RAPID TEST METHOD FOR EVALUATING POTENTIAL ALKALI REACTIVITY OF AGGREGATES BY AUTOCLAVING TREATMENT

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In order to reduce the testing time for evaluating the potential alkali reactivity of aggregates in less than one week, a rapid test method using autoclaving treatment of mortar bars is examined.
The effect of various testing conditions was investigated on aggregates with various kinetics and several reactivity characterization criteria were tested.
About 50 samples of aggregate were tested under set conditions. Correlation with the mortar-bar method, chemical test is discussed.

INTRODUCTION

Amongst the accelerated tests for identifying the potential alkali-reactivity of aggregates used in concrete, autoclave tests on mortar test bars, proposed since 1986 by Japanese and Chinese researchers, offer the possibility of reducing to a few days the time needed for characterizing an aggregate using relatively simple equipment.

Despite these advantages, few studies have concentrated on verifying and comparing the different autoclave tests, taking into account the fact that experimental conditions and the criteria for characterizing aggregates differ considerably from test to test.

Our experiments with autoclave tests on mortar began with an attempt to test different French and Canadian aggregates using a method very similar to that proposed by Tamura et al (1, 2). After these unsuccessful tests, we tried to determine the experimental conditions and characterization criteria which would facilitate identification of reactive aggregates, taking into account the work of Nishibayashi et al (3). The study resulted in the definition of an original autoclave test capable of triggering an alkali-aggregate reaction within the mortar, similar to that observed in pathological in-situ cases.

IDENTIFICATION OF AGGREGATES

Six aggregates were selected for developing the autoclave test; they were identified by petrographic inspection and accelerated tests (Table 1).

Four of the aggregates contain reactive minerals aAnd have already presented pathological symptoms in structures due to alkali-aggregate reactions. The other two have been identified as non-reactive.

The reactive aggregates were chosen in such a manner as to cover the wide spectrum of kinetics of reaction with alkalis. In fact, the Chambon and Spratt aggregates, with rapid kinetics, are easily identified as potentially reactive by the different accelerated tests, whereas the potentially reactive nature of the Sudbury and Light Posdam aggregates, with slow kinetics, is not always demonstrated by accelerated tests.

The non-reactive aggregates chosen were, firstly pure calcareous Omya limestone, used as a test control, and secondly dark Posdam aggregate, from the same source as the light Posdam aggregate but, unlike the latter, containing non-reactive minerals.

OPTIMISATION OF EXPERIMENT CONDITIONS

Initial conditions

Three mortar test bars measuring 4x4x16 cm were prepared, using:

600 g of the aggregate being tested (0.16-0.63 mm / 0.63-2.5 mm / 2.5-5.0 mm = 2/5/3),
600 g of Omya aggregate with the same particle size,
600 g of HPR portland cement (0.98% Na_2O equivalent),
300 g of NaOH solution to adjust the Na_2O equivalent content of the cement to 2.5%.

After being left to set for one day in the mould and one day in water, the test bars were autoclaved in water for two hours at a relative pressure of 0.05 MPa (111°C). After this, they were left to cool till 20°C in the autoclaving water for 20 hours.

Variations in the length, weight, ultrasonic pulse velocity, and dynamic modulus of elasticity of the test bars were measured, as were the compressive and bending strengths.

Three aggregates, Chambon, Spratt and Omya, were tested under these conditions, which are very close to those recommended by the Tamura autoclave test.

AGGREGATE	PETROGRAPHY
CHAMBON (FR)	Sericite micaschist including medium-grained quartz, fine-grained and crypto-crystalline quartz, chlorite and recrystallized carbonates
SPRATT (CAN)	Fine-grained, micritic or sparitic limestone with dolomite including detrital micro-quartz and fine-grained silica between carbonate grains
SUDBURY (CAN)	Quartzitic sandstone including akrose, chlorite, sericite and feldspar; also olivine and pyroxene gabbro; also alterate recrystallised rock including epidote, albite and chlorite.
POSTDAM light (CAN)	Fine-grained quartzite including undulose extinction quartz and few phyllosilicates between the quartz grains
POSTDAM dark (CAN)	Sandstone with uniform extinction rounded quartz grains and carbonate rock cement
OMYA (FR)	Pure recrystallized calcite limestone

AGGREGATE	STRUCTURE DETERIORATION CASES	MORTAR-BAR TEST [(a)] 6 months exp (%)	CONCRETE PRISM TEST [(b)] 8 months exp (%)	MICRO-BAR TEST [(c)]	KINETIC TEST [(d)]
CHAMBON (FR)	observed	0.138	0.169	potentially reactive	potentially reactive
SPRATT (CAN)	observed	0.313	0.128	potentially reactive	potentially reactive
SUDBURY (CAN)	observed	0.027	0.073	potentially reactive	potentially reactive
POSTDAM light (CAN)	observed	0.021	0.083	potentially reactive	potentially reactive
POSTDAM dark (CAN)	not observed	0.020	0.038	n.a.	n.a.
OMYA (FR)	not observed	0.013	0.011	nonreactive	nonreactive

[(a)] NF P 18 585 mortar-bar test corresponding to ASTM C 227 test

[(b)] NF P 18 587 conctete prism test corresponding to CAN3-A23.2-14A test

[(c)] NF P 18 588 mortar micro-bar test

[(d)] Chemical kinetic test : future NF P 18 5...

TABLE 1 : Identification of aggragates used for autoclave treatment

CHARACTERIZATION CRITERIA	AGGREGATE		
	OMYA	SPRATT	CHAMBON
Visual cracking	not observed	not observed	not observed
Variation of ultrasonic pulse velocity (%)	-1.6	-2.1	-2.1
Variation of dyn. Young's modulus (%)	-13.1	-9.3	-7.9
Expansion (%)	+0.031	+0.038	+0.031
Variation of weight (%)	-0.03	+0.08	+0.08
Flexural srength (MPa)	5.6	5.4	5.9
Compressive strength (MPa)	24.4	25.5	25.6

TABLE 2 : Characterization of aggregates by autoclave accelerated test in initial conditions

The results obtained (Table 2) are similar for the three aggregates, in contradiction to the reactivity noted in the concrete structures and the results of the other accelerated tests (Table 1). If the characterization criteria put forward by Tamura are used (absence of visible cracking, less than 15% reduction in dynamic modulus of elasticity, less than 5% reduction in ultrasonic pulse velocity), the three aggregates tested would be considered to be non-reactive.

