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EFFECTIVENESS OF MINERAL ADMIXTURES IN REDUCING ASR EXPANSION

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ABSTRACT

This paper investigates the effectiveness of eight mineral admixtures in reducing the alkali silica(te) reactivity of cement mortar. The admixtures include one class F fly ash, a condensed silica fume, a ground fiberglass, three glass containing waste materials, and two inert fillers (carbon and calcium carbonate). Mortar bar expansion, the change in evaporable water content, the evidence of reaction and the composition of reaction products were studied. It was found that shale is the only reactive component of the sand and the effectiveness of admixtures in reducing alkali-silica reaction depends largely on the chemical composition of the admixtures. Ground fiberglass is very effective in controlling the alkali silica reaction, while inert fillers have only a "dilution effect". It is thought that admixtures reduce ASR expansion by two reactions: (1) pozzolanic reaction that reduces the permeability of concrete and entraps some fraction of the alkali ions; (2) alkali silica reaction that consumes most of the available alkali ions and thereby prevents the reaction between alkalis and aggregate.

Introduction

Concrete deterioration due to alkali-silica reaction(ASR) is a serious problem throughout the world. However, the mechanism of the reaction is not well understood. One theory explains the ASR as following: (1) OH^- attacks the reactive aggregate and provokes its dissolution, (2) dissolved silica(te) reacts with alkalis (Na^+ or K^+) to form alkali-silica gels, (3a) expansion of concrete is due to the osmotic pressure generated by alkali silica(te) gels which are confined within a semipermeable membrane of cement paste[1]; (3b) the expansion is a consequence of the formation and subsequent widening of cracks due to mechanical pressure exerted by the reaction products[2][3]; (3c) the expansion of concrete depends on the type of reaction products, i.e. swelling alkali-silica gel or non-swelling lime-alkali-silica gel. Expansion will occur only when the swelling alkali-silica gel is formed. The Ca^{++} ion concentration in concrete controls the formation of the type of reaction products[4][5]. Different theory has been proposed by Chatterji[6], the main points of which are delineated as follows: (1) During the reaction, OH^-, Na^+, K^+ and Ca^{++} ions penetrate reactive grains. The rate of penetration is determined by the size of the ions in solution. (2). More Ca^{++} ions are left in the liquid phase because their size is larger. (3) During the penetration, some fraction of the silica is set free to migrate away from its original site. (4) The concentration of Ca^{++} in the environment controls the rate at which dissolved silica diffuses out of the grains. (5) An expansion occurs if more materials penetrate a reactive grain than silica migrates out of the grains. There are many other theories proposed by different researchers[7][8] and it seems none of these theories are generally accepted. Despite the complexity of the alkali-silica reaction, mineral admixtures such as condensed silica fume (CSF) and fly ash (FA) have been used successfully to control the ASR expansion. The various proposed mechanisms of control can be summarized as follows[6][8-14]. (1). As a result of pozzolanic reaction between mineral admixtures and cement hydrates, the permeability of the cement paste is decreased, and consequently the mobility of ions in concrete is reduced. (2). The improved strength developed by the mineral admixtures provides higher resistance to the expansive stresses produced by ASR. (3). Mineral admixtures reduce the alkalinity of the pore solution. (4) Mineral admixtures deplete $Ca(OH)_2$ in the cement paste, (5) The secondary hydrate produced by pozzolanic reaction entraps alkali ions. It should be mentioned that not all researchers agree on all the points summarized here because of different views on the ASR mechanisms.

This study investigated the influence of eight mineral admixtures on the alkali-silica(te) reactivity of cement mortar. These admixtures included pyrolysed sheet molding composites (PSMC) and its components, pyrolysed automotive fluff (PAF), condensed silica fume (CSF) as well as fly ash (FA). PSMC is a waste material obtained from pyrolysis of glass fiber reinforced automotive plastics, and consists of about 55% $CaCO_3$, 35% fiberglass and 10% carbon. It has been proved to be pozzolanic and to increase the strength of concrete[15][16]. The composition of PAF is currently unknown, but the main components of PAF are similar to those in PSMC. In this study, all the above mentioned admixtures have been individually incorporated into cement mortar made with shaly sand, and their influence on ASR has been studied. Four research techniques were applied. First, an accelerated mortar bar expansion method was used to study the ASR expansion. Second, an optical microscope was used to observe the reaction with the aggregate. Third, an interval evaporation method (see test methods for details) was used to study the change in evaporable water content due to ASR. Fourth, an energy dispersive spectroscopy (EDS) microanalysis was used to study the reaction products leached out from the mortar samples during the expansion test.

