

# Infrared Spectroscopy in the Evaluation of Aggregates in A.S.R. Deteriorated Concretes from Many Parts of the World: Comparison with Other Methods

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## ABSTRACT

To verify the suitability of infrared spectroscopy for the evaluation of microstructural disorder in concrete aggregates and, to determine their potential alkali-reactivity. Fifteen concrete samples drawn in several countries from A.S.R. deteriorated structures were examined. The optical microscopy has confirmed the close relationship between disordered aggregates and their alkali-reactivity. The sensitivity of infrared spectroscopy to microstructural disorder variations of the samples was exploited and a disorder coefficient ( $C_D$ ) obtained from a simple graphic elaboration of I.R. spectrum was defined. On the ground of collected data, a mutual relation between the  $C_D$  values and the alkali-reactivity of aggregates was determined.

## 1. INTRODUCTION

Hirche (1975) proposed the use of IR spectroscopy to evaluate the potential reactivity of aggregates with the alkalis in cement.

This technique is based on the correlation between microcrystalline disorder and reactivity and it was used, in a modified form, to study the aggregates of concretes, degraded by ASR, and to verify if this method can provide information on the potential reactivity of siliceous aggregates.

Petrographic examination was used as a standard reference test.

Subsidiary information was obtained by means of microscopic and X-ray analyses.

## 2. MATERIALS AND METHODS

The information on the examined concrete samples(\*) and the results of the analyses are summarised in Table 1. Optical analyses were carried out on each concrete, with the stereomicroscope and on thin section, to identify the petrographic nature of the aggregates and the type of alteration which occurred at the cement paste-aggregate interface.

When possible X-ray analysis was carried out on the reaction products found in the concrete pores.

Afterwards, the aggregates of each sample were carefully separated from the mortar, washed with dilute HCl and water and ground to a grain size  $< 40 \mu\text{m}$ . In order to calculate the «coefficient of disorder» ( $C_D$ ), I.R. analysis was performed on the powders following a protocol previously described (Bachiorrini et al., 1986).

For heterogeneous aggregates, this protocol was modified (Bachiorrini 1987) assuming as transmittance zero that of the  $\nu_3$  band ( $\sim 1100 \text{ cm}^{-1}$ ) of the  $\text{SiO}_4$  groups;  $\nu_1$  ( $\sim 800 \text{ cm}^{-1}$ ) was assumed as the analytical band (Figure 1).

(\*) We are grateful to the scientists who supplied the samples and the information concerning them.

Table 1 - Source and characteristics of the samples

Samples ( $\Delta t$ )	Source	Cement Kg/m <sup>3</sup>	% Na <sub>2</sub> O eq.	Environment Conditions		C <sub>d</sub>	Petrographic examination of aggregates
				°C	R.H. (%)		
No. 1 (7)	Italy B,i	300	1.1	10/25	100	365	Essentially calcareous very-fine grained; strained quartz, chert
No. 2 (1)	Italy B,s	400	0.9	0/40	90/100	260	Essentially calcareous, chert, strained quartz, chalcedony (traces)
No. 3 (5)	G.B. B,i	530	1.2	0/30	—	340	Gneiss, strained quartz, feldspars, micas (c); chert, chalcedony (f)
No. 4 (8)	G.B. b,i	400	1.1	- 5/25	60/100	365	Calcareous (c); chert, chalcedony, strained quartz (f)
No. 5 (3)	W. Germ. B,s	340	0.9	- 20/30	65	225	Quartzose, feldspars, strained quartz, chert, chalcedony, limestone (traces)
No. 6 (-)	W. Germ. d,s	300	—	- 20/30	65/100	340	Strained quartz very-fine grained, feldspars, micas, chert very fine grained, chalcedony
No. 7 (3)	Sweden B,s	280	0.42	18/20	75/95	285	Quartzose, feldspars, chert (traces) (c); strained quartz and carbonates (f)
No. 8 (6)	Japan b,i	350	—	0/30	30/90	435	Effusive, glassy, partially recrystallized, chalcedony (c); strained quartz and feldspars (f)
No. 9 (10)	USA d,i	400	—	—	—	160	Strained quartz, quartzites, feldspars
No. 10 (0.5)	Austral. b,s	—	1.1	10/40	50/95	245	Effusive with strained quartz and feldspars
No. 11 (0.5)	Austral. d,s	—	1.1	0/40	70/90	235	Quartzose with strained quartz, micas and feldspars
No. 12 (4)	East Afr. b,s	375	1.1	20/40	60/90	340	Granite with very strained quartz, feldspars and micas
No. 13 (20)	Sout Afr. b,s	—	—	0/25	—	185	Quartzose with strained quartz, micas and feldspars

