

**SESSION 4**

**Reaction Products  
and Petrographic Examination**

**Keynote Lecture**

**Dr. Micheline Regourd-Moranville**

In concretes containing siliceous limestone aggregates Durand and Bérard [4] observed a gel occurring as small veins within the aggregate. The chemical composition measured with a SEM equipped with an energy dispersive spectrometer was also relatively constant in five samples from different structures in the Quebec province of Canada :

51 % SiO<sub>2</sub>, 12 % CaO, 8 % K<sub>2</sub>O, 6 % Na<sub>2</sub>O, 2 % FeO

To prevent the dissolution or alteration of gels methanol was used as a lubricant during the polishing of concrete samples.

Calcium is always present in the gel and more in the textured gel than in the massive gel. As concrete pore solutions are poor in calcium the Ca<sup>2+</sup> ions in the solid reaction products is not obvious. It is known that potassium silicate is formed at pH between 11.3 and 12.1, rather far below the 13.5 measured in concrete. So after Diamond [5] the decreasing in pH can locally dissolve Ca(OH)<sub>2</sub>. Calcium ions would be then able to diffuse to the reaction site and to penetrate into the reacting aggregate. This had been illustrated in opal grains by Kawamura, Takemoto and Hasaba [6]. A depletion of Ca in the cement paste at the interface with the gel has also been observed in the case of sand grains [7] or quartzite [3]. Chatterji, Jensen, Thaulow and Christensen [7] considered that free calcium ions near a reactive grain are the controlling factor in the penetration of Na<sup>+</sup>, K<sup>+</sup> and OH<sup>-</sup> ions in the aggregate and the diffusion of silicate ions out of this aggregate. Davies and Oberholster [8] removed alkalis from reaction products by soaking mortar and concrete specimens in water. Their results on exuded and surface gels or spongy gel suggest that alkali ions Na<sup>+</sup> and K<sup>+</sup> are absorbed rather than chemically bound in alkali silicates (Table 1).

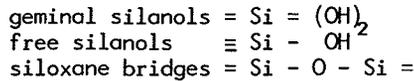
Table 1. Semi-quantitative EDS analysis of massive and spongy gels at the surface of mortar prisms and concrete pats immersed in 1N Na OH solution [8]

Oxide	Massive gel		Spongy gel	
	unwashed	washed 96 hrs	unwashed	washed 8 hrs
% in weight				
SiO <sub>2</sub>	49.4	61.9	18.5	56.6
CaO	28.2	31.9	20.8	43.4
K <sub>2</sub> O	-	-	4.1	-
Na <sub>2</sub> O	22.5	-	56.6	-
Ca/Si atomic	0.61	0.55	1.20	0.82

Based on the view that gels only adsorb Na<sup>+</sup>, K<sup>+</sup> and Ca<sup>2+</sup> ions Natesaiyer and Hover [9] considered that silica surface possesses cation exchange properties. In that way the uranyl ion UO<sub>2</sub><sup>2+</sup> has been chosen for identifying the gel. Under ultraviolet light uranyl ions fluoresce and localise the gel zones. Another example has been done by Poole, Lachlan and Ellis [10] using copper ions from a solution of cuprammonium sulfate. The gel gets stained blue and the amount of copper ions incorporated into the gel is related to the volume of gel formed during the alkali-silica reaction.

The microstructure of gels and the nature of their chemical bonds have been approached through the study of synthetic gels. Synthetic silicate gels

prepared from silica gel in Na OH solution by Liu [11] appeared under Transmission Electron Microscope as globules the size of which varied from 10 to 36.000 Å for SiO<sub>2</sub>/Na<sub>2</sub>O ratio from 1 to 20. Up to SiO<sub>2</sub>/Na<sub>2</sub>O = 1 to 10 globules were dispersed and agglomerated for SiO<sub>2</sub>/Na<sub>2</sub>O = 20. It was expected that a globule was constituted of [SiO<sub>4</sub>] [Si<sub>2</sub>O<sub>7</sub>] [Si<sub>4</sub>O<sub>13</sub>] chains able to condense into domains of a complex gel. Each domain was in this structure environned by Na<sup>+</sup>, OH<sup>-</sup> and H<sup>+</sup>OH<sup>-</sup>. When Ca<sup>2+</sup> ions were present in the solution they were expected to migrate towards the globule which resembled to C-S-H with particle size between 300 and 500 Å. Tuel [12] also studied silica precipitated from basic solutions and appearing as colloidal aggregates of 500 - 5.000 Å size. Using Magic Angle Spinning and Crossed Polarisation Nuclear Magnetic Resonance, three species were identified at the silica surface. They were :



