EVALUATION OF ALKALI-SILICA REACTION EVOLUTION IN CONCRETE USING ULTRASONIC TESTS

ÉVALUATION DE L'ÉVOLUTION DE LA RÉACTION ALCALI-SILICE DANS LE BÉTON PAR LES MÉTHODES ULTRASONIQUES

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ABSTRACT

Concrete structures may be at risk to deteriorate by alkali-silica reaction (ASR), when they are maintained under the conditions supporting the reaction. This reaction causes the expansion of concrete and generates microcracking in the interior part and macrocracking on the surface of concrete. This transformation can lead to serious durability problems in concrete structures, which decrease their technical and economical management. The aim of this study is to monitor the damage due to ASR progression using mechanical and non-destructive tests on specimens of various scales: mortars, laboratory concrete and cores collected from a large hydraulic structure.

Two Canadian crushed aggregates were used: Spratt limestone as a reactive aggregate and Limeridge limestone as a non-reactive aggregate. Two mortars and two concretes were made from these aggregates. Mortars were made according to Canadian standard CSA A23.2-25A and stored at 38°C in 1 molar NaOH solution and concretes were made and stored at 38°C in humid air (R.H>90%) according to Canadian standard CSA A23.2-14A. Also two types of concrete cores containing reactive aggregates (siliceous clayey limestone and Potsdam sandstone) from two concrete locks affected by ASR were kept at 38°C in 1 molar NaOH solution. Expansion and mass variation of all specimens were measured regularly during the reaction evolution.

In a first step, the properties of aggregates were characterized and their reactive silica content was measured. Non-destructive test methods were based on measurement of ultrasonic pulse velocity and resonant frequencies (longitudinal and transversal) of mortar, concrete and concrete cores. Also some other investigations were performed on the specimens: compressive and tensile strength, modulus of elasticity, non linear acoustic test, petrographic analysis by SEM and Damage Rating Index measurement.
The main observations and recommendations are:

- Measurement of elastic modulus of elasticity is the best mechanical test to assessing ASR damage in concrete.
- The relation was observed between the expansion of mortars and the variation in its petrography features.
- There was a good relationship between expansion and the degree of damage associated by ASR in concrete and concrete cores assessed by Damage Rating Index method.
- Measurement of non linear parameters and resonant frequencies are the best non-destructive techniques for assessing ASR progression. A direct correlation was shown between the expansion of concrete cores and the reduction of dynamic modulus and increasing the nonlinear parameters.
RÉSUMÉ

Les structures en béton peuvent être endommagées par la réaction alcali-silice (RAS), quand elles sont maintenues dans les conditions soutenant la réaction. Cette réaction provoque l'expansion du béton et génère les microfissures dans la partie intérieure et macrofissures sur la surface du béton. Cette transformation peut diminuer la durabilité des structures en béton, ce qui diminue ses compétences techniques et augmente des frais de gestion des ouvrage. L'objectif de cette étude est l'évaluation des dommages provoqués par l'évolution de la RAS en utilisant des essais mécaniques et non-destructifs sur des échantillons de différentes compositions : mortiers, béton de laboratoire et carottes de forage.

Deux granulats canadiens ont été utilisés: Le calcaire Spratt comme un granulat réactif et le calcaire Limeridge comme un granulat non-réactif. Deux mélanges de mortier et de béton ont été préparés avec ces granulats. Les mortiers ont été préparés selon la norme canadienne CSA A23.2-25A et entreposés à 38°C dans la solution NaOH 1 molar et les bétons ont été fabriqués et entreposés à 38°C en air humide (H.R. > 90%) selon la norme canadienne CSA A23.2 - 14A. Des carottes de forage contenant les granulats réactifs (Calcaire siliceux et grès de Potsdam) provenant de deux écluses atteint de RAS ont également été conservées à 38°C dans la solution NaOH 1 molar. L'expansion et la variation de masse de toutes les éprouvettes ont été mesurées à intervalle régulier.

Plusieurs études ont été considérées: Dans la première étape, les granulats ont été caractérisés et leur teneur en silice réactive a été mesurée. Les essais non destructifs (la mesure de la vitesse ultrasonores et la fréquence des résonances et essais non linéaires), essais mécaniques, examen pétrographique quantitatif et examen pétrographique par MEB ont été effectués sur ces éprouvettes. Les principales conclusions sont les suivantes:

- La mesure du module d'élastique de béton est le meilleur essai mécanique pour l'évaluation des dommages provoqués par la RAS.
• Une relation a été observée entre l’expansion des barres des mortiers et de la variation des microstructures observées par MEB.

• Il y a eu une bonne corrélation entre l’expansion et le dommage causé par la RAS, pour le béton de laboratoire et les carottes de béton évalués par la méthode DRI.

• L’étude de la fréquence de résonance et acoustique non linéaire sont les meilleures techniques non-destructives pour l’évaluation de la progression de la RAS. Une corrélation directe a été établie entre l’expansion des carottes de béton et la réduction de module dynamique et augmentation des propriétés non linéaires.

Mots-clés : réaction alcalis-silice, essais non destructifs, expansion résiduelle, acoustique non linéaire, pétrographie, caractérisation, béton, mortier
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GLOSSARY

ACR: Alkali-carbonate reaction
ASR: Alkali-silica reaction
AAR: Alkali-aggregate reaction
BLG: Concrete containing Limeridge limestone
BSP: Concrete containing Spratt limestone
BB: Concrete core extracted from Beauharnois lock
BCSC: Concrete core extracted from Côte-St-Catherine lock
DRI: Damage Rating Index
$E_L$: Dynamic modulus of elasticity calculated from longitudinal resonant frequency
$E_T$: Dynamic modulus of elasticity calculated from transversal resonant frequency
MLG: Mortar containing Limeridge limestone
MSP: Mortar containing Spratt limestone
SEM: Scanning electron microscopy
UPV: Ultrasonic pulse velocity
XRD: X-Ray diffraction
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CHAPTER 1

INTRODUCTION

1.1 Context
This study was undertaken in 2005 and proceeds under the supervision of professors Patrice Rivard and Jamal Rhazi at Civil Engineering Department of the University of Sherbrooke. This thesis is part of a large research project that aims at evaluating the residual expansion of concrete affected by alkali-silica reaction with non-destructive techniques and chemical analysis. The Rock Mechanic Group in civil engineering department has been studied various aspects of alkali-silica reaction in concrete for many years.

Concrete structures can be deteriorated by various physical and/or chemical factors: freezing/thawing cycles, carbonation, chloride attack on the steel reinforcement, sulphate attack, alkali-aggregate reaction (AAR), etc. AAR is a chemical reaction between certain types of aggregates and hydroxide ions (OH\textsuperscript{−}) associated with alkalis in the cement, under humidity conditions. Usually, the alkalis come from the Portland cement, but they may also come from other ingredients in the concrete or from the environment. Under some conditions, the reaction may result in damaging expansion and cracking of the concrete. Concrete deterioration caused by this reaction is generally slow, but progressive.

The cracks facilitate the entry of de-icing salt solutions that may cause corrosion of the reinforcing steel, thereby accelerating deterioration and weakening a structure. The map cracking due to AAR generally becomes visible when concrete is 5 to 15 years old. AAR is a severe form of deterioration and measures need to be taken to minimize it.

The AAR-related problems were first identified in the early 1940's in California (U.S.A) (Fournier et al., 2000). Since then, it has been recognized in more than 50 countries around the world. It is likely that this problem exists in a large number of countries.

In Canada, the first documented case of AAR was investigated in a bridge in Montreal, Quebec, in 1953 (Swenson, 1957). Since then, numerous cases have been reported in Ontario, Quebec and the Maritimes. However, cases of this reaction are less common in the Prairie Provinces and
British Columbia because the alkali content of the cements produced there has been relatively low. The distribution of potentially reactive aggregate sources and documented cases of alkali-aggregate reactivity are shown in Figure 1.1.

![Figure 1.1 Map of Canada showing locations of structures affected by alkali-aggregate reaction or sources of known reactive aggregate (Grattan-Bellew et al., 2002)](image)

There are two types of alkali-aggregate reaction:

**Alkali-silica reaction**

This is the most common form of AAR. The alkali-silica reaction (ASR) on concrete occurs by reaction between certain silica phases in the aggregate with alkali and hydroxide ions in the pore solution of the hydrating cement to produce a hydrous alkali silicate gel (Glasser, 1992). This gel can swell by incorporation large amounts of water, causing severe and irreversible expansion. This reaction also can lead to cracking, increased permeability, and decreased strength of the concrete (Figure1.2). Massive concrete structures, such as dams, are particularly susceptible to the damage caused by ASR, because of the availability of water and because massive gravity dams usually do not contain enough steel reinforcement to restrain the expansion. This type of reaction can be divided into two groups based on the evolution rate of the reaction as a consequence of the rock origin. It will be explained in the section 2.1.
Alkali-carbonate reaction

Alkali-Carbonate Reaction (ACR) is similar to ASR in that the alkaline environment of concrete attacks the dolomite aggregates. This reaction has been debated for a long time. In particular the dolomitic limestone from Kingston, in Ontario, Canada has been studied extensively. However, all dolomite aggregates are not reactive. Feldman et al. (1961) explored the sorption and expansion isotherms of the dolomitic limestone from Kingston. They found evidence of gel like material and concluded that the mechanism was similar to that of ASR. So they used low alkali cement and pozzolanic materials to stop the expansion caused by this reaction but without success.

Gillot (1963) has reported that certain clayey dolomitic limestone containing 40-60% of dolomite or calcite and 5-10% acid-insoluble residues, taken from Kingston, were highly reactive aggregate. Since then, the numerous occurrences of reactive carbonate aggregates in concrete have been reported in the North America (Ozol, 1994). This reaction can be explained as:

$$\text{CaMg(CO}_3\text{)}_2 + 2\text{MOH} \Rightarrow \text{CaCO}_3 + \text{Mg(OH)}_2 + \text{M}_2\text{CO}_3 \tag{1}$$

Where, M presents alkali ion. The formed $\text{M}_2\text{CO}_3$ reacts with $\text{Ca(OH)}_2$ produced from cement hydration to regenerate alkali ions.
\[ M_2CO_3 + Ca(OH)_2 \rightarrow CaCO_3 + 2 MOH \]  \hspace{1cm} (2)

For equation (1), the sum of volume of the solid products is less than that one of reactant, thus dedolomitization could not be the cause of expansion. In fact, the dedolomitization just simply developed the channel for access of moisture by clays (Gillot, 1964, Tang et al, 1989, 1991, Deng et al, 1993). The clay then expands by imbibing water, including pressure large enough to crack the concrete.

This reaction usually occurs early and structures may show cracking within 5 years after construction. Over time, the ACR products create a "rim" around the aggregate, weakening the bond and creating microcracks and voids. Cracks allow ingress of potentially deleterious materials like water, sulfates and chlorides inside concrete, which in turn can lead to durability issues such as freeze/thaw damage, sulfate attack or steel corrosion.

Once ACR begins, it will continue until the reactants (that is, dolomite and hydrated lime) are exhausted. Therefore, structural damage caused by ACR can only be repaired by complete replacement of affected members. This reaction is less common and cases in Canada to date are limited to parts of Eastern Ontario (Figure 1.3). ACR is relatively rare because aggregates susceptible to this reaction are usually unsuitable for use in concrete for other reasons, such as strength potential. The mechanism of this reaction is different from that of alkali-silica reaction and it is not discussed in this study.

![Figure 1.3](image)  

Figure 1.3  Map-pattern cracking and extrusion of joint filling material due to expansion of concrete affected by alkali-carbonate reactivity in a sidewalk in Kingston, Ontario (Grattan-Bellew et al., 2002)
Extensive research has been carried out on ASR over the past six decades. Main area of research in Canada and worldwide are addressed (Bérubé et al., 2000):

1. Better understanding of the mechanism involved its deleterious process in concrete
2. Identifying the rock types or specific petrographic fancies at risk and developing quick and reliable test methods to assess the potential alkali-reactivity of concrete aggregate
3. Developing specification for preventing ASR in new concrete structure
4. Developing management action for existing concrete structure affected by AAR (diagnosis of the cause of distress, risk for future distress and actual structure integrity and repair method).

For a concrete structure affected by ASR, it is difficult to find if the reaction is completed and if not, what the reaction degree and the expansion level are, how much additional expansion can be expected and for how long.

Despite decades of study, the chemistry of alkali-silica reaction remains poorly understood, especially at the molecular scale. Yet, several studies have been carried out on the mechanism of ASR in the siliceous aggregates (Dent-Glasser and Kataoka, 1981, Chatterji, 1989, Wang and Gillot, 1991, Dron and Brivot, 1992, Prince and Perami, 1993, Wilson et al., 1994, Lombardi, 1997, Bulteel et al., 2000). However, only few works was carried out on the mechanism of ASR in the concrete containing the siliceous limestone aggregates (Monnin, 2005). This study is directed on the evaluation of residual expansion of concrete containing limestone aggregates affected by ASR.

1.2 Objectives
The objective of this thesis is to assess various non-destructive methods regarding their effectiveness to characterize the effects of the alkali-silica reaction on mortars and concrete specimens. The contribution of this work is to gather results on cementitious materials at different scales, from mortar bars, concrete prisms and cores collected from a large hydraulic structure affected by ASR. Detailed petrographic exams were conducted in order to better understand how the microstructure damaged by ASR affects the results obtained from non-destructive tests.
1.3 Thesis structure

This document contains ten Chapters. Following the introduction, chapter 2 presents the general knowledge on the ASR. In chapter 3, objective and experimental program is described. Chapter 4 presents material and their characteristics. Chapter 5 presents the characterization of aggregates. In chapter 6, the results of expansion and non-destructive tests and microscopic analysis of mortars and chemical tests are presented, respectively. The results of tests on concretes made in laboratory and concrete cores are described in chapter 7 and 8, respectively. In chapter 9, the results of experimental program will be discussed. The last chapter is conclusions and recommendations for further studies.
CHAPTER 2

LITERATURE SEARCH ON ALKALI-SILICA REACTION

2.1 Different types of alkali-silica reaction

According to the literature, there are basically two types of alkali-silica reaction:

a) Alkali-silica reaction that occurs with poorly crystalline or metastable silica minerals and volcanic or artificial glasses, which have significant initial alkali reactivity: Volcanic glass and opal are two typical examples. The rapid cooling of magma when it is exposed to the atmosphere or the environment, the atoms do not have enough time to accommodate themselves and form crystalline network forms. Therefore, crystal formation cannot occur and glass becomes a potentially reactive substance. Its thermal history and severe overcooling from larger amounts of glass that can either form govern the amount of glass in a volcanic rock. They react quickly with deleterious effects about one year after the structure building, but this period is also depends on environmental conditions (Ponce et al., 2006). Aggregates containing such materials may cause deterioration of concrete when the reactive component is present in amounts as small as 1%.

b) Alkali-silica reaction that occurs with various varieties of silice such as chalcedony, cryptocrystalline, and macrogranular quartz. These minerals have undergone deformation and significant thermal changes due to the number of tectonic processes acting during the evolution of the earth’s crust. This group shows a slow or delayed reaction with deleterious expansive effects that can be seen after 6 to 10 years, or even at later ages. In other cases however, particularly when exposed to deicing salts, cracking may occur in 5 years or less. Aggregates containing such forms of silice may cause deterioration of a concrete when the reactive component is present in amounts as small as 5% by mass of the aggregate and there is a high alkali content. According to Canadian experience this category also includes several slowly expanding aggregates in which microcrystalline quartz is thought to be the reactive component.
Rocks such as greywacke, argillite, quartzite, hornfels, granite and granite gneiss are some. Such rock types may not show cracking and deterioration for up to 20 years.

These reactions introduce by their mechanism swelling and cracking that is deleterious for concrete durability. The formation and evaluation of amorphous and poorly crystallized phases could induce a variation of the volume of the aggregate and consequently participate in this swelling. Figure 2.1 shows the various aggregates and their reaction patterns regarding ASR (Idorn, 1997). An important factor to the deleterious activity of the aggregates is their size distribution.

Figure 2.1 Cracking in different type of aggregates (Idorn, 1997)
2.2 Principal methods of reaction mechanism

One mechanism of reaction that is confirmed by most of the researchers is hydroxide attack of silica causing formation of ionized site and then breaking up of siloxane bonds.

\[ 2\text{SiO}_2 + 2\text{OH}^- \rightarrow 2\text{SiO}_2^{5/2} + \text{H}_2\text{O} \]  \[ \text{[1]} \]

\[ \text{SiO}_2^{5/2} + \frac{1}{2} \text{H}_2\text{O} + \text{OH}^- \rightarrow \text{H}_2\text{SiO}_4^{2-} \]  \[ \text{[2]} \]

We can classify different models of mechanism according to three processes:

- Topochemical process: the reaction is hold in solid-liquid interface constituted by interstitial solution and aggregate. In this reaction, it is not necessary that all of the aggregate come in to the solution.
- The second process is dissolution-precipitation. In this model the reaction is hold in interstitial solution when the elements have passed under ionic form in different spaces.
- There is also a new chemical method to quantify ASR. This method is based on the measurement of the reaction degree. This process concerns the properties of product precipitating from dissolution of silica.

2.2.1 Topochemical process

For the first time, in 1981, Dent-Glasser and Kataoka (1981a) proposed a reaction mechanism explaining deterioration of poorly crystalline silica. This reaction is demonstrated as follow:

- Ionization of silanole by the hydroxyl ions.
  \[ \text{SiO}_{5/2}\text{H} + \text{OH}^- \rightarrow \text{SiO}_{5/2}^{4} + \text{H}_2\text{O} \]  \[ \text{[3]} \]
- The neutralization of the sites ionized by alkaline (Na\textsuperscript{+} or K\textsuperscript{+}).
  \[ \text{SiO}_{5/2}^{4} + \text{Na}^+ \rightarrow \text{SiO}_{5/2}\text{Na} \]  \[ \text{[4]} \]
- Formation of silanole due to siloxane bond breaking up by the hydroxyl ions attack
  \[ 2\text{SiO}_2 + \text{OH}^- \rightarrow \text{SiO}_2^{5/2} + \text{SiO}_{5/2}\text{H} \]  \[ \text{[5]} \]
Silanole group formed by breaking up of siloxane bonds is then ionized by hydroxyl ions and neutralized like shown in equation 3 and 4. Figure 2.2 shows mechanism of reaction.

![Reaction](image)

**Figure 2.2** Reaction described by Dent-Glasser and Kataoka (1981a)
(a): Ionization of silanol and neutralization of the sites ionized by alkaline
(b): Formation of silanol due to siloxane bond breaking

In the solution containing sufficient alkalis, breaking up of siloxane bond continues with dissolution of silica. Then, the silanol of residual silica will be completely ionized (Dent-Glasser and Kataoka, 1981a). According to Fernandez (Fernandez, 1992) ionized silanol proportion is function of pH and also, ionization degree will increase with increasing pH of solution in balance with silica gel. For example, the ionized silanol proportion is 0.25 for the pH of 10.5 whereas; this proportion is 0.5 for the pH of 11.5.

Thus, aggregates composed of amorphous or poorly crystalline silica will tend to dissolve more readily in the inherently high pH pore solution in concrete. Well-crystallized or dense forms of silica, such as quartz, are relatively inaccessible to alkaline-hydroxide solution, and dissolution only occurs at the surface, at a very slow rate.

These first stages are allowed by a large number of other authors. Prince and Perami (Prince and Perami, 1993) showed also that silica attack is done by hydroxyl ions. However, some others like
Wilson (Wilson et al., 1994) believed that siloxane bonds are broken up by the alkalis ions and not by hydroxyl ones.

In 1992, another mechanism similar to that of Dent-Glasser and Kataoka (1981) was proposed by Pool (Pool, 1976), but in this method, the role of calcium is presented.

Generally, Ca\(\text{OH}_2\) performs two main functions in ASR (Wang and Gillot, 1991):

1) Source of supply of OH\(^-\) ions

The attack of alkali on silica is the result of attack by OH\(^-\) on the Si-O-Si bridge in a silica grain. Therefore, the concentration of OH\(^-\) is primary important in alkali-silica reaction. A saturated calcium hydroxide solution has a pH value of 12.5. In Portland cement concrete, the pH of pore solution is greater than 12.5 due to the presence of alkali. It is generally agreed that the higher the pH, the more severe the attack on siliceous material. In concrete containing reactive silica, the OH\(^-\) will be depleted by interaction with siloxane groups, but once the OH\(^-\) will be released by ionization of Ca\(\text{OH}_2\). In other words, the Ca\(\text{OH}_2\) maintains a high pH value in the pore solution.

2) The source of Ca\(^{2+}\) ions

Ca\(^{2+}\) ions can exchange by alkali ions in the swelling alkali-silica complex to produce a non-swelling lime-alkali-silica complex. This process increases the alkali ions needed for further production of a swelling alkali-silica complex by exchange with protons on silanol groups. Therefore, Ca\(\text{OH}_2\) provides the source of Ca\(^{2+}\) needed to release alkali ions in the alkali-silica complex.

On the other hand, the Ca\(^{2+}\) ions concentration in the environment controls the rate of high silica diffuses out of the grains. The higher the Ca\(^{2+}\) concentration of the environment, the lower the
rate at which silica diffuses out of the grains and the higher the rate at which cations diffuse into the grains (Chatterji, 1989).

The fact that alkali ions diffuse to the reaction sites before Ca\(^{2+}\) ions, is explained below:

- They are surrounded by fewer number of molecule of water. So, calcium ions penetration depends to the aggregates deterioration degree. When they are sufficiently deteriorated, the calcium ions can penetrate inside of them (Poole, 1992).
- According to the model proposed by Chatterji (Chatterji et al., 1986), more of the smaller ions (Na\(^{+}\) and K\(^{+}\)) will follow the penetration of OH\(^{-}\) ions than the large ion of calcium, although both types of cations will penetrate reactive silica grains. The reaction involves dissolution of exterior part of aggregate and calcium ions controls amount of diffusion. In fact, silica diffusion is slow down by its precipitation with calcium ions in forming the C-S-H or C-K-S-H.
- Electrostatic energy of calcium ion is more than those of alkalis and it can release one part of alkalis of gel that will be available to continue the reaction and reproduce alkali-silica gel (Wang and Gillot, 1991).
- More alkali ions are available at the first stage. The solubility of NaOH and KOH is much higher than that of Ca (OH)\(_2\) (Table 2.1). In other words, alkali ions are more readily available for the exchange reaction. Also, when alkali and Ca (OH)\(_2\) are in the same system, the solubility of Ca (OH)\(_2\) is greatly suppressed due to the common ion effect (Figure 2.3). So the alkalis are more available than lime.
- The hydraulic radius is another factor that affects on diffusion of the cations to the reaction sites. The hydraulic radius of Na\(^{+}\) and K\(^{+}\) ions is much smaller than that of Ca\(^{2+}\) ions (Table 2.2) and this tend to increase the rate of diffusion of Na\(^{+}\) and K\(^{+}\) ions (Wang and Gillot, 1991).
TABLE 2.1 SOLUBILITY OF ALKALIS AND Ca (OH)$_2$ IN WATER
(Wang and Gillot, 1991)

<table>
<thead>
<tr>
<th>Alkali</th>
<th>Solubility (g/100cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>At 0°C</td>
</tr>
<tr>
<td>NaOH</td>
<td>42</td>
</tr>
<tr>
<td>KOH</td>
<td>107 (at 15°C)</td>
</tr>
<tr>
<td>Ca (OH)$_2$</td>
<td>0.185</td>
</tr>
</tbody>
</table>

TABLE 2.2 HYDRAULIC RADIUSES OF CATIONS AND ELECTROSTATIC ENERGY BETWEEN CATIONS AND O$^-$(Wang and Gillot, 1991)

<table>
<thead>
<tr>
<th>Cations (R)</th>
<th>Hydraulic radius</th>
<th>R-O distance</th>
<th>Electrostatic energy $(Z_1Z_2/d)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na$^+$</td>
<td>3.3</td>
<td>4.65</td>
<td>0.21</td>
</tr>
<tr>
<td>K$^+$</td>
<td>3.1</td>
<td>4.45</td>
<td>0.22</td>
</tr>
<tr>
<td>Ca$^{2+}$</td>
<td>4.2</td>
<td>5.55</td>
<td>0.36</td>
</tr>
</tbody>
</table>

Figure 2.3 Solubility of CaO in alkaline solution (Wang and Gillot, 1991)
2.2.2 Dissolution-precipitation process

According to this model, ASR occurs by the setting of silica ions in interstitial solution. Stark (Stark et al., 1993) reported the case of mortar made by Opal of Beltane. The observation of alkali-silica gel by optical microscope has demonstrated the large quantity of this gel in the cement paste. They mentioned that the central part of silica stays visible and the peripheral part is dissolved. In this theory, hydroxyl ions, silica and alkalis migrate in to the solution and alkali-silica gel is formed outside of aggregates. Generally this reaction can occur as follow (Dron and Brivot, 1992; Lombardi, 1997):

1) First siloxane bond breaking up by hydroxyl ions attack and then pass in the solution.
\[
\text{SiO}_2 + \text{OH}^- + \text{H}_2\text{O} \Rightarrow \text{H}_3\text{SiO}_4^- \quad [6]
\]

2) Dissolution of Ca (OH)$_2$ in order to balance hydroxyl ions: In solution, the cations of Ca$^{2+}$ close to Ca (OH)$_2$ tend to migrate toward the zone containing less calcium ions:
\[
\text{Ca(OH)}_2 \Rightarrow \text{Ca}^{2+} + 2\text{OH}^- \quad [7]
\]

3) When all of the elements (OH$^-$, Na$^+$, K$^+$, Ca$^{2+}$ and Si$^{4+}$) are contacted in the solution, the gel of reaction that its composition depends to the proportion of elements will be formed.
\[
\text{H}_3\text{SiO}_4^- + 1.43 \text{Ca}^{2+} (n - 0.57) \text{H}_2\text{O} \Rightarrow \left[\text{SiO}_2 + 0.43 \text{CaO} - n\text{H}_2\text{O}\right] + 0.86 \text{H}^+ + \text{OH}^- \quad [8]
\]

4) Alkali ions are adsorbed just one time in the reaction product and they do not release from it (Davies and Oberholster, 1988; Dron and Brivot, 1993). According to the result of chemical analysis of reaction product, interstitial solution was observed in the solid phase (Lombardi, 1997). So the role of calcium ions in this model is more important than that presented by Chatterji (1986).
According to this theory, pressure of swelling is caused by crystallization of reaction product. But, this theory could not be found, because in the term of cement hydration, the form of all reaction products is crystalline.