Experimental Details

1. Materials

The sand used in this study was shaly sand from the Leamington area of Ontario. It was graded according to ASTM C227 Standard. The cement used was ordinary Portland cement (ASTM type I).

The abbreviations and definitions of mineral admixtures used in this study are as follows:

(1) PSMC	is finely ground pyrolysed sheet molding composite. Its composition is about 35% glass, 55% $CaCO_3$, and 10% of carbon.
(2) RG	is finely ground fiberglass originally used in making sheet molding composites. The chemical composition of the glass is given in Table 1.

(3) $CaCO_3$	is an inert filler originally used in making sheet molding composites.
(4) PG	is finely ground fiberglass separated from PSMC, it consists of mostly glass plus a small amount of calcium carbonate and carbon.
(5) C+Ca	is the material left after separating the glass from PSMC and it consists mostly of calcium carbonate and carbon plus a small amount of glass.
(6) PAF	is finely ground pyrolysed automotive fluff (also referred to as automotive shredder residue). The composition of PAF is currently unknown but the major components of PAF are similar to those of PSMC.
(7) CSF	is a condensed silica fume from Ontario Hydro. Its chemical composition is given in Table 1.
(8) FA	is class F fly ash from Ontario Hydro, Its chemical composition is given in Table 1.

TABLE 1.
Comparison of Chemical Composition of Mineral Admixtures and Cement

Oxide	CSF	FA	Glass	Cement
SiO_2 %	92.7	46.79	52-56	20
Al_2O_3 %	0.17	23.99	12-16	6
CaO %	0.17	3.15	16-25	63
Fe_2O_3 %	0.29	16.35	0-0.8	3
MgO %	0.59	0.81	0-5	1.5
SO_3 %	0.36	1.16	-	2
Na_2O+K_2O %	1.11	0.61	0-2	1
B_2O_5 %	-	-	5-10	-
P_2O_5 %	0.06	-	-	-
LOI %	3.24	3.71	-	2

2. Sample preparation

The composition of the control mortar was 1 part cement to 2.75 parts of sand by mass, and the water to cement ratio by mass was 0.5 for all samples. The samples with admixtures were made by adding 10% of the admixture by mass of the control mortar before mixing (mineral admixture to cement ratio was 0.425, water to cementitious ratio is 0.35). Melment L-10 superplasticizer was used to adjust the workability so that all the samples would have the same water to cement ratio and comparable workability.

Mortar bars (2.5 x 2.5 x 21 cm) were made to test ASR expansion and to study the evaporable water. After the expansion tests, three 2.5 cm cubes were cut from each mortar bar for microscopy studies. The rest of the mortar bars were then used for water evaporation study using an interval evaporation method which will be discussed in the next section. 5 cm cubes were also made to test the compressive strength at different ages.

3. Test Methods

An accelerated mortar bar method was used to test ASR expansion. The method is similar to MTO LS-620[17] and NBRI[18] accelerated mortar bar method: the mortar bars were immersed in 1 N NaOH solution at 80 °C. The linear expansion of the samples was recorded every three days for the first 4 measurements and then every week for a total of eight weeks. Comparative evaluation of test methods for assessing the effectiveness of mineral admixtures in suppressing expansion due to alkali-aggregate reaction have been reported by other authors [12][19]. They concluded that the accelerated mortar bar method yields results in good agreement with those of the CSA Concrete Prism Method. It is suggested the accelerated mortar bar method may give a good indication of the effectiveness of pozzolans in reducing expansion in concrete due to ASR.