NMR was able to distinguish between geminal and free silanols that IR could not do. Precipitated silicas were porous with many surface irregularities. If the silanol distribution was uniform there were more protons concentrated inside micropores corresponding to clusters. On the contrary polarisable siloxanes were located in a superficial layer relatively homogeneous. NMR appears as a promising technique for elucidating the AAR mechanism and the composition of reaction products particularly if silanols are considered as the first species involved in the reaction between silica and alkalis [13].

Massive gel (fig. 1) is usually found in the concrete of every structure altered by alkali-silica reaction [14]. Gels can also appeared grainy, spongy, foliated (fig. 2). On massive gels have been observed calcite microcrystals [1]. Ršumović and Cmijanič [15] considered that carbonate is an intermediate product as observed by Infra Red Spectroscopy. The gel of alkali silicate is partially or totally destroyed by calcium silicate and calcium carbonate. Iiyama, Kusano and Tokunaga [16] revealed the AAR "histological" evolution with carbonate as a late product and a consequence of a secondary deterioration.

2.1.2. Crystals occur as roses, lamellas, fibers, needles and filaments (fig. 3). Compared to massive gels they can occasionally contain Al, Fe, Na (Table 2)

Table 2. Composition of some crystals by EDS [14]

Oxide % in weight	Rose crystals			Needle crystals		Blade crystals
SiO <sub>2</sub>	68.6	71.1	76.9	63.7	60.3	71.2
Al <sub>2</sub> O <sub>3</sub>	-	-	4.6	-	-	-
CaO	18.2	17.1	9.4	8.9	1.1	11.6
Na <sub>2</sub> O	-	-	-	13.1	8.4	-
K <sub>2</sub> O	13.2	11.8	9.1	14.3	18.2	17.2

Bérubé and Fournier [2] found in the presence of quartzite a relatively constant composition of rose crystals with a large amount of alumina may be from feldspars or chlorites.

56 - 63 % Si<sub>2</sub>O<sub>3</sub> , 20 - 27 % Al<sub>2</sub>O<sub>3</sub> , 8 - 11 % K<sub>2</sub>O, 6 - 8 % CaO  
 XRD patterns of crystals exhibit numerous peaks with the major d. values

