ASSESSMENT AT THE LABORATORY SCALE OF SEVERAL FACTORS AFFECTING ASR REACTION, TO BUILD A MODEL VALIDATION DATABASE

Geneviève FORAY - Stéphane POYET - Bruno CAPRA
(LaM – UMLV, Cité Descartes – 5 boulevard Descartes, 77454 Marne la Vallée Cedex 2, FRANCE)
Alain SELLIER
(LMDC - INSA/UPS – Dpt GC, 135 avenue de Rangueil, 31077 Toulouse cedex 4, FRANCE)
Jean-Michel TORRENTI
(IRSN / DICST / Pg – BP 17, 92262 Fontenay aux Roses Cedex, FRANCE)
Hélène TOURNIER
(EDF / DER, Site des Renardières, Route de Sens, Ecuelles, 77818 Moret-sur-Loing, FRANCE)
Eric BOURDAROT
(Direction de l'Equipement, CIH, Savoie-Technolac, 73373 Le Bourget-du-Lac, FRANCE)

ABSTRACT

Expansion and deterioration due to ASR will continue in an affected structure as long as reactives (Mineral, Humidity and Alkalis) are not consumed and reactives are able to diffuse towards reactive aggregates. Management of an affected structure requires therefore to know accurately the level of reaction an the kinetic of reaction at any time. To gain such information, a dynamic exchange between model, experiment at the lab scale, and case study is needed. In this paper, the laboraty scale study associated to the research program is discussed.

ASR is produced using a reactive limestone, a cement with high alkali level, and addition of sodium hydroxide to hydration water. Length, mass, and dynamic modulus of mortar samples kept at 60°C are measured during two years. Characterization test are made as well : compressive strength, mercury porosimetry, initial degree of saturation and MEB-SEM on mortar, X ray fluorescence, soluble silica content and mercury porosimetry on reactive sand. In the first part, influence of reactive particle size and distribution of reactive particle size on the development of AAR is studied. Results show that there is a coupling factor when reactives particles of different sizes are present within the mortar. In a second part, influence of relative humidity is studied. Results are discussed and compared with those found in the literature. A database structure is proposed to store experimental results, experimental frame.

Keywords: Alkali Silica Reaction, experimental work, aggregate size grading, expansion, porosity.

1 INTRODUCTION

Alkali-Silica Reaction (ASR) is a chemical reaction between reactive silica of some aggregates and ions contained in the pore solution of concrete (alkalis, hydroxide and calcium ions) [1] [2]. This reaction requires a sufficient amount of water to take place inside concrete. Two theories are communly used to explain the consequences of this reaction . The firs one suppose formation of some new products, ASR Gel and on the second one suppose volume change of the aggregate. The first one is applied in this work. Reaction products, often called 'ASR gels', have various compositions depending on local chemical equilibrium [2] [3]. Those gels precipitate within the porosity close to each reactive aggregate. When the gel volume near an aggregate is greater than the available porosity, a stress distribution appears [4]. As chemical reactions continue, the gel volume increases and the stress distribution incrases as well.

Main consequences of this pathology are cracking, swelling and loss of mechanical characteristics (tensile and compressive strengths, Young's modulus...) [5] which may impair durability and safety of affected structures.

Owners of affected strucures have to develop a preventive maintenance to overcome ASR problems. Structural works have to be scheduled, keeping in mind that the structure must remain cost effective. Moreover, a daily maintenance is often needed to ensure service life of the strucutre and users security.

The effect of the size of aggregates and aggregate size range have already been studied [6,7,8,9,10]. Nevertheless, the available results are contradictory. The effect of water incomes were studied as well [11, 12, 13], but little work was done on the history of the incomes [14].

The aim of this paper is to develop a database structure to store experimental results issued from our test as well as those issued from litterature. This experimental database will then be linked with a modelling one.

Our approach is based on a three scale modelling link to experimental work at each scale [15]. The first scale consists in understanding and modelling local chemistry phenomena, the second scale takes into account interactions between cement paste and reactive aggregates on one hand, and on the other hand interactions between different aggregates of a representative elementary volume (REV) [16,17]. The third and last modelling level is the macro-mechanical one which allows to evaluate state of damage on concrete structures [18].

2 EXPERIMENTAL WORK

2.1 Background

The experimental work at the laboratory scale focus on two distinct problems :

- Effect of aggregate size and aggregate size grading on the development of ASR.