An interval evaporation method was used to study the evaporable water in mortar samples. The principle of the method is to examine the amount of water evaporated from the samples within certain intervals at different temperature. The test method consists of: a) drying the samples at 65 °C for 24 hours, b) immersing the samples in water for more than 3 days followed by boiling for 6 hours, c) allowing the samples to cool in water for 24 hours and measuring the weight of samples, d) drying the samples according to the conditions given in Table 2. The amount of water evaporated from the samples was measured after each drying interval, and then the cumulative evaporation of water from the samples was calculated.

TABLE 2.
Interval Evaporation Method, Drying Conditions

Drying Intervals	**Drying Temperature (°C)**	**Drying Time (hours)**	**Cumulative Drying Time (hours)**	**Cumulative Evaporation (%)**
D1	23	24	24	Measured and Calculated
D2	65	24	48	
D3	105	24	72	
D4	105	24	96	
D5	105	24	120	
D6	105	24	144	

Experimental Results

1. Effectiveness of PSMC and other mineral admixtures in reducing ASR expansion

The influence of PSMC and other mineral admixtures on ASR expansion is shown in FIG.1. It should be mentioned that for the accelerated test, an expansion in excess of 0.1% at 12 days was considered to be deleterious and this criterion is thought to be equivalent to 0.05% at 1 year in ASTM C227 method[18][19]. The main purpose of this study, however, was to compare the relative effectiveness of different mineral admixtures in mitigating the ASR expansion. Therefore, the absolute value of expansion is less important. The following facts are revealed in Fig 1.:

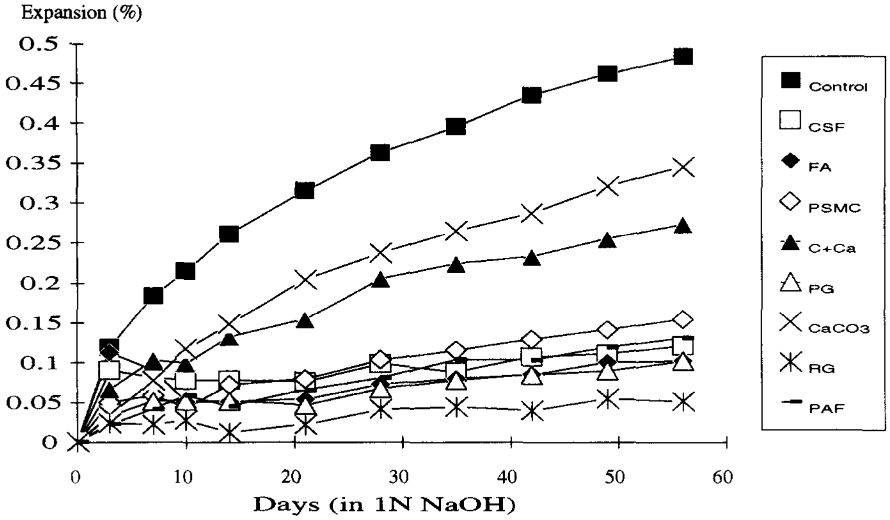


FIG. 1
Influence of Mineral Admixtures on ASR Expansion of Cement Mortar

(a). All the samples with admixtures show less expansion than that of control mortar.

(b). The effectiveness of PSMC and PAF in controlling ASR expansion is comparable to that of CSF and FA. However, the glass is the most important component in PSMC and PAF contributing to the effect. In descending order, the glass content of the admixtures related to pyrolyzed products are RG (100%) > PG > PAF > PSMC > C+Ca > $CaCO_3$ (0%). The effectiveness in controlling ASR expansion, as shown in Figure 1, gives exactly the same order.

(c). Of all the admixtures tested, ground fiberglass is the most effective material mitigating ASR expansion although the SiO_2 content of the glass is much less than that of CSF.