2.2.3 Model of quantification the reaction degree (Bulteel et al. 2000)

Bulteel et al. (2000) presented the new chemical method for quantitative measurement of the ASR degree. In fact it is the diagnosis technique that could be specific and quantitative to relate expansion to the reaction degree. This method is applied to a crushed natural reactive aggregate (Silex) kept in contact with an alkaline solution and saturated lime. This chemical system is designed to model the concrete capillary pores alkaline solution, in contact with siliceous reactive aggregates. (Figure 2.4)

![Diagram of model of Bulteel (Bulteel et al. 2002)](image)

The dissolution degree is measured by a selective acid treatment, and the nature of silica into solution ($Q_0$) is characterized by liquid NMR (Nuclear magnetic resonance) spectroscopy. The remaining silica is composed of $Q_4$ tetrahedrons and $Q_3$ protonated sites identified by solid NMR spectroscopy. These protonated sites are measured by thermogravimetry analysis.
\[
2 \text{SiO}_{5/2} \text{H} \rightarrow 2 \text{SiO}_2 + \text{H}_2 \text{O}
\]

It should be noted that the aggregate which is used in this model is a siliceous aggregate containing more than 99% silica. Thus the result of thermogravimetry analysis is only associated to the transformation of Q₃ to Q₄. But the reliability of this model for quantifying the reaction degree has not already been used to the other types of aggregates containing impurities. For example, the presence of clay in the matrix of aggregate could influence the results of thermogravimetry analysis. This topic will be discussed in chapter 6.

It has been shown that the formation of Q₃ sites prevails on dissolution as the reaction progresses and contributes to an internal silica gel generation.

The Q₃ site formation and the silica dissolution are controlled by the hydroxide diffusivity in reaction grains and by hydroxide adsorption on the solid surface. These values could be measured as follow:

\[
n = \frac{\text{moles of Q₃ sites}}{\text{moles of initial silica}}
\]

\[
\alpha = \frac{\text{moles of dissolved silica}}{\text{moles of initial silica}}
\]

Where, \( n \) and \( \alpha \) are Q₃ site content and dissolution degree, respectively.

The formation of Q₃ tetrahedrons causes a swelling of the aggregate and a significant increase of its specific pore volume due to microcracking (Bulteel et al., 2002). The hydroxide diffusivity increases with the pH and the ionic strength of the solution (Chatterji, 2000). At a constant pH and ionic strength, the hydroxide adsorption decreases with the increasing size of the hydrated cations.

Also, according to this method, the mortar swelling can be considered as proportional to the aggregate swelling by the increase in the pore volume. But the increase in pore volume due to
$Q_4 \rightarrow Q_3$ expansive transition induced by swelling of the aggregate is not proportional to the degree of the reaction.

Monnin (Monnin et al., 2004) reported the same model-reactor allows quantifying various reaction degrees in siliceous limestone aggregates. According to their results, as ASR progresses, the formation of $Q_3$ tetrahedron prevails on the formation of $Q_0$ tetrahedron and the transition of $Q_4 \rightarrow Q_3$ on the siliceous limestone is expansive; so the origin of the expansion in the aggregate seems to be an internal granular swelling.

As explained, this chemical system models the reaction between pore alkaline solution and aggregate present in concrete. But the role of fine aggregate as the other part of concrete component has not been determined. On the other hand, concrete elements may contain the siliceous fine aggregate in their structure. Therefore, using this model could not be reliable for quantifying the ASR degree in a core sample extracting from a concrete structure.

2.3 Signals of ASR

Expansion, microcracking, loss of cement paste-aggregate bond, reaction product (gel) and reaction rim are the main results of ASR in concrete. But concrete could be affected by the other factors such as freezing and thawing, mechanical loading, salt attack and shrinkage that could produce symptoms similar to those of ASR. So, the occurrence of this reaction in concrete should be confirmed. For instance, freezing and thawing does not occur in warm climates and sulphate attacks only occur in the concrete in contact with ground water.

2.3.1 Cracking

The main effect caused by this expansive reaction is cracking of concrete surface. Internal cracks spreading from expanding particles into the cement paste and macrocracking the surface of affected structure. Dron and Brivot (1993) proposed that cracks in concrete affected by ASR are filled by ASR gel. Hydrated gel is diffused far from the aggregate into micro pores and channel
connecting them. Then, the gel reacts with Ca\(^{2+}\) ions and expands to cause cracking of concrete. But in this model it was not obvious why the gel generates expansion pressure in the pores. Also, the cracking of aggregate under this compressive pressure was not described.

Bazant et al., (2000) proposed that expansive pressure accumulated in the boundary of aggregate induced cracking of concrete. According to this theory, pressure is initially released by pushing ASR gel in to accessible micro pores near the aggregate surface. When the pore around of aggregate is completely filled by gel, further formation of gel induces expansive pressure between cement paste and the unaffected concrete pore. The expansive pressure is finally released by cracking the cement paste. In this model, although expansive pressure is applied on all parts of concrete structure, the cracking of cement paste should not attend cracking of aggregate. According to analysis of crack patterns in concrete affected by ASR, Idorn (2001) concluded that expansive pressure due to formation of ASR gel is accumulated inside the reacting aggregate. Heterogeneous expansion of aggregate during the time of reaction causes tension in aggregate that induces the crack of both the aggregate and surrounding cement paste.

The cracking of concrete due to ASR may take several forms depending to the design, geometry and other stresses operating on it. The pattern cracking in plain or lightly reinforced concrete is the network of cracks that widen with time. The cracks from polygons of seemingly sound areas, roughly square to hexagonal in form and quite variable in size, are often 10 to 15cm in one direction. With time, the larger network of cracks may subdivide into smaller networks (Gillot, 1975). But in more heavily reinforced or prestressed concrete, they become directional, in the direction of the largest applied stress. If prestress is lost with age, they tend to take the form of map cracking as further random cracks appear (Shayan, 1994).

The environmental condition of structure and its composition must also be considered. For example, in the case of a floor slab of concrete, the cracks are found in the part in contact with the ground. Thus, the presence of crack in the parts of structure exposed with cycle of drying and damping, heating and loading is more remarkable. Also, less strained parts of structure show more obvious signs of ASR cracking than the confined part of the elements (Shayan, 1994).
The type of aggregate used in concrete is another important factor influencing on the form of cracking. For example, the swelling of Potsdam sandstone is isotropic and it generates fewer cracks compared with Spratt limestone (Rivard, 2002).

2.3.2 Reaction rim

One particular spectrographic feature associated with ASR is the growth of the dark reaction rim at the periphery of aggregate particles (Figure 2.5). In fact, reaction rim is a zone surrounding the aggregate, between the particle and the cement paste. Identifying the reaction rim can sometimes be difficult around dark aggregates. It plays a significant role in the deterioration of concrete. ASR does not cause the deterioration of concrete, if ASR is completed before the formation of the reaction rim (Ichikawa and Miura, 2007).

Figure 2.5 Reaction rim surrounding Potsdam particles (Rivard, 2002)

The chemical composition of this zone can be different from the inner part of the particle and related to ion exchange processes between silica in aggregate and alkali hydroxides present in concrete pore solution. A few studies carried out on reaction rim in the case of opal aggregate, from reactive aggregate to cement paste are reported as follow (Thaulow et al., 1975; Pool, 1976; Wang, 1991; Brouxel, 1993):
• A rapid decrease in SiO$_2$ content at aggregate contact followed by a slow decrease.
• A rapid increase in CaO content at aggregate contact followed by a slow increase. For instance, Thaulow showed a calcium-rich reaction zone inside the aggregate.
• Maximum alkali content in the middle of the reaction rim, which slowly decreases toward the cement paste.

However, all rocks do not react like opal. For example, the reaction rim in the Potsdam sandstone is mainly composed of more than 99.5% silica (Rivard, 2000). As the reaction progresses and the expansion level raises, a dark rim is developing at the internal boundary of the aggregate particle. At 0.04% expansion, rims are generally difficult to identify. But at an expansion level of 0.06%, the rims are about 2mm thick and at the expansion level of 0.12-0.14%, the reaction rim is countable and affects nearly all the aggregate particles.

2.4 The concept of the pessimum proportion

Numerous studies have shown that there is a certain concentration or proportion of reactive aggregate present in an otherwise inert aggregate that cause a maximum expansion in concrete made from it. This proportion is referred as a pessimum proportion (Ozol, 1975). Similarly, according to Ozol, there is also a pessimum proportion for alkali content, which will give maximum expansion. Hobbs (Hobbs, 1988) suggested that both alkali concentration and percentage of reactive aggregate component should be considered together and pessimum can be corresponding to the ratio of SiO$_2$/Na$_2$O. He presented a graphical model explaining relationship between SiO$_2$/Na$_2$O and Expansion of concrete (Figure 2.6).

This figure presents expansion of four areas:

A: Reactive silica content is very low and the quantity of reaction product is too negligible to crack the concrete. So this expansion cannot be deleterious.
B: The reaction continues after concrete hardening, reactive silica content is sufficient to be expansive in concrete and expansion increases with increasing the SiO$_2$/Na$_2$O ratio.

C: The reaction continues after concrete hardening and the concrete is cracked but the rate of reaction slows down with increasing amount of SiO$_2$ in SiO$_2$/Na$_2$O ratio.

D: Reactive silica content is too high and the reaction occurs before hardening of the concrete. So, reaction product cannot make swelling and cracking of concrete.

![Graphical model of pessimum](image)

Figure 2.6 Graphical model of pessimum, Water/Cement = 0.4, Aggregate/Cement = 2.75 and Na$_2$O$_{eq}$ = 6 kg/m$^3$ (Hobbs, 1988)

In this example, the pessimum of reactive silica content is 2.5% of total mass of aggregate. There is not any pessimum if the aggregate contains more than 2.5% of reactive silica or fewer than it.
Such a critical level will depend on the nature of the reactive material in the mortar or concrete and the mobility of pore fluids within the sample. This mobility will, of course, depend on the porosity and permeability of the concrete, and this will depend on a number of factors, but perhaps most important among these will be the water/cement ratio.

2.5 Composition and other characteristics of the alkali-silica gel

ASR gels are almost responsible for expansion and damage due to ASR in concrete. Gel may be present in cracks and within aggregate particles. The best technique for the identification of ASR is the examination of concrete in thin section, using a petrographic microscope. Alternatively, polished sections of concrete can be examined by scanning electron microscopy (Figure 2.7). This gel is usually amorphous to X-ray diffraction and optically isotropic (John et al., 1998). Reactive indices are low (between 1.45-1.51) and crystallization of older gel into various compounds can take place. It is reported by various authors that ASR gel in concrete tend to change composition over time, usually accumulating calcium from the surrounding cement paste.
The compositions of ASR gel vary widely, especially in CaO content. The compositions of ASR gels in field concretes typically fall into the range \((\text{Na}_2\text{O}+\text{K}_2\text{O})/\text{SiO}_2 = 0.05-0.6\) and \((\text{CaO} + \text{MgO}) / \text{SiO}_2 = 0-0.2\) (molar ratios) (Struble, 1979 and Knudsen et al., 1975). The compositions, especially Ca contents, vary significantly with age and distance from the reaction site. Many gels identified petrographically as ASR gel has the composition very similar to C–S–H (Diamond, 2000, Thomas, 2001 and Knudsen, 1975). Helmuth and Stark (1992) concluded that compositionally, ASR gels can be regarded as two-component mixtures containing variable proportions of alkali calcium silicate and alkali silicate components with nearly fixed compositions.

Generally, alkalis that exist in C-S-H gel may incorporate the hemi-crystalline C-S-H structure and also be trapped in the layer space of C-S-H gel as a free state or may be adsorbed on the surface of C-S-H. Because C-S-H has a huge surface to volume ratio, among alkalis existing in C-S-H gel, the adsorbed alkalis are the dominating ones. Hence it is the adsorption ability of C-S-H surface for alkalis to decide the fixating alkalis capability of C-S-H gel. But it is not enough deep to study on the adsorbing alkalis ability of C-S-H surface and the contributing factors.

The alkali silicate end member is thought to have a composition of 17\% \((\text{Na}_2\text{O}+\text{K}_2\text{O})\) and 83\% \(\text{SiO}_2\), and the alkali calcium silicate end member a composition of approximately 6.6\% \((\text{Na}_2\text{O}+\text{K}_2\text{O})\), 52.6\% \(\text{CaO}\) and 40\% \(\text{SiO}_2\). In contrast, Diamond (2000) concluded that ASR gel is neither uniformly low nor high in Ca, but varies from one grain to another. Generally, gel rich in calcium are considered non-swelling, but some Ca is necessary for the formation of gel. In fact, significant expansion occurs only when sufficient reactive \(\text{Ca(OH)}_2\) is available, that systems without \(\text{Ca(OH)}_2\) exhibit little expansion despite evidence that ASR has occurred.

The appearance of ASR gels has been described many times. Macroscopically, they have been described as transparent and resinous in appearance with variable viscosity and when they are altered, they become white and hard, with desiccation cracks similar to those observed in layers of dried mad (Poole, 1992).
The SEM (Scanning electron microscopy) observation of ASR at high magnification shows that they generally appear to be composed of very fine nodules and the dry mass appear to be finely porous and it displays a characteristically spongy texture.

2.6 The principal factors influencing on alkali-silica reaction

It is widely accepted that the three essential components necessary for ASR-induced damage in concrete structures are:

1) Reactive forms of silica in the aggregate

2) High-alkali (pH) pore solution

3) Sufficient moisture

Also, there are other factors that influence on the rate of reaction that will be mentioned next.

2.6.1 Reactive silica

The use of reactive aggregates in concrete is necessary for ASR to occur. The term of reactive refers to aggregates that tend to breakdown under exposure to the highly alkaline pore solution in concrete and subsequently react with the alkali-hydroxides (sodium and potassium) to form ASR gel. It is important to note that not all siliceous aggregates are prone to ASR. The inherent reactivity of aggregates depends on several factors, including aggregate structure and aggregate texture.

- Aggregate structure

In nature, rocks contain the variety of mineral of the silica group, where silica usually has different structural states. There are two types of reactive aggregate that could be classified by their reaction rate (Section 2.1). The behaviour and manifestations in concrete are directly related to the mineralogy of the aggregates, different compositions and their own microstructures.
All these reactive materials have some common characteristics. All of them contain SiO₂ as a major component and have either a disturbed or open structure. Also both external and internal surfaces of all siliceous materials are covered with silanol, SiO₁/₂H groups. As Hornain (Hornain 1993) underlined it, when crystalline network of aggregate is similar to that of quartz (there is a few defect) the risk of alkali-silica reaction will be reduced.

The other parameters determining reactivity of different types of silica are the length L of Si-O liaison and inter-tetrahedron angle of α in Si-O-Si that are presented in Figure 2.8 (Couty, 1993a, 1993b, 1999). This angle can vary between 120° and 180° and determine energy of Si-O liaison by silica reactivity. The nuclear magnetic resonance test has confirmed that this angle in quartz and opal are 144, 7°±0, 7° and 152, 6°±6, 9°, respectively (Couty and Fernandez, 1996).

![Figure 2.8](image)

Figure 2.8 Representation of a siloxane bond between two silica tetrahedron defining angle α and length of silica-oxygen liaison (Bulteel, 2000)

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Aggregate texture

The factors concerning aggregate texture are granularity, micro porosity and micro cracking. All these factors influence on reaction surface that is the important parameter influencing on the rate of alkali-silica reaction.
2.6.2 High-alkali pore solution

The presence of sufficient alkalis is another required ingredient for ASR. The source of alkali can be from any of the following:

- Portland cement.
- Supplementary cementing materials such as fly ash, slag and silica fume.
- Aggregates.
- Chemical admixtures.
- External sources such as seawater and de-icing salts.
- Wash water (if use).

Of the above materials, Portland cement is the main contributor of alkali. The alkali present in Portland cement is in the form of potassium oxide (K$_2$O) and sodium oxide (Na$_2$O). The quantity of alkalis in Portland cement is typically expressed as follows:

$$\text{Na}_2\text{O}_{\text{eq}} = \text{Na}_2\text{O} + 0.658\text{K}_2\text{O}$$

The alkali content of Portland cements generally ranges from 0.3-1.3%. Interstitial solution analysis by Roux (Roux et al., 1992) shows that alkali concentration can fall quickly because of its consumption in some solid phase formation such as C-K-S-H. Thus, increasing in alkalis content of interstitial solution increases its consumption by solids phases and increase hydroxyl ions concentration.

Although the percentage of alkalis in Portland cement is relatively low, in comparison to other oxides or compounds, the bulk of the alkalis ultimately resides in the pore solution of concrete, and it is the associated hydroxide (OH$^-$) concentration (necessary to maintain charge balance) that produces the inherent high pH in the pore solution (13.2 to 14.0). Based on Stanton's early work (1940), it was proposed for many years that expansion due to ASR is unlikely to occur when the alkali content of the cement is below 0.6% Na$_2$O$_{\text{eq}}$. This value has been cited in various specification limits and was adopted as part of the American Society for Testing and Materials standard ASTM C 150.
One part of the alkali hydroxide of the concrete pore solution cannot contribute to ASR:

- Alkali hydroxide could induce gypsum dissolution to form portlandite and soluble alkali sulphate. This reaction raises the sulphate concentration of the solution instead of the hydroxide concentration.
- A partial drying of the concrete decreases, more than increases, the alkali hydroxide concentration in pore solution.

In interstitial solution, OH\(^{-}\), Na\(^{+}\), K\(^{+}\) and Ca\(^{2+}\) are responsible of reactive silica deterioration and increasing their concentration accelerates the rate of reaction. Also, the concentration of alkali and sulphate are higher than that of Ca\(^{2+}\) (Gillot et al., 1973; Gillot, 1980; 1983, 1989; Regourd 1989). Figure 2.9 (Diamond, 1983) illustrates increasing of potassium and sodium ions concentration and reducing in calcium ion content and its solubility \(K_s = [\text{OH}^-]^2 \cdot [\text{Ca}^{2+}]\).

![Figure 2.9](image-url)  
**Figure 2.9** Development in 24 hours of sodium, potassium, calcium, and sulphate and hydroxide concentration in interstitial solution of Portland cement paste (Diamond, 1983)
The increasing in hydroxide ion concentration causes decrease in calcium ion concentration. Up to about 8 h, the pore liquid contains a mixture of $\text{Ca}^{2+}$, $\text{OH}^-$, $\text{Na}^+$, $\text{K}^+$ and $\text{SiO}_4^{2-}$ ions. After that time the concentration of $\text{Ca}^{2+}$ and $\text{SiO}_4^{2-}$ drop quickly and those of alkali and hydroxyl ions increase sharply.

However, it is now recognized that limiting the alkali content of Portland cement is not, by itself, an effective way of preventing ASR-induced damage, because this approach does not control the total alkali content of the concrete mixture. Therefore, limiting the maximum alkali content of concrete is the preferred approach when specifying alkalis level. Nixon and Sims (1992) reported that maximum permissible alkali contents between 2.5 and 4.5 kg/m$^3$ Na$_2$O$_{eq}$ have been specified by various countries and agencies, with the allowable alkali content sometimes varying depending on aggregate reactivity.

Figure 2.10 illustrates the effects of the alkali content of concrete on expansion, using ASTM C 1293 (this test will be described in section 2.7). Using an expansion limit of 0.04 percent, the graph shows that laboratory concrete containing less than 3.0 kg/m$^3$ Na$_2$O$_{eq}$ was generally resistant to excess expansion, even after 2 years of testing. Although laboratory tests have shown that keeping the total alkali content below 3.0 kg/m$^3$ Na$_2$O$_{eq}$ is an effective method of limiting expansion, field structures have exhibited damage with even lower alkali loadings, especially when alkalis have also been contributed by the aggregates in the mixture or by external sources, such as de-icing salts. Thus, when considering imposing a limit on the alkali content for a given concrete mixture, consideration should be given to the aggregate type and reactivity, exposure conditions, and nature of the structure.

There has been significant debate regarding the impact of alkalis from supplementary cementing material (SCMs) on ASR. However, limits are placed on the total alkali content of the SCMs, and replacement levels (by mass of cement) are specified, based on the chemistry of the SCMs.
As previously mentioned, alkalis also can be released from certain aggregates within concrete, thereby increasing the alkali content of the mixture (Thomas et al., 1992; Stark and Bhatty, 1986). Stark and Bhatty (1986) reported that certain aggregates such as feldspar, argil, nepheline and volcanic aggregate can release alkalis equivalent to 10 percent of the Portland cement content under extreme conditions. So, it's particularly important to ensure the aggregate cannot release significant amount of alkalis in the concrete pore solution.

The total alkalis content within a given concrete mixture may also be increased by the penetration of alkalis from external sources such as seawater, ground water (containing sulphates), de-icing salts, and superplasticizers. Jun-Zhe et al. (2004) showed using sodium salts as an anti-freezing admixture considerably increased the expansion due to ASR of mortars containing reactive aggregates. Moreover, Nixon (1982) demonstrated that seawater (used as part of the batch water) raises the OH⁻ concentration in the pore solution and resulted in higher concrete expansion values.

In the case of high-performance concretes, they are made using a high superplasticizer dosage. As most naphthalene and melamine superplasticizer are neutralized with soda, the use of a high dosage of superplasticizer increases the availability of alkali ions within the interstitial solution and therefore could result an increase in the expected expansion, as founded by Wang and Gillot (1989). Also, microstructural analysis indicated that superplasticizers in the mortar changed

![Figure 2.10 Effect of Alkali Content on Expansion using ASTM C 1293 (Thomas, 2001)](image)
physico-chemical properties of gel products such as viscosity, solubility and moisture adsorption capacity and surface tension. However, amount of alkali brought by the superplasticizer is very low compared with the amount brought by the cement, but they could be more concentrated near the surface of reactive aggregate. This can be explaining the reason that some researchers use calcium naphthalene superplasticizer when they have the least doubt of the potential reactivity of the aggregates used in high-performance concrete.

2.6.3 Sufficient Moisture

Available moisture is important when we consider the potential for ASR-induced damage in field structures. Concrete mixtures comprised of highly reactive aggregates and high-alkali cements have shown little or no expansion in certain very dry environments. Likewise, local differences in moisture availability within the same structure have resulted in vastly different performance within that structure. Specifically, portions of the structure exposed to a constant or steady source of moisture (due to poor drainage or poor detailing) have exhibited significant ASR-induced damage, while other portions of the structure that remain essentially dry have shown little or no damage.

Role of water in ASR (Larive, 2000)

1. Reactive concrete specimens do not adsorb more water than non-reactive ones: water motive not affected by ASR. Osmosis or imbibitions is not the major cause of swelling
2. ASR-expansion increases with external water supply. The determination of expansion in various storage conditions can be used to assess the influence of water content on concrete swelling.
3. Irregular external water supplies irreversibly affect concrete expansion.
4. Concrete swelling can reach 0.1% even without water ingress, thus leading to potential structural degradation.
5. Expansion and ASR-products formation both stop when concrete loses more than a certain amount of water and they can be reactivated very quickly when water comes back.
Sellier et al. (2004) reported 0.04% shrinkage at each drying step (14 days) on ASR deformation that might be due to the loss of water by the ASR gel itself. After ASR gel forms, a minimum relative humidity of 80% is required to cause significant expansion due to ASR. Data supporting the importance of moisture on expansion are shown in Figure 2.11, where four different reactive aggregates were stored under different moisture conditions, and the expansion of concrete prisms (ASTM C 1293) was assessed (Pedneault, 1996). In this experiment, concrete that was maintained in an environment with less than 80% relative humidity did not undergo significant expansion (expansion was less than 0.04 percent after 2 years). In fact, when the humidity is less than 80%, the mobility of aggressive ions in interstitial solution and availability of water for dissolving silica will be reduced.

