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Rapid evaluation of the threshold alkali level for alkali-reactive siliceous aggregates in concrete

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Abstract

A modified version of the ultra-accelerated concrete prism expansion test in alkaline solutions at 150°C (testing concrete mixes at varying alkali content of concrete) was investigated in order to ascertain the reliability of this test method for evaluating the threshold alkali levels (TAL) of alkali-reactive siliceous aggregates in concrete. Five natural sands of known field performance were tested and their TAL values determined by the ultra-accelerated test were compared with those obtained from concrete prism expansion tests at 38°C and 100% RH. A correlation between the threshold alkali level of the sands and their microstructural disorder coefficient, Cd, as measured by infrared spectroscopy, was also attempted. The modified ultra-accelerated concrete prism test proved to be a rapid and reliable method for evaluating the threshold alkali level of reactive siliceous aggregates in concrete, provided that an expansion limit of 0.11% at 3 weeks was taken as the reactivity judgement criterion for this test. The threshold alkali level proved to be an appropriate reactivity parameter for the selection of alkali-reactive siliceous aggregates. Also, the existence of a straight-line relationship between TAL and Cd suggested the possibility of developing a new test methodology for estimating TAL, based on Cd measurements by infrared spectroscopy. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Alkali-reactive siliceous aggregates; Concrete; Threshold alkali level

1. Introduction

The use of alkali-reactive siliceous aggregates in concrete can cause severe damage to concrete structures due to the development of the expansive alkali-silica reaction (ASR).

Visible cracking in the affected structures is observed typically more than 10 years after construction. In severe exposure conditions, however, structures may be damaged in much shorter times.

Recommended preventive measures for the use of ASR-susceptible aggregates in concrete are: (1) limiting the alkali content of the concrete by reducing the cement content and/or by using a low-alkali Portland cement, and (2) replacing part of the Portland cement with supplementary cementing materials (SCM), such as pozzolan, fly ash, ground granulated blast furnace slag, condensed silica fume, provided the selected SCM-cement combination is proven “safe” by long-term expansion testing.

In many countries, a safe alkali content of the concrete, derived from concrete mix materials and expressed in terms of kg Na_2O equivalent (Na_2O_{eq})/m^3 of concrete, has been recommended or prescribed for the use of potentially reactive aggregates. The alkali limit is commonly in the range 2 to 4.5 kg Na_2O_{eq}/m^3, although, in some countries (for example, France, Ireland, and South Africa), the actual value varies depending on the nature of the reactive aggregate. [1]

Undoubtedly, an alkali level of 2 to 4.5 kg Na_2O_{eq}/m^3 could be insufficient to cause expansive reactions for certain types of aggregates [2], whereas it could be too restrictive for other varieties of aggregates [3], since the nature of the aggregate greatly affects the value of the threshold alkali level (TAL) for ASR expansion, i.e., the minimum alkali level necessary to promote deleterious expansion in concrete.

Furthermore, it must be considered that wetting/drying cycles, humidity gradients, freezing/thawing cycles and electric currents can cause migration and

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concentration of alkali ions in concrete [4–7]. Enhancement of the alkali concentration in concrete could also arise when concrete structures are exposed to external sources of alkali (for example, deicing salts).

As a consequence of the migration and concentration of alkali ions, alkali-aggregate reactions may be accelerated and localized deleterious reactions may be initiated even if the native alkali content of the concrete is below the TAL of the particular aggregate.

Therefore, if the exposure conditions of the particular concrete structure are likely to lead to increased alkali concentration, the design of the safe alkali content of the concrete should consider the threshold alkali level for the specific aggregate to be used, the alkali provided by the concrete mixture materials (native alkali content), and the enhancement of the alkali concentration that could be derived from the exposure conditions; the last being estimated from past field experiences.

If the safe alkali content of the concrete, thus calculated, is incompatible with the engineering properties required for the specific structure, then the particular aggregate has to be rejected and replaced with an aggregate having a higher threshold alkali level, or appropriate SCM-cement combinations have to be used.

Such a design approach requires the preliminary knowledge of the threshold alkali levels for all the aggregates available for the construction project.