2. Observation of the evidence of ASR by use of optical microscopy

From the microscopic study, it was found that all the samples show some evidence of reaction around sand particles (reaction rim and cracking of the sand particles), but the degree of reaction was different. For the control sample and the sample with inert filler (calcium carbonate), the reaction was much more severe than the other admixture samples. This is in agreement with the expansion results in which the control and $CaCO_3$ samples show greater expansion than those of the other admixture samples. For the samples with ground fiberglass (RG) and CSF, the evidence of reaction was confined merely to a few small particles close to the exterior surface of the samples. There was no sign of reaction in the center portion of the sample. It was also observed that the reaction took place only around shale particles. Shale is not widely used as aggregate for concrete because of its poor physical properties. But in this study, shale was found to be alkali-silica reactive when using a rapid mortar bar expansion method.

3. Chemical Composition of Shale Particles and the Reaction Products Studied by EDS Microanalysis

Table 3 lists the chemical composition of shale and the reaction products of mortar samples as studied by the EDS microanalysis. It can be seen that after alkali reaction, the sodium content of the shale is increased but the silica content is decreased. This indicates that during the reaction, Na^+ ions penetrated into the shale particles while Si has been dissolved and leached out. Alkali-silica gel has been found in the solution in which control mortar was immersed for the expansion test. Similar product was also found in the solution containing CSF samples. It should be mentioned that the composition of reaction products shown in Table 3 was not pure ASR gel but the mixture of NaOH and ASR gel. It can also be seen from Table 3 that the chemical composition of the substance found in the solution (in which mortar samples with ground glass were immersed for expansion test) contained little reaction products since about 90% of the substance is NaOH which is from the 1N NaOH solution. This indicates that alkali silica gel was not formed in the samples with ground fiberglass or it was not leached out.

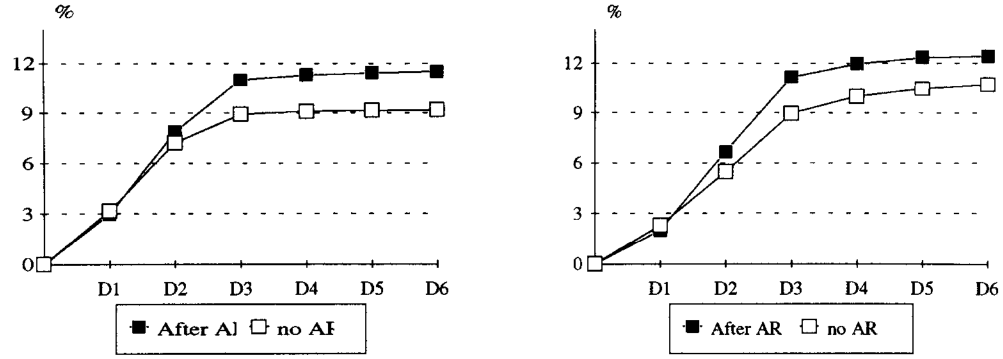
TABLE 3
Chemical Composition of Shaly Sand and Reaction Products Found in Solutions in Which Mortar Samples with Different Admixtures were Immersed for Expansion Test

	Unreacted Shale Particles	**Reacted Shale Particles**	**Reaction Products in Solutions**		
			Control Mortar	**Mortar with Glass**	**Mortar with CSF**
Na_2O	0.14	9.16	28.69	88.26	34.98
Al_2O_3	22.34	25.75	7.43	2.73	2.76
SiO_2	61.05	51.08	57.07	3.77	56.05
SO_2	1.05	2.70	1.95	0.58	0.00
K_2O	3.94	4.96	4.86	1.22	6.12
CaO	4.11	1.06	0.00	1.29	0.00
FeO	7.37	5.29	0.00	2.14	0.10