However measurement on plain and reactive concrete specimens proved that water supply cause new ASR-swelling if the maximal potential swelling has not been reached before due to the lack of water (Multon et al., 2004).

![Figure 2.11 Effect of relative humidity on expansion using ASTM C 1293 (Pedneault, 1996)](image-url)
2.6.4 Environmental conditions

Concrete elements undergoing ASR and exposed to cyclic exposure to sun, rain, and wind or portion of concrete piles in tidal zones often show severe surface cracking because of induced tension cracking in the surface layer under the expansive thrust of the inner concrete core (Stark and Depuy, 1987). Laboratory experiments performed on concrete cylinders incorporating a highly reactive aggregate and subjected to different combinations of exposure conditions showed that (Bérubé et al., 1996):

1. Test cylinders subjected to wetting and drying cycles expanded significantly less but showed more extensive surface cracking than those constantly stored at 100% humidity and 38\(^\circ\) C.
2. Test cylinders exposed to freezing and thawing cycles expanded significantly more and showed more surface macro cracking than those constantly stored at 100% humidity and 38\(^\circ\) C.

Surface cracking due to the ASR can accelerate the overall deterioration of concrete through process such as corrosion of reinforcing steel, freezing and thawing and sulphate attack. On the other hand, ASR can be induced or accelerated once a concrete element incorporating potentially reactive aggregate has cracked due to one or many of the above deleterious mechanism.

2.6.5 Temperature

Many laboratory investigations have shown that increasing temperature raises ASR expansion rate but may result in lower ultimate expansion. AS reported by Diamond et al. (1981), at high temperature reaction and expansion are initiated early and develop rapidly, but as reaction continues both rate of reaction and rate of expansion slow down. By contrast, reactive concretes and mortar stored at lower temperature react more slowly, but eventually expansion reaches the same level and then exceeds the expansions attained at the higher temperature (Figure 2.12).
This phenomenon can be related to the loss of viscosity of reaction product with increasing the temperature (Capra, 1997). So, reaction products will be more fluid and dissipate in cement paste without any swelling. In the other hand, some aggregate such as Potsdam sandstone do not produce expansion product in concrete at 80 °C (Bérubé et al., 1994) and reactive silica dissolves rapidly before making expansive reaction. The maximal expansion level of this aggregate is observed at the temperature around 40 °C.

Massive concrete elements may be relatively more at risk regarding ASR because of the time required to dissipate the heat resulting from cement hydration. Also high temperature gradients generated at early ages in massive concrete elements can cause micro cracking with the risk of accelerating moisture ingress and consequently the development of ASR.

2.6.6 Concrete permeability and water-to-cement ratio

The availability of moisture is critical to the development of deleterious or excessive expansion due to ASR. A lower water-cement-ratio (W/C) in concrete generally leads to improve mechanical properties, lower internal free water content, lower concrete permeability and reduced movement of moisture inside the concrete. Laboratory investigations have shown that,
for ordinary Portland cement concrete with w/c in the range of 0.35 to 0.55, a reduction in the W/C generally resulted increase of durability and reduction of permeability and ASR expansion (Bérubé and Fournier, 2000). But in the case of concrete with w/c of higher than 0.50, increase in the w/c reduces alkali concentration in the pore solution and reduces ASR expansion.

Numerous cases of ASR in hydraulic dams were reported involving mass concrete with low cement factors, and high W/C and permeability characteristics. In these elements, reaction rate are generally slow but the excess hydration water is likely present in sufficient amounts to sustain ASR for prolonged periods of time (Stark, 1990).

2.6.7 Air entrainment

The use of air entrainment in concrete was reported to reduce expansion due to ASR (Pleau et al., 1989), but no enough to prevent adequately deleterious expansion and cracking due to ASR. Bérubé (Bérubé et al., 1994) reported that using air entraining in concrete containing Kingston and Spratt has no influence on reducing the ASR expansion, but it could be beneficial in the other concrete containing Beauceville Tuff and Potsdam sandstone. Figure 2.13 shows that the higher air content results in lower expansion. The gel expanding into the available capillary pores or voids, without stressing the structure of the cement matrix, could explain this.
Concrete: moist cured, 20 °C;
opalline silica particle size 150-300 μm

Figure 2.13 Influence of air content in hardened concrete on the relationship of expansion and age: W/C = 0.41 and Na$_2$O = 5 kg/m$^3$ (Hobbs, 1988)

2.6.8 Size of aggregate

Many studies have been conducted on the effect of reactive aggregate size on ASR. Wood (1986) showed that for opalline aggregate, ASR expansion is maximum in the particle range of 70-85 μm and using larger or smaller reactive silica reduce the expansion. Other studies on opalline silica showed that using particle size between 20 and 50 μm increase mortar expansion, but below a particle size of 20 μm no abnormal was observed (Hobbs et al., 1979 and Diamond, 1974). Also, Hobbs (1988) results showed that the greatest expansion occurs with the finest gradation (Figure 2.14). Kuroda et al., (2004) reported the expansion of mortar increased as the particle size of reactive aggregate decreases when the volume of the reactive aggregate is the same and resulted the expansion of mortar bar can be expressed by function of the total surface area and the volume of reactive aggregate.
Nevertheless, at very small sizes, in the order of microns, a pozzolanic effect could be expected to reduce the amount of expansion. Another study on limestone aggregate showed no deformation for diameter below 160 μm (Sellier et al., 2004). Also, the relation between expansion and crack is different for the aggregate with different particle sizes. Chengzhi et al. (2004) found that for the aggregate with the particle size of 150-800 μm, the cracks do not be found on the surface of specimen by microscope for the expansion level of 0.175% and when the expansion reaches 0.274%, cracks are visible. In the case of aggregate with the particle size of 5-10mm, cracks are found on the surface of all specimens, when the expansion is more than 0.04%. Therefore, when the particle size of aggregate is larger, it is easier that concrete cracks even for the low expansion level. The reason is that expansion stress caused by ASR could be well distributed when aggregate is smaller.
It seems that three distinct effects should be considered (Ramyar et al., 2005):

- The reaction rate that would be faster for smaller particle size.
- Long-term expansion that could be larger for coarse aggregate.
- The angularity of particles has negligible effect on ASR expansion for very large and very small particles.

2.7 Laboratory test method for assessing

This section provides an overview of available laboratory test methods to assess ASR of aggregates and measure the effectiveness of various methods to mitigate ASR in concrete. There are several standard test methods to assess ASR. In Canada, among the laboratory tests specified, only two are recommended as suitable tests for assessing ASR: ASTM C 1260 and ASTM C 1293.

- ASTM C 1260

ASTM C 1260, is often referred to the accelerated mortar bar test, has been adopted by various countries and agencies, including the United States and Canada. The test entails casting mortar bars that contain the subject aggregate (either coarse or fine), which is processed to a standard gradation. The mortar bars are then removed from their moulds after 24 hours and placed in water at room temperature. The temperature of the water is then raised to 80° C in an oven, and the mortar bars are stored in this condition for the next 24 hours. After the bars are removed from the water, they are measured for initial length and then submerged in a 1 normal (N) NaOH solution at 80° C, where they are then stored for 14 days. Length change measurements are made periodically during this storage period. The total expansion at the end of the 14-day soaking period typically is used in specifications, although the expansion limits specified by different agencies vary. For example, the expansion criteria established by ASTM 1260 and CSA A23.2-25A are as follows:
ASTM C 1260 expansion criteria:
< 0.10% is considered non reactive, 0.10% to 0.20 % is considered potentially reactive and more than 0.20 % is considered reactive

CSA A23.2-25A expansion criteria:
> 0.15 % is considered reactive (all aggregates except limestone)
> 0.10 % is considered reactive (limestone aggregates only)

ASTM C 1260 is recognized as a very severe test method because of the extreme test conditions, specifically the use of a highly alkaline storage solution and high temperature. Because of this inherent severity, the test has been shown to identify some aggregates as being reactive, even though they have performed well in concrete prism testing (ASTM C 1293) and in field applications (Bérubé and Fournier, 2000). Therefore, an aggregate should not be rejected solely based on ASTM C 1260 results unless the reactivity is confirmed using ASTM C 1293. Whenever data are available from both ASTM C 1260 and ASTM C 1293 results should govern.

- ASTM C 1293

ASTM C 1293, commonly referred as the concrete prism test, is generally considered the most accurate and effective test in predicting the field performance of aggregates. In this test, concrete is cast with cement content of 420 kg/m$^3$. The cement is required to have equivalent alkali content between 0.8-1.0 % and additional alkalis (NaOH) then are added to the mixing water to obtain a total alkali content of 1.25 % (by mass of cement), which equates to a total alkalis content in the concrete mixture of 5.25 kg/m$^3$. Concrete prisms are cast, cured for 24 hours at 23° C, and then stored over water at 38° C. Expansion measurements are taken at regular intervals for 1 year. When testing SCMs or lithium compound, the test typically is carried out for 2 years. This relatively long period for conducting ASTM C 1293, either 1 or 2 years, has been the major drawback for the test and has limited its use somewhat. An expansion limit of 0.04% typically is specified, as this value has been reported to correlate well with cracking of test prisms. This expansion limit (0.04%) is referenced in the appendix to ASTM C 1293. As part of the most recent guidance provided by CSA (2000a), the expansion limits for the concrete prism test (CSA A23.2-14A) were delineated further to assess aggregate reactivity as follows:
CSA A23.2-14A expansion criteria:
< 0.04% is considered non-reactive
0.04% to 0.12% is considered marginally reactive
> 0.12% is considered highly reactive

ASTM C 1260 and ASTM C 1293 are the two recommended tests for ASR. ASTM C 1293 generally is considered to be more representative of field performance. To supplement these tests, a petrographic evaluation of a given aggregate (ASTM C 295) is also suggested, but not required. A thorough petrographic evaluation provides useful information about the types and amounts of minerals present in an aggregate, and can be used to identify a wide range of reactive components. However, because of inherent difficulties in identifying all potentially reactive phases within aggregate, petrographic analysis findings alone should not be used to accept or reject a given aggregate, but rather to supplement the findings of other laboratory evaluations.

Field performance histories of aggregates, supplemented with petrographic analysis of field concrete (ASTM C 856) containing the subject aggregate, also provide fairly qualitative and subjective result. Also, as in the case of petrographic analysis of aggregates (ASTM C 295), field performance evaluations should not be used solely to accept or reject a given aggregate for use in new structures.

The other tests methods for alkali-silica reactivity are:

- ASTM C 227: Potential alkali-reactivity of cement-aggregate combinations (mortar-bar method)
- ASTM C 289: Potential alkali-silica reactivity of aggregates
- ASTM C 294: Constituents of natural mineral aggregates
- ASTM C 295: Petrographic examination of aggregates for concrete
- ASTM C 342: Potential volume change of cement-aggregate combinations
- ASTM C 441: Effectiveness of mineral admixtures or GBFS in preventing excessive expansion of concrete due to alkali-silica reaction
- ASTM C 856: Petrographic examination of hardened concrete
- Accelerated concrete prism test (modified ASTM C 1293)
• ASTM C 1567: Potential alkali-silica reactivity of combinations of cementing materials and aggregate (accelerated mortar-bar method)

2.8 Destructive and non-destructive test methods for evaluating the damage of concrete affected by ASR

2.8.1 Destructive tests methods

Destructive tests are usually used to evaluate the quality of mortar and concrete specimens made in laboratory or the core samples extracted from a concrete structure. These tests can be ordinary tests such as mechanical tests or special tests for evaluation the state of ASR damage in concrete such as Damage Rating Index method (DRI) and Stiffness Damage Test (SDT). These methods are explained as follow:

- **Damage Rating Index**

A system called the Damage Rating Index (DRI) introduced by Dunbar and Grattan-Bellew (1995) and then has been used by some other authors (Shrimer, 2000, Rivard, 2000, 2002, Smaoui et al., 2004, Saint-Pierre, 2007). This method is based on the determination by petrographic examination of the internal damage in concrete. It takes into account several defects such as the abundance and distribution of reaction products and the magnitude of internal microcracking, within the aggregate and in the cement paste. In this method, a polished specimen of concrete is examined at a certain magnification, and the number of features associated with ASR is counted. The total number of each feature is then multiplied by weighting factors. Each factor is given a weighting factor to reflect its importance in propagating damage in concrete. The total of the contribution of each feature multiplied by its weighting factor become the Damage Rating Index (DRI) for the sample. The DRI is normalized to a standard area (currently 100cm$^2$).

The higher the DRI, the greater the amount of ASR damage sustained by the concrete, as evidenced in the feature identified in the petrographic examination of the concrete specimens. The features listed in Table 2.3 are those, which are currently used in this diagnostic method, for
examining concrete from existing structure (the weighing factors of this table have been modified according to P.E. Grattan-Bellew).

**TABLE 2.3 WEIGHTING FACTOR FOR EACH FEATURE IN DRI METHOD**
(Dunbar and Grattan-Bellew, 1995)

<table>
<thead>
<tr>
<th>Feature</th>
<th>Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Closed cracks in aggregates</td>
<td>0.75</td>
</tr>
<tr>
<td>Open cracks in aggregates</td>
<td>4</td>
</tr>
<tr>
<td>Cracks with gel in aggregates</td>
<td>2</td>
</tr>
<tr>
<td>Debonded aggregate</td>
<td>3</td>
</tr>
<tr>
<td>Reaction rim around aggregate</td>
<td>0.5</td>
</tr>
<tr>
<td>Crack in cement paste</td>
<td>2</td>
</tr>
<tr>
<td>Cracks with gel in cement</td>
<td>4</td>
</tr>
<tr>
<td>Air voids with gel</td>
<td>0.5</td>
</tr>
</tbody>
</table>

Saint-Pierre (2006) reported that DRI was directly influenced by expansion. The damage as measured by DRI increased with increasing the expansion. The magnitude of DRI and the types of defects detected by this method may be related to the types of aggregate used in concrete. For example, this value in concrete containing Spratt limestone was more important than that of concrete containing Potsdam sandstone (Saint-Pierre, 2006). The reaction rim is usually observed on the surface of concrete containing Potsdam sandstone and the thickness of rim may indicate the degree of ASR damage in concrete (Section 2.3.2).

- **Stiffness damage test**

Stiffness Damage Test (SDT) was proposed by Chrisp (Chrisp et al., 1989). It is based on the cyclic compressive loading of concrete core sample (5 cycles) between 0 and 10MPa (Smaoui et al., 2004). They discovered that the value of modulus of elasticity measured at the time of the first loading was significantly less for sample extracted from cracked concrete than for sample
from undamaged concrete and also the area occupied by the stress-strain curve was clearly larger than that for undamaged concrete. But the results obtained from SDT seem to depend on the diameter of specimen and type of aggregate involved (Smaoui et al., 2004).

2.8.2 Non-destructive test methods

Non-destructive test (NDT) methods have been widely used to evaluate existing structure, to detect imperfections, weaknesses and deterioration.

These methods can be the in-situ tests such as visual inspection and surface cracking measurement to evaluate current condition of concrete or they could be based on collection of series of concrete cores selected from different depths of structure for examination in the laboratory to evaluate the alkali-silica reactivity and future deterioration. The following provide brief details of the values that could be measured by NDT techniques more commonly for assessing the alkali-silica reaction in concrete.

- Visual inspection

Visual inspection can provide a wealth of information that may lead to positive identification of the cause of observed distress. Optical magnification allows a more detailed view of local areas of distress such as microcracking. Borescopes, fibrescopes and digital video camera are used in the visual inspection.

- Surface cracking

The institution of structural engineering (ISE, 1992) proposed surface cracking mapping method that consists of measuring the width of cracks intersecting at least five parallel lines 1m long, traced on the concrete and separated by at least 250mm from one another. The total deformation due to ASR and other deleterious mechanism is assumed to be equal to the sum of the widths of all intersected cracks divided by the total length of the reference lines. This method has the advantage of being non-destructive, inexpensive, and easy to perform and easily repeated for monitoring changes with time.
- Ultrasonic pulse velocity

Ultrasonic methods have been used since the mid 1940s for concrete testing. The most commonly measured parameter is the ultrasonic pulse velocity (UPV). The basic procedure is to measure the travel time of an ultrasonic pulse generated by a transducer, passing through concrete (Popovics, 1998). Longitudinal wave can be measured in a more reliable manner than other waves because:

- The velocity of the longitudinal wave is greatest.
- The energy loss is smallest.
- Their amplitude is largest.
- It is relatively simple to separate them from other wave types.

Usually, this test determines the velocity of propagation of compression wave in concrete. With the measuring of the transmission time and calculation of the pulse velocity, concrete properties such as uniformity of the concrete, cavities and cracks could be determined (Whitehurst, E.A., 1951) Table 2.4 provides the classification commonly used for assessing concrete quality with regards to UPV.

<table>
<thead>
<tr>
<th>Pulse velocity, m/s</th>
<th>Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Above 4570</td>
<td>Excellent</td>
</tr>
<tr>
<td>3660 to 4570</td>
<td>Generally good</td>
</tr>
<tr>
<td>3050 to 3660</td>
<td>Questionable</td>
</tr>
<tr>
<td>2130 to 3050</td>
<td>Generally poor</td>
</tr>
<tr>
<td>Below 2130</td>
<td>Very poor</td>
</tr>
</tbody>
</table>

TABLE 2.4  CLASSIFICATIONS FOR USING VELOCITY AS AN INDICATOR OF QUALITY OF CONCRETE (WHITEHURST, 1951)
This technique has several inherent advantages: it penetrates the full depth of members; velocity measurements can be taken quickly, repeatedly and accurately. According to the arrangement of the transducers, properties can be calculated over the whole transverse section.

The most important factors that affect the measured ultrasonic pulse velocity and should be considered are:

- Moisture content; an increase in moisture content increases the pulse velocity.
- Presence of reinforcement oriented parallel to the pulse propagation direction; the pulse may propagate through the bars and result in an apparent pulse velocity that is higher than through concrete.
- Presence of cracks and voids; these can increase the length of the travel path and result in a longer travel.

Goueygou et al. (2003) reported that velocity decreases with increasing water to cement ratio, porosity and permeability of concrete. Presence of cracks and voids and uniformity of concrete increases the length of the travel path and result in a longer travel time. Thus, the ultrasonic pulse velocity could be useful for assessing ASR in concrete. Ohtsu et al. (2003) have estimated concrete properties by elastic wave methods. According to their results, compressive strength of concrete could be determined by ultrasonic wave.

The type of aggregate used in concrete is another important factor influencing on the result of UPV tests. UPV of limestones is around 6000 m/s (Sulub, 1990, Rivero, 1996 and Carrillo, 2000) whereas the UPV of Potsdam sandstone is between 1400 m/s and 4300 m/s. Thus, it may be estimated that the UPV of concrete containing limestone aggregate would be higher than that of concrete containing Potsdam sandstone. This topic will be approved in section 8.3.
Also, the depth of the surface crack in damaged concrete can be determined according to RILEM NDT1. By measuring $T_c$ and $T_o$ at more than three locations along the surface crack, the depth $d$ is estimated:

$$d = L \left[ \left( \frac{T_c}{T_o} \right)^2 - 1 \right]^{1/2},$$

Where:

$L$: Distance between the driving and the receiving sensors after the calibration

$T_c$: Time-of-flight on cracked surface

$T_o$: Time-of-flight on the non-cracked surface

If ordinary concretes haven’t been affected by ASR, this velocity rises until 30 days (hardening of the concrete) and then it decrease slightly (thermal dilation). In the other hand, in concrete containing reactive aggregate, the velocity rises between 1 and 10 days and then fall until 30 days because of appearance of the cracks and finally it increases until 80 days, when ASR gel fills of the cracks. It’s claimed by some authors that a crack opening of 0.025mm in concrete will cause total reflection of acoustic waves (Bernstone et al., 2004).

Also the result of test on concrete prisms containing different kinds of aggregate demonstrates that velocity is sensitive to ASR-induced cracking in the concrete. Blight (Blight et al., 1986) reported that ASR damage made significant reduction in the UPV of concrete surface whereas this value in the interior of concrete structure did not reduced. Okada (1986) suggested that the UPV of ASR damaged concrete is generally less than 3500m/s, when this value was measured in site and the UPV of core samples extracted from the structure varied between 3000m/s and 3700m/s. Akashi et al. (1986) reported the relationship between the reduction of mechanical properties and pulse velocity in concrete affected by AAR. They concluded that the degree of deterioration of concrete damaged by ASR could be estimated by the spectral analysis of ultrasonic pulse waves passing through the concrete by applying the linear system. Following their test, Amasaki and Takashi (1989) observed the energy of the response of spectrum frequency of ultrasonic wave is a good indicator of degradation by AAR. But Pleau et al. (1989) concluded pulse velocity is not a good indicator of development of alkali aggregate reaction. This value did not change during the reaction development.
Ono and Taguchi (2000) observed the reduction of pulse velocity and compressive strength of concrete affected by AAR. Velocity reduces with an increase in free expansion (Gallo et al., 2006). Pulse velocity of reactive concrete decreases 4% for the expansion degree of 0.2% (Saint-Pierre, 2006). On the other hand, some author observed pulse velocity is relatively insensitive to strength for mature concrete.

Bakker et al. (2003) investigated the ASR in two-dimensional reinforcement configuration in the concrete bridge decks (no shear connection between the top and bottom reinforcement) by NDT. They measured expansion in the bridge deck in vertical direction by vibrating wire sensors placing vertically in the bridge deck. This type of sensors consists of a thin wire, anchored at both ends, and an electric coil. An electric field sets the wire into vibration. As the concrete expands, the strain in the wire increases, resulting in a higher vibrating frequency.

Gudmundsson (2004) introduced two similar instrument based on sending and receiving ultrasonic waves (pulse-echo): a relatively simple crack depth instrument and a more complex flaw detector. The main conclusion of this study on concretes affected by ASR is that these instruments are very sensitive to damage concrete by their mapping and measure crack depth with a quick, accurate and inexpensive method that cover large area of concrete without the need to drill cores from the structure.

- **Resonant frequency**

Measuring resonant frequency is a NDT method based on the propagation of a stress wave through concrete. This technique consists of generating a wave by using a mechanical impact. This test measures the fundamental transverse, longitudinal and torsional resonant frequencies of concrete prism and cylinders for the purpose of calculating dynamic modulus of elasticity, the dynamic modulus of rigidity and dynamic Poisson’s ratio (Figure 2.15).

A steel ball tapped lightly against the surface normally causes the impact. The required diameter of the ball depends on the depth of concrete and the minimum size of the significant defect. A larger ball produces a higher energy pulse that can detect flaws deeper in the specimens, but may not detects that are smaller or closer to the surface.
As resonant frequencies are related to physical properties of the structure, it could be measured in concrete damaged by ASR in order to detect the global changes of the physical properties such as modulus of elasticity or the losses of the bending rigidity. Ludwig (1981) has reported that resonant frequency of concrete affected by AAR reduces with the expansion of specimen. Monette (Monette et al., 2000) has confirmed this hypothesis by observation of 7.8% reduction in dynamic modulus calculated from resonant frequency of an ASR affected concrete. Siegert (Siegert et al., 2005) reported amount of stiffness reduction based on resonant frequency of ASR damaged concrete beam in laboratory condition in the range of 12 to 20%. Also the results of Saint-Pierre (Saint-Pierre, 2006) show that longitudinal resonant frequency decreased by 12% in concrete containing reactive siliceous limestone for the expansion level of 0.2%.

![Diagram of resonant frequency modes](image.png)

**Figure 2.15** Location of impact and needle pickup in resonant frequency method (ASTM C 215)
Stark (2003) reported that it would be possible to determine AAR by non-destructive acoustic test procedures such as ultrasonic scanning, resonant frequency and natural-period-of-vibration techniques. In order to eliminate the influence of the mechanical coupling and to obtain high speeds of measurements and short distances between measuring points, laser techniques could be used which allow the development of ultrasonic scanning procedures into no contacting measuring procedures. In the first basic tests the laser excitation of vibrations was demonstrated in various materials (concrete, aerated concrete, brick, marble). Another advantage of the laser excitation is the technical possibility of exciting P- and S-waves in one measuring process. The investigations into the laser reception of sound waves were made with a laser scanning vibrometer PSV 300F. The assessment of the microstructure of this concrete layer plays an important role in investigations into the durability of concrete (e.g. frost resistance, alkali-aggregate reaction). An additional advantage is that building parts can be examined from a greater distance without scaffolding and marking of measuring points.

