DEVELOPMENT OF A NEW SHORT-TERM CONCRETE PRISM TEST METHOD (SCPT) TO IMPROVE PREDICTABILITY FOR POTENTIAL ALKALI REACTIVITY OF AGGREGATES

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DEVELOPMENT OF A NEW SHORT-TERM CONCRETE PRISM TEST METHOD (SCPT) TO IMPROVE PREDICTABILITY FOR POTENTIAL ALKALI REACTIVITY OF AGGREGATES

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ABSTRACT

DEVELOPMENT OF A NEW SHORT-TERM CONCRETE PRISM TEST METHOD (SCPT) TO IMPROVE PREDICTABILITY FOR POTENTIAL ALKALI REACTIVITY OF AGGREGATES

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Alkali-aggregate reaction (AAR) is a chemical deterioration process which happens in concrete due to the presence of reactive aggregate, sufficient alkalis and moisture. The analysis for the reactivity may be very difficult because AAR has sophisticated and slow progression behaviour. Analysing the aggregates' structure in detail is primarily necessary before preparation of concrete mixture. Therefore, fast, simple, and reliable tests should be carried out.

In the first step of the study, seven pilot new test methods taking 28 days were performed on 8 types of aggregates to develop a new method, which would a reliable accelerated performance for predicting potential alkali reactivity of aggregates. RILEM Accelerated Mortar-Bar Test (14 days) and Concrete Prism Test (20 weeks) were applied on these aggregates to analyse the degree of correlation between the developed new methods. Common specifications of these methods are usage of 50 mm x 50 mm x 200 mm concrete prisms, 16 mm maximum aggregate size, storage temperature at 60 ^{o}C and soak solution with different ingredients. These solutions were prepared by using variable chloride based de-icers with sodium hydroxide. After completion of the preliminary study, two new methods in addition to seven methods were performed on new 24 types of aggregates by modifying preliminary best methods to improve correlation. Data obtained from totally 32 types of aggregate shows that a new method entitled as short-term concrete prism (SCPT) test can be applicable due to good correlation with Concrete Prism Test Method and relative compliance with field performances of test aggregates.

Keywords: Alkali-aggregate Reaction, Short-term Concrete Prism Test, Chloride Based De-icers, Correlation

ÖZ

AGREGALARIN POTANSİYEL ALKALİ REAKTİVİTESİNİN ÖNGÖRÜLEBİLİRLİĞİNİ ARTIRMAK İÇİN YENİ BİR KISA DÖNEM BETON PRİZMA TEST YÖNTEMİNİN (SCPT) GELİŞTİRİLMESİ

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Alkali-agrega reaksiyonu (AAR) reaktif agrega, fazla miktarda alkalilerin ve nemin olması durumunda, betonda oluşan kimyasal bir bozulma sürecidir. AAR'nin karmaşık ve yavaş ilerleme özelliğinden dolayı, reaktivitenin analizi çok zor olabilir. Beton karışımı oluşturulmadan, agrega yapısının detaylı olarak incelenmesi öncelikle gereklidir. Bu yüzden, hızlı, basit ve güvenilir testler yapılmalıdır.

Çalışmanın ilk aşamasında, agregaların alkali-reaktivitesini güvenilir, hızlandırılmış bir biçimde tespit etmek için yeni bir deney metodu tasarlamak için, 28 gün süren yedi ön çalışma amaçlı test metodu, 8 agrega üzerinde denenmiştir. RILEM Hızlandırılmış Harç Çubuğu (14 gün) ve Beton Prizma Test (20 hafta) metotları ile yeni metot sonuçlarının korelasyon derecesini tespit etmek için agrega numuneleri üzerinde uygulanmıştır. Tasarlanmış yeni metotların ortak specifikasyonları, 500 mm x 50 mm x 200 mm boyutlarında beton prizmaların, en büyük tane boyu 16 mm agrega kullanımı, 60 ^{o}C koşullandırılma sıcaklığı ve farklı içerikleri olan solüsyonlardır. Değişen ölçülerde klorür esaslı buz çözücüleri, sodyum hidroksitle birlikte kullanarak solüsyonlar hazırlanmıştır. Ön çalışma tamamladıktan sonra, korelasyonu iyileştirmek için, önceki yedi metoda ek olarak iki yeni metot, ön çalışma metotları revize edilerek 24 yeni agrega tipi üzerinde denenmiştir. 32 tip agrega üzerinde yapılan deneylerin sonuçları, gerek sonuçların beton prizma deneyiyle uyumundan gerek agregaların saha performanslarıyla göreceli uyumundan dolayı yeni bir kısa dönem beton prizma test yönteminin uygulanabilirliği gösterilmiştir.

Anahtar Kelimeler: Alkali-agrega Reaksiyon, Kısa Dönem Beton Prizma Testi, Klorür Esaslı Buz Çözücüler, Korelasyon

To our only and sweet daughter,
Gülru Şevval

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LIST OF ABBREVIATIONS

ABBREVIATIONS

AAR : Alkali - Aggregate Reaction

ACR : Alkali - Carbonate Reaction

AMBT : Accelerated mortar bar test

ASR : Alkali - Silica Reaction

CPT : Concrete prism test

DNM*i* : Designed new methods for the latter experimental study

G*i* : Aggregates used in the preliminary experimental study

NG*i* : Aggregates used in the latter experimental study

PH : Proportional Harmony

PNM*i* : Pilot new methods designed for the preliminary experimental study

RH : Relative Humidity

SCPT : Short-term concrete prism test

CHAPTER 1

INTRODUCTION

1.1 General

The need for construction works has irrepressibly increased day to day since especially the beginning of the 20th century due to the rapidly rising world population and urbanization. Therefore, availability, abundance and economy of construction materials have become a priority interest for human being [Islam & Akhtar, 2013]. Today, concrete consisting of mainly aggregate, water and binding material, cement or another alternative binder like as lime, gypsum, silica fume, furnace slag, fly ash etc. is the most critical material for construction works. The complicated reactions can occur between concrete constituents such as alkali-aggregate reaction [Naik, 2008; Ingham, 2010]. It may be defined as a reaction between various constituents of aggregates in concrete and cement alkalis that may be from other sources. There are noted three types of the alkali-aggregate as follow:

i. alkali-silica reaction (ASR)
ii. alkali-carbonate reaction (ACR)
iii. alkali-silicate

ASR is the most common, so this study will focus on it. ASR is a chemical process emerging in concrete through the reaction between reactive silica in concrete aggregates and dissolved hydroxyl ions in concrete pore, mostly from cement. This reaction causes a gel formation expanding by absorbing water. This gel can generate tensile stress having potential to make serious deteriorations in concrete structures. In other words, a deformation of expansion ratio as much as 0.04-0.05% is enough to provoke cracking in concrete structure because tensile strength of concrete is 7-11% of its compressive strength [Swamy, 1994].

ACR is a chemical reaction occurring between dolomitic limestones and metal hydroxides in the pore solution of concrete. Alkali-silicate reaction is defined as a reaction of rocks such as greywacke, filite or argilite with metal hydroxides in concrete pore solution. It is a reaction that develops more slowly than alkali-silica reaction. Among these reactions, ASR is the most common type of reaction that attracts the attention of researcher [Hobbs, 1988].

The damage in concrete from ASR was firstly demonstrated by Stanton nearly 75 years ago. In Turkey, the first document about ASR was published in 1975 [Kocaçıtak, 1975]. This document stated that 30% of the aggregates used in some dams in Turkey were potentially reactive according to chemical test method (ASTM C289). It was reported that the mentioned aggregates had been used with fly ash having lower alkali content in these dam structures, so there was no sign of damage. In 1975, 85% of the cement produced in Turkey was known to have a low alkali content. Nevertheless, a significant rise in alkali content of the cement has happened on account of that cement factories transmitted from the wet production method to dry process. That is namely, the equivalent alkali content of cements produced in 1996 was between 0.81% and 0.97%, and this current state has not been changed significantly today [Andiç-Çakır et al., 2012].

First damage from ASR in Turkey was determined in 1995 just as widespread cracking observed in some roadway bridges in İzmir region. As a result of the research, it was understood that reactive glassy rhyolite originated from Gediz River and Nif Stream beds caused ASR formation if used over 3% in natural course and fine aggregates [Katayama, 2012]. Further studies and investigations have revealed that the reaction resulted from not only the aggregates from Izmir region (1) but also the aggregates from Aliağa (2) [Çopuroğlu et al., 2007]., Niğde (3) [Korkanç & Tuğrul, 2004], Afyon (4) [Ramyar, 2013], Sivas (5) [Erik & Mutlutürk, 2004], Fırat river basin (6) [Aşık et al., 2004], Deriner Çoruh River basin (7) [Sağlık et al., 2003], Ankara (8) [Bektaş et al., 2008] ve Sakarya (9) [Yıldırım et al., 2011]

Provided that test results of long-term observation indicate good performance for concrete aggregates including high alkali content, there is no need to experiment for detection of their potential alkali-reactivity. Otherwise, it is necessary to carry out experiments for determining whether certain aggregate-cement combinations may cause a harmful alkali-aggregate reaction or not.. The countries apply the test methods by selecting the most appropriate ones for themselves not more. Therefore, the test methods may vary from country to country. The major reasons for these varieties can be seen as those every country has a method developed specifically for themselves and petrographic properties of aggregates may show considerable variances in different countries [Andiç-Çakır, 2007].

Figure 1.1 The location of aggregates, potentially reactive for AAR, in Turkey [Ramyar, 2013]

Field examination of concrete structure may firstly be used for examining and observing ASR process [ACI Committee 221. 1998]. In this method, concrete structure over 10 years old, exposed to deterioration processes can be examined with regard to the physical appearance, ASR gel existence, cracking pattern etc [Stark, 1991a, 1991b].

Although field performance is a decisive indicator for potential reactivity of aggregates, it cannot give an exact idea due to the following reasons:

i. Absence of sufficient information about aggregates used in the structures.
ii. Absence of sufficient information about the factors affecting ASR such as amount of cement, alkalinity of cement, and curing conditions etc.
iii. Differences in external effects affecting concrete structures such as moisture, freeze-thaw, wetting-drying, sea-water and de-icing salts [Berube & Fournier, 1992].

For these reasons, laboratory tests involving all types of aggregates to be exposed to similar conditions are needed for acceptable evaluation of aggregates in terms of AAR.

There are too many test methods to detect aggregates in terms of alkali-reactivity. Petrographic examination, chemical methods, mortar bar methods, autoclave methods, concrete prism methods are nowadays applied for assessing AAR. Mortar bar methods such as ASTM C1293, ASTM C227, Canadian CSA A23.2-25A, RILEM AAR-2 take 16 days while concrete prism test methods take months or even years, very long time period. In some laboratory experiments, the reaction is accelerated by abnormally high cement content, alkali addition or testing at high temperatures. Therefore, test methods should be evaluated by considering two main factors. Firstly, under such abnormal conditions, some siliceous components may react at very different speeds than under normal conditions. Secondly, the physical effects of the reaction can be very different. These tests can only provide reliable results when compared with field conditions or tests performed on normally cured samples (long-term concrete prism tests). Discussion on AAR test methods are elaborately mentioned in Chapter 3.

1.2 Scope of Research

Scope of this research study is divided into two parts; preliminary and latter experimental studies at large. Processes of the preliminary experimental study are (1) to select test aggregates, (2) to prepare soak solutions with changing content, and (3) to perform standard test methods; AMBT and CPT and also MABT, (4) finally, to apply new developed test methods on these aggregates to accomplish the research objectives. 5 types of sandstone from Kizilirmak River basin, 1 type of sandstone from Ceyhan River basin, 1 type of granite from İzmir and 1 type of sandstone from Coruh River basin were tested with a high alkali cement type to prepare concrete mixes in the preliminary experimental study. "RILEM AC-219 AAR-2 Detection of Potential Alkali-Reactivity—Accelerated Mortar-Bar Test Method for Aggregates" and "RILEM AAR-4.1—Detection of Potential Alkali-Reactivity—Test Method for Aggregate Combinations Using Concrete Prisms" and also modified AMBT (at 60 ^{o}C) were applied with the aim of controlling the result of developed new test methods. These new methods were developed by using soak solution including NaOH and chloride based deicers in different ratios, narrowed concrete prisms (50 mm x 50 mm x 200 mm) and modified size distribution of aggregate within a specified size grade interval of available test methods. In preliminary experimental study, different soak solutions which were (1) 0.3N NaOH+1% NaCl + 1% $CaCl_2$, (2) 0.3N NaOH+2% NaCl, (3) 0.3N NaOH+2% $CaCl_2$, (4) 0.3N NaOH+2% $MgCl_2$, (5) 0.35N NaOH+1% $CaCl_2$, (6) 0.35N NaOH+2% $CaCl_2$ were prepared to design the best appropriate test method giving excellent correlation with standard test methods and field performance of aggregates. After completion of preliminary investigation, two new methods developed by making necessary arrangements in the previous better methods were also applied to all 32 types of aggregates from different resources and having different petrographic and mineralogical structures. Furthermore, petrographic and mineralogical evaluation were performed on some types of aggregates to detect the effects of these properties on potential alkali-reactivity. Lastly, an observational research was started to examine existing concrete structures prepared by the test aggregates to gather information about their field performances, so reliability of the developed new method can be checked by an extra way. All samples prepared for this experimental study are given as follow with their numbers and dimensions:

i. 120 mortar bars in dimension, 25 mm x 25 mm x 285 mm, for AMBT and MABT
ii. 96 concrete prisms in dimension, 75 mm x 75 mm x 300 mm, for CPT
iii. 252 concrete prisms in dimension, 50 mm x 50 mm x 200 mm, for the new designed methods.

Chapter 1 of the thesis includes introduction part, the theoretical considerations of alkali-aggregate reaction takes part in Chapter 2 and Chapter 3 includes literature review. Chapter 4 consists of materials and methods and Chapter 5 explains experimental study and development of the new method. Chapter 6 contains results of experimental study and analysis of data to improve correlation. Lastly, Chapter 7, Chapter 8, Chapter 9 are respectively composed of discussion, conclusions and recommendation.

1.3 Research Significance

Compared with the 20 week-concrete prism test method, the new method will be completed in a much shorter period of 28 days. Unlike the accelerated mortar-bar test method (AMBT), it will be free from aggressive crushing and manipulation of the aggregates, as well as alkali leaching from test specimens observed generally in CPT methods. The comparative study performed by using 32 types of aggregate has proved that the 28-day expansion results of the new method can show a high degree of correlation with 20-week expansion results of CPT method. These results infer the viability of the new method as an alternative reliable method for evaluating AAR.

1.4 Principal Objectives to Contribute to the Knowledge

i. To develop a more practical and a reliable new method to detect potential alkali-reactivity of aggregates by using miniature concrete prisms.

ii. To regulate test duration as 28 days considering completion duration of all necessary initial type tests in the standard of concrete aggregates (EN 12620).

iii. To minimize lacks of recommended standard test methods, such as false-positive/negative test results, excessive crushing of aggregates, excessive test condition in accelerated mortar bar test methods; long test duration and alkali leaching in concrete prism test method.

iv. To make this new method applicable in the laboratories in Turkey.

    Explanation: The laboratories serving in aggregate tests have narrowed concrete prisms with pin (50 mm x 50 mm x 200 mm) because they can perform the drying shrinkage test of aggregates (EN 1367-4) that is one of initial type tests in the standard of concrete aggregates by using these concrete prisms. On the contrary, most of the laboratories do not have standard concrete prisms used in standard long-term concrete prism tests. Therefore, usage of 50 mm x 50 mm x 200 mm concrete prisms can allow for enabling the applicability of this new method widely in Turkey.

v. To investigate the effects of (a) chloride based de-icers (salts), (b) type of cement, (c) storage temperature, (d) size distribution of aggregates, (e) dimension of test specimen, (f) petrographical, origin, mineral composition, mechanical and physical properties of test aggregates on potential alkali-reactivity.

vi. To validate the new method to be developed by comparing with field performance of aggregates in existing concrete structures.

CHAPTER 2

THEORETICAL BACKGROUND

The general outline of this chapter comprises of general information about the alkali-aggregate reaction (AAR), types of AAR, general overview about the alkali-silica reaction (ASR); chemistry and mechanism of ASR, role of calcium in ASR, the effectual factors affecting the process of ASR; composition of ASR products, diagnosis of ASR damage and lastly mitigation measures of ASR effect.

2.1 Alkali-Aggregate Reaction

Alkali-aggregate reaction occurs when alkali hydroxides of cement or other external resources and some reactive constituents of aggregates react in a humid environment. After the start of the reaction, deterioration process starts to appear on the surface of concrete structures at different shapes such as map cracking, pop outs, fragmentation and spalling [Swamy, 1992]. The potential reactivity of concrete aggregates should be determined by test methods before construction because the symptoms of the deterioration process begins to appear in a few years after the concrete structure is built up. Therefore, AAR damages may be threatening the service life of the concrete structures unless preventive measures are taken.

In the 1920s and 1930s, severe cracks were observed on the surface of some concrete structures in California, USA though they were constructed in accordance with the construction techniques and standards. In the 1940s, Stanton demonstrated that the main reason for these damages appeared on concrete structures was the alkali-aggregate reaction between the main components of concrete. Stanton's experimental studies showed that the reason of the damage was the expansion from reaction between opal aggregate and high alkaline cement [Stanton, 1940; Swamy, 1992; Mindess & Young 1981; Andiç-Çakır, 2007].

Aggregates contain different types of reactive components that classify alkali-aggregate reaction (AAR) into three main types. These types are Alkali-carbonate reaction (ACR), alkali-silicate reaction and alkali-silica reaction (ASR) [Bektaş, 2002].

2.1.1 Alkali-Carbonate Reaction

The reactions of some dolomitic rocks with cement alkali are called the alkali carbonate reaction (ACR). Reactive rocks mostly contain larger rhombic dolomite crystals scattered in fine-grained calcite and clay matrix. Calcite is one of the mineral forms of calcium carbonate; dolomite is the general name of calcium-magnesium carbonate. The dolomite crystals in the matrix of the reactive rock may be sparsely dispersed or closer to each other or more dense. Most of the sparse crystals have a uniform crystal shape, while others often have a more irregular crystal geometry [Swamy, 1992].

ACR formation is not as frequent as ASR. One reason for this is that the aggregates susceptible to ACR are not used much due to other factors such as low strength in concrete. Another reason is that potential reactive rocks for ACR are not geologically widespread. ACR was first described by Swenson in 1957. Swenson observed closure and cracking of joints on some concrete pavements in Ontario due to excessive expansion six months after construction. Unlike the alkali-silica reaction, Swenson realized that the reaction occurred between clayey calcitic dolomite aggregate and cement alkalis [Swenson & Gitlott 1964]. ACR expansion forms when alkali ions and water molecules migrate to the fine-grained area surrounding the dolomite crystal. In particular, the migration of these pressure-producing products into the crystal as they crystallize, the growth and rearrangement of the non-dolomitization products create expansion [Farny & Kosmatka, 1997; Andiç-Çakır, 2007].

There is no definite property or crack geometry to describe the reaction in field where concrete exposed to ACR. The resultant cracks are a reaction of the concrete against internal stresses and may vary depending on the shape and moisture condition of the element. The petrographic analysis of concrete can be used to determine whether the cause of the cracks is due to ACR [Ozol, 1994]. However, microscopic examination revealed no reaction product gel as in ASR. Instead, brucite and calcite formed as a result of the dolomitization reaction may be observed [ACI Commitee 221, 1998].

Comparing ASR and ACR cracks, ASR cracks in concrete damage the element in two ways; (1) internal cracks of aggregate parts and their extension towards mortar and paste, (2) elongation of existing cracks due to migration and subsequent expansion of ASR gel. The main cause of ACR damage is the propagation of cracks in the reactive coarse aggregate into the mortar and paste. On the other hand, visual manifestations caused by both reactions are typically analogous. Map-cracking, displacement, pop-outs, exudations are noted manifestations of ASR and ACR [ACI Commitee 221, 1998].

2.1.2 Alkali-Silicate Reaction

Alkali silicate reaction occurs in alkaline-rich concrete containing argilite and greywacke aggregate. The reaction process of such rocks with alkalis is so slow and not fully understood. The silicate minerals in these aggregates expand and cause damage to the concrete. Expansion of each piece of rock occurs when the pre-dry aluminosilicate surfaces in the microcrystalline portions of these rocks absorb water. There is a direct proportion between expansion percent and porosity ratio of the microcrystalline material. This reaction is less common than alkali carbonate and alkali silica reactions [Thomas et al., 2011].

2.1.3 Alkali-Silica Reaction

Alkali-silica reaction (ASR) is a chemical reaction forming in concrete structure due to reactive siliceous content in aggregate and alkali hydroxide ions contained in pore solution of concrete. The three main requirements for formation of ASR expansion are as follow (Figure 2.1):

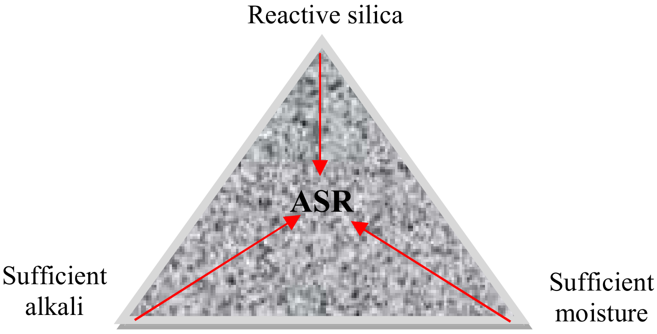
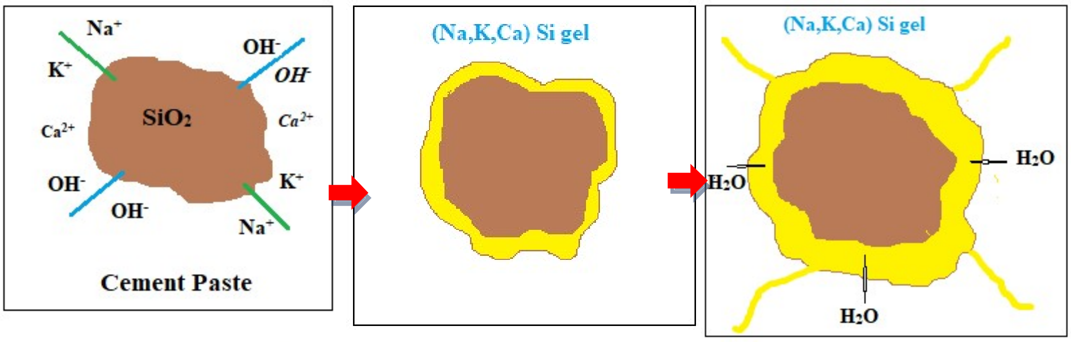


Figure 2.1 The necessities for formation of ASR expansion

i. Reactive silica components in aggregate.
ii. Pore solution with higher pH (> 13.2) and alkalis [Tang & Fen, 1980].
iii. Sufficient moisture that means RH≥ 85% [Chatterjii et al, 1989].



**Concrete pore solution is dominated by Na, K and OH (with minor amounts of Ca)
If the silica in the aggregate is reactive, the OH and then the Na & K will react with the SiO_2**

**The production of the reaction is in the alkali-silica gel composed of Na, K, Ca and Si
The gel forms araund and within the aggregate**

The gel imbibes water from surrounding cement paste and expands Eventually, the swelling pressures may excedd the tensile strength of the surrounding paste and cause expansion & cracking of the concrete

Figure 2.2 The order for ASR formation [Thomas et al., 2013]

As a reaction product, a gel having calcium content in different proportions starts to be prone to grow out of absorbed water due to its hygroscopic property. This gel creates tensile stresses in a concrete structure, causing expansion and naturally cracking in concrete with map shaped. The order of ASR formation is schematically explained in Figure 2.2 [Thomas et al., 2013].

2.2 Chemical Comprehension of Alkali-Silica Reaction

ASR occurs because the hydroxyl ions existing in concrete pore solution react with particular silica within the aggregate structure. Silica (SiO_2) is fundamentally composed of siloxane bunches (≡Si-O-Si≡), however, crystalline silica especially at the surface is cluttered and surface oxygen are hydroxylated (indeed in immaculate water) shaping silanol bunches (≡Si-OH) (Figure 2.3) [Thomas et al., 2013].

Dissolution of silica is initiated by the existence of hydroxyl ions (OH^-) in a high concentration. Main steps of this dissolution are (1) neutralization of the silanol groups; (2) assault towards the siloxane groups. The equations for these reactions can be explained as follow [Thomas et al., 2013].

$$\equiv Si\text{-}O\text{-}Si + OH^- \rightarrow Si\text{-}O^- + H_2O \qquad \text{Eqn.2.1}$$

$$\equiv Si\text{-}O\text{-}Si\equiv + 2OH^- \rightarrow 2Si\text{-}O^- + H_2O \qquad \text{Eqn.2.2}$$

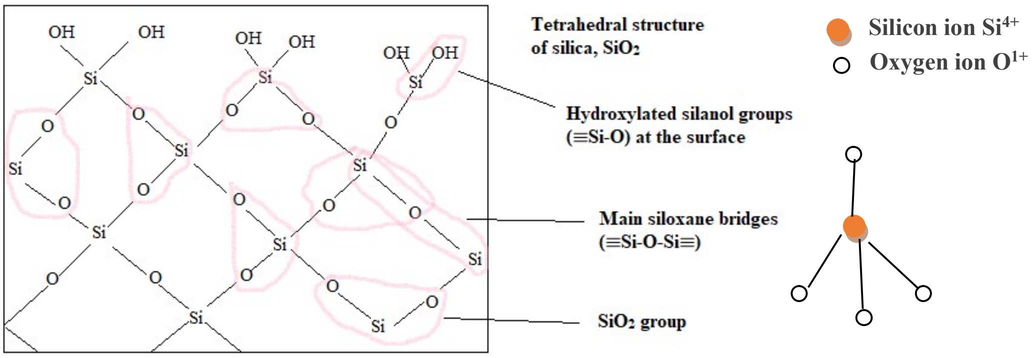


Figure 2.3 The Siloxane Network [Thomas et al., 2013]

After the breakage of siloxane bridges, the structure of silica network starts to be destroyed (Figure 2.4). $Si\text{-}0^-$ ions having a negative charge draws the alkali ions like as sodium (Na^+) and potassium (K^+) having a positive charge existing aplenty in concrete pore solution. Moisture content is a major reason for formation of alkali silicate solution, the output product of ASR. Nevertheless, this gel mainly composed of calcium, potassium and silica occurs because of precipitation of silica in solution with existence of calcium. Dissolution of silica reduces the hydroxyl ion concentration and so the pH of the solution decreases. Figure 2.5 shows that solubility of silica will go on by the time the concentration of the concrete pore solution attains the equilibrium curve of the silica-pH in a framework consisting of silica and alkali hydroxide. The data obtained from the studies on pessium effect ,which will be elaborately mentioned in the following parts, reveal that premier ratio of $Si0_2/Na_2O$ specifies the ultimate concentration of silica. The case is further complicated in concrete presumably owing to plenty of calcium that depletes the concentration of silica and additionally ensures hydroxyl ions to the pore solution. Eventually, equilibrium term in concrete structure are gradually achieved [Thomas et al., 2013; Iler 1979; Urhan 1987].

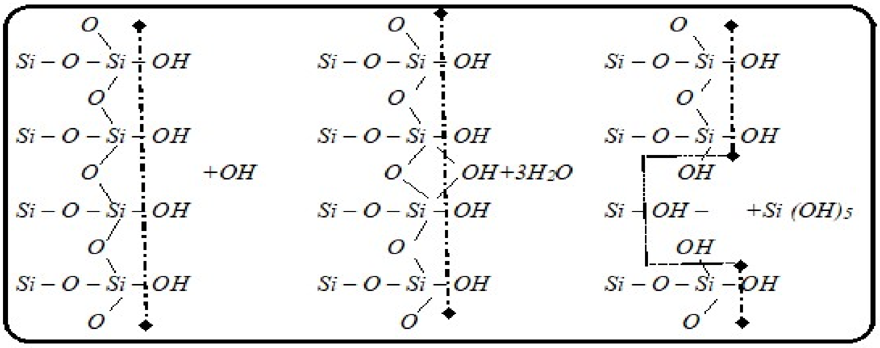


Figure 2.4 Dissolution of Silica [Iler 1979; Urhan 1987]

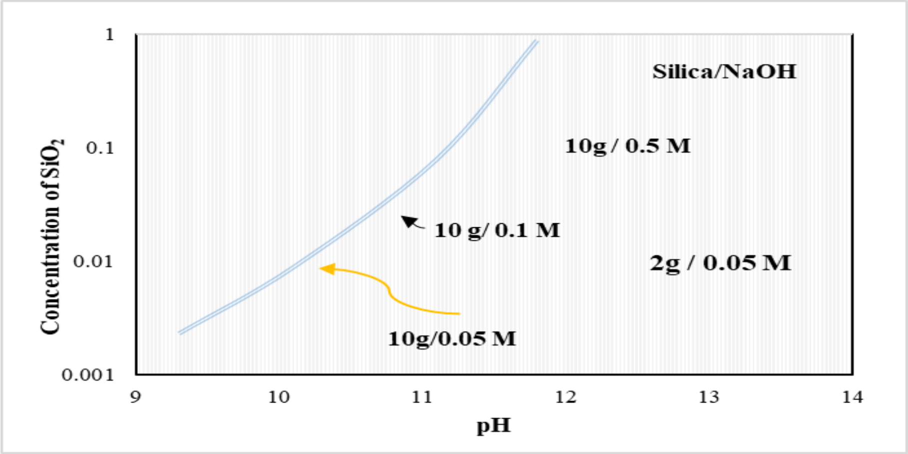


Figure 2.5 The curve of Equilibrium Solubility (SiO_2-pH) [Glasser & Kataoka 1981b]

As for other studies on ASR chemistry, Chatterji has proposed a mechanism concept including principal points as below:

a) Ionization of ≡Si-OH groups existing on the reactive particles' surface starts and then a negative charge density is picked up by the reactive particle [Chatterji, 2005].

b) Increase in pH and ionic strength of the surrounding solution causes a rise in the charge density belonging to a particle. The reasons for this increase are surface adsorption and penetration of OH^- in the particle stated by extent of the attendant hydrated positive ions. Throughout this continuum, ions such as calcium (Ca^{2+}), sodium (Na^+), hydroxyl OH^- and water (H_2O) are pumped in the particle as the penetration of alkalis such as potassium (K^+), sodium (Na^+), calcium (Ca^{2+}), barium (Ba^{2+}) [Chatterji, 2005].

c) Supplementation an irrelevant electrolyte such as sodium chloride NaCl to the pore solution can lead to the ionic strength of a solution to rise. Similarly, extra sodium hydroxide (NaOH) raises the ionic strength, pH at 10 and silica dissolution. On the other hand, sodium hydroxide (NaOH) reduce calcium (Ca^{2+}) concentration due to common effect of the ions [Chatterji, 2005].

d) A disintegration of inside ≡Si-O-Si≡ bonds and release of silica develop with the diffusion of sodium hydroxide (OH^-). The released silica may penetrate into the particles that is hampered by calcium ion (Ca^{2+}) [Chatterji, 2005].

e) Entrance of ions (calcium, sodium and hydroxide) and water that is absorbed from surroundings the particle into potentially reactive granule cause a generation of explosive pressure. This pressure decreases with increasing dissolved and diffused silica. The process of explosive pressure generation is explained with two main steps in Figure 2.6 [Chatterji, 2005].