4. Amount of evaporable water in mortar samples as influenced by ASR

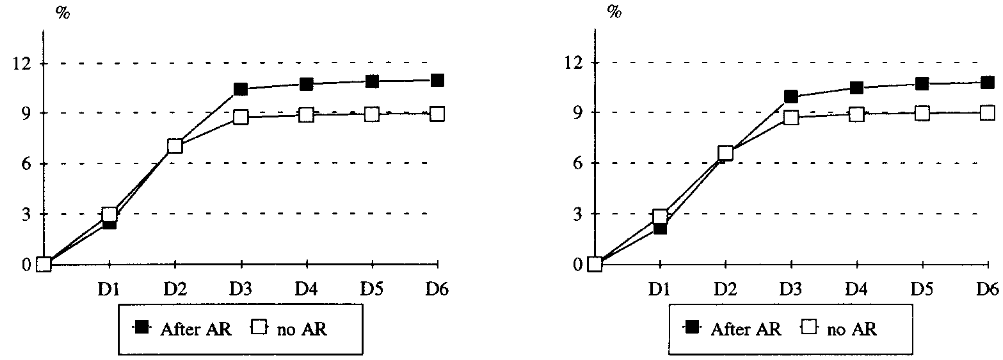
Comparisons of the evaporable water content in samples after ASR testing and samples not subjected to the ASR test are given in FIG.2 and FIG.3. The evaporation curves in FIG.2 show that for mild drying conditions, about same amount of water was removed from AR and non-AR samples. But under elevated drying temperature conditions, more water was removed from AR samples than from non-AR samples, and the amount of total evaporable water was higher for the samples which had undergone the ASR test. The samples with the above mentioned characteristics include the control mortar and the mortar with inert fillers. Except for the CSF specimens, these samples showed higher expansion as indicated in FIG.1.

The evaporation curves shown in FIG.3 are quite different from these shown in FIG.2 Under mild drying conditions, less water was evaporated from the AR samples. At elevated temperature drying conditions, more water came out from the AR samples than from the non-AR samples. The amount of total evaporable water was slightly higher for the samples that experienced the ASR. The samples in this group have one thing in common: they contain admixtures which consist of wholly or partly of ground fiberglass except fly ash, which has, however, a composition similar to that of glass.



(a). Control Mortar

(b). Mortar with CSF



(c). Mortar with $CaCO_3$

(d). Mortar with C+$CaCO_3$

FIG. 2
Influence of ASR on Water Evaporation of Mortar Samples - Type I

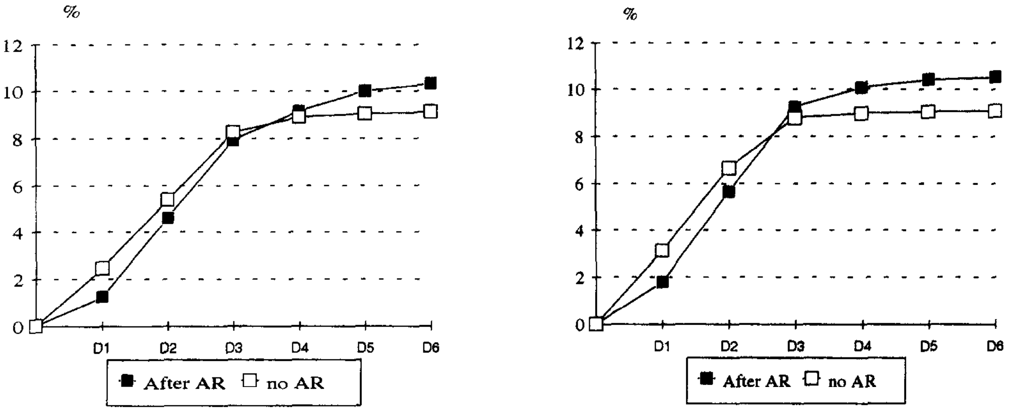
Discussions

1. Accelerated Mortar Bar Method and Concrete in Natural Environmental Conditions.

When using the accelerated mortar bar method, the mortar samples are exposed to 1N NaOH solution at 80 °C temperature. This condition is much more severe than concrete experiences in natural environment conditions. Some aggregates which tested reactive using this method may perform satisfactorily in ordinary service. MTO LS-620[18] suggests that when an aggregate is found reactive using the accelerated mortar bar method, additional studies, possibly using alternative methods, may be appropriate to develop further information on its potential reactivity. The main purpose of this study was to compare the relative effectiveness of different mineral admixtures in reducing ASR expansion; the accelerated mortar bar method was considered to be sufficiently suitable and fast for this purpose.