- **Non-linear acoustics**

Non-linear acoustic method is a sensitive method to ASR damage in concrete without high dependency on testing conditions. Briefly, when the fundamental ultrasonic frequency interacts with a material such as concrete, the non-linear interaction between the ultrasonic wave and the material generates harmonics. As damage increases, the magnitude of the non-linear interaction increases and causes a greater portion of the fundamental frequency to be converted to higher harmonics. This method is sensitive and enable to detect the microcracks which could not been detected by linear methods (Donskoy, 1997)

Stauffer (Stauffer et al., 2005) studied UPV, resonant frequency and non-linear acoustic methods for evaluation of damage on concrete specimens by loading them in compression. The results showed that increasing damage may result the increasing in harmonic generation levels. But UPV and resonant frequency test methods were generally insensitive to damage up to approximately 60% of the ultimate strength of the concrete. This method is less dependent on the repeatability of the coupling of the transducers (Wu et al., 2000) and much more sensitive to
lower damage states than pulse velocity method (Wu et al., 2000, Kodjo, 2008). Kodjo (2008) showed the parameters measured by non-linear acoustic tests are too sensitive to ASR damage. These parameters increase significantly by expansion.

- **Acoustic Emission**

Acoustic Emission (AE) technique is a non-destructive test that offers the possibility to listen to the damage process when and where it occurs. This technique is a passive method which detects energy released when microstructural changes in a material take place. The AE sensors, mostly PZT transducers, resonant or broadband, are used to pick up the elastic stress wave from the material and convert them into the signal. The possibility of evaluation the ASR damage in mortar and concrete by AE technique were already studied in reactive and non-reactive mortar and concrete made in laboratory. This method was performed by counting of the number of detected signals produced by cracking the aggregates due to ASR (Taylor, 1997). The results indicated the number of detected signals in reactive mortar and concrete were much more than that of non-reactive mortar and concrete.

2.8.3 Effect of concrete characteristics and environmental condition on the results of non-destructive tests

Type of aggregate used in concrete and their content directly influence on compressive strength and the results of NDT. Some type of aggregate such as limestones and granite are very dense and concretes containing these types of aggregates have the higher compressive strength. In sections 7.3.1 and 8.3.1, the high UPV of concrete containing limestone aggregates severely affected by ASR will be demonstrated.

The high water to cement ratio cause the high porosity of concrete and reduces the UPV (Lafhaj, 2006). Popovics (Popovics, 1998) reported the pulse velocity was less affected by the porosity in concrete damage by freezing and thawing than the resonant frequency. The losing in internal structure of concrete caused by repeated freezing and thawing reduced the modulus of elasticity less than dynamic modulus calculated from resonant frequency.
Also, more the W/C of concrete in higher, more the effect of humidity is important. In High performance concrete, humidity has no any influence on the results of UPV. But in ordinary concrete, the UPV of a saturated concrete is up to 5% more than that of dry concrete proportionate the moisture content (Rhazi, 2004). In a full saturated mortar, longitudinal velocity decreases from 5100m/s to 4300m/s, when the porosity rises from 8% to 14%. But when the mortar is dried, the variation of porosity reduces the longitudinal velocity from 4500m/s to 4100m/s (Lafhaj, 2006).

The variation of temperature of concrete specimens between 5 and 30°C has a little influence on the results of UPV. This value increases with increasing the temperature (Table 2.5).

<table>
<thead>
<tr>
<th>Temperature of concrete (°C)</th>
<th>Correction of velocity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Dry concrete</td>
</tr>
<tr>
<td>60</td>
<td>+5</td>
</tr>
<tr>
<td>40</td>
<td>+2</td>
</tr>
<tr>
<td>20</td>
<td>0</td>
</tr>
<tr>
<td>0</td>
<td>-0.5</td>
</tr>
<tr>
<td>&lt;-4</td>
<td>-1.5</td>
</tr>
</tbody>
</table>

2.9 Effects of ASR on engineering properties of concrete

2.9.1 Compressive strength

The reduction in compressive strength was found to depend upon the type of specimen used and the expansion level reached. Swamy and Al-Asali (1988) observed 40% reduction in compressive strength of concrete containing reactive aggregate (Opal) and alkali content of 5.2Kg/m³ for the expansion level of 0.6%. Also, Pleau (Pleau et al., 1989) reported no
significant reduction in compressive strength for expansion levels less than 0.12%, but reduction of 30-50% for expansion in order of 0.25%. Wood (Wood et al., 1989) reported a significant alteration of stress-strain curves due to AAR as measured during the stiffness damage test. Concretes affected by ASR typically showed significantly higher strains at the peak stress, and were consequently more ductile than unaffected concretes.

2.9.2 Tensile strength

According to Swamy and Al-Asali (1988), for the expansion level between 0.05 and 0.10%, the reduction in tensile strength (indirect test) is around 30%. Reduction in tensile strength may require special consideration since losses of 40 to 80% were reported depending on the test method used and the expansion level reached.

Nixon and Bollinghaus (1985) suggested the use of tensile-to-compressive strength ratio as a good indicator for assessing internal damage due to ASR. In a sound concrete, this typically varies from 0.07 to 0.11. In investigations dealing with AAR, it was suggested that a ratio <0.06 was indicative for internal deterioration due to AAR and freezing and thawing cycles.

2.9.3 Flexural strength and modulus of elasticity

Flexural strength and modulus of elasticity are essential engineering properties for the concrete element to keep its integrity and carry on its intended functions (Swamy, 1995). A number of tests have shown that these properties are highly sensitive to change occurring in the internal structure of the concrete due to AAR, and those losses in static modulus of elasticity and flexural strength could lead to substantial reductions in flexural rigidity and structural stiffness of affected members.

Hobbs (Hobbs, 1987) reported the results of mechanical tests on 14 years old high performance concrete affected by ASR ($f'_c = 70\text{-}90\text{MPa}$). The results showed that the reductions in mechanical properties have been less than those of ordinary concrete (10-15% in compressive resistance, 15-20% in tensile strength and 20-40% in modulus of elasticity).

According to Clark (1990), there is no significant reduction in modulus of elasticity for expansions < 0.05%. Losses in modulus of elasticity and flexural strength between 20 and 60%
were reported for expansion ranging from 0.1-0.3% and could even reach 80% for the former at very high expansion levels. Important reduction in the modulus of elasticity can be obtained even at low expansion level or when compressive strength of the affected concrete is still increasing.

Batic (Batic et al., 2004) reported the reduction of flexural tensile strength near 40% that was higher than the reduction observed in compressive strength (25%). However this reduction is not as drastic as the reduction in static modulus of elasticity. Figure 2.16 shows stress-strain curve of concrete affected by ASR ($\varepsilon_\text{ex} \times 10^3 = \text{ASR expansion in the direction of uniaxial loading}$).

![Stress-strain curves for concrete with variable ASR expansion (Wen, 1993)](image)

Figure 2.16 Stress-strain curves for concrete with variable ASR expansion (Wen, 1993)
CHAPTER 3

OBJECTIVE AND EXPERIMENTAL PROGRAM

3.1 Objective

Concrete structures may be at risk to damage by ASR, when they are maintained under the conditions supporting the reaction. This reaction is one of the main causes of concrete deterioration in Canada. The characterization of the state of ASR damage in concrete and the optimal management of the structures affected by ASR requires finding the answers to the following questions:
- What is the state of ASR damage in concrete?
- How long the reaction will continue?
- Which will be the highest level of expansion?

Evaluation of the state of ASR damage in concrete appropriated several studies in Civil engineering Department of the University of Sherbrooke. These investigations have been focused on the residual expansion of concretes containing two types of reactive aggregates (Spratt limestone and Potsdam sandstone) by measurement the variation of alkalis content in the pore solution, petrography analysis and also monitoring the reaction progression with ultrasonic pulse velocity on laboratory concrete specimens.

In this study, ASR evolution will be followed by chemical test in mortar and non-destructive tests in mortar, laboratory concrete and concrete core.

Chemical analysis is based on the modeling of reaction which has already been developed by Bulteel (2000) and Monnin (2005). According to this mechanism, it would be possible to determine the number of reactive silica and dissolved silica at different reaction degrees in mortars affected by the reaction.
ASR progression in concrete by non-destructive test (NDT) methods is the main part of this study. Development of non-destructive methods, aiming at monitoring ASR evolution, would help managing the structures rehabilitation works. Thus, the evolution of damages associated with ASR is monitored based on ultrasonic wave propagation (measurement of ultrasonic pulse velocity) and acoustic tests (measurement of resonant frequencies and nonlinear acoustical properties) in order to establish criteria associated to each method.

In order to achieve this objective, non-destructive test methods were used during the reaction progression in mortar bar (25x25x75mm), laboratory concretes specimens (100x200 mm) and concrete cores extracted from two ASR-affected locks kept in accelerated conditions. Also, the reaction evolution will be monitored by various tests such as expansion measurement, mechanical tests, and petrographic examination. By knowing the influence of ASR damage on physical, mechanical and petrographic features, it can be possible to determine:

- The residual expansion as a regular parameter for measurement the reaction degree
- The effect of ASR damage on the structure of mortar, concrete and concrete cores
- Relationship between expansions and the sensitivity of each method to ASR development

3.2 Experimental program

To be able to compare the effect of reactive aggregate on concrete, two limestone aggregates with similar geological nature were used: Limeridge limestone as the control aggregate and Spratt limestone as the reactive aggregate. The main reason that limestones were selected for this work is that most structures affected by ASR have been built with this type of aggregates.

In the first step, properties of aggregates were characterized and their reactive silica content was measured. Then, chemical test would be performed on mortar at different stages of ASR damage in order to determine the potential for residual expansion of affected structure by measurement the quantity of reactive silica and dissolved silica content of aggregate.
In the next step, destructive and non-destructive tests were carried out to measure the variation of physical and mechanical properties of mortar and concrete affected by ASR. Experimental program of this study is explained follow (It is schematized in Figure 3.1).

![Experimental programme](image)

**Figure 3.1 Experimental program**

- **Characterization of aggregates:**

  In a first stage, the aggregates were ground to obtain a [0.16-0.63mm] size distribution. The characterization of aggregate consists of:

  - Chemical composition by X-Ray flourescence examination;
  - Crystallography analysis by X-Ray diffraction in order to determine presence of crystalline phase in aggregate and their degree of crystallinity;
• Petrographic analysis by SEM (scanning electron microscopy);
  - Tests on mortars:

• Expansion measurement and mass variation;
• Chemical test on mortars in order to measure the quantity of different forms of silica (Q₄, Q₃ and dissolved silica);
• Petrographic analysis by SEM (scanning electron microscopy);
• Compressive strength of cubic specimens;
• Non-destructive tests;
  - Ultrasonic pulse velocity
  - Dynamic modulus of elasticity (E_d) calculated from longitudinal resonant frequency

- Tests on laboratory concretes:

• Expansion measurement and mass variation;
• Mechanical tests:
  - Compressive strength;
  - Tensile strength;
  - Static modulus of elasticity;
• Non-destructive tests:
  - Ultrasonic pulse velocity;
  - Dynamic modulus of elasticity (E_d) calculated from transversal and longitudinal resonant frequencies;
  - Nonlinear frequency shift;
• Petrographic examination according to the Damage Rating Index method

- Tests on cores extracted from locks affected by ASR:

• Expansion measurement and mass variation;
• Non-destructive tests:
  - Ultrasonic pulse velocity;
- Dynamic modulus of elasticity calculated from transversal and longitudinal resonant frequencies;
- Nonlinear frequency shift;
- Petrographic examination by Damage Rating Index method

3.2.1 Characterization of aggregates

The first step of this study is the aggregate characterization. In order to determine the mineralogical composition and the mode of formation of aggregates, petrographic examination was performed on aggregates. The basic information on petrographic examination of aggregate is described in ASTM C 295.

- Chemical composition by X-Ray fluorescence

The chemical composition of the aggregates was determined by X-ray fluorescence to measure the percentage of silica and other various elements in the aggregates. Energy dispersive X-ray fluorescence technology (ED-XRF) provides one of the simplest, most accurate and most economic analytical methods for the determination of the chemical composition of many types of materials. It is non-destructive and reliable, requires no, or very little, sample preparation and is suitable for solid, liquid and powdered samples.

- SEM (Scanning electron microscopy)

By scanning an electron probe across a specimen, high-resolution images of the morphology or topography of a specimen, with great depth of field, at very low or very high magnifications can be obtained. Compositional analysis of a material may also be obtained by monitoring secondary X-rays produced by the electron-specimen interaction. Thus detailed maps of elemental distribution can be produced from multi-phase materials or complex, bioactive materials.
- **X-Ray diffraction**

The analysis by X-Ray diffraction made it possible to determine the crystalline phases present in the aggregates. Diffraction occurs as waves interact with a regular structure whose repeat distance is about the same as the wavelength. It happens that X-Rays have wavelengths on the order of a few angstroms, the same as typical interatomic distances in crystalline solids. X-Ray can be diffracted from minerals that, by definition, are crystalline and have regularly repeating atomic structures. A diffraction sample records the X-Ray intensity as a function of 2-theta angle.

3.2.2 Test on mortar

For each aggregate, a mortar conforming to CSA A23.2-25A (Test method for detection of alkali-silica reactive aggregate by accelerated expansion of mortar bars) was prepared and stored in 1 molar NaOH solution at 38 °C.

- **Mortar bar expansion**

In order to detect the potential for deleterious expansion of mortar, length change and mass variation of three mortar bars have been measured periodically during two years.

- **Chemical test**

After the measurement of the expansion of mortar bars, the reaction progression had to be measured by chemical test. According to the model of Bulteel (Bulteel et al., 2000) and Monnin (Monnin, 2005), two reactions steps are taken into account in the mechanism:

- Formation of $Q_3$ sites due to the first siloxane bond breaking up by attack of hydroxide ions.

$$2 \text{SiO}_2 + 2 \text{OH}^- \rightarrow 2\text{SiO}_3^{2-} + \text{H}_2\text{O}$$
• Dissolution of silica due to continued attack of hydroxide ions on the $Q_3$ sites to form silica ions

\[
\text{SiO}_{5/2}^+ + \frac{1}{2} \text{H}_2\text{O} + \text{OH}^- \rightarrow \text{H}_2\text{SiO}_4^{2-}
\]

The aggregates have to be extracted from the cement matrix. The extraction method is a chemical treatment using a cold 1M HCl solution based on the protocol developed by Garcia-Diaz (Garcia-Diaz et al., 2005). During this attack, the cement paste and C-S-H/C-K-S-H phase produced by the reaction are removed and $Q_3$ sites are protonated. After this attack, the remaining aggregate is constituted of $Q_4$ and $Q_3$. The silica content of remaining aggregate could be checked by X-ray florescence.

The content of $Q_3$ sites is obtained by a thermal treatment of the residual silica at 1000°C, silanole groups is condensed back to silica $Q_4$ and release water as follow:

\[
2 \text{SiO}_{5/2} \rightarrow 2 \text{SiO}_2 + \text{H}_2\text{O}
\]

The released water measured by thermogravimetry allows the calculation of the number of $Q_3$ for a given time:

\[
n_{Q_3}(t) = 2 \left( \frac{M_{H_2O}}{M_{H_2O}^{\text{mol}}} \right)
\]

Where:

$M_{H_2O}$ is in gram

$M_{H_2O}^{\text{mol}} = 18$ g/mol

So, the $Q_4$ for a given time could be calculated as follow:

\[
n_{Q_4}(t) = (\frac{M_{SO_2}}{M_{SO_2}^{\text{mol}}}) - n_{Q_3}(t)
\]

Where:

$M_{SO_2}$ is in gram
Therefore the number of moles of dissolved silica will be calculated as follow:

\[ nQ_0(t) = (nQ_4(0) - nQ_4(t)) + (nQ_3(0) - nQ_3(t)) \]

Based on above data, the two degree of reaction will be defined as:

- The change of \( Q_3 \) in mortar bar during the reaction:
  \[ dnQ_3(t) = nQ_3(0) - nQ_3(t) \]

- The formation of \( Q_0 \) in mortar bar during the reaction:
  \[ dnQ_0(t) = nQ_0(t) \]

- **SEM (scanning electron microscopy)**

SEM was used to show cracking, ASR gel, aggregate-paste interphases and the reaction rim. The manifestation of reactivity provides graphic evidence of the presence of deleterious ASR reaction product and also provides definitive morphological and compositional information.

- Resonant frequency and Pulse velocity

The following values were measured by NDT technique for assessing the ASR in mortar and concrete:

- **Ultrasonic pulse velocity:**

This test determines the velocity of propagation of compression wave in concrete. It is an automatic measurement by determination of transmission time (Figure 3.2) using an ultrasonic instrument (TICO) with 54 KHz transducers. This method was explained in section 2.8. According to ASTM C 597-97, an ultrasonic pulse velocity of mortar and concrete is calculated as follows:
\[ V_L = \frac{L}{\Delta T} \]

Where:

\[ V_L = \text{Pulse velocity, m/s} \]

\[ L = \text{distance between transducers, m} \]

\[ \Delta T = \text{transit time, s} \]

Figure 3.2  A view of ultrasonic pulse velocity measurement

Resonant frequency:

Measuring resonant frequency is a NDT method based on producing large amplitude vibrations through concrete. This method was explained in section 2.8. In order to determine resonant frequency of each aggregate, 3 mortar bars were used. Then resonant frequency was measured according to ASTM C 215-02 (Standard test method for fundamental transverse, longitudinal, and torsional resonant frequencies of concrete specimens) (Figure 3.3).
According to ASTM C 215-02, dynamic modulus of elasticity ($E_d$) can be calculated from fundamental longitudinal resonant frequency of concrete prism as follow:

$$E_d = DM \times f_f^2$$

Where:
- $f_f$ = Fundamental longitudinal frequency, Hz
- $M$ = Mass of specimen, Kg
- $D = 4 \left( \frac{L}{bt} \right), \frac{N.s^3}{Kg.m^2}$
- $L$: Length of specimen, m
- $t, b$: dimension of cross section of prism

Also, the dynamic modulus of elasticity can be calculated from fundamental transversal resonant frequency as follow:

$$E_d = C \times M \times f_T^2$$
Where:

\( E_d \): Dynamic modulus of elasticity

\( f_T \): Transversal vibration frequencies of concrete

\( M \): Mass of specimen (Kg)

\[
C = 0.9464 \left( \frac{L^3T}{bt^3} \right) \text{ for a prism}
\]

\( L \): Length of specimen, m

\( t, b \): dimension of cross section of prism

\( T \): correction factor that depend to the ratio of \( v/3.464 \) for a prism and Poisson coefficient

- **Compressive strength**

According to ASTM C 109/109M 99 (Standard test method for hydraulic cement mortar), compressive strength of mortars was determined using 2-in cubes specimens. The mortars used in this test were prepared like those prepared for mortar bar test. Three specimens from a batch of mortar for each age were made. After curing, 6 cubes were placed in saturated lime at 23°C and the others were placed in 1 molar NaOH solution at 38°C to accelerate ASR.

3.2.3 Test on concretes

- **Expansion measurement and mass variation of concrete**

For each mixture (reactive and non-reactive), 4 prisms and 24 cylinders were cast. In order to evaluate potential reactivity of aggregates, length change and mass variation of prisms were measured during 100 weeks (Figure 3.4)
- Compressive strength

This test consists of applying a compressive axial load to moulded cylinders at a rate, which is within a prescribed range until failure occurs (Figure 3.5). The end of compression test specimens that have not been plane within 0.002in (0.05mm) is sawed to meet that tolerance. Testing machine and its accuracy were in accordance with ASTM C 39/C 39M-03.
- **Splitting tensile strength**

This test consists of applying a diametric compressive force along the length of the cylindrical concrete specimen at a rate that is within a prescribed range until failure occurs (Figure 3.6). This loading induces tensile stresses on the plane containing the applied load and relatively high compressive stresses in the area immediately around the applied load. Tensile failure occurs rather than compressive failure because the areas of load application are in a state of triaxial compression, thereby allowing them to withstand much higher compressive stresses than would be indicated by a uniaxial compressive strength test result. The testing machine conform to the requirements of ASTM C 39/C 39M.

![Figure 3.6 A view of measuring the tensile strength of concrete](image)

Tensile strength of specimen is calculated as follow:

\[ T = \frac{2P}{\pi LD} \]

Where:

- **T**: Splitting tensile strength (MPa)
- **P**: Maximum applied load indicated by the testing machine (N)
- **L**: Length (mm)
- **D**: Diameter (mm)
- **Static modulus of elasticity**

This test provides a stress to strain ratio value of concrete applicable within the customary working stress of 40% of ultimate concrete strength (Figure 3.7).

![Figure 3.7 A view of measuring the static modulus of elasticity](image)

- **Petrographic examination by the Damage Rating Index method**

In order to confirm and quantify ASR damage in concrete, a petrographic examination was conducted according to the Damage Rating Index. This method was performed on polished section at 100 weeks.

- **Resonant frequency and ultrasonic pulse velocity**

The procedure and instruments of these tests on concrete are similar to those of mortars in section 3.2.2. According to ASTM C 215-02, the dynamic modulus of elasticity determined from fundamental longitudinal resonant frequency of cylindrical concrete specimens as follow:

\[
E_d = D \times M \times f_L^2 
\]

$E_d$: Dynamic modulus of elasticity

$f_L$: Longitudinal vibration frequencies of concrete (Hz)
M: weight of specimen (kg)

\[ D = 5.093 \left( \frac{L}{d^2} \right) \text{ for a cylinder} \]

Also, the dynamic modulus of elasticity from fundamental transversal resonant frequency is calculated as follow:

\[ E_d = C \times M \times f_t^2 \]

\( E_d \): Dynamic modulus of elasticity

\( f_t \): Transversal vibration frequencies of concrete (Hz)

\( M \): weight of specimen (kg)

\[ C = 1.6067 \left( \frac{L^3 T}{d^4} \right) \text{ for a cylinder.} \]

\( T \): Correction factor that depend to the ratio of \( K/L \) (\( K = d/4 \) for a cylinder) and Poisson coefficient.

- **Nonlinear frequency shift**

Nonlinear acoustic method was explained briefly in section 2.8. It refers to acoustical waves in compressible media with small and finite amplitude. These waves are described by nonlinear equations following from the general governing equations of continuous media for moderate-amplitude displacements when the nonlinear terms are small compared with the linear one. There are several methods for measurement of nonlinear effects. Resonant frequency shift is a sensitive measurement of the amplitude dependence of the resonant frequency that can be used for calculation of the average modulus of elasticity and wave velocity. Measurements are made of upward and downward swept frequency response over the frequency interval in order to present resonance peak shift. Acceleration is frequently measured from which strain is calculated (Ostrovsky et al., 2001) (Figure 3.8).
Figure 3.8 Examples on nonlinear behaviour (from Ostrovsky et al., 2001): a) Resonance acceleration response of polyvinyl chloride (PVC) for several drive levels. b) Resonance acceleration response of Fontainebleau sandstone, for increasing drive. c) Time and frequency domain signals from relatively low amplitude, but nonlinear, drive levels (right), and at large drive levels (left).

Two coefficient of nonlinearity (δ and β) of concrete samples are determined from measurement the frequency shift as follow:

δ: The slope of normalized frequency shift with the strain amplitude

β: The slope of second-harmonic amplitude with the fundamental amplitude square

\[
\beta = 4 \frac{c_0^2}{\omega \pi} \times \frac{A_2}{A_1^2} = 4 \left( \frac{\lambda}{\pi} \right)^2 \times \frac{A_2}{A_1^2}
\]
3.2.4 Test on concrete cores

Preparation of concrete core specimens will be described in section 4.3.3. The tests on concrete core specimens are:

- Expansion measurement and mass variation;
- Petrographic examination by Damage Rating Index method;
- Ultrasonic pulse velocity and resonant frequencies;
- Nonlinear frequency shift.

The procedure of tests on concrete core is similar with those of concrete made in laboratory.
CHAPTER 4
MATERIALS AND THEIR CHARACTERSTICS

4.1 Cement

The cement used in mortar and concrete is an ordinary Portland cement type 10 from the company of Ciment St-Laurent. The composition by weight of the cement is shown in Table 4.1.

