A COMPARISON OF EXPANSION TESTS FOR ALKALI-REACTIVITY OF AGGREGATES, BASED ON A KINETICS APPROACH – PART I - LIMIT RATES IMPLICIT IN REACTIVITY TEST CRITERIA

Luis Mayor Gonzalez[1*], António Santos Silva[2], Said Jalali[1]

[1] Department of Civil Engineering, University of Minho, Guimarães, Portugal

[2] Materials Department, National Laboratory for Civil Engineering (LNEC), Lisboa, Portugal

Abstract

Among the properties measuring aggregates reactivity towards alkali-silica reaction (ASR), linear dilatation of mortar bars or concrete prisms is often used in several tests for determining aggregate reactivity under various conditions, criteria and procedures of measuring and accelerating the reaction. As reactivity is an essentially kinetic property, the expansion limits in such tests, or even tests results for the same aggregate, should be kinetically inter-related. This paper presents a kinetic relationship proposal for critical reaction rates, evaluated from criteria of NF P 18-590, ASTM C 1260, and ASTM C 1293 standard test-methods. For this, the critical expansion rates of these tests were corrected for a reference 1N alkalinity and depicted as Arrhenius plot. The high correlation of the regression line evidences linearity of the proposed model, which was assumed as criterion of kinetic compatibility between tests. The discussion includes a comparison with other tests and literature comments on their evaluation.

Keywords: Expansion tests, alkali reactivity, ASR, kinetics, aggregate

1   INTRODUCTION

The alkali-silica reaction (ASR) or, in general, the alkali-aggregate reaction (AAR) is a system of physical and chemical transformations with global volume increase, between the alkaline concrete pore solution, some silica containing aggregates, and portlandite, $Ca(OH)_2$. Main factors affecting the reaction rate are alkalis, aggregate reactivity, humidity, temperature and presence of calcium ions. Absence or lowering of any of these factors is reported as enough to reduce or arrest the reaction.

The aggregate reactivity varies with origin, composition and geologic history. In Ireland flints are tested innocuous, but as reactive in England and North of France. Bektas et al 2008 [1] refer to differences in crystallinity. Nixon 1992 and Hobbs 2000 refer to the allowed use in U.K of aggregates with reactive components, but above pessimum contents [2, 3]. Le Roux 1984 [4] hints that metamorphization or weathering, even slight, may change reactivity, e.g., by straining quartz grains [5]. Rayment 1992, 1996 [6, 7] has shown flint reactivity to depend on weathering by the amount of external cortex. Wigum 1997 [8] refers to variations in composition and mechanical deformation of minerals reflecting regional variations. In other situations, limestones and rocks became reactive by siliceous inclusions in grains, veinlets or matrix impregnation invisible to the naked eye [5]. In Portugal, the more common reactive rocks are granites or gneisses, quartzites, greywackes and limestones with flint nodules [9].

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*   Correspondence to: lmgonzalez@mail.telepac.pt

With such sensitivity to slight changes, evaluation of the alkali-aggregate reactivity is especially important. Thomas et al 2006 [10] refer that an aggregate is classified as reactive by comparison with its field record; as this requires long periods of observation, expansion field tests on concrete block are a second best comparison basis. Aggregates or concrete mixes tested in these conditions may constitute a basis to validate other shorter tests or expedite criteria presently in use.

The ASR reactivity test of aggregates is often measured by linear expansion of mortar bars or concrete prisms, but many other material properties, inherent or affected by the reaction, are reported [11-14]. Several aggregate reactivity tests based on linear expansion differ in experimental conditions, procedures and criteria, as shown in Table 1. The tests are not exactly equivalent, as they classify differently some aggregates, due to national test variations and small differences in, e.g., bar size, effective humidity around them and criteria. The text of ASTM C 1293 [15], in Appendix X1, item X1.5, includes a comparison of a few relevant tests.

Briefly, in ASTM C 227 test [16], Grattan Bellew 1983, 1997 [17, 18] suggests limits ca four fold lower; Berubé et al. 1992 [19] found the test ASTM C 1260 [20] to yield some false positives, as confirmed by other authors [21-23]; the test ASTM C 1293 [15] is generally considered as the one better fitting field records; Thomas 2006 refers not being of his knowledge disagreements of reactive field records in normal utilization, with the ASTM C 1293 test, except for a few cases reported for highways with high alkali contents [10].