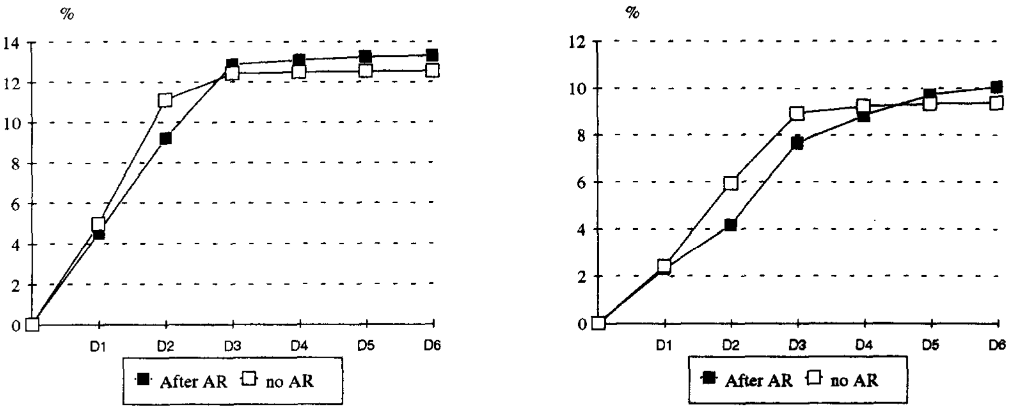
2. Source of Alkalis

Availability of alkalis in concrete is one of the necessary conditions for ASR to take place. Alkalis can come from cement, mineral admixtures and from outside sources. When using a mineral admixture in concrete, it is important to know how the mineral admixtures will influence the alkalinity of the pore solution and many publications have been devoted to this



(a). Mortar with Fly Ash

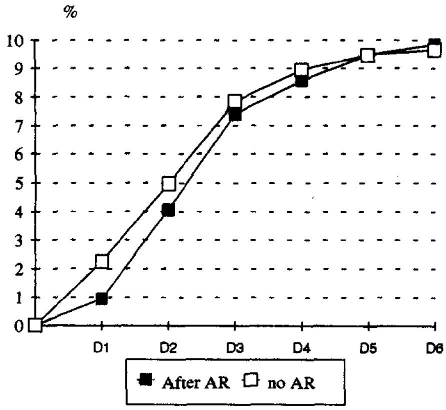
(b). Mortar with PSMC



(c). Mortar with PAF

(d). Mortar with PG

FIG.3
Influence of ASR on Water Evaporation of Mortar Samples - Type II



(e). Mortar with Ground Glass

topic [10][13][14]. However, when using accelerated mortar bar method, alkalis are continuously supplied from the outside source, therefore the alkali content of mineral admixtures is not of primary importance.

3. Evaporable Water Content and ASR Expansion

Evaporation of water from mortar samples is closely related to the pore structure of the mortar. There are generally four types of water in hydrated cement paste (hcp): free water, adsorbed water, interlayer water and chemically combined water[20]. Free water is the water held in large capillary pores. It behaves like bulk water, and is easy to remove. Adsorbed water is the water close to the solid surface. These water molecules are physically adsorbed onto the surface of solids in the hydrated cement paste (hcp). Removing adsorbed water from hcp is more difficult than removing free water. Interlayer water is the water associated with the C-S-H structure. The interlayer water is lost only during prolonged high temperature drying. Chemically combined water is the water that is an integral part of the structure of various cement hydration products. This water can not be removed by drying. Water attracted by alkali-silica gels is mostly adsorbed water which is also difficult to remove. The amount of evaporable water is also influenced by the type of hydrates, but only in interlayer water.