At present, no standard test method exists for determining the TAL values for potentially reactive aggregates in concrete. However, according to Stark [8] and Ross and Shayan, [9] the alkali tolerance of aggregates could be estimated by the accelerated mortar-bar expansion test at 80°C using NaOH solutions of varying concentrations.

As reported in our recent paper [3], the TAL value for a specific aggregate in concrete may be determined from the results of concrete prism expansion tests at 38°C and 100% RH, when these tests are performed using the same concrete mix proportions, storage conditions and expansion limits as those established by the Canadian standard CSA A23.2-14A-M94 test method [10], but the alkali content of the concrete is varied through addition of NaOH to the mix water (in the standard test the alkali content of the concrete is fixed at 5.25 kg Na_2O_{eq}/m^3).

Unfortunately, this method requires a long test period (12 months), which is in contrast with the short lead time for many construction projects.

In 1994, Criaud et al. [11] developed an ultra-accelerated concrete prism expansion test which may be used to reliably predict the risks of ASR expansion for actual field concrete compositions within a few weeks.

This test method consists of immersing concrete prisms (70 × 70 × 280 mm) in an alkaline solution at 150°C for 3 weeks and monitoring their length changes periodically. The composition of the alkaline solution is adapted to match as closely as possible the composition of the interstitial pore solution within the concrete. A provisional expansion limit of 0.11% at 3 weeks was proposed by the above Authors as the reactivity judgment criterion.

Obviously, no particular alkali content of the concrete was established for the ultra-accelerated concrete prism test and, consequently, this test, at least in its original version, cannot be used for the ASR assessment of siliceous aggregates.

In the present study, a modification of the ultra-accelerated test was made which consisted essentially of testing concrete mixes incorporating a specific aggregate at varying alkali content of concrete. The modified ultra-accelerated concrete prism test was investigated in order to ascertain its reliability for evaluating the threshold alkali level of a number of Italian natural siliceous sands of known field performance. The TAL values obtained from concrete expansion tests at 38°C and 100% RH [3] were used for comparative purposes.

2. Materials and methods

Five natural sands, designated by letters *A* to *E*, were tested in this study. These sands have already been characterized for ASR in previous works [3,12] by using test methods based either on the aggregate or on cement-aggregate combinations.

Table 1 gives the petrographic characteristics of the sands tested together with their alkali-reactivity status (non-reactive: NR; potentially reactive: PR), established by field service records, results of infrared spectroscopy technique [13] (determination of the microstructural disorder coefficient, Cd, of the silica lattice of the aggregate through the broadening of v_1 bands corresponding to SiO_4 groups in the IR spectrum), ASTM C1260 mortar-bar expansion test in a 1M NaOH solution at 80°C, and Canadian standard CSA A23.2-14A-M94 test method. Detailed information relating to the materials and test procedures employed for obtaining the data of Table 1 is given in the previous papers [3,12].

All the sands, except *D*, have shown reaction in service (Table 1) and their expansive behaviour was typical of slowly reactive aggregates. In particular, all the concrete structures incorporating sand *C* have exhibited damage due to ASR while no sign of ASR was observed in some structures incorporating sand *A*, *B* or *E*. Therefore, on the basis of field performance data, sand *C* was considered to be more reactive than sands *A*, *B* and *E*. The petrographic examination of the reactive sands showed that their reactivity could be attributed to the presence of significant amounts of rhyolitic rocks, containing strained quartz, or flint.

Table 1
Test results for alkali-silica reactivity of sands investigated

		Aggregate				
		A	*B*	*C*	*D*	*E*
Petrographic examination		carbonate rocks with flint and traces of chalcedony	same source as *A*, but with crushed particles	rhyolitic rocks	dolomitic and gneissic rocks with limestone, quartz, and quartzite	siliceous sand with quartzite, flint, and stressed quartz
Field service record		PR	PR	PR	NR	PR
IR method	Cd (cm^{-1})	134	144	192	100	143
Judgment criteria:						
$Cd \leqslant 120$: not reactive	Diagnosis	PR	PR	PR	NR	PR
$120 < Cd \leqslant 200$: slowly reactive		(slowly reactive)	(slowly reactive)	(slowly reactive)		(slowly reactive)
$200 < Cd \leqslant 300$: rapidly reactive						
$Cd > 300$: pozzolanic behaviour						
ASTM C1260 (a)	*E*%	0.21	0.26	0.41	0.07	0.37
Interpretation of results: (b)	at 14 days					
E% at 14 days < 0.10%: not reactive	Diagnosis	PR	PR	PR	NR	PR
E% at 14 days > 0.20%: potentially reactive						
CSA A23.2-14A-M94	*E*%	0.015	0.017	0.075	0.013	0.019
Judgment criteria	at 1 yr					
E% < 0.04% at 1 yr: not reactive	Diagnosis	NR	NR	PR	NR	NR
E% $\geqslant$ 0.04% at 1 yr: potentially reactive						