<table>
<thead>
<tr>
<th>Element</th>
<th>Amount (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>20.9</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>5.1</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>2.1</td>
</tr>
<tr>
<td>CaO</td>
<td>63.7</td>
</tr>
<tr>
<td>MgO</td>
<td>2.1</td>
</tr>
<tr>
<td>SO₃</td>
<td>3.3</td>
</tr>
<tr>
<td>Na₂Oₑᵥ</td>
<td>0.89</td>
</tr>
<tr>
<td>C₃S</td>
<td>54.0</td>
</tr>
<tr>
<td>C₂S</td>
<td>19.2</td>
</tr>
<tr>
<td>C₃A</td>
<td>9.9</td>
</tr>
<tr>
<td>C₄AF</td>
<td>6.3</td>
</tr>
</tbody>
</table>

4.2 Aggregates

Two different limestone aggregates were used as coarse aggregate in concrete. The result of chemical analyses on these aggregates is shown in Table 4.2.
TABLE 4.2  BULK CHEMICAL COMPOSITIONS OF COARSE AGGREGATES

<table>
<thead>
<tr>
<th>Element</th>
<th>Amount (%)</th>
<th>Spratt limestone</th>
<th>Limeridge limestone</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>10.4</td>
<td>3.0</td>
<td></td>
</tr>
<tr>
<td>CO₂</td>
<td>38.6</td>
<td>43.0</td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>45.7</td>
<td>51.5</td>
<td></td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.33</td>
<td>0.35</td>
<td></td>
</tr>
<tr>
<td>K₂O</td>
<td>0.12</td>
<td>n.d.</td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>1.50</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.08</td>
<td>n.d.</td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.72</td>
<td>1.0</td>
<td></td>
</tr>
</tbody>
</table>

4.2.1  Spratt limestone (reactive limestone)

This aggregate that is used as coarse aggregate in reactive concrete is obtained from Spratt quarry in Ontario province of Canada. It primarily consists of calcite with minor amounts of dolomite and about 10% insoluble residue. The reactive component of the rock is reported to consist of 3% to 4% of microscopy chalcedony and black cherts, which is finely dispersed in the matrix. Density and adsorption of this aggregate are 2.69 g/cm³ and 0.48 %, respectively (Rogers, 1999).

4.2.2  Limeridge limestone (non-reactive limestone)

This aggregate that is used as coarse aggregate in non-reactive concrete is dolomitic limestone slightly metamorphic obtained from Lime-Ridge quarry in Quebec province of Canada. Density and adsorption of this aggregate are 2.70 g/cm³ and 0.44 %, respectively.
4.2.3 Fine aggregate

Non-reactive sand, which used as the fine aggregate in concretes is a limestone aggregate obtained from the company of Aimé Côté in the area of Cantley (Sherbrooke area). Density and adsorption of this aggregate are 2.71 g/cm$^3$ and de 0.81 %, respectively.

4.3 Mixture characteristic, fabrication and storage of the test specimens

4.3.1 Mortar

The mortars consist of one part cement and 2.25 part of sand proportioned by mass and the water/cement ratio of 0.50 were prepared. Aggregates were graded in accordance with the requirements provided in Table 4.3 (CSA A23.2-25A).

TABLE 4.3 GRADING REQUIREMENTS OF AGGREGATE USING IN MORTAR

<table>
<thead>
<tr>
<th>Passing</th>
<th>Retained</th>
<th>Mass (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5mm</td>
<td>2.5mm</td>
<td>10</td>
</tr>
<tr>
<td>2.5mm</td>
<td>1.25mm</td>
<td>25</td>
</tr>
<tr>
<td>1.25mm</td>
<td>630 μm</td>
<td>25</td>
</tr>
<tr>
<td>630 μm</td>
<td>315 μm</td>
<td>25</td>
</tr>
<tr>
<td>315 μm</td>
<td>160 μm</td>
<td>15</td>
</tr>
</tbody>
</table>

Materials were mixed in accordance with the procedure described in ASTM C 305-99. The moulds were filled with mortar. The mortar bars were then removed from their moulds after 24 hours and placed in water at room temperature. Then the temperature of the water rose to 38° C and the mortar bars were stored in this condition for the next 24 hours. The bars were then removed from the water, they were measured for initial length and submersed in a 1 molar NaOH solution (Each litre of 1 molar NaOH consists 40g NaOH + 1L H$_2$O) at 38° C, where they were then stored for 2 years (Figure 4.1).
4.3.2 Concrete

Two concrete mixtures were made. Each mixture consisted of 4 prisms (75 × 75 × 285mm) and 24 cylinders (100 × 200mm). Spratt limestone and Limeridge limestone were used as the coarse aggregates in reactive and non-reactive concrete, respectively. Concretes consisted of coarse aggregate, non-reactive sand as the fine aggregate, cement, sodium hydroxide and water. Coarse aggregate was graded in accordance with the requirement in Table 4.4. The proportions of concrete mixture are showed in Table 4.5. The properties of fresh concretes are shown in Table 4.6. As coarse aggregate used in BSP (Spratt limestone) are angular and Limeridge limestone aggregates present in BLG are smooth, the slump of BSP in much less than that of BLG.

| TABLE 4.4  GRADING REQUIREMENTS OF COARSE |
|-----------------|-----------------|
| Passing | Retained | Mass (%) |
| 20mm | 14mm | 33.3 |
| 14mm | 10mm | 33.3 |
| 10mm | 5mm | 33.3 |

| TABLE 4.5  CONCRETE MIXTURE PROPORTIONS |
|-----------------|-----------------|
| Cement content (kg/m$^3$) | 350 |
| Coarse to fine aggregate ratio | 60/40 |
| Water to cement ratio | 0.50 |
| Alkali content of concrete mixture (kg/m$^3$) | 4.5 |
TABLE 4.6 FRESH CONCRETE PROPERTIES

<table>
<thead>
<tr>
<th></th>
<th>Non-reactive concrete</th>
<th>Reactive concrete</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>22.5</td>
<td>21.5</td>
</tr>
<tr>
<td>Slump (mm)</td>
<td>110</td>
<td>50</td>
</tr>
<tr>
<td>Air content (%)</td>
<td>1.1</td>
<td>1.5</td>
</tr>
</tbody>
</table>

The moulds were filled with concrete and consolidated with a tamping tool to ensure that no large air voids occur and the proper compaction were achieved. Then, they were demoulded 24 ± 4 hours after moulding and initial length of prisms were measured. All the concrete specimens were placed in a humid room for 7 days. The prisms and cylinders specimens were then placed in a vertical position inside the storage container shown in Figure 4.2 and stored in the oven at 38° C. 24 hours before each measurement of expansion, the container of prisms specimens were placed at 23° C.

![Figure 4.2](image.png)

Figure 4.2 A view of concrete prisms stored in the oven at 38° C in humid air (H.R> 90%)

4.3.3 Concrete cores from St. Lawrence Seaway locks

The St. Lawrence Seaway is one of the most comprehensive inland navigation systems in the world (Figure 4.3). This is the system of locks, dams and canals linking a heartland of North America with the Atlantic, Ocean via, the Great Lakes and the St Lawrence River. The St.
Lawrence Seaway proper extends from Montreal to Lake Erie. The Montreal/Lake Ontario section encompasses a series of canals, dredged channels and seven locks over 300km (five Canadian and two American) enabling ships to navigate between the St. Lawrence River and Lake Ontario. Initial construction work began in 1954 and involved corporation from the Canadian and American government. It was officially opened in 1959.

All of the seven locks of the Montreal/Lake Ontario section of the Seaway (St. Lambert, Côte Ste-Catherine, Lower and Upper Beauharnois, Bertrand H. Snell, Dwight D. Eisenhower and Iroquois) are 233.5m long, 24.4m wide and 9.1m deep over the sill.

Figure 4.3  A view of the St Lawrence Seaway
St. Lambert, Côte Ste-Catherine, Lower and Upper Beauharnois are the concrete structures currently affected by alkali-silica reaction (Gaudreault, 2000). Figure 4.4 shows typical symptoms of ASR on a polished core taken from St-Lambert lock. The presence of reactive aggregate, high alkali content of cement and availability of water made the susceptibility of these concrete locks to ASR damage and macrocracking of the surface of concretes. The ASR damage in Beauharnois lock (containing Potsdam Sandstone) and Côte Ste-Catherine lock (containing clayey siliceous limestone) were investigated in this study.

The concrete cores investigated in this study were extracted from concrete locks and brought to the laboratory of the University of Sherbrooke in December 2005. The location of the cores taken from Beauharnois lock and Côte Ste-Catherine lock are shown in Figures 4.5, 4.6, 4.7 and 4.8, respectively. The core diameter was 80 mm and their lengths were about 185 mm for Beauharnois and 170 mm for Côte-St-Catherine. The condition of the cores has been assessed by various mechanical and non-destructive tests (Rivard, 2006). Then, the concrete cores were placed in moisture room at 21°C. In May 2007, 6 specimens of each concrete were selected: 3 specimens for non-destructive tests and 3 specimens for expansion measurements (Table 4.7). In

Figure 4.4  Typical symptoms of ASR observed on a polished core taken from St-Lambert lock: cracks in limestone aggregates and cement paste filled with reaction product, reaction rim around particles
order to evaluate influence of exposure condition on concrete, the cores were collected from different depths where the concrete have been more or less severely affected by ASR.

Figure 4.5 A view of Beauharnois lock

Figure 4.6 Localization of concrete cores extracted from Beauharnois lock
Figure 4.7 A view of Côte Ste-Catherine lock

Figure 4.8 Localization of concrete cores extracted from Côte Ste-Catherine lock
### TABLE 4.7 DEPTHS OF CONCRETE CORES FROM THE SURFACE OF THE LOCKS

<table>
<thead>
<tr>
<th></th>
<th>Expansion test</th>
<th>Non destructive tests</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Beauharnois</strong></td>
<td>8.8m</td>
<td>2.7m</td>
</tr>
<tr>
<td></td>
<td>11.2m</td>
<td>7.7m</td>
</tr>
<tr>
<td></td>
<td>4.5m</td>
<td>13.9m</td>
</tr>
<tr>
<td><strong>Côte Ste Catherine</strong></td>
<td>4-4.2m</td>
<td>1.95-2.05m</td>
</tr>
<tr>
<td></td>
<td>1.7-1.9m</td>
<td>5.2-5.4m</td>
</tr>
<tr>
<td></td>
<td>4.8-5.0m</td>
<td>6.2-6.4m</td>
</tr>
</tbody>
</table>

Stainless gauge studs were installed on the 3 specimens selected for longitudinal expansion measurements. After measuring their initial length, the cores were placed in water at 38° C in an oven and kept in these conditions for the next 24 hours. Then, the cores were removed from water and their lengths were measured again. Finally, all the cores were submersed in a 1 molar NaOH solution at 38° C for accelerating ASR.
CHAPTER 5

CHARACTERIZATION OF AGGREGGATES

Limestones are the sedimentary rocks mainly composed of the mineral calcite (calcium carbonate, CaCO₃). Limestones often contain variable amounts of silica in the form of chert or flint, as well as varying amounts of clay, silt and sand as disseminations, nodules, or layers within the rock.

5.1 Chemical composition of aggregates

The chemical composition of the aggregates was determined by X-Ray fluorescence to measure the percentage of silica and various elements in the aggregates. Chemical composition of two aggregates was shown in section 4.2. The chemical composition of these aggregate shows that they are mainly made up of limestone (CaO + CO₂ = 94.5% and 84.3% for Limeridge limestone and Spratt limestone, respectively). Moreover, SiO₂ is in larger amounts in Spratt. SiO₂ constituted 10.4% and 3% of Spratt and Limeridge, respectively.

5.2 Ray diffraction

The analysis by X-Ray diffraction made it possible to determine the crystalline phases present in the aggregates and their degree of crystallinity. The spectrum of X-Ray diffraction of each aggregate is the subject of Figures 5.1 and 5.2. The essential minerals, which are observed in the aggregates, are:

- Quartz (SiO₂)
- Calcite (CaCO₃)
- Albite (Na(Si, Al)O₈)
- Anorthite ((Ca, Na) (Si, Al)₄O₈)
The crystalline phase present in Limeridge is quartz made up of networks of tetrahedron well crystallized comprising little defect. In Spratt limestone, the width of line is more important than that of Limeridge limestone and its intensity is much less. It consists of badly crystallized networks comprising defects. The position of silica peak in both aggregate is the same ($2\theta = 26.64$), which corresponds to the same type of silica ($\text{SiO}_2$).

5.3 Microstructural analysis by scanning electron microscopy (SEM)

The samples prepared by crushing rocks were observed by means of scanning electron microscopy Figure 5.3 shows scanning electron micrograph of Limeridge rock specimen (SEM). There were a few pores and their dimension does not exceed 10 µm. The fractured surface of this aggregate is very dense and consists of calcium carbonate.

The microstructure of Spratt (Figure 5.4a) is different. It was observed more porous and less dense than Limeridge specimen and many pores of 1-10µm were scattered on the surface of aggregate. Figure 5.4b shows the less porosity of fractured surface of Spratt.

The elemental maps obtained by microanalysis probe in the SEM (Figures 5.5 and 5.6) show the distribution of different elements that were mainly made up of calcium and different types of silica closely overlapping in the carbonated matrix. In Limeridge limestone, only a small part of surface was covered by silica and its distribution was not homogeneous. In Spratt limestone, the presence of silica was more remarkable and it was distributed on entire surface of aggregate.

Above results allows better understanding of the relation between reactivity of aggregates and their petrographic characterization. Spratt limestone is a reactive aggregate due to its higher silica content, which was poorly crystallized, but homogeneously distributed on the aggregate texture and Limeridge limestone is a non-reactive aggregate due to the lower silica content, which was well crystallized and heterogeneously were found on the aggregate texture.
Figure 5.1  Crystalline phases of Liméridge limestone with SiO$_2$ phase

Figure 5.2  Crystalline phases of Spratt limestone with SiO$_2$ phase
Figure 5.3  SEM micrograph of Limeridge limestone  
(A: surface and B: fractured surface of Limeridge limestone)

Figure 5.4  SEM micrograph of Spratt limestone  
(A: surface and B: fractured surface of Spratt limestone)
Figure 5.5  Elemental map of Limeridge limestone by microanalysis probe in the SEM

Figure 5.6  Elemental map of Spratt limestone by microanalysis probe in the SEM
CHAPTER 6

TESTS ON MORTARS

6.1 Expansion measurement and mass variations

6.1.1 Expansion measurement

Figures 6.1 and 6.2 show the expansion of mortar bars containing Limeridge limestone (MLG) and Spratt limestone aggregate (MSP). In MLG, during the first 17 weeks, reaction rate was too slow and expansion was lower than 0.046%. Then, it was raised gradually until 0.41% at 92 weeks. Since then, deformation was too little to be considered. As shown in Figure 6.1, the expansion level of this mortar is higher than limit mentioned in CSA A23.2-25A (section 2.7). According to CSA A23.2-25A, Limeridge limestone maintains in 1 molar NaOH at 80°C is a non-reactive aggregate with the maximum expansion level of less than 0.1% at 50 days. But in this study, MLG obtained this expansion level at 26 weeks and also the duration of reaction was too long that could be due to lower temperature. Thus, regarding the low reaction rate, increasing immersion period of expansion measurement could be a solution to better recognition the reactivity of this aggregate.

In MSP (Figure 6.2), the expansion was less than 0.04% during the first 4 weeks and then it increased sharply to 0.93% at 61 week. The highest average of the expansion rate in MSP reached 0.07% per month between 12 and 43 weeks. Then, it slowed down and did not exceed 1.06% at 93 weeks. From 100 weeks, mortar bars have been curving and it was not possible to continue the expansion measurements (Figure 6.3).

According to above results, Spratt limestone shows significant reactivity while Limeridge limestone exhibits a lower reactivity. In MSP, reaction started earlier and the expansion rate was higher than that of MLG. The final expansion of MSP was about 2.6 times the MLG.
Figure 6.1  Expansion of mortar bars containing Limeridge limestone

Figure 6.2  Expansion of mortars bar containing Spratt limestone
6.1.2 Masse variations

At the same time of expansion measurement, the mass of each mortar bar was measured. Figures 6.4 and 6.5 show the mass variation of MLG and MSP during the reaction development, respectively. The mass variation increased significantly with the expansion and when the expansion stopped, this value decreased slightly that could be the sign of the end of water adsorption by mortar.

In MLG, the mass of mortar increased by about 1% during the first 48 hours, and then increased slightly to 2.44% at 79 weeks for an expansion level of 0.372% and then it slightly decreased. In MSP, during the first week, this value was 0.8% and then rose until 3.83% at 93 weeks for the maximum expansion level of 1.06%.

In both mortars, mass variation increased with the expansion and during the first weeks, increasing the mass was more significant due to adsorbing the solution. However, in MSP, the increase of the mass of mortar was higher at higher expansion levels. Figure 6.6 illustrates the relation between mass variation and expansion of both mortars specimens followed about the same pattern.
Figure 6.4  Mass variations of mortar bar containing Limeridge limestone

Figure 6.5  Mass variations of mortar bar containing Spratt limestone
6.2 Compressive strength of mortars

Compressive strength of mortars was measured at 28 and 91 days and the results are shown in Table 6.1 and Figure 6.7.

In MLG, compressive strength of mortar soaked in 1 molar NaOH was less than those soaked in saturated lime according to what was expected. The reductions of strength were 3.5% and 14.5% at 28 and 91 days, respectively. In MSP, the condition of storage did not influence of compressive strength of mortar at 28 days. However, at 91 days, compressive strength of specimens soaked in 1 molar NaOH was 13.2% less than that of specimens soaked in saturated lime, which is about the reduction observed with MLG.

Moreover, at 91 days, compressive strength of MSP soaked in NaOH was 7% and 11% higher than that of MLG stored in saturated lime and NaOH, respectively.
TABLE 6.1  COMPRESSION STRENGTH OF MORTARS  
(CV: COEFFICIENT OF VARIATION)

<table>
<thead>
<tr>
<th></th>
<th>28 days (MPa)</th>
<th>91 days (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MLG</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mortar stored in saturated lime</td>
<td>62.1 CV = 1.87</td>
<td>69.4 CV = 1.07</td>
</tr>
<tr>
<td></td>
<td>57.3 ( f_c' = 57 )</td>
<td>71.0 ( f_c' = 69 )</td>
</tr>
<tr>
<td></td>
<td>57.5</td>
<td>68.0</td>
</tr>
<tr>
<td>Mortar stored in 1 normal NaOH</td>
<td>55.2 CV = 1.94</td>
<td>58.7 CV = 0.55</td>
</tr>
<tr>
<td></td>
<td>57.1 ( f_c' = 55 )</td>
<td>57.5 ( f_c' = 59 )</td>
</tr>
<tr>
<td></td>
<td>52.0</td>
<td>59.1</td>
</tr>
<tr>
<td>MSP</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mortar stored in saturated lime</td>
<td>60.7 CV = 0.85</td>
<td>71.9 CV = 2.51</td>
</tr>
<tr>
<td></td>
<td>59.4 ( f_c' = 60 )</td>
<td>75.6 ( f_c' = 76 )</td>
</tr>
<tr>
<td></td>
<td>59.2</td>
<td>76.7</td>
</tr>
<tr>
<td>Mortar stored in 1 normal NaOH</td>
<td>60.9 CV = 1.07</td>
<td>66.0 CV = 0.54</td>
</tr>
<tr>
<td></td>
<td>63.7 ( f_c' = 61 )</td>
<td>65.6 ( f_c' = 66 )</td>
</tr>
<tr>
<td></td>
<td>61.6</td>
<td>66.7</td>
</tr>
</tbody>
</table>

Figure 6.7  Compressive strength of cubic mortars
Relation between expansion of mortar bars and compressive strength of cubic mortars

As shown in Figure 6.8, the expansion of MLG was much less than the expansion limit mentioned in CSA A23.2-25A (0.1%) during the first 3 months. The compressive strength of this mortar was increased by 7.3% between 28 and 91 days.

In MSP, in spite of relatively high expansion level (0.2%) at 91 days, this value increased by 7.6%. These results may show that this expansion level in this mortar is too low to influence on compressive strength development of mortar.

The reason that why the compressive strength of MLG at 91 days is less than that of MSP can be related to the variation of microstructure and weakening the aggregate-cement paste interphases in MLG and production of ASR gel on the surface of MSP. The microstructure of mortars will be explained in section 6.4.

![Graph showing relation between compressive strength and expansion of mortars](image)

**Figure 6.8** Relation between the results of compressive strength and expansion measurement of mortars stored in 1 normal NaOH at 28 and 91 days
6.3 Non-destructive tests (NDT)

As mentioned in section 3.2.2, NDT tests have been performed according to the methods described in ASTM C 215-02 and ASTM C 597-97. However, these standards are designed for concrete specimens. On the other hand according to ASTM C 215-02, specimens used in resonant frequency test have to be made in accordance with ASTM C31, ASTM C192 or ASTM C 42. Also, the lateral dimension of specimens used in pulse velocity test has to be at least:

\[ d \geq \lambda \left( \lambda = \frac{\text{Velocity}}{\text{frequency}} \right) \]

The mortar specimens that were used for NDT had the same lateral dimensions of mortar bar used for expansion measurement (25×25mm). The length of MLG and MSP specimens were around 0.30 m and 0.24 m, respectively. Therefore, in a mortar bar with the minimum ultrasonic pulse velocity of 3500m/s, and for a pulse frequency of 54 kHz, the minimum lateral dimension of specimens must be at least 65mm that means the lateral dimensions of mortar bars used in this study were too low and can influence the results of ultrasonic pulse velocity test.

There are a few investigations on effect of specimen geometry on resonant frequency measurement tests. According to ASTM C 215 resonant frequency test, best results are obtained when the ratio of length to lateral dimension of specimens is between 3 and 5 (or at least 2). Lafhaj (Lafhaj et al., 2005) studied relation between porosity and permeability of mortar with ultrasonic parameters. The mortar specimens were cylinders of diameter 37 and 10mm height. The height of specimens were chosen too small to allow better transmission of ultrasonic wave. Tandon (Tandon et al., 2006) reported in specimens with L/D of 2, dynamic modulus of elasticity is similar (with 10% variation) independent of diameter to those of the specimens with L/D of 1.5 and 1.3. This ratio in our mortar bars is too high (between 10 and 12).

It must be pointed out that mortar and concrete do not have the same structure. The density of mortar used in this study is slightly less than of those of concretes. Also, the small size of sands
in mortar accelerates the reaction rate and increases damage even in the inner part of mortars (It will be discussed in section 6.4).

Another significant point that will be illustrated in the results of non destructive tests is that since the reaction rate in mortar is influenced by the type of aggregate, reduction in pulse velocity and resonant frequency does not occur at the same rate or in proportion to expansion.

These parameters would influence on the results of resonant frequency and pulse velocity. Thus, for the same test method on mortar bars, it is primarily important to find a correction factor for each value. The dimensions of mortar bars used for this purpose are 0.025x0.025x0.075m. The correction factor of each mortar was calculated from the ratio of the dynamic modulus of elasticity of mortar bar of 0.075m heights to that of 0.30m.

6.3.1 Pulse velocity

This test is a measurement of transmission time of stress waves. The calculation of the ultrasonic pulse velocity (UPV) allows estimating the presence of cracks and voids and uniformity.

In MLG, UPV increased gradually until 8.4% at 6 months, when the expansion reached 0.103% (expansion limit). Then, it decreased by 5% until one year for an expansion level of 0.28% and then it varied between 3.1 and 8% (Figure 6.9).

In MSP, the initial pulse velocity was 2.9% more than that of MLG (Figure 6.10) and also there is a significant difference between the pulse velocities of mortar at different times of reaction (Figure 6.11). During the first month, it rose by 2.3% and then it reduced by 5% during the next two months, when the expansion reached 0.2%. Since then, this value varied between -10.4% and 8.5%. The highest value was reached at 20 months with the highest expansion level.

It was confirmed by other authors that ASR can be often a surface phenomenon in concretes and cannot always extent completely to the core. In fact, pulse velocity measurement in concrete structure showed that a thin shell of deteriorated concrete surrounding an almost unaffected interior causes the reduction of this value. Therefore in a thin mortar bar with the small lateral
dimension microcracks formation and deterioration can be found in the entire volume of mortars. The internal deterioration of mortar bars will be shown in micrographs of following section.

Figure 6.9 Variation of pulse velocity with expansion

Figure 6.10 Pulse velocity in mortars
As shown in Figure 6.11, there is a significant variation in pulse velocity of MSP during the reaction development. This variation could be related to the volume of produced gel and volume of pore and microcracks in mortar. In fact, at 38°C, the reaction rate is slower than the reaction rate at 80°C and mortar has more time to adsorb ions of solution and produces ASR gel. So the volume of produced gel at lower temperature may be higher. On the other hand, when the duration of reaction is longer, produced gels have enough time to fill pores and microcracks of mortars. Therefore, the tensile stress caused by producing ASR gel would be reduced which leads to reduction in microcracking and raising pulse velocity. The results of petrographic analysis by SEM (Section 6.4) confirmed that when the pulse velocity of mortars affected by ASR increases, it may be the sign of high volume of produced gel that filled voids and some parts of microcracks. The above information can describe why the pulse velocity of MSP after 70 weeks was more than that of MLG. Furthermore, in section 6.4.2, it will be demonstrated that the generation of microcracks on entire surface of mortar minimized this value at 52 weeks.