As the results show in Figure 2 and Figure 3, the total evaporable water content in AR samples is higher than in non-AR samples, and the additional amount of evaporable water comes mostly from the adsorbed water. It is thought that this additional amount of water could be the water adsorbed by alkali-silica gel or by the micro cracks generated by the ASR. Therefore, the difference in total evaporable water between AR and non-AR samples, that is, the amount of additional evaporable water(AEW) may serve as an indication for the amount of alkali-silica gel produced by the alkali-silica reaction. The relationship between AEW and ASR expansion is given in FIG.4. As can be seen, if the AEW is less than 1.4%, the expansion of the samples is negligible. This means that ASR did generate some alkali-silica gel in these samples, but the

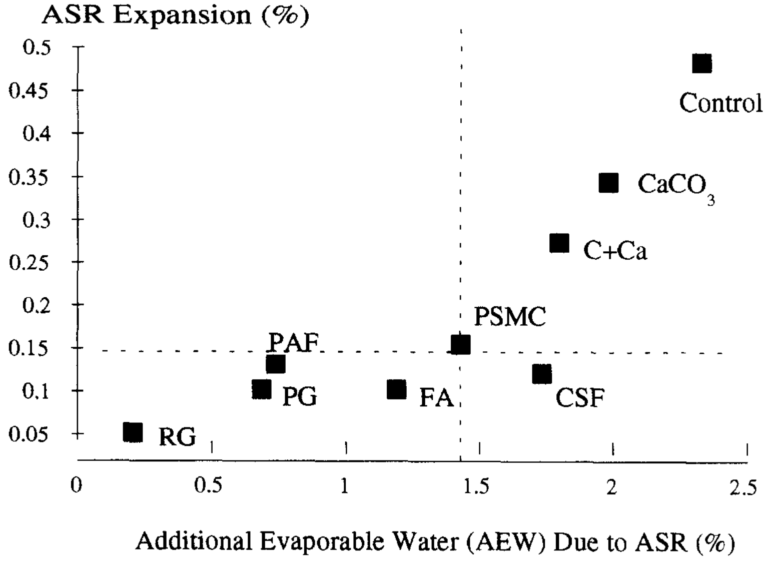


FIG.4
Relationship Between Additional Evaporable Water and ASR Expansion

amount of the gel was not enough to cause significant expansion. When the AEW is over 1.4%, then the ASR expansion increased rapidly with the increase of AEW. (samples with CSF are an exception).

4. Pozzolanic Reaction and Alkali-Silica Reaction

Pozzolanic reaction is expressed by many researchers as following[8][21][22][23]:

$$SiO_2\,(s) + Ca^{++}\,(aq) + 2OH^-\,(aq) \rightarrow xCaO.SiO_2.yH_2O\ (CSH)$$

Alkali-silica reaction can be expressed as following [8]:

$$SiO_2 + 2Na^+(K^+) + 2OH^- \rightarrow Na_2(K_2)SiO_3.H_2O$$

There are similarities between these two reactions. It is possible that in the first reaction some alkali metal ions can be incorporated into secondary CSH hydrates , and it is also possible that lime-alkali-silica gel will form in the second reaction when some Ca^{++} ions are incorporated. Therefore, the products from both reactions could be form part of a spectrum of compositions. It has been suggested that reaction products of different chemical composition (from low viscosity gel to CSH) may be observed in the same concrete[8]. But secondary CSH hydrates can improve the properties of concrete while expansive alkali-silica gel will damage the concrete if produced in a large quantity. In the presence of mineral admixtures and under a strong alkali environment, both pozzolanic and alkali-silica reactions can take place simultaneously. However, when admixtures are present, the alkali-silica reaction is not only between the alkali and the aggregate but also between the alkali and the mineral admixtures. Therefore, the gel produced can be distributed evenly within the concrete and can be accommodated by the concrete. In addition, not all the gels produced are expansive. One important consequence of pozzolanic reaction and alkali-silica reaction is that both of them will reduce the alkali content in the concrete. Another consequence is that both of them will reduce the permeability of concrete and further reduce the free movement of alkali ions. These two consequences will prevent or reduce the potential reaction between alkalis and aggregate.