[a] A Portland cement (phase composition: $C_3S = 49.43\%$; $C_2S = 26.48\%$; $C_3A = 7.01\%$; $C_4AF = 9.95\%$) with a native alkali content of 1.15% Na_2O_{eq} and a Blaine specific surface area of 500 m^2/kg was used in this test.

[b] Not conclusive results for 14-day expansions (*E*%) between 0.10% and 0.20%.

Concrete mixes were prepared using each of the sands tested, a non-reactive coarse aggregate (from the same source as sand *D*) in a standard gradation, a low-alkali (0.55% Na_2O_{eq}) Portland cement, and distilled water. Reagent-grade NaOH pellets were dissolved in the mix water to provide appropriate alkali contents in the concrete mixes. Table 2 gives the chemical and physical characteristics of the Portland cement used along with its phase composition calculated by the Bogue method.

The concrete mix proportions were in accordance with the CSA A23.2-14A-M94 test procedure. The alkali content of the concrete was varied in the range 2.3 to 9.0 kg Na_2O_{eq}/m^3 by adding appropriate amounts of NaOH to the mix water. The alkali content of 2.3 kg Na_2O_{eq}/m^3 was achieved with no addition of NaOH to the concrete mix, i.e., with the native alkali content of the cement used.

From each concrete mix, three prisms, 70 × 70 × 280 mm in size, were cast, demoulded after 1-day storage in air at 20°C and RH > 90%, and subsequently cured in alkaline solutions at 150°C, according to the test procedure developed by Criaud et al. [11]

The composition of the alkaline solution in contact with the concrete prisms (essentially, Na^+, K^+ and OH^- ions) was calculated on the basis of the water/cement ratio of the concrete mix, the amount of NaOH added to the mix water, and the soluble alkali of each of the components of the concrete. Alkali solubilities were taken as 100% for Portland cement and 0% for fine and coarse aggregates. The alkaline solutions were prepared using distilled water and appropriate amounts of NaOH and KOH pellets.

Cylindrical stainless steel containers were used for the treatment, and each cylinder (height = 58.0 cm; internal diameter = 11.0 cm) contained one vertical prism immersed in approximately 2.5 l of alkaline solution. The containers were tightly closed and put in an oven at 150°C. Every week, the containers were cooled down under water and, after equilibration at 20°C, the length changes of the prisms were measured using a micrometer. The prisms were subsequently put back in their containers for another week of curing. Length changes were monitored up to 5 weeks of curing.

Table 2
Chemical and physical characteristics of portland cement used

SiO_2	21.20
Fe_2O_3	3.40
Al_2O_3	4.40
CaO	63.10
Free CaO	0.14
MgO	1.20
SO_3	3.30
Mn_2O_3	0.04
TiO_2	0.09
P_2O_5	0.10
SrO	0.06
Na_2O	0.27
K_2O	0.43
Na_2O eq	0.55
Specific surface area (Blaine) (m^2/kg)	400
Autoclave expansion (%)	0.02
Potential compounds (%)	
C_3S	51.50
C_2S	22.10
C_3A	5.90
C_4AF	10.50

The percent linear expansion of the prisms at a given curing time was determined as the average expansion of three specimens. The coefficient of variation for expansion measurements within a set of specimens was always less than 4%.