b = bridge; d = dam; B = building; s = surface; i = inside

f = in fine fraction c = in coarse fraction

( $\Delta t$ ) = time elapsed between construction and ASR appearance in years

### 3. RESULTS AND DISCUSSION

In the examined concretes it was observed that the alteration affected especially cherts and strained quartz and, to a lesser extent, feldspars.

In general it can be affirmed that the cherts showed different degrees of alteration. Those with a very fine grain or with zones of fibrous structure, typical of chalcedony, showed a strong dissolution and sometimes more or less complete substitution of the siliceous material by an ultrafine carbonate-like material of new formation (Figure 2).

For coarse cherts, however, the alteration was observed at the interface with the cement paste, with the formation of an ultrafine material like that described above; the grain boundaries in that case appeared either strongly indented or deeply lobate.

Quartz alteration occurred above all where the quartz showed a marked undulatory extinction and therefore greater crystalline disorder. These observations confirm the correlation between the microstructural disorder of the aggregates and the extent of alteration by ASR (Cembureau 1977).

In addition, pores in the concretes appeared to be partially filled by a whitish material, which in some cases was of an amorphous, siliceous nature, and in others of a siliceous-calcareous nature (Figure 3). This material was also found along some cracks in the cement paste and in some aggregates and can be attributed to ASR.

In some samples (nos. 6, 9, 11, 13) optical and X-ray analyses have also shown the presence of ettringite, in the concrete pores, confirming the possible connection between sulphate alteration and ASR (Pettifer & Nixon, 1980).

As regards the relationship between the  $C_d$  values and petrographic examination, aggregates with  $C_d$  lower than 200 were found to contain reactive phases, like strained quartz (Poitevin & Regourd, 1983). Those with  $C_d$  between 200 and 300 contain phases with higher reactivity (for example more or less fine chert) as well as strained quartz. Finally, those with  $C_d$  greater than 300 contain chert very fine-grained or with zones of chalcedoneous structure or vitreous phases, altered micas and feldspars in greater quantities.

It seems therefore that as the microstructural disorder  $C_d$  of the aggregate, increases, its reactivity towards alkalis increases too.

When the disorder increases too much ( $C_d > 300$ ) the aggregate begins to show a behaviour comparable to that of pozzolans ( $C_d > 450$ ) and therefore the consequences of ASR are minimised.

The time, elapsed between use of the concrete and the appearance of distress due to AAR, is assumed as an index of the aggregate reactivity. A curve with a maximum was obtained by plotting this time versus the  $C_d$  values (Figure 4). This curve is like those presented by Hobbs (1980) and the more general one elaborated by Nilsson and Peterson (1983).

It is obviously not possible to find a rigorous correlation between the  $C_d$  values and the times of the appearance of alteration since the various concretes had different alkali and cement contents and were exposed to quite different weather conditions.

It was found, however, that the equivalent  $\text{Na}_2\text{O}$  content in the concretes, as shown in Table 1, is higher than  $3 \text{ Kg/m}^3$  except in the case of sample no. 7 (Report of a Working Party, 1983).

### 4. CONCLUSIONS

It is clear that the foregoing statements are based on a limited experience. Experiments should be carried out on a much higher number of samples so that the above-mentioned correlations can have a wider statistical basis.

