AN AUTOCLAVE MORTAR BAR TEST FOR ASSESSING THE EFFECTIVENESS OF MINERAL ADMIXTURES IN SUPPRESSING EXPANSION DUE TO AAR

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An accelerated autoclave mortar bar method is used to evaluate the effectiveness of one ground granulated blast furnace slag, two condensed silica fumes and three different pulverized fly ashes in the presence of two very alkali-silica reactive aggregates: a siliceous limestone and a rhyolitic tuff. The results were compared to those obtained with the CAN/CSA -A23.2-14A Concrete Prism Method and the ASTM C 9-Proposal-P 214 Accelerated Mortar Bar Method. Good correlation was obtained between the three methods using a limit of 0.1% expansion in the autoclave test.

INTRODUCTION

The use of mineral admixtures is one of the most popular solutions proposed to suppress expansion due to the alkali aggregate reaction (AAR). However, with a given reactive aggregate, one cannot use just any mineral admixture in any proportion. Most reactive aggregates behave differently, thus laboratory testing is called for. This paper discusses the ability of an autoclave mortar bar method for assessing the effectiveness of mineral admixtures in the presence of two very alkali-silica reactive aggregates from Canada, a siliceous limestone (Spratt, Ottawa) and a rhyolitic tuff (Beauceville, Quebec). These aggregates reacted expansively in a number of structures in Ontario (Rogers and Hooton (1)) and Quebec (Bérubé and Fournier (2)).

MATERIALS AND METHODS

The expansive behavior of these two very reactive aggregates has been investigated in combination with six mineral admixtures: one ground granulated blast furnace slag (GBFS), two condensed silica fumes (CSF) and three pulverized fly ashes (PFA). The chemical composition of all admixtures and cements used are given in Table 1. The CSF's and PFA's were chosen in order to have very different alkali contents.

Mortar bars tested by autoclave were made with the two aggregates and various mineral admixture contents at a water/cement of 0.5. The mixture characteristics are given in Table 2. The samples were tested according to the slightly modified procedure of the ASTM Standard Test Method for Autoclave Expansion of Portland Cement (C151) (3). The mortar bars, with the alkali content increased to 3.5% (Na_2O equiv.) of the mass of cement (cement A, high alkali), were cured for two days in a moist cabinet (23°C), then for 5 hours in the autoclave under low temperature-pressure conditions (130°C-0.17 MPa or 25 psi). These test conditions were proposed by Fournier et al. (4). These authors evaluated the potential alkali-silica reactivity of 40 carbonate aggregates produced in the St. Lawrence Lowlands (Quebec). Afterwards, the method was also applied to more than 60 other siliceous and silicate aggregates and the results are reported at this conference by Bérubé & Fournier (5). A very similar autoclave testing method was first proposed by Tang et al. (6) who also tested mineral admixtures (Tang and Han (7)).

TABLE 1 -- Chemical composition of mineral admixtures and cements

	PFA			CSF		GBFS	CEMENT		
	A	B	C	A	B	A	A	B	C
Oxide (%)									
SiO_2	42.20	53.90	32.59	94.17	74.60	36.60	20.53	20.66	20.05
Al_2O_3	21.60	20.90	17.93	0.21	0.59	8.00	5.46	4.62	4.78
Fe_2O_3	27.60	3.52	5.94	0.32	6.54	0.67	2.50	3.11	3.38
TiO_2	-	-	1.17	0.00	0.00	-	0.25	0.28	0.26
MnO	-	-	0.03	0.07	0.38	-	0.05	0.05	0.04
MgO	0.92	1.11	4.12	0.47	1.56	13.70	2.79	2.31	2.32
CaO	1.87	12.00	20.70	0.50	0.40	37.20	63.33	61.76	62.94
Na_2O	0.66	2.74	8.08	0.00	1.68	0.44	0.25	0.20	0.26
K_2O	2.55	0.50	0.72	1.17	2.97	0.31	1.14	0.82	0.42
P_2O_5	-	-	0.59	0.04	0.00	-	0.15	0.26	0.06
Cr_2O_3	-	-	0.01	0.00	0.03	-	0.00	0.01	0.07
SO_3	1.10	0.09	1.48	0.12	0.81	3.97	2.84	2.98	2.75
LOI	1.85	0.57	0.96	2.77	7.34		0.90	2.82	3.00
Total	100.35	95.33	94.32	99.84	96.90	100.89	100.19	99.88	100.33
Alk.(Na_2O eq.)	2.34	3.07	8.55	0.77	3.63	0.64	1.00	0.74	0.54
*Avail. Alk. (%)									
Na_2O	0.36	1.18	6.01	0.23	0.33	0.18			
K_2O	1.00	0.20	0.58	0.60	0.94	0.21			
Na_2O eq.	1.02	1.31	6.39	0.62	0.95	0.32			