As a result of these unsuccessful initial tests, we attempted to identify experiment conditions which would allow differentiation between aggregates with different reactivity.

Na_2O equivalent content

The mortar was enriched with alkalis to an Na_2O equivalent content of 4% and the other experiment conditions remained constant (Figure 1).

The increase in the Na_2O equivalent content from 2.5% to 4% resulted, in the case of the Chambon aggregate, in a significant increase in the length and weight of the test bars, together with a reduction in the ultasonic pulse velocity and the dynamic modulus of elasticity. These variations are much less significant in the case of the pure calcareous Omya limestone.

The Na_2O equivalent content of 4% was therefore used for the rest of the tests.

It was noted that the increase in alkalinity of the mortar resulted in a decrease in the mechanical strengths irrespective of the nature of the aggregate tested.

Autoclaving pressure

Having established the Na_2O equivalent content of the mortar at 4%, the autoclaving relative pressure was increased to 0.15 MPa (127°C). The Chambon, Sudbury and Omya aggregates were tested under these conditions (Figure 2).

With the increased autoclaving pressure, the variations in the length and weight of the mortar made with Chambon aggregate almost doubled, and cracks appeared on the surface of the test bars. The results obtained allowed this aggregate to be identified as potentially reactive according to the Tamura criteria.

On the other hand, the mortar using Sudbury aggregate produced results very close to those for the mortar using the non-reactive Omya aggregate, the latter showing only very slight sensitivity to the increase in the autoclaving pressure. So the Sudbury aggregate would be identified as non-reactive according to the Tamura criteria.

Therefore the increase in the autoclaving pressure and the increase in the alkali content are conditions which allow identification of the reactivity of the Chambon aggregate (with rapid kinetics), but are nevertheless inadequate for characterizing the slow reactivity of the Sudbury aggregate.

Autoclave curing time

The autoclave curing time was extended to five hours with an adjusted Na_2O equivalent content of 4% and an autoclaving pressure of 0.15 MPa. The Chambon, Spratt, Sudbury and Omya aggregates were tested (Figure 3).

Extension of the autoclave curing time allowed differentiation between the aggregates according to their potential reactivity. We note in particular that the expansion and mass gain values of the mortar made with Sudbury aggregate almost doubled.

The variations in the ultasonic pulse velocity and in the dynamic modulus of elasticity do not, however, follow a coherent relation. With increased autoclaving time, the Spratt aggregate produces low values, close to the Omya aggregate, whilst the values for the Chambon and Sudbury aggregates stand out from the latter.

A study of the standard deviation of values also confirms that measurement of the length variation (+/-3.5%) and weight variation (+/- 8%) of mortars is more reliable than measurement of the ultrasonic pulse velocity variation (+/-25%) or the dynamic modulus of elasticity variation (+/-10%) for the purpose of identifying the potential reactivity of the aggregates tested.

It should be noted that the mechanical strengths of the different mortars seem, overall, to be independent of the potential reactivity of the aggregates.

Size of test specimens

Tests using an adjusted Na_2O equivalent content of 4% and an autoclaving pressure of 0.15 MPa for two hours, were carried out on 2cm x 2cm x 16cm test specimens made with Chambon and Sudbury aggregate, and compared with the preceding tests on 4cm x 4cm x 16cm test specimens (Figure 4).

The reduction in section of the test specimens significantly amplifies the reduction in the ultasonic pulse velocity and the dynamic modulus of elasticity of the mortars made with these potentially reactive aggregates. On the other hand, it adversely affects the length and weight variations.

Taking into account the previous results on the reliability of the different criteria for characterizing aggregates, a reduction in section of the test specimenss was not adopted for the rest of the study.

It should also be noted that failure in bending can occur when a small load is applied to certain reduced section test specimens, thus preventing the mechanical strengths of the mortar from being identified. This behaviour confirms the deep cracking of the mortars, in keeping with the greater reduction in the ultrasonic pulse velocity and the dynamic modulus of elasticity. In this case, the reduction in expansion and weight gain of the smaller section (2cm x 2cm) test bars can only be explained by the supposition that the expansive products formed are pushed towards the exterior, assisted by the deep cracking of the material and the reduction in section.

Relative quantity of aggregate to be tested

All the foregoing tests were carried out on a mix of 50% of the aggregate being tested and 50% of non-reactive aggregate (Omya), in accordance with the preparatory conditions of the Tamura test. We increased the relative quantity of the aggregate being tested to 100%, whilst keeping the ratio of total sand/cement equal to 2. The tests were carried out using Spratt, Sudbury, Light Posdam, Dark Posdam and Omya aggregates, with an Na2O equivalent content of 4%, on 4cm x 4cm x 16cm test bars, and with an autoclaving pressure of 0.15 MPa and a curing time of 5 hours (Figure 5).

Replacement of non-reactive aggregate with the aggregate being tested increases, in all cases, the differences between the aggregates according to their reactivity to alkalis. As a result, this modification to the composition of the mix was adopted.

Water/cement ratio

All the mortars previously studied were prepared using 300 g of NaOH solution, as recommended by the Tamura test method. When the mortar is enriched to 4% Na_2O equivalent, this quantity of solution results in a water/cement ratio of around 0.46 for the mortar. It is clear that this ratio varies as a function of the Na_2O equivalent content of the cement (in our case, 0.98%).

In order to eliminate this problem, which could affect the reproducibility of the test, we decided to use 300 ml of NaOH solution for mixing. Given this condition, the quantity of mixing water remains practically constant and corresponds to a water/cement ratio of 0.50 with an accuracy of ±0.1%, whatever the quantity of NaOH added to the water as required to increase the Na_2O eq. content of the cement to 4%.