However, from the available results, it seems possible to indicate reliably that aggregates that have  $C_d < 120$  are not reactive (Bachiorrini, 1987); reactive in a long time those with  $C_d$  between 120 and 200; reactive in a short time those with  $C_d$  between 200 and 300; reactive in a medium time those aggregates with  $C_d > 300$ .

If more extensive researches should confirm the validity of this method, a rapid and simple technique for forecasting the reactivity of siliceous aggregates and their behaviour at work should be available.

### 5. REFERENCES

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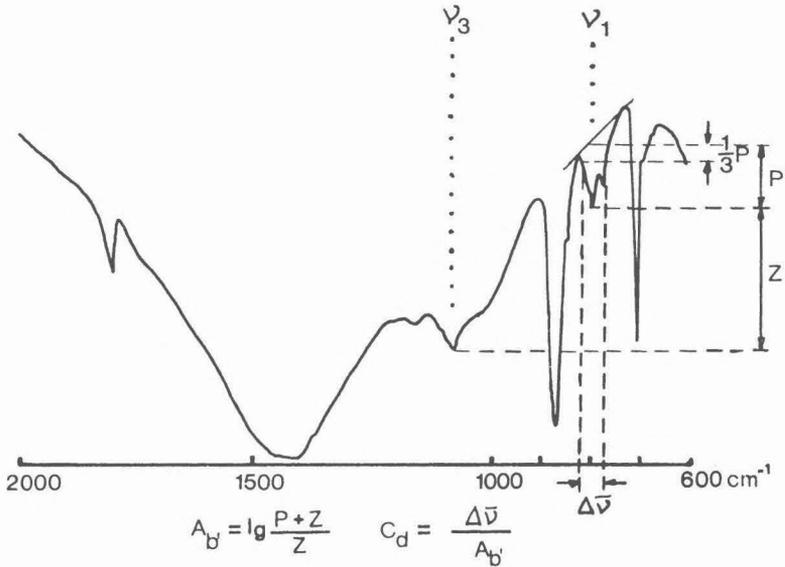


Figure 1 - Example of  $C_d$  calculation from the spectrum of an heterogeneous aggregate (sample no. 2)

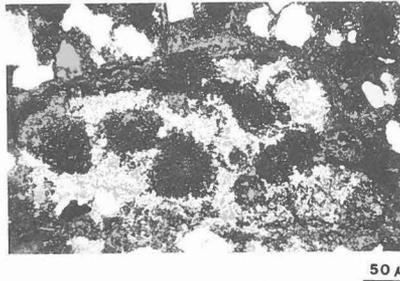


Figure 2 - Grain of very fine-grained chert with replacement of a large amount of chert with carbonate of new formation (sample no. 3) (Nicol X).

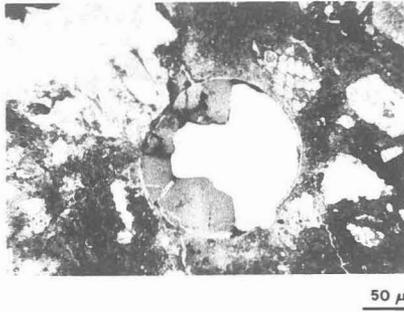


Figure 3 - A Pore in the cement paste partially filled by ASR products (sample no. 1) (polarizer).

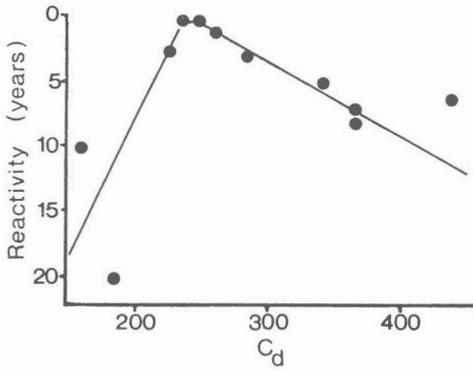


Figure 4 - Reactivity of the aggregates versus their  $C_d$  values.