* According to ASTM C311

In addition, CSA concrete prisms were made as a reference with a total cement + admixture content of 350 kg/m^3, rather than 310 kg/m^3, and with a fixed water/cement of 0.5, rather than controlling the slump. The medium alkali cement B was used for most concrete mixtures, with the alkali content boosted to 1.25% Na_2O eq. of the mass of cement by adding NaOH to the mix water (cement B + NaOH) (Table 2). A low alkali control was also made with the cement C (0.54% Na_2O eq.), without the addition of NaOH (Table 2).

The two tests above were also compared with the Accelerated Mortar Bar Method ASTM C9-Proposal-P214. The bars were made in accordance with ASTM C227, with a fixed water/cement of 0.5. Some tests were also made with the alkali content of the bars increased to 1.25% (Na_2O eq.) of the mass of cement (cement A + NaOH) because the higher the initial alkali content, the higher was the expansion obtained by Bérubé & Duchesne (8) after 14 days of immersion when using mineral admixtures. These mix characteristics also appear in Table 2.

The microstructure of the reaction products developed in the mortar bars during the autoclave treatment was studied under the SEM (JEOL JSM-840A equipped with an EDXA). These products were compared with those found in the concrete prisms as well as in the mortar bars tested in the ASTM C9-Proposal-P214 Method.

TABLE 2 -- Mix characteristics of the samples tested

Parameter	Autoclave mortar bar	Mortar bar ASTM C9-P214	Concrete prism CSA A23.2-14A
w/c	0.5	0.5	0.5
(Cement + min. adm.) / aggregate	1 : 2.25	1 : 2.25	-
Cement content	-	-	350 kg/m^3
Mineral admixture (% cement repl.) (% weight)	CSF: 0, 5, 10, 15% PFA: 0, 20, 40% GBFS: 0, 35, 50%	CSF: 0, 5, 10, 15% PFA: 0, 10, 20, 30, 40% GBFS: 0, 35, 50, 65%	CSF: 0, 5, 10% PFA: 0, 20, 40% GBFS: 0, 35, 50%
Cement alkali content (Na2O eq.)	A+ NaOH: 3.5%	A: 1.0% A+NaOH: 1.25%	B+NaOH: 1.25% C: 0.54% (low alk.)
Storage conditions	Pressure: 25 psi Temp.: 130°C	Immersion NaOH 1N, 80°C	100% RH Temp.:38°C
Time limit	5 hours	14 days	1 year
Criterion	Exp. <0.1%	Exp.<0.1%	Exp.<0.04% or < low alk. control

RESULTS AND DISCUSSION

Evaluation of the autoclave mortar bar method for assessing the effectiveness of mineral admixtures

The expansion values for all testing methods with the two reactive aggregates are given in Table 3. This table presents the expansion of mortar bars after 5 hours of autoclaving (25 psi), and after 14 days in NaOH 1N at 80°C. The results of concrete tests after 6, 12 and 18 months are also given. Silica fumes showed a pessimum effect by 12 months for the concrete made with the rhyolitic tuff. This behavior was not due to alkali leaching because it was not observed with the Spratt limestone. Fly ashes did not show a pessimum effect, which is usual for this type of admixture because the minimum content used in the concrete prism (20%) was too high to show this effect.