These data also emphasize that there is no unique relationship between expansion and loss or gain in pulse velocity in a thin mortar bar, when the expansion is more than 0.25%.
6.3.2 Dynamic modulus of elasticity

Figure 6.12 shows the dynamic modulus of elasticity ($E_d$) in mortars calculated from longitudinal resonant frequency. In MLG, this value increased by 14.5% during the first 28 days and then there was a reduction of 6.9% at 3 months (Figure 6.13), for the low expansion level of 0.04% (Figure 6.14). During the next three months, this value increased 4.7%, for the expansion level of less than 0.1%. The maximum reduction occurred between 6 and 9 months, when the expansion rose from 0.1 to 0.2%. Since then, it gently increased with a few variations. In this mortar, the maximum dynamic modulus of elasticity was observed (27.7GPa) at 14 and 28 days with the expansion levels of 0.023% and 0.024%, respectively and the minimum value was 21.6GPa for the expansion level of 0.36%.

In MSP, the initial dynamic modulus of elasticity was 14% less than that of MLG (Figure 6.12). This value increased by 37% during the first three months, when the expansion reached 0.26%. During the next 6 months, there was a significant decrease of 33%, when the expansion increased from 0.26% to 0.71%. Since then it raised gently with a few variation. At 100 weeks, this value was 23.3GPa for the highest expansion level of 0.105%.

The comparison between modulus of elasticity of two mortars shows the $E_d$ of MLG was higher than that of MSP during the reaction development (Figure 6.12) and it was more sensitive to damage caused by ASR. In spite of lower reactivity of MLG, the reduction of this mortar dynamic modulus of elasticity started earlier and for lower expansion level. In MLG, this value was quite lower (with 8.5% reduction) for an expansion level of 0.36%, whereas in MSP the highest reduction was 8.7% for an expansion level of 1.04%.

As shows in above result, there were significant variations in dynamic modulus of elasticity of both mortars at different ages, which did not related to the expansion. In section 6.4, it will be shown that the small lateral dimension of mortar bars allowed the reaction development on entire volume of mortar bars and generate microcracks and high volume of ASR gel during the reaction progression. Another important point is sensitivity of NDT methods to the variation in the microstructure of mortars. In section 6.4, it will be shown that in MSP, pulse velocity decreased...
since the microcracks were generated on mortar, whereas, dynamic modulus of elasticity decreased when the width of microcracks reached around 10 μm.

Figure 6.12 Dynamic modulus of elasticity in mortars

Figure 6.13 Variation of dynamic modulus of elasticity in mortars
6.3.4 Conclusion on the results of non-destructive tests on mortars

- There is not unique relationship between the expansion and the variation of the results of pulse velocity and dynamic modulus calculated from longitudinal resonant frequency of ASR-affected mortars.
- Variation of pulse velocity and dynamic modulus did not follow the similar pattern of behaviour in two mortars. Thus it can be suggested that the mortars were not deteriorated in the same way (it will be explained in the following section).
- Dynamic modulus of elasticity was largely varied with increasing the expansion. It was more sensitive to damages due to ASR.

6.4 Microscopic properties of mortars

Studies on the fractured surfaces of mortars were performed by using Scanning electron microscope at a magnification varying from 200 to 10000X. Observations were performed at each stop by moving around and following paste/aggregate bond zones and cracks. It would be noticed that the small size of fracture surface of mortar could not give the good representation of bulk microstructure. Also the preparation of sample crushing and drying with various kinds of
vacuum may introduce microcracks and alter microstructures. This topic has been discussed by some authors (Diamond, 1994, Hornain, 1994 and Olivier, 1985). Thus, the results obtained from SEM test should be evaluated bearing such factor in mind.

6.4.1 MLG observation

At 24 hours, the surface of aggregate was sound with a few microcracks (width \( \leq 1 \mu m \)) on the surface of cement paste and around aggregates that would be caused by drying shrinkage of mortar (Figure 6.15).

![Figure 6.15 SEM petrology of MLG at 24 hours](image)

At 14 days, with the low expansion level of 0.022\%, there were some microcracks (width \( \geq 1 \mu m \)) on the cement paste and around the aggregates. Also, there were some microcracks filling by gel on the surface of aggregate. ASR gel formed around the aggregate and in microcracks (Figure 6.16).

Expansion level slightly rose between 14 and 28 days (0.024\%). The number of microcracks on the surface of aggregates (width \( \geq 1 \mu m \)) and cement paste (width \( \leq 1 \mu m \)) increased and the gels are more visible around the aggregates (Figure 6.17).
After 3 months, the expansion of mortar reached 0.04%. The microcracks with the dimension of 1-10 μm are on the surface of aggregates and cement paste. The most important point that was observed in Figure 6.18 is the formation of microcracks with the width of more than 2 μm in aggregate-cement paste interphases. In fact, the presence of non-reactive aggregate in mortar makes the reaction only on the external surface of aggregate and generates the microcracks in this zone.

As explained in literature, the mechanical properties of concrete are mainly controlled by presence of transition zone. This zone has lower stiffness than the bulk paste. Thus, when the volume of transition zone is not negligible, this may significantly affect the overall modulus of elasticity of mortar and concrete (Mehta and Monterio, 1993 and Cohen and Goldman, 1994).
Another study by Ramesh (Ramesh et al. 1996) has shown that shear modulus of transition zone influence directly on the mortar modulus of elasticity. This relation is linear and the mortar modulus of elasticity decreased from 31.5 to 16.7GPa, which corresponds to the reduction of the volume and increasing the elastic modulus of transition zone.

According to the results of compressive strength and dynamic modulus of elasticity of MLG at 3 months, the formation of microcracks in aggregate-cement paste interphases occurred with a 6.9% reduction regarding the mortar dynamic modulus of elasticity and makes the compressive strength of this mortar 9.8% less than that of MSP.

These results show when the width of cracks around aggregate is 2\mu m, this might be the critical crack width that makes the remarkable decrease in longitudinal resonant frequency and dynamic modulus of elasticity calculated from this value.

![Figure 6.18 SEM petrography of MLG at 3 months](image)

After 9 months, expansion of mortar bars was 0.203%. The surface of aggregates is more deteriorated with microcracks and gel and the volume of gel is higher (Figure 6.19). The cracks are wider (1 \leq w \leq 10 \mu m) on the entire surface of mortar. Although the microcracks around the aggregate are particularly filled by gel, but the high expansion level that raised the numbers and width of microcracks even on the surface of aggregate, minimized dynamic modulus of elasticity and reduced pulse velocity.
After 19 months, expansion of mortar bars was 0.372%. The surface of aggregate and cement paste was covered by map cracks (in some cases, $w \geq 10 \mu m$) (Figure 6.20). The microcracks that had formed around aggregates were filled by high volume of gel (more than $20 \mu m$).

6.4.2 MSP observation

At 24 hours, expansion was very low (0.012%). Microstructure of this mortar at 24 hours was similar to that of MLG (Figure 6.21).
After 14 days, with the low expansion level of 0.023%, the reaction product (gel) was visible around the aggregate and on the cement paste and the surface of aggregate (Figure 6.22). Also the surface of aggregate was particularly deteriorated by the reaction and covered by gel. The structure of gel formed around aggregate was spongy.

After 28 days, although expansion was 0.038%, the gel is more visible around the aggregates and on the surface of cement paste. Microcracks (w<1 μm) cover all the surface of cement paste (Figure 6.23).
After 3 months, the expansion of mortar reached 0.26%. The microcracks (w<1 μm) covered the surface of cement paste (Figure 6.24). In some cases the width of microcracks seemed more than 1 μm. The gel is more concentrated around the microcracks. This variation in the texture of mortar was followed by the significant decrease in ultrasonic pulse velocity.

After 6 months, the expansion was 0.52%. This expansion level produces the map cracks with the width near 10 micrometer on all the surface of cement paste (Figure 6.25). The surfaces of aggregates were too porous and the volume of produced gel is too high and it filled the voids and some parts of microcracks that caused a 8.6% increase in ultrasonic pulse velocity. It seems that the formation of wide microcracks of 10 micrometer cause the significant reduction in the mortar dynamic modulus of elasticity.
After 9 months, expansion of mortar was 0.71% that shows the high degree of reaction with the formation of holes and voids within the mortar and high volume of gel (Figure 6.26). The cracks with the width of more than 10 \( \mu \)m are found on cement paste. These variations in mortar structure caused 25% reduction in dynamic modulus of elasticity.

Between 9 and 12 months, expansion increased gently to 0.86%. Finer aggregates were completely deteriorated and they could not be seen under microscope (Figure 6.27). The surface of large aggregate was more deteriorated than the cement paste. Aggregates exhibited numerous microcracks, which contributed to minimize the pulse velocity of mortar (-10.4%). However, the width of cracks was less than those observed at 9 months (w<10 \( \mu \)m), which had led to an
increase of 5.2% of the dynamic modulus of elasticity. After 15 months expansion was 0.88%.
All the surface of mortar was covered by microcracks ($1 \leq w \leq 10 \mu m$) (Figure 6.28).

At 20 months, expansion reached 1.04% that was the highest expansion level. The surface of mortar was more deteriorated and the microcracks were wider (in some cases more than $10 \mu m$) (Figure 6.29).

Figure 6.27  SEM petrography of MSP at 12 months

Figure 6.28  SEM petrography of MSP at 15 months
6.4.3 Conclusion on the results of SEM

- SEM observations were in agreement with the results of expansion measurements.
- The amounts of microcracks in mortar were clearly increased with expansion. SEM images show that the cracks associated with ASR varied from 1 to 10 μm and in some cases more than 10 μm after 15 months in both mortars.
- In MLG, the most important aggregate degradation was observed on the surface of aggregates. In MSP, the entire volume of aggregate was deteriorated.
- In MLG, the microcracks were widest in cement-aggregate interphases and followed the boundaries of larger aggregate particles. These cracks were the critical cracks that made the significant reduction in the results of resonant frequency measurement.
- In MSP, the microcracks were observed on the surface of aggregate and cement paste, and there were not microcracks in aggregate-cement paste interphases. In this mortar, the longitudinal resonant frequency was increased with increasing the width of microcracks. In spite of ongoing reaction in MSP, the dynamic modulus of elasticity decreased when the width of microcracks reached near 10 μm, whereas the reduction in UPV occurred when the microcracks are wider than 1 μm.
- The reason for self-healing in physical properties of mortars might be formation of ASR gel and filing some parts of voids and microcracks. In other words, there could be the
competition between volume of produced gel and the volume of microcracks formed by the reaction that made the big difference in the results of non-destructive tests.

- Although the structure of a reactive mortar and a concrete containing reactive fine aggregate is not the same due to the presence of coarse aggregate in concrete, but the results of NDT and petrographic analysis by SEM in mortars can give an idea about the microstructure and physical properties of concrete containing reactive fine aggregate damaged by ASR.

6.5 Chemical tests

At different stages of reaction development (24 hours, 14 and 28 days, 3, 6, 9, 15 and 20 months), one bar from each mortar was removed from the solution, dried and then was ground to obtain the finesse of 80 μm. When the reaction stopped in both mortar, the chemical tests on ground mortars started. The specimen of MSP at 24 hours was the first ground mortar subjected to chemical test according to the procedure explained in section 3.2.2. The test was performed on three specimens in order to determine the average of the number of reactive silica ($Q_3$) and dissolved silica ($Q_4$) in mortar. Table 6.2 presents the results of chemical tests on three MSP specimens at 24 hours.

**TABLE 6.2 THE RESULTS OF CHEMICAL TEST ON MSP AT 24 HOURS**

<table>
<thead>
<tr>
<th>Specimen Identification</th>
<th>a</th>
<th>b</th>
<th>c</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specimen Mass (mg)</td>
<td>27.1</td>
<td>54.96</td>
<td>19.36</td>
</tr>
<tr>
<td>Mass variation (%)</td>
<td>-8.9</td>
<td>-1.2</td>
<td>-13.8</td>
</tr>
<tr>
<td>Mass of water (mg)</td>
<td>2.41</td>
<td>0.66</td>
<td>2.7</td>
</tr>
<tr>
<td>$nQ_3(t) = 2 \left( \frac{M_{H_2O}}{M_{H_2O}^{mol}} \right)$</td>
<td>$0.27 \times 10^{-3}$</td>
<td>$0.07 \times 10^{-3}$</td>
<td>$0.3 \times 10^{-3}$</td>
</tr>
<tr>
<td>$nQ_4(t) = \left( \frac{M_{SO_2}}{M_{SO_2}^{mol}} \right) - nQ_3(t)$</td>
<td>$0.18 \times 10^{-3}$</td>
<td>$0.85 \times 10^{-3}$</td>
<td>$0.02 \times 10^{-3}$</td>
</tr>
<tr>
<td>$Q_3(t)$ (%)</td>
<td>60</td>
<td>8</td>
<td>94</td>
</tr>
<tr>
<td>$Q_4(t)$ (%)</td>
<td>40</td>
<td>92</td>
<td>6</td>
</tr>
</tbody>
</table>
Although the samples were selected from a dry and homogenate ground mortar, the results showed the significant difference between the mass variation of samples and the proportions of $Q_3$ and $Q_4$ calculated from this value. As explained in section 3.2.2, mass variation is directly related to the transformation of $Q_3$ to $Q_4$. This method has been already performed on a siliceous aggregate (Silex) and two types of siliceous limestone aggregate with prosperity as a quantitative method.

Therefore, it can be concluded that there is another source of water in samples that made the diffraction in the results. Limestone aggregates, which were used in this study, contain some impurities such as clay in their structure. Clay is a material which shows plasticity through a variable range of water content and can be hardened when dried. They impart plasticity and variable amounts of water trapped in their structure. This water could be evaporated during the thermogravimetry test and made it impossible to measure only water released by transformation of $Q_3$ to $Q_4$. So, this method cannot use to determine the reaction degree in mortars containing limestone aggregates.
CHAPTER 7

TESTS ON CONCRETES

In this section the results of tests on laboratory concrete mixtures are presented. All properties measured in this study were taken on at least three specimens and the data presented here are the average of measurements conducted on all three.

7.1 Expansion measurement and mass variation

7.1.1 Expansion measurement

The expansion of the two mixtures was measured regularly and their trends are shown in Figures 7.1 and 7.2. The average expansion on each figure is calculated from four concrete prisms. The average of expansion of BLG and BSP is presented in Figure 7.3.

The non-reactive concrete (BLG) did not show significant expansion. The first sign of reaction was observed after 34 weeks; the expansion reached 0.009% at 45 week. The average of expansion during these weeks was 0.0009% per week. Since then, the rate varied between 0.0003% and 0.009%.

In reactive concrete (BSP), the expansion curve consisted of 3 stages: The first 13 weeks was the beginning of reaction and the expansion was too low to be observed (less than 0.005%). Then it increased to 0.066% at 34 week (0.003% per week). Between 34 and 52 week, the expansion rate decreased (0.0005% per week). After 52 week, it raised sharply until 0.122% at 72 week (0.0028% per week). Since then, the third stage started and the reaction rate slowed down. The maximum expansion level was 0.13% at 91 weeks, which is around three times more than expansion limit mentioned in CSA A23.2-14A.
Figure 7.1  Expansion of concrete prisms containing Limeridge limestone

Figure 7.2  Expansion of concrete prisms containing Spratt limestone
Figure 7.3 Average expansion of reactive and non-reactive concrete mixtures

Saint-Pierre (2006) investigated the reactivity of Spratt limestone by measurement the expansion of a laboratory concrete stored in 1molar NaOH solution at 38°C. Although the composition of concrete used in his study was almost the same with BSP composition, but the final expansion level of concrete specimens stored in NaOH solution was about 0.2%. These results show the final expansion of concrete stored in NaOH solution is much more than that of concrete with high alkali content and stored in the oven at 38°C in humid air (H.R> 90%)

7.1.2 Mass variations measurement

Mass variation of concrete prisms is illustrated in Figures 7.4 and 7.5. Figure 7.6 shows the average mass variation of BLG and BSP. In BLG, the mass variation gently increased to 0.31% at 58 weeks. Since then, there was a little variation in this value. In BSP, the mass of specimens did not increase during the first stage of expansion evolution. Then it rose to 0.42% at 83 weeks, followed by a few decrease during the next months.
Figure 7.4  Mass variations of concrete prisms containing Limeridge limestone

Figure 7.5  Mass variations of concrete prisms containing Spratt limestone
7.1.3 Relation between expansion and mass variations of concretes

The relation between expansion and mass variation of concretes is illustrated in Figure 7.7. In BLG, during the first 21 weeks, the loss of mass appeared with the negative variation of expansion. Then specimen mass raised with a very low rate of expansion. The ratio of expansion to gaining mass was around 0.03%. In BSP, expansion of concrete prisms occurred with increasing the specimens mass. The ratio of expansion to mass gain of these specimens was around 0.3%. In the other word, this ratio in BSP was 10 times the BLG.
7.2 Mechanical tests

7.2.1 Compressive strength

Compressive strength of concretes is presented in Table 7.1 and Figure 7.8. According to these results, compressive strength of BLG increased to 5.6, 35 and 49% after 3, 12 and 23 months, respectively. In BSP, this value increased to 9, 22 and 36% after 3, 12 and 23 month, respectively. At 28 days, compressive strength of BLG was 7.7% more than that of BSP that could be related to the higher density and lower air content of BLG (the initial density of BLG was 2.3% higher and the air content was 26% lower than BSP). At 3 months, this value of BLG was only 4.4% more than that of BSP.

As it explained in section 7.1, during the first 8 months, no sign of expansion were observed in BLG. Also, the expansion of BSP at 3 months was 0.005% that was too low to make microcracks on the surface of concrete. But this level of expansion can fill some part of voids and make the rate of strength development of BSP more than that of BLG (Figure 7.9).
### TABLE 7.1 COMPRESSIVE STRENGTH IN CONCRETES
(CV: COEFFICIENT OF VARIATION)

<table>
<thead>
<tr>
<th></th>
<th>28 days (MPa)</th>
<th>3 months (MPa)</th>
<th>12 months (MPa)</th>
<th>23 months (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BLG</td>
<td>39.7 CV=1.79</td>
<td>42.3 CV=1.5</td>
<td>55.0 CV=2.3</td>
<td>58.3 $f'_c=59.1$</td>
</tr>
<tr>
<td></td>
<td>42.9 $f'_c=39.8$</td>
<td>42.7 $f'_c=42.5$</td>
<td>52.7 $f'_c=54.9$</td>
<td>59.8</td>
</tr>
<tr>
<td></td>
<td>39.9</td>
<td>39.9</td>
<td>57.2</td>
<td></td>
</tr>
<tr>
<td>BSP</td>
<td>37.4 CV=1.79</td>
<td>41 CV=1.2</td>
<td>46.1 CV=2.4</td>
<td>50.9 $f'_c=50.6$</td>
</tr>
<tr>
<td></td>
<td>37.1 $f'_c=37.3$</td>
<td>40.3 $f'_c=40.7$</td>
<td>39.2 $f'_c=46.4$</td>
<td>50.2</td>
</tr>
<tr>
<td></td>
<td>39.1</td>
<td>42.6</td>
<td>46.6</td>
<td></td>
</tr>
</tbody>
</table>

Figure 7.8  Compressive strength developments in concretes

At 12 and 23 months, the compressive strength of BSP was 15.6% and 14.4% less than that of BLG. Although, the value of expansion in BSP was 0.076% and 0.13% at 12 and 23 months (Figure 7.3) and these expansion levels are significantly more than expansion limit mentioned in CSA A23.2-14A, the rate of compressive strength development of damaged concrete did not reduced significantly by the reaction (Figure 7.9).
Figure 7.9  Variation of compressive strength development in concretes

It should be considered that when a concrete sample damaged by ASR is subjected to compressive strength test, the microcracks which formed on the surface of sample due to ASR will be closed during the test. In other words, compressive strength of concrete cannot be influenced significantly by ASR development.

7.2.2 Tensile strength

The concrete tensile strength is given in Table 7.2 and in Figure 7.10. In BLG, this value increased to 12.5% at 3 months. At 12 months, the failure was not diametric and it occurred on the corner of specimens. The results at 12 months could not be reliable to use in this section. In BSP, this value increased to 8.3% and 22.2% at 3 and 12 months, respectively. At 28 days and 3 months, tensile strength of BLG was 16.6% and 23% higher than that of BSP.

As it was described in section 2.9.2, using tensile-to-compressive strength ratio could be a good indication of internal damage due to AAR (Nixon and Bollinghaus, 1985). This ratio in a sound concrete varies from 0.07 to 0.11, but in a concrete damaged by ASR or freezing and thawing cycles, this ratio is less than 0.06. The tensile-to-compressive strength ratio in BSP at 28 day, 3
and 12 month was 0.097, 0.094 and 0.095, respectively. Thus this expansion level (0.076% at 12 months) was too low to influence the tensile strength of concrete.

TABLE 7.2 TENSILE STRENGTH IN CONCRETES (CV: COEFFICIENT OF VARIATION)

<table>
<thead>
<tr>
<th></th>
<th>28 days (MPa)</th>
<th>91 days (MPa)</th>
<th>365 days (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BLG</td>
<td>3.6 CV= 0.3</td>
<td>3.9 CV= 0.5</td>
<td>4.23 f&lt;sub&gt;r&lt;/sub&gt; = 4</td>
</tr>
<tr>
<td></td>
<td>4.2</td>
<td>4.8</td>
<td>3.78</td>
</tr>
<tr>
<td></td>
<td>4.2</td>
<td>4.8</td>
<td>3.78</td>
</tr>
<tr>
<td>BSP</td>
<td>3.6 CV= 0.15</td>
<td>4.40 CV= 0.3</td>
<td>4.52 f&lt;sub&gt;r&lt;/sub&gt; = 4.4</td>
</tr>
<tr>
<td></td>
<td>3.5 f&lt;sub&gt;r&lt;/sub&gt; = 3.6</td>
<td>3.8 f&lt;sub&gt;r&lt;/sub&gt; = 3.9</td>
<td>4.23</td>
</tr>
<tr>
<td></td>
<td>3.8</td>
<td>3.9</td>
<td>4.23</td>
</tr>
</tbody>
</table>

Figure 7.10 Tensile strength development
7.2.3 Static modulus of elasticity

In Table 7.3 and Figure 7.11, modulus of elasticity in concretes affected by ASR is presented. At 28 days and 3 months, there is a few difference between the modulus of elasticity of two concretes (2.8% and 4.4%). But at 12 and 23 months, this value in BLG was 31.5% and 59.3% more than that of BSP.

### TABLE 7.3 STATIC MODULUS OF ELASTICITY IN CONCRETES (CV: COEFFICIENT OF VARIATION)

<table>
<thead>
<tr>
<th></th>
<th>28 days (GPa)</th>
<th>3 months (GPa)</th>
<th>12 months (GPa)</th>
<th>23 months (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>BLG</strong></td>
<td>37</td>
<td>37</td>
<td>40</td>
<td>44</td>
</tr>
<tr>
<td></td>
<td>E = 37</td>
<td>E = 37.5</td>
<td>E = 40.1</td>
<td>E = 43</td>
</tr>
<tr>
<td></td>
<td>37</td>
<td>38</td>
<td>40.18</td>
<td>42</td>
</tr>
<tr>
<td><strong>BSP</strong></td>
<td>36</td>
<td>36</td>
<td>29.8</td>
<td>27</td>
</tr>
<tr>
<td></td>
<td>E = 36</td>
<td>E = 36</td>
<td>E = 30.5</td>
<td>E = 27</td>
</tr>
<tr>
<td></td>
<td>36</td>
<td>36</td>
<td>30.9</td>
<td>27</td>
</tr>
</tbody>
</table>

![Figure 7.11 Elastic modulus of elasticity in concretes](image)

Figure 7.11 Elastic modulus of elasticity in concretes
In BLG, this value was raised to 1.6%, 8.4% and 16.2% at 3, 12 and 23 months, respectively. But in BSP, it was constant between 28 days and 3 months and then it decreased by 15.3% and 25% at 12 and 23 months, respectively (Figure 7.12).

![Graph showing variation of modulus of elasticity in concretes](image)

**Figure 7.12** Variation of modulus of elasticity in concretes

In BSP, even the low expansion level of less than 0.0044% at 3 months prevented the increase in modulus of elasticity. It has been already confirmed by other authors (section 2.9.3). Also, the expansion levels of 0.076 and 0.13% caused the remarkable reductions of 15% and 25% in modulus of elasticity. In section 7.3.2, the results of static modulus of elasticity will be compared with those of dynamic modulus of elasticity.

### 7.2.4 Conclusion on the results of mechanical tests

Figure 7.13 presents the summary of mechanical properties variation with expansion in BSP during 23 months. The result of mechanical tests shows the sensitivity of these properties to damage caused by ASR. This reaction did not cause significant reduction on compressive and
tensile strength of concretes, when the expansion was less than 0.08%. The modulus of elasticity is the most sensitive property due to damage caused by ASR.