Tests expansions value depend on the conditions. As reactivity is essentially of a kinetic nature, such differences should agree with known effects of major kinetic factors like alkalinity and temperature. Several authors suggest care in test comparisons. Grattan-Bellew 1983 alerts to possible high temperatures reactions, not usually present [17]; Cyr and Guisbergues 2002 indicate different time scales for pre and after cracking stages [24]; Lenzner 1983 considers that at high temperature, expansion may start in unconsolidated concrete reducing initial expansion[25]; Chatterji 2005 highlights differences in field and laboratory conditions[26].

Not ignoring such useful and good sense alerts, this paper tries to highlight kinetic inter-relations among standard tests NF P 18-590 [27], ASTM C 1260 and ASTM C 1293, in a wide range of temperature and alkalinity. The correlation proposed is compared with other tests referred to in Table 1.

2. PROCEDURE

For the 3 mentioned tests, critical expansions were converted to reaction rates, corrected for alkalinity to arbitrarily pre-fixed conditions, and represented as an Arrhenius plot.

The **main assumptions** were:

- extent of reaction proportional to strain and measured by it;

- reaction rate is almost constant up to 0.10-0.15% for aggregates which expansion is near the upper limit value for nonreactives (referred to in this paper as **quasi reactive aggregates**), as shown for several tests in Table 1, and confirmed as approximation for several aggregates as displayed in Figure 1.

- alkalinity of the pore solution may be estimated by equation of Helmut 1993 [28], from the alkalis contents of the cement, and the water/cement ratio (w/c),

- reaction rate proportional to alkalinity, i.e., concentration of OH^-, defined as 1M in ASTM C 1260, and as given by equation (1) for ASTM C 1293, ASTM C 227 and NF P 18-590 tests. In the longer ASTM tests, concentration is corrected for leaching. Short duration test NF P 18-590 assumed no correction.

- kinetically consistent equivalent reaction rates of a reaction yield a linear Arrhenius plot. **The linearity of such plot is assumed as criterion of kinetic consistency between tests,** meaning that the same reaction step is controlling, not specifying it, nor implying all other transformations to be the same.

2.1.   Rationale and Background

In each test, the critical limits were set by experiment. To each criterion corresponds a critical rate of strain or reaction. A hypothetical virtual aggregate with such strain rate is considered. If all tests were fully consistent, such aggregate would be classified the same way. This is not exactly true for every real aggregate, but it is assumed to be so for most of them, otherwise tests would have been discarded.

Comparing tests is then to compare expansion criteria after given times, i.e., implicitly, rates of reaction at different test conditions. The relevant varying conditions are assumed as the main factors known to affect ASR (all other factors were neglected as they are kept constant or have lesser effects). Rates may then be corrected for alkalinity (proportionally) and temperature (following Arrhenius equation), as humidity is constant. To simplify, after corrections for alkalinity, the critical reaction rates of the tests should yield a straight line in Arrhenius plot.

Finally, reaction rates meaning is less evident in experimental terms. However, as they were assumed constant for quasi reactive aggregates, rates may be expressed by the inverse of their <u>equivalent ages</u>, i.e., the time needed to reach a common reference strain arbitrarily fixed at 0.10%, assuming a constant rate.

2.2   Assumptions made, in detail:

An almost linear **expansion** or constant rate is suggested for quasi reactive aggregates by the tests expansion limits, e.g., the ASTM C 1260. Then, critical reactivity means critical rate. These test limits aren't though exactly equivalent, as a hierarchy exists in case of divergence. The approach to linearity is shown in Figure 1 in real aggregates. Some cases evidence no proportionality, but just linearity beyond a point *ca* 0.02%, after which the rate becomes constant, so that the same assumption can be made just with a translation.

Alkalinity of the pore solution

Alkalinity of the pore solution is related by different authors with w/c and cement alkalis contents and by others with the total alkalis in concrete. The first situation is modeled by Helmut 1993 equation [28],

$$[OH^-] \text{ , mol/L} = 0.339 \text{ Na}_2\text{O}_{eq} \text{ \% / (w/c)} + 0.022 \pm 0.06 \qquad (1)$$

Equation (1) models just the effect of hydration and partial ion adsorption in concrete consolidation.