Figure 1 compares the expansion values of the different combinations (cement+admixture) tested in the autoclave mortar bar method (5 hours) and in the concrete prism method (18 months). A 0.1% limit for the autoclave test is in quite good agreement with the results from the reference concrete prism test according to an expansion limit ≤ the one obtained for the low alkali control at 18 months. However, three admixture-cement combinations (Fig.1) that are non-reactive in concrete according to this limit induced excessive expansion in the autoclave test. These combinations contain 20% PFA-B(Spratt and tuff) and 10% CSF-A (Spratt). On the other hand, one combination with 40% PFA-C expanded just a little in the autoclave mortar bar in the presence of the rhyolitic tuff (0.082%) while it produced high expansion in the concrete prism test (0.142%-18 months).This mixture contained the highest alkali content (60% cement with 3.5% Na_2O equiv. + 40% PFA-C with 8.55% Na_2O equiv.). The low expansion in the autoclave test may suggest that alkali content may also exhibit pessimum behavior, with the expansion decreasing when the alkali content of the mixture exceeds a certain threshold. A similar behavior has been observed by Bérubé et al. (9) for concrete prism tests made with the same aggregate, when increasing the cement content.

TABLE 3 – Expansion values

AGGREGATE		RHYOLITIC TUFF					SPRATT LIMESTONE				
METHOD		CAN/CSA A23.2-14A			AUTO-CLAVE	ASTM C9-P214	CAN/CSA A23.2-14A			AUTO-CLAVE	ASTM C-9 P214
MIX		Percent expansion at different ages					Percent expansion at different ages				
		6m	12m	18m	5h	14d	6m	12m	18m	5h	14d
0% control		.152	.243	.250	.279	.513	.161	.276	.301	.324	.323
CSF-A	5%	.015	.322	.368	.230	.200	.010	.183	.232	.165	.143
	10%	-.009	.006	.015	.108	.054	.000	.007	.013	.139	.080*
	15%	-	-	-	.046	.040	-	-	-	.009	.035
CSF-B	5%	.096	.333	.368	.261	.270	.036	.221	.257	.262	.273
	10%	-.005	.048	.097	.151	.120	.000	.019	.055	.176	.119
	15%	-	-	-	.064	.049	-	-	-	.013	.068*
PFA-A	10%	-	-	-	-	.274	-	-	-	-	.225
	20%	-.011	.009	.023	.130	.120	.003	.011	.027	.170	.141
	30%	-	-	-	-	.055	-	-	-	-	.052
	40%	-.010	-.013	-.011	.050	.035	-.025	-.029	-.034	.073	.030
PFA-B	10%	-	-	-	-	.257	-	-	-	-	.221
	20%	-.011	.004	.013	.220	.110*	-.013	-.006	-.002	.200	.100*
	30%	-	-	-	-	.047	-	-	-	-	.040
	40%	-.011	-.014	-.014	.045	.032	-.014	-.016	-.016	.055	.037
PFA-C	10%	-	-	-	-	.457	-	-	-	-	.327
	20%	.166	.237	.261	.211	.375	.076	.134	.149	.326	.391
	30%	-	-	-	-	.337	-	-	-	-	.349
	40%	.063	.121	.142	.082	.289	.034	.081	.095	.167	.351
GBFS-A	35%	-.004	.035	.043	.116	.140*	.002	.007	.016	.118	.126
	50%	.004	.007	.006	.059	.039	.000	-.002	-.003	.052	.040
	65%	-	-	-	-	.027	-	-	-	-	.017
low alk. control		-.003	.002	.001	-	-	.000	.010	.014	-	-

* 1.25% Na_2O eq. mass of cement

Figure 2 compares the expansion values obtained in the concrete prism test and in the ASTM C9-Proposal-P214 mortar bar test. Five admixture combinations non-reactive in concrete induced excessive expansion (<0.04% after 18 months). However, this number falls to two when using the other criterion (expansion< low alkali control after 18 months). These two mixtures contained 20% PFA-B (Tuff and Spratt aggregates). On the other hand, one combination with 10% CSF-A (Tuff) expanded just a little in the ASTM C9-Proposal-P214 (0.054%) while it produced expansion just higher than the low alkali control (0.015%) after 18 months. The mixture with 40% PFA-C (Tuff) is the only one which expanded more than its low alkali control after 18 months in the concrete test method, and less than 0.1% in the autoclave test. The mixture with 10% CSF-A (tuff) presents the same behavior when tested in the ASTM C9-Proposal-P214 test.