- Effect of exterior relative humidity kept constant or balancing between high and low level. The balance rythm may be either long (28days) or short (14 days).

2.2 Reactive aggregates

Aggregates used come from two sources. NR is a non reactive sand R is a reactive limestone. Both sources of aggreates are first washed and sieved. Three granular class are defined :

A small one	C1, 0,08<diameter<0,16mm
An intermediate one	C2, 0,63<diameter<1,25mm
A large one	C3, 2,50<diameter<3,15mm

By combination of those a model sand with a discrete granular composition is build (Fig. 1). Level of reactive aggregates in the sand vary from 0 to 75%. Seven types of reactive sand are build, each one having a distinct reactive size grading, but the same overall grading.

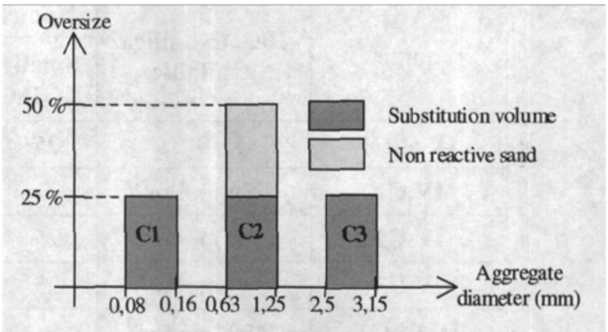

Fig. 1: aggregate size grading of the sand

In the first set, there is only one class reactive, i.e. 25% of the aggregate are reactives and comes either from C1, C2 or C3 (Table 1). In the second set, there is a combination of two reactive classes, i.e. 50% of the aggregates are reactives and come either from C1+C2, C2+C3, C1+C3 (Table 1).

Table 1 : List of aggregate size grading and reactivity

name	Reactive silica available	Non reactive sand (%)			Reactive sand (%)		
		Small C1	Inter. C2	Large C3	Small C1	Inter. C2	Large C3
IV CRéf	0	25	50	25	-	-	-
IV C1	336 mol/m^3	-	50	25	25	-	-
IV C2	336 mol/m^3	25	25	25	-	25	-
IV C3	336 mol/m^3	25	50	-	-	-	25
IV C1C2	2*336 mol/m^3	-	25	25	25	25	-
IV C1C3	2*336 mol/m^3	-	50	-	25	-	25
IV C2C3	2*336 mol/m^3	25	25	-	-	25	25
IV C1C2C3	3*336 mol/m^3	-	25	-	25	25	25

In the third set, There is a combination of three reactive classes, i.e. 75% of the aggregates are reactive C1+C2+C3 . The last set is the reference with no reactive aggregates.

2.3 Experimental frame

The mortar is composed of the model sand , cement, water, and sodium hydroxyde. The alkali are dissolved in water 30 minutes before the test. The formulation is : Cement=537.8 kg/m3, Water=268.8 kg/m3, Sand=1612.8 kg/m3, Na_2O_{eq}=2.4%.

The test program was done twice with two different cements. As the results were the same for both, only one type of cement is presented here.

Specimens are demolded after 10 days at 20°C, wrapped in plastic foil and kept at 95% RH to avoid loss of water. Specimens are then kept at 60°C and 95%RH for reactive aggegate study and target humidity for water incomes study.

Length, mass and dynamic modulus are measured on a regular basis. Length change, mass change and dynamic modulus change with respect ot the reference population are then computed.

Compression strength, porosity, SEM-EDAX observations are made ponctually.

Some separate tests are made on the reactive sand to get a better understanding of its reactivity.

2.4 Characterisation of the aggregate

R aggregates have a bimodal porosity (3 and 130nm) while reactive one have a single porosity band (120nm). The aggregates were analysed by X ray fluorescence and thermogravimety. The results are similar for the three size observed [12].

The silica dissolution tests are contradictory. The first set of experiments shows that silica quantity increases with aggregate size (from 200 to 600ppm at 70days). The duplicate shows that intermediate and large aggregates produce the same level and rate of dissolution. MIP before and after the test shows that dissolution of silica creates a new aggregate porosity in the domain 0,02/2,5 micron, this for all size of aggregates.