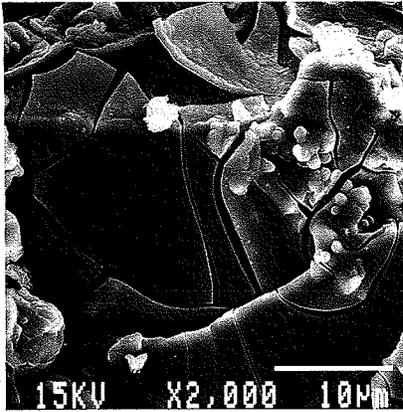


Fig. 1 Massive gel

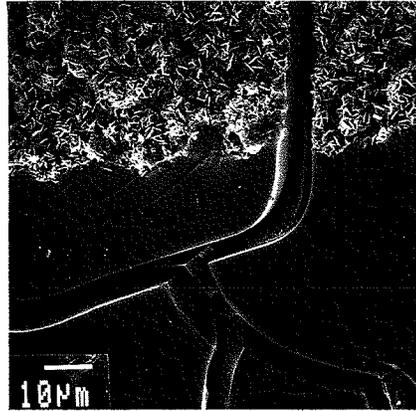


Fig. 2 Textured gel on massive gel



Fig. 3 Lamellar and rosette - like crystals

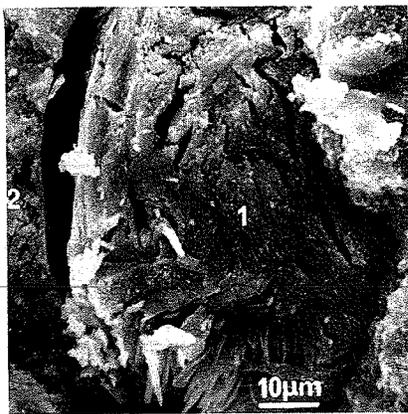
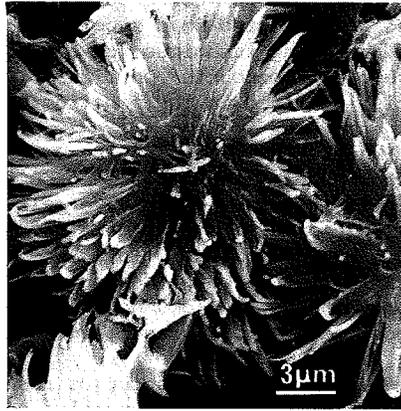


Fig. 4 Massive ettringite (1) in a pore of the cement paste (2)

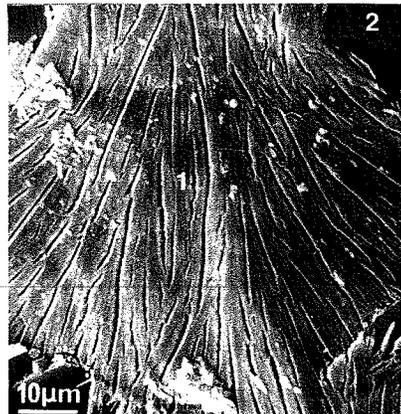


Fig. 5 Ettringite (1) on the surface of a quartzite aggregate (2)

at 12 Å, 8 Å, 3 Å. Davies and Oberholster [14], Shayan and Lancucki [17] tried to isolate the coexisting silicates by heat treatment. However differences in the loss of moisture lead to different XRD spacings. Okenite  $\text{Ca}_{10}\text{Si}_8\text{O}_{46}\cdot 18\text{H}_2\text{O}$  and zeolites  $(\text{Na Al SiO}_4)_{12}\cdot 2\text{H}_2\text{O}$  or  $(\text{Ca}_6\text{ Al SiO}_4)_{12}\cdot 30\text{H}_2\text{O}$  have been suggested. Mullick and Samuel [18] also found different silicates of composition  $\text{Na Si}_7\text{O}_{13}(\text{OH})\cdot 3\text{H}_2\text{O}$  and  $\text{K}_2\text{Ca}(\text{SiO}_4)_2\cdot \text{H}_2\text{O}$  with quartzite,  $(\text{K}_2\text{Na}_2\text{Ca})_{16}\text{Si}_{32}\text{H}_2\text{O}$  with granitic aggregates.

When washed in distilled water but the K rosette phase excepted crystalline phases found as internal reaction products in mortars and concretes exchange their alkali for calcium (Table 3).

Table 3. Semi-quantitative EDS analysis of the rosette crystals found in cracks and air voids of mortars immersed in alkaline solutions, before and after immersion in distilled water [8]

Oxide % in weight	1 N Na OH solution			1 N KOH solution		
	unwashed	washed 2 hr	washed 84 hr	unwashed	washed 44 hr	washed 382 hr
$\text{SiO}_2$	71.7	73.6	71.0	63.5	67.3	67.0
$\text{CaO}$	12.4	17.8	26.5	20.7	22.6	21.7
$\text{Na}_2\text{O}$	14.8	8.6	1.7	-	-	-
$\text{K}_2\text{O}$	0.4	-	0.8	15.8	10.1	11.3
Ca/Si atomic	0.18	0.26	0.40	0.35	0.36	0.35

So it seems that Ca ions substitute for alkalis probably by an ion-exchange reaction, the source of calcium could be  $\text{Ca}(\text{OH})_2$ . The Ca - bearing compound is also after Kodama and Nishino [19] a key in explaining the AAR mechanism which is not as simple as a reaction between alkalis in the pore fluids and reactive silica [8]. The substitution of Ca<sup>2+</sup> for alkalis leads to the formation of alkali hydroxides. Until the disappearance of  $\text{Ca}(\text{OH})_2$  the regenerated alkali hydroxides are able to react with silica.