The autoclave mortar bar test as well as the ASTM C9-Proposal-P214 mortar bar test are considered powerful screening tools but cannot be used for rejecting job cements because they are overly severe on numerous innocuous mixtures containing innocuous aggregates (Bérubé & Fournier (10),(8)). Nevertheless,the autoclave mortar bar method led to results in very good agreement with those from the CSA concrete prism test for 22 of the 26 mixtures tested, using expansion limits of < low alkali control (at 18 months) for the concrete test and < 0.1% at 5 hours for the autoclave test. The agreement is similar with the ASTM C9-Proposal-P214 mortar bar method which agreed with the concrete test in 23 cases out of 26. Also, the ASTM method and the autoclave method detected all alkali-reactive mixtures, except for one case each.

Admixture contents required to perform each testing method

The minimum admixture contents needed to meet the performance criterion are given in Table 4 for each mineral admixture and for each aggregate tested. The values were obtained from Figs. 3 (Autoclave), 4 (Concrete) and 5 (ASTM C9-P214) which show the effect of the mineral admixture content on expansion. They apply strictly to the very reactive aggregates tested, and different contents may be needed with other reactive aggregates.

TABLE 4 -- Minimum admixture contents required to meet each criterion

Method	AUTOCLAVE		ASTM C9-P214		CAN/CSA A23.2-14A			
Criterion	5 hours		14 days		18 months			
	Exp. <0.1%		Exp. < 0.1%		Exp. < 0.04%		Exp. ≤ LA control	
Material	Spratt limestone	Rhyolitic tuff	Spratt limestone	Rhyolitic tuff	Spratt limestone	Rhyolitic tuff	Spratt limestone	Rhyolitic tuff
CSF-A	12	11	8	8	9	9	10	10
CSF-B	13	13	11	11	10	>10	~11	>10
PFA-A	33	26	24	22	18	17	21	21
PFA-B	34	35	20	21	16	16	18	19
PFA-C	>40	38	>>40	>>40	>40	>40	>40	>40
GBFS-A	38	39	39	40	29	35	36	44

Two criteria have been suggested for the reference test (CAN/CSA A23.2-14A Concrete Prism Test). An expansion <0.04% after 1 year is the limit suggested in the CSA Standard when testing at 38°C with an alkali content of 310 kg/m^3. In this study, the alkali content was 350 kg/m^3, but a part of the cement was replaced by mineral admixture. The second criterion (expansion ≤ low alkali control) appears more severe but is realistic, particularly when testing cement-admixture combinations containing less than 3.9 kg/m^3 of alkali, which is the specified alkali level in the CSA test; otherwise very low expansions may occur with reactive aggregates whether or not using mineral admixtures. In this study, the limits were extended to more than one year, as suggested by many workers (8).

To meet this criterion for the CSA A23.2-14A method, the mixtures must contain 10% CSF-A, >10% CSF-B, 21% PFA-A, 18-19% PFA-B, >40% PFA-C and 36-44% GBFS-A for controlling expansion. The autoclave test was severe for assessing the performance of the low alkali PFA's. Indeed, to meet the limit of <0.1% expansion, the mixtures must contain 26-33% PFA-A, 34-35% PFA-B, 38% and more PFA-C, 11-12% CSF-A, 13% CSF-B, and 38-39% GBFS-A. Finally, when tested in the ASTM C9-Proposal-P214 mortar test, the mixtures must contain 8% CSF-A, 11% CSF-B, 22-24% PFA-A, 20-21% PFA-B, >40% PFA-C, 39-40% GBFS-A for controlling expansion. This testing method was not severe enough for testing silica fumes.

Microstructure of the reaction products

As shown in Fig.6, the reaction products developed in the mortar bars during the autoclave test were similar to those found in the concrete prisms as well as in the mortar bars tested in the ASTM C9-P214. Massive gels were found lining pores of the cement paste in close association with rosette-like deposits (Fig. 6-A). Such deposits were not observed after autoclaving in the study by Fournier et al.(4). Also, gels with microreticular or sponge-like textures were found in pores of the cement paste with rosette-like deposits along microcracks (Fig. 6-B). These silico-calco-alkaline massive and rosette-like reaction products were also found in the concrete prisms.