2.5 Description of the aggregate

The reactive aggregate is a limestone. Two different forms of siliceous materials are identified. At the macroscopic level siliceous nodules similar to cherts that have the composition and texture of chalcedony and/or opal and mainly consists of microcrystalline silica. At the microscopic level, « diagenetic silica », forming a « grid » between 4 and 10µm [19] .

3 AGGREGATE SIZE AND AGGREGATE GRADING

3.1 Experimental results with 25% substitution by reactive aggregates, influence of diameter.

The large C3 and intermediate C2 aggregate formulation show similar expansion caractericstics up to 0,12 and 0,15% at 60 days (Fig. 2). The formulation with small C1 R aggregates shows very little deformation (less than 0,015%) (Fig 2). There is more dispersion in small R aggregates results. We lack data to define a relation between estimated specific surface of aggregate and RAS deformation. There is mainly two behaviours for the aggregates studied here, small diameter gives almost no deformation / Others produce large deformation.

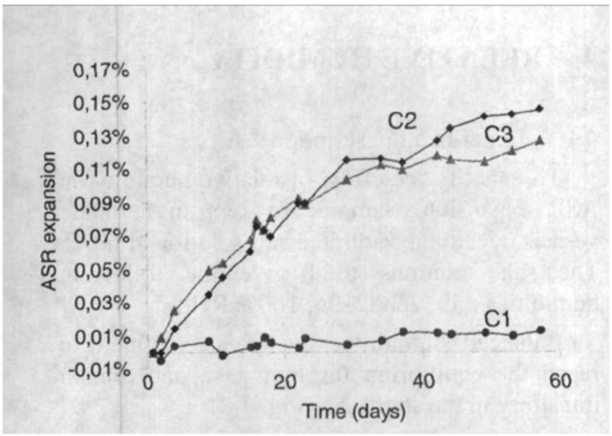

Fig. 2: ASR expansion for several reactive sand grading and 25% of reactive elements

3.2 Experimental results with 50% and 75% substitution by reactive aggregates, influence of grading

Figure 3 compares deformation measured on a single reactive aggregate to deformation recorded when two grades of reactive aggregates are used. The quantity of reactive silica is doubled, while alkali quantities remain equal. Indeed, the deformations measured increased but are much less than the addition of individual (At 60 days : intermediate : 0,15% - large : 0,13% - combination 0,18%, i.e. 70% of the sum of single grade deformation) .

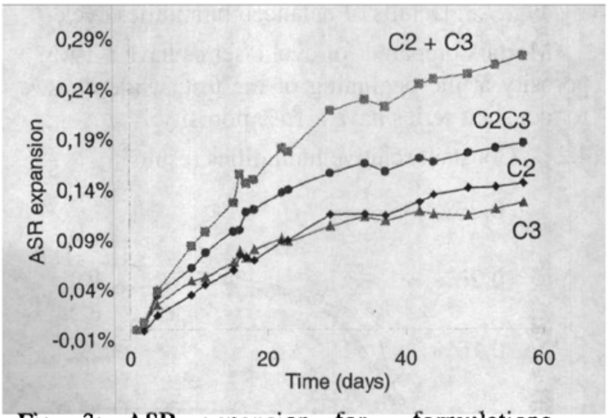

Fig. 3: ASR expansion for formulations, combination effects

Those results are confirmed when the quantity of reactive silica is increased from 50 to 75% by substituing small R aggregates to a sand already including intermediate and large ones . Deformation are 30% less when the small aggregates are included.

Figures 4 shows the RAS deformation measured when small aggregrates are combined with large ones. The addition of small C1 aggregates as a reducing effect on deformation (large alone : 0,14% - combination : 0,12%, i.e. 70% of the sum or 20% less than the large C3 alone).

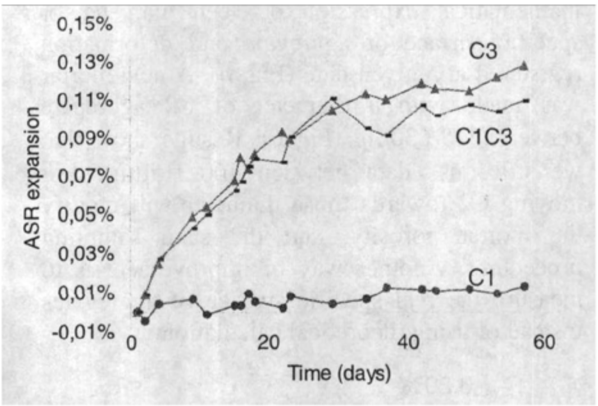

Fig. 4: ASR expansion for formulations, combination effects with the small class

Measurements of the dynamic modulus on sample confirms the expansion measurements.