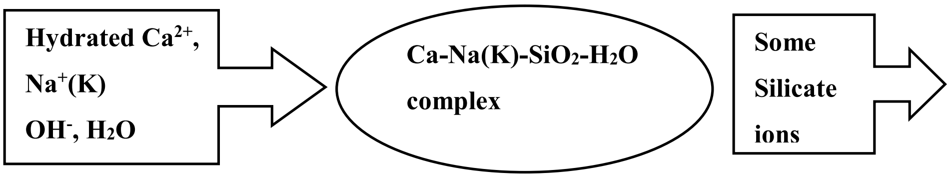


Figure 2.6 The process of pressure generation [Chatterji, 2005]

2.3 The Mechanism of Formation of the Alkali-Silica Gel

Figure 2.7 shows a clear photograph of a thin concrete section affected by ASR. Because of the reaction, the inserted flint particle, observed in cement paste, starts to expand and then crack. The crack forming in cement paste and aggregate structure surrounds the cement paste over time and is partly filled with the gel, main product of ASR, openly seeming obvious between cement paste and aggregate particle [Thomas et al., 2013].

Although many studies have been performed to comprehend the mechanism of formation of the alkali-silica gel, an important progression has not been achieved. Unclear points for the mechanism could not be still resolved. Different theories have been put forward to explain this mechanism since the 1930s when ASR was firstly observed.

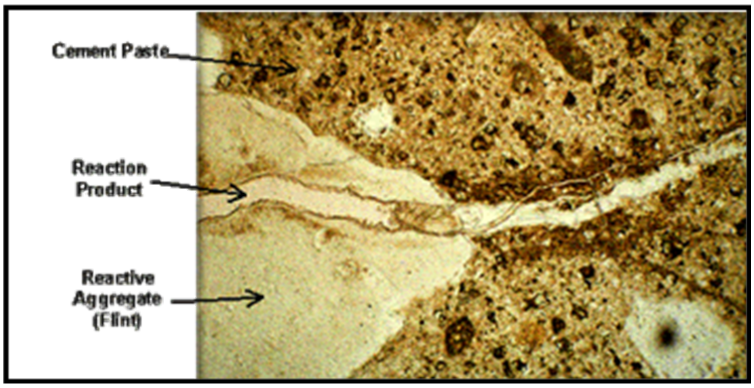


Figure 2.7 Thin-section cut of ASR-damaged concrete [Page & Page, 2007]

Absorption theory explains that ASR expansion depends mainly on the density, growth rate, and physical properties of the gel. As the growth rate of the gel is slow, the internal stresses can be prevented due to penetration of the gel into concrete. On the other hand, as the growth rate of the gel is fast, internal stresses are high enough to expand and crack concrete structure [Hobbs, 1988].

Osmotic pressure theory means that cement paste acts as an impermeable membrane against silicate ions. This membrane permits diffusion of water, hydroxyl ions and alkali ions, but prevents the diffusion of silicate ions. In this case, each region of that the reaction forms creates an increased pressure in the cement paste wrapped by this membrane. Hansen claims that the passage of the concrete pore solution through this membrane accelerates the reaction [Hansen, 1944]. However, Vivian criticized this theory by saying that the cracking continues after the reaction and the cement paste acts as an impervious membrane are impossible [Vivian, 1950].

C-S-H shell theory expresses that in case of the existence of calcium hydroxide $Ca(OH)_2$, hydroxyl and alkali ions from the concrete pore solution breaks into the aggregate matrix including reactive silica. On condition that alkali and hydroxyl ions enter, the diffusion of silica and penetration of ions inside the pore solution may occur owing to disintegration of ≡Si-O-Si≡ bonds. The diffusion of silica is limited whereas penetration of materials into the aggregate goes on in the event of higher intensity of calcium hydroxide and alkali salts in the concrete pore solution. This incompatibility creates an extensive force causing internal stress within the aggregate [Chatterji, 1987].

Calcium/alkali exchange theory states that an extra gel formation appears as calcium ion (Ca^{2+}) is absorbed by the formed gel in place of alkali ions reacting with the ambient of concrete pore solution. This gel hereby takes up further gap and so produces extensive forces, which create cracking in concrete structure [Rotter, 1995].

Prezzi et al. have modelled the expansion by the Guoy-Chapman **electrical double layer** theory of colloid chemistry. On the solid surfaces in the liquid, electrical charge, accumulations occur because of isomorph ion displacement, chemical reactions or ion adsorption. The first ion layer in contact with the layer is fixed and consists of positive ions that can be held by the negative charges on the solid surfaces. The second layer consists of negative charges in the pore solution with residual positive charges. The second layer is wider and the electrical potential decreases as it moves away from the solid. Algebraic sum of the forces in both layers is zero. An expansion happens in the solid surroundings of the second layer because the expansion of its width increases according to the load condition. The expansion of the layer becomes evident by the increase in the alkaline cations passing here. Cations' ability to take place is the function of their valences, densities and ion diameters. For example, the potassium cations are first retained in the region of oxygen, which is not bonded to the silica, and upon being saturated, in the region of the oxygen-bonded to silica, i.e. in the siloxane region. However, the lithium ion is more initially retained by both regions. This property provides a significant privilege to lithium cations [Prezzi et al., 1995].

The greater the valence of the counter ions in the double layer, or the greater the density of these ions, the smaller the thickness of the double layer and the impulse forces in the presence of water.

Therefore, ASR products, with high amounts of Na_2O_{eq} and low amounts of CaO/ Na_2O_{eq} in the mortar bars containing natural pozzolan, fly ash and slag, produce higher expansions [Prezzi et al., 1995].

According to Diamond, the composition of the ASR product in concrete undergoes change over time by collecting calcium ion from the cement paste. The calcium content of the ASR product varies depending on the location in the concrete and age of the product [Diamond, 2000]. Powers and Steinour claim that density ratio of the calcium ion / alkali ion in the concrete space solution controls the gel expansion. If this ratio is high, a complex calcium-alkali-silica-hydrate product, a type of non-swelling (or limited to swell) could be expected to occur. Conversely, a gel comprising of alkali-silicate-hydrate with swelling type and including small amount of calcium becomes apparent [Powers & Steinour, 1955]. Wang and Gillot explain that the presence of an excess of calcium hydroxide in the medium can accelerate the reaction even though they agree that the calcium-alkali-silicate gel has a non-swelling property [Wang & Gillot, 1989]. Researchers have pointed out that calcium ions cause the release of the alkalis in ASR product to the ambient by substituting them and the released alkalis increase the expansion by reacting with reactive silica in the ambient [Ramyar, 2013].

According Chatterji et al., calcium hydroxide accelerates the entrance of water molecules into the reactive particle with sodium, calcium and hydroxyl ions. High concentration of calcium ions also reduces the leakage of silicate ions from reactive particles to outside. As a result, it is explained that the expansion forms by ways of the entrance of ions such as sodium, calcium and hydroxyl more than the silicate ions leaking out into the reactive particles. In a mixed electrolyte medium with calcium hydroxide and sodium chloride, smaller ions such as sodium ions can be monitored more easily than the hydroxyl ions entering the inside as comparing them with the larger calcium ions [Chatterji et al., 1986; Chatterji, 1989; Chatterji & Thaulow, 2000]. Helmuth and Stark have concluded that ASR products are a mixture of alkaline silicate hydrate gel and weak crystalline calcium silicate hydrates containing different amounts of water and alkali [Helmuth & Stark, 1992].

2.4 The Role of Calcium on the Gel Expansion.

Many experimental investigations have made inferences that the presence of large amount of calcium, as calcium hydroxide $Ca(OH)_2$ is the only main reason for remarkable ASR expansion. In the event of lack of $Ca(OH)_2$, silica is inclined to remain in pore solution due to dissolution although there is a plenty of reactive silica and alkali hydroxides. A number of proposals about the role calcium on the mechanism of ASR has been introduced as follows but this role could not be fully clarified [Thomas et al., 2013].

i. Calcium substitute alkalis that provide reproduction of alkalis for subsequent reactions so it advances alkali recycling [Thomas, 2001 ; Hansen, 1944].
ii. Calcium hydroxide supplies a storage of hydroxyl ions in order to provide a solution with high concentrated hydroxyl [Wang & Gillott, 1991].
iii. Calcium, in high level in the solution, precludes silica from diffusion [Chatterji, 1979; Chatterji & Clausson-Kass, 1984].
iv. In lack of calcium, the dissolution of silica occurring in the solution with alkali hydroxide does not cause damage in the concrete structure [Thomas, 1991; Diamond, 1989].
v. Many studies have proven that pozzolans are very useful material to minimize the expansion in the concrete structure due to ASR and provide the depletion

of calcium hydroxide by means of pozzolanic reaction that decreases the quantity of calcium. Bleszynski and Thomas have alleged the role of pozzolans on the alkali-silica reaction with an experimental study. According to this study, no expansion was observed in concrete consisting of a reactive type of aggregate with fly ash in 40% ratio although it had been stored at 80 ^{o}C and in 1 M NaOH for 4 years. The cement paste with migrated ASR gel and saturated by it surrounds reactive particles but no damage appears (Figure 2.7) [Bleszynski & Thomas, 1998]. On the contrary, some researchers reveal that calcium oxide (CaO) may lead concrete consisting cement replacement materials such fly ash, furnace slag or silica fume to expand [Wang & Gillott, 1991 ; Tang et al., 1983].

vi. Concretes or mortars which are prepared with a reactive type of aggregate and stored in a solution with higher alkali concentration is hampered from expansion through extraction of calcium hydroxide. Chatterji used filtration method to extract the calcium hydroxide in concrete while Thomas made this process by carbonating the calcium hydroxide within mortar bars in an ambient including plenty of carbon dioxide [Chatterji, 1979 ; Thomas, 2001].

Even if the role of calcium hydroxide, $Ca(OH)_2$ on expansion mechanism is not fully perceived, it is obvious that the reaction to cause concrete damage needs calcium hydroxide. Therefore, damage from ASR expansion in concrete may be inhibited by reducing calcium content. Therefore, pozzolonic reactions can reduce the expansion by means of depleting calcium hydroxide [Thomas, 2013].

2.5 Effectual Factors Affecting the Process of Alkali-silica Reaction

Alkali-silica Reaction is an extremely complex reaction to examine in detail. As mentioned above, the three essential elements required for the formation of the ASR are the presence of reactive silica, high alkaline content and water in the environment. Many factors affecting ASR which are types of reactive silica, aggregate size, pessimum effect, alkali content of concrete, moisture effect, temperature effect, mineral admixtures and other external sources (e.g chloride based de-icers) are extensively discussed below.

2.5.1 Types of Reactive Silica

Concrete aggregates may origin from rocks, minerals or synthetic materials. Potentially reactive aggregates are listed in Table 2.1.

Table 2.1 Potentially reactive rocks and minerals [Thomas, 2013]

Reactive mineral	Chemical composition	Physical character
Opal	$SiO_2 \cdot nH_2O$	Amorphous
Chalcedony	SiO_2	Crystalline, strained, fractured, fibrous
Quartz	SiO_2	Crystalline
Tridymite	SiO_2	Crystalline
Cristobalite	SiO_2	Crystalline
Siliceous glass	Siliceous+(Al_2O_3 in less)	Glass
Rhyolitic	Siliceous+(Al_2O_3& Fe_2O_3 in less)	Glass, cryptocrystalline
Reactive rocks		
Opaline cherts	Rhyyolites and tuffs	Opaline concretions
Chalcedonic cherts	Dacites and tuffs	Fractured, strained
Quartzose cherts	Andesites and tuffs	--
Siliceous Limestone	Siliceous shales	
Siliceous dolomites	Phylites	

Silica (SiO_2) presents in the structure of most rock, but this does not mean that all siliceous aggregates bring about detrimental expansion in concrete structure. To give a striking example, both of the minerals named as quartz and opal have largely silica content. In contrast with having similar chemical content, quartz is generally non-reactive while opal is potentially reactive. Dissimilarity of these minerals in views of their silica solubility and crystalline structure can be clearly seen in Figure 2.7.

Amorphous structure may provoke the mineral to be unstable for alkali-silica reactivity at high pH. Therefore, the minerals consisting of largely of opal having amorphous structure are prone to react with concrete pore solution that causes the concrete structure to expand. On the contrary, quartz having well-crystallized structure may not be seen among the potentially reactive types of aggregate without considering other important factors such as temperature effect, ambient conditions, alkali content of concrete, the design life of concrete structure [Thomas, 2013].

As summary of Table 2.1, silica minerals such as opal, volcanic glass, strained or microcrystalline quartz, tridymite, chert, and cristobalite can be defined as reactive materials of the alkali silica reaction. Argillite, shale, chert, sandstone, flint, gneiss, granite, quartize, greywacke, hornfels, arkose, carbonate rocks, and arenite are best known examples of rock types consisting of the reactive minerals mentioned in the previous sentence. Nevertheless, many studies have proved that information about chemical composition/structure properties of materials are not enough to decide whether they are reactive or not. Some aggregates, for instance, composing of largely granite may be determined as non-reactive according to test results [Thomas, 2013]. Similarly, in this experimental study, the test results of granitic aggregates verify this idea as mentioned in Chapter 6.

Reactive substances in aggregates can be examined by petrographic examination or the chemical test method but the performance of the aggregates in mortar or concrete can be tested by many different test methods, which will be discussed in depth in Chapter 7.

Silica minerals having a substantially defective (amorphous or poorly crystalline) structure like opal, artificial or volcanic glasses and cristobalite are too inclined to react and create expansion in one or two years at very little amounts even in proportionally 1%. On the other hand, different forms of quartz such as in strained or microcrystalline, cryptocrystalline not enough reactive to cause rapidly progressive reaction so deleterious damage in concrete composing of this silica form does not occur rapidly [Thomas, 2013].

Quartz varieties, such as cryptocrystalline, microcrystalline or filtered quartz, react more slowly so they cause damage after longer time. They are not present in larger amounts than crystalline and amorphous silica [Thomas, 2013].

2.5.2 Aggregate Size

Many studies have proved that the particle size of the reactive aggregate is also effective on the damage caused by ASR. Figure 2.8 (a) gives an illustrative graph for particle size effect on ASR expansion designed according to works of Stanton applied for a type of reactive siliceous aggregate. The data obtained from these works manifested that the maximum expansion was observed at mortars prepared with size distribution ranging from 0.18 mm to 0.60 mm [Stanton, 1940].

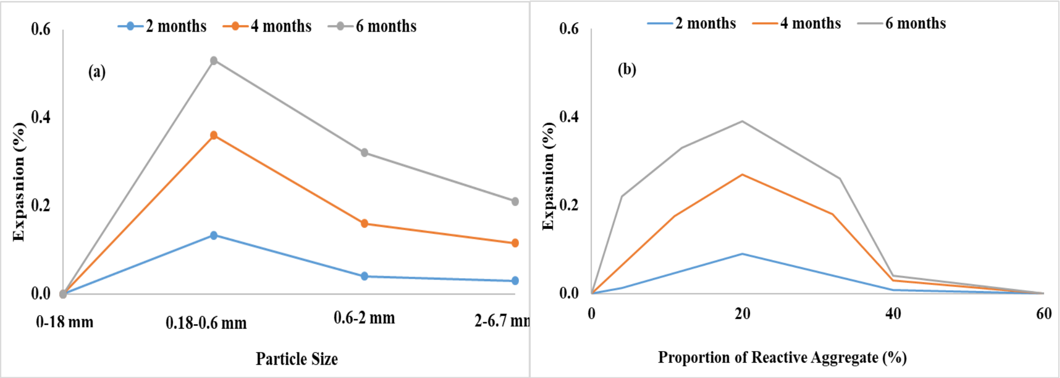


Figure 2.8 Particle size versus expansion percent (b) Proportion of reactive aggregate versus expansion percent [Stanton, 1940]

Vivian concluded that for the opal aggregate, as the aggregate size is between 0.07-0.85 mm, the expansions are maximum; the larger and smaller grains expand [Vivian, 1950]. Other researchers using opal silica stated that as the reactive particle size was reduced to 0.05 or 0.02 mm dimensions, the mortar expansions increased, but high expansion was not observed at lower than 0.02 mm [Han & Fang, 1984; Hobbs & Gutteridge, 1979; Diamond & Thaulow, 1974]. Mehta emphasized that as the reactive aggregate having a size distribution between 1 and 5 mm is used, the expansion was at maximum rate [Mehta & Monterio, 1993].

In the study performed by Kawamura et al., mortar bars were prepared by using 10% of the total aggregate in seven different sizes with aggregate/cement ratio of 0.75. These samples were cured at 38 °C for 50 days in moisture boxes. The total alkali content of the mortar is nearly 1.6% by weight of cement. The samples prepared using the finest opal (<0.074 mm) showed no significant expansion. 30-day expansion of samples containing medium-sized opal was measured as 0.3%. On the other hand, the samples containing two most coarse (2.5-5.0 mm and 1.2-2.5 mm) opal have lower expansion than others do [Kawamura et al., 1983]. The reason for this was asserted as the fact that the coarse portion is not affected by the same amount of reaction compared to the fine [Helmuth & Stark, 1992].

Kodama and Nishino prepared mortar mixtures containing reactive andesite in two different grain sizes (<0.15 mm and 0.15-5 mm).

The alkaline content of the normal Portland cement they used was 0.85% Na_2O_{eq} and the cement content of the mixture was nearly 610 kg/m^3. The 56-day expansion of the mixture containing 0.15-5 mm andesite was higher than 0.2%, whereas the expansion of the mixture containing andesite smaller than 0.15 mm was about 0.02% [Kodama & Nishino, 1986]. The data in this study is in contradiction with that in Vivian's study. The contradiction between different researches may be due to the difference between the reactivity of the aggregates used in the studies, as well as from different cement / aggregate ratios, different alkali content and environmental conditions.

It has been determined that the expansion of the concrete come into existence in the early stages if only reactive fine aggregate is used in fine aggregate and it becomes fixed in the future. On the other hand, if only coarse aggregate is used as reactive aggregates, the expansion may be slow and occur much later [Nishibayashi &Yamura, 1992].

Zhang et al. suggest that ASR expansion increases as the particle size decreases for the siliceous aggregate from 0.15 to 10 mm. As the aggregate size increases, the ratio of cement / aggregate giving maximum expansion decreases [Zhang et al., 1999].

Two important impacts should be considered in this regard; 1) the reaction speed of the reactive aggregate having a small particle size is higher, 2) the expansion of the coarse aggregate may be greater if long-term expansions are taken into account [Andiç-Çakır, 2007].

Ramyar et al. investigated the effect of reactive aggregate size and aggregate angularity on ASR expansion. In this study, the crushed natural aggregate, obtained by breaking one type of natural round aggregate with the coarse particles of the same aggregate was used in different sizes instead of the non-reactive limestone aggregate. Aggregate gradation was selected according to ASTM C1260 accelerated mortar bar method and used in the proportions specified in the standard. According to test result obtained by 14-day ASTM C1260 test method, the results of crushing and natural aggregates containing 25% reactive aggregates in medium particle size were quite different from each other. The size effect of the reactive particles is more explicit in the crushing aggregate. Although the angularity is not effective in the smaller and larger aggregate particles, the effect of angularity on expansion in medium sized particles becomes more of an issue. Moreover, the total expansion of the samples in which the reactive particle sizes were separately tested were higher than the control sample, completely consisting of reactive aggregates [Ramyar et al., 2005].

The reason for this may be an assertion that too many reaction zones create a barrier that affects each other [Shayan, 1992].

The use of a non-reactive aggregate is ideal, but it is not always a practical solution. The best way to know about the performance of the aggregate is to have a good information on its field performance. However, there may be no field record for each aggregate. Therefore, it is possible to get an idea about the quality of the aggregate, mostly through experiments that measure the potential reactivity of the aggregate. In order to improve the quality of the aggregate, the selection process called enrichment can be applied. In some cases, this process may result in the removal of a large portion of the reactive aggregate but as well discharging a quantity of high-quality aggregate. A number of reactive aggregates can be used by mixing with a non-reactive limestone aggregate to reduce the effect of ASR. As applying this process, it is important to take into account aggregate types showing pessimum proportion. Like in reactive thin material or marine aggregates, the elimination of the part containing alkaline by washing is another aggregate improvement process [ACI Committee 221. 1998; Andiç-Çakır, 2007; Farny & Kosmatka, 1997].

2.5.2 Pessimum Effect

Stanton stated that the expansion does not increase continuously with the increase of the reactive aggregate ratio. Mortar bars showed the expansion percent, which varied according to the proportion of reactive aggregate, limestone, having higher silica content in the mixture as, Figure 2.8 (b) shows. As a reactive aggregate, natural sand, was used at 20%, the maximum expansion was observed. On the other hand, the proportion of reactive aggregate in the mixture increased a dramatic value such as 6 %, the expansion decreased significantly even get close to zero expansion at the ratio more than 60% [Stanton, 1940].

There is no linear relationship between the potential reactive components in the concrete and the expansion. The Proportion of the reactive components which provides the highest expansion is called “pessimum rate”. As the ratio of the reactive components is higher or lower than this, the expansion decreases. The pessimum rate is also for the alkaline content of the concrete as in the reactive material in the aggregate [Hobbs, 1988; Ozol, 1975; Poole, 1992; Grattan-Bellew, 2001].

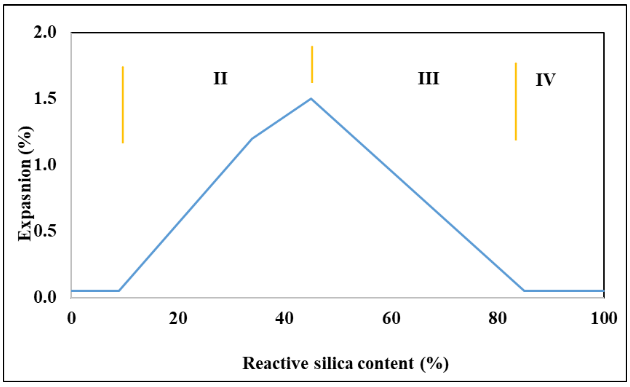


Figure 2.9 Pessimum behaviour divided into four regions [Hobbs, 1988]

Hobbs divided the pessimum behaviour graph into four regions in Figure 2.9 and also particularly described it in Table 2.2 [Hobbs, 1988].

Table 2.2 Effect, explanation of pessimum behaviour regions [Hobbs, 1988]

Region	**Activity**	**Clarification**
I	Reaction starts but cracking does not appear	Gel is not enough to initiate structure damage
II	Reaction continues, if sufficient reactive silica is available, cracking may start	Expansion is the maximum percent if reactive silica is entirely used up Alkali/silica ratio may not affect gel composition.
III	Reaction continues, if sufficient reactive silica is available, cracking may start	Expansion does not continue if alkali level is not enough to keep up the reaction As silica content increase, process of gel expansion starts to decelerate.
IV	Reaction continues but cracking comes to the end	Although reactive silica content is very high and speed of reaction is very fast, gel formation starts to stop due to completion of concrete hardening

Knowing the possible pessimum rate of the reactive aggregates can be determined by the trial mixes prepared at different proportions of the reactive components. In general, it is known that the use of mineral admixture instead of a sufficient portion of the cement is effective in order to reduce ASR expansion in concretes with a pessimum rate. Nevertheless, it is stated that some fly ash can lead to higher expansion when used at a very low rate, especially in place of low-alkaline cement. The reason for this is stated because of the water-soluble alkalis added to the system by the fly ash [Lindgard et al., 2012; Buck & Mather 1987].

2.5.3 Alkali Content of Concrete

Portland cement is clearly the main source of alkaline in concrete. Additionally, water, aggregate, mineral admixtures and chemical admixtures are other important internal sources. There may also be an alkaline input from the external environment to the concrete. The most important external source is chloride based de-icers [Engin, 2015].

Concrete is a basic alkaline material. The main reason for this is cement-based salts. The most important source of alkali for ASR is Na^+ and K^+ ions. The effect of other alkaline ions, on ASR is very low. Therefore, the mass percentage of Na^+ and K^+ ions is used in the calculation of alkali equivalents [Engin, 2015].

$$Na_2O_{eq} = Na_2O + 0.658 * K_2O \qquad \text{Eqn.2.3}$$

where: Na_2O: sodium oxide , K_2O: potassium oxide

Cements with an alkaline content of less than 0.6% are defined as low alkaline cement. This ratio is calculated by Eqn. 2.3. The alkaline content of cement in world generally varies between 0.2 and 1.3 %. Cement with higher alkaline content; 1.65 % or higher may be available in worldwide [Thomas et al., 2013; Engin, 2015].

As the reactive silica dissolves only in the high pH solution, the ASR occurs at a high concentration of hydroxide ions. However, as the reactivity of the aggregate increased, it has been found that even at lower levels of alkalinity, the reaction can occur. Therefore, it is useful to use cement with low alkaline content in case that it is necessary to use suspicious aggregate [Engin, 2015].

2.5.3.1 Pore Solution Composition

Alkalis which are extremely soluble in concrete pore solution can master the concrete pore solution if not they are as high as other oxides in terms of mass percentage. Eqn. 2.4 gives data for calculation of hydroxyl concentration $[OH^-]$ as follows [Helmuth et al., 1993].

$$[OH^-]= 0.0339Na_2O_{eq} / (w/c)+0.022 \pm 0.06 \text{ mol/L} \qquad \text{Eqn.2.4}$$

With the presence of reactive aggregate in mortar/concrete, the alkali-silica reaction occurs and cause the alkali content to decrease the hydroxyl concentration. Alkalinity of the pore solution decreases with time for control concrete due to a possibility of occurrence of a reaction between the inert aggregate (culmination of many aggregates) and alkali hydroxides. On the other hand, this decrease can be considered as very low if it is compared with that in concrete consisting of reactive flint sand. The existence of flint sand cause depletion of alkalis in rather slower than beltane opal does because reach to steady-state matter takes 1 year while that of Beltane opal takes 4 weeks. Nevertheless, the concentration of hydroxyl ion at steady-state is nearly 0.27 mole/l $[OH^-]$, a value which is almost same with in the Beltane opal (0.28 mole/l $[OH^-]$). Diamond defined threshold value of alkali concentration leading to outbreak of alkali silica reaction as 0.25 mol/l or higher [Diamond et al., 1981; Thomas, 1998].

In case of potential of ASR, some European countries and Canada limit the total alkaline content of concrete to 3 kg / m^3. In the UK, mineral and chemical additives, and alkalis from some aggregates and the mixture water are also included to the total alkali content. In Canada, the alkaline content of these materials is not taken into account unless Na_2O_{eq} exceeds 4.5% in fly ash and 1% in the slag. In the United States, it is recommended to use a cement with Na_2O_{eq} content of less than 0.6% instead of limiting the total alkaline content of concrete. However, it has been reported that there is a risk of ASR formation because of the reasons such as alkaline migration, high reactivity aggregate use, alkalis from mineral and chemical additives, mixed water or aggregate in concrete made with low alkaline cement [Farny & Kosmatka, 1997 ; Sibbick & Page, 1991]. It should be taken into account in the calculation of the total reactive alkaline if the amount of alkali from concrete other than cement is over 0.2% kg / m^3. It is also necessary to determine the safe alkaline content for different aggregates [Ramyar, 2013; Andiç-Çakır, 2007].

In addition to the alkalis from the materials forming the concrete, the alkalis that the hardened concrete takes as a result of contact with seawater, some ground water and de-icing salts should also be considered. The amount of alkali absorbed from such sources depends on the permeability of the concrete; the elapsed time concrete is exposed to the water containing alkali and the type of alkali source [Swamy, 1992].

2.3.3.2 Alkali Recycling

In the concrete pore solution, formation of ASR gel ($CaO-Na_2O/K_2O-SiO_2-H_2O$) which also contains little calcium leads to decrease in amount of alkali ions such as calcium and potassium and hydroxyl ions. It is still an accepted fact that some alkaline over time replaces with calcium ions and alkalis are let out back to the pore solution probably containing reactive silica. In 1944. Hansen come up with this subject [Hansen, 1944]. Microanalysis of concrete implemented by electron probe indicated that the gel appeared in inside or near to aggregates had low calcium and high alkali contents but calcium content increased as the formed gel moved away from aggregates, probably because of close commitment with cement paste having lots of calcium [Knudsen & Thaulow, 1979].

As a summary of various studies, it is more logical that gradual transition from ASR gel with low viscosity to calcium-silicate-hydrate (C-S-H) on the occasion of the transfiguration of the gel physically and mechanically. Figure 2.10 explains the alkali recycling schematically in terms of changes in alkali ions, viscosity, water absorption, mechanical strength and structure of gel. In spite of very low viscosity, the gel having a poor calcium contents is liable to swell because of its high capacity. On the contrary, the gel having a rich calcium contents is not liable to swell due to its more rigid structure. Although not clearly proclaimed, the chemical composition of ASR gel provokes damage in cement paste because viscosity and swelling capacity of the gel are adequate to do it [Urban, 1987].