![Graph showing variation of mechanical properties of BSP with expansion](image)

**Figure 7.13** Variation of mechanical properties of BSP with expansion

Above results are in accordance with those of other authors explained in section 2.9.

### 7.3 Non destructive tests

This section shows the results of non destructive tests in the reactive concrete (BSP) and the non-reactive concrete (BLG). All properties measured in this section were taken on three specimens and the data presented are the average of measurements on all three. It should be noted that NDT were performed on specimens at room temperature, when they had already removed from the oven that could induce losing temperature and humidity of samples and reducing pulse velocity proportionate the variation of above parameters. Therefore, NDT results have to be evaluated bearing such factor in mind.
7.3.1 Ultrasonic pulse velocity

This technique is sometimes used as a mean of checking the quality of concrete in a structure affected by ASR. It depends on the presence of open cracks and the absence of gel in the cracks. Figure 7.14 shows the ultrasonic pulse velocity (UPV) of two concretes. This value in two concretes was always more than 4000m/s. The high level of UPV in concrete containing limestone aggregate has already investigated by Sulub (1990), Rivero (1996) and Carrillo (2000). They reported UPV of limestone rocks is between 5900m/s and 6100m/s, depending to porosity and density of rock. Thus, velocity of concrete containing this type of aggregate would be high.

In BLG, this value was always more than 4575m/s that mean its high quality (Table 2.4 in section 2.8). In BSP, in spite of the ongoing reaction, UPV of damaged concrete was higher than 4400m/s that mean it is still in good condition. In BLG, UPV was raised 4.8% during the first 10 months and then it varied between 4650 and 4950m/s during the next months (Figure 7.15).

In BSP, this value was reduced slightly (around 1%) during the first 6 months, followed by 1.6% increase in the next 3 months. From 9 months, it decreased to 6.1% at 23 months. Figure 7.16 illustrates the variation of UPV caused by ASR damage in BSP. This value decreased around 1%, even for the low expansion level of 0.004% at 3 months. Between 6 and 9 months, when the expansion raised from 0.046 to 0.068%, this value increased to 1.6%. As the UPV is the surface phenomenon the reason for self-healing in UPV could be the formation of ASR gel on the concrete surface that filled some parts of microcracks generated by the reaction. From 9 months, UPV decreased with increasing expansion. Pulse velocity decreased by 6.1% at the highest expansion level of 0.13%. These results show the low sensitivity of ultrasonic pulse velocity of reactive concrete to ASR damage for the low expansion levels. In other words, UPV decreases when ASR has sufficiently progressed in concrete and expansion is relatively high.
Figure 7.14  Pulse velocity in concretes

Figure 7.15  Variation of pulse velocity in concretes
7.3.2 Dynamic modulus of elasticity calculated from resonant frequencies:

Figure 7.17 presents the dynamic modulus of elasticity of concrete calculated by longitudinal resonant frequency ($E_L$) and transversal resonant frequency ($E_T$). In BLG, $E_L$ was raised from 40.9GPa to 48.8GPa. In BSP, the initial $E_L$ was 44.6GPa. Then it decreased by 7.4% at 6 months followed by a 8.3% increase during the next 5 months (Figure 7.18). Since then, it decreased and levelled at 23 months with the value of 40.5GPa. Another important point is the significant difference between $E_L$ of two concretes after 12 months. Form 12 months; this value of BLG was always at least 6.5GPa more than that of BSP.

The initial $E_T$ of BLG was 47.2GPa, which was 1GPa more than that of BSP at the same time. Since 3 months, dynamic modulus of BLG was always more than that of BSP and the difference between the concrete dynamic modulus of elasticity increased with time. The maximum dynamic modulus of BLG occurred at 11 months with 6.1% increase followed by 1.4GPa variation during the next months. But in BSP, this value decreased and reached to minimum at 17 months with a 17.3% reduction and since then there were a few variations in this value.
Figure 7.17 Dynamic modulus of elasticity in concretes

Figure 7.18 Variation of dynamic modulus of elasticity in concretes
Figure 7.19 shows the variation of the BSP dynamic modulus of elasticity as a function of expansion. This figure shows the high sensitivity of dynamic modulus of elasticity to expansion. $E_L$ decreased by 7.4% for the expansion level of 0.046%. Then, it rose until the expansion level of 0.076 and then it decreased by 10.2% for the expansion level of 0.128%. $E_T$ decreased from 3 months, when the expansion was 0.004% and minimized for the expansion level of 0.123.

![Figure 7.19 Variation of dynamic modulus with expansion in BSP](image)

The results of the concrete dynamic modulus of elasticity indicate that these values were higher than that of static modulus of elasticity. The dynamic modulus of elasticity calculated from the resonant frequency refers to almost purely elastic effects and it could not be affected by creep. For this reason, $E_d$ was higher than that of $E_p$.

On the other hand, the ratio of static modulus to dynamic modulus, which is always smaller than unity, is higher the higher the strength of concrete and increases with age. In BLG, this ratio increased from 0.78 to 0.86 between 28 days and At 23 months, respectively. But, in BSP, it
decreased from 0.81 to 0.67. These results suggest ASR damage in BSP and lower strength of this concrete.

7.3.3 Nonlinear acoustics tests

At 6, 12 and 18 months, concrete cylinders were subjected to nonlinear acoustics tests. Table 7.4 shows normalized frequency shift versus strain amplitude (δ) and second-harmonic amplitude versus fundamental amplitude squared (β) in BLG and BSP, respectively. In BLG, both parameters remained quite constant, confirming that ASR has not occurred and concrete is not damaged. On the contrary, significant increases were measured with time regarding BSP concrete. Figures 7.20 and 7.21 show that these parameters are directly influenced by the expansion and the effect of ASR damage on δ is more important. In BSP, δ increased around 120% and 230% and β increased to 70% and 98% at 12 and 18 months, respectively.

<table>
<thead>
<tr>
<th></th>
<th>BLG</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>6 months</td>
<td>12 months</td>
<td>18 months</td>
<td>6 months</td>
<td>12 months</td>
<td>18 months</td>
<td></td>
</tr>
<tr>
<td>δ</td>
<td>41281</td>
<td>43133</td>
<td>43960</td>
<td>30937</td>
<td>68076</td>
<td>102107</td>
<td></td>
</tr>
<tr>
<td>β</td>
<td>1550</td>
<td>1582</td>
<td>1612</td>
<td>3403</td>
<td>5773</td>
<td>6735</td>
<td></td>
</tr>
</tbody>
</table>
7.3.4 Discussion on the results of non-destructive tests on concrete

The results of NDT show the reduction at various levels of the concrete properties. It seems that there is no unique relationship between expansion and loss in UPV and dynamic modulus of...
elasticity. Among the linear acoustics tests, the sensitivity of the modulus of elasticity calculated from transversal resonant frequency is highest with about 17% reduction and pulse velocity of concrete is the least sensitive test with the maximum reduction of 6.1%. Thus, effect of ASR on stiffness is more critical than the effect on microcracking. Another important point found from these results is the particularly increase of these values at one year that would be caused by formation of ASR gel as a self healing product and improving the concrete structure. This explanation could also justify the low reduction in tensile strength of concrete at one year. The results of nonlinear tests show the significant sensitivity of nonlinear parameters to expansion and ASR damage. All these results give confidence in using these non-destructive methods to identify and assess the damage caused by ASR. The variation of this property associated with ASR is similar to those of concrete damaged by freezing and thawing cycles. It was reported (Popovics, 1969) that, in concrete damaged by freezing and thawing cycles, pulse velocity is less affected by porosity than the resonant frequency. Observing the less reduction of longitudinal resonant frequency than that of transversal resonant frequency supports this opinion. Also, the transverse vibration produces higher stress in the concrete specimen than the ultrasonic pulses, which makes the effect of porosity more pronounced (Popovics, 1998).

7.3.5 Comparing the results of non destructive tests on mortar and concrete

Non-destructive tests on concretes showed the pattern of variation of these values with time is not similar with those of mortars. In concrete specimens, ASR was a surface phenomenon, but the results of petrographic analysis of mortars showed in mortar bars with thin section, the reaction extends completely in to the core of the section. In fact, using sands with the small size of less than 5mm in mortars and the small lateral dimensions of mortar bars accelerate deterioration of mortars and damaged them profoundly. Thus the reaction progression caused the high variation in their physical properties that induces the variation of NDT methods.

These results are similar to the results of resonant frequency of reactive mortars reported by Ludwig (1981) who studied the effect of ASR development on the resonant frequency of mortars containing opaline sandstone. He observed significant variations in resonant frequency during the reaction progression and relatively increases at the highest expansion levels. The
complementary discussion on the results of NDT in mortars and concretes will be presented in chapter 9.

7.4 Petrographic examination by Damage Rating Index method

According to Damage Rating Index method (Section 2.8), petrographic examination of two concretes was performed on polished section of concretes attained the maximum expansion level (23 months) in order to quantify the degree of ASR damage. The expansion level of specimens was 0.0036% and 0.13% in BLG and BSP, respectively. Figures 7.22 and 7.23 show the petrography of concretes at 23 months. This observation may explain some results obtained with the other tests. The results of this examination are presented in Table 7.5 and Figure 7.24.

In BLG, ASR damage is too weak that concerned to its low expansion level (0.0036%) and non-reactivity of aggregate used in this concrete. As it confirmed by Rivard (Rivard, 2002), the number of cracks observed on the surface of Limeridge limestone aggregate may be formed before incorporating in concrete mixture. Another significant feature is the cracks in aggregate-cement paste interphases (Debonded aggregate) that constituted 47.3% of damage in BLG.

In BSP, degree of damage was much higher than that of BLG (1144). This value is mainly consists of microcracks and microcracks with gel in aggregates (72%). In spite of high reactivity of this concrete, cracks in aggregate-cement paste interphases only constituted 8% of damage.
Figure 7.22 Petrography of BLG at 23 months

Figure 7.23 Petrography of BSP at 23 months
**TABLE 7.5 THE RESULTS OF PETROGRAPHIC EXAMINATION IN CONCRETE**

<table>
<thead>
<tr>
<th>Number of petrographic indices (Equivalent for 100cm$^2$)</th>
<th>BLG</th>
<th>BSP</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Expansion: 0.0036%</td>
<td>Expansion: 0.13%</td>
</tr>
<tr>
<td>Closed cracks in aggregate</td>
<td>36.1</td>
<td>61.1</td>
</tr>
<tr>
<td>Open cracks in aggregate</td>
<td>0</td>
<td>438</td>
</tr>
<tr>
<td>Cracks with gel in aggregates</td>
<td>8.3</td>
<td>324.1</td>
</tr>
<tr>
<td>Debonded aggregate</td>
<td>51.4</td>
<td>91.7</td>
</tr>
<tr>
<td>Reaction rim around aggregate</td>
<td>1.6</td>
<td>4.4</td>
</tr>
<tr>
<td>Crack in cement paste</td>
<td>11.1</td>
<td>108.3</td>
</tr>
<tr>
<td>Cracks with gel in cement</td>
<td>0</td>
<td>64.8</td>
</tr>
<tr>
<td>Air void with gel</td>
<td>0</td>
<td>2.5</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>108.6</strong></td>
<td><strong>1144</strong></td>
</tr>
</tbody>
</table>

![Bar chart](image.png)

Figure 7.24  ASR damage of concretes quantified by DRI method
CHAPTER 8

TESTS ON CONCRETE CORES

8.1 The initial characterization of concrete cores

Concrete cores drilled from the two locks described in section 4.3.3 were first subjected to various destructive and non-destructive tests when they were brought to the laboratory of the University of Sherbrooke (2006). The summary of the tests conducted on concrete cores from various depths is presented in Table 8.1 (Rivard, 2006)

<table>
<thead>
<tr>
<th></th>
<th>BCSC</th>
<th>BB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultrasonic pulse velocity (m/s)</td>
<td>4020 – 4732</td>
<td>3586 – 4330</td>
</tr>
<tr>
<td>Average</td>
<td>(4345)</td>
<td>(3992)</td>
</tr>
<tr>
<td>Dynamic modulus of elasticity (GPa), $E_L$</td>
<td>27.2 – 41.3</td>
<td>21.7 – 32.7</td>
</tr>
<tr>
<td>Average</td>
<td>(35.9)</td>
<td>(26.8)</td>
</tr>
<tr>
<td>Compressive strength (MPa)</td>
<td>31.2 – 37.3</td>
<td>25.4 – 44.6</td>
</tr>
<tr>
<td>Average</td>
<td>(34.7)</td>
<td>(36.8)</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>3.1 – 4.9</td>
<td>4 – 5.6</td>
</tr>
<tr>
<td>Average</td>
<td>(4)</td>
<td>(4.8)</td>
</tr>
<tr>
<td>Static modulus of elasticity (GPa)</td>
<td>23.3 – 35.3</td>
<td>15.8 – 24.4</td>
</tr>
<tr>
<td>Average</td>
<td>(29.1)</td>
<td>(20.4)</td>
</tr>
</tbody>
</table>

BB: Concrete cores extracted from Beauharnois lock
BCSC: Concrete cores extracted from Côte-Ste-Catherine lock
8.1.1 The initial characterization of BB

The reaction rim was observed around almost all aggregates of core samples extracted from concrete lock (Figure 8.1). Figures 8.2, 8.3 and 8.4 show the compressive strength, tensile strengths and static modulus of elasticity of core taken at different depths. The compressive strength of the core extracted from 0.24m of surface was the lowest (25MPa), indicating the high degradation of concrete surface. The compressive strength of other cores varied between 32MPa and 45MPa and there was not the relevance between the compressive strength and the depth of concrete. Indirect tensile strengths varied between 4.0 MPa and 5.6 MPa, with an average of 4.8 MPa that is comparable with a sound concrete.

There was a significant variation between the static modulus of elasticity of cores taken from different depths. This value varied between 16GPa and 24GPa, with an average of 20GPa. No tendency seemed to arise from the results. Among mechanical properties of core samples, only the static modulus of elasticity was less than that of an ordinary concrete. The results of static modulus of elasticity were comparable with those of laboratory concrete containing Potsdam sandstone affected by ASR (Saint-Pierre, 2006). The low values of static modulus of elasticity of cores and very low compressive strength of concrete surface may indicate the damage due to the environmental conditions such as wetting and drying cycles, freezing and thawing cycles or ASR.

The Ultrasonic Pulse Velocity (UPV) did not seem to follow any significant trend (Figure 8.5). UPV varied between 3586 m/s and 4330 m/s that point the difference in the quality of concrete cores from different lengths of drilling. The dynamic modulus of elasticity calculated from longitudinal resonant frequency (\( E_d \)) varied between 22GPa and 33GPa, with an average of 27GPa (Figure 8.6). The low values of dynamic modulus of elasticity indicate a loss of concrete rigidity. The low UPV and dynamic modulus of elasticity measured on the cores, as well as the presence of microcracks, gel and reaction rim on concrete surface emphasised that ASR has already started in this lock. In addition, based on the results of expansion test on laboratory concrete containing Potsdam sandstone (Rivard et al., 2002), it could be estimated that the
presence of dark reaction rim around the majority of ASR reactive aggregates is induced by expansion level higher than 0.06%.

Figure 8.1  petrographic features of BB cores before subjected to residual expansion test

Figure 8.2  Compressive strength vs depth in BB
**Figure 8.3**  Tensile strength vs depth in BB

**Figure 8.4**  Static modulus of elasticity vs depth in BB
Figure 8.5  Ultrasonic pulse velocity vs depth in BB

Figure 8.6  Dynamic modulus of elasticity vs depth in BB
8.1.2 The initial characterization of BCSC

Figure 8.7 shows the petrographic features of a core samples subjected to initial characterization. The presence of some microcracks and gel on the surface of aggregate and cement paste indicated that this concrete structure has already slightly affected by ASR. Figures 8.6, 8.7 and 8.8 show the compressive strength, tensile strengths and static modulus of elasticity of the core samples at different depths, respectively. The compressive strength of the specimens varied between 31MPa and 37MPa, with an average of 35MPa, which is comparable with a standard concrete and shows almost good quality for an old concrete structure since the cement hydration has already been completed. However, there was no relevance between the compressive strength and the depth of concrete. Indirect tensile strengths varied between 3.1MPa and 4.9MPa, with an average of 4.0MPa. A tendency shows that the tensile strength decreased from the surface to the depth.

As reported by Nixon (Nixon et al., 1985), the tensile-to-compressive strength ratio of concrete could be a good indication of internal damage due to ASR (section 2.9.2). This ratio in sound concrete varies from 0.07 to 0.11, but for damage of concrete due to ASR it is less than 0.06. In BB and BCSC, this ratio was 0.13 and 0.11 respectively, which shows measurement of compressive and tensile strength of concrete cores affected by ASR is not a reliable method to evaluate the real state of ASR damage concrete structures.

There was a significant variation in the static modulus of elasticity at different depths. This value varied between 23GPa and 35GPa, with an average of 29GPa. No tendency seemed to arise from the results. Contrary to the results of compressive strength and tensile strength measurement of core samples, which are reasonable, the average of static modulus of elasticity is less than that of an ordinary concrete. The results of laboratory concretes containing siliceous limestone aggregates (Chapter 7) showed a high static modulus of elasticity of 43GPa for BLG and a low value of 27GPa for BSP at higher expansion level. It has also resulted that among mechanical tests used for evaluation of ASR progression in laboratory concrete, only static modulus of elasticity is sensitive to ASR damage and compressive strength and tensile strength were less
sensitive. Therefore the modest values of static modulus of elasticity show the ASR has already affected the concrete.

Figure 8.7 Petrographic features of BCSC cores before subjected to residual expansion test

Compressive strength (MPa)

Depth (m)

Figure 8.8 Compressive strength vs depth in BCSC
Figure 8.9  Tensile strength vs depth in BCSC

Figure 8.10  Static modulus of elasticity vs depth in BCSC
The ultrasonic pulse velocity (UPV) did not seem to follow any specific trend (Figure 8.9). UPV varied between 4020 m/s and 4732 m/s, with an average of 4345 m/s. The low coefficient of variation (4%) of UPV may suggest that the concrete had similar quality for the entire length of drilling. Although the high UPV of core samples may indicate very good quality of the hardened concrete, it also can be related to the high UPV of limestone aggregate present in concrete. The dynamic modulus of elasticity calculated from longitudinal resonant frequency varied between 27GPa and 41GPa, with an average of 34GPa (Figure 8.10). These values did not indicate a particular tendency. The lowest $E_d$ of BSP affected by ASR was 40GPa, which was almost higher than those of core samples. These results confirm the evidence of ASR such as map cracking and ASR gel were the results of ASR damage. It was observed that the size and the volume of coarse aggregate in cores are not the same in all samples (Figure 8.7). Therefore, the significant difference between the results of mechanical and NDT of core samples from different depths can be the heterogeneity of concrete mixture.

On the other hand, the difference between UPV and dynamic modulus of elasticity may be related to the difference between the compositions of concretes. Some parameters such as water/cement ratio, cement content and density and the volume of aggregate in concrete can influence on the results of non destructive tests. In BCSC, the higher UPV of core samples was due to the higher pulse velocity of limestone aggregate embedded in concrete (Section 2.8). In BB, ASR attack and concrete composition could induce the lower value in the results of non destructive tests in these core samples.

It should be noted that although the Potsdam sandstone has lower density than limestones, but the proportion of aggregate in BB samples is higher than that of an ordinary aggregate. The measurement of density of core samples showed this value in both BB and BCSC samples were almost the same (between 2490 kg/m$^3$ and 2570 kg/m$^3$).
Figure 8.11 Ultrasonic pulse velocity vs depth in BCSC

Figure 8.12 Dynamic modulus of elasticity vs depth in BCSC
8.2 Expansion measurement and mass variation

8.2.1 Expansion measurement

The residual absolute reactivity of the Potsdam sandstone of BB and siliceous clayey limestone of BCSC specimens was determined by testing core samples in 1 molar NaOH solution at 38°C, using the procedure detailed by Bérubé and Fournier (2000). Due to this procedure, absolute reactivity of aggregates in concrete cores is determined, and classified according to Table 8.2 (Bérubé and Fournier, 2000).

<table>
<thead>
<tr>
<th>Expansion (%) at one year</th>
<th>Reactivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 0.04</td>
<td>Negligible</td>
</tr>
<tr>
<td>0.04 to 0.08</td>
<td>Low</td>
</tr>
<tr>
<td>0.08 to 0.12</td>
<td>Moderate</td>
</tr>
<tr>
<td>0.12 to 0.2</td>
<td>High</td>
</tr>
<tr>
<td>&gt; 0.2</td>
<td>Very high</td>
</tr>
</tbody>
</table>

The expansion of two concrete was measured regularly and their curves are shown in Figures 8.13 and 8.14. In BB, there was significant expansion during the first 3 weeks due to increasing the volume of core samples when they were stored in NaOH solution. Then it was increased gradually during the next months. The highest expansion level of 0.1% was measured at 41 weeks and then there is almost no expansion. The average of reaction rate in this concrete was around 0.002% per week. Also, from 20 weeks, there is significant difference between the expansions of cores from various depths of concrete structures that may describe their heterogeneity. The results of residual expansion test and initial characterization of BB cores confirm that BB had already affected by ASR and reached its highest expansion level.
In BSCS, the expansion increases significantly until 0.417% at 86 weeks and the expansion curves show that ASR expansion has not stopped in core samples, which show the very high reactivity of siliceous clayey limestone present in BCSC. Since 24 weeks, the difference between the expansions of core samples extracted from 4.1m and 4.9m of surface and that of extracted from 1.8m was around 0.06% that confirms the heterogeneity of concrete in the various depths of the lock.

The average of expansion of two concretes is presented in Figure 8.15. The average of maximum expansion level in BB and BCSC was 0.1% and 0.417%, respectively. It should be noted that the maximum level of expansion in BCSC cores is very high due to their storage conditions which accelerate the ASR evolution and the residual expansion of BCSC structure shall be less than that of core samples.

![Figure 8.13](image.png)

**Figure 8.13** Expansion of concrete cores extracted from Beauharnois lock (BB)
Figure 8.14 Expansion of concrete cores extracted from Côte-St-Catherine lock (BCSC)

Figure 8.15 Expansion of concrete cores
8.2.2 Mass variations measurement

Mass variation of concrete cores that was measured at the same time of expansion measurement is presented in Figures 8.16 and 8.17. In both concretes, the significant increase in the mass of specimens was observed at the first time of measurement that would be induced by the adsorption of solution into the volume of specimens. In BB, the mass variation increased by 0.83% during the first week. This value reached 1.14% at 22 weeks followed by a few decreases in the next weeks. In BCSC, this value increased by 1.44% during the first week and since then it was gradually increased to 2.37% at 87 weeks.

Figure 8.18 shows the mass variation of BCSC during the first week was 73% more than that of BB and from then, the mass variation rate of BCSC was always higher than that of BB.

![Figure 8.16](image-url)  
**Figure 8.16** Mass variations of concrete cores extracted from Beauharnois lock
8.2.3 Relation between expansion and mass variations of concretes

Mass variation can be compared with the expansion of concrete cores (Figure 8.19). As explained in previous section, in both concretes, the mass of samples significantly increased...
during the first 3 weeks due to the adsorption of solution by specimens. In BB, the initial mass variation of 0.89% occurred at an expansion level of 0.051%. Then, it increased slightly with a few variations at longer ages. At 50 weeks, expansion reached 0.098% and mass variation was 1.13%. The latency of expansion and mass variation of this concrete may emphasize this concrete was already affected by ASR and reached to extreme expansion level, before subjected to expansion measurement test in laboratory.

In BCSC, initial mass variation was higher than that of BB (1.49) and the initial expansion was less than that of BB (0.031%) that means BCSC samples adsorbed more volume of solution for the lower expansion level. The surface of BCSC cores used for expansion tests was already covered by a microcracks network that may induce higher increases in the mass of specimens at early age. At longer ages, the expansion increased significantly with a gradual increase in mass.

Furthermore, the comparison between the ratios of expansion and the mass variation of two concretes shows the higher mass variation of BCSC for the same levels of expansion that may be related to the higher volume of produced gel for the same expansion level.

![Figure 8.19 Relation between expansion and mass variation of concrete cores](image)
8.3 Petrographic examination by Damage Rating Index method

The degree of ASR damage in the cores before and after residual expansion test was quantified by Damage Rating Index method (Section 2.8). In BCSC, concrete cores used for non-destructive tests were collapse at 50 weeks, when the cores used for expansion measurement were still seen intact.

The results of DRI in BB are presented in Tables 8.3 and Figure 8.20. First, the degree of ASR damage for three specimens from 4m to 17m of concrete surface were measured and then after the residual expansion measurement, the extent of ASR damage were determined by DRI method on a core from 7.7m of concrete surface. The petrography features of concrete after residual expansion test is shown in Figure 8.21.