3.3 MIP results on mortar

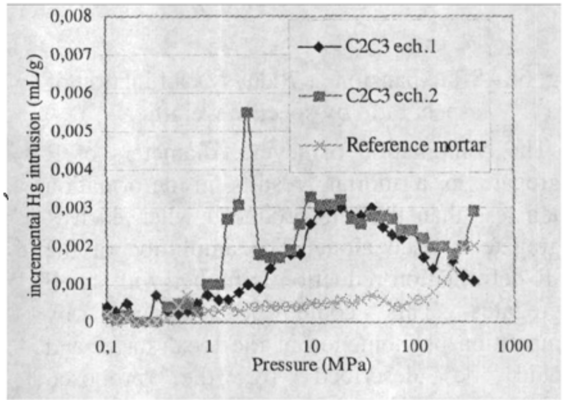

Fig. 5: Compared MIP analysis of reactif and reference mortar.

Mortars have a 14% Porosity at the beginning of the test. The repartition between micro, meso and macro porosity is 55/24/21%. A porosity domain, 0,02/2,5 µm (assuming that pore are spheric), is measured in mortar including reactive sand (Fig 5). This porosity is similar to the porosity measured on aggregates alone after the Si dissolution test. Further tests should confirm wether this porosity is associated to ASR development.

3.4 Conclusion on aggregate size and grading

The aggregates studied produce more or less mortar deformation depending on the diameter used. Our kind of aggregates like those studied by monteiro [20], jin [19] and guedon[10] shows almost no deformation for diameter below 160 micron. A different behaviour is found when studying opaline, deformations increase below a threshold diameter of 50 micron [10].

We need more data to find a simple mathematical expression between diameter or specific surface of aggregate and deformation registered at a given date (Fig. 6). A new mortar was made with a diameter of C2 aggregate between 315-630µm (Fig 6). Results show that we still miss data between 160-315µm). But moving C2 towards those diameter will modify the mortar porosity, and the sand sampling procedures. Another way of improvement is to measure the real specific surface of aggregates instead of using theoretical calculation.

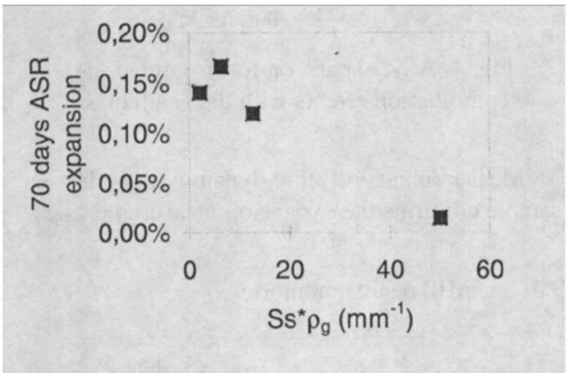

Fig. 6: ASR expansion at 70days as a function of aspect ratio by specific weigth

The combination of several diameters of R aggregate in a mortar, results in deformation much less than the one produced when each R aggregate is used alone. The amplitude of the ASR deformation reduction is higher with small aggregates. This could be explained by competition phenomenon at the local stage, and should be described by the proposed modelisation [16]. For example in a small and large R aggregates mortar, alkali could react either with the small or the large aggregate. Reacting with the small one, it will not be available any more for the large one. Moreover, the ASR gel produced will have more space (interfacial transition zone) to spread in and will produce less pressure on the cement paste. This will result in less overall ASR deformation.

MIP curves of granular material and mortar show a new porosity domain, after dissolution test and after 70days ASR expansion. This domain stands in the range of 0,02 to 2,5 µm and its origin has to be determined.

4 RELATIVE HUMIDITY

4.1 Experiment description

To quantify the effect of relative humidity on ASR expansion. Sample are kept in hermetic vessels over some saturated salt solution at 60°C. The salt solutions used give the following humidities : 59, 76, 82, 96, 100% RH.