Increasing the water/cement ratio from 0.46 to 0.50 results in an attenuation of the differences measured between the aggregates (Figure 6). However, examination of the spread of results shows improved reliability, which justifies the adoption of this modification in the test procedures.

WORKING METHOD ADOPTED

All the preceding results allowed a new working method to be defined for autoclave tests, permitting the potential alkali-reactivity of aggregates to be identified.

Three prismatic test bars (4cmx4cmx16cm) are made up using:

1200 g of the aggregate to be tested, in the form of sand (2.5-5.0 mm / 0.6-2.5 mm / 0.15-0.6 mm = 3/5/2)
600 g of Portland cement with a high alkali content
300 ml of NaOH solution, such that the Na2O equivalent content of the cement is adjusted to 4%.

After being kept for the first 24 hours in moulds in a high humidity tank, and then out of the moulds and in water for 24 hours, the test bars are immersed in the autoclaving water and autoclaved for 5 hours at a relative pressure of 0.15 MPa (127°C). The test specimens are left in the autoclaving water for 18 hours while they cool to ambient temperature (20°C). Variation in the length of the test bars before they are put into the autoclave and after cooling is measured.

The autoclaving conditions selected for the test are similar to those proposed by Nishibayashi et al. These test, however, suggests that 2.5cmx2.5cmx28.5cm test bars be made using a ratio of sand/cement of 2.25, a water/cement ratio of 0.45, and the cement to be alkali-enriched at 1.5% Na_2O equivalent.

Our tests demonstrated that increasing the section of the test bars to 4cm x 4cm and the Na_2O equivalent content of the mortar to 4% allowed differentiation of the aggregates according to their reactivity. More particularly, the choice of a 1.5% Na_2O equivalent content for the Nishibayashi test has already been challenged as insufficient, by Hooton and Rogers (4) and by Fournier et al (5).

MICROSTRUCTURAL STUDY OF AUTOCLAVED MORTARS

The MEB study of mortars made with Chambon and Sudbury aggregates (Figures 7 to 12), tested by autoclave accelerated test using the above conditions, demonstrates the formation of gels similar in appearance and composition to those observed in pathological cases of alkali-aggregate reaction in structures.

Several gel facies have been observed:

- smooth-textured amorphous crackled gel,
- amorphous crackled gel, smooth textured or exhibiting start of recrystallisation,
- crackled and recrystallised gel, with microreniform or finely honeycombed texture,
- gel with finely honeycombed texture, sprinkled with lamellar tufts, often rosette-shaped.

They confirm that autoclaving produces an acceleration in the alkali-aggregate reaction processes of aggregates, without modifying the products formed:

The formation of gels is more frequent and widespread in the case of Chambon aggregate than in the case of Sudbury aggregate, in keeping with the kinetics of reaction of each aggregate and the expansion measured in the mortar.

In the case of Sudbury aggregate, gels are found on the circumference of the aggregates or in the imprints of the aggregates. In the case of Chambon aggregate, gels are also found in the CSH of the paste and in vacuoles in the mortar where they end up migrating.

VALIDATION OF TEST AND COMMENTS

At the present time, 52 aggregates have been tested by autoclave accelerated test using the method proposed. The results obtained are shown in Table 3 and compared with all data currently available for these aggregates.

The number of tests is not sufficient to permit reliable statistical correlation linking characterization by autoclave test with that by other accelerated tests or by experience of the in-situ use of aggregates. It nevertheless permits a distinction to be made between the approximate values of two critical threshholds which split the length variations measured by the autoclave test into three different classes:

- the class of size variation values greater than or equal to 0.30%, corresponding to potentially reactive aggregates characterized as such by all characterization tests,

AGGREGATE	PETROGRAPHY	STRUCTURE DETERIORATION CASES	AUTOCLAVE ACCEL. TEST expansion (%)	MORTAR-BAR TEST 6 months exp (%)	CONCRETE PRISM TEST 6 months exp (%)	MICRO-BAR TEST	KINETIC TEST
FR - T6336	Flint		0.593		potentially reactive	0.25	
FR - CHAMBON	Micaschist, crypto-quartz, carbonates	observed	0.518	0.138	0.169	potentially reactive	potentially reactive
ANT - CBB2	Metamorphicized eruptive rock	observed	0.466				
ANT - CDP	Quartzitic microdiorite + microgabbro	observed	0.401				
FR - AFROBRG	Limestone with inter-grained silica	observed	0.394	potentially reactive	potentially reactive		potentially reactive
FR - PCTRN	Limestone with inter-grained silica	observed	0.393	potentially reactive	potentially reactive		potentially reactive
B - OG1-7/20	Siliceous sandstone with mica	observed	0.373		potentially reactive		
FR - LA			0.367		> 0.208		
B - OG2-14/20	Limestone with inter-grained quartz	observed	0.366		potentially reactive		
USA - T4894	Flint		0.361		potentially reactive	0.22	
B - OG1-2/7	Siliceous sandstone with mica	observed	0.360		potentially reactive		
B - OG2-4/14	Limestone with inter-grained quartz	observed	0.359		potentially reactive		
FR - PCOPL	Opaline quartz		0.356				
FR - LDSB			0.345	0.371	potentially reactive		potentially reactive
FR - LDGR			0.339	potentially reactive	> 0.072		potentially reactive
FR - LVDR			0.312	0.114			potentially reactive
CAN - SPRATT	Limestone with inter-grained silica	observed	0.307	0.313	0.128	potentially reactive	potentially reactive
FR - LTRROB	Quartite with crypto-quartz and silica		0.306				
FR - LC			0.306		> 0.042		potentially reactive
FR - PCBR	Quartzite	observed	0.303	potentially reactive			
FR - LCHB2	Micaschist with carbonates	observed	0.300	potentially reactive	potentially reactive	potentially reactive	potentially reactive
FR - VSPR4			0.296				
FR CBTBOUAFL	Siliceous rock	observed	0.284				potentially reactive
ANT - CBB1	Metamorphicized basaltic lava	observed	0.284				
FR - LCVX			0.274	0.020	0.038		potentially reactive
FR - CBTBRX	Quartzite	observed	0.253	potentially reactive	potentially reactive		potentially reactive
FR - T4981	Siliceous rock		0.225	potentially reactive		0.13	
CAN - SUDBURY	Quartzitic sandstone + gabbro	observed	0.204	0.027	0.073	potentially reactive	potentially reactive
ANT - CMVER	Microgabbro with feldspar and quartz		0.202				
CAN - POSTDAM light	Quartzite	observed	0.181	0.021	0.083	potentially reactive	potentially reactive
ANT - CBB4	Metamophicized basaltic lava	observed	0.171				
FR - LGRD			0.166	0.024	0.048		potentially reactive
ANT - SMB2	Alterated rock with feldspar and quartz	not observed	0.126				
FR - VGNR3			0.111				
FR - CAMPBRN			0.108				non reactive
CAN - PITTSBURG	Dolostone with clay impurities	observed	0.107	0.050	0.273		
FR - LB			0.105	0.019			
FR - CFNBL	Crystilisazed quartz	not observed	0.089				
CAN - POSTDAM dark	Quartz sandstone + carbonate cement	not observed	0.076	0.020	0.038		
FR - T113H	Pure calcareous limestone	not observed	0.062	non reactive	non reactive	0.05	
FR - OMYA	Pure calcareous limestone	not observed	0.061	0.013	0.011	non reactive	non reactive
FR - PIZZ	Limestone with quartz	not observed	0.059	non reactive	non reactive		non reactive
FR - CBTLBCL		not observed	0.037				non reactive
ANT - CMDF	Alterated rock with feldspar and quartz	not observed	0.031				
ANT - SMB3B4	Alterated rock with feldspar and quartz	not observed	0.027				
ANT - CBB3	Eruptive rock	not observed	0.025				
ANT - CHHI	Alterated rock with feldspar and quartz	not observed	0.022				
ANT - CHH2	Alterated rock with feldspar and quartz	not observed	0.021				
FR - AFRBOUL	Pure calcareous limestone	not observed	0.019				
ANT - CBTMDF	Alterated rock with feldspar and quartz	not observed	0.014				non reactive
ANT - CBTHH1	Alterated rock with feldspar and quartz	not observed	0.013				non reactive
ANT - SMV2	Alterated rock with feldspar and quartz	not observed	0.013				