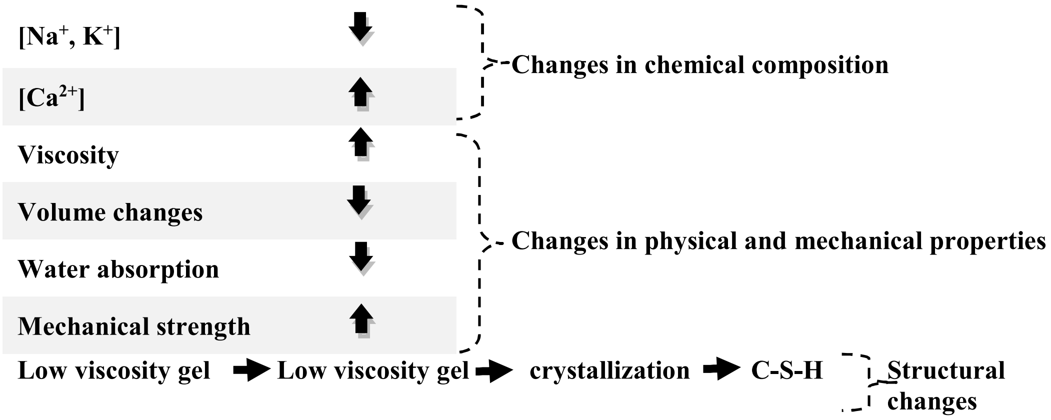


Figure 2.10 Alkali Recycling Process [Urban, 1987]

2.5.4 Moisture Effect

Moisture in concrete structure is important to facilitate the migration of alkali ions into reaction zones and to provide expansion of the formed ASR gel. Accordingly, in order to prevent moisture and alkaline-containing solutions from entering the concrete, reducing w/c ratio and applying a proper curing process in sufficient time can be considered as the simplest way to reduce the permeability of concrete. However, this way of permeability reduction may not be advantageous for ASR because the low water content causes the pH of the pore solution to be high and also the ASR expansion formed in a concrete with a low pore ratio creates more stress. Reducing permeability by using mineral admixtures may be the best way to prevent the negative effects mentioned above [Ramyar, 2013].

The water content of the structures affected by the ASR is generally expressed by the relative humidity (RH), which reflects the thermodynamic state of the pore solution. It is known that this value is difficult to measure in structures and the results are not very reliable. Nevertheless, with a number of other factors, it has been reported that the critical RH for the formation of ASR in the concrete is between 80-90% [Larive et al., 2013]. Local weather conditions in terms of moisture contents cause damage from ASR in different intensities even in same concrete structure [Thomas et al., 2013]. Coating the concrete element in contact with the humid environment may reduce ASR expansion.

Surface coatings are also used for repair purposes to stop the reaction's progress after the start of the reaction damage [Stark et al., 1993].

2.3.5 Temperature Effect

Like in most chemical reactions, the alkali silica reaction accelerates with increasing temperature. In addition, it is known that some aggregates which are reactive at normal temperature such as flint cause gel formation at high temperatures. Moreover, the temperature affects the water absorption of the gel which is the second stage of the reaction, causing the micro-cracks to occur, and subsequent damage to the concrete. Diamond observed that at higher temperature, the reaction and expansion started earlier and continued fast and both the speed of the reaction and the expansion decreased with time but that the reaction progressed slowly at low temperature and the expansion gradually approached or exceeded the level seen at higher temperature over time [Diamond, 1981].

Different explanations have been introduced for these observations. Slower expansion process at low temperature is attributed to slower reaction and slower migration of alkalis to the reaction site. It is also known that the maximum expansion pressure occurs when the gel has a certain water content. It is explained that the gel becomes progressively liquid, leaks into cracks and reduces pressure formed by it after this level. More expansion may occur as the time to reach maximum expansion pressure for the gel extends at low temperature. An alternative hypothesis is that a gel of different structure or composition forms at different temperatures and that the gel causing more expansion pressure forms at low temperature [Swamy, 1992].

2.5.6 Other Effectual Factors

Factors such as permeability of concrete, external alkalis, de-icer salts and steam curing also affect ASR [Ramyar, 2013].

Some studies have emphasized that the moisture and alkaline movements gets harder in concretes with reduced permeability by some precautions such as low w/c ratio, usage of mineral admixtures etc. Therefore, the ASR expansion decreases [Farny & Kosmatka, 1997; Stark, 1995].

The alkalis, in the de-icer salts, seawater, groundwater and industrial wastewaters can also increase the ASR expansion in especially cracked or permeable concretes. Protecting against these alkalis needs preventing the ingress of alkali by reducing permeability of concrete or using protective coatings [Farny & Kosmatka, 1997].

Sibbick and Page have claimed that the ASR damage, which may occur in concrete in contact with salty water, is higher than expected, and in this case, being at the limit of 3 kg / m^3 Na_2Oeq for total alkaline content of the concrete is not protective way [Grattan-Bellew, 1994].

More expansion was observed in the mortars exposed to steam cure with extra alkaline and gypsum containing reactive aggregate as compared to those without gypsum [Shayan & Ivanusec, 1996]. The researchers have reported that this effect occurs because of delayed ettringite form (DEF). The same experiment showed that the formation of DEF alone did not create the same damage when done by using a non-reactive aggregate. Moreover, that the ASR expansion, occurred as steam cure was applied, caused greater damage by being joined to DEF expansion [Ramyar, 2013].

Among other factors, chloride based de-icers (de-icer salts) will be mentioned in detail in the following part because they were used in new method trials as a content of solution in this experimental study.

2.5.6.1 Effect of Chloride Based De-icers (de-icer salts)

As known, the most important drawback of chloride-based de-icers in concrete structure is to lead reinforcement to corrosion. The grade of free chlorides is connected with chloride dosage and cation types and chlorides can also be in concrete as bound to hydration product [Al-Hussaini et al., 1990]. Some experimental studies have revealed that chloride based de-icers (especially alkali salts) generally provoke alkali silica reaction. These alkali salts like as NaCl, $CaCl_2$. KCl may bring about more expansion in concrete structure than that with alkali hydroxides [Chatterjiet et al., 1987]. Sirivivatnanon has stated divalent or cations like as calcium (Ca^{2+}) don't cause expansion in concrete as much as monovalent cations like as sodium and potassium (Na^+, K^+) do [Sirivivatnanon et al, 1987]. Soak solution with chloride based salts cause chloroaluminates formation in mortar or concrete. Expansion percent in mortar or concrete specimens exposed to de-icer salt solution are affected by concentration of these solutions and storage conditions such as relative humidity, temperature etc. [Desai, 2010].

a) Sodium chloride (NaCl)

Comprehensive study has been carried out on what extent sodium chloride (NaCl) affects ASR in mortar or concrete. Equation 2.5 explain that chloroaluminate form due to replacement of SO_4^{-2} ions from ettringite by Cl- from NaCl [Desai, 2010].

$$2\,NaCl + Ca(OH)_2 \rightarrow 2\,NaOH + CaCl_2 \qquad \text{Eqn. 2.5}$$

$$3CaO.Al_2O_3.3CaSO_4.32H_2O + CaCl_2 \rightarrow 3CaO.Al_2O_3.3CaCl_2.32H_2O + 3CaSO_4$$

Sodium chloride (NaCl) leads concentration of hydroxyl ion to rise in pore solution because chloride may be forced to penetrate into ASR products [Kawamura&Ichise, 1990; Kawamura&Komatsu, 1997]. In mortar specimens, consisting of reactive aggregate exposed to soak solution with sodium chloride (NaCl) as an extra chemical material, concentration of hydroxyl (OH^-) ion decrease slightly in pore solution of theirs and concentration of Cl^- ion decrease significantly at reaction period of 12-24 hours. On the other hand, concentration of hydroxyl (OH^-) and concentration of Cl^- ion decrease little at the same period in the pore solution of samples including a non-reactive aggregate. In summary, Kawamura expressed chloride ions as a probable reason for flash expansion in early period of ASR [Kawamura & Ichise, 1990].

b) Calcium chloride ($CaCl_2$)

Calcium hydroxide ($Ca(OH)_2$) in free state and Calcium chloride ($CaCl_2$) in a high concentration may trigger concrete structure to deteriorate [Barsione, 1984]. Chemical reactions in mortar or concrete cause the formation of various component groups such as calcium chloride hydrates ($3CaO.CaCl_2.12H_2O$, $3CaO.CaCl_2.2H_2O$) and chloroaluminates ($3CaO.Al_2O_3.3CaCl_2.32H_2O$) which may be considered as sources of existence of chloride in pore solution. Moreover, another component named as calcium silicate hydrate (CSH) absorbs Cl^- ions [Desai, 2010; Chatterji, 1978]. Many experimental studies have shown that mortar or concrete specimens exposed to soak solution with calcium chloride ($CaCl_2$) does not expands as much as those exposed to soak solution with sodium chloride (NaCl) [Kawamura & Ichise, 1990; Prezzi et al., 1998].

c) Potassium chloride (KCl)

Potassium chloride and calcium chloride, which are de-icer salts with monovalent cations (Na^+, K^+) affects the mechanism of ASR in pretty much the same behaviour [Desai, 2010; Chatterji et al., 1987].

d) Magnesium chloride ($MgCl_2$)

Desai stated that magnesium chloride ($MgCl_2$) with divalent cation (Mg^{2+}) does not have significant effect on ASR expansion. However, the reaction between magnesium chloride ($MgCl_2$) and calcium silicate hydrate (CSH), cementitious material, produces magnesium silicate hydrate, non-cementitious so it can damage concrete structure [Desai, 2010].

2.6 Composition of ASR Products

The ASR composition and texture vary according to the composition of the pore solution, the type of reactive silica, the reaction temperature and the density of the reacted products. It is also known that the feature in question of these products change with the time and position in the concrete [Thaulow et al., 1996; Kawamura et al., 1998]. The gel in the cracks of the reacted concrete is usually transparent and resinous. A significant difference between the viscosities of the gel samples can be also found. Some gels are so liquid that it can fill the pores of concrete completely or partially by flowing through the cracks. In general, the gels become white and dry state over time by being carbonized due to their contact with air [Poole, 1992].

The knowledge about the composition and morphology of ASR products has increased with the use of scanning electron microscopy (SEM). These investigations have shown that the gel contains different proportions of silica and less but still different amounts of calcium and alkali. It is known that the K_2O / Na_2O ratio of the clinker varies from 1 to 3 except for those containing very low alkali, and this is reflected in the gel composition. It is explained that this ratio and the relative amount of calcium and alkaline changes in the concretes exposed to the effects of external alkalis such as seawater and laboratory test environment, Na_2O or K_2O or deicer salts added to the concrete mixture to accelerate the reaction [Lindgard et al., 2012; Brouxel,1993].

The formation of ASR products is known to be affected by reactivity potential, speed and being in pessimum rate of the aggregate in addition to petrographic properties, structure and texture of its [Lu et al., 2006; Diamond & Thaulow, 1974]. However, it is also reported that aggregate properties are not the only factor. Quite different gels having various calcium content were identified in unlike or equivalent positions of the same sample [Lindgard et al., 2012; Peterson et al., 2006; Fernades, 2009].

Calcium content was found in the cracks in the gel away from the coarse aggregate due to the replacement of the alkalis with calcium. It was determined that the expansion was due to the calcium content of the gel rather than the degree of the reaction or the amount of gel formed [Lindgard, 2012; Kawamura et al., 1998; Kundsen and Thaulow, 1975].

In a study based on visual inspection of the ASR gel, amorphous gels were found in the cracks and air voids in the cement paste. ASR products having rosaceous crystalline structure were identified in the coarse aggregate grains [Thaulow et al., 1996]. Moranville-Regourd classified the reaction products in two ways: gels and crystals and described the gels as solid (dense) or spongy looking [Moranville-Regourd, 1989]. Variability in the appearance of the gel may be due to the type or amount of shrinkage during drying in SEM, as well as gels in different compositions.

Typical morphologies of the crystalline products such as layered or rosaceous, needle or rod-shaped and leafy were seen [Andiç-Çakır, 2007]. ASR products with different morphology and structure are shown in Figure 2.12.

The reaction products produced resulting from accelerated mortar bar test and the ASR products seen in the actual structures are described as similar in terms of morphological In general, the amorphous gel is considered to crystallize over time. The following mechanisms have been proposed that describe the crystallization of the gel. Some researchers have stated that the gel is crystallized owing to drying [Cole & Lancucki, 1983]. Thordal and Thaulow stated that crystallized gels are generally found in aggregates and less frequently in cracks in cement paste [Thaulow et al., 1996; Thordal & Thaulow, 1990].

The chemical composition difference between the crystalline products and those in amorphous structure may be due to the chemical variation of the water between pore solution of cement paste and aggregate cavities [Ramyar, 2013]. Kurtis et al. declared that alkali silica gel becomes crystallized in calcium and sodium hydroxide solutions, ASR products formed in concrete are generally amorphous and crystalline gel is less common [Kurtis et al., 1997; Kurtis et al., 1998]. Peterson et al. found an excess of crystallized ASR product in the concrete samples of a building constructed in 1890s [Peterson et al., 2006]. Results obtained from the investigations, show that the density of sodium and potassium ion in amorphous gel is less than that of crystallized product but calcium ion density is higher. Furthermore, it has been emphasized that amorphous gels have a more variable composition than crystal products. The age and excess of crystal product of examined concrete signal that crystallization is a formation occurring with time [Ramyar, 2013].

Mostly, investigations using EDS (Energy Dispersive Spectrometry) on completely dried samples has shown that the gel composition varies over a wide range. This difference is due to the fact that some gel samples are exposed to carbonation before inspection. Moreover, the other reason for difference is considered as that the normalization of the elements corrected by modern computer and determined by microanalysis to 100% and that the elements with atomic number less than 11 cannot be determined by this microanalysis [Poole, 1992].

Although the chemical composition of ASR products is different, it can be simply explained in three classes; (1) silica partially reacted and swollen (2) comparatively pure alkaline silicate solutions or gels containing various alkali and water content (3) two-component simple mixtures in almost constant composition (excluding water content) [Ramyar, 2013]. Kirkpatrick's research confirms this possibility and claims that alkali silica gel is a mixture of phases of alkali-silicate-hydrate and calcium-silicate-hydrate [Kirkpatrick, 1991]. Furthermore, it is emphasized that the reaction zone around the beltane opal particles is composed of sodium silicate and sodium calcium silicate [Gutteridge & Hobbs, 1980].

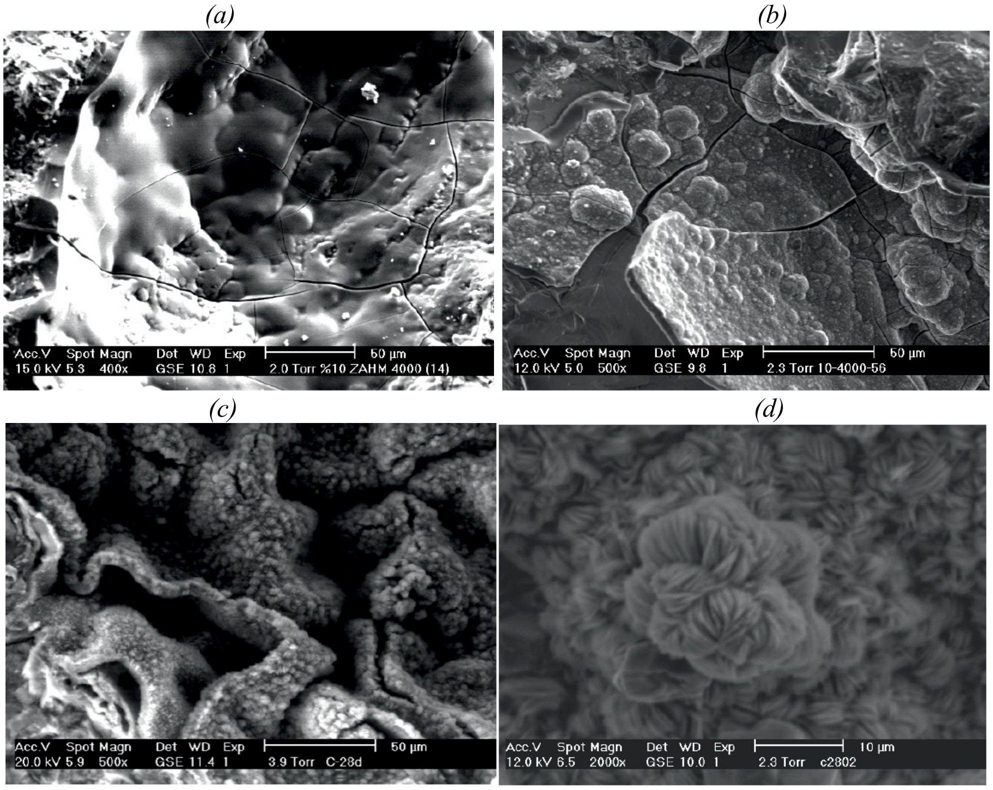


Figure 2.11 (a) Solid ASR product with 400 times magnification (b) Semi-crystalline cracked ASR product with 500 times magnification (c) Rosaceous ASR product with 2000 times magnification (d) Rosaceous ASR product forming in layers with 500 times magnification [Andiç-Çakır, 2007]

2.7 Diagnosis of ASR Damage

ASR expansion creates problems in terms of structural and usage in concrete structures due to cracks formed by it. Conventional diagnostic methods are based on the determination of cracks on the surface and determination of drilling core strength taken from damaged concrete [Ramyar, 2013]. However, damage caused by ASR in concrete affects the modulus of elasticity and tensile strength (both flexural and direct tensile strength) rather than concrete compressive strength [Hafçı, 2013]. ASR damage can also be detected by petrographic analysis of concrete. In this method, reactive aggregate and ASR products can be determined by examining the concrete sections with a microscope. However, this method may not give precise opinion about the extent of the damage. As clearly seen in Figure 2.12 (a), the ASR gel may be present in or around the aggregate. The expansion, cracks, superficial residues, fragmentation and colour change in the concrete can be taken into consideration in order to detect ASR damage [Ramyar, 2013].

Expansion and cracking, superficial residues, fragmentation and displacement in concrete structure due to ASR are useful for diagnosis of ASR damage.

2.7.1 Expansion and Cracking

The ASR expansion begins with gel formation in the reactive aggregate or on the surface and absorption of water by the gel. The gel absorbing water forms a pressure stress of approximately 10 MPa in each direction. As a result of the pressure, cracks in the cement paste surrounding the aggregate, which corresponds to the volume increase, occur and generally make an angle of 120 ° with each other.

These cracks spread by starting from aggregate in star-shaped with three-four-arms. Common type of map-cracking is observed in unstrained and non-reinforced concrete due to the joining of ASR cracks (Figure 2.12 (b)) [Figg, 1987]. In strained structures, cracks are directed to the direction of stress [Poole, 1992]. ASR cracks are formed in this direction because the reinforcement is parallel to the main stress. Nevertheless, unlike corrosion cracks, these cracks form between the reinforcement and not on the reinforcement. Cracks in a bridge pier, which are formed by the effect of load and ASR, appear in Figure 2.12 (c). As for concrete structure like as slab having evenly distributed reinforcement, map-cracking in rectangular-shaped was observed as in Figure 2.12 (d) [Swamy, 1992; Figg, 1987].

Hobbs expressed that macro-cracks occur at a width of 0.1 mm to 10 mm and a depth of 25-50 mm on the surface of the concrete elements affected by ASR [Hobbs, 1988]. In further process of the ASR damage, closure and cover throw of the joint, and regional displacement of some parts of the concrete elements were also observed [Ramyar, 2013].

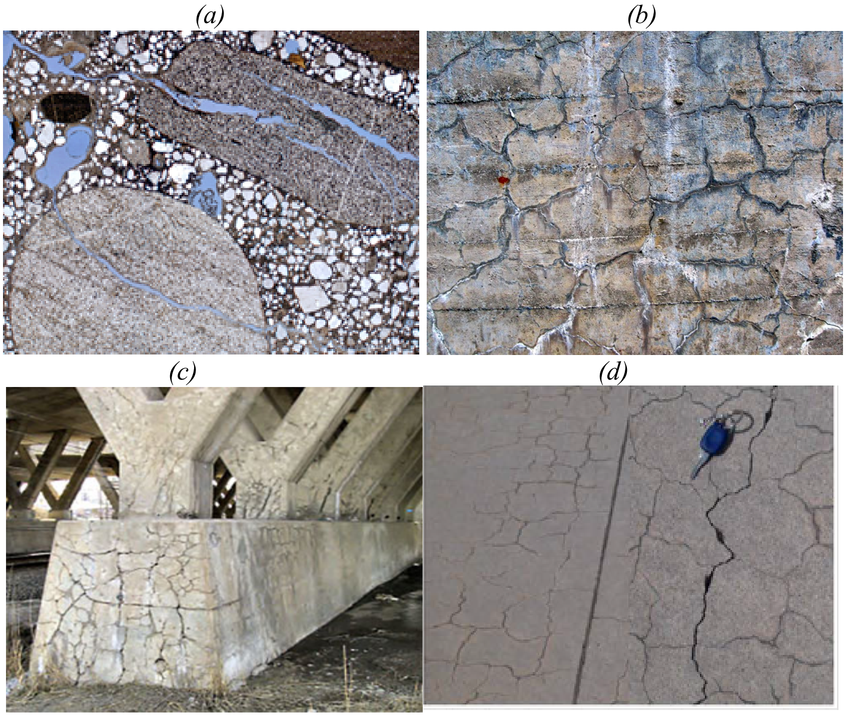


Figure 2.12 (a) ASR gel with blue colour (b) Common type of map-cracking from ASR [Engin, 2015] (c) ASR crack on a bridge pier [FHWA, 2010] (d) ASR cracks on the path and taxiway of İzmir Airport [Andiç-Çakır, 2007]

2.7.2 Superficial Residues, Fragmentation and Displacement

If the ASR gel is in low viscosity, it accumulates in the voids in the concrete surface and according as moisture condition; it creates an aqueous area and gains a white by slowly carbonating. Moisture stains and traces observed in snail-shaped, can be also used in the diagnosis of ASR symptoms [Palmer, 1997]. In other cases, ASR gel or calcium carbonate can be seen in cracks. They leave residues with different colours varying from white to dark grey. These residues may be useful to diagnose ASR damage [Farny & Kosmatka, 1997]. Fragmentation is the removal of the cone-shaped part from the surface because of expansion of the gel or aggregate part close to the concrete surface [Poole, 1992; Palmer, 1988]. Fragmentation is generally seen in the concrete containing very reactive aggregate or that exposed to stream cure [Figg, 1987].

ASR does not always occur at the same intensity all over the structure. Therefore, some elements in the structure may displace relative to the other. For example, it is seen in Figure 2.13 that one of the concrete elements displaces 50 mm relative to the other in a dam parapet [Poole, 1992].

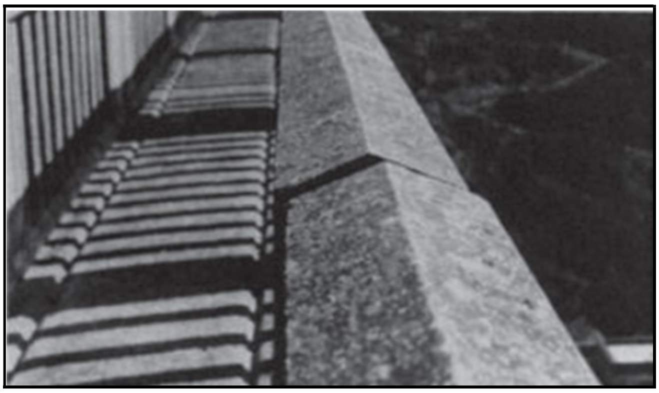


Figure 2.13 Parapet of Val de Mare Dam, Jersey [Poole, 1992]

2.8 Mitigation Measures of ASR Effect

Many studies have carried out to find methods for minimizing ASR effect on concrete structure. In general, the strategies for mitigation of ASR effect are counted as follows:

i. Aggregate should be used according to petrographic analysis results. In suspicious case, the aggregate should be considered as deleterious and necessary measures should be taken.
ii. If that the aggregates are deleterious or suspicious is known, cement with low alkali content should be preferred.
iii. Pozzolans such as fly ash, blast furnace slag and silica fume etc. should be used to reduce the alkali content and increase the durability of the concrete.
iv. Water/cement ratio should be low.

v. The aggregate known as potentially reactive or suspicious should be mixed in concrete with a non-reactive aggregate.

vi. The inhibitors of alkali silica reaction should be utilized [Engin, 2015].

CHAPTER 3

LITERATURE REVIEW

Many studies have been carried out on available AAR test methods. Moreover, new method developments have been improved by only a few researchers. In this experimental study, a number of test variables was studied to find the most compatible test method with accepted field performance by trying different soak solution, storage temperature, mould dimension and cement type. The literature review consists of (1) the development of available and standard test methods, (2) designed new methods to improve reliability for alkali-silica reactivity of aggregates, (3) comparison of laboratory performance tests for ASR, (4) effect of petrographic properties of aggregates on alkali-silica reactivity, (5) influence of chloride based de-icers on the process of alkali-silica reaction

3.1 Development of Available Test Methods to Detect AAR

Laboratory methods for the detection of AAR include the following objectives; (1) to determine whether the aggregate type is a potential reactive, (2) to determine the reactivity of the cement-aggregate combination in concrete and whether the aggregate has a slow / late expansion type as the reactivity is triggered, (3) to determine the maximum expansion of concrete over time regarding as certain environmental conditions and mixing ratios [Farny & Kosmatka, 1997].

The process of ASR expansion occur for a long time (decades) in normal concrete exposed to normal curing condition. Therefore, the reaction was accelerated in a variety of ways as designing laboratory test methods measuring ASR expansions on mortar and concrete samples. One or more of the following methods are applied together to accelerate the reaction;

i. To increase the alkali concentration.
ii. To use of silica having high-reactivity potential.
iii. To apply higher temperature.

The results obtained from applied test methods may give uncertain data if test conditions move away from real environmental conditions [Thomas & Innis, 1999].

The history of standard, modified and specially developed test methods by researchers to detect alkali reactivity of aggregates will be explained together as below chronologically.

3.1.1 Stanton Method (1940)

Stanton was known as the first person to introduce alkali silica reaction to knowledge in 1940. He first started to research on ASR and test methods to detect potential reactivity of aggregates in 1938. He worked for the sake of this for five years. For designing the test method, Stanton tried various specimens in different geometries such as mortar bars, 1 in. x 1 in. x 10 in., mortar cylinders, 4 in (long) x 2 in (diameter), concrete prisms, 2 in. x 2 in. x 11.25 in. and different aggregate combinations in proportion and maximum aggregate size such as a sample containing % 50 fine aggregate (sand) and 50 % coarse aggregate with 0.75 in. aggregate in maximum size. This test method was applied by processes of wetting, drying in both continuously and alternately in room conditions in terms of temperature and relative humidity. The specimens were cured at storage container with water and air for a duration varying from 80 days to 5 years. As conditioning the test specimens, heating and cooling cycle was performed on them at temperature ranging from nearly 21 ºC to 66 ºC. After applying test procedures, he concluded that some mineral constituents like as cherts, limestones in impure form and shales could lead concrete to expand deleteriously if cement used in concrete had sufficient alkali content. The expansion was not observed in concrete containing cement with lesser than 0.6% of alkali content even though concrete was prepared with reactive aggregates. Moreover, usage of pozzolanic material was determined as a preventive measure to minimize deleterious effect of ASR. In one respect, this first ASR test method pioneered the creation of a basic logic for ASTM C 227 standard test method for cement-aggregate combinations [Stanton, 1940; Latifee, 2013].

3.1.2 ASTM C227 / Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (1950)

ASTM C227 was first introduced in 1950 referring to Stanton method (1940) and revised lastly in 2010 but withdrawn in 2018. This test method is based on the measurement of expansion of mortar bars produced by aggregate which would be tested in views of alkali silica reactivity. Aggregates are brought to the specified gradation in the standard; if necessary, crushing process is also applied. The cement to be used in the field or the reference cement with an equivalent alkali content of 0.6 % Na_2O_{eq} may be used. The cement, whose alkali content is usually 1.0 to 1.2 % Na_2O_{eq}, is used. In this experiment, concrete prismatic moulds, 25 mm x 25 mm x 285 mm in size, is discharged with concrete mix having aggregate in the specified gradation and aggregate/cement ratio 2.25. Four mortar samples are at least prepared. Mortar bar samples are cured at 38 °C, on water, at 100% relative humidity. The length measurements are recorded with a precision of 0.002 mm for a period of 12 months or more starting from 14 days involving extra measurement in 1,2,3,4,6 and 9 months. According to ASTM C33 standard, for non-reactive aggregates, the expansion should be lesser than 0.10% at six months and 0.05% at three months. Longer expansions should also be recorded to understand the differences in aggregate reactivity [Farny & Kosmatka, 1997]. If there is no result for 6 months, expansion limit for 3 months can be taken as the basis. it is recommended that additional experiments should be performed for the final assessment in case of that the 6-month expansions exceed the limit value [Andiç-Çakır, 2007].

3.1.3 ASTM C289. Quick Chemical Method (1952)

ASTM C289 was first applied in 1952 but withdrawn in 2016**.** This method was improved by hardworks of Mielenz [Mielenz et al., 1950; Mielenz et al., 1958]. It was used to estimate the potential reactivity of siliceous aggregates. ASTM C289 test method gave compatible results with petrographic properties and field performance of aggregates in a certain extent.

Other chemical methods are listed below with the countries in which they are used (or developed):

i. Weight loss method (Germany)
ii. Gel pat method (UK)
iii. Osmotic cell experiment (USA)
iv. Chemical shrinkage method (Denmark) [Andiç-Çakır, 2007].

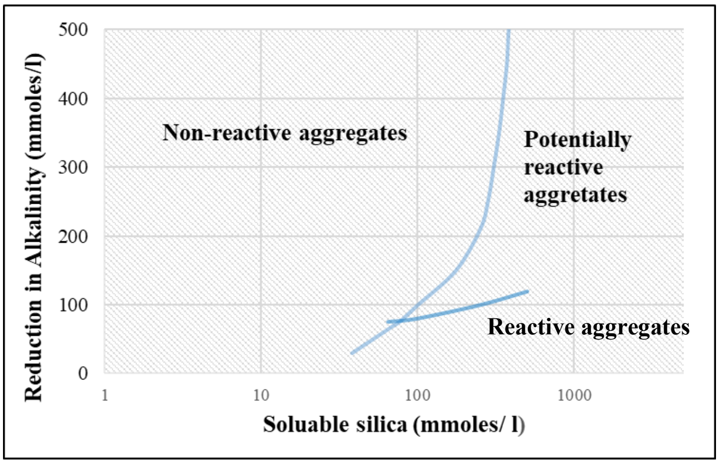


Figure 3.1 Graph of reduction in alkalinity quantity versus dissolved silica (ASTM C289)

In the ASTM C289 chemical method, three samples, each of 25 g., are prepared by crushing and sieving aggregate. This material is reacted in an alkaline solution (1N NaOH solution). After 24 hours, the reduction in the alkalinity of the solution with dissolved silica within the aggregate is determined. These values are marked on the graph shown in Figure 3.1. It is determined that the aggregate is harmful, potentially harmful or harmless to the region [Farny & Kosmatka, 1997].