3. Results and discussion

Fig. 1(a)–(e) shows the expansions of concrete prisms incorporating each of the sands investigated as a function of their curing time in alkaline solutions at 150°C, when the alkali content of the concrete mixes was 2.3, 4.5. 6.6 or 9.0 kg Na_2O_{eq}/m^3, and the concentration of OH^- ions in the alkaline solutions was 393, 768, 1126 or 1536 mmoles/l, respectively.

For all the concrete mixes tested, expansions developed rapidly within the first 2 weeks of curing and then progressively levelled off. For most mixes, expansions were largely completed after about 3 weeks.

Increasing alkali content of the concrete generally resulted in an increased expansion of the concrete prisms.

At a fixed alkali content of the concrete, the greatest ultimate expansions (0.10–0.25%) were always observed for the concrete mixes incorporating sand *C*, while the lowest ultimate expansions were always found for the specimens made with sand *D* (0.06–0.11%). These results agreed well with the diagnoses of the ASTM C1260 mortar bar test and the infrared spectroscopy technique (Table 1), both indicating sand *C* as the most reactive and sand *D* as non-reactive.

In particular, these two test methods classed sand *D* as innocuous and the other four sands as potentially reactive: this diagnosis was in accordance with the field performance of the sands investigated. Also, on the basis of the judgment criteria established by the IR method [13] and reported in Table 1, all the reactive sands were classed as slowly reactive (Cd values within the range 120–200 cm^{-1}), but sand *C* had a Cd value (192 cm^{-1}) which was near the lower limit of the domain corresponding to rapidly reactive aggregates.

In developing the ultra-accelerated concrete prism test, Criaud et al. [11] proposed a provisional expansion limit of 0.11% at 3 weeks as the reactivity judgment criterion. This limit was derived from the correlation between the