Balanced humidities cycles are defined to reach the equilibrum for long test, and remain transitory in the short one (Fig. 7,8)

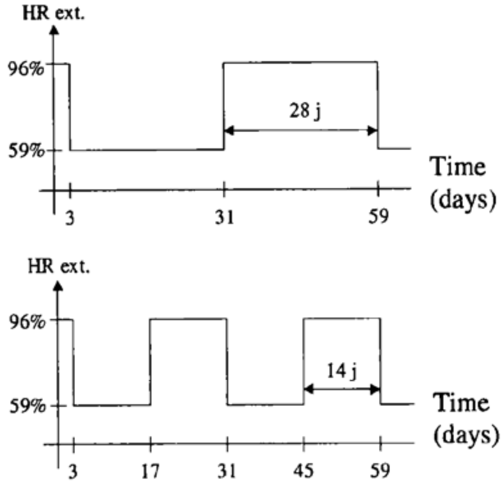

Fig. 7,8: Details of balanced humidities cycle

Mortars prepared for cyclic series have a 19% porosity at the beginning of the test, while does for constant series have a 15% porosity.

4.2 Constant relative humidities results

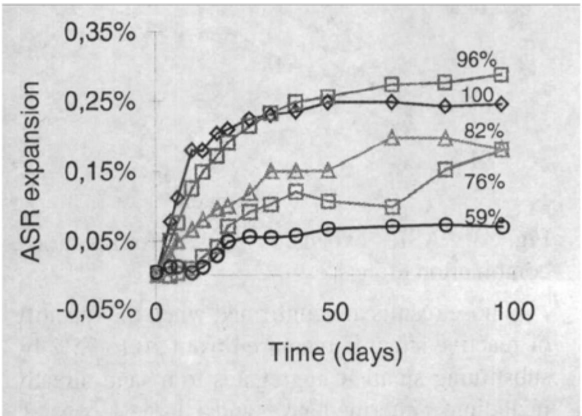

Fig. 9 : ASR expansion under imposed constant relative humidities

When water availability increase, ASR expansion increase (Fig. 9). A cross-over between 96% and 100% RH curve is observed. This is due to condensation of water droplet along 100% RH samples, that cause alkali leaching [14,15].

The step decrease below 82% in ASR deformation seems to be associated with loss of capillary water from the sample. The cutting of capillary channel, slow down ionic diffusion and lower the probability that all reactive element are present to dissolve silica and form ASR gel.

All samples conserved above 82% gain weigth, those at 82% HR are at equilibrum, those lower loose weigth [12].

4.3 Balancing humidities

Our first goal is reached, mass variation of the two series are very different. Consequences on ASR deformation should be present.

Mass variations of samples show that the first cycle produce more amplitude in weigth than the following (Fig 10). Short cycles are scattered in the wetting part. Long cycle seems to procuce structural modification in mortar : The weigth gain gets lower as the number of cycle increases, while the minium weigth reached increased.

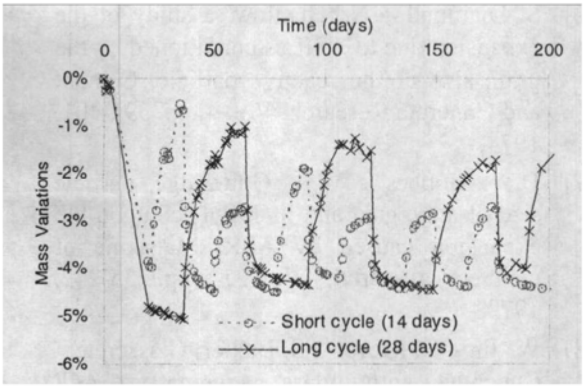

Fig. 10 : Mass variation, humidities cycles.

At equivalent "wet time" (2short/1long, 4short/2long) the ASR expansion are first equal, then the curves separate and long cycles show 10% more expansion (Fig. 11, 0,21% and 0,23%).

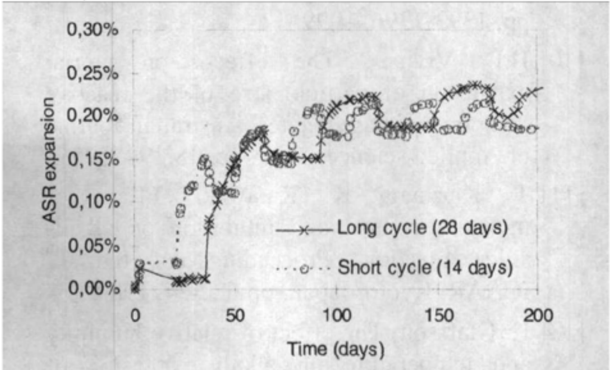

Fig. 11 : ASR expansion, humidities cycles.