CONCLUSION

The autoclave mortar bar method described in this study led to results in very good agreement with those from the CSA Concrete prism test, using a limit of 0.1% expansion in the autoclave test. The results from this test confirmed that the effectiveness of mineral admixtures against ASR depends on their chemical composition, particularly on the alkali content, even though the bars were made with an alkali content of 3.5% Na_2O equiv. of the cement mass. The two accelerated tests remain good tools to estimate the minimum content needed to prevent excessive expansion due to ASR. An autoclave limit of 0.1% gives a conservative measurement of mineral admixture content that should be used in the mixtures. Nevertheless, this method appears severe for testing low alkali pulverized fly ashes. For this type of admixture, the ASTM C9-Proposal-P214 is more appropriate. In the same way, the autoclave method should be chosen for testing silica fumes because the ASTM method gives minimum admixture contents lower than those proposed by the concrete test method. Finally, the reaction products developed during the autoclave treatment was similar to those found in the others testing methods.

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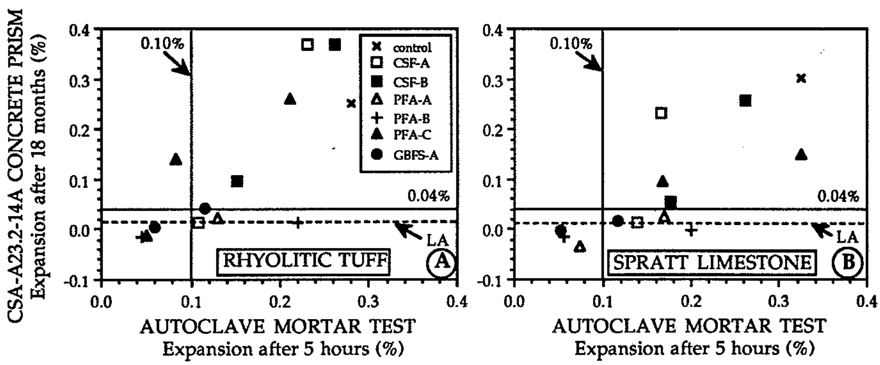

FIGURE 1 -- Relation between the expansion values at 18 months in the CSA concrete test and at 5 hours in the autoclave test. A) Rhyolitic tuff B) Spratt limestone

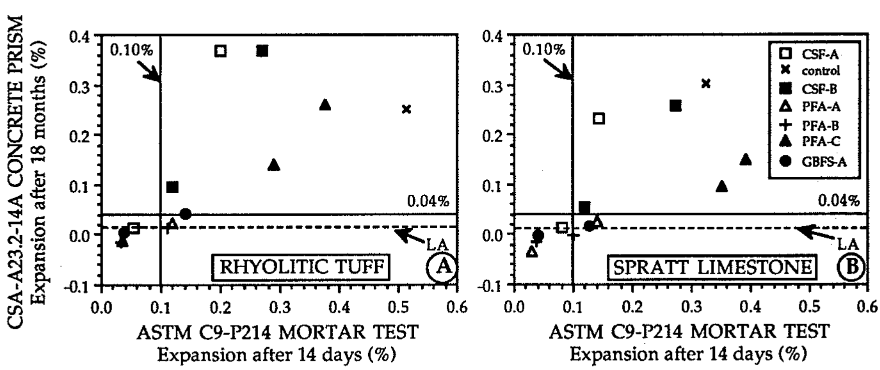

FIGURE 2 -- Relation between the expansion values at 18 months in the CSA concrete test and at 14 days in the ASTM test. A) Rhyolitic tuff B) Spratt limestone