2.1.3. Secondary products. In addition to carbonates or hydrocarbonates a secondary deterioration of concrete has been related to the formation of ettringite [11 - 20 - 21]. Jones and Poole [20] and Jones [21] have clearly observed large quantities of ettringite which replaced alkali silica gel. Ettringite can occur infilling voids of the cement paste (fig. 4) or on the aggregate surface (fig. 5).

## 2.2. Alkali-Carbonate Reaction : Dedolomitisation

In their study of reactive carbonates from Kingston, Canada, Tang, Liu and Han [22] observed by optical microscopy and SEM/EDAX and on Ar ion beam thinned sections a layer of oriented crystals of brucite surrounding dolomite rhombs, after immersion in a KOH solution. The reactive carbonate was constituted of rhombic crystals of dolomite distributed in a clay matrix mixed with fine grained calcite. The  $\text{Mg}(\text{OH})_2$  layer of 2 µm thickness corresponded to the diffusion of  $\text{M}^+$ ,  $\text{OH}^-$  ions and water molecules through the clay matrix towards the

dolomite crystals. The expansion was related to the growth and rearrangement of the reaction product at the surface of dolomite crystals and in a restricted area.

Grattan-Bellew and Lefebvre [23] did not find any dedolomitisation in concretes prepared with the Kingston rock. However these concretes had already expanded. In this case the dedolomitisation could be considered as a secondary effect.

In fact in many cases the alkali-silica reaction is also observed with carbonate rocks. In Canada three types of reactive silica have been found in limestone aggregates [2 - 4 - 24]. They are :

1. silicified fossils : sponge needles, brachiopods
2. diffused silica appearing as a fine network at the boundary of carbonate grains
3. nodules or veins of cherts.

### 3. PETROGRAPHIC EXAMINATION

Petrography is used for the diagnosis of possible AAR in damaged concretes and characterisation of reactive aggregates. Thin sections and polished sections examined under optical microscopy and fractured surfaces observed under scanning electron microscope are able to characterise AAR products and mineral distribution in aggregates [25]. A very useful report of a working party on the diagnosis of alkali-silica reaction has been published by the British Cement Association [26].

#### 3.1. Petrography of damaged concrete

After a careful site inspection including records of cracking, discoloration, efflorescence, exudation, pop-outs, the sampling should include cores and sometimes powdered drillings [26]. Samples should be protected against desiccation and carbonation.

3.1.1. Thin sections The petrographer is able to describe the cement paste and its interface with aggregates, the rock type of aggregates, the presence of gels and the map cracking. Gels frequently exhibit drying shrinkage cracks. They are located either around aggregates or in cracks and voids. The cement paste-aggregate interface and filled cracks darken by gel impregnation. Versus time gels can get structured and birefringent. In fluorescence impregnated thin sections flint particles can appear almost fully dissolved and replaced by a swelling gel with cracks in and around aggregates and with an extension into the cement paste [27].

3.1.2. Polished sections. Reaction products can be seen under light microscope around aggregates in a dark rim (siliceous aggregates) or a white rim (dolomitic aggregates). More details are offered by electron optical methods like SEM/EDS and AEM giving also the elemental composition of gels and crystals [3 - 14 - 17 - 18 - 19].

3.1.3. Fracture surfaces. The microstructure of gels and crystals has been illustrated in fig. 1, 2, 3. Since the Ottawa conference, reaction products were observed in core samples from concrete structures like pillar girders, retaining walls, floor paving of houses in Japan [16], a bridge in Australia [17], a dam in India [18], viaducts and road bridges in Canada [22] and dams in USA [29]. Three types of deleterious AAR mechanisms have been illustrated in canadian concrete [24] (i) a peripheral zone in the case of massive granitic

rock, (ii) a bulk swelling of the Postdam orthoquartzite, (iii) veinules of silica gel within the Trenton limestone.