TABLE 3 : Characterization of aggregates

- the class of values lower than 0.30% and greater than or equal to 0.15%, corresponding to potentially reactive aggregates characterized as such by at least one characterization test,
- the class of values lower than 0.15%, corresponding to non-reactive aggregates, characterized as such by all characterization tests.

In this classification, we must mention a single failure, concerning the Canadian Pittsburg aggregate, which has caused problems in structures and is characterized as potentially reactive by the test on concrete. The expansion obtained by the autoclave accelerated test is 0.10%, which places this aggregate in the non-reactive aggregates class.

This failure may be explained in the first place by the mineralogical nature of the aggregate (dolostone with clay inclusions), which results of a alkali-carbonate type reaction in the presence of alkalis. It is likely that this type of reaction is not favoured under autoclaving conditions. It should be noted that the dolomitic nature of this aggregate makes it impossible to characterize by means of the micro-bar test or the kinetic chemical test.

Tests on a large number of aggregates are in course at present, with a view to testing the boundaries of validity of the autoclave test and determining the reliability of the length variation threshholds permitting the aggregates to be identified. The autoclave test offers two major advantages over that procedure:

- the simplicity of the equipment needed (witch apart from the laboratory equipment needed for standardised cement tests, is one autoclave),
- the speed of characterization (only three days from mixing of the paste).

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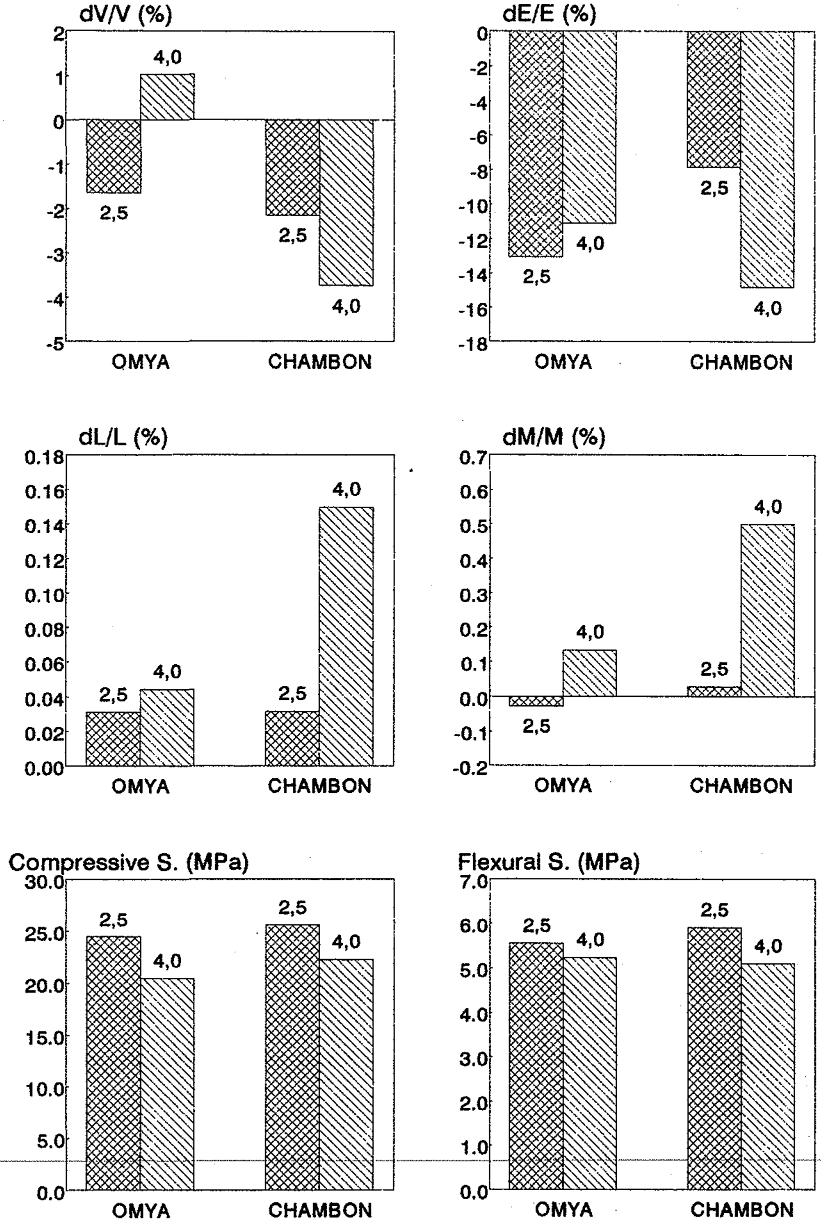