A sharp decrease in net ASR expansion (0,04%) is recorded at each drying branch (Fig12). Shrinkage of the cement paste matrix is already taken as the deformation plotted are those recorded on reactive samples minus those of a reference sample. It migth be that the ASR gel formed losses water as well. This loss produces a volume diminution, that in turn relieves pressure on the matrix, and causes an expansion decrease.

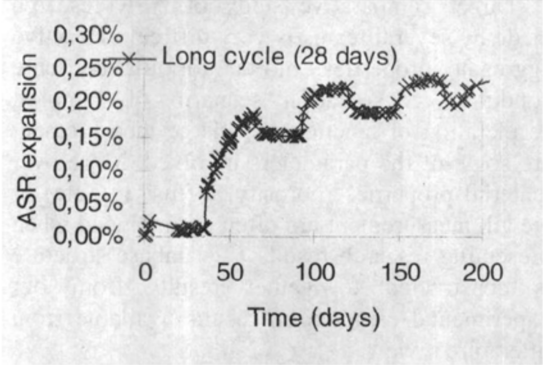

Fig. 12 : ASR expansion, Long 28 days cycles.

4.4 ESEM observations

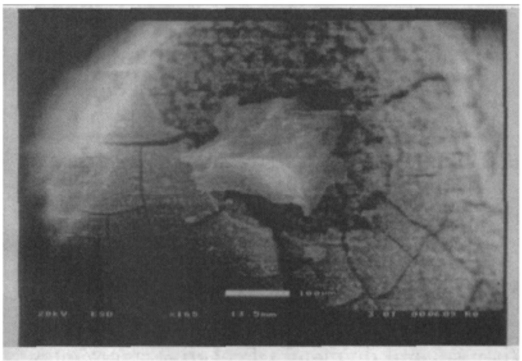

Fig. 13 : ESEM observation, of ASR gel.

ASR gel (Fig13) observed is localised in a 50 µm pore near an aggregate. EDAX analysis gives the following ratio (Ca/Si = 1.05, Na+K/Si = 0.3). A map cracking is observed on the surface of ASR gel. The crack patterns densifies during the observation as the sample losses some more water.

4.5 Conclusions on relative humidities

Our experiment results show that ASR deformation increases when water availability increases. This confirms experiments made by several researchers [11,12,13]

The balancing cycle results show that equivalent wet time does not apply. The history amplitude and frequency of water incomes modifies the deformation recorded.

A 0,04% shrinkage is measured at each drying step on RAS deformation. This migth be due to loss of water by the ASR gel itself.

To model accuratly ASR reaction, the availability of water should be taken inot account

5 DATABASE

Direct comparative study of ASR research made by several teams is very difficult. Reactive aggregate properties, mortar formulation, cure condition, conservation scenario of samples, acceleration of reaction, means of measurement are some of the parameters involved. More over material properties (porosity, diffusivity, etc) or weigth measurement are often not included when presenting research results. A database structure is constructed to gather results from our experimental campain an results available from litterature review.

6 CONCLUSION

This experimental study confirms that aggregate size grading and aggregate size greatly influence ASR deformation.

Each diameter of reactive aggregates has its own ASR gel formation kinetics. Simple relation does not exist between ASR deformation and aggregate specific surface. This will imposed reflexion to build acccurate micro-mecanical model.

When several diameter of reactive aggregates are present together, competition phenomena appear, and result in coupling factor.

Experiments made on water incomes show that water availability greatly influences ASR deformation. To compare ASR deformations data, water availability at the local stage or at least weigth variations of each sample have to be considered.

7 ACKNOWLEDGEMENT

Some of the characterisation tests were made with the facilities of other research laboratories. We acknowledge R. Combes, G2S, UMLV for conducting ESEM observations. We thank as well S. Maximilien, GEMPPM, INSA lyon for the access provided to mercury intrusion porosimetry.

8 REFERENCES

[1] L.S. Dent Glasser – N. Kataoka, The chemistry of alkali-aggregate reaction, Cement and Concrete Research, Vol. 11 n°1, 1981, pp. 1-9.