There is not yet a correlation between the amount of gels and their expansive forces leading to the deterioration of concrete. Some UK damages corresponded to relatively small amounts of gel [26]. It seems also difficult to differentiate between a reaction leading to a low amount of gel and the early stage of reaction. So it is important to know the age of the concrete structure before concluding.

### 3.2. Petrography of aggregates

Microscopic observations of damaged concretes also characterise aggregates which have reacted with alkalis. They are numerous and several lists have been published including the type of rock (metamorphic, sedimentary, volcanic, igneous) and potentially alkali-silica reactive components (opal, chalcedony, glass, cristobalite, crypto-crystalline quartz). Some rocks which have reacted in a country may not react in another one [26]. This is the case for hornfels respectively in South Africa [3] and United Kingdom [26]. The importance of petrography in the assessment of aggregates has been emphasized by Sims [30] who described a new procedure adapted to UK aggregates (flints and cherts mostly) based on the separation into appropriate petrographic groups. Andesites, rhyolites, cherts and siliceous shales are reactive rocks in Japan. Wakisaba, Moriya and Kawano [31] are able to estimate the alkali-reactivity of these rocks using assemblage and amount of minerals. Felsic minerals (high silica) and glasses with little bridging oxygen are highly reactive. Mafic minerals (low silica) are slightly reactive. Japanese volcanic rocks have also been classified into four zones by Katayama and Kaneshige [32]. This classification based on mineral assemblages includes (1) the unaltered zone (2) the slightly zone (3) the smectite zone (4) the chlorite zone. The potential reactivity with alkalis of these rocks has been found decreasing from the first zone to the last one.

Beside reactive silicas polycrystalline aggregates can contain altered minerals like feldspars or clays able to release alkalis and to swell. A petrographic examination of aggregates will identify these minerals and inclusions either under polarising microscope or scanning electron microscope.

3.2.1. Thin sections examined under polarising light reveal in siliceous aggregates which have reacted in concrete, quartz with undulatory extinction, plagioclase and mica (fig. 6), altered striated feldspars (fig. 7) and chalcedony (fig. 8). Biotite can be chloritised [33]. Plagioclases sometimes contain inclusions of micas (damouritisation and seritisation) [34]. Microcrystalline quartz is the main mineral of novaculite and chert. It is very often associated with strained quartz. Gratton-Bellew [35] concluded from a study of granitic aggregates that microcrystalline quartz was more reactive than quartz grains with high undulatory extinction angles. Vivian [36] also emphasized the importance of the crypto-crystalline silica content of aggregates in their potential reactivity. Opal in sandstones from Argentina have highly reacted when their content was close to the pessimum value [37]. Cristobalite in its  $\alpha$  variety and volcanic glass in a bronzite andesite are responsible for the damage of the Hanshin Expressway in Japan [38]. In deteriorated canadian railway bridge piers reactive orthoquartzite sandstone was constituted of quartz grains of 0.33 mm size cemented by a fine siliceous matrix [39].

Some carbonate rocks also contain reactive silica as silicified fossils [24 - 40 - 41] and intraclasts in a micrite matrix. The amount of silica is between 7 and 15 % [2].

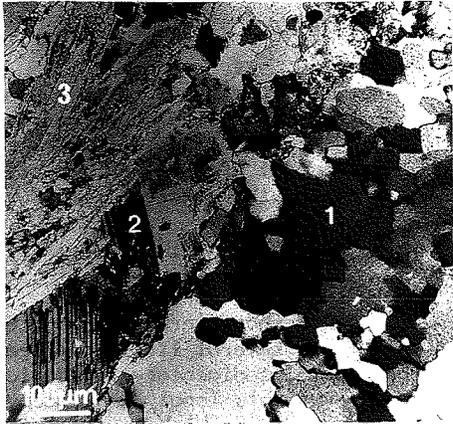


Fig. 6 Thin section of a granite :  
1 = quartz with undulatory extinction  
2 = plagioclase, 3 = mica