FIG 1 : Influence of the Na2O equivalent content on the behavior of mortar bars submitted to autoclave accelerated test

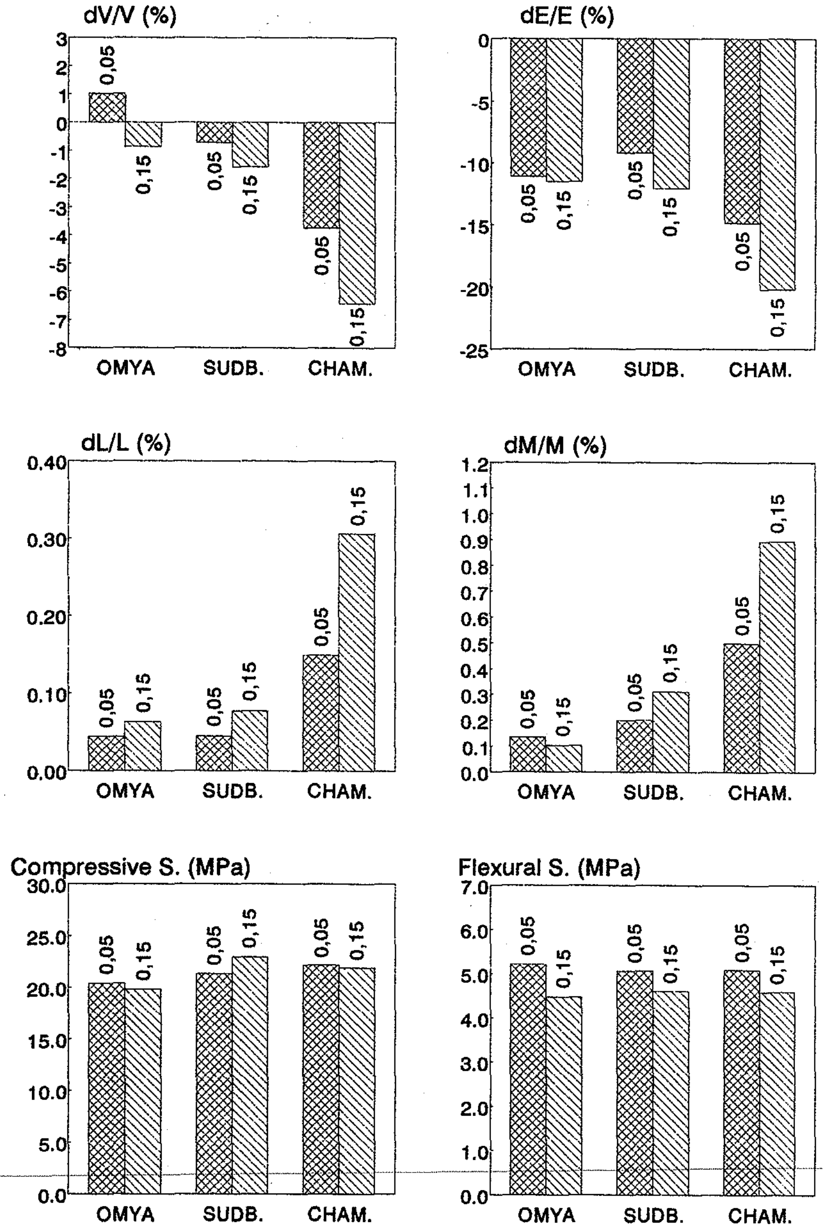

FIG 2 : Influence of the autoclaving pressure (temperature) on the behavior of mortar bars submitted to autoclave accelerated test