Other effects on alkalinity are due to the reaction (a measurable effect [29, 30]), to alkalis leaching under saturating humidity, and to alkalis liberation in certain aggregates (Le Roux considered these able to trigger the reaction [4]). For modeling purposes this work considers all effects included in the effect of leaching under saturating conditions. In ASTM C 1260 test conditions, the alkali is in large excess and the reaction effect is minimized, i.e., alkalinity is practically constant, at least in the early stage. The reaction rate, assumed proportional to alkalinity, is assumed as the concentration of OH$^-$.

Humidity

Humidity is a main factor on ASR. The reaction nearly stops at Relative Humidity (RH) below 80%, accelerating with humidity increase up to a maximum *ca* 95%; above this, leaching supposedly counteracts this trend. Capra 1998 [31] used a factor $(\text{RH,\%}/100)^8$ as effect of humidity in non saturating conditions.

Temperature

For thermoactivated transformations, it is expected that their rate constants follow Arrhenius law:

$$\ln (1/K) = k1. \exp (-Ea/RT) \qquad (2)$$

where K is the kinetic constant of the reaction, k1 is the pre-exponential constant, Ea is the apparent energy of activation, and T is the absolute temperature, in Kelvin degrees. Equation (2) applies if the controlling reaction/step is the same.

3   PROPOSED APPROACH
3.1   Assumptions summary
The assumptions used are presented in section 2.2 as: reaction proportional to strain, constant reaction rate for **quasi reactive aggregates**, alkalinity of the pore solution given by Helmut equation, reaction rate proportional to alkalinity, reaction rates constant following Arrhenius law.

3.2   Modelling the leaching effect
The effect of humidity in saturating conditions was studied experimentally by Rogers and Hooton 1989, 1991 [32, 33], Rivard 2003 [34] and Lindgard 2010 [35]. Lining with absorbent coating the concrete prisms or container walls, to promote humidifying, may induce a significant reduction of the expansion; Rivard 2003 has shown condensation to occur on the surface of the concrete, leaching alkalis from concrete[34]. In the case of NF P 18 590 test-method, the bars are immersed in water, but leaching was neglected as the test is very short. This assumption should however be verified, if possible.

External diffusion controlled leaching model
Gonzalez et al. 2011 presented a model for **leaching** [36] assuming continuous condensation, overflowing as film running down over the prism surface; the diffusion from prism surface, S, into this film was supposed as controlling and modeled assuming uniform concentration inside the prism, Cas.

$$Cas = Caso \cdot \exp(-Kt) \tag{3}$$

In the beginning, Cas = Caso (the initial concentration) and in the end (for t= infinite) Cas = 0

Knowing alkali losses at a certain age, (1- Cas / Caso), Equation (3) yields the value of K, and enables at any age to evaluate the concentration Cas and its average value during the test. Thomas 2006 [10] refers a loss at 12 months of 35% of alkalis in conditions similar to ASTM C 1293. Equation (3) yields a constant of 0.00118 and, for an initial contents of 1.25% Na_2O_{eq}, alkalis, values of 1.01% average and 0.81% Na_2O_{eq} final. Without other experimental data, these values were generalized until better information is available.

This model corresponds to descriptions commonly found. It is very rough, though, and departs from original leaching data at intermediate times, overestimating the average alkali contents, as shown in Figure 2.

Internal diffusion controlled leaching model
Another possible model assumed leaching as a process controlled by pore diffusion, leaving an inner, higher concentration region (assumption consistent, e.g., with the higher viscosity expected in this region).

The interface was assumed as plane, a well studied model (see, e.g., Levenspiel 1972 [37]), yielding

$$Cas = Caso \cdot (1 - K'(t)^{1/2}) \tag{4}$$

For the leaching data used, the same final value requires $K'_o = 0.01832$. A fit to minimize squared X deviations yields $K'_x = 0.01952$, while minimum squared time deviations yields $K'_t = 0.01924$. For this last K'_t value, which seemed best, a concrete 1.25 % Na_2O_{eq}, at 365 days, has 36% of its alkalis leached, final contents of 0.79% Na_2O_{eq} and 0.96% average. For 0.90% Na_2O_{eq} initial, has final value of 0.56%, and 0.69% average.