5. Effectiveness of CSF and Ground Fiberglass in Mitigating ASR

Many researchers have found that CSF is the most effective material to control ASR[11][12] but others have questioned the long term effectiveness of CSF[10]. In this study, it was found that ground fiberglass is more effective than CSF. As shown in Table 3, alkali-silica gel (low viscosity) was found in CSF samples as well as in the control mortar, but no gel was found in glass containing samples. Figure 4 suggests that there was a considerable amount of gels (AEW) in CSF samples, but little in glass bearing samples. Explanations are given as follows:

(1). The optimum amount of admixtures to control ASR differs for every admixture. In this study, all the admixtures were used in the same amount, which may not be the optimum for all of them.

(2). The chemical composition for CSF and fiberglass is different (Table 3). CSF contains mostly SiO_2. Under strong alkali condition and at high temperature, both pozzolanic and alkali-silica reactions are expected to take place in samples with CSF. Ground fiberglass contains not only SiO_2 but also Al_2O_3 and CaO. The presence of Al_2O_3 and CaO lowers the solubility and the rate of dissolution of silica glasses[8]. Once dissolved, alumina reacts instantaneously with lime[24]. Thus the pozzolanic reaction may dominate in the samples with ground fiberglass.

(3). The products of pozzolanic reaction (secondary CSH) can incorporate alkali metal ions into their structure and reduce the alkalinity of the pore solution. At the same time, the secondary CSH hydrates fill the adjacent pores and reduce the permeability of concrete, consequently, reducing the free movement of alkali ions. When there is a continuous supply of alkali from a outside source (as the case of accelerated mortar bar method), the latter effect is more important because it will slow down (if not stop) the supply of alkalis to concrete. The formation of alkali-silica reaction products in a small amount may also reduce the alkalinity of the pore solution and the permeability of concrete. But some of these reaction products are expansive. There is a limit to the amount which the concrete will accommodate without expanding, even when they are distributed evenly. Ideally, the alkali reaction will not result in expansive products. This appears to be the case when using ground fiberglass in which alkali-silica gel were not found. In CSF samples, the amount of alkali-silica gel produced is relatively high, but the expansion of the mortar samples is not as large (Figure 4.). It is thought that the higher strength of CSF samples resist the expansion pressure by the gels.

6. Effectiveness of $CaCO_3$ and the "Dilution Effect".

$CaCO_3$, when added into concrete in finely ground form, may influence the hydration process of cement and consequently the composition of hydration products[25]. However, many researchers believe that $CaCO_3$ is chemically inert to concrete, and the influence of $CaCO_3$ on concrete properties is mainly physical[26][12]. With regard to ASR, the results shown in Figure 1 indicate that the expansion of mortar samples with $CaCO_3$ was reduced, though not as much as that of samples with other pozzolans. Since the solubility of $CaCO_3$ is very low under a strong alkali environment, it is thought that the reduced expansion by $CaCO_3$ is mainly due to the "dilution effect". That is, the addition of 10% mineral admixture to the mortar reduced the unit concentration of reactive aggregate and alkalis in the mortar samples, and therefore reduced the ASR expansion.

Conclusions

(1). Among the mineral admixtures investigated in this study, ground fiberglass is most effective in controlling ASR expansion. The effectiveness of PSMC and PAF is comparable to that of fly ash and condensed silica fume. However, the only effective component of PSMC and PAF in controlling ASR is the finely ground fiberglass. Inert fillers contained in PSMC and PAF such as carbon and calcium carbonate are not effective in controlling ASR in concrete. The lower expansion caused by these inert fillers is believed to be mainly due to the "dilution effect".

(2). In the presence of pozzolanic mineral admixtures, and under strong alkali condition, both pozzolanic reaction and alkali silica reaction can simultaneously take place. These reactions consume alkali ions and reduce the permeability of concrete. The overall effect is to reduce the possibility of detrimental reaction between alkali and aggregate.

(3). Shale is generally not considered as an alkali silica reactive aggregate since it is not normally used in concrete - it tends to deteriorate it under a variety of climatic conditions.

However, shaly sand containing shale in the finer sizes is occasionally found acceptable. This research shows that the deterioration of concrete containing shale may also be partly due to alkali reactivity.

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