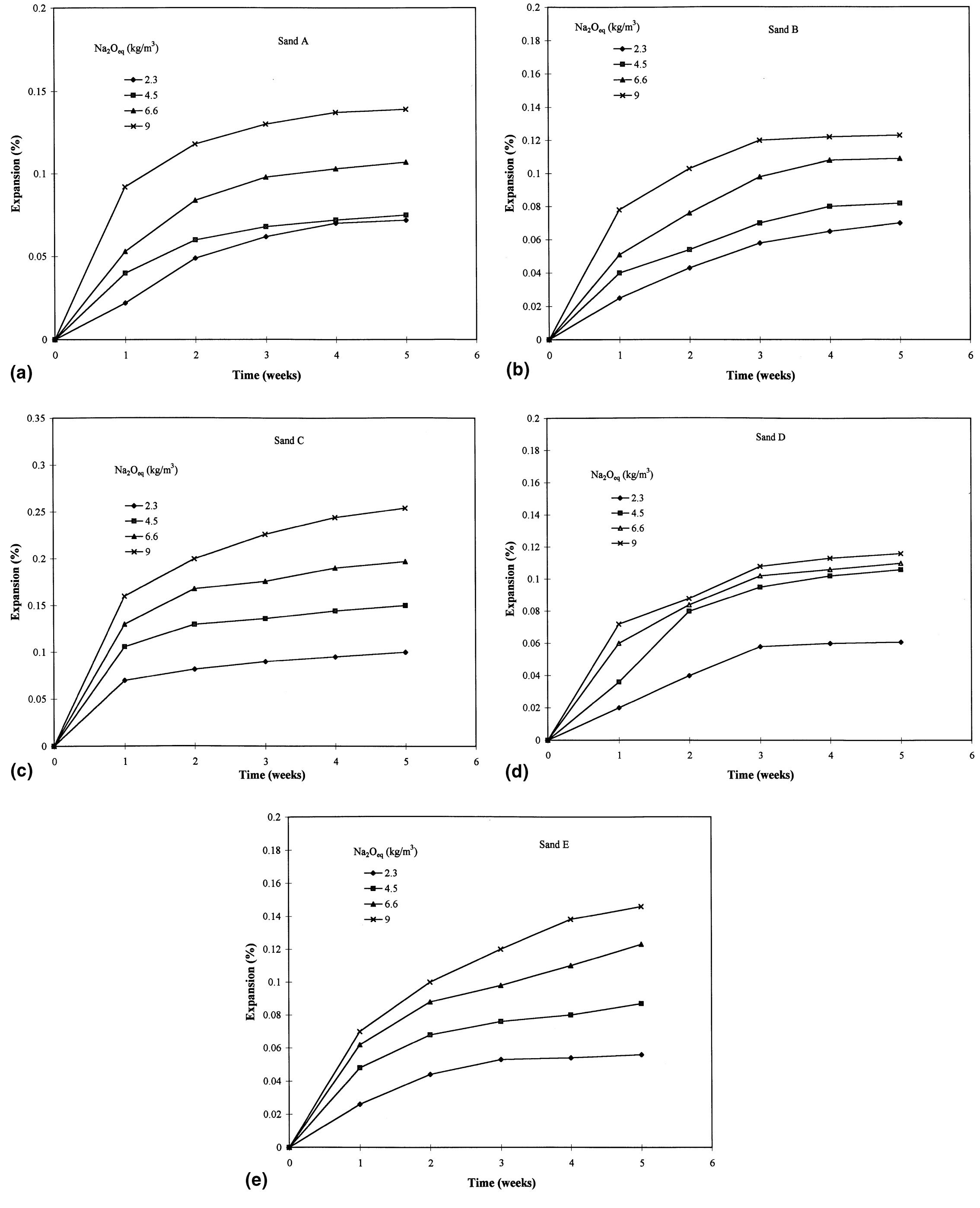

Fig. 1. (a)–(e) Expansion curves for concrete prisms immersed in alkaline solutions at 150°C.

3-weeks expansions of concrete prisms in alkaline solutions at 150°C and the 16-weeks expansions at 60°C and 100% RH. If the 1-year expansions at 38°C and 100% RH were taken as a basis, a slightly higher expansion limit (about 0.14%) was found for the ultra-accelerated test. However, this limit was not considered by the above Authors, because of the uncertainty arising from the correlation with a very limited number of data at 38°C.

In order to better define the judgment criterion for the ultra-accelerated concrete prism test, in the present study the expansion data at 150°C (Fig. 1 (a)–(e)) were correlated with those obtained on the same concrete mixes at 38°C and 100% RH in a previous work [3]. This correlation was limited to concrete mixes with alkali contents of 4.5, 6.6 and 9.0 kg Na_2O_{eq}/m^3. For mixes incorporating sand *C*, data were also available for comparing the expansions at an alkali content of 2.3 kg Na_2O_{eq}/m^3.

The 3-weeks expansions in alkaline solutions at 150°C are shown plotted against 1-year expansions at 38°C and 100% RH in Fig. 2. The vertical dotted line represents the expansion limit of 0.04% at 1 year, which has been adopted as the reactivity judgment criterion in the CSA A23.2-14A-M94 test. The two horizontal dotted lines correspond to the expansion levels of 0.11% and 0.14% at 3 weeks, which have been hypothesized as possible reactivity judgment criteria for the ultra-accelerated concrete prism test.

Thus, the plot of Fig. 2 can be divided in four zones. Points on zones 1 and 3 indicate agreement between the verdicts of the two test methods (non-reactivity for zone 1 and reactivity for zone 3), while points on zones 2 and 4 indicate disagreement between the two test methods.

From Fig. 2 it can be seen that, for all the concrete mixes tested, there was agreement between the verdicts of the two test methods only if the expansion level of 0.11% at 3 weeks was taken as the reactivity judgment criterion for the ultra-accelerated concrete prism test.

On the basis of this expansion limit and the data in Fig. 1(a)–(e), it was then possible to determine the threshold alkali level for each sand investigated, by plotting the 3-weeks expansions of the concrete mixes as a function of the alkali content of the concrete.

As shown in Fig. 3, the threshold alkali levels were found (by interpolation and, for sand *D*, by extrapolation) to be 3.3 kg Na_2O_{eq}/m^3 for sand *C*, 7.5 kg Na_2O_{eq}/m^3 for sand *A*, 7.6 kg Na_2O_{eq}/m^3 for sand *B*, 7.7 kg Na_2O_{eq}/m^3 for sand *E*, and 9.7 kg Na_2O_{eq}/m^3 for sand *D*. These results confirmed the great influence of the nature of aggregate on the TAL value, and also explained the different field performances of the sands investigated.