Figure 2 depicts alkali losses in weight fraction, X=(1-Cas/Caso), against time, for the leaching data of Thomas 2006 [10] and estimates for final value, time fit or X fit criteria. The internal diffusion control, plane surface model, fits data better, by any of these criteria, than the earlier model (equation 3). As earlier, results are extrapolated to other concretes; a better support for such assumption needs an experimental program.

3.3 Equivalent reaction rate at a reference concentration

For test comparison, the relevant reaction rate is not the obtained from experimental data, but the equivalent reaction rate estimated at a pre-fixed alkalinity, reference concentration, 1 M.

The experimental rate of reaction is $v_e = \delta_e/t_e$, where δ_e, the strain measured after time t_e, is assumed, for quasi reactive aggregates, as the limit strain defined in the end of the reference test. Being proportional to the concentration used in each test, C_e, may be corrected for the reference concentration C_S:

$$v_s = v_e \, (C_s/C_e) = \delta_e/t_e \, (C_s/C_e). \tag{5}$$

3.4 Time equivalent at a reference strain

Arrhenius plots are valid for reaction rates, strains and times being easier to understand. For assumed constant rate, plots may use instead an equivalent age t_S, or time to reach a reference strain, δ_S, fixed as 0.10%.

$$t_S = t_e \, (C_e/C_s) \, (\delta_S/\delta_e) \tag{6}$$

The equivalent age, a virtual value, is listed for each test in Table 1, valid for quasi reactive aggregates.

4. RESULTS

4.1 Comparison of expansion tests

Critical rates and equivalent times representation in Arrhenius plot

From the experimental conditions listed in Table 1, the critical expansion rate and equivalent ages were estimated for each test, as presented in Table 2.

The equivalent time for the selected expansion tests NF P18-590, ASTM C 1260, and ASTM C 1293, [15, 20, 27] are plotted in Figure 3 along a regression line with high correlation coefficient. As the points refer to limit reactivity reaction rates, the line separates then a reactive field (higher reaction rates, above the line) from a non reactive one. Variations are expected in experimental conditions, even within allowed tolerances, broadening the fields delimiting line. The sensitivity to temperature settings allowance of ± 2°C, at 38 and 80 °C, affect the results, as depicted by the dashed lines in Figure 3. Other variations may be present.

Comparison

Other tests from Canada, Norway and Germany, based on linear expansion, were also represented in the same plot (Figure 3), for comparison.

In immersed bar tests, AMBT, at 80 °C in NaOH 1M, the limit values for expansion range from 0.08 to 0.15 (Berube et al. 1992, Hooton and Rogers 1992, Wigum 1995 [19, 38, 39]). The ASTM C 1260 defines the limit as 0.10%, referred to by some authors (Lobo 1998, Hooton 1994) as too severe [21, 22]. In Figure 3, the exactly co-linear position for this test-method in relation to the other two corresponds to 0.11% at 14 days, or 0.10% at 12.7 days. Both these values equal the limits for tests AMBT in Norway (for fine and coarse) and Germany, respectively [23] [Wigum 2006. In Norway, expansion limits may differ for coarse (0.08%) and fine (0.14%) fractions if tested separately (using an innocuous complement). In Canada, the test uses 0.15% as limit (0.10% for limestones and other aggregates [40][Chak and Chan 2004]).

Expansion tests using concrete prisms in saturated container were also considered. Equivalent ages for ASTM C 227 [16], (corrected for leaching) is plotted at two alkalis levels in concrete, 0.90 and 1.25% Na_2O_{eq},

inside the reactive field, what is in agreement with comments that it may yield false negatives. In this test, the correction value for leaching used the same as discussed for ASTM C 1293, so that the two tests differ only in the criteria, for 1.25% case. Other similar tests in saturating conditions over water are cited from German test reviews [23,44] at 40 ºC and 70 ºC may also be close to the limiting line (the leaching correction factor considered was the same as for 38 ºC, so that some deviation, particularly at 70 ºC are to be expected. Bar size also is different for the 40 ºC test.

