIR, New Zealand) asked how much thaumasite was mixed with the ettringite. Was this very common, he said, or was it just a localised phenomenon.

Dr M Regourd said that they also had found thaumasite mixed with ettringite and she though that in the mini sample they had had such a mixture. However, it was very difficult to identify thaumasite in the presence of ettringite by X-ray diffraction analysis. They could also have identified them by electron microscopy, but again they were not easy to distinguish. These two minerals had been found in some concrete stored in sea water and it was thought that the thaumasite was a product of the decomposition or transformation of ettringite. This transformation had been observed in a very old dyke in Cherbourg, in France, and also in a concrete which had been immersed in the Mediterranean sea. The authors thought that the formation of thaumasite also represented a stage in the evolution of the microstructure.

Mr P Rossouw (SAR & H) asked the author to comment on the relative absence of sodium in the samples investigated.

Dr M Regourd replied that the cement in both samples came from South Africa and said that the samples contained more potassium than sodium. The potassium could also have come from aggregates such as K-feldspar.

Prof U Ludwig (RWTH, Aachen, West Germany) asked whether thaumasite would contribute to the destruction of concrete in the same manner as ettringite.

Dr M Regourd replied that it would and added that they had found a lot of thaumasite in a hydraulic lime from Marseilles in which there was very little alumina because it had consisted mostly of C_2S and calcium hydroxide. Wherever thaumasite had been found all the material had been destroyed around that point.

DISCUSSION

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Dr J Gebauer (Holderbank, Switzerland) asked whether air entrainment would help prevent the gel from moving into the cement paste and expanding, but Dr Regourd replied that she did not know.

Dr P E Grattan-Bellew (NRC, Ottawa, Canada) commented that it had been shown in some of the early papers that air entrainment did seem to help reduce expansion with the alkali-silica reaction. However, with Canadian slowly expanding siliceous aggregates, similar to those found in South Africa, air entrainment had not had any appreciable effect.

Prof S Diamond (Purdue University, USA) said that in the northern hemisphere cases of alkali-silica reaction were almost always complicated by frost, salt application and other factors. However, the reaction occurred even in South Africa where freezing and apparently salt application were not factors. From what Dr Regourd had said the cracks might have opened up by the action of expanding gel, but once open the concrete was subject to a variety of alternative processes as witness the ettringite and thaumasite. In his view such processes might serve to continue the destructive action within the concrete without necessarily having too much to do with the primary alkali-aggregate attack. It was just a consequence of having cracks in the concrete and ion mobility. When the concrete got wet, water poured in through the cracks.

Dr M Regourd reported that they had found several similar cases of expansion in a sample from another country. Ettringite appeared near the aggregate in the cracks and they had not yet determined whether it was the gel or the ettringite, or both which had caused the expansion. What she could say was that some gels were very compact — for example where they had used fumed silica condensate. They had also found a very compact CSH with the fumed silica.

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