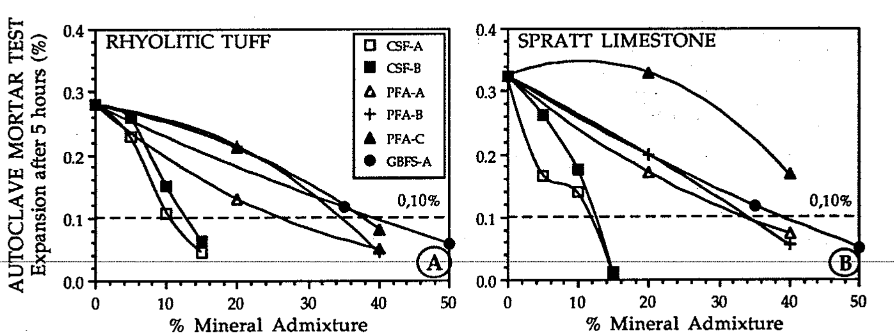

FIGURE 3 -- Expansion values measured at 5 hours in the autoclave mortar bar test as a function of the mineral admixture content. A) Rhyolitic tuff B) Spratt limestone

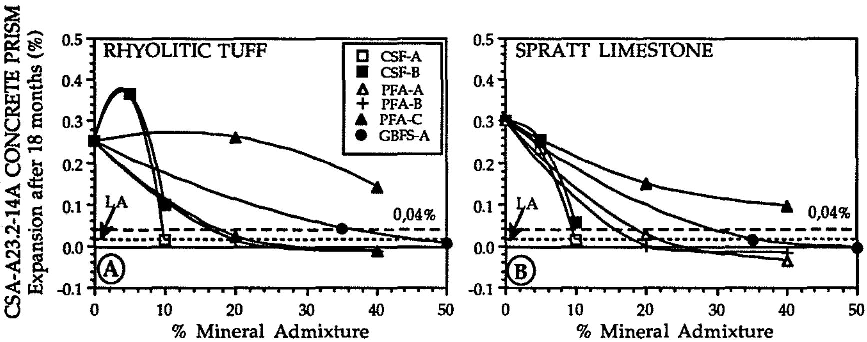

FIGURE 4 -- Expansion values measured at 18 months in the CSA-A23.2-14A concrete prism test as a function of the mineral admixture content. A) Rhyolitic tuff B) Spratt limestone

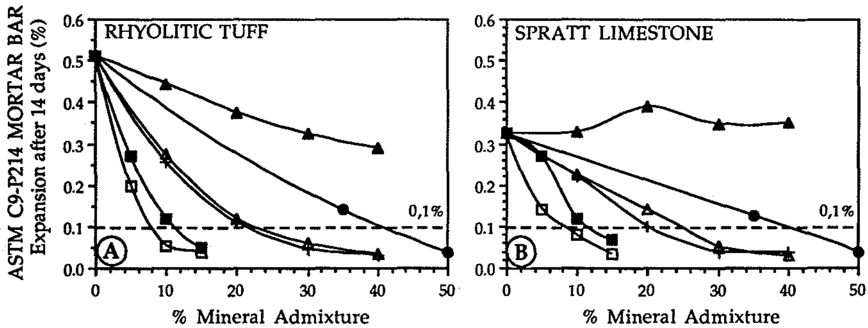

FIGURE 5 -- Expansion values measured at 14 days in the ASTM C9-P214 mortar bar test as a function of the mineral admixture content. A) Rhyolitic tuff B) Spratt limestone

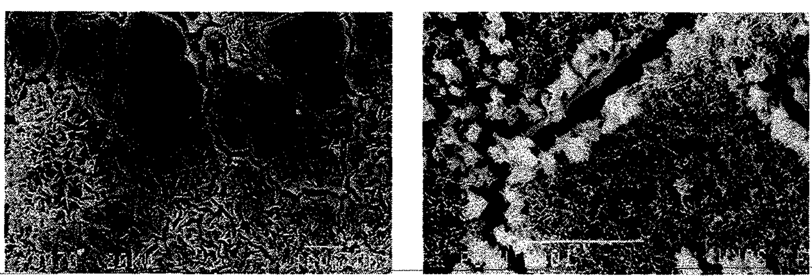

FIGURE 6 -- SEM micrographs of mortar bars at 5 hours of steam curing in the autoclave.
A) Rhyolitic tuff with 5% CSF-A
B) Spratt limestone with 20% PFA-C