Other common autoclave test, the microbar test, was not considered as it uses more than one temperature. For comparison with the present model, intermediate expansion measurements and the alkalis losses after each temperature cure are required what would imply a special experimental program.

Evaluation

The results found are close to comments from authors on these tests under different perspectives, to which this kinetic approach may lend apparently a certain rationale, indicating a possible kinetic consistency between tests. However, it allows just an approximate comparison and must be considered as preliminary, needing to include ill or not yet quantified effects of factors of known relevance like leaching, size and size distribution, and to better quantify the effect of factors already considered such as alkalinity and humidity.

As underlined above, the points in this plot are not exactly equivalent. The criterion of ASTM C 1293 is more significant, whereas ASTM C 1260, that may yield false positives, is somewhat shifted downward, to the reactive side; such a downward shift makes sense, as the alkalinity decreases more slowly in this test.

The criteria dispersion for the AMBT evidences a near but not full equivalence. It results from varying size or other unaccounted sub-factors in the nature and properties of the aggregates, shape or size of the specimens. In alkalis liberating aggregates a limit of 0.08% is referred to in Hooton and Rogers 1992 [38].

This near but not exact equivalence agrees with the known complexity of the reaction, with a multitude of factors acting on the expansion. When a test fixes limit values for specified conditions, the effect of other lesser factors is ignored, or these factors are considered have no variation at all.

To underline a positive comparison in spite of a still incomplete knowledge and modeling of the underlying phenomena, the authors considered the results as the **tests being not kinetically inconsistent.**

4.2   Other expansion tests

The comparison procedure proposed doesn't apply directly to complex tests, like the microbar test AFNOR P18-588, with intermediate cure at 100 ºC before autoclave at 150 ºC [41]. Future work may study the application of this test and the chemical test, ASTM C 289, to the procedure proposed.

5.   CONCLUSIONS AND RECOMMENDATIONS

Kinetic consistency was checked between ASTM C 1293, ASTM C 1260 and NF P 18-590 tests.

The comparison carried out assumed constant expansion rate for quasi critical reactivity. In some tested aggregates, the linearity of expansion for quasi reactive aggregate was observed only above *ca* 0.02%.

The effect of the main factors (alkalinity, humidity and temperature) used models found in literature. For leaching effect a model is proposed. The consistency criterion adopted was the linearity of the Arrhenius plot of the reaction rate after correction by the effect of the other factors to a same comparison basis.

The proposed approach has lead to a linear Arrhenius plot, evidencing a global coherence between tests conditions and related criteria. Effect of relevant factors variation was considered for alkalinity, temperature and leaching; others were not considered at all, either because are constant or are neglectable. Considering tests temperature allowances, a temperature allowed variation was obtained (dashed lines, Figure 1). A few other linear expansion tests were added, and found to fall within or close to this allowed variation.

These results only concern the presence of a common controlling step. Other steps may exist or be added, and vary, even changing the overall mechanism, but they are kinetically irrelevant, if they are faster.

Present conclusions are constrained by the simplified assumptions and lack of precision, difficult to avoid in such complex reaction. Their quality may be improved, namely:

i) by improving the used models, namely regarding alkalinity variation due to ASR and leaching.

ii) by extending the proposed approach to other standard expansion tests, mainly those using linear expansion, as well as to data from expansion tests of aggregates in different tests (results for some aggregates are presented in part II of this paper). If results are positive, extend to tests based on different properties.

iii) by improving the accuracy of reading experimental data, namely expansion and temperature and, when relevant, alkalinity and its evolution

ACKNOWLEDGEMENTS

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Table 1 - Summary of test parameters for selected linear expansion techniques (revisions in Silva 2005 [42], Wigum 2006 [23], Chak and Chan 2004 [40] and respective equivalent times (ages). Adapted from [36]. The subscript "e" designates test or experimental, and "s" designates reference or standard.