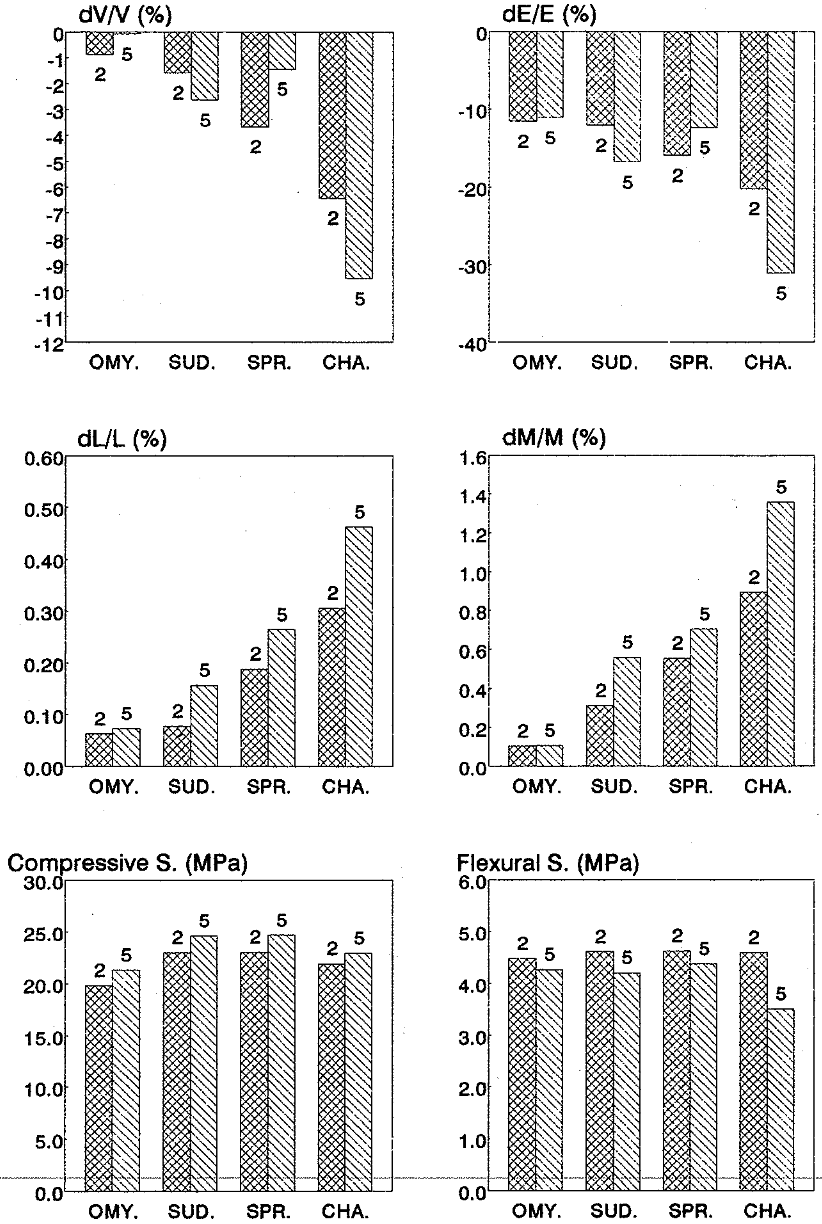

FIG 3 : Influence of the autoclave curing time on the behavior of mortar bars submitted to autoclave accelerated test

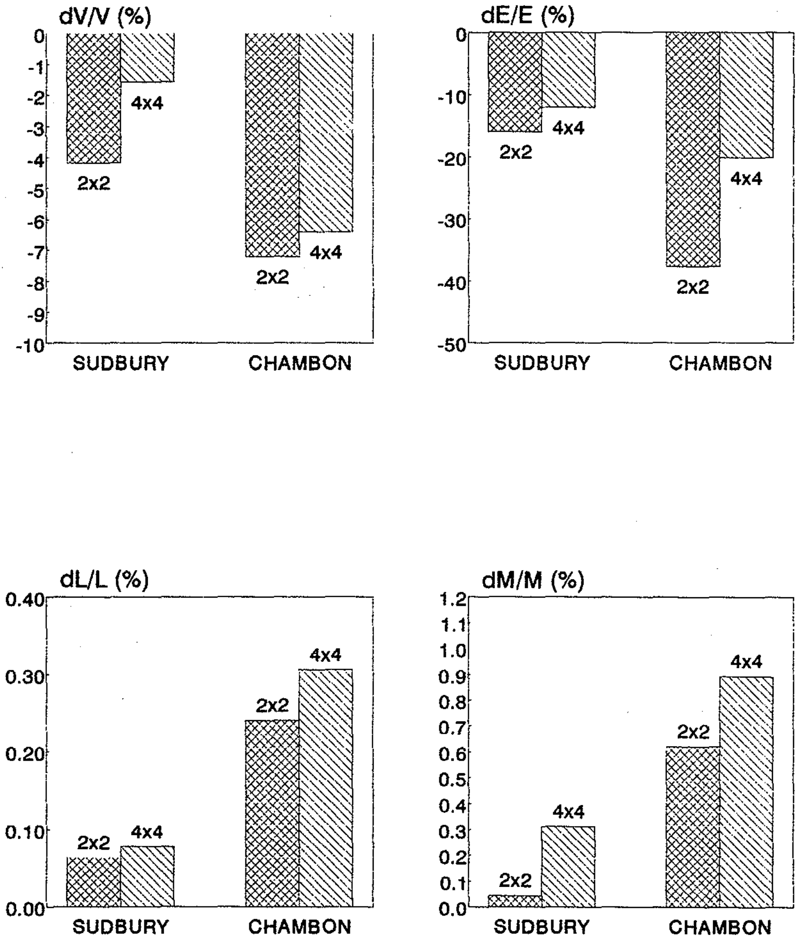

FIG 4 : Influence of the section of mortar bars on the autoclave accelerated test behavior