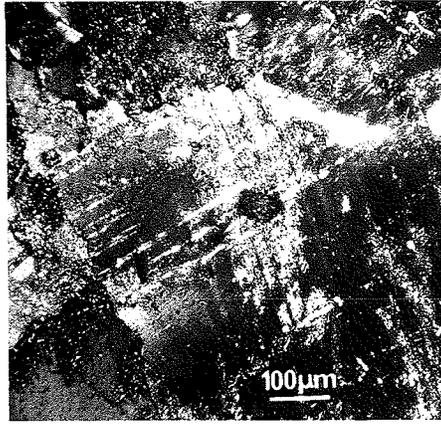


Fig. 7 Thin section of a granite with  
altered striated feldspar and phyllitic  
deposits

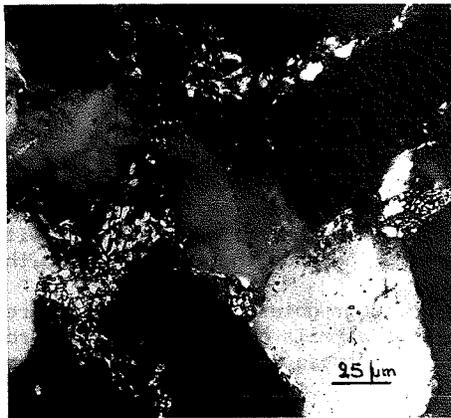


Fig. 8 Thin section of a quartzite :  
fibrous chalcedony between quartz  
grains

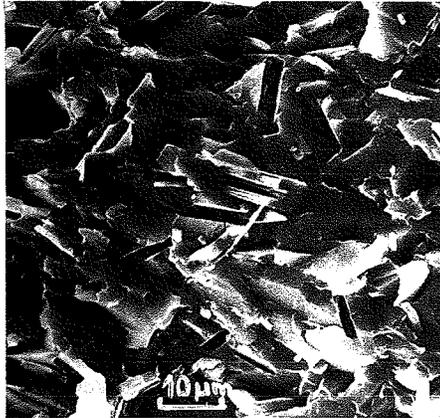


Fig. 9 SEM. Seritisation in a granodiorite :  
mica spangles on a feldspar  
grains

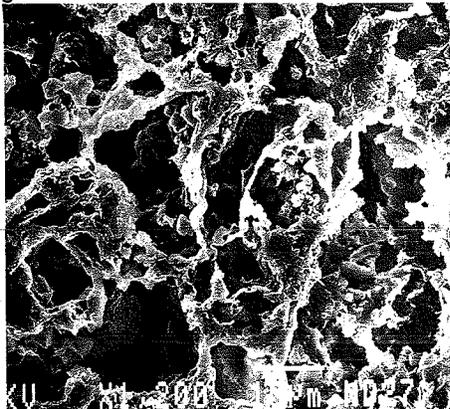


Fig. 10 SEM. Limestone aggregate silica  
matrix after dissolution of carbonate

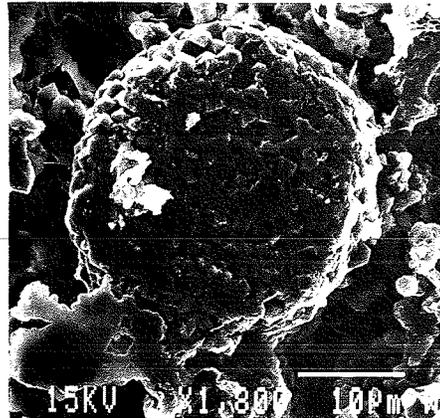


Fig. 11 SEM. Limestone aggregate  
Raspberry like - grains of pyrite in  
the silica matrix

3.2.2. Fracture surfaces observed under SEM give details on the roughness, porosity, alteration and grain boundaries of the different minerals. The sericitisation is identified in an altered feldspar (fig. 9). Clay inclusions are visible on sand grains and are identified by their elemental analysis. Secondary minerals can be amphibole hornblend, pyroxene, epidote, ilmenite, glauconite and pyrite.

In limestone aggregates the fine silicate matrix between microsparite crystals [2] has an alveolar structure after the dissolution of carbonate in acetic or chlorhydric acid (fig. 10). Pyrite has also been identified as raspberry-like grains (fig. 11).

3.2.3. Polished sections as reactivity indicators. The SEM examination of polished sections of aggregates immersed in alkaline solutions at 80°C for three days can be an helpful accelerated test [25]. After the treatment quartzite grains which react slowly exhibit a corroded surface. A foliated deposit rich in silica and containing calcium has been observed on a granite and micas were exfoliated.