**TABLE 8.3 THE RESULTS OF PETROGRAPHIC EXAMINATION IN CONCRETE CORES (BB)**

<table>
<thead>
<tr>
<th>Number of petrographic indices (equivalent for 100cm$^2$)</th>
<th>BB1 initial 4.1-5.6m</th>
<th>BB2 final 7.7m</th>
<th>BB3 initial 12.1-13.1m</th>
<th>BB4 initial 16.6-16.8m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Closed cracks in aggregate</td>
<td>20.5</td>
<td>22</td>
<td>10.9</td>
<td>19.6</td>
</tr>
<tr>
<td>Open cracks in aggregate</td>
<td>152.4</td>
<td>76</td>
<td>50</td>
<td>109.5</td>
</tr>
<tr>
<td>Cracks with gel in aggregates</td>
<td>21.4</td>
<td>28</td>
<td>8.3</td>
<td>11.9</td>
</tr>
<tr>
<td>Debonded aggregate</td>
<td>96.4</td>
<td>0</td>
<td>12.5</td>
<td>32.1</td>
</tr>
<tr>
<td>Reaction rim around aggregate</td>
<td>43.5</td>
<td>43</td>
<td>32.8</td>
<td>35.1</td>
</tr>
<tr>
<td>Crack in cement paste</td>
<td>183.3</td>
<td>70</td>
<td>52.1</td>
<td>64.3</td>
</tr>
<tr>
<td>Cracks with gel in cement</td>
<td>90.5</td>
<td>36</td>
<td>54.2</td>
<td>14.3</td>
</tr>
<tr>
<td>Air void with gel</td>
<td>9.52</td>
<td>3</td>
<td>9.4</td>
<td>1.8</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>617</strong></td>
<td><strong>278</strong></td>
<td><strong>209</strong></td>
<td><strong>289</strong></td>
</tr>
</tbody>
</table>
Figure 8.20  ASR damage of BB cores quantified by DRI method before and after residual expansion test

Figure 8.21  Petrography features of BB after residual expansion test
The results showed the highest level of damage in BB1 from 4m of the surface. The presence of open cracks in aggregate and debonded aggregates were most significant in this sample, which indicated the extent of damage on the surface of concrete lock. However, the higher petrographic indices of damage in this sample could induce by combination of ASR damage and other environmental condition such as freezing/towing. Furthermore, reaction rim was observed around nearly all aggregates and there were a few differences between the quantitative index of reaction rim of cores (before and after the residual expansion test) that may confirm the ASR was already exhausted in this concrete lock. The low final DRI value of the BB2 is in agreement with the above explanations.

The DRI values of BCSC cores are presented in Table 8.4 and Figure 8.22. The initial DRI values of BCSC1 and BCSC2 from 2 to 5.7m of concrete surface were much less than that of BCSC3 from 5.3m after residual expansion test, which point the high ASR damage in BCSC3 due to the ASR evolution. On the other hand, there were a few differences between initial DRI values of BCSC cores. However, the presence of cracks and cracks with gel in cement paste in BCSC1 were observed. Therefore, it may be concluded that BCSC has already slightly affected by ASR damage and it has the very high potential of alkali-silica reactivity.

Figure 8.23 shows the petrography features of BCSC 4.1m sample for the highest expansion level of 0.359%. The DRI value in BCSC after the ASR evolution was very high (1392), which indicates a high degree of ASR-damage. This value, measured at an expansion level of 0.27%, is about 22% higher than that of the laboratory concrete containing Spratt limestone (BSP with a final expansion level of 0.13%). In BSP, the microcracks and gel were mainly observed within the aggregate particles, whereas in BCSC the microcracks and gel were observed on throughout the concrete.
### TABLE 8.4 THE RESULTS OF PETROGRAPHIC EXAMINATION IN CONCRETE CORES (BCSC)

<table>
<thead>
<tr>
<th>Number of petrographic indices (equivalent for 100cm$^2$)</th>
<th>BCSC1 initial 2.7-4.3m</th>
<th>BCSC2 initial 4.3-5.7m</th>
<th>BCSC3 Final 5.3m</th>
<th>BCSC4 initial 16.2-17.7m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Closed cracks in aggregate</td>
<td>17.8</td>
<td>22.9</td>
<td>51</td>
<td>19.5</td>
</tr>
<tr>
<td>Open cracks in aggregate</td>
<td>85.7</td>
<td>155.6</td>
<td>329</td>
<td>29.2</td>
</tr>
<tr>
<td>Cracks with gel in aggregates</td>
<td>28.6</td>
<td>36.1</td>
<td>214</td>
<td>22.9</td>
</tr>
<tr>
<td>Debonded aggregate</td>
<td>71.4</td>
<td>58.3</td>
<td>132</td>
<td>71.9</td>
</tr>
<tr>
<td>Reaction rim around aggregate</td>
<td>0</td>
<td>0</td>
<td>7</td>
<td>3.1</td>
</tr>
<tr>
<td>Crack in cement paste</td>
<td>183.3</td>
<td>172.2</td>
<td>443</td>
<td>154.2</td>
</tr>
<tr>
<td>Cracks with gel in cement</td>
<td>80.9</td>
<td>44.4</td>
<td>214</td>
<td>54.2</td>
</tr>
<tr>
<td>Air void with gel</td>
<td>17.3</td>
<td>4.2</td>
<td>2</td>
<td>30.2</td>
</tr>
<tr>
<td>Total</td>
<td>485</td>
<td>494</td>
<td>1392</td>
<td>385</td>
</tr>
</tbody>
</table>

Figure 8.22 ASR damage of BCSC cores quantified by DRI method before and after residual expansion test

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Figure 8.23 shows the petrography features of BCSC 4.1m sample for the highest expansion level of 0.359%.

In BB, the value of DRI was much less than that of BCSC (288). In this concrete, the volumetric ratio of aggregate to cement paste was higher than ordinary concrete and the microcracks on aggregate and cement paste and the reaction rim were constituted the main part of petrographic deterioration. The significant difference between the DRI of two concrete cores shows that the state of ASR damage in concretes related to the type of aggregate present in concrete. It was already confirmed by other author that in concrete containing Potsdam sandstone, the value of DRI is much less than that of concrete containing limestone (Rivard, 2002 and Saint-Pierre, 2006).

8.4 Non destructive tests

This section shows the variation of ultrasonic pulse velocity and dynamic modulus of elasticity calculated from the resonant frequencies. It has to be noted that the coarse aggregates present in BCSC cores that were used for NDT were very large (in some cases larger than the core section) and some particles were debonded from the cement paste. The variations in petrographic feature of cores were visible even with visual observation. More the reaction progressed in cores, more the aggregates expanded and the number and dimension of macrocracks between aggregate and cement paste increased. From 40 weeks, the dimension of
macroracks was even more than 1mm. After 50 weeks, the specimens were too deteriorated and two BCSC core collapsed.

8.4.1 Ultrasonic pulse velocity

Figure 8.24 shows the ultrasonic pulse velocity (UPV) of two concretes. The first measurement showed the UPV of BCSC was 55 m/s more than that of BB. In BB, the UPV was always higher than 3660 m/s that may point the good quality of this concrete during the reaction development (Table 2.4). Figure 8.25 shows a significant variation between the UPV of BB at different ages. UPV of BB decreased to 6% for the expansion level of 0.1% at 41 weeks.

In BCSC, there was a few variation of UPV during the first 24 weeks and then it increased by 4.8% at 29 weeks. Since then it decreased and minimized at 46 weeks (-1.5%) when the expansion reached 0.25%. Furthermore, in both concrete, in spite of the ongoing reaction, UPV was higher than 3000 m/s that means this method did not indicate the “real” state of damage due to ASR in concrete.

Figure 8.24 Pulse velocity of concrete cores
In BB, although the residual expansion was less than that of BCSC, there were significant variations between UPV at different ages. However, the collapsing of two BCSC samples due to ASR damage did not allow determining the average of UPV for the higher expansion level, but it seems that measurement of UPV cannot give the idea about the state of damage in core samples.

Figure 8.26 shows that the variation of pulse velocity with expansion. In BB, UPV was too sensitive to the reaction rate development and varied between -6% and +4.8%. But these variations did not relate to the expansion. In BCSC, there were a few variations in this value when the expansion was less than 0.16%. This value increased by 6.5%, when the expansion increased from 0.16% to 0.187%. The relation between expansion and the reduction of UPV was observed when the expansion increased up to 0.19%.
8.4.2 Dynamic modulus calculated from resonant frequencies

Figure 8.27 shows the dynamic modulus of elasticity calculated from longitudinal resonant frequency ($E_L$) and transversal resonant frequency ($E_T$) of concrete cores. There were a few differences between $E_L$ and $E_T$. In BCSC, $E_L$ was always less than $E_T$ that may be related to the formation of several transversal cracks on the surface of core samples. The initial $E_L$ of BCSC was 36GPa, which was 5.1GPa higher than that of BB. This value in both concretes decreased and reached 21GPa and 17.1GPa in BB and BCSC, respectively. In BB, $E_L$ decreased by 34% during 87 weeks and the variations were not proportionate with time (Fig 8.28).

But in BCSC, $E_L$ decreased by 51%. The initial $E_T$ of BCSC was 39.5GPa, which was 10.1GPa higher than that of BB. This value in both concretes decreased and reached 19GPa and 19.8GPa in BB and BCSC, respectively.
Figure 8.27 Dynamic modulus of elasticity calculated from resonant frequencies

Figure 8.28 Variation of dynamic modulus of elasticity in concrete cores
Figure 8.29 shows the different rate of decreases in dynamic modulus of elasticity with expansion in two concretes. In BB, $E_L$ decreased by 23% for the expansion level of 0.08% and when the expansion increased from 0.076 to 0.098, the reaction development made a few variations in this value. Although the expansion stopped in this concrete after 350 days, $E_L$ decreased by 34.4% during the next 8 months. In BCSC, this value decreased 11% for the expansion level of 0.23% and then it significantly decreased by 47.1% when the expansion reached 0.0.42%. In BCSC, $E_T$ increased by 1.8% during the first month and then it decreased to 18.8%, for the high expansion level of 0.27% at 50 weeks. In BB, the $E_T$ significantly decreased to 30.1% during 50 weeks, when the expansion level was 0.098%.

![Figure 8.29 Variations of dynamic modulus of elasticity with expansion](image)

8.4.3 Nonlinear frequency shift

At the last time of NDT measurements, a core sample of each concrete was subjected to nonlinear acoustics tests. Voltage excitation of piezoelectric actuators was varied linearly from 100 to 1500V. Figure 8.30 shows resonant frequency of BB varied from 7610HZ to 7530Hz. In BCSC, this value decreased from 10570Hz to 10265Hz (Figure 8.31). The frequency shift in BCSC (305Hz) was higher than that of BB (80Hz) which monitors the higher ASR damage in BCSC.
Also, the results show that nonlinear response depends on the type of concretes and the state of ASR damage in concrete. Figures 8.32 and 8.33 show the nonlinear parameters (δ and β) in BB and BCSC. In BCSC, both parameters were higher than that of BB. Above results show more a concrete is affected by ASR, more its nonlinear parameters (δ and β) will be important.

Figure 8.30  Resonant frequency shift in BB

Figure 8.31  Resonant frequency shift in BCSC
Figure 8.32 Normalized frequencies shift versus strain amplitude (b)

Figure 8.33 Second-harmonic amplitude versus fundamental amplitude squared (b)
8.4.4 Discussion on the results of non-destructive tests on concrete cores

The results of non-destructive tests show the reduction in dynamic modulus of elasticity calculated from resonant frequencies. Figures 8.34 and 8.35 show the variation of these properties with expansion in BB and BCSC, respectively. In both concretes, there is no relationship between UPV and expansion. It should be noted that the low UPV of BCSC at highest expansion level of 0.417% was due to the formation of several wide and deep microcracks on the surface of only specimen that has not been already collapsed.

In BB, there was a good relationship between $E_T$ and expansion, but $E_L$ was influenced directly by the expansion, when the expansion increased to 0.076%. Both $E_L$ and $E_T$ decreased around 35%, even when the expansion had been already exhausted.

In BCSC, both $E_L$ and $E_T$ was influenced directly by the expansion and the effect of ASR on $E_L$ was more critical than the effect on $E_T$. In this concrete, $E_L$ had 1.1 times the decrease of $E_T$.

Figure 8.34 Variation of UPV, $E_L$ and $E_T$ with expansion in BB
Figure 8.35  Variation of UPV, $E_L$ and $E_T$ with expansion in BCSC

Table 8.3 present the last results of expansion measurement and NDT methods on concrete cores. In BCSC with higher final expansion level, the results of linear acoustics tests are less than those of BB whereas the nonlinear properties of this concrete are higher. The above results confirm the reliability of nonlinear acoustics tests for evaluation the state of ASR damage in concrete cores.

In the next chapter the results of NDT on concrete core will be compared with those of concrete made in laboratory.

<table>
<thead>
<tr>
<th>Expansion (%)</th>
<th>Linear acoustics tests</th>
<th>Nonlinear acoustics test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>UPV (m/s)</td>
<td>$E_d$ (GPa)</td>
</tr>
<tr>
<td>BB</td>
<td>0.098</td>
<td>3970</td>
</tr>
<tr>
<td>BCSC</td>
<td>0.417</td>
<td>3520</td>
</tr>
</tbody>
</table>
CHAPTER 9

DISCUSSION

This chapter discusses the results obtained in previous chapters and suggests the application of reliable methods for evaluation of field structures.

9.1 Comparison between the results of mortars, laboratory concretes and concrete cores

At first, it should be noted that the specimens used in this study were stored at 38 °C and before NDT measurement they were removed from their container and placed at room temperature (around 20°C). In mortars bars with the small lateral dimension the variation of temperature may be faster and the difference between the UPV of specimens might be greater. The results obtained from NDT methods should be evaluated bearing such factor in mind.

9.1.1 Expansion measurement and mass variation

The comparison between final expansion level of BSP (0.13%) and BCSC (0.417 %) may show the higher reactivity of siliceous clayey limestone aggregate used in Côte-Ste-Catherine lock. However, the higher residual expansion of limestone aggregate used in Côte-Ste-Catherine lock could be related to the environmental conditions of lock such as the availability of alkalis ions which influence on the ASR progression rate and the residual expansion of cores. As it was discussed in section 9.1, the initial characterization of cores showed almost good quality of BCSC cores. Therefore, it could be concluded that Côte-Ste-Catherine lock has not severely affected by ASR.
As the ASR had already started in concrete lock, before the core samples extracted from the structure, it could be estimated that the final expansion level of laboratory concrete containing this aggregate would be higher. Figure 9.1 shows the expansion of concretes and concrete cores versus mass variation. In BSP and BCSC, the expansion and mass variation followed almost the same pattern.

Figure 9.1 Relation between expansion and mass variation in concretes and concrete cores

Rivard (Rivard et al., 2002) studied the reactivity of Potsdam sandstone in concrete. In the concrete containing an alkali content of 4 kg/m³, the final expansion level was 0.093% and in another concrete containing higher alkali content of 5.25, this value was 0.213%. In BB, the residual expansion of concrete cores was 0.1%. The petrographic feature of core samples and the result of initial characterization of concrete showed that concrete lock had been already affected by ASR.

9.1.2 Non-destructive test methods
- Ultrasonic pulse velocity (UPV)

Figure 9.2 shows the difference between ultrasonic pulse velocity in mortars, laboratory concretes and cores. UPV of mortars were always less than those of laboratory concretes due to their lower density. Although the aggregates present in BLG, BSP and BCSC are limestone aggregates, the initial UPV of BCSC was around 340 m/s less than that of BSP at highest expansion level. Also, Figure 8.9 showed that UPV of core samples extracted from surface to 6 m depth of BCSC varied between 4000 m/s and 4400 m/s. The lower initial UPV of BCSC confirmed this concrete had already been damaged by ASR.

In BCSC, the variation of UPV at older age was not similar to that of BSP. In BSP, UPV decreased, when the expansion reached 0.076%. But in BCSC, there were a few differences between the initial and final UPV.

Figure 9.2  Ultrasonic pulse velocities of mortars, concretes and concrete cores
Figure 9.3 Variation of ultrasonic pulse velocities in reactive concretes

Although the dynamic modulus of elasticity ($E_d$) laboratory concrete was closely related to expansion, the $E_d$ of mortars did not vary in the same way during the reaction evolution (Figure 9.4). As described in section 6.3 and 7.34, the results of NDT on mortars were influenced by various parameters such as dimensions of specimens, small size of aggregate present in mortar and high final expansion levels in mortars.

In BCSC, the initial dynamic modulus of elasticity was 36GPa which was at least 4GPa less than dynamic modulus of elasticity of BSP for the high expansion level of 0.128%. At older ages, the variation of this value with expansion was almost similar to that of BSP (Figure 9.5).

These results indicate that the sensitivity of dynamic modulus of elasticity not only related to the expansion progression, but also it may depends to the other parameters such as type of aggregate present in concrete cores and the degree of ASR damage in core samples at the initial time of measurement.
Figure 9.4 Dynamic modulus of elasticity of mortars, concretes and concrete cores

Figure 9.5 Variation of Dynamic modulus of elasticity in reactive concretes
- Nonlinear properties
Figures 9.6 and 9.7 show the nonlinear behaviour of laboratory concretes and cores. Although the $\delta$ was directly influenced by the expansion in BSP, but in BCSC, which was significantly more reactive than BSP, the $\delta$ was only 2.3% higher than BSP. Also in BLG, which was a non-reactive concrete, this parameter was always more than that of BSP at 6 months. Thus, it could be inferred that this parameter strongly depends to the type of concrete. It is recommended that this parameter should be measured at various stages of reaction development in the core samples.

Figure 9.7 shows a good relationship between the nonlinear parameter $\beta$ in all concretes and cores and their expansion level. In the other word, this parameter was less influenced by the type of concrete and could be used as a reliable NDT method for evaluation the state of ASR damage in concrete.

Figure 9.6  Normalized frequencies shift ($\delta$) vs expansion

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Figure 9.7 Second-harmonic amplitude (\(\beta\)) vs expansion

9.2 Reliable methods for assessing ASR in concrete cores

Regarding to the results of tests on laboratory concretes and concrete cores, measurement of static modulus of elasticity as a mechanical test and measurement of dynamic modulus of elasticity and nonlinear properties as the non-destructive methods are best methods for initial characterization of core samples. The residual ASR damage in concrete may be evaluated by expansion measurement test, measurement of dynamic modulus and nonlinear properties. Also petrographic examination by DRI method may determine the state of internal damage in concrete. Measurement of second-harmonic amplitude, which correlates very well with residual expansion, can be used in laboratory to assess the deterioration progress in concrete affected by ASR.

9.3 Assessment of ASR in field structure

Generally, assessment of the current condition of a concrete structure affected by ASR and evaluation of the residual damage include various steps. The preliminary visual inspection allows to estimate the cause of distress and in the case of ASR damage assess the location of damage, severity of degradation and the exposure condition induced the reaction.
NDT methods are needed to find damage in concrete structures and to determine the internal integrity of the structures. Depending on the type of investigated structure, it could be possible to perform NDT methods for in-site monitoring of damage. Measurement of resonant frequency is a recommended example. Also, measurement of surface cracking as an inexpensive and repeatable test can easily be performed for monitoring damage with time.

As described in section 2.6.3, in a field concrete containing reactive aggregate and high alkali content, when the relative humidity is less than a minimum relative humidity, ASR does not occur. The minimum relative humidity depends on the type of reactive aggregate in concrete. This value in concretes containing Spratt limestone and Potsdam sandstone is 65% and 90%, respectively (Berube et al., 1994). Albert (Albert et al., 1987) reported measurement of humidity could be considered as a non-destructive test in-site. They observed R.H. of 85% from 75mm and 350mm of the surface of concrete. By knowing this parameter one can estimate the possibility of the reaction progression in concrete. Grabe (Grabe et al., 2000) showed the possibility of reducing ASR expansion by measurement the humidity of concrete using electric manual probes.

However, in order to confirm the cause of the deteriorations observed during the visual inspection and evaluation the residual evolution of reaction, concrete core samples have to be collected from the concrete structure. The diameter of cores should be at least three times the maximum size of aggregates and their length should be two to four times their diameter. For concrete with aggregate < 35mm, a core diameter of 100mm is the most practical. The core diameter can influence on the results of tests in humid air; the greater the specimens diameter, the lower usually the expansion in the short term, but the higher in long term due to less alkali leaching (Bérubé, 1994). Also, the samples should not be taken too close to the surface and the first 10cm of concrete should be avoided (Bérubé et al., 2004).

Furthermore, the variation in mixture proportion, exposure condition, stress condition and internal deterioration within a single structure could lead variation in the test results. So, in order to have a good coverage of this variation, it was recommended to take samples from various parts of structure (Bérubé et al. 2004).
After coring, all cores should be wrapped immediately in order to prevent drying and wait one week at least before subjecting to expansion test to reach a relative hygrometric equilibrium. The petrographic examination of samples before and after the expansion test will help in the interpretation of the test results and determination the potential for residual expansion.
CHAPTER 10

CONCLUSIONS AND RECOMMENDATION

Civil engineering department of the University of Sherbrooke developed the methodology allowing evaluation of the state of ASR damage in concrete in recent years. There has always been a need for test methods to measure the in-place properties of concrete for quality assurance and for evaluation of existing conditions. Non-destructive tests do not impair the function of the structure and permit re-testing at the same locations to evaluate changes in properties with time. The objective of this study was evaluation of ASR evolution in concrete, using chemical and non-destructive tests. A methodology was proposed to examine these methods on mortar, laboratory concrete and concrete cores collected from two concrete locks.

The experimental program was started by preliminary examination of limestone aggregates presents on mortars and concretes and then the reaction progression in mortar, concretes and concrete cores was measured using above methods.

The following conclusions can be drawn from this Ph.D. thesis:

- There was a good relationship between expansion and mass variations of mortars and reactive concrete. This relation in both mortars followed almost the same pattern.
- Petrographic analysis by SEM is a reliable method to follow the gradual variation in microstructure of the mortar affected by ASR. In mortar containing non-reactive aggregate (Limeridge limestone), microcracks and gel were mainly observed within the aggregate particles and at aggregate-cement paste interphases. Whereas, in reactive mortar, ASR induces the microcracks and gel on entire volume of aggregate and cement paste.
- In mortar, the small lateral dimension of mortar bar (25×25mm) causes ASR damage in the entire volume of the mortar and using fine aggregate accelerate the ASR rate. Therefore, ASR damage in mortar bar may be much higher and more profound than
damage in concrete. Thus, although these results clearly show the variations in microstructure of affected mortar during the reaction evolution, but they cannot give an idea about the variation in microstructure of concrete damaged by ASR.

- There was a significant variation in the results of NDT in mortars and the sensitivity of these results did not related to the reaction progression. The combination of the results of SEM analysis and NDT showed this variation could be related to the volume of produced gel and volume of pore and microcracks in mortar. The reason for self-healing in physical properties of mortars might be formation of ASR gel that filled some parts of voids and microcracks.

- Although the structure of reactive mortar and concrete containing reactive sand are different due to the presence coarse aggregates in concrete, but the results of the nondestructive tests and SEM analysis on mortar bars can give an idea about the microstructure and physical properties of concrete containing reactive sand and damaged by ASR.

- ASR has already affected BB and the expansion level was estimated to be more than 0.06%. The residual damage in this structure will induce 35% reduction in dynamic modulus of elasticity.

- As BCSC samples used for expansion test are still expansive, expansion measurement has to be continued until the reaction been exhausted. Siliceous clayey limestone aggregate embedded in BCSC is much more reactive than Spratt limestone. The ASR damage has almost the same effect on dynamic modulus of elasticity of BSP and BCSC according to the expansion level of concretes.

- There were a good relationship between nonlinear parameters and expansion in laboratory concretes. Although the δ is directly influenced by the expansion, it depends to the type of concrete. The β is not influenced by the type of aggregate and the measurement of this parameter is strongly recommended as a reliable NDT method for assessing ASR damage in the concrete and estimating the expansion level. These measurements are easier to perform than expansion measurement.
Following the experimental program accomplished in this study, these recommendations are presented as the supplementary investigations:

- Chemical test aiming at quantifying the degradation of silica will be investigated on mortars containing two reactive aggregate (Spratt limestone and Potsdam sandstone).
- The measurement of tensile strength of concrete during the ASR evolution is recommended to find the influence of ASR on this mechanical property.
- The methods of nonlinear acoustics must be continued in order to evaluate their effectiveness to distinguish the ASR from the other damage mechanisms.
- The measurement of DRI during the ASR progression in concrete is recommended to explain the relationship between the variation of the petrographic properties of core samples and the variation in the parameters measured by the methods ND.
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