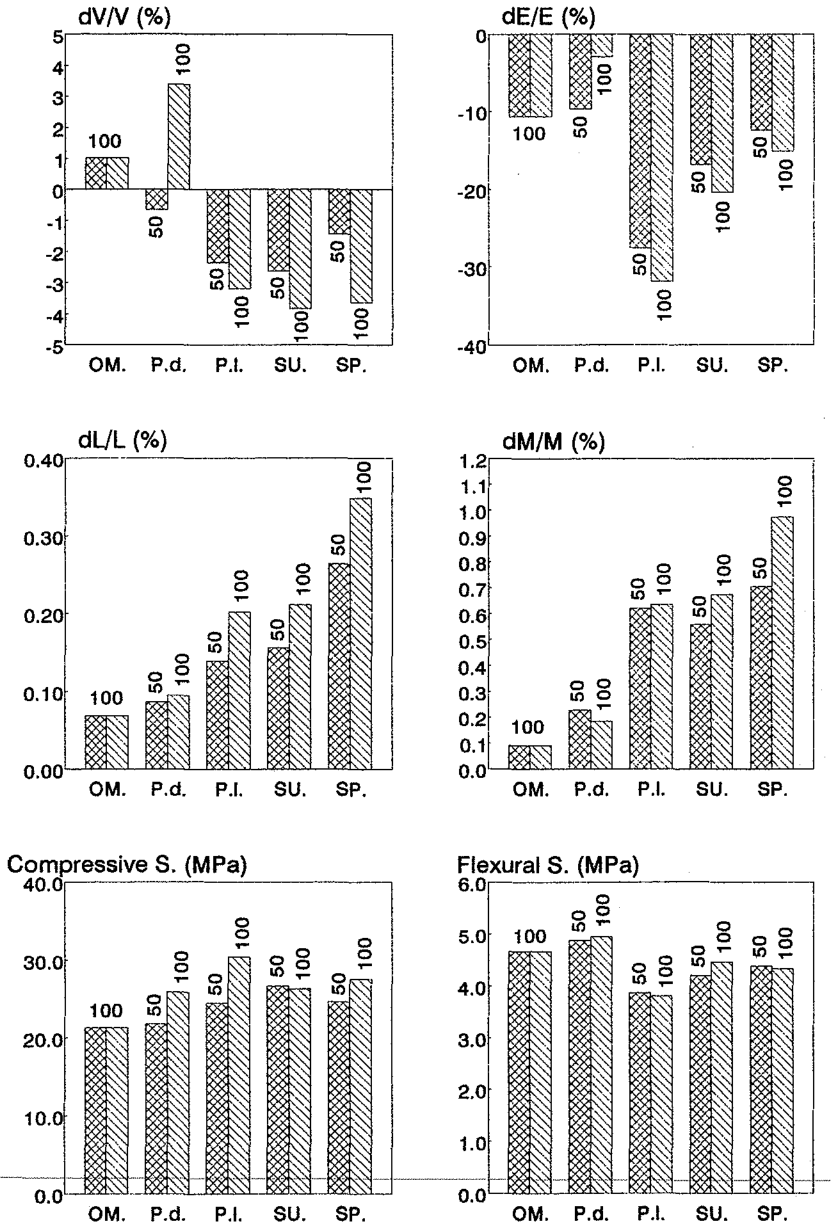

FIG 5 : Influence of the aggregate relative quantity on the behavior of mortar bars submitted to autoclave accelerated test

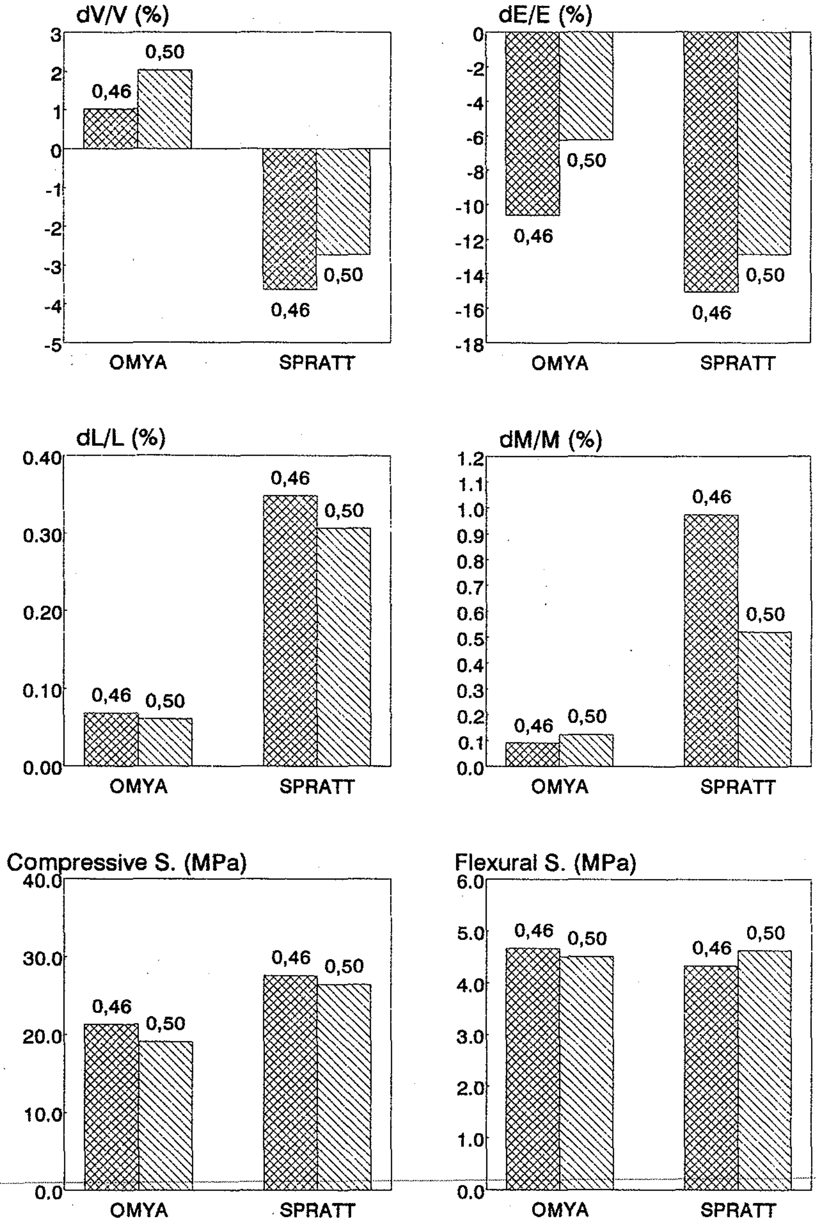

FIG 6 : Influence of the water/cement racio on the behavior of mortar bars submitted to autoclave accelerated test

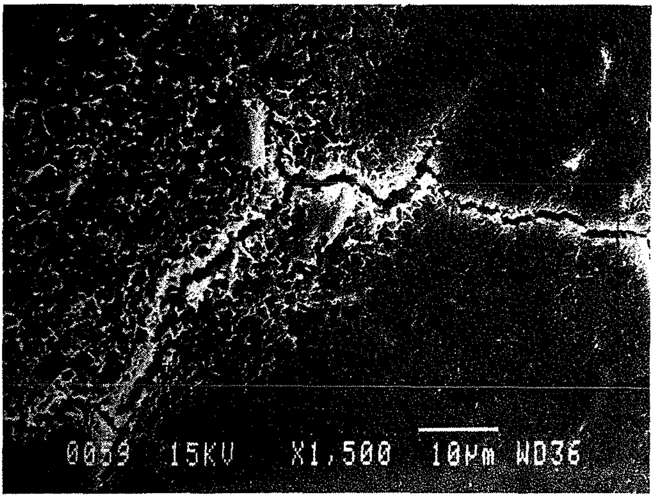

FIG 7 : Sudbury aggregate autoclaved mortar (x1500), start of recrystallisation of cracked amorphous gel