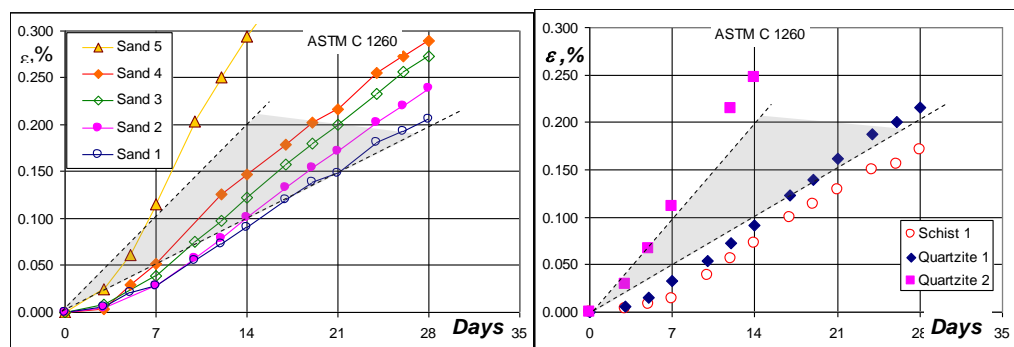| Designation	[16] ASTM C 227 USA		[15] ASTC C 1293 USA	[20] ASTM C 1260 USA	[27] NF P 18-590 France	[23] NB 21 (Norway)	[43] Alkali Richtlinie (Germany)	[43] Alkali Richtlinie (Germany)	[43] Alkali Richtlinie (Germany)
Abbreviation	Mortar		CPT	AMBT 1	autoclave	AMBT 2	AMBT 3	CPT Alt	CPT 2
Bar size , mm	25x25x285		75x75x250	25x25x285	40x40x160	40x40x160	40x40x160	40x40x160	100x100x500
Conditions, humidity	Container, saturating over water		Container, saturating over water	Immersed, NaOH 1M	Immersed, water	Immersed, NaOH 1M	Immersed, NaOH 1M	Container, saturating over water	Container, saturating over water
Alkalinity: [OH-] solution, w/c, Na_2O_{eq} cem	not specified ; 0.9-1.25% assumed		1.25%	NaOH 1M	4%	NaOH 1M	NaOH 1M	2.5%	1.3%
Ages for readings: **h**ours, **d**ays, **m**onths	3, 6, 12 m		0, 7,28,56 d 3,6,9,12 m	0, 3,7,10,14, 21, 28 d	5 h ± 10 min	14 d	13 d (previously, 14 d)	28 d	9 m
Critical expansion	in ASTM C 33 [44]: 0.05% at 3 m 0.10% at 6 m		0.04%@12m	10%@14d 20%@28d	0.15%@5d	0.08% (coarse), 0.11%(fine,coarse) 0.14% (fine)	0.10% at 13d	0.15%@28 d Previously, 0.20%@21d	0.06% at 9 m
Observations	Being too permissive, may yield false negatives		Yields best results face to performance records	Being too strict, may yield false positives	Considered too strict	Criterion allows to separate fines, coarse, and both altogether		Alternative process Expansions read at 20ºC.	
t, ºC	38		38	80	127	80	80	70	40
δ_e , %	0.10		0.04	0.10 - 0.20	0.15	0.11 (fine, coarse) 0.14 (fine) 0.08 (coarse)	0.10	0.15	0.06
t_e , days or hours	181 d		365 d	14-28 d	5.25 h	14 d	13 d	28 d	9 m
C_e concentn or from Helmut eqn, M	0.49	0.67	0.74	1	2.73	1	1	1.32 (est)	0.70 (est)
Na_2O_{eq},% — Initial	0.89	1.25	1.25		4			2.5	1.3
Na_2O_{eq},% — Average during leaching	0.688	0.955	0.955	-	-	-	-	1.91	0.99
Equivalent expansion rate, %/day	0.00113	0.000825	0.0001478	0.00714	0.399	0.00786	0.00769	0.0041	0.00032
Equivalent time (or age), days	88.5	121	677	14	0.25	12.7(fine, coarse)	13	24.6	313