[2] R. Dron – F. Brivot – T. Chaussadent, Mécanisme de la réaction alcali-silice, Bulletin des laboratoires des ponts et chaussées, n°214, 1998, pp. 61-68.

[3] J. Lombardi – A. Perruchot – P. Massard – C. Larive, Etude comparée de gels silico-calciques produits des réactions alcali-granulats dans les bétons et de gels synthétiques types, Cement and Concrete Research, Vol. 26 n°4, 1996, pp. 623-631.

[4] A. Sellier – B. Capra, Modélisation physico-chimique de la réaction alcali-granulats, apport au calcul des structures dégradées, Revue Française de Génie Civil, Vol. 1 n°3, 1997, pp. 445-481.

[5] K. Okada – S. Nishibayashi – M. Kawamura, Proceedings of the 8th ICAAR, Kyoto, 1989 17-20 July, 885p.

[6] S. Diamond – N. Thaulow, a study of the expansion due to ASR as conditioned by the grain size of the reactive particle, Cement and Concrete Research, Vol. 4, pp. 391-401, 1974.

[7] D.W. Hobbes – W.A. Gutteridge, particle size of aggregate and its influence upon the expansion caused by ASR, Magazine of concrete research, Vol. 31, pp. 235-242, 1979.

[8] W. Jin – C. Meyer – S. Baxter, Glasscrete – Concrete with glass aggregates, ACI Materials Journal, Vol. 97, pp. 208-213, 2000.

[9] C. Zhang – A. Wang – M. Tang – B. Wu – N. Zhang, Influence of aggregate size and aggregate size grading on ASR expansion, Cement and Concrete Research, Vol. 29, pp. 1393-1396, 1999.

[10] H.E. Vivians, The effects on mortar expansion of particle size of the reactive component in aggregates, Australian Journal of Applied science, Vol. , pp. 488-94, 1951.

[11] T. Kurihara, K. Katawaki, Effecs of moisture control and inhibition on alkali-silica reaction, Proceedings of the 8th ICAAR, Kyoto, Japan, pp 622-34, 1989.

[12] H. Olafsson, The effect of relative humidity and temperature on alkali expansion of mortar bars, Proceedings of the 7th ICAAR, Ottawa, Canada, pp 461-65,1986.

[13] F. Tomosawa, K. Tamura, M. Abe, Influence of water content on alkali-aggregate reaction, Proceedings of the 8th ICAAR, Kyoto, Japan, pp 881-85, 1989.

[14] C. Larive, A. Laplaud, O. Coussy, The role of water in alkali-silca reaction, Proceedings

of the 11th ICAAR, Quebec, 2000 11-16 June, 1402p.

[15] S. Poyet, Etude de la dégradation des ouvrages en bétons atteints par la réaction alcali-silice : Approche expérimentale et modélisation numérique multi-échelles des dégradations dans un environnement hydro-chémo-mécanique variable, PhD thesis UMLV, 2003, 238p.

[16] S. Poyet – A. Sellier – G. Thevenin-Foray – B. Capra – J.-M. Torrenti – H. Tournier-Cognon – E. Bourdarot, Modelling of Alakali-silica Reaction, part 1 : Influence on aggregate size range on chemical modelling of ASR, Proceedings of the 12th ICAAR, Pékin, Chine, 2004.

[17] S. Poyet – A. Sellier – G. Thevenin-Foray – B. Capra – J.-M. Torrenti – H. Tournier-Cognon – E. Bourdarot, , Modelling of Alakali-silica Reaction, part 2 : Influence of water on ASR, Proceedings of the 12th ICAAR, Pékin, Chine, 2004.

[18] S. Poyet – A. Sellier – G. Thevenin-Foray – B. Capra – J.-M. Torrenti – H. Tournier-Cognon – E. Bourdarot, , Modelling of Alakali-silica Reaction, part 3 : Modelling of structural effects induced by ASR,, Proceedings of the 12th ICAAR, Pékin, Chine, 2004.

[19] S. Guedon, et al, Study on the tournai limestone in antoing cimescaut quarry petrological, chemical and alkaly reactivity approach , Proceedings of the 11th ICAAR, Quebec, 2000 11-16 June, 1402p.

[20] P.J.M. Monteiro, K. Shomglin, et al, Effect of aggregate deformation on alkali-silica reaction, ACI Materials Journal, Vol 98., pp 179-83,2001