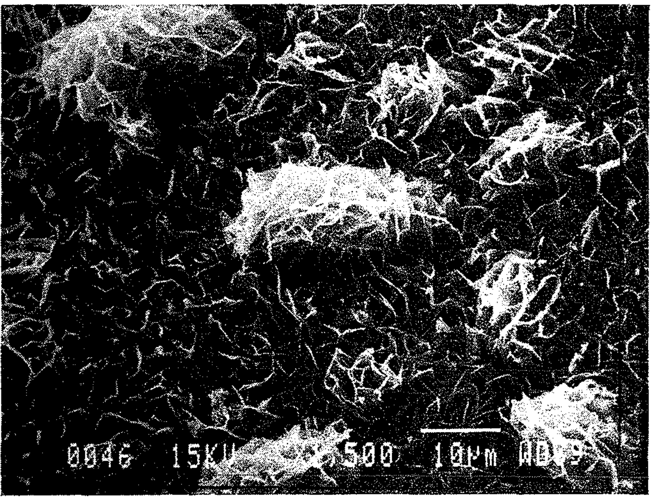

FIG 8 : Sudbury aggregate autoclaved mortar (x1500), rosette-shaped gel

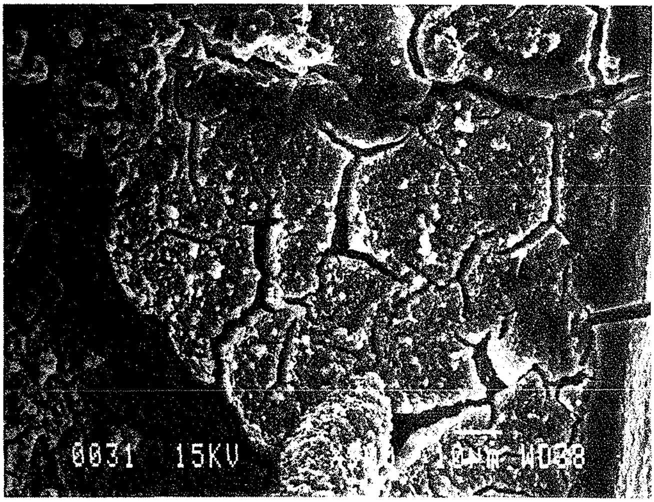

FIG 9 : Chambon aggregate autoclaved mortar (x900), gel with microremiform texture

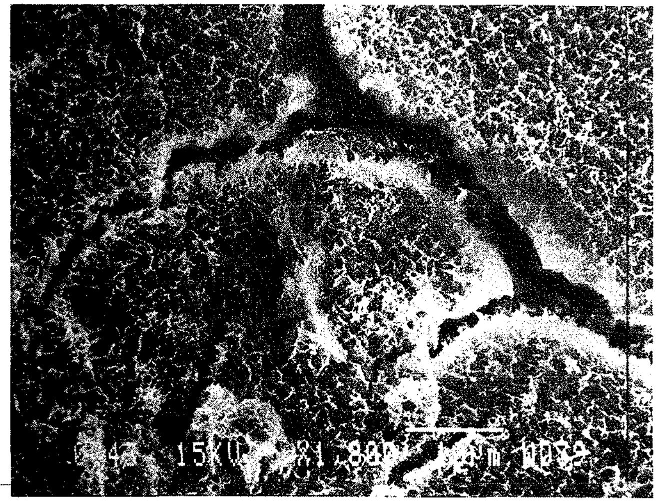

FIG 10 : Chambon aggregate autoclaved mortar (x1800), gel with fine honeycombed texture

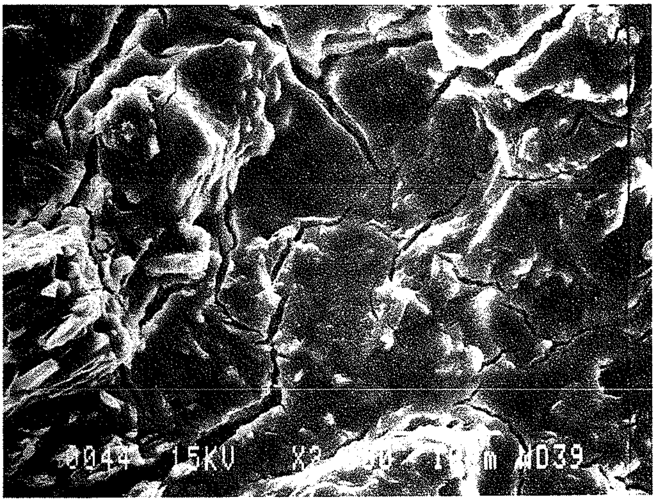

FIG 11 : Chambon aggregate autoclaved mortar (x2000), **CSH covered with** smouth-textured cracked amorphous gel

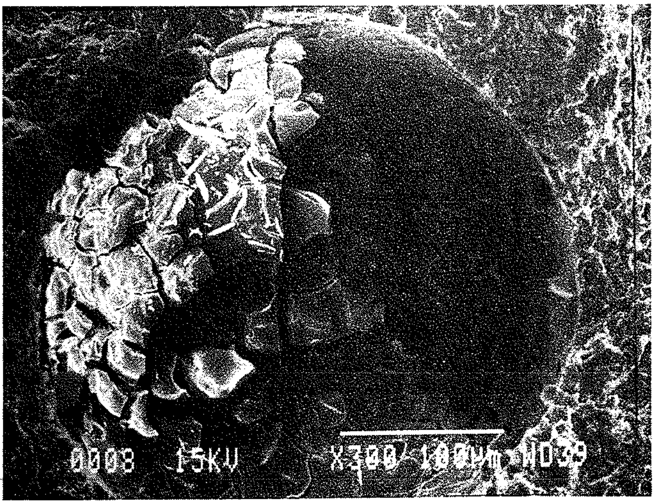

FIG 12 : Chambon aggregate autoclaved mortar (x300), **void filled with** smouth-textured cracked amorphous gel