3.2.4. Infra red spectroscopy as reactivity indicator. Hirche [42] evaluated the amount of silanol groups in siliceous minerals by IR spectroscopy through the OH vibrations. Opal, flint, sandstone, glass, all reactive with alkalis showed larger absorption than well crystalline quartz. Bachiorrini [43] applied a "disorder coefficient" Cd to different siliceous minerals through the broadening of  $\nu_1$  bands corresponding to  $\text{SiO}_4$  groups. Using as standards some aggregates which have not reacted with alkalis three domains have been tentatively fixed for the potential reactivity i.e.

- Cd < 120 : aggregates are not reactive
- 120 < Cd < 200 : aggregates are slowly reactive
- 200 < Cd < 300 : aggregates are rapidly reactive
- Cd > 300 : aggregates are meanly reactive

This method has been tested on deteriorated concretes from different countries [44]. Aggregates with Cd < 200 contained strained quartz. Those with 200 < Cd < 300 contained fine chert and those with Cd > 300 contained very fine chert or chalcidony or vitreous phases, altered micas and feldspars. When Cd was high (Cd > 450) the aggregate was getting like a pouzzolanic material. Silica microinclusions in carbonate rocks are also able to react with alkalis. There is a close relation between the time of cracks appearance in concrete and their coefficient of disorder [45].

#### 4. DISCUSSION

Since the last conference in Ottawa in 1986 most of AAR cases which have been published are related to the alkali-silica reaction even with dolomitic limestone aggregates. Reaction products observed visually (exudation, white rims around aggregates) or under microscopy occur as gels and crystals of alkaline silicates. Their composition and structure are not yet known precisely. Their elemental composition has been approached by energy dispersive spectrometry on wavelength dispersive spectrometry on the solid skeleton after the free water removing and has to be considered as a semi-quantitative determination [37]. Gels appear close to silica gel used as a chromatographic support [9]. Silica surfaces are sites of cation exchanges and  $\text{Na}^+$  or  $\text{K}^+$  previously adsorbed on the gel can be easily replaced by  $\text{Ca}^{2+}$  [8]  $\text{Cu}^{2+}$  [10] or  $\text{UO}_2^{2+}$  [9]. After immersion in distilled water, exuded and surface gels loose their alkalis and get a tobermorite-like structure [8]. Versus time amorphous and massive gels transform into textured silicates some close to C-S-H but lower in calcium compared

to Portland C-S-H. If the role of calcium ions has been found essential for the development of reaction products [5 - 6 - 7 - 8] it is not yet fully elucidated. The crystal structure of roses and lamellae has also been approached through the XRD patterns. Many silicates have been suggested, they all contain alkalis, calcium and hydroxide ions and water molecules. Some crystals were found different from okenite [17] and were not surely identified after a heat treatment. So the composition and structure need more research. Maybe the solid and proton nuclear magnetic resonance and transmission electron microscopy are techniques to be used for future work.

No clear correlation has been found between the amount of gel and expansive forces responsible for the deterioration of concrete. This is also a way to be explored. Quantitative analysis of cracks density may be by image analyser after a careful preparation of samples should be undertaken.

Petrographic examination of aggregates appears very important. Disordered silicas as determined by IR spectroscopy [43], silicified fossils or diffused silica in limestone aggregates [2 - 4] altered feldspars, chlorites, pyrite are all minerals involved in the reaction. Secondary reactions as carbonation of the cement paste and formation of ettringite are complementary types of deteriorations of concrete.

In conclusion the alkali-aggregate reaction is very complex and not a simple reaction between silica and alkalis from the pore solution. In some conditions the reaction is not far from a pozzolanic reaction (content of opal largely higher than the pessimum value) giving hydrates close to that of cement paste, C-S-H or tobermorite (cation exchanges between calcium and sodium or potassium). These conditions have to be precised. We urgently need mathematical and mechanistic models for each type of alkali-aggregate reaction and possible preventive measures.

## 5. ACKNOWLEDGMENTS

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