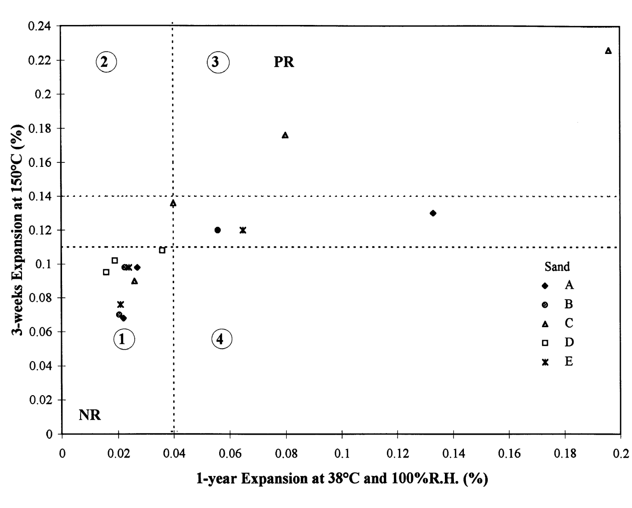

Fig. 2. Correlation between the 3-weeks expansions at 150°C and the 1-year expansions at 38°C and 100% RH.

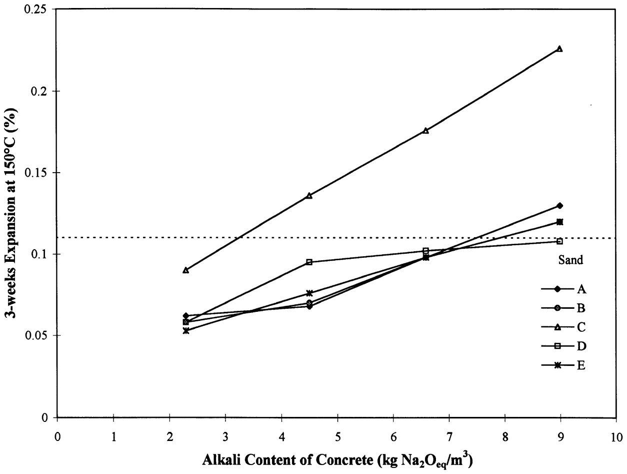

Fig. 3. Determination of threshold alkali levels from expansion data at 150°C.

If it is considered that: (1) the native alkali content of the concrete structures is commonly 4 to 5 kg Na_2O_{eq}/m^3, and (2) under particular exposure conditions, the alkali content of certain zones of the concrete structures may even double as a consequence of migration and/or penetration of alkalies in concrete [7], it may be explained why sand *C*, which has a TAL value of 3.3 kg Na_2O_{eq}/m^3, has always shown reaction in service, whereas sand *D*, which has an estimated TAL value of 9.7 kg Na_2O_{eq}/m^3, has shown no sign of alkali–silica reaction in concrete structures commonly designed on native alkali levels of 4 to 5 kg Na_2O_{eq}/m^3. The same considerations may also explain the different expansive behaviour of sands *A*, *B* and *E*, depending on the exposure conditions of the concrete structures.

A practical inference is that there is a problem correlating the safe concrete alkali contents estimated from laboratory tests with those for field concretes. Also, if concrete is designed on a native alkali content of 4 to 5 kg Na_2O_{eq}/m^3, the selection of aggregates for structures subjected to severe exposure conditions could be very problematic since, in such cases, aggregates with TAL above 9 kg Na_2O_{eq}/m^3 should be used.

The data in Fig. 3 also show that the change in expansion with alkali content of concrete was greatly affected by the type of aggregate tested. Instead of the step-wise change of expansion reported to commonly happen when concrete prisms are moist cured at 20°C or 38°C [3,14], different slopes were observed. In particular, for concrete mixes incorporating sand *C* or *E*, a straight-line relationship was found between expansion and alkali content.