Figure 1 - Results of test ASTM C 1260 for five sands (a) and two quartzites and a schist (b); the expansion is nearly linear in quartzite 2. Shadowed area is the zone critically reactive.
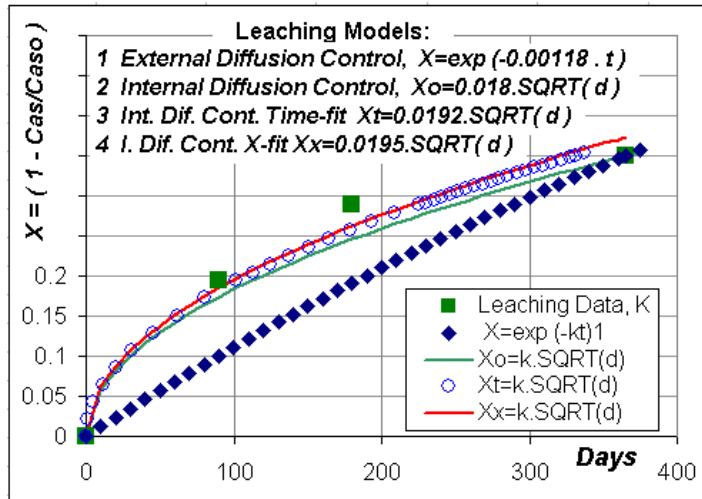
Figure 2 Comparison of models controlled by external and by internal diffusion. In ordinates, fractional losses of alkalis.
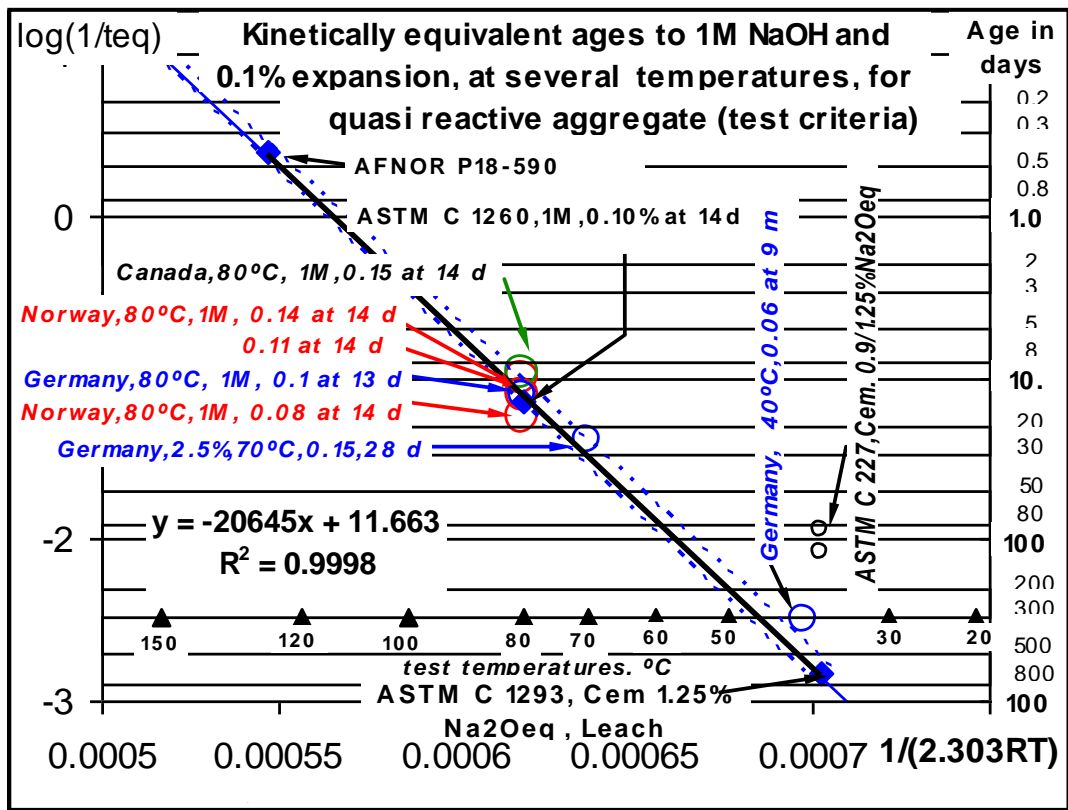

Figure 3 - Arrhenius plot relating ASTM C 1260, ASTM C 1293 and NF P18-590, in comparison with a few other expansion tests (see text). Dashed lines delimit approximately effect of temperature variation of ± 2 °C. (as allowed, i.a., by the ASTM C 1260)