This method is reliable for very reactive aggregates; however, it may give false results for slow reactive aggregates. Some aggregates may exhibit low expansion in concrete in spite of having a high amount of soluble silica. Therefore, the test method does not always give reliable results [Stark et al., 1993]. This test is convenient and quick to classify aggregates, but the results need to be verified by other test methods [ACI Committee 221, 1998].

3.1.4 The Conrow Test, Standard Test Method for Potential Volume Change of Cement-Aggregate Combination (1952)

ASTM C 342 (withdrawn in 2001) described as the conrow test method is primarily a modified mortar bar method to detect potential reactivity of aggregate for ASR [Conrow, 1952]. In test procedure, mortar bars are exposed to various conditions in terms of temperature and moisture. Measurements for expansion change are performed at regular intervals for one year. This test method does not define an expansion limit but ASTM subcommittee work has stated the expansion limit as 0.020 % for one year [Latifee, 2013].

3.1.5 ASTM C295 Petrographic Examination (1954)

ASTM C295 was first validated in 1954. ASTM C295 / C295M - 18a Standard Guide for Petrographic Examination of Aggregates for Concrete has been approved as the last version in 2018. RILEM AAR-1 and BS 812: part 104 are seen as equivalent standards of its [Latifee, E, 2013]. Evaluation of aggregate can be done by macroscopic or microscopic methods. In macroscopic analysis, aggregate sample is divided into groups and similar rock types are classified. Microscopic analysis is a more extensive and time-consuming procedure. Analysis is thus usually carried out with fewer aggregate samples. Petrographic microscopes are mostly used and thin sections of aggregates are examined under polarized light. The refractive index and specific gravity of the powdered aggregate sample are also evaluated [Farny & Kosmatka, 1997].

Petrographic microscopy has many different functions. An experienced petrograph using a petrographic microscope can examine the sample under flat, diagonal and polarized light. After this, he/she determines the location of hazardous pieces and whether alteration occurs or not [Swamy, 1992]. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) can also be used for the characterization of the mineral composition of the aggregate sample. Analytical techniques such as x-ray diffraction and infrared spectrography are also applied to characterize the crystal structure and texture of silica within the aggregate [Andiç-Çakır, 2007].

Since petrographic analysis of the aggregate is time consuming, small samples are studied. For this reason, the selected sample must represent the aggregate. It is useful to evaluate the relationship between the results of petrographic analysis and the records of the aggregate maintained during the service life of the aggregate. The results of petrographic analysis cannot give an idea about whether aggregate will show harmful expansion in concrete. Other test methods should be applied to determine this [Farny & Kosmatka, 1997].

The specifications, such as the British Highways Specification, specify undesirable phases in the aggregate to control the alkali-silica reaction. Accordingly, aggregates may be accepted as harmless if 95% and more of the fine and coarse aggregates are composed of aggregates of non-reactive type; if they are not contaminated by reactive silica minerals such as opal, tridimite and cristobalite and also if they do not contain more than 2% of the total cherry, flint or chalcedony. Because of the pessimum rate, if the total content of chert or flint in the aggregate is more than 60% by mass and does not contain the aforementioned siliceous minerals, it may be still considered to be harmless. Quartz should not contain quartzite and more than 30% of quartz undergoing metamorphism by mass [Swamy, 1992].

These techniques are very useful in determining the mineralogical structure of aggregate and the amount of these minerals in its. Nevertheless, it is important to note that none of these techniques can accurately determine whether ASR damage will occur. In addition to environmental impacts, factors such as bulk density, porosity, particle size distribution, amount of reactive particles, aggregate shape and roughness play an important role in ASR expansion. Therefore, it should be noted that petrographic analysis is always the first step in determining the suitability of concrete aggregates.

3.1.6 ASTM C1293 Concrete Prism Test (CPT), (1950s)

Although the basic framework of this test had been laid in the 1950s by Swenson and Gillott, the first generation was presented in 1973 in Canada [Swenson&Gillott, 1964]. The aim of this experiment is to obtain an idea about the alkali silica reactivity of aggregates by means of measurement of the length change of concrete prisms affected by ASR. It is recommended to obtain information on the petrography of aggregates before the application of the test.

The aggregate to be applied to the test is prepared by mixing the fine aggregate with the non-reactive coarse aggregate. If the reactivity of the coarse aggregate is measured, the material is mixed with non-reactive fine aggregate. After that, it is brought to a certain gradation and poured into 75 mm × 75 mm × 285 mm moulds. The cement content of the samples is 420 ± 10 kg / m^3 and the water / cement ratio should be adjusted to provide workability between 0.42 and 0.45. The equivalent Na_2O_{eq} content of the cement used is increased to 1.25% by mass by adding NaOH to the mixture water. After 24 hours, the first length measurements of the moulded samples are taken and they are stored at a temperature of 38 ° C, on water (in a humid environment) as specified in the standard. Length measurements are periodically measured. The use of roving in the storage container is also available in this method.

Expansion limit is determined as 0.04% by taking average of three samples at the end of one year period. On the other hand, expansion limit for material combination is 0.04% for two years. CPT is generally applied as an extra to other test methods. Both fine and coarse aggregates can be tested by this test method. RILEM AAR-3, CSA A23.2-14A (Canadian) and BS 812: Part 123 (British) test methods are equivalent to ASTM C 1293.

This method has two main drawbacks, which will be mentioned as follow:

1) Time consuming: Concrete prism test lasts 1 or 2 years varying by the goal of the test application. This long-term test method is not practical at all for detecting aggregates to be used in specific construction planned to be completed in a short period.
2) Alkali Leaching: The samples are not completely stored at water or soak solution so convective air current appears within storage container, which can cause leaching of alkali from concrete prism. Therefore, less expansion may be observed in concrete prisms exposed to CPT method as compared to concrete blocks exposed to storage conditions having same alkali content. The research made by Thomas et al revealed that nearly 20% of alkalis drain from the concrete prisms within 90 days and even 35% of those drain from prisms within 1 year [Thomas et al., 2005].

3.1.7 Osmotic Cell Test (1955)

Osmotic cell test was improved in 1955 to detect both chemical and physical impacts of alkali silica reaction [Verbcek&Gramlich, 1955]. This method utilizes only inflatable pressure with dissolving by passing over the mechanic of mixing material impacts that make test data of mortar or concrete incomprehensible. A distinct work of particular versions, inspection and direct monitoring of some physical and chemical views of ASR mechanism have been applied by osmotic cell test [Latifee, E, 2013]. Stark developed this method to assess aggregates in terms of alkali silica reactivity [Stark, 1983]. However, this method has an important drawbacks that ASR gel may not appear as osmotic cells forms really [Latifee, 2013].

3.1.8 Gel Pat Test (1958)

Qualitative analysis is performed to detect ASR by the gel pat method that introduced first in England in 1958. In this test, after curing, small parts of aggregates are poured into cement paste pats that are granulated to bright to light the surface of tested aggregate. Mortar specimens consisting of aggregates with flat, cut surface, are stored in the alkali soak solution for 3 days. In case of that the aggregate tested is determined as reactive, ASR gel appears throughout its, so the amount of reactive components by percent can be predicted. Although the gel pat test is an easy way to observe potential reactivity of aggregates, it does not numeric data for degree of reactivity, so the gel pat test is not accepted as a standard test method [Berube & Fournier, 1993; Latifee, 2013].

3.1.9 Rock cylinder method /ASTM C 586 (1966)

Hadley's work had been a pioneer in the emergence of this method before approval of original form of ASTM C 586 in 1966 [Hadley, 1964]. Rock Cylinder Method is a test method that measures the expansion characteristics of carbonated rocks. An example of a small rock cylinder 35 mm in length and 9 mm in diameter is immersed in an alkali solution (1N NaOH) at room temperature. For example, length expansions of the rock cylinder are measured for a period of one year or more. Generally, the expansion tendency reveals within a month. Specimens with a 28-day expansion of 0.10% or more may show harmful expansion under normal service conditions. Some researchers have suggested a 0.20% expansion limit for 16 weeks consider the fact that the rock cylinder shrinkages before the expansion [Newlon et al., 1974; Ryell et al., 1974; Ozol, 1975].

This test method has many disadvantages. It is notably difficult to obtain a sample representing the rock and the duration of the experiment is long. Variations in the nature of the rock, such as whether free expansion is allowed in the experiment (expansion of the concrete is restricted) affect the experimental results. The expansion character of the aggregate can be estimated by this method. However, ASTM C 586 test is not an experimental method that can decide on the usability of the aggregate alone since the expansion of the concrete is dependent on other factors such as the water cement ratio, the content of the binding material and the alkalinity of the concrete [Farny & Kosmatka, 1997; Ozol, 1994].

3.1.10 Concrete Cube Test (1973)

The concrete cube test was introduced by Dahms and Bonzel in 1973. It is named as the Dahms cube test which is a qualitative way to assess aggregates for ASR [Dahms, 1977]. In this test method, aggregate to be test is mixed in concrete specimens, 300 mm cube, to detect for ASR in terms of observing cracking and gel leakage from concrete structure. The cube specimens are exposed to 95% moisture in a humid room with 40 ºC temperature. Moreover, mixed materials are poured into 100 mm cube moulds and moulded specimens are stored at 65% RH and 20 ºC but not immersed completely in water. The Dahms cube test is not accepted as a standard test method for ASR due to absence of a quantitative result [Latifee, 2013].

3.1.11 Nordtest Accelerated Alkali-Silica Reactivity Test/ Building Method 295 (1978)

Jensen and Chatterji designed Nordest Building Method 295 as known also "saturated NaCl bath method", by modifying and accelerating available mortar-bar tests in 1978. This test method is only used to assess reactive fine aggregates in Denmark and not used another country. In first part of test process, three mortar prisms in 40 mm x 40 mm x 160 mm dimension are stored in water for 27 days. Prismatic mortar bars which have larger and shorter dimensions than those of ASTM samples are prepared by proportions as 1:3 cement/aggregate ratio and 0.5 water/cement ratio. These mortar bars are cured in water for 4-5 weeks and after curing, initial length measurement is taken and they are exposed to saturated NaCl solution at 50 °C [Chatterji, 1978].

3.1.12 Japanese Industrial Standard (JIS)

Japanese Industrial Standard (JIS A1146) is a kind of mortar test method and its last revision was published in 2017. Mortar bars are prepared by proportions as 1:2 of cement/aggregate ratio and 0.5 of water/cement ratio and supplying NaOH to regulate cement alkali content as 1.2%. Initial measurement is taken 1 day later after preparing the mortar bar specimens and the specimens are exposed to 100% RH at 40 ºC in a sealed container for 6 months. The length measurement are periodically taken for each week.

3.1.13 Chinese Autoclave Test method (1983)

Tang et al. committed Chinese autoclave test method in 1983. The test specimens are prepared by 10 mm x 10 mm x 40 mm bars and are stored at 150 ºC so the test can be completed in a very short period, 1 day. This short period is seen an advantage, but non-reactive aggregate cannot be assessed by Chinese autoclave test method. Expansion limit in this method is described as 0.10 percent for 6 hours [Tang et al., 1983].

3.1.14 ASTM C 1260 Accelerated mortar bar test (1986)

Oberholster and Davies were first people to proposed AMBT (Accelerated mortar bar test) by modifying ASTM C227 in 1986. 25 mm x 25 mm x 285 mm mortar bars are used in this method while 50mm x 50mm x 285mm concrete prisms are used in ASTM C227 [Oberholster&Davies, 1986]. The test method is based on the principle of measuring the length change of mortar samples stored at high temperature and in a high alkaline solution [Farny & Kosmatka, 1997]. First approval and last revision dates of ASTM C1260 as a standard are 1989 and 2014 in turn.

This test measures the potential reactivity of the aggregate instead of behaviour of certain cement-aggregate combinations. This method accelerates the reaction by making the condition of curing more excessive and give opportunity for determining reactivity of the aggregates within 16 days. As stated in other methods, it is recommended that the petrographic analysis of the aggregates should be performed and that whether the expansion is due to ASR or not should be examined if the expansion occurs above the limits before the applying the test [Andiç-Çakır, 2007]. The mass of aggregate specified in the standard is 2.25 times that of the cement, and the mortar mixture with a water / cement ratio of 0.47 is prepared.

25 mm × 25 mm × 285 mm mortar samples poured into moulds after 24 hours are extracted from the moulds taken and the lengths of specimens are measured. Specimens are kept in 80 ºC water for 1 day after removing them from the moulds and the initial length are taken. Specimens are exposed to 1 N NaOH soak solution at 80 ºC and their length measurements are periodically taken at 3,7,11 and lastly 14 days. The standard does not give a value about the alkali content of cement. The reason for this is that the pore solution alkalinity of the samples increases due to curing conditions.

The expansion percentage obtained by this test method are evaluated as follows:

i. If expansions are below 0.10%, aggregates may show harmless behaviour.
ii. If expansions are greater than 0.20%, aggregates may indicate potentially harmful expansion.
iii. If expansions are between 0.10% and 0.20%, aggregates may show either harmful or harmless behaviour, in other words, they can be described as suspicious. Therefore, before reaching a decision about aggregates, investigation of the cause and extension of measurements up to 28 days are suggested.

Different organizations can determine different expansion criteria according to their local experience. The materials to be used in concrete can be selected as considering the expansion level of the aggregate [Farny & Kosmatka, 1997].

CSA A23.2-25 A (Canadian), RILEM AAR-2, DD 249 (British) and RTA T363 (Australian) standards are common equivalent standard test methods of ASTM C1260.

Application of this test method is so widespread in all world due to being practical way to detect aggregates for ASR in a short time. On the other hand, this method has some critical drawbacks as follows:

i. Pulverizing aggregates excessively might lead their reactivity to decrease due to modification at practicable of reactive silica in aggregate.
ii. High-test temperature accelerates the reaction more than the necessary that cause unreal environmental test conditions. Specimens are stored at 80 °C which is so high that may cause false results for test aggregate.
iii. Concrete mixture cannot be detected by this method because only fine aggregates are tested in a mortar bar [Latifee, 2013].

3.1.15 Uranyl Acetate Gel Fluorescence Test (1988)

This method is an indirect way to assess hardened concrete in views of existence of ASR products by controlling petrographic examination report of hardened concrete structure. Hardened concrete exposed to a solution including uranyl acetate is monitored by ultraviolet light (UV). Uranyl acetate can cause fluorescence generation due to ASR products. Although being a rapid and practical method, it requires professionalism to carry out it [Natesaiyer & Hover, 1988; 1989].

3.1.16 Simple Chemical Method (1989)

The Simple Chemical Method is a practical way because detection of aggregate takes only 24 hours. Some soak solution with KCl, HCl are used to dissolve 100 g aggregate at 70 °C. Main disadvantage of this test method is that it cannot give quantitative data about potential reactivity of test aggregate [Chatterji, 1989].

3.1.17 Autoclave Mortar Bar Test, 1991

Autoclave Mortar Bar Test was first introduced by Fournier et al. in 1991 as a rapid test method that can be completed within only a few days. This experiment is applied on mortar bars suitable for ASTM C227. In this test, water / cement ratio is 0.5 and alkali content is increased to 3.5% Na_2O_{eq} by adding NaOH to the mixing water. Samples are cured at 100% RH and 23 °C in two-day period before entering the autoclave. In the autoclave, the samples are stored at 130 ºC for 5 hours under pressure of 0.17 MPa. Length measurement of samples are taken both before they enter the autoclave and after samples are cooled down to 23 ºC and expansion generation is recorded. This method shows good correlation with ASTM C1260-accelerated mortar bar method but poor correlation with ASTM C1293-concrete prism test [Fournier et al., 1991].

3.1.18 Accelerated Concrete Prism Test, 1992

Ranc and Debray developed a test method that was one of modified available concrete prism test methods by increasing exposure temperature from 38 ºC to 60 ºC in 1992 [Ranc&Debray, 1992]. From this date, the expansion limit was proposed as 0.04% in the light of the data obtained from many studies. Further experimental studies showed that this test method can give good correlation with concrete prism test taking 1 year or longer by keeping in view correlation coefficient (R^2=0.98) [Thomas et al., 2006]. Nevertheless, this test method could not solve the problems in the standard concrete prism test such as long time consuming and alkali leakage.

3.1.19 Duncan Method (1992)

This method is executed by curing of four mortar bars at 100% RH and 64 ºC. These mortar bars are prepared according to specifications in ASTM C227. An expansion limit of 0.05% was proposed for 16 weeks [Swamy, 1992].

3.1.20 Japanese Autoclave Test (1992)

Japanese Autoclave Test was originally approved in 1992 and numerated as JIS A 1800s that is a standard method to detect concrete structure for ASR [Kishitania et al., 1994]. After samples of mortar bars are cured for two days, their concrete mixture consisting of pulverized aggregate, cement, water and standard sand are stored with NaOH soak solution in very hot water (127 ºC) in a pressure vessel providing 0.15 MPa gauge pressure for four hours. Samples of mortar bar made of crushed sample aggregate, standard sand, cement and NaOH solution are placed in boiling water in a pressure vessel (gauge pressure 0.15 MPa, temperature 127 °C) for 4 hours after 2-day curing. Evaluation of aggregate for reactivity is made by observing changes in firstly length as well as dynamic modulus of elasticity and UPV (ultrasonic pulse velocity) of concrete. This method has common drawbacks like accelerated test methods [Latifee, 2013].

3.1.21 Modified Gel Pat Test (1993)

This gel pat test modified by Fournier in 1993 examined the reactivity on polished concrete pieces differently. 25 mm x 75 mm x 75 mm concrete parts were stored for 56 days in a soak solution with 1N NaOH at 38 ºC. The gel pat test rating (GPTr) was improved in order to gauge quantity of formed gel on these parts. 65 types of carbonate aggregate originated from Canada were tested by this method to check the reliability. According to results, GPTr showed very strong correlation with available test methods in Canada [Berube & Fournier, 1993].

3.1.22 USACE (1994)

US army corps of engineers modified mortar bar test (USACE) is specially designed to evaluate aggregates predicted as react slowly by modifying ASTM C227 (a direct method). Mortar bars are exposed to 60 ºC and 100% RH for 1 year. Before applying the test, petrographic examination of aggregate is inspected and siliceous aggregates with strained quartz is used in more than 20 percent by mass [Munir et al., 2017].

3.1.23 Concrete Microbar Test (2000)

Xu et al. improved concrete microbar test (CMT) to be used in measuring alkali carbonate reaction in 2000. After then, this method was applied to detect both ASR and ACR by Grattan-Bellew et al. in 2003. Good correlation between CMT and standard concrete prism test was observed [Xu et al., 2000; Grattan-Bellew et al., 2003].

The difference between this method and the accelerated mortar bar method is (i) usage of 40 mm × 40 mm × 160 mm moulds and (ii) the preparation of concrete microbar samples using a gradation aggregate between 12.5 mm and 4.75 mm. The aggregate cement ratio of the mixture is 1.0 and water/cement ratio is be 0.33. The amount of water is allowed to be changed to ensure workability. Like in the accelerated mortar bar test, the alkali content of the cement is not so important because the alkalinity of the pore water is very high due to the alkalis added from the outside. Microbar test specimens are exposed to a soak solution with 1N NaOH at 80 ^{o}C like in AMBT. The expansions measured in this experiment show linearity for 30 days [Xu et al., 2000; Grattan-Bellew et al., 2003; Andiç-Çakır, 2007].

If the concrete microbar test results and concrete prism test results are compared, siliceous limestones causing ASR expansion may show a different relationship with other aggregates showing carbonate reactivity (greywacke).

Like in these types of siliceous limestone, there is no correlation between the expansion results of the accelerated mortar bar method and the concrete prism experiment. However, with two exceptions, all aggregates determined to be reactive in the concrete prism test were also found to be reactive in the accelerated mortar bar test. The reason is still unclear for the lack of correlation between the accelerated mortar bar test and the concrete prism test. In this experiment, 30-day expansion limit has been determined as 0.14% for siliceous limestones and 0.04% for other aggregate types [Xu et al., 2000; Grattan-Bellew et al., 2003; Andiç-Çakır, 2007].

3.1.24 A Modified Test Method to Assess Recycled Concrete (2000)

This method developed by Gress in 2000 is an accelerated modification of AMBT (ASTM C1260) and CPT (ASTM C1293) to assess the reactivity of recycled aggregate because these available standards can be applied on only usual aggregates. 280 mm x 76.2 mm x 76.2 mm concrete prism and 76.2 mm cube specimens are prepared to measure expansion ratio [Gress, 2000]. This accelerated modified test method is seen as an effective method for accelerating the reactivity.

3.1.25 ASTM C1105 (2008) Length Change of Concrete Due to Alkali-Carbonate Rock Reaction

ASTM C1105 is the most reliable test method used for the measurement of alkali carbonate reactivity. Six concrete prism samples are poured from aggregate and cement to be used in the field (or Type I cement or II). 1-year test duration is ideal for the experiment. However, 3 or 6-month expansion results can also be used to decide if the time is limited. Expansions of 0.030% at 1 year, 0.025% at 6 months or 0.015% at 3 months indicate that the aggregate may potentially show carbonate reactivity.

This experiment not only gives an idea about the reactivity of the aggregate, but also allows to decide on the behaviour of the cement-aggregate combination. However, that the experiment takes a long time is a disadvantage. The aggregate subjected to ASTM C1105 should be examined by petrographic analysis according to ASTM C295 and or ASTM C586 should be performed on it. This test method is named Canadian CSA A23.2-14A.

Although similar sample sizes and curing conditions are used with ASTMC 1105, the higher alkali level of cement in the Canadian version aggravates the conditions. Expansion limits of the Canadian standard have been accepted as 0.010% for 3 months and 0.025% for one year. Therefore, it is possible to determine non-reactive aggregates according to ASTM standard as reactive materials with Canadian standard.

3.1.26 Universal Accelerated Test to Detect Aggregates for ASR and ACR (2008)

The universal accelerated test is performed by using 40 mm x 40 mm x 160 mm microbars and aggregate gradation consisting of only 2.5-5.0 mm fraction. Therefore, it can be seen as a type of modified Chinese microbar test. After cured in water at 80 ºC for 24 hours, Specimens are stored in a soak solution including 1N sodium hydroxide at 80 ºC for 28 days and then the expansion ratio is determined. The studies on trial of this method have suggested expansion limit for 14 days as 0.093% [Lu et al., 2008].

3.1.27 Microwave Method (2013)

Donnell et al. suggested microwave method based on principles of that mixture component percent of mortar specimens is same as in ASTM C1260. Storage is at seal container filled with 38 ºC water for 36 days and then measurements are performed by microwave method. Microwave parameters may vary with process of gel generation and allurement of free water [Donnell et al., 2013].

3.2 Designed New Methods to Improve Reliability for Alkali Reactivity of Aggregates

Prominent new method trials studied by researchers will be mentioned in this part in addition to available test methods. However, these new designed test methods have not yet standardized although correlation analyses were made by comparing them with standard test methods, especially CPT methods.

3.2.1 Ultra-accelerated CPT method (2005)

Berra et al. developed a concrete prism test method for ASR that tried on different types of natural aggregates originated from Italy. In this method, concrete prism specimens are prepared by concrete mixtures with various alkali contents and test aggregates and stored in an alkaline soak solution at 150 ºC (very unrealistic condition) for 21 days. Standard CPT method is used to control the results of Ultra-accelerated CPT method [Berra et al., 2005].

The results showed that ultra-accelerated CPT method may be determinative rapid way to detect alkali silica reactivity of natural aggregates only for ones defined as susceptible and potentially reactive. Most concrete specimens with defined aggregates give suitable expansion percentages for 21 days with CPT method's for 1 year while some of aggregate types show poor correlation with CPT method's. Comparison analysis suggests that it is rational to specify expansion limit as 0.12% for 21 days [Berra et al., 2005].

3.2.2 Miniature Concrete Prism Test (2013)

Latifee presented a new rapid test method designated as "miniature concrete prism test (MCPT)" to assess reactivity of test aggregates in 2013. Instead of 75 mm x 75 mm x 285 mm prisms like in ASTM C1293 (CPT), prepared mixtures are poured into 50 mm x50 mm x 285 mm moulds in this method. Specimens are removed from the moulds nearly 24 hours after poured and then measurement of their initial lengths are recorded. The specimens are stored for extra 24 hours ta 60 ºC water, totally 48 hours after casting. Length measurements of the specimens are takes as zero day length (l_0) by help of a comparator. After these operations, length measurements are regularly taken at 3, 7, 10 days; 2, 3, 4, 6, 8, 10 and 7 weeks at 60 ºC not at ambient condition [Latifee, 2013].

As for evaluation of experimental results, correlation analysis for this method was made by compared its data with that obtained from AMBT and CPT. The researcher states that this method gives more reliable data than ASTM C1260 (AMBT) and provides good correlation with CPT when compared its 8-week expansion results and CPT's 1-year ones [Latifee, 2013]. 33 different types of aggregates having various petrographic properties were test by MCPT while available AMBT and CPT examined only 12 types of them to seek the degree of correlation.

Figure 3.2 clearly indicates a well correlation (R^2=0.99) between the data obtained from MCPT and CPT methods. Expansion limit to define whether test aggregate was reactive or not was taken as 0.040% like in ASTM C1293 for both methods [Latifee, 2013].

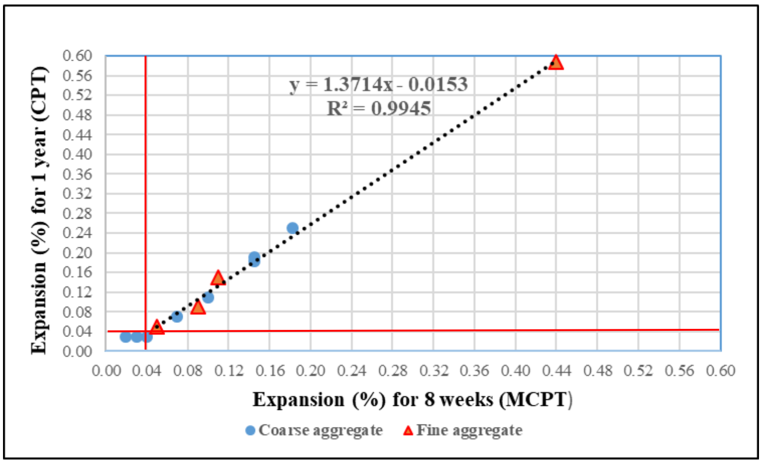


Figure 3.2 Correlation analysis of MCPT for 8-week data with CPT for 1 year [Latifee, 2013]

As similar to data in the previous figure, Figure 3.3 indicates that correlation analysis, made regardless of very reactive aggregates showing more expansion than 0.2% at 8 weeks, refers to good correlation (R^2=0.97) for the same data of MCPT and CPT [Latifee, 2013].

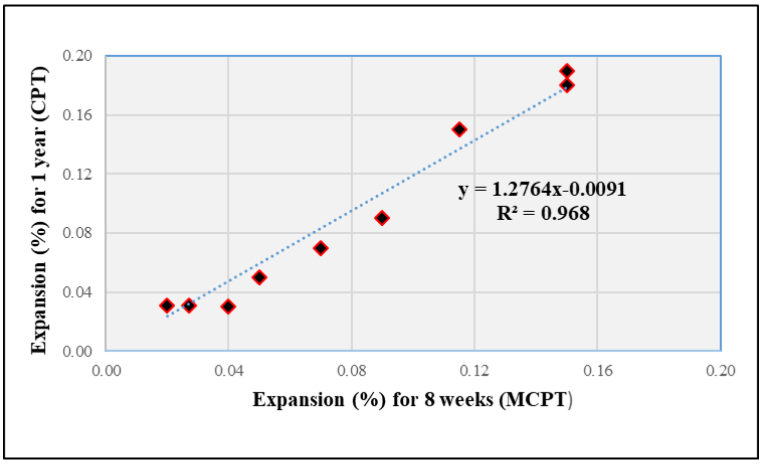


Figure 3.3 Correlation analysis of MCPT for 8-week data with CPT for 1 year regardless of very reactive aggregates [Latifee, 2013]

3.2.3 An Improved Test Method of Chinese Standard Test Method (2013)

Wang et al improved available Chinese mortar bar test methods numerated as JTG E42 and TB/2922.5 (accelerated) applied to detect the reactivity of slate aggregates. Depending on the common principles of mentioned standard methods such as exposure to temperature and humidity, this new method tends to get close to realistic field experience. For example, external alkalis is not used like in Chinese accelerated mortar method. Specimens prepared by slate aggregates having 0.8-2.5% alkali content were stored at 80 °C water for 21 days and 3,7,14 and 21-day expansion ratios are determined. Table 3.1 gives the data obtained from this improved new method in views of increasing alkali content [Wang et al., 2013].

Data indicates not so good correlation with available accelerated mortar bar. For example, 14 day expansion in the available method is 0.46% as that in the improved new method is 0.31%. These data point out a huge deviation but the data may be rated to find a linear equality like as 0.46/0.31=1.5. In general, the new improved test can be applicable for slate aggregates due to stable results and repeatable data [Wang et al., 2013].

Table 3.1 Data from the new method [Wang et al., 2013]

#Specimen	Alkali content (%)	Expansion (%)			
		3 day	7 day	14 day	21 day
1	0.8	0.01	0.02	0.02	0.02
2	1.0	0.01	0.02	0.03	0.03
3	1.2	0.03	0.05	0.07	0.06
4	1.5	0.07	0.17	0.19	0.18
5	1.8	0.14	0.26	0.31	0.32
6	2.0	0.11	0.22	0.28	0.28
7	2.5	0.10	0.21	0.26	0.26

3.3 Comparison of Laboratory Performance Tests for ASR

Heck made a correlation study for three standard test methods which were ASTM C289 (Chemical Method), ASTM C227 (Mortar Bar Method) and ASTM C295 (Petrographic Examination). Potential reactivity of aggregates were examined due to these methods by using siliceous and limestone sand in various mass ratio and three different size distributions of aggregates. Test results prove that a poor correlation appears between the chemical method and mortar bar. Mortar bars prepared by reactive siliceous and limestone sand in 20-10-5-2 % by mass show expansion over the reactivity limit that is 0.05% for 3 months so they can be defined as potentially reactive material. That indicates compatible results with the chemical method. However, the reason for the poor correlation is that mortar bars all did not expands over 6-month expansion limit (0.1), some of them exceeded 1-year expansion limit.

As for petrographic examination applied for determination of potentially reactive constituents in aggregates to be tested, not good correlation between petrographic examination and mortar bar method could not be obtained although the results of petrographic examination was seen to correlate well with ones of the chemical method. Moreover, a linear equality formed between the proportions of reactive constituents and the amounts of dissolved silica obtained from ASTM C289 (Chemical Method) [Heck, 1983].

Islam and Akhtar have evaluated susceptible aggregates by applying standard test methods such as petrographic examination (ASTM C295), accelerated mortar bar method (AMBT), concrete prism test (CPT) and modified version of AMBT and CPT to evaluate the aggregates in views of field performance petrographic and mineralogical properties of innocuous aggregates for ASR. The results of this study showed that, an appropriate method combination including different method should be established to find a right assessment method for alkali silica reactivity of aggregates since not all methods yield reliable results on a single basis [Islam & Akhtar, 2013].

Whiting detected 12 types of concrete pavements for which were considered as in early damaged manner due to alkali-silica reactivity by performing standard test method like as ASTM C1260. ASTM C1293. The data obtained these standardized methods created a serious contradictory view to assess reactivity of aggregates. Mortar bars consisting of sands tested by AMBT exceeded expansion limit (0.02% for 14 days in ASTM C1260) but concrete prisms tested by CPT showed expansion varying nearly from 0.03 to 0.04 so they were determined as potentially not reactive because of lower expansion than CPT expansion limit (0.04% for 1 year). Concrete prisms with opaline shale deteriorated and spall, pop-up formed on their surface (Figure 3.4) despite of low expansion appeared in them [Whiting, 2013].

Shayan applied mortar bar test (MBT), accelerated mortar bar test (AMBT), autoclave test and concrete prism tests (CPT) on five different sand under different conditions. Mortar bar specimens for MBT were cured at 40 ºC and 100% relative humidity. The content of cement was adjusted by adding NaOH to the mixture water so that the Na_2O_{eq} was 1.38 and 1.80. For AMBT, the curing temperature was kept at 80 ºC, but the alkali content of the solution was adjusted to 0.5, 0.75 and 1 N. In the autoclave method, the mortar specimens were prepared by adding NaOH to the mixing water that increasing the alkali content to the levels of 2.50 and 3.50 Na_2O_{eq} and then cured at 127 ºC for 4-5 hours in the autoclave. In the concrete prism test, non-reactive basalt was used as coarse aggregate and cement was adjusted by adding

NaOH to the mixing water with 2 different Na_2Oeq values, 1.38 and 1.80. Samples prepared using 460 kg/m^3 cement were cured at 40 °C [Shayan et al., 1994].

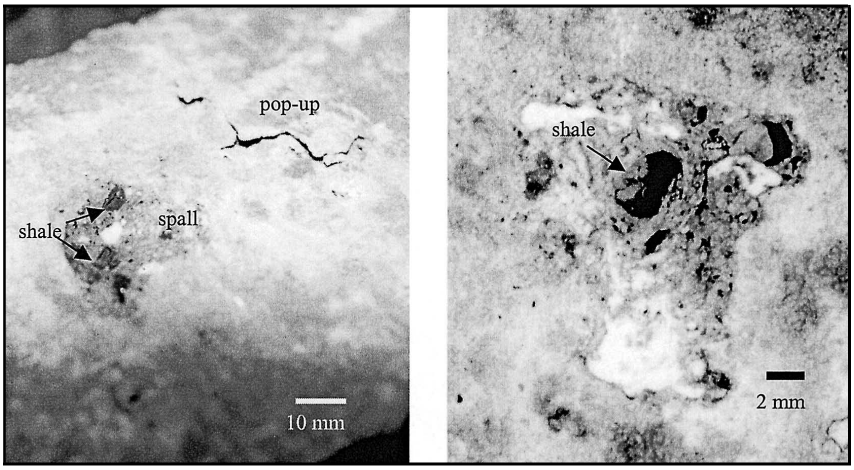


Figure 3.4 Diagnosis of ASR damage on concrete with opaline shales [Whiting, 2013]

Not all the aggregates investigated were reactive because they showed an expansion under the limit of 0.04% in the mortar bar test. However, samples cured in standard 1N NaOH in the AMBT showed reactivity due to exceeding the expansion value of 0.10% between 10 and 17 days. That these samples exceed the 0.20% limit for 21 days are even so indicative that they are reactive [Shayan et al., 1994].

Rogers proposed limits for the AMBT are as follows; 0.15% for 14 days, 0.33% for 28 days, 0.48% for 56 days [Rogers, 1993]. However, these limits do not provide suitable results for some slowly expanding aggregates. According to the results of the autoclave, only one of the sands gives the impression that it is reactive [Shayan et al., 1994].

Grosbois and Fontaine compared CPT and AMBT with a comprehensive study on different types of aggregates. This study deals with test methods for different types of aggregates separately. One-year test limit for CPT was 0.04%; For AMBT, the 14-day expansion limit is 0.10% and 0.15%. Accordingly, the following situation was observed for different aggregate types. Carbonate Rocks; Many reactive and non-reactive aggregates give accurate diagnosis by both methods. However, in some of the aggregates, which were found to be reactive with CPT, the same diagnosis could not be established. The opposite situation was not observed. Sandstone and other sedimentary rocks; most of them show expansion over the limits. However, the extreme expansion values measured with AMBT performed in standards remain insufficient. Volcanic and metamorphic rocks; mostly both experiments put the diagnosis. However, AMBT shows a higher expansion compared to CPT in some mafic rocks. The results show that AMBT is not able to make a reliable diagnosis for carbonaceous aggregates. Higher expansion limits may be recommended for granite and other metamorphic rocks. No suitable results can be obtained from AMBT and CPT for sandstones and other sediments [Grosbois & Fontaine, 2000].

Wigum clarified the results of the Norwegian alkali aggregate research program called NORMIN 2000. In this study, three different work groups including related experts were set up. Work areas of these groups were petrographic analysis, accelerated mortar bar test and field performances of the concrete structures [Wigum, 2000].

Within the scope of petrographic research, counting point was carried out by taking a thin section from the aggregates, which were 1-2 mm to 2-4 mm in dimensions, and the amount of the reactive material was indicated in percentage by volume. Up to 1000 points were counted for each sample. As a result of the petrographic analysis, it is concluded that the aggregates are not reactive if the total amount of the types of rocks considered to be potential reactive is less than 20%. In the accelerated mortar bar method, 40 mm × 40 mm × 160 mm mortar samples are used. If the 14-day expansion values are lower than 0.10%, they defined test aggregate as non-reactive; if between 0.10% and 0.25%, they indicate slowly expanding aggregate. Expansion values greater than 0.25% are an important signal for very reactive aggregate [Wigum, 2000]. In the light of the studies performed, different researchers may reach different results because of statement that petrographic analysis is a method depending on the experience and skill of petrography. In order to be clearer, a list classifying rock types simply is made. In addition, samples consisting of reactive materials close by 20% is recommended to be exposed to the AMBT method before classification [Wigum, 2000].

Jensen and Fournier applied AMBT and CPT on 7 types of aggregate, which were black sandstone, red sandstone, quartzite, rhyolite, filite, mylonite and cataclasite. These reactive coarse aggregates as well as non-reactive fine aggregates were used in CPT. In AMBT, correlation between 40 mm × 40 mm × 160 mm samples used in the RILEM method and 25 mm × 25 mm × 285 mm samples used in ASTM has been found as 0.60xASTM=RILEM. Moreover, the researchers state that size of the storage containers at CPT affects the expansion values [Jensen & Fournier, 2000].

Xu et al. provide an accelerated method based on curing at 80 ºC, developed by small concrete samples for the determination of alkali carbonate reactivity. They examined 20 dolomitic limestone and limestone samples with different geological characteristics by this method and the autoclave method [Xu et al., 2000].

The autoclave method is applied at temperature of 150 ºC and 10% KOH alkaline solution for 6 hours. Samples for this method are prepared with 5-10 mm aggregate, and 1.5% equivalent Na_2O cement alkalinity. This method is similar to the Chinese autoclave method but contains different sized aggregates. In the accelerated 80 ºC method, moulding and procedures before curing are the same but the material and test conditions differ from as follows:

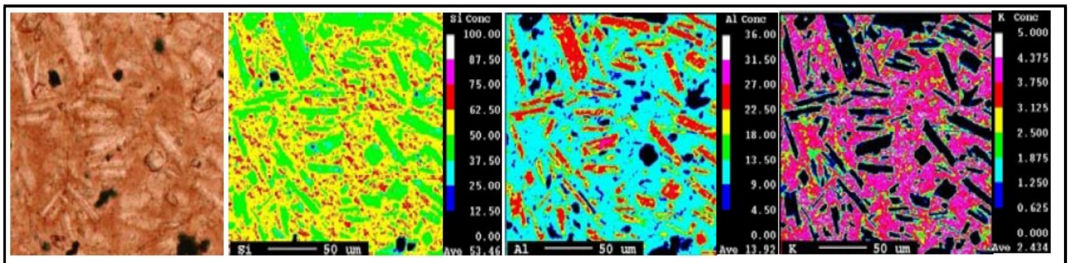
i. Water/cement=0.3, aggregate/cement=1.0.
ii. KOH is added to soak solution to make alkali content increase to Na_2O_{eq} 1.5%.
iii. Alkali cure: 1N (molar) NaOH.
iv. Size of samples: 20 mm x 20 mm x 80 mm and 40 mm x 40 mm x 160 mm.
v. Aggregate size: 0.8-1.25 mm, 1.25-2.5 mm, 2.5-5 mm, 5-10 mm [Xu et al., 2000].

Researchers who emphasize that there is no change in the cement hydration products and the hydration process in especially 80 ºC experiments indicate that the maximum expansion of carbonate aggregates comes from the competent with aggregate size of 5-10 mm. However, it has been clearly seen in the other dimensions examined that the expansions increase as the aggregate size decreases. Herein, a pessimum size for ACR may be mentioned. These studies are recommended as an expansion limit of 0.1% at the end of 4 weeks for the concrete microbar test compared to the autoclave method and field surveys. As both the 80 ºC and 40 ºC cures of the same samples were compared, the expansions at both temperatures were constant in the non-reactive aggregates, while the expansions at the high temperature in the reactive aggregates increased. This gives the impression that the temperature does not have any detrimental effect other than increasing AAR expansions [Xu et al., 2000].

3.4 Effect of Petrographic Properties and Mineralogical Composition of Aggregates on Alkali-Silica Reactivity

Petrographic properties and mineralogical composition of aggregates give a general opinion about crystalline structure of reactive silica because it is critical in high performance concrete for detecting inclination of aggregates to ASR. Ali et al. analysed 20 types of aggregates in views of their petrographic features, textural characteristics and mineralogical composition. Most samples mainly compose of quartz particles in form of micro/poly-crystalline, micro-cracked, stiff, xenomorphic and elongated. Potential reactivity of aggregates increases as the amount of Silica ($Si0_2$) increases if high alkali cement is used. Therefore, cracking, expansion and deterioration of concrete structure are unavoidable due to ASR. Damaged reinforced concrete structure is vulnerable to corrosion so its service life may unintentionally drop. Micro-cracks in undulated and elongated quartz particles may open alkali solution the way to cement paste where more suitable for reaction formation. In addition to dense silica ratio, test rocks include plenty of phyllosilicate minerals, which are very prone to swell up and expand in high degree due to their absorption characteristic. Swelling of these minerals such as biotite, chlorite and muscovite can generate tremendous pressure at interfacial transition zone which is an expression used for border between aggregate and cement paste. Apart from phyllosilicate minerals, a fine-grained plagioclase and K-feldspar may be seen as potentially reactive constituents of rocks to create a risk for high performance concrete [Ali et al., 2014].

Kawabata detected widely some types of rocks such as andesite and volcanic origin ones in Japan where andesite composes of nearly 25% of all crushed aggregate in views of petrographic characteristics. On the other hand, samples were also tested by quick chemical and AMBT (A1146-Japanese Standard) methods and mass proportion of alkali content in cement was limited to virtually 1.2% and stored at 40 °C. Figure 3.5 gives an example of imtime mapping obtained by optical microscope analysis and electron probe microanalysis (EPMA) on a glassy type of andesite (An(M)) [Kawabata et al., 2007].



(a) Optical (b) Si (c) Al (d) K

Figure 3.5 Image mapping of An(M) obtained by optical microscope and electron probe microanalysis [Kawabata et al., 2007]

Moreover, scatter diagram in Figure 3.6 clarifies the phases (plagioclase or pyroxene) of Si (silica) and Al(alumina) by providing handy measurement of points. These analyses on An(M) show clearly that phenocrysts and glass phase in matrix form can be monitored with no presence of alteration. K (Potassium) is situated in merely in matrix form of glass not in phenocrysts due to its liquid form. An(M) does not consist of silica [Kawabata et al., 2007].

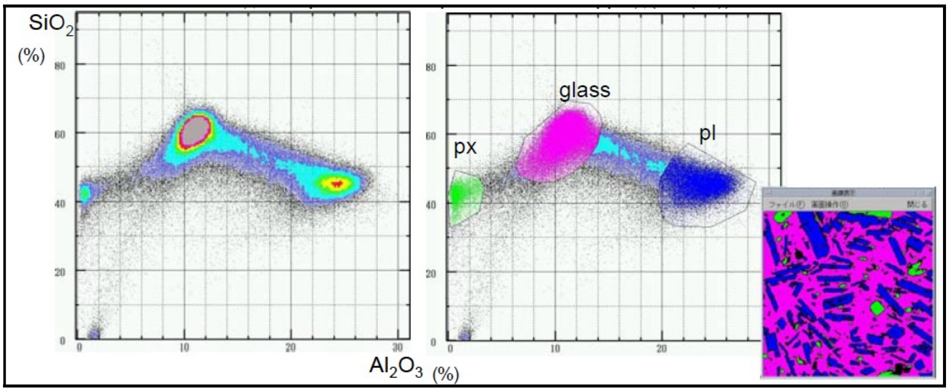


Figure 3.6 An(M) scatter diagram with Al_2O_3 and SiO_2 [Kawabata et al., 2007]

More complex diagram is countered as in Figure 3.7 showing a type of andesite named as An(A) due to presence of alteration. Concentration of potassium appears in a high level as if being altered glass. Additionally, presence of silica mineral defined as cristobalite by X-ray Powder Diffraction (XRD) is explicitly observed in Figure 3.7 [Kawabata et al., 2007].

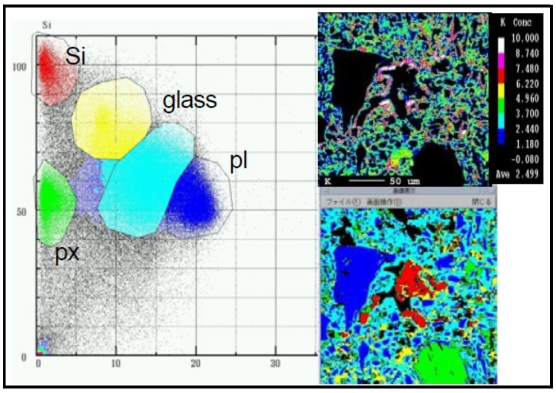


Figure 3.7 An(A) scatter diagram with Al_2O_3 and SiO_2

In general, this study exhibits that dissolution conduct and composition of pore solution of an andesite have important effect on its reactivity and petrographic examination. They can also provide valuable information about which constituents are innocuous for ASR.

Castro et al. analysed five types of aggregate quantitatively by applying XRD method on their polished surface. Petrographic examination on these aggregates, selected as both reactive and non-reactive and originated from 4 different countries, indicates that the aggregates consists of remarkable diversities in ingredient lithology. Figure 3.8 gives a random texture microphotograph of rock types examined in this petrographic study. In this study, optical petrography was firstly used to detect susceptible constituents for ASR. After that, extensive evaluation on granulated particles and polished surface was performed thanks to XRD method. Results of further petrographic examination shows that highly fine-granulated quartz containing may be seen as potentially harmful constituents of aggregates. Thus, it is thought as the reason for high expansion percentage because of its excess silica [Castro et al., 2012].

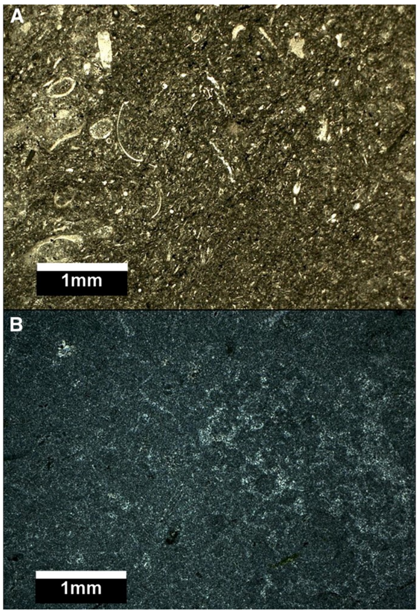


Figure 3.8 A random texture microphotograph of test rock types [Castro et al., 2012]

3.5 Influence of Chloride Based De-Icers on the Process of ASR

Desai evaluated effectiveness degree of chloride-based de-icers on ASR process by conducting AMBT methods at 80 ºC (standard form) and at 38 ºC (modified form). Mortar bars included fused silica as a reactive material and sandstone as a non-reactive material with cement having alkali content in different ratio. Data obtained from these tests indicates that mortar bar samples stored at 3% NaCl solution and KCl may expand more than those stored at NaOH like in standard AMBT if fused silica is used. On the other hand, $CaCl_2$ and $MgCl_2$ have been observed as inconclusive on increasing alkali silica expansion for samples with same contents. De-icing salts invalidated the effect of alkali content from cement because they provided redundant alkali to the reaction. The reasons for more expansion in samples exposed to the solution with chloride-based de-icers are explained by that chloro-aluminates appear as a reaction product and chlorides initiate generation of alkali hydroxides [Desai, 2010]. The results are given according to various soak solutions as below.

3.5.1 Deionized water

Figure 3.9 reveals that alkali content of cement has major effect on magnitude of expansion for mortar samples stored at deionized water. It is remarkable that samples stored at lower temperature give higher expansion even if cement with higher alkali content is used. The samples with fused silica expanded in drastic rate for 7 days but after that expansion decelerated likely due to depletion of alkalis to continue the reaction [Desai, 2010].

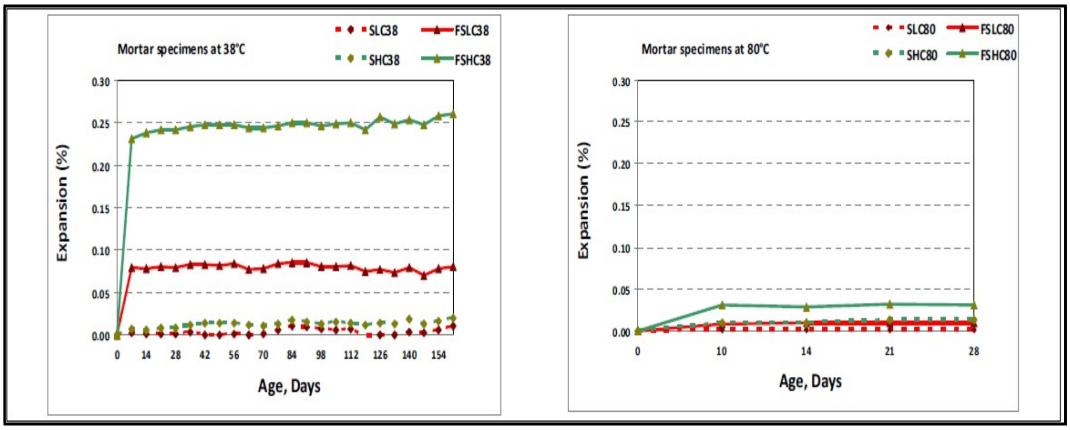


Figure 3.9 Graphical data for mortar samples stored at deionized water [Desai, 2010]

3.5.2 3% Sodium chloride (NaCl) by weight

The results clearly show that sodium chloride increase expansion rates in mortar bar because it supplies sufficient alkalis for the reaction. As shown in Figure 3.10, at 38 ºC, the mortar samples with fused silica showed highest expansion after 147 days, nearly 2.2% whereas the highest expansion at 80 ºC was measured as 2.0% [Desai, 2010].

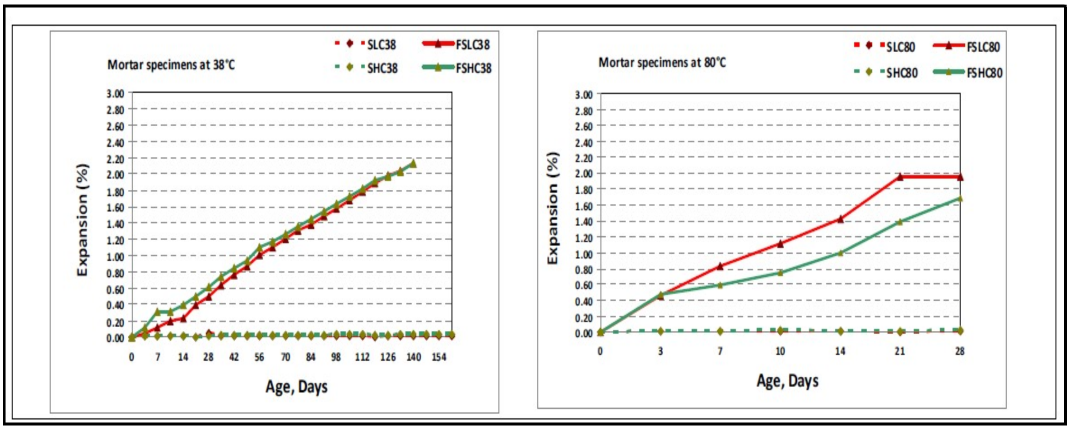


Figure 3.10 Graphical data for mortar samples stored at soak solution with 3% NaCl [Desai, 2010]

3.5.3 3% Calcium chloride ($CaCl_2$) by weight

The results of this experimental study revealed that $CaCl_2$ has inconsiderable influence for accelerating ASR both temperatures of 38 ºC and 80 ºC if compared samples stored at $CaCl_2$ soak solution with those stored at deionized water because expansion seems to be affected by alkali content of cement (Figure 3.11) [Desai, 2010].

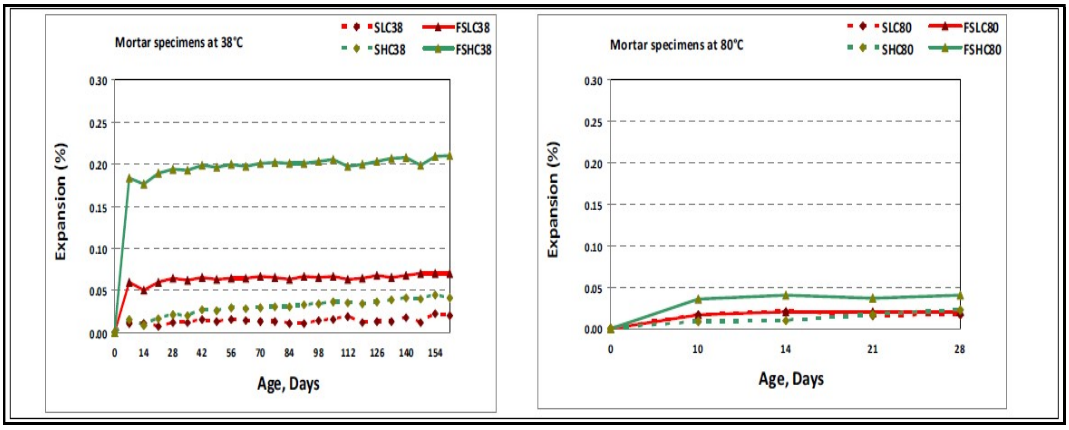


Figure 3.11 Graphical data for mortar samples stored at soak solution with 3% $CaCl_2$ [Desai, 2010]

3.5.4 0.5N Sodium hydroxide (NaOH) and 3% sodium chloride (NaCl) by weight

Figure 3.12 openly exhibits that samples stored at this soak solution give higher expansion rates than those stored at standard solution consisting of 1N NaOH. 161-day expansion of mortar bar samples at 38 ºC reached up to 2.5% as their 28-day expansion is 1.5%. That demonstrates crucial effect of Cl^- ions on ASR. Moreover, another inference of the results may be seen as that storage temperature increase expansion of samples [Desai, 2010].

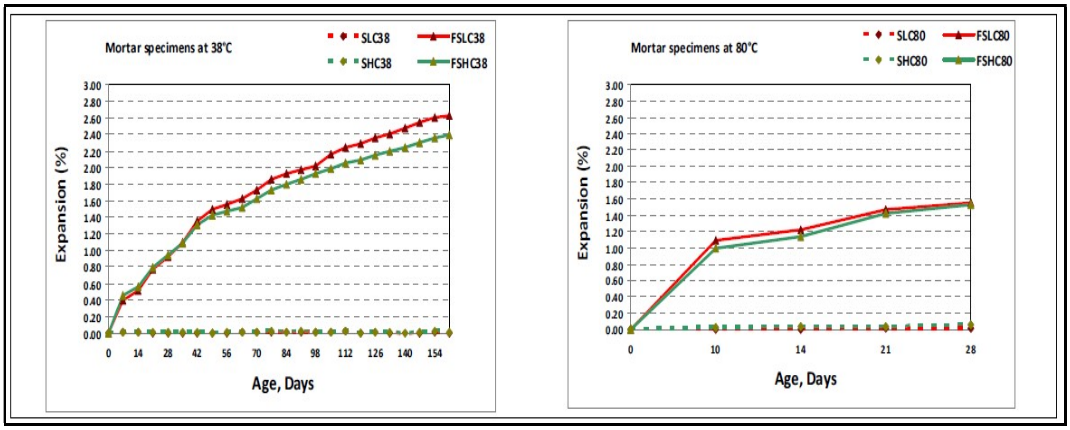


Figure 3.12 Graphical data for mortar samples stored at blended soak solution with 0.5N NaOH and 3% NaCl [Desai, 2010]

3.5.5 3% Magnesium chloride ($MgCl_2$) by weight

As seen in Figure 3.13, $MgCl_2$ does not affect ASR expansion of mortar samples as compared these samples exposed to $MgCl_2$ with those to deionized water. Like as $CaCl_2$, alkali content of cement seems to determinant factor for degree of expansion [Desai, 2010].

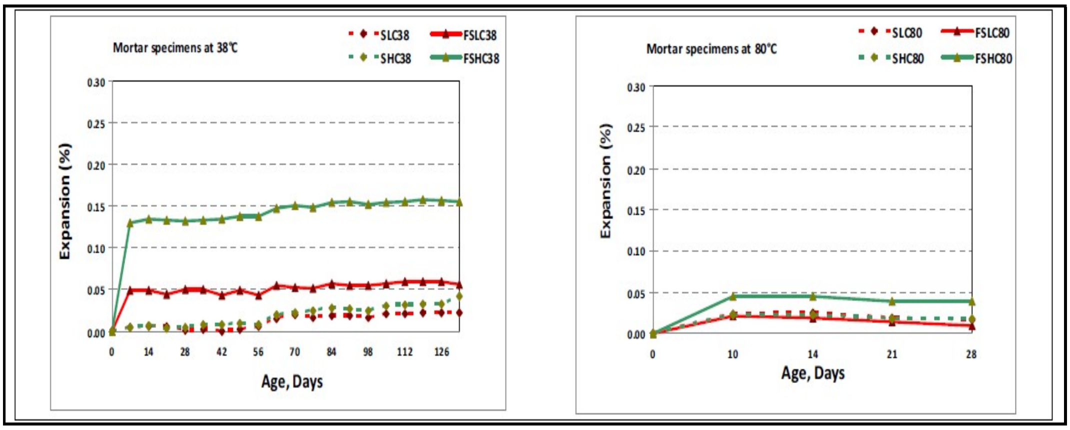


Figure 3.13 Graphical data for mortar samples stored at soak solution with 3% $MgCl_2$ [Desai, 2010]

Nixon et al. have examined the influence of sodium chloride and synthetic seawater on what degree they affect the process of ASR by using them as a mixing material of test specimens not in soak solution. The results show that they can increase OH$^-$ ion concentration and thus accelerate formation of ASR products causing deleterious expansion if reactive aggregates are used [Nixon et al., 1988].

Berube conducted an experimental investigation on concrete cylinders, 5 inch in radius, by making up different storage conditions for them. Test specimens were prepared with cement having different alkali content and a very reactive aggregate and stored at 38 °C. For starters, cylinder specimens were exposed to two various soak solution with NaCl in both 3% and 6% and then to moist environment at 100% RH. Lastly, a complex cycle includes 12-day moist exposure, 2-day drying process, storage at a soak solution with 3% NaCl for 3 hours. Figure 3.14 shows that all concrete cylinders with low alkali content did not expand notably while those with high alkali content all went beyond the expansion limit (0.04 % for 365 days) at even 26th week. Concrete cylinders with high alkali content which were stored at soak solutions with NaCl expanded less if compared them with specimens exposed to moist air at 100% RH and the cycle. This remarkable inference is identified with the fact that NaCl or seawater does not sufficiently contribute hydroxyl ion concentration provoking a jump in pH value of the pore solution that makes the concrete vulnerable to formation of ASR [Berube et al., 2003].

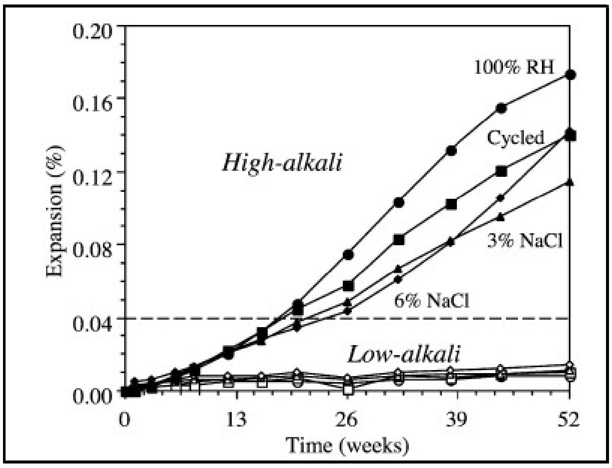


Figure 3.14 Graphical data for expansion values of test concrete cylinders [Berube et al., 2003]

Katayama et al. have evaluated the effect of sea salts or sodium chloride on ASR process by drilling cores from available concrete structures such as bridges, vents settled in seawater and experienced ASR damage. As mentioned Berube's study, adding NaCl may not provoke a significant rise in hydroxyl ion concentration so slow entrance into concrete structure by means of cracking in the cores. In this study, SEM (scanning electron microscopy) analysis was carried out on the samples and the SEM images are given Figure 3.15 (I, J) showing clearly ASR gel consisting of Cl^- ions [Katayama et al., 2004].

Kurtis et al. have utilised three lithium additives registered as LiOH, LiCl, and $LiNO_3$ to investigate their effectuality in a limited scope on only ASR expansion process because of available incomprehensible points for mechanism. An expansion mortar bar test method was carried out in order to practise this experimental study. Many soak solutions including 0.7N sodium, hydroxide, lithium additives in various ratios and calcium ions not used in all specimens were prepared to expose mortar bars. The results to be notable were that firstly, lithium additives could lower expansion in mortar bars if adequately used. Secondly, the threshold ratio $(Li_2O/)/(Na_2O_{eq})$ to perform this reduction was determined as between 0.5 and 1 by taking into account that the aggregate was reactive. Thirdly, in case of presence of redundant lithium additives, for example $(Li_2)/(Na_{eq})$=1.5 molar, expansion reduction did not go on [Kurtis et al., 2004].

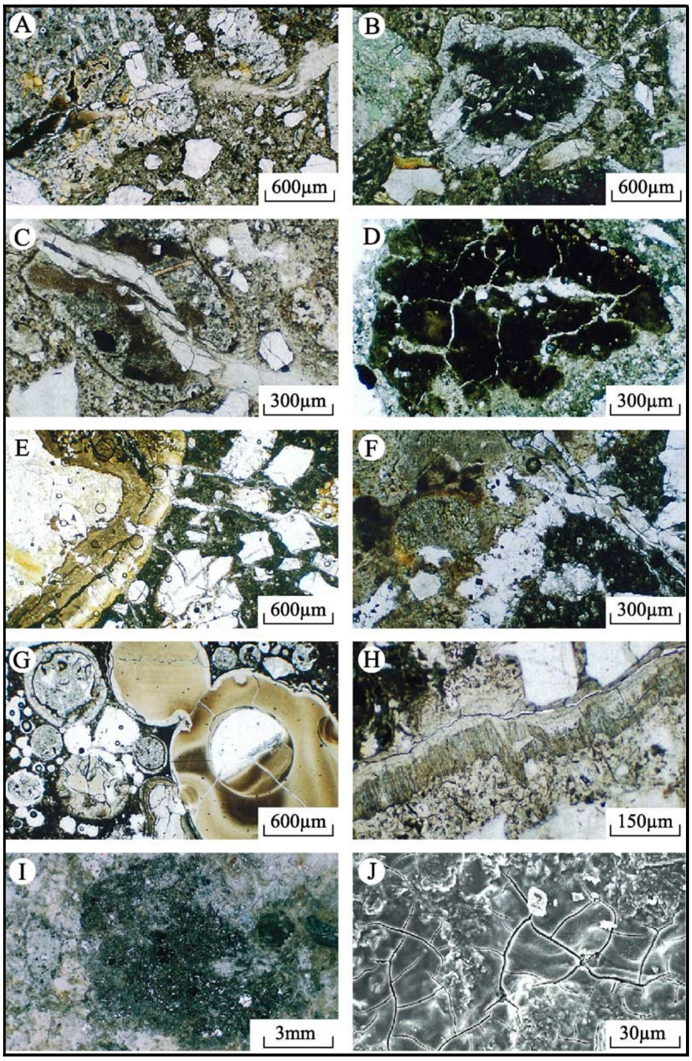


Figure 3.15 SEM images of samples exposed to NaCl in different forms [Katayama et al., 2004]

CHAPTER 4

MATERIALS AND METHODS

32 types of aggregates received from different stone quarries or river basin in Turkey, 2 types of cement, sodium hydroxide (NaOH) and chloride based de-icers such as sodium chloride (NaCl), calcium chloride ($CaCl_2$), potassium chloride (KCl), magnesium chloride ($MgCl_2$) were used for development of a new method test to improve predictability for potential alkali reactivity of aggregates. The accelerated mortar bar test (RILEM AAR-2) and the concrete prism test (RILEM AAR-4.1) methods were performed on all types of aggregates to check reliability of the results of the new methods to be developed.

4.1 Materials

4.1.1 Aggregates

Eight types of aggregates, which are known well as potentially reactive or suspicious, were examined to perform the preliminary experimental study. To begin with, developed seven new methods were applied on preliminary test aggregates and then best ones of these methods were selected according to their degree of correlation with the standard test methods. After that, the second stage of the experimental study was started by making necessary improvement in the selected methods to obtain better correlation. Finally, designed two new methods were applied on extra 24 types of aggregate (totally 32) were also tested for the latter experimental study to finalize the study for a new method. Table 4.1 provides information about petrographic identification, origin, results of mechanical and physical test for all test aggregates. Standard test methods; “TS EN 1097-6/ Determination of particle density and water absorption”, TS EN 1097-5/ Determination of the water content by drying in a ventilated oven” and “TS EN 1097-2/ Determination of resistance to fragmentation” were applied to find the numerical data of **ρ_a**=apparent particle density; **ρ_{rd}**= oven-dried particle density; **ρ_{ssd}**= saturated and surface-dried particle density; **WA_{24}**= water absorption after immersion for 24 h; **W_C**= water content (% by mass); **LA**= the Los Angeles coefficient for fragmentation” of all test aggregates. Except for Los Angeles abrasion test, the other methods were separately conducted for both fine aggregates (FA) and coarse aggregates.

4.1.2 Cement

Two types of CEM I 42.5 R specified by related standards “ASTM C150 or TS EN 197-1” were used to prepare mortar bars or concrete prism. Test methods of related RILEM standards practised in this experimental study stipulate alkali equivalent as Na_2O_{eq}=0.9-1.3% so the cements were selected in accordance with this requirement.

Table 4.2 shows chemical compositions of these cements with calculated alkali equivalent from the formula (%Na_2O+0.658% K_2O). In the preliminary experimental study, only type I with higher alkali content was used for all samples. On the other hand, both types of CEM I 42.5 R were used in next step of this experimental study to observe the effect of cement type on AAR.

4.1.3 Sodium Hydroxide (NaOH)

NaOH is the most important chemical material used in soak solutions. It provides alkali content of concrete to reach up to 1.25% Na_2Oe in accordance with the test standards for AAR. The technical grade sentences only one directive that NaOH must be leastways 98 percent purity.

4.1.4 Chloride Based De-icer (Salts)

4.1.4.1 Sodium Chloride (NaCl)

Sodium chloride is one of most common de-icers used usually in preventing freezing which may occur on highway. If used at nearly 23% concentration, solutions with

NaCl can show resistance to freezing up to at least -21 °C. The melting point of sodium chloride salt is 801 °C. Just as melting, the decomposition does not occur immediately. It turns into steam at 1440 °C. It is solid, pure, colourless and crystalline. Sodium chloride in nature, with dissolved form in the sea exists in the bed of the inner seas as rock salt and dried in dried form [Desai, 2010].

4.1.4.2 Calcium Chloride ($CaCl_2$)

Calcium chloride ($CaCl_2$) is one of the ionic de-icers in solid form at room temperature. It can be produced directly from limestone in addition to the Solvay method. $CaCl_2$ should be stored in closed container due to hygroscopic structure. Because $CaCl_2$ is a solid chemical in hygroscopic and ionic structure, it should be stored in air-tight containers. The process of $CaCl_2$ dissolution in water and alcohol is known as exothermic. It has properties of moisture retention and low freezing point. For example, a solution with nearly 30% concentration of $CaCl_2$ is observed to freeze at -51 °C. $CaCl_2$ functions as a source of calcium ions in the soak solutions and causes precipitation due to insolubility of calcium compounds [Desai, 2010].

4.1.4.3 Magnesium Chloride ($MgCl_2$)

Magnesium chloride ($MgCl_2$) is an inorganic and irregular compound. The solution's freezing degree with nearly 22% concentration is -33 °C. As heated, magnesium chloride is soluble in alcohol and water partially hydrolysed, but not completely. $MgCl_2$ is obtained from natural brines and its purest compound from sea products in Japan [Desai, 2010].

Table 4.1 Petrographic identification, origin and physical properties of test aggregates

Preliminary experimental study& Latter experimental study									
Group name	**Petrographic identification**	**Origin**	**Mechanical and physical properties**						
				ρ_a	ρ_{rd}	ρ_{ssd}	WA_{24} (%)	W_C (%)	LA (%)
G1	Siliceous (sedimentary) sandstone	Kizilirmak Basin/ Korgun Özcay Stream/ Cankiri	FA	2.67	2.61	2.66	2.0	4.8	--
			CA	2.68	2.62	2.66	1.1	1.6	22.5
G2	Granite	Bergama/İzmir	FA	2.74	2.67	2.71	1.9	0.1	--
			CA	2.73	2.68	2.70	0.8	0.0	18.7
G3	Polygenic river sandstone	Ceyhan River Basin/ Cakit Stream/Karaisalı-Adana	FA	2.67	2.61	2.65	1.6	1.5	--
			CA	2.68	2.62	2.66	0.9	0.9	20.8
G4	Siliceous river sandstone	Kizilirmak Basin/ Korgun Kartepe Stream/Cankiri	FA	2.67	2.61	2.65	2.7	3.5	--
			CA	2.66	2.59	2.63	1.6	1.2	19.9
G5	Siliceous river sandstone	Kizilirmak Basin/ Konak Stream/ Cankiri	FA	2.67	2.61	2.65	2.5	2.1	--
			CA	2.68	2.60	2.65	1.4	0.9	20.2
G6	Recrystallized limestone	Kizilirmak Basin/ AktepeVillage/ Avanos-Nevsehir	FA	2.66	2.59	2.64	2.2	0.1	--
			CA	2.65	2.60	2.62	1.2	0.1	23.4
G7	Siliceous river sandstone	Kizilirmak Basin/ Dogantepe Village/ Cankiri	FA	2.68	2.60	2.65	2.3	1.4	--
			CA	2.67	2.61	2.64	1.2	0.5	20.6
G8	Meta sandstone	Coruh River Basin/Bayburt	FA	2.69	2.61	2.67	2.2	1.7	--
			CA	2.67	2.63	2.66	1.0	0.8	19.4
Latter experimental study									
NG9	River Sandstone	Filyos Stream Basin/Caycuma-Zonguldak	FA	2.69	2.62	2.67	1.6	0.5	--
			CA	2.67	2.62	2.64	0.8	0.2	21.6
NG10	Perlite/ rhyolitic glass	Mollaköy/ Erzincan	FA	2.43	2.31	2.42	1.8	0.1	--
			CA	2.40	2.30	2.41	2.7	0.0	41.2
NG11	Calcirudite	Gediz River Basin/Sancaklı-İgdecik -Manisa	FA	2.70	2.63	2.68	1.6	0.3	--
			CA	2.68	2.61	2.66	0.9	0.1	29.3
NG12	Dolomite	Region/Dagbelen Village/Bodrum-Mugla	FA	2.72	2.64	2.67	1.1	0.0	--
			CA	2.70	2.66	2.67	0.6	0.0	22.7
NG13	Sandstone	Bartın River Basin/ Terkehaliller	FA	2.70	2.63	2.69	2.8	0.4	--
			CA	2.73	2.64	2.70	1.6	0.2	21.1
NG14	River Sandstone	Dicle River Sandstone/ Bismil-Diyarbakır	FA	2.66	2.59	2.64	3.5	4.3	--
			CA	2.65	2.60	2.64	1.7	1.2	20.6

Table 4.1 continued

NG15	Sandy Limestone	Filyos Stream Basin/Caycuma Zonguldak	FA	2.65	2.58	2.63	3.5	4.3	--
			CA	2.66	2.60	2.64	1.7	1.2	20.6
NG16	Meta Sandstone	Cebeci rock quarry/ Dereboyu-Istanbul	FA	2.71	2.62	2.69	1.4	0.0	--
			CA	2.72	2.66	2.69	0.7	0.0	23.4
NG17	Sparite	Saadet Village /Afyonkarahisar	FA	2.72	2.65	2.68	1.4	0.1	--
			CA	2.72	2.64	2.68	0.6	0.0	22.0
NG18	Siliceous Sand	Bozköy Dip Region /Manisa	FA	2.69	2.62	2.67	1.6	0.5	--
			CA	2.68	2.62	2.65	1.0	0.2	22.9
NG19	Marble	Konya Road 13. Km/ Afyonkarahisar	FA	2.67	2.62	2.65	1.1	0.5	--
			CA	2.68	2.63	2.65	0.6	0.2	26.5
NG20	Granite	Bulancak/Giresun	FA	2.72	2.67	2.69	1.0	0.0	--
			CA	2.70	2.67	2.69	0.4	0.0	20.3
NG21	Basalt	Kosebucagi/Ordu	FA	2.82	2.78	2.80	0.5	0.0	--
			CA	2.81	2.79	2.80	0.1	0.0	16.7
NG22	Granite	Erdek/Balıkesir	FA	2.71	2.66	2.69	1.3	0.0	--
			CA	2.70	2.66	2.68	0.5	0.0	21.6
NG23	River Sandstone	Sakarya River Basin/Selcuk Village/ Bilecik	FA	2.67	2.57	2.65	1.8	3.4	--
			CA	2.68	2.58	2.66	0.6	0.8	19.7
NG24	Olivine Basalt	Horozgedigi Region/Aliga-İzmir	FA	2.85	2.80	2.82	0.4	0.0	--
			CA	2.84	2.81	2.82	0.1	0.0	15.3
NG25	Calcitic Dolomite	Selcuklu/Konya	FA	2.71	2.65	2.68	1.5	0.0	--
			CA	2.70	2.65	2.68	0.5	0.0	23.7
NG26	Limestone (large of quartz, andesite,granite)	Aksu River Basin/Narlı-Kahramanmaras	FA	2.71	2.65	2.69	1.4	0.1	--
			CA	2.70	2.66	2.68	0.6	0.0	24.5
NG27	Marble	Bögecik Village Ayrancı/Karaman	FA	2.66	2.62	2.64	1.2	0.1	--
			CA	2.67	2.63	2.64	0.7	0.0	25.0
NG28	Quartz	Yesilcay stream Basin /Sile İstanbul	FA	2.68	2.64	2.67	2.2	0.4	--
			CA	2.66	2.63	2.65	0.8	0.2	17.9
NG29	Half Recrystallized Limestone	Topluca Village /Bartın	FA	2.72	2.66	2.69	0.9	0.0	--
			CA	2.71	2.67	2.70	0.4	0.0	23.9
NG30	Basalt	Umurbey Dam Gokkoy/Lapsseki-Canakkale	FA	2.89	2.82	2.83	0.3	0.0	--
			CA	2.88	2.83	2.84	0.1	0.0	14.1
NG31	Dacitic Tuff	Eksisu Region/Erzincan	FA	2.38	2.26	2.35	2.9	0.2	--
			CA	2.36	2.25	2.36	3.4	0.3	51.8
NG32	River Sandstone	Yesilirmak Basin/ Çarşamba Samsun	FA	2.65	2.60	2.64	2.0	1.9	--
			CA	2.64	2.59	2.63	0.9	0.5	21.0

Table 4.2 Chemical compositions of the standard cements [Cimsa, 2018; Bolu Cimento, 2018]

Chemical Composition (%)											
Type I											
CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	**Na_2O**	**K_2O**	Cl	IR	LOI	Na_2O_{eq}
64.05	20.31	5.38	3.02	1.45	3.31	**0.64**	**0.89**	0.013	1.28	1.69	**1.23**
Type II											
63.48	20.48	5.87	3.24	1.58	3.09	**0.51**	**0.65**	0.015	1.20	1.71	**0.94**

4.2 Test Methods

"RILEM AAR-2 Accelerated Mortar Bar Test" (AMBT) and "RILEM AAR-4.1 Concrete Prism Test" (CPT) methods were conducted to evaluate potential alkali reactivity of aggregates and to check the developed new methods for reliability of results. Test procedures of AMBT and CPT are shortly described as below.

4.2.1 Accelerated Mortar Bar Test (RILEM AAR-2)

RILEM AAR-2 Accelerated Mortar Bar Test is designed to evaluate swiftly concrete aggregates for AAR. Usage of this method is widespread due to completion of test in a short time meaning 14 days. The mortars prepared by fine aggregates are cast into 25 mm x 25 mm x 285 mm mortar bars (Figure 4.1).

Figure 4.1 Moulds

After removal from the moulds, length measurements of prepared samples are record as first length (*l*). The samples are firstly cured in a water at 80 °C for 1 day and the length measurement is taken with a tapeline and recorded as l_0. After that, the samples are stored at a soak solution with 1N NaOH at 80 ºC in a sealed container for 14 days and the length measurements are taken with a digital comparator as seen in Figure 4.2 (a). Expansion percentages of the samples are calculated regarding as 14-day length measurement (l_{14}) in accordance with the formula; $100x(l_{14}-l_0)/l$. 0.2% expansion limit is described in this method to determine reactivity of the aggregates. In addition, the aggregates showing expansion between 0.1% and 0.2% are approved as susceptible materials for AAR formation.

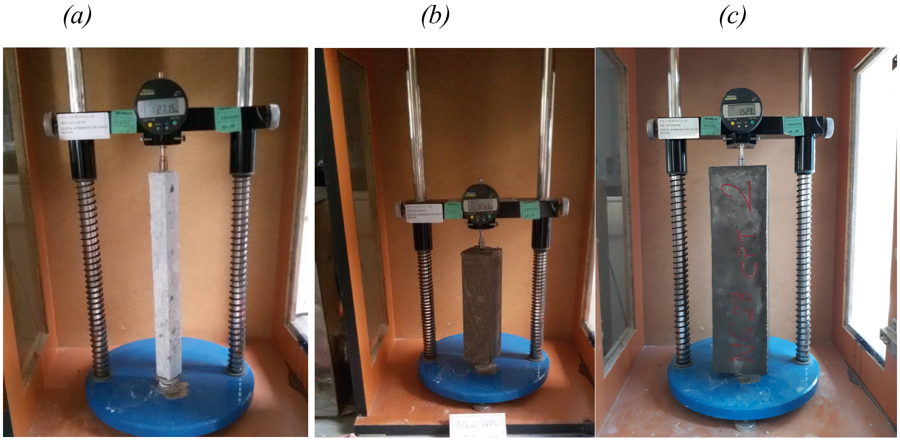


Figure 4.2 Length measurement of the specimens

Aggregate size distribution applied in the related standard are quantized in Table 4.3.

Table 4.3 Size distribution of the aggregates in AMBT

Sieve Size (mm)	**4**	**2**	**1**	**0.5**	**0.25**	**0.125**
Retained on Sieve (%) by mass	0	10	35	60	85	100

4.2.2 RILEM AAR-4.1 Concrete Prism Test (CPT)

RILEM AAR-4.1 is an accelerated CPT method taking only 20 weeks while ASTM C1293 long-term CPT takes 1 or 2 years. This method is applied by providing an exposure condition with 100% RH and at 60 ºC to examine both fine and coarse aggregates for AAR. Expansion limit for that period is 0.04% as it is 0.03% for 15 weeks. Many researches have proved that RILEM AAR-4 CPT method shows good correlation with field performance of aggregates, so it was designated as a reference method to check reliability of developed new methods in this experimental study.

RILEM AAR-4.1 CPT method specifies the mould dimension as 75±5mm x 75±5mm x 250±50 mm. Accordingly, 75 mm x 75 mm x 300 mm (Figure 4.1) concrete prisms were preferred, so their all sizes are 1.5 times more than those (50 mm x 50 mm x 200 mm) used in new method trials. Size distribution of test aggregates used for the CPT is in Table 4.4 that manifests usage of 40% fine and 60% coarse aggregates in concrete mixture. Size distribution of the aggregates is elaborated according to the specified interval in Table A1 of RILEM AAR-4.

Table 4.4 Size distribution of the aggregates in CPT

Sieve Size (mm)	**22.4**	**16.0**	**8.0**	**4.0**	**2.0**	**1.0**	**0.5**	**0.25**	**0.125**	**Pan**
Retained on Sieve (%) by mass	0	10	30	60	65	75	85	95	98	0

CHAPTER 5

EXPERIMENTAL STUDY AND DEVELOPMENT OF THE NEW METHOD

In this experimental study, different new method trials were performed to establish the most acceptable method for evaluation of aggregates in view of alkali reactivity. Experimental studies are divided into two part as preliminary and latter. New method trials were studied by using many solutions with different contents, different types of cement and modifying storage temperature. General principles of the new method trials will be mentioned and all process of the experimental study on development of a new method will be elaborately scrutinized in this chapter.

5.1 General Principles of the New Methods

5.1.1 Mix Design of Test Specimens

Material proportions of an example concrete mixture prepared for new method samples by mass are 28.6% fine aggregate, 42.9% coarse aggregate, 19.0% cement and 9.5% water. These proportions mean that 1 m^3 concrete compose of 1658 gr aggregate (746 g fine, 912 g coarse aggregate), 440 g cement and 220 g water. On the other hand, numerical values of water absorption, water content of aggregates may change material proportions according to equation 5.1. With data from Table 4.1, an example calculation for G1 aggregate is as follows and accordingly, mix design of 1 m^3 concrete is given in Table 5.1.

$W_{extra} = [(WA_{24} - W_c)/100] \times [(\text{quantity of the aggregate}) / (1+WA_{24} / 100)]$ Eqn. 4.1

Given data;

For fine G1 aggregates; water absorption (WA_{24}) = 2.0%, water content (W_c)=4.8%

For coarse G1 aggregates; water absorption (WA_{24}) = 1.1%, water content (W_c)=1.6

For fine aggregate,

$$W_{extra} = [(2.0 - 4.8) / 100] \times [(746) / (1 + 2.0 / 100)] \approx -0.7 \text{ kg (Formula 5.2)}$$

For coarse aggregate,

$$W_{extra} = [(1.1 - 1.6) / 100] \times [(912) / (1 + 1.1 / 100)] \approx -0.2 \text{ kg (Formula 5.3)}$$

Table 5.1 An example mix design of 1 m^3 concrete prepared by G1 aggregate

	Size (mm)	Size distribution (%)	Quantity (kg/m³)	Mass (%)
Coarse aggregate	4/16	55	912-(-0.7) = 912.7	39.4
Fine aggregate	0/4	45	746-(-0.2) =746.2	32.2
Cement	--	--	440	19.0
Water	--	--	220-0.7-0.2= 219.1	9.4
Total	--	100	2318	100

5.1.2 Size Distribution of Aggregates

In this experimental study, the maximum size of aggregate was specified as 16 mm unlike in standard RILEM AAR-4 CPT method (22.4 mm). Researchers have shown that limiting the maximum size of aggregate from 22.4 mm to 16 mm does not cause any significant impact on size of surface area exposed the reaction as much as grinding coarse aggregate to sand sized particles. In addition to reducing the maximum size of aggregate, proportions of fine and coarse aggregates were modified as 55% coarse and 45% fine instead of 60% coarse and 40% fine according to the specified interval in Table A1 of RILEM AAR-4. On the other hand, cumulative mass percentage of aggregate passing 16 mm must be specified as 100 due to modification of maximum aggregate size. Consequently, aggregate size distribution for development of a new method was designed by adhering to Table A1 in RILEM AAR-4 except 16-22.4 mm interval. Detailed grain size distribution of aggregates used in this new method study is explained as in Table 5.2 with recommended range for passed aggregate on Sieve (%) by mass in RILEM AAR-4.1 CPT method. In addition, it can be graphically seen in Figure 5.1.

Table 5.2 Grain size distribution of test aggregates

Sieve size (mm)	**16**	**11.2**	**8**	**4**	**2**	**1**	**0.5**	**0.25**	**0.125**	**Pan**
Retained on sieve (%) by mass	0	20	30	55	67	79	89	96	99	100
Passed on sieve (%) by mass	100	80	70	45	33	21	11	4	1	0
Recommended range for passed on sieve (%) by mass	75-95	--	45-70	35-55	25-45	20-35	10-25	4-12	1-8	0

The main objective of these modifications is to increase workability of concrete mixture, so application of the method to be designed will be more practical and preparation of test specimens in laboratory conditions will be easier as compared with the standard CPT method.

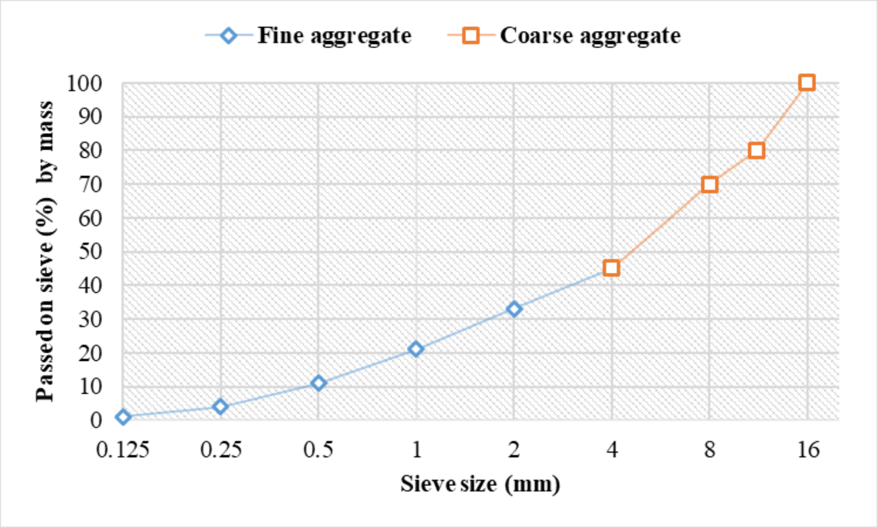


Figure 5.1 Size distribution of test aggregates

5.1.3 Exposure Conditions

To get over alkali leaching in 50 mm x 50 mm x 200 mm concrete prisms, six new soak solutions for test specimens prepared by G*i* aggregates were designed for the preliminary experimental study as seen in Table 6.1. In this phase of this experimental study, seven new methods developed by using NaOH and different chloride based de-icers in different ratios were applied on 8 types of aggregates and the results were also compared with standard AMBT and CPT methods.

Figure 5.2 Test specimens for study of developing a new method

In addition to these seven new methods, two new methods developed by the same way were conducted on these preliminary aggregates (G*i*) and also new 24 types of aggregate (NG*i*) to provide best correlation with the test results of CPT. All test specimens prepared for development of a new method were stored at a closed sealed container to prevent alkali leaching from concrete prisms and exposed to storage temperature of 60 ºC (Figure 5.2).

5.1.4 Test Temperature

Temperature of 80°C is commonly used in accelerated mortar bar test methods to speed process of the reaction. Many studies have revealed that such a high temperature does not reflect real environment condition, so it may raise the possibility of false negative/positive test results. Therefore, temperature of 60°C is more reasonable for storage environment than 80°C if period of the test and real environment condition are considered. 60°C is not a temperature that cannot be seen in nature because concrete structure may be subjected to this temperature in some places, for example, around the Ecuadorian of the world and very close temperatures are also observed in Turkey. Temperature of 60°C can accelerate the reaction in an enough degree to complete the test for AAR in a reasonable time though it does not considerably disrupt the reaction kinetics. Many investigations have claimed that this temperature has not an important negative effect on the hydration productions. On the other hand, temperature of 60°C may cause unsteady phases of sulfo-aluminate and surplus sulphate accumulation in pore solution. However, this undesirable sulphate may not be seen as a significant problem because sufficient alkalis are provided by NaOH and chloride based salts used in the soak solutions prepared for designing new methods. In short, this temperature can provide short test period without disrupting the main process of the reaction and the products of the concrete mixtures.

5.1.5 Dimension of Test Specimens

The reasons for selection of 50 mm x 50 mm x 200 mm narrowed concrete prisms to prepare test specimens are those (1) the preparation of test specimens is more practical than in the standard CPT method; (2) accessibility of the narrowed concrete prisms with pins is more easier because they are commonly used in “TS EN 1367-4 determination of drying shrinkage test” which must be applied to check the conformity of aggregates to the standard “TS 706 EN 12620- Concrete Aggregates” in Turkey; (3) all sizes of 50 mm x 50 mm x 200 mm concrete prisms are 1.5 times smaller than 75 mm x 75 mm x 300 mm used in the standard CPT method, so the correlation study between new developed test methods and CPT method may be more rational. Examples for dimensions of the specimens used in this study are given in Figure 5.3.

Figure 5.3 Dimensions of test specimens

5.2 Process of Experimental Study

The general framework of experimental studies is also divided into two parts as preliminary and latter experimental study. Chloride based de-icers were used as fundamental materials to develop a new method. Different soak solutions, narrowed concrete prism (50 mm x 50 mm x 200 mm), modified temperature in storage and measurement, different size distribution of aggregate were extensively applied to achieve the aim of this study. Table 5.3 explains the experimental program in a general framework. In first step, the new methods were developed by using soak solutions which were (1) 0.3N NaOH+1% NaCl + 1% $CaCl_2$, (2) 0.3N NaOH+2% NaCl, (3) 0.3N NaOH+2% $CaCl_2$, (4) 0.3N NaOH+2% $MgCl_2$. After data obtained from previous test methods, it needed that these aggregates were storage at new 2 soak solutions which were (5) 0.35N NaOH+1% $CaCl_2$, (6)0.35N NaOH+2% $CaCl_2$ to design the best appropriate test method having excellent correlation with standard test methods considering the results of first trials. PNM1 and PNM2 were considered as two separate methods though the solution prepared for them was the same. The reason for this separation was that the lengths of the test specimens in PNM2 were measured at 20 ºC while the length measurements of the other specimens were performed at 60 ºC. After completion of preliminary experimental study, one new method providing better correlation with the CPT method is modified and so last two new methods were developed. As main part of the latter experimental study, these two methods were applied to all 32 types of aggregates. Measurement processes for expansion rate are same in all method as specified in the standard test methods. The test aggregates are originated from different resources and have different petrographic and mineralogical structures as seen in Table 4.1. Furthermore, petrographic and mineralogical evaluation were examined on most types of aggregates to detect the effects of these properties on AAR according to the test result reports obtained by different institutions or laboratories.

General specifications of all new methods about mix design of test specimens, size distribution of aggregates, storage ambient, test temperature and dimension of test specimens are elaborately discussed in chapter 4. In this experimental study, the number of prepared specimens are briefly counted as (1) 6 (25 mm x 25 mm x 285 mm) mortar bars, 3 (75 mm x 75 mm x 300 mm) concrete prisms, and 18 (50 mm x 50 mm x 200 mm) narrowed concrete prisms for 8 types of aggregates used in the preliminary experimental study; (2) 3 (25 mm x 25 mm x 285 mm) mortar bars, 3 (75 mm x 75 mm x 300 mm) concrete prisms, and 6 (50 mm x 50 mm x 200 mm) narrowed concrete prisms for 32 types of aggregates used in the latter experimental study. As an example, all specimens prepared for one type of aggregate are given in Figure 5.4 in the preliminary experimental study.

Figure 5.4 All specimens prepared for G1 aggregate

Table 5.3 Experimental Program

Test Method	**Test Period**	**Soak Solution**	**Mould Dimension**	**Storage Temp.**	**Cement Type**	**Notation**
Preliminary Experimental Study						
RILEM AAR-2/ Mortar Bar Test	14 days	**1N NaOH**	25x25x285 (mm*mm*mm)	80 ºC	Type I	AMBT
Modified RILEM AAR-2/ Mortar Bar Test	14 days	**1N NaOH**	25x25x285 (mm*mm*mm)	60 ºC	Type I	MABT
RILEM AAR-4.1/Concrete Prism Test	20 weeks	**No Soak Solution 100%RH**	75x75x300 (mm*mm*mm)	60 ºC	Type I	CPT
Pilot New Method-1	28 days	**0.3N NaOH** **1% NaCl** **1% $CaCl_2$**	50x50x200 (mm*mm*mm)	60 ºC 20 ºC (meas.)	Type I	PNM1
Pilot New Method-2	28 days	**0.3N NaOH** **1% NaCl** **1% $CaCl_2$**	50x50x200 (mm*mm*mm)	60 ºC	Type I	PNM2
Pilot New Method-3	28 days	**0.3N NaOH** **2% NaCl**	50x50x200 (mm*mm*mm)	60 ºC	Type I	PNM3
Pilot New Method-4	28 days	**0.3N NaOH** **2% $CaCl_2$**	50x50x200 (mm*mm*mm)	60 ºC	Type I	PNM4
Pilot New Method-5	28 days	**0.3N NaOH** **2% $MgCl_2$**	50x50x200 (mm*mm*mm)	60 ºC	Type I	PNM5
Pilot New Method-6	28 days	**0.35N NaOH** **1% $CaCl_2$**	50x50x200 (mm*mm*mm)	60 ºC	Type I	PNM6
Pilot New Method-7	28 days	**0.35N NaOH** **2% $CaCl_2$**	50x50x200 (mm*mm*mm)	60 ºC	Type I	PNM7
Latter Experimental Study						
Designed New Method-1	28 days	**0.35N NaOH** **1.25% $CaCl_2$**	50x50x200 (mm*mm*mm)	60 ºC	Type I	DNM1
Designed New Method-2	28 days	**0.35N NaOH** **1% $CaCl_2$**	50x50x200 (mm*mm*mm)	60 ºC	Type II	DNM2

CHAPTER 6

RESULTS OF EXPERIMENTAL STUDY AND ANALYSIS OF DATA

In this chapter, the results obtained from both preliminary and latter experimental studies are analysed to obtain a good correlation between the developed new methods and the CPT method. Analyses for the process of experimental study are as follows.

6.1 Preliminary Experimental Study

In first step of this experimental study, seven pilot new test methods were performed on 8 types of aggregates which are different types of sandstones or limestones from basins of Kizilirmak River, Ceyhan River, Coruh River and a granite from Bergama/Izmir in Turkey (Table 4.1) in order to examine the correlation between the new test methods and CPT method that is a good indicative for field performance of aggregates.

6.1.1 Expansion Limits

Expansion limits should be definitely remembered before proceeding to the evaluation of test results because available test methods are considered as control methods for the proposed new methods. In “RILEM AAR-2 Accelerated Mortar-Bar Test Method for Aggregates” and in “RILEM AAR-4.1 Test Method for Aggregate Combinations Using Concrete Prisms” expansion limits are as in Tables 6.1 and 6.2.

Table 6.1 Expansion limits in the AMBT method

Expansion (percent) for 14 days	**Aggregate Reactivity**
0.0 – 0.1	Non-reactive
0.1– 0.2	Suspicious
> 0.2	Potentially reactive

Table 6.2 Expansion limits in the CPT method

Expansion (percent)	Aggregate Reactivity
0.0 – 0.03 for 15 weeks	Non-reactive
0.0 – 0.04 for 20 weeks	Non-reactive
> 0.03 for 15 weeks	Potentially reactive
> 0.04 for 20 weeks	Potentially reactive

6.1.2 Analysis Method

Regression analysis and calculation of correlation coefficient (R^2) are specified fundamental ways to analyse the data obtained from the experimental studies. In the preliminary experimental study, comparing the pilot new methods data with the CPT data needs to create a time mapping as follow to establish a correlation between them.

i. t ≡7 days for PNMs ≡ 28 days for CPT.
ii. t ≡14 days for PNMs ≡ 10 weeks for CPT.
iii. t ≡21 days for PNMs ≡ 15 weeks for CPT.
iv. t ≡28 days for PNMs ≡ 20 weeks for CPT.

In the time mapping, five times the expansion days of PNMs are equalized to those of CPT method. However, this equalization is only not valid for 7-day expansion of PNMs because the rates of expansion at the beginning of the reaction may vary in long-term and short-term methods.

Regression analysis is an analysis method used to measure the relationship between two or more variables. It allows estimations of unknown future events from known findings. Regression develops a forecast equation by using the relationship between dependent and independent variables and the concept of linear curve. The univariate regression model describing the linear relationship between the dependent variable and the independent variable is $Y = aX + b$ where Y= the value of the dependent variable; X= the value of the independent variable; a= the slope of the regression line; b= intersection value of regression line (constant value) [Foley, B., 2018]. The adaptation processes of this model to this experimental study is as follows: the linear regression line has a formula which is $Y = aX_i + b$, where X is expansion in PNMs, Y is expansion of CPT and a&b are variable coefficients according to test data.

Correlation is a statistical technique that allows us to quantify whether there is a relationship between two or more variables. If there is a relationship, the amount and direction of this relationship can be determined by this technique. The degree of correlation between the two variables is called the correlation coefficient (R^2). The calculated correlation coefficient is between -1 and +1. It cannot be less than -1 and greater than + 1. If the correlation is positive (+), the variables are changed in the same direction. The correlation coefficient + is that the two variables are in the same direction, whereas the negative (-) indicates an inverse relationship between the two variables. If the increase or decrease in the variables is not interconnected, the correlation becomes zero. This also means that there is no relationship between variables. When the correlation is 1, the relationship is perfect and when it is 0 there is no relationship [Foley, 2018].

Correlation coefficient:

i. As we get closer to +1.00, the relationship between two variables increases in the same direction. One of the variables increases and the other increases.
ii. As we approach -1.00, the relationship between the two changes increases in the opposite direction. One of the variables increases while the other decreases.
iii. The closer to 0.00 the relationship between the two variables decrease.

The formula of correlation coefficient (R^2) is defined as below:

$$R^2 = \sum (x_i - x_m) * (y_i - y_m) / \left[\sum (x_i - x_m)^2 * \sum (y_i - y_m)^2\right]^{1/2} \qquad \text{Eqn. 6.1}$$

where: x_i = the x-sample values, x_m= the mean of the x-sample values, y_i = the y-sample values, y_m= the mean of the y-sample values.

In light of this information, parameters for excellent correlation between the data obtained from PNMs and CPT is explained as below:

$Y=X_i$ that is a=1; b=0 and $R^2= |1|$ (ideal equation)

In order to decide the best appropriate test method, it is very important to take into consideration the values of "a", "b" coefficient and R^2 on integrated basis. In this study, evaluation criteria have been specified as the priority criteria that is value of "a" closest to 1, next criteria value of R^2 closest to 1 and b closest to 0 to design ideal method. To accept R^2 as the primary parameter may mislead because it is possible that the time mapping cannot meet the temporal equation fully. In addition, closeness of expansion data in PNMs for 28 days and in CPT for 20 weeks is very important criteria in choice of the best method.

6.1.3 Test Results of Preliminary Experimental Study

The test results of preliminary experimental study are in Table 6.3 on average of three samples for all each method. The Appendix shows the raw data of all samples in detail.

Table 6.3 Test results of preliminary experimental study

G1 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.219	0.332	0.395	0.607			
MABT	Av:	0.059	0.164	0.238	0.282			
CPT	Av:	0.005	0.012	0.019	0.023	0.041	0.048	0.051
PNM1	Av:	0.026	0.051	0.060	0.068			
PNM2	Av:	0.018	0.034	0.054	0.063			
PNM3	Av:	0.028	0.081	0.132	0.159			
PNM4	Av:	0.008	0.025	0.037	0.044			
PNM5	Av:	0.008	0.011	0.019	0.024			
PNM6	Av:	0.012	0.025	0.042	0.053			
PNM7	Av:	0.013	0.023	0.039	0.046			

Table 6.3 continued

G2 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.039	0.070	0.097	0.122			
MABT	Av:	0.021	0.030	0.045	0.055			
CPT	Av:	0.001	0.003	0.004	0.007	0.014	0.016	0.018
PNM1	Av:	0.012	0.016	0.022	0.030			
PNM2	Av:	0.004	0.005	0.018	0.025			
PNM3	Av:	0.018	0.024	0.030	0.036			
PNM4	Av:	0.002	0.005	0.011	0.014			
PNM5	Av:	0.002	0.003	0.007	0.009			
PNM6	Av:	0.004	0.007	0.012	0.015			
PNM7	Av:	0.002	0.008	0.012	0.016			
G3 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.113	0.194	0.242	0.274			
MABT	Av:	0.055	0.073	0.104	0.141			
CPT	Av:	0.004	0.007	0.009	0.011	0.017	0.021	0.023
PNM1	Av:	0.006	0.015	0.023	0.031			
PNM2	Av:	0.004	0.011	0.018	0.025			
PNM3	Av:	0.019	0.030	0.042	0.054			
PNM4	Av:	0.002	0.009	0.019	0.031			
PNM5	Av:	0.001	0.006	0.010	0.013			
PNM6	Av:	0.003	0.010	0.022	0.034			
PNM7	Av:	0.005	0.011	0.024	0.034			
G4 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.337	0.454	0.478	0.493			
MABT	Av:	0.196	0.282	0.328	0.353			
CPT	Av:	0.019	0.030	0.035	0.040	0.047	0.061	0.076
PNM1	Av:	0.021	0.048	0.083	0.112			
PNM2	Av:	0.018	0.021	0.067	0.106			
PNM3	Av:	0.028	0.074	0.121	0.184			
PNM4	Av:	0.016	0.025	0.058	0.081			
PNM5	Av:	0.004	0.011	0.018	0.030			
PNM6	Av:	0.019	0.027	0.061	0.083			
PNM7	Av:	0.017	0.023	0.050	0.068			
G5 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.209	0.353	0.379	0.421			
MABT	Av:	0.112	0.145	0.185	0.238			
CPT	Av:	0.012	0.017	0.022	0.027	0.036	0.044	0.049
PNM1	Av:	0.016	0.021	0.042	0.070			
PNM2	Av:	0.007	0.014	0.025	0.031			

Table 6.3 continued

PNM3	Av:	0.020	0.028	0.039	0.061			
PNM4	Av:	0.009	0.011	0.028	0.038			
PNM5	Av:	0.003	0.007	0.014	0.021			
PNM6	Av:	0.007	0.013	0.030	0.040			
PNM7	Av:	0.009	0.013	0.023	0.033			
G6 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.172	0.267	0.344	0.378			
MABT	Av:	0.091	0.124	0.146	0.214			
CPT	Av:	0.012	0.018	0.020	0.022	0.026	0.033	0.037
PNM1	Av:	0.016	0.024	0.037	0.055			
PNM2	Av:	0.003	0.016	0.020	0.041			
PNM3	Av:	0.020	0.032	0.050	0.076			
PNM4	Av:	0.003	0.010	0.021	0.032			
PNM5	Av:	0.002	0.007	0.018	0.022			
PNM6	Av:	0.006	0.013	0.026	0.038			
PNM7	Av:	0.002	0.014	0.024	0.034			
G7 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.210	0.347	0.374	0.413			
MABT	Av:	0.098	0.139	0.175	0.232			
CPT	Av:	0.009	0.015	0.019	0.027	0.034	0.037	0.046
PNM1	Av:	0.014	0.018	0.039	0.074			
PNM2	Av:	0.009	0.016	0.025	0.034			
PNM3	Av:	0.022	0.030	0.043	0.066			
PNM4	Av:	0.009	0.012	0.030	0.040			
PNM5	Av:	0.002	0.007	0.013	0.019			
PNM6	Av:	0.014	0.018	0.034	0.049			
PNM7	Av:	0.006	0.012	0.024	0.042			
G8 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.124	0.216	0.264	0.296			
MABT	Av:	0.061	0.085	0.103	0.136			
CPT	Av:	0.005	0.009	0.012	0.016	0.021	0.027	0.032
PNM1	Av:	0.011	0.017	0.028	0.042			
PNM2	Av:	0.010	0.015	0.028	0.041			
PNM3	Av:	0.022	0.030	0.042	0.054			
PNM4	Av:	0.004	0.011	0.022	0.038			
PNM5	Av:	0.001	0.005	0.008	0.016			
PNM6	Av:	0.007	0.012	0.028	0.040			
PNM7	Av:	0.005	0.014	0.031	0.043			

6.1.4 Analysis of the Results

A type of aggregate called G1 aggregate was detected in all methods and found as potentially reactive because expansion percentages overly exceed the limits defined in standard test methods. 14-day expansion of G1 aggregate in AMBT is measured as 0.332% over the expansion limit (0.2%) and it also shows 0.051% expansion exceeding the expansion limit (0.04%) for 20 weeks in CPT as seen in Figure 6.1. Most investigations on potential alkali-reactivity reveal that long-term CPT is acceptable as decisive test method to determine whether the aggregate is reactive or not, so it is logical that this method primarily can be referenced to evaluate the test results of the pilot new methods. Experimental studies on G1 aggregate show that PNM6 seems to provide best correlation with CPT data among all methods (Figure 6.2) if compared the 28-day expansion of PNM6 that is 0.053% and 20-week expansion of CPT that is 0.051% as seen in Table 6.3.

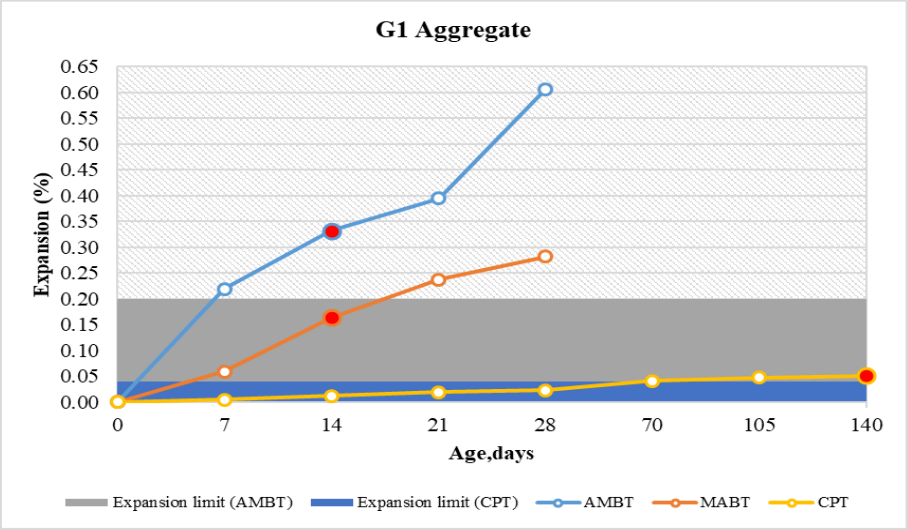


Figure 6.1 Expansion results of G1 aggregate according to the standard test methods

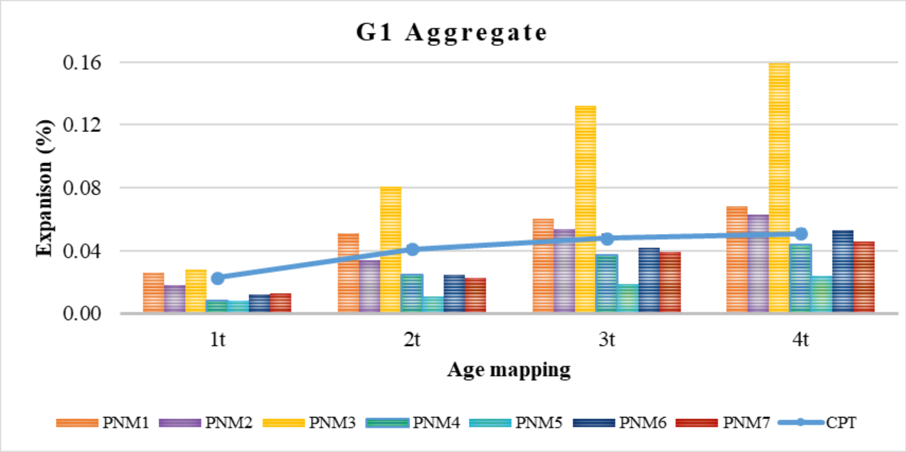
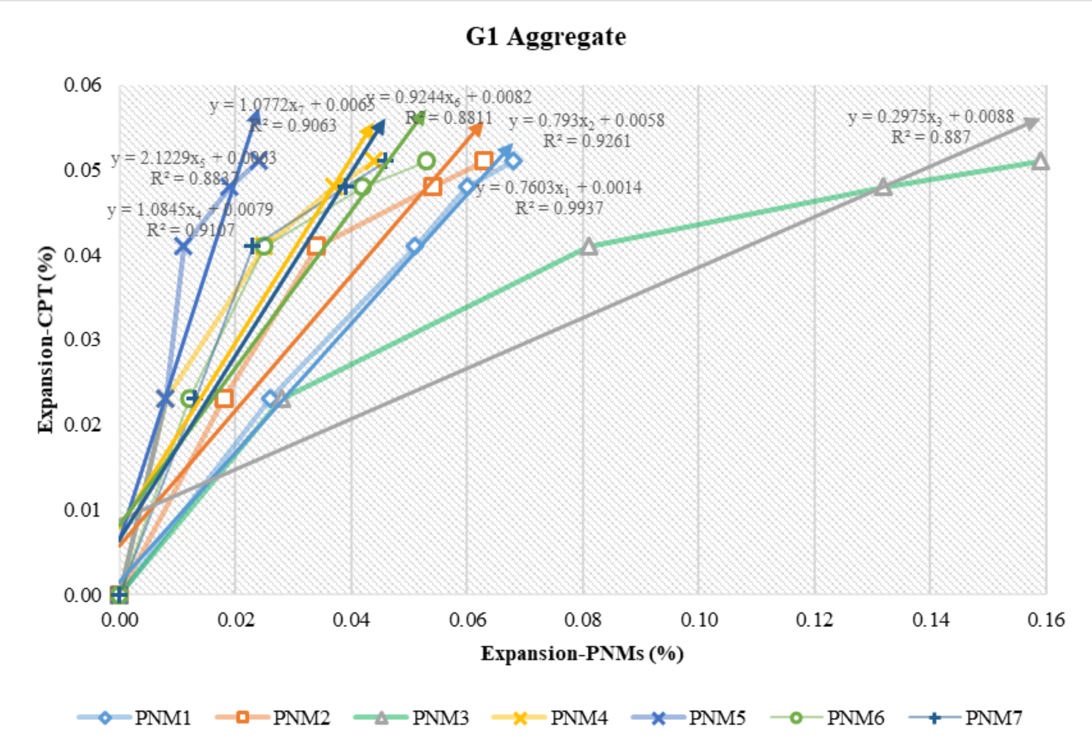


Figure 6.2 Comparison of pilot new methods data with CPT data for G1 aggregate

On the other hand, regression analysis (Figure 6.3) for G1 aggregate data reveals that PNM7 provides best correlation for all test period because the line equation, $Y=1.0772X_7+0.0065$; ($R^2=0.9063$), is most compatible with ideal equation, $Y=X$. Only if considering correlation coefficient ($R^2=0.9937$), PNM1 may be conceivable but Figure 6.3 shows definitively poor correlation with CPT data. PNM4 gives an equation $Y=1.0845X_4+0.0079$ ($R^2=0.9107$), so it can be remarkable. If comparing with PNM7 equation, PNM4 equation has a little better correlation in view of R^2 coefficient but better for “a” and “b” coefficients. For all these parameters, PNM7 can be assessable as the best method for G1 aggregate.



Method	**Regression Line Equation ($Y=aX_i+b$)**	**Correlation Coefficient (R^2)**
PNM1	$Y=0.7603X_1+0.0014$	0.9937
PNM2	$Y=0.7930X_2+0.0058$	0.9261
PNM3	$Y=0.2975X_3+0.0088$	0.8870
PNM4	$Y=1.0845X_4+0.0079$	0.9107
PNM5	$Y=2.1229X_5+0.0063$	0.8837
PNM6	$Y=0.9244X_6+0.0082$	0.8811
PNM7	**$Y=1.0772X_7+0.0065$**	**0.9063**

Figure 6.3 Regression analysis of pilot new methods data and CPT data for G1 aggregate

Table 6.3 including data obtained from the tests on G2 aggregate shows clearly that a type of non-reactive granite was examined in this part of the study. G2 aggregate shows 0.070% expansion for 14 days in AMBT and 0.018 expansion for 20 weeks in CPT. This data states that G2 aggregate expanded noticeably under expansion limits of both AMBT and CPT methods (Figure 6.4). It can even be seen as not suspicious material for alkali-reactivity due to 0.018% lower expansion than the related limit (0.1% for 14 days) in the AMBT.

Regression analysis on the data of the expansion results of G2 aggregate reveals that PNM7 has best correlation with CPT data if observing the expansion percent as period of both tests completed. 20-week expansion in CPT is 0.018%; 28-day expansion in PNM7 is 0.016%, which are very closer to each other as seen in Figure 6.5. In the same line, regression analysis of the results in Figure 6.6 indicates that PNM7 is observed as the best method because value of "a" coefficient in the regression line equation, $Y=1.0491X_7+0.0030$, is closer than that in the other methods though it has not the best correlation coefficient value $R^2=0.8965$.

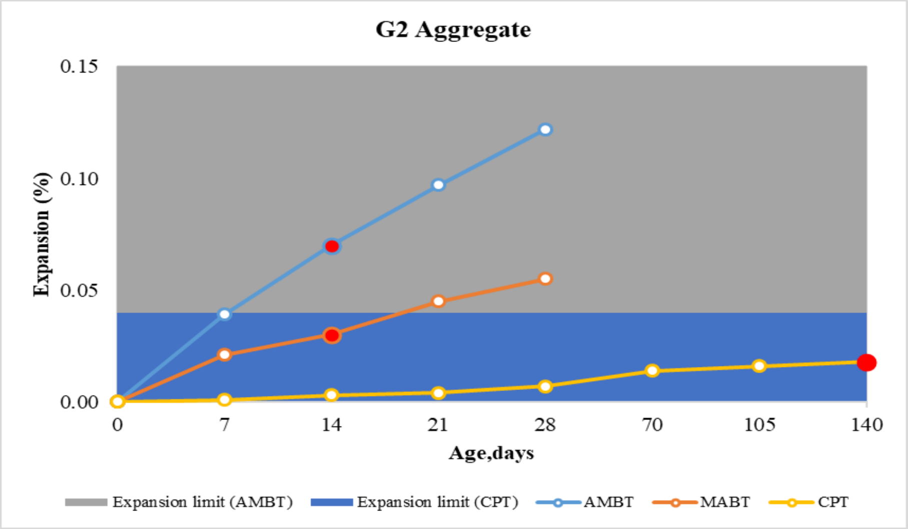


Figure 6.4 Expansion results of G2 aggregate according to the standard test methods

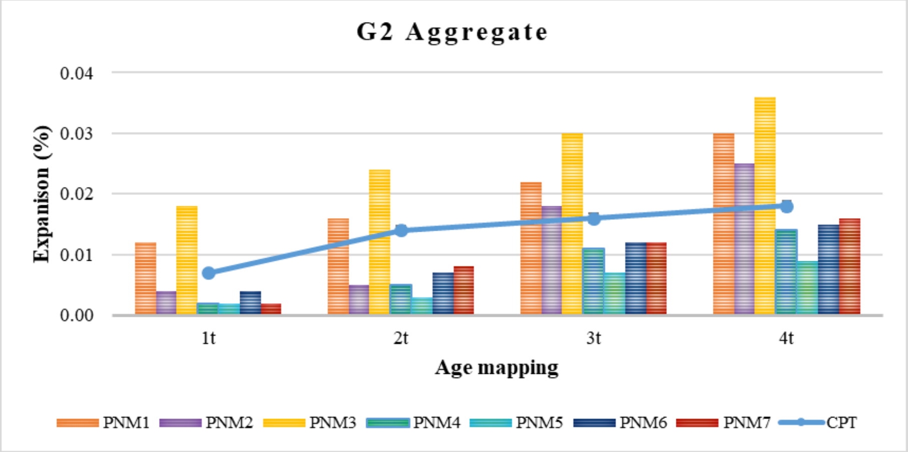
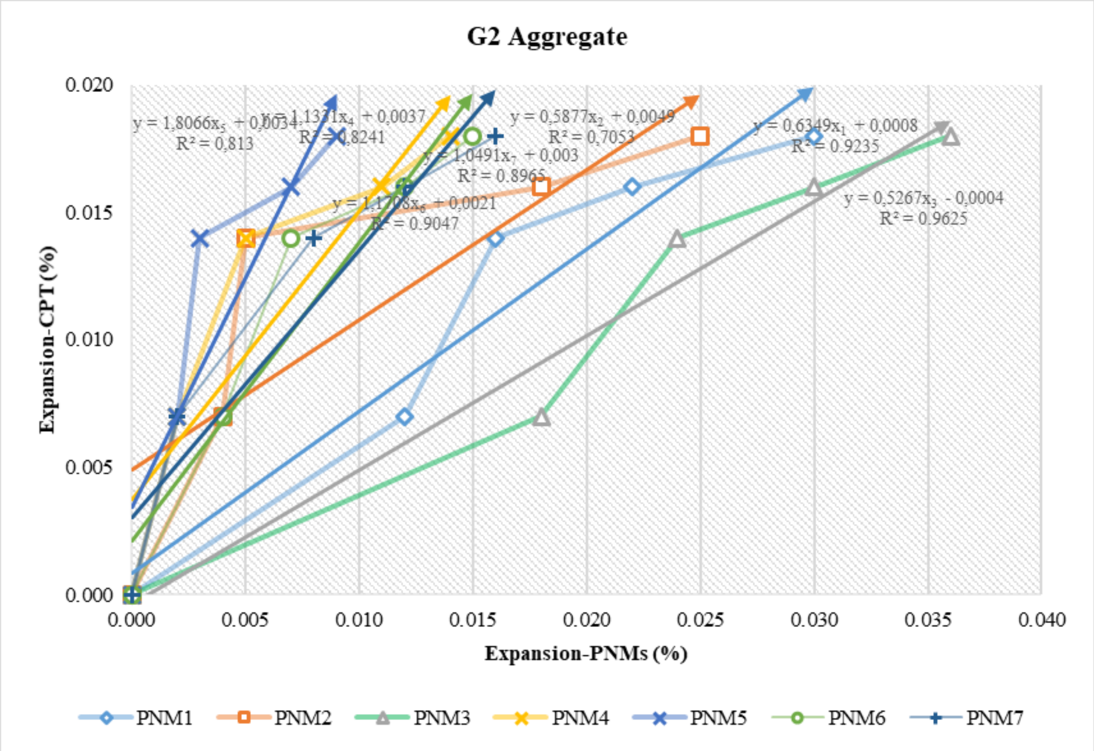


Figure 6.5 Comparison of pilot new methods data with CPT data for G2 aggregate



Method	Regression Line Equation (Y=aX$_i$+b)	Correlation Coefficient (R^2)
PNM1	Y=0.6349X$_1$+0.0008	0.9235
PNM2	Y=0.5877X$_2$+0.0049	0.7053
PNM3	Y=0.5267X$_3$-0.0004	0.9625
PNM4	Y=1.1331X$_4$+0.0037	0.8241
PNM5	Y=1.8066X$_5$+0.0034	0.8130
PNM6	Y=1.1708X$_6$+0.0021	0.9047
PNM7	**Y=1.0491X$_7$+0.0030**	**0.8965**

Figure 6.6 Regression analysis of pilot new methods data and CPT data for G2 aggregate

According to data from AMBT in Table 6.3, G3 aggregate is not precisely described as reactive because average expansion of G3 aggregate is 0.192 % that a bit lower than the limit, 0.2% for 14 days, so it can be described a suspicious material. Figure 6.7 indicates that G3 aggregate can be defined as a non-reactive material because 20-week expansion of G3 aggregate detected by CPT method is 0.023%, lower than the expansion limit, 0.04%.

Comparing the expansion results of CPT for 20 weeks and PNMs for 28 days, G3 aggregate tested by PNM2 shows 0.025% expansion so this method can be seen as the most suitable method according to the latest results (Figure 6.8).

Regression analysis data of G3 aggregate does not point out a good correlation between PNMs and CPT but only PNM2 shows the best correlation because it has regression line equation of $Y=0.8441X_2+0.0046$ that may be most ideal (Figure 6.9). Correlation coefficient (R^2) is accepted as an evaluation criterion but not the primary parameter due to possible lack of the time mapping. The truth of this logic has been proved with an impressive example that PNM3 provides the best correlation coefficient ($R^2=0.9567$) while it shows very poor correlation in view of the regression line equation, $Y=0.4349X_3-0.0018$, far from the ideal equation.

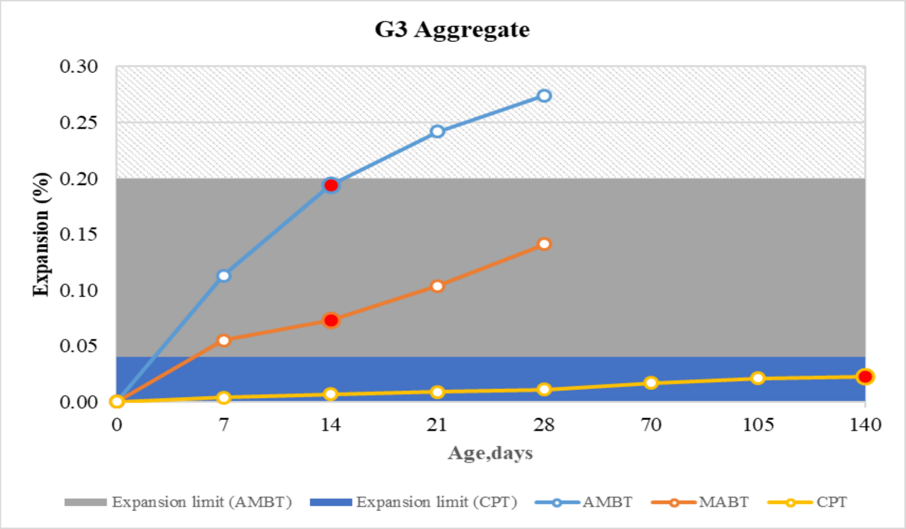


Figure 6.7 Expansion results of G3 aggregate according to the standard test methods

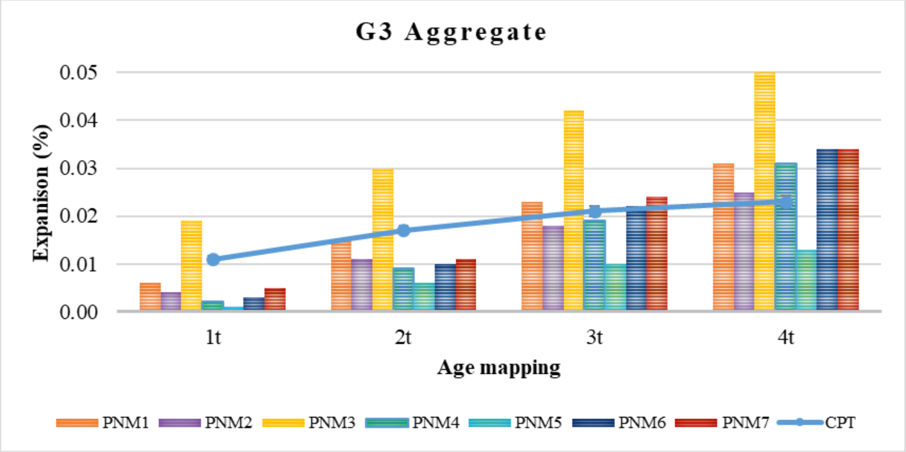
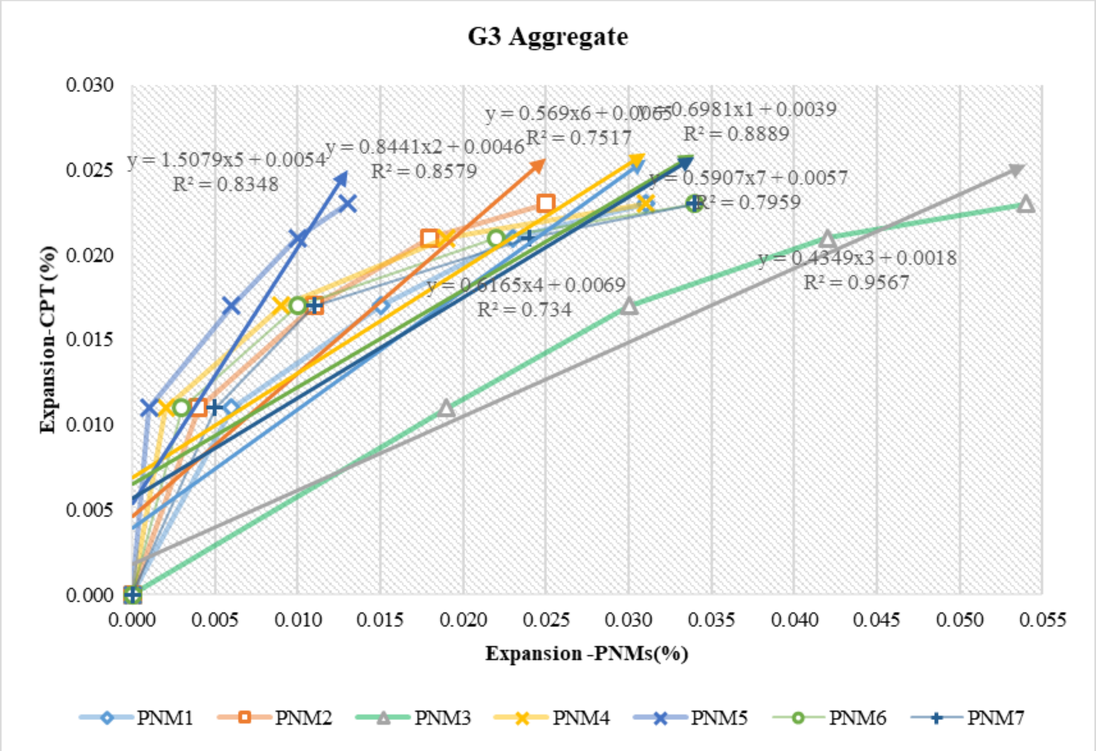


Figure 6.8 Comparison of pilot new methods data with CPT data for G3 aggregate



Method	Regression Line Equation (Y=aX+b)	Correlation Coefficient (R^2)
PNM1	$Y=0.6981X_1+0.0039$	0.8890
PNM2	**$Y=0.8441X_2+0.0046$**	**0.8579**
PNM3	$Y=0.4349X_3-0.0018$	0.9567
PNM4	$Y=0.6165X_4+0.0069$	0.7340
PNM5	$Y=1.5079X_5+0.0054$	0.8130
PNM6	$Y=0.5690X_6+0.0054$	0.8348
PNM7	$Y=0.5907X_7+0.0057$	0.7959

Figure 6.9 Regression analysis of pilot new methods data and CPT data for G3 aggregate

Data in Table 6.3 reflects that G4 aggregate is exactly reactive type by virtue of 0.454 % expansion in AMBT for 14 days and 0.076% expansion in CPT for 20 weeks (Figure 6.10).

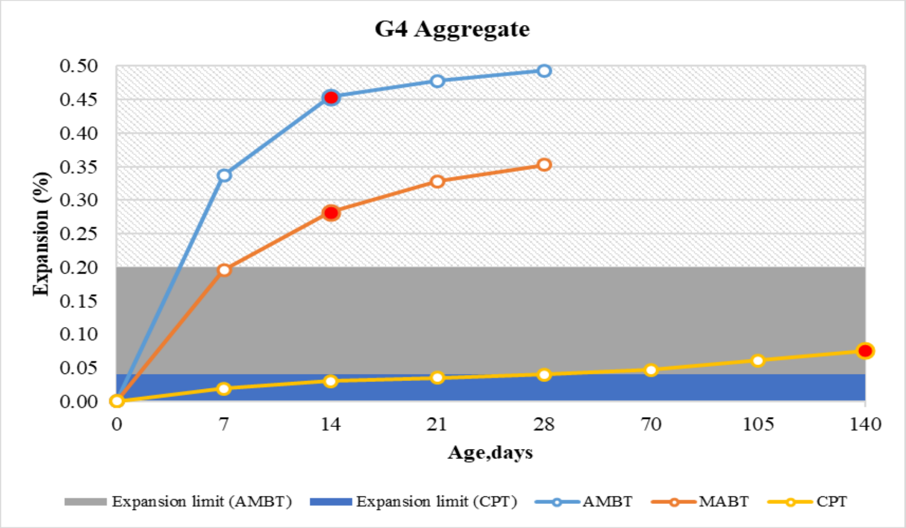


Figure 6.10 Expansion results of G4 aggregate according to the standard test methods

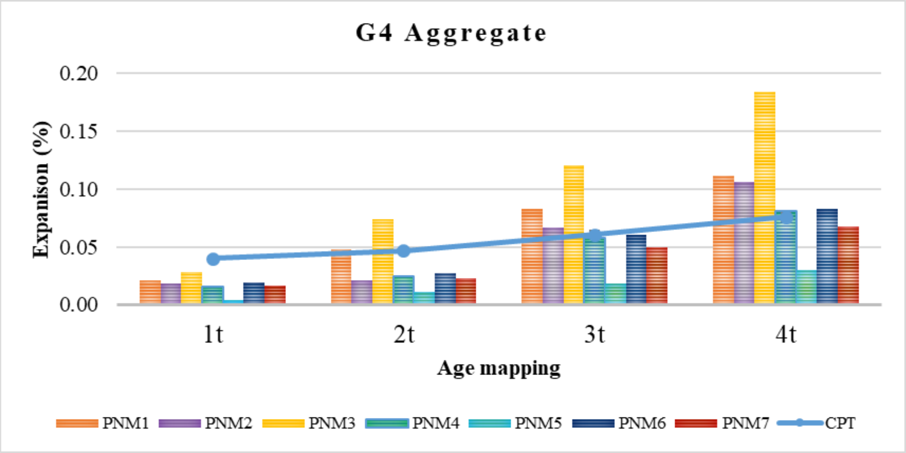
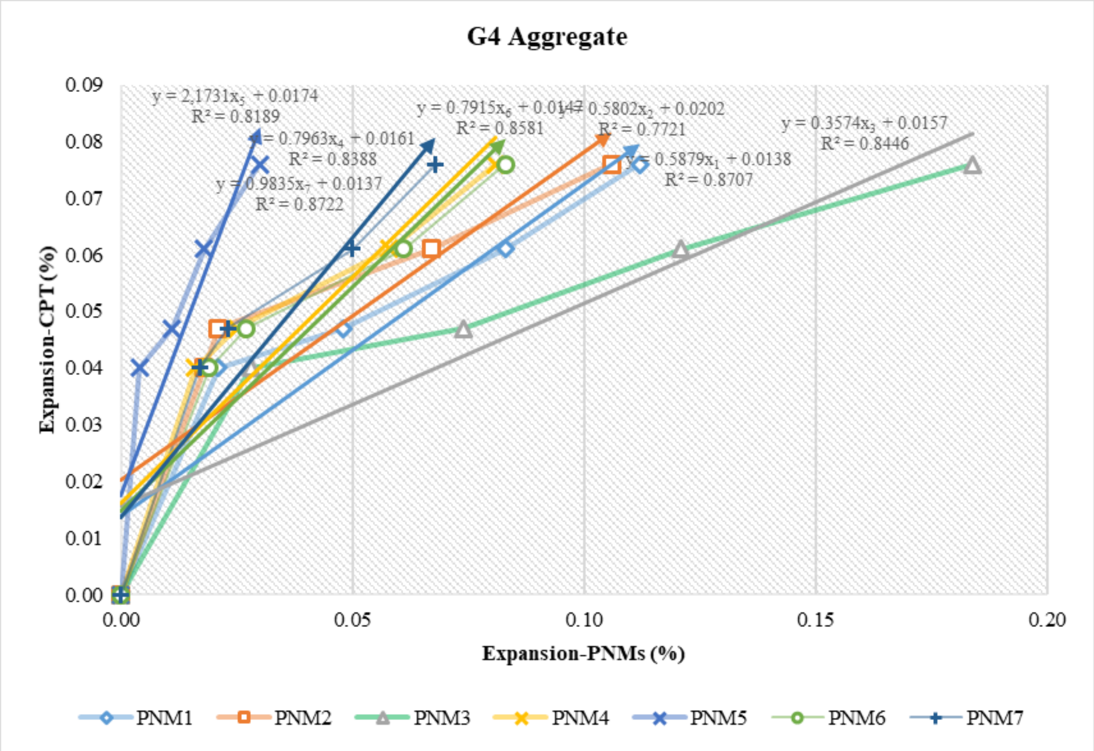


Figure 6.11 Comparison of pilot new methods data with CPT data for G4 aggregate

The expansion rates exceed the limits largely for both standard methods as clearly seen on Figure 6.10. After 28 days, the most compatible result with that in CPT

(0.076% expansion for 20 weeks) is provided by PNM2 method applied on G4 aggregate showing 0.081% expansion. Moreover, G4 aggregate shows 0.282% expansion for 14 days even in MABT that applied at 60 ºC. This expansion is a very high rate because most reactive aggregates even does not expand so much (Figure 6.11).

Moreover, expansion rates in PNM6 (0.083%) and in PNM7 (0.068%) may be considered as another closer values to one in CPT. On the other hand, if analysing regression line equation on Figure 6.12, PNM7 has a perfect equation, $Y=0.9835X_7+0.0137$, in view of only "a" coefficient value of 0.9835 but no good indicator of "b" and R^2 like in all other method. As considering all parameters, PNM7 is determined as the best one and PNM2, PNM4 can be seen alternative methods for G4 aggregate.



Method	Regression Line Equation (Y=aX+b)	Correlation Coefficient (R^2)
PNM1	$Y=0.5879X_1+0.0138$	0.8707
PNM2	$Y=0.5802X_2+0.0202$	0.7721
PNM3	$Y=0.3574X_3+0.0157$	0.8446
PNM4	$Y=0.7963X_4+0.0161$	0.8388
PNM5	$Y=2.1731X_5+0.0174$	0.8130
PNM6	$Y=0.7915X_6+0.0147$	0.8581
PNM7	$\mathbf{Y=0.9835X_7+0.0137}$	**0.8722**

Figure 6.12 Regression analysis of pilot new methods data and CPT data for G4 aggregate

Figure 6.13 shows that G5 aggregate is a typical reactive sandstone that may be risky for usage in concrete structure because it expands over the expansion limits of both AMBT and CPT methods. G5 aggregate shows 0.353% expansion for 14 days in AMBT and 0.049% expansion for 20 weeks in CPT.

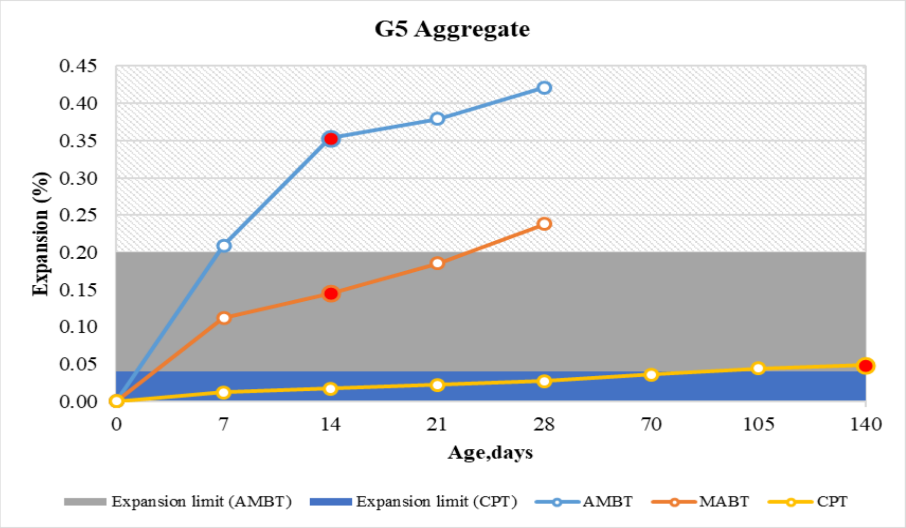


Figure 6.13 Expansion results of G5 aggregate according to the standard test methods

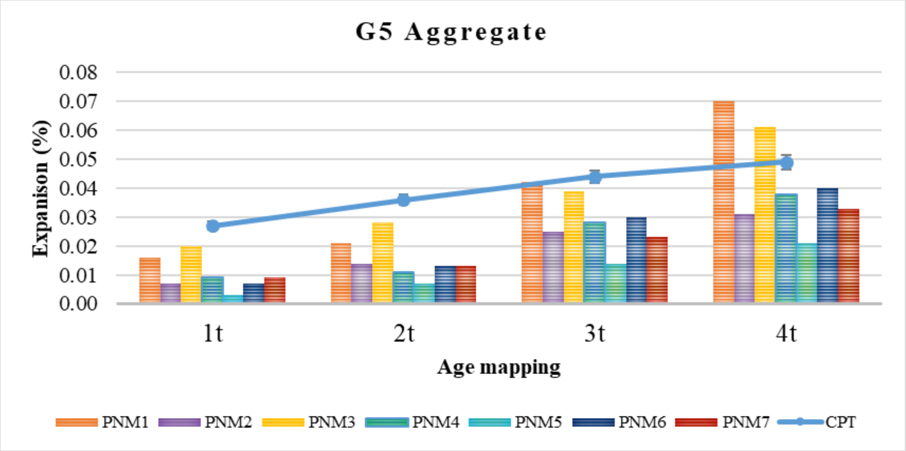
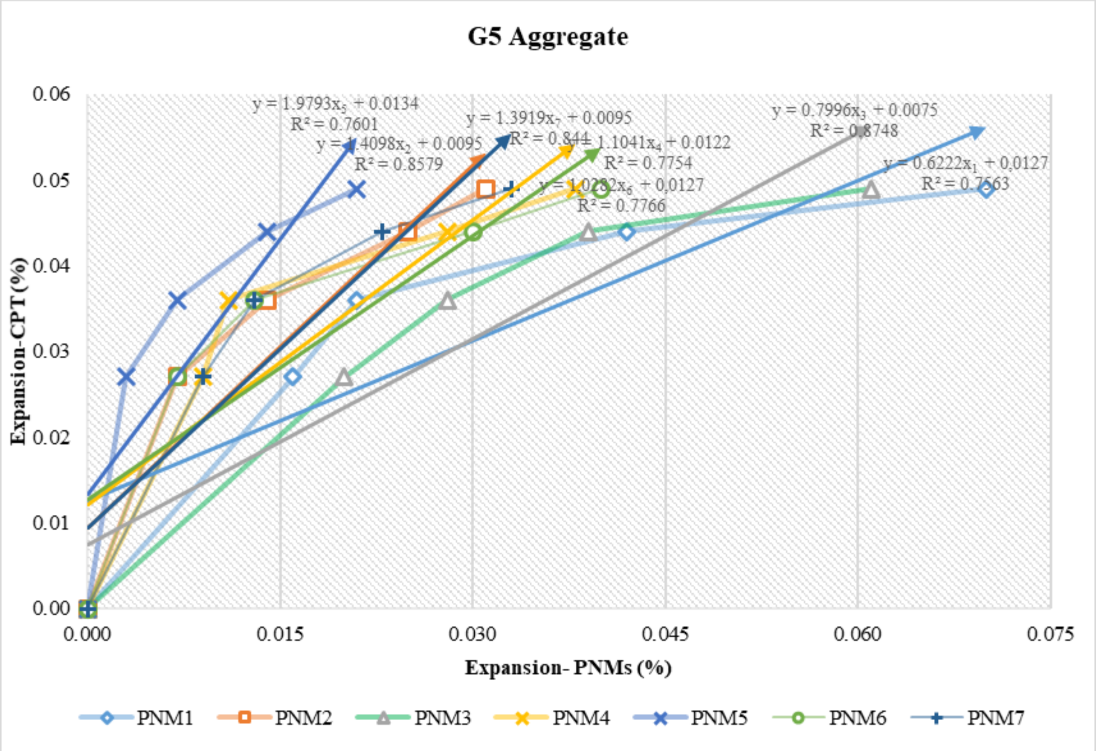


Figure 6.14 Comparison of pilot new methods data with CPT data for G5 aggregate

Correlation Parameters obtained from regression analysis of G5 aggregate indicate that excellent correlation with CPT is not observed in all new pilot methods as seen in Figure 6.15. PNM6 having regression line equation of $Y=1.0282X_6+0.0127$ is determined as the best method for G5 aggregate due to good “a” coefficient that much close to ideal number “1”. The other important parameter to support this choice is that PNM6 give most reasonable data, 0.040% expansion for 14 days, as comparing data in CPT, 0.048% expansion for 20 weeks (Figure 6.14). For G5 aggregate, PNM4 can be evaluated an alternative method though correlation parameters are not so good.



Method	Regression Line Equation (Y=aX+b)	Correlation Coefficient (R^2)
PNM1	$Y=0.6222X_1+0.0127$	0.7563
PNM2	$Y=1.4098X_2+0.0095$	0.8579
PNM3	$Y=0.7996X_3+0.0075$	0.8748
PNM4	$Y=1.1041X_4+0.0122$	0.7754
PNM5	$Y=1.9793X_5+0.0134$	0.7601
PNM6	**$Y=1.0282X_6+0.0127$**	**0.7766**
PNM7	$Y=1.3919X_7+0.0095$	0.8440

Figure 6.15 Regression analysis of pilot new methods data and CPT data for G5 aggregate

As seen in Figure 6.16 and in Table 6.3, G6 aggregate shows 0.267% expansion in AMBT, so it is detected as a potential reactive material. However, expansion of 0.037% in CPT does not mean potential reactivity but critical value for the long-term limit. Moreover, Table 6.2 expresses the expansion limit as 0.030 % for 15 weeks. Accordingly, G6 aggregate is considered as a dangerous material for alkali reactivity because of 0.033% expansion for this period. If regarding as the last values, PNM6 gives almost the same result, 0.037% expansion for 28 days with that in CPT, 0.038% expansion for 20 weeks (Figure 6.17). This is a strong reason to choose PNM6 as the best appropriate method.

Figure 6.18 including regression analysis parameters of G6 aggregate reflects that PNM7 with line equation of $Y=0.8504X_7+0.0110$ is determined as a little better correlation in view of “a” coefficient (0.8504). On the other hand, last expansion value, “b” coefficient, correlation coefficient (R^2) are more logical in PNM6 with line equation of $Y=0.8184X_6+0.0100$, so the best compatible method is preferred as PNM6 for G6 aggregate.

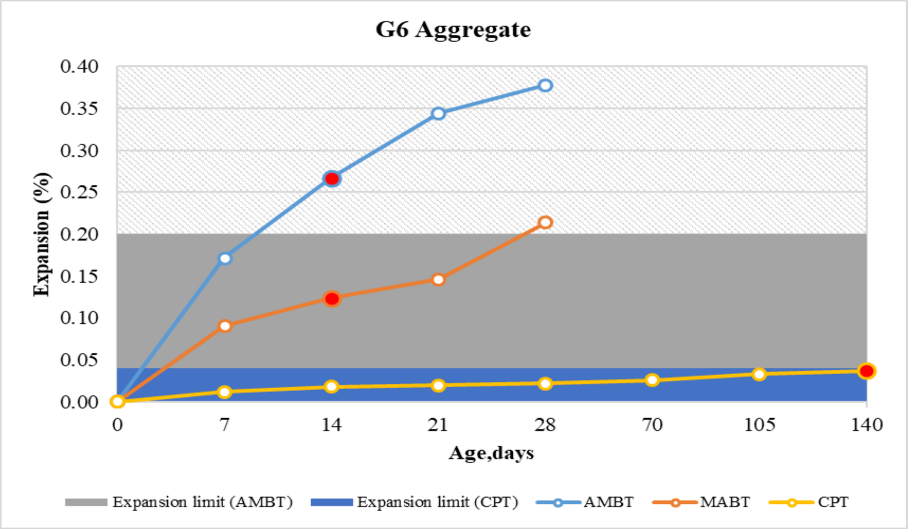


Figure 6.16 Expansion results of G6 aggregate according to the standard test methods

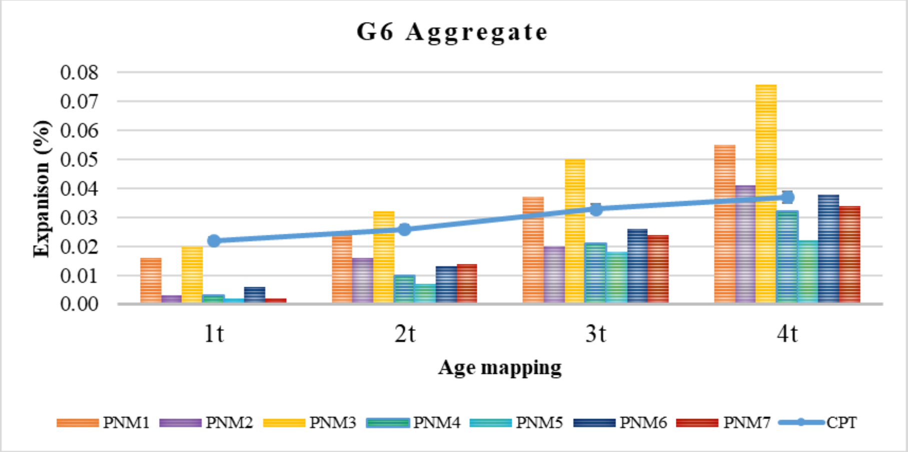
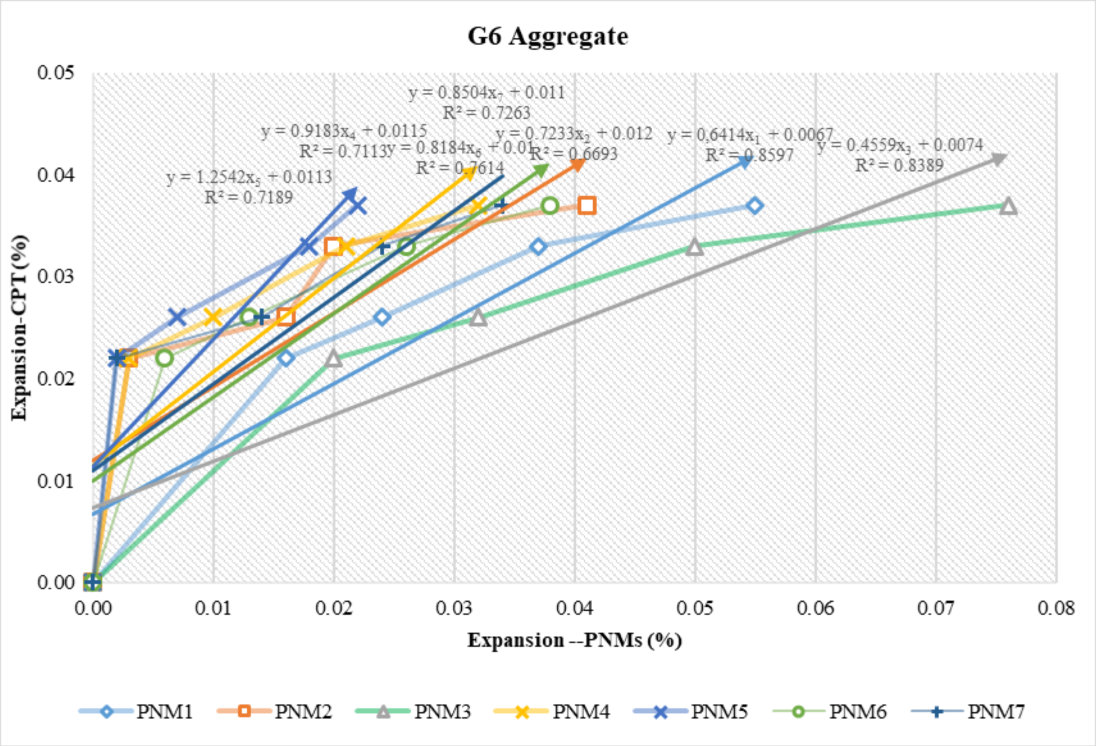


Figure 6.17 Comparison of pilot new methods data with CPT data for G6 aggregate



Method	Regression Line Equation (Y=aX+b)	Correlation Coefficient (R^2)
PNM1	$Y=0.6414X_1+0.0067$	0.8597
PNM2	$Y=0.7233X_2+0.0095$	0.6693
PNM3	$Y=0.4559X_3+0.0074$	0.8389
PNM4	$Y=0.9183X_4+0.0115$	0.7113
PNM5	$Y=1.2542X_5+0.0113$	0.7189
PNM6	$\mathbf{Y=0.8184X_6+0.0100}$	**0.7614**
PNM7	$Y=0.8504X_7+0.0110$	0.7263

Figure 6.18 Regression analysis of pilot new methods data and CPT data for G6 aggregate

According to data in Table 6.3, G7 aggregate is potentially reactive because it exceeds the 0.2% expansion limit even on 7th day. G7 aggregate tested by AMBT shows 0.210% expansion for 7 days and 0.347% expansion for 14 days (Figure 6.19). When CPT method is applied on this sandstone named as G7 aggregate, it is observed as a reactive type of aggregate due to 0.046% expansion for 20 weeks that exceeds 0.04% expansion limit specified in the control concrete prism test method.

0.353% expansion in AMBT for 14 days and 0.046 % in CPT that exceeding the limits in both standard methods. Expansion observed in PNM6 is 0.049 % for 28 days, which is a value closest to 0.046 % in CPT for 20 weeks (Figure 6.20).

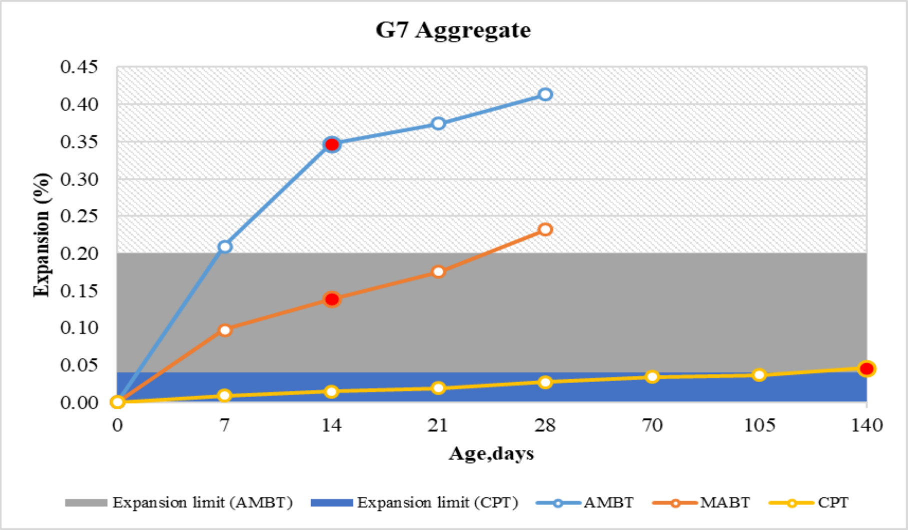


Figure 6.19 Expansion results of G7 aggregate according to the standard test methods

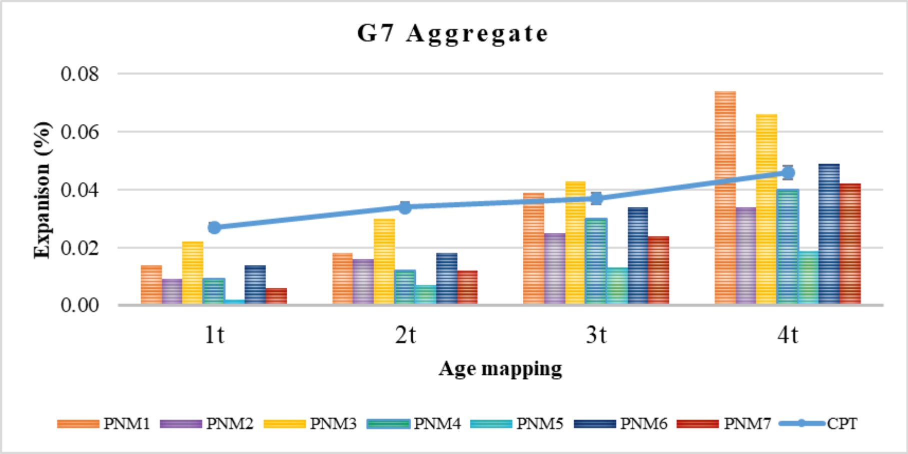