From Table 1 or Fig. 3 it can be seen that testing concrete mixes at a fixed alkali content may lead to misleading conclusions about the alkali-reactivity of susceptible aggregates. Thus, if concrete mixes incorporating each of the sands investigated had been tested using the CSA A23.2-14A-M94 concrete prism test (38°C; 100% RH; 5.25 kg Na_2O_{eq}/m^3) (Table 1) or the ultra-accelerated concrete prism test at an alkali content of 5.25 kg Na_2O_{eq}/m^3 (Fig. 3), then all the sands, except sand *C*, were innocuous. This diagnosis, however, would be in contrast to the poor field performance of sands *A*, *B* and *E* and the verdicts of ASTM C1260 and infrared spectroscopy (Table 1).

Furthermore, when concrete mixes were tested with the ultra-accelerated test at an alkali content of 6.6 kg Na_2O_{eq}/m^3, corresponding to a OH^- ion concentration of 1.13 M in the alkaline solution, it was not possible to differentiate the expansivities of the sands investigated (expansions of ±0.10% at 3 weeks), except sand *C* (Fig. 3). Conversely, the results of the ASTM C1260 test (1M NaOH solution at 80°C) indicated wide differences in the expansivities of these sands (Table 1): this discrepancy was difficult to explain. However, it must be considered that: (a) the results of the ASTM C1260 and ultra-accelerated concrete prism test were obtained using two different Portland cements (Tables 1 and 2), and (b) as reported in a previous paper [12], both the native alkali content and the Blaine specific surface area of the Portland cement were found to be significant factors in affecting mortar bar expansions in the ASTM C1260 test, at least when this test was used to assess the alkali-reactivity of slowly expanding aggregates. Therefore, further work is needed to investigate the influence of the type of Portland cement on the expansion of concrete mixes in the ultra-accelerated concrete prism test.

These arguments clearly demonstrated the great difficulty of the concrete prism expansion tests in detecting the potential alkali-reactivity of slowly expanding siliceous aggregates and, at the same time, supported the validity of using the threshold alkali level as a reactivity parameter for the selection of ASR-susceptible aggregates.

Fig. 4 compares the TAL values determined by the modified ultra-accelerated concrete prism test with those obtained from concrete prism expansion tests at 38°C and 100% RH [3].

For sands *A*, *D* and *E*, the TAL values at 150°C were slightly higher than the respective values at 38°C, while the opposite was observed for sands *B* and *C*. The greatest percentage difference between the TAL values at 38°C and 150°C was found for sand *C* and it amounted to about 25%. From these data, and also bearing in mind the complexity of the test procedures adopted and the uncertainty involved in the TAL determination, it can be concluded that there was a good correspondence between the TAL values determined by the two test methods, if the expansion limits of 0.11% at 3 weeks and 0.04% at 1 year were taken as reactivity judgment criteria for the ultra-accelerated and standard concrete prism tests, respectively.

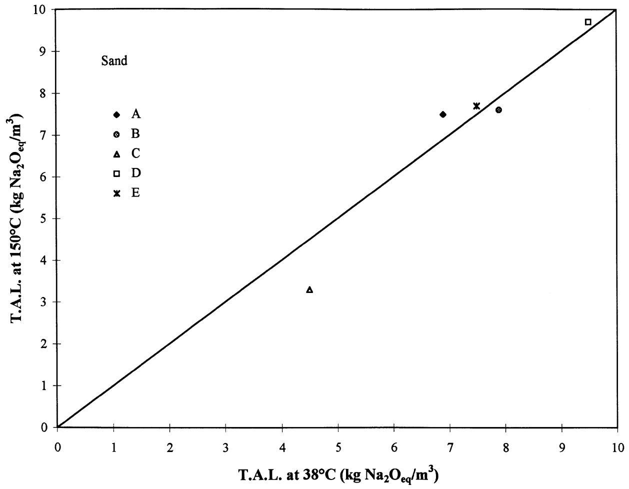

Fig. 4. Comparison between the TAL values obtained from the results of the ultra-accelerated concrete prism test and the concrete prism test at 38°C and 100% RH.

In a recent work [15], Hobbs observed that the approximate ages at which deleterious ASR expansion was induced in concretes made with a number of UK siliceous aggregates and different alkali contents, and stored at 38°C, 20°C and externally, were approximately in the ratio 1:4:7, respectively. When expansions for the concrete mixes were plotted against normalized age, the expansion curves were broadly similar, thus indicating that the alkali–silica reaction rates for concretes subjected to the three exposure conditions were approximately in the ratio 7:4:1. Also, no concrete with original alkali content below 4.8 kg Na_2O_{eq}/m^3 exhibited deleterious expansion. These results suggest that no significant change in TAL should be expected if concrete expansion tests are performed at 38°C or at normal exposure temperatures, although the latter test would be 7 times slower than the former one.

On the basis of the above considerations it can be concluded that the modified ultra-accelerated concrete prism test may be used as a rapid and reliable method for evaluating the threshold alkali levels for aggregates susceptible to ASR at normal exposure temperatures, provided that an expansion limit of 0.11% at 3 weeks is taken as the reactivity judgment criterion for this test.

The different TAL values exhibited by the sands investigated (Fig. 3) were consistent with their different alkali-reactivity as indicated by the results of the infrared spectroscopy technique (Table 1).

Sand *C* having the highest Cd value (192 cm^{-1}), exhibited the lowest TAL value (3.3 kg Na_2O_{eq}/m^3), while sand *D*, which was characterized by the lowest Cd value (100 cm^{-1}), exhibited the highest TAL value (9.7 kg Na_2O_{eq}/m^3). Also, for sands *A*, *B* and *E*, which were

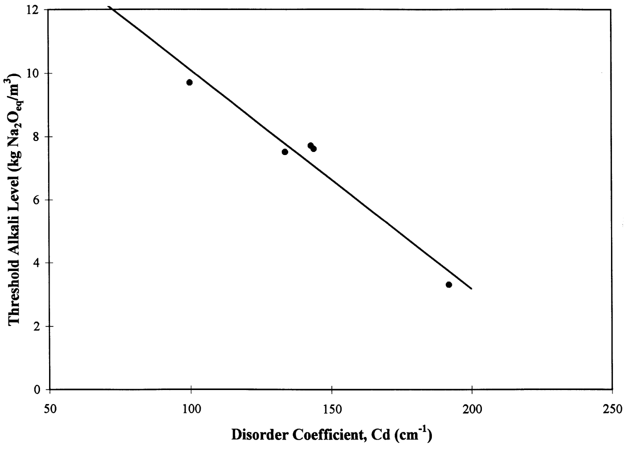

Fig. 5. Relationship between the threshold alkali level and the disorder coefficient, Cd, of the sands tested.

characterized by similar Cd values (134–144 cm^{-1}), no significant difference between the TAL values (7.5–7.7 kg Na_2O_{eq}/m^3) was observed.

Fig. 5 depicts the relationship between the coefficient of microstructural disorder, Cd, of the sands tested and their threshold alkali level determined by the modified ultra-accelerated concrete prism test.

A straight-line relationship was obtained and this relationship can be expressed as:

$$\mathrm{TAL} = 17.07 - 0.07\ \mathrm{Cd}$$

with a determination coefficient, R^2, of 0.95.

This relationship applies for Cd values not higher than 300 cm^{-1}, because siliceous aggregates with higher Cd values behave as pozzolanic materials [13].

If this equation could be validated by a greater number of data, collected on a large variety of reactive aggregates, then the determination of the disorder coefficient, Cd, of such aggregates could be a rapid and simple method for estimating their TAL values at normal exposure temperatures.

4. Conclusions

The modified ultra-accelerated concrete prism expansion test (testing concrete mixes at varying alkali content of concrete) proved to be a rapid and reliable test method for evaluating the threshold alkali level for alkali-reactive siliceous aggregates in concrete, provided that an expansion limit of 0.11% at 3 weeks was taken as the reactivity judgment criterion for this test.

Comparison between the results of the concrete prism tests and the field performance of the sands investigated, supported the validity of using the threshold alkali level as a reactivity parameter for the selection of alkali-reactive siliceous aggregates.

There was a straight-line relationship between the threshold alkali level of the sands tested and their microstructural disorder coefficient, Cd, as measured by infrared spectroscopy. This relationship suggested the possibility of developing a new test methodology for estimating the threshold alkali level of siliceous aggregates, based on the determination of their disorder coefficient, Cd, by infrared spectroscopy. However, much work would be needed to consolidate the data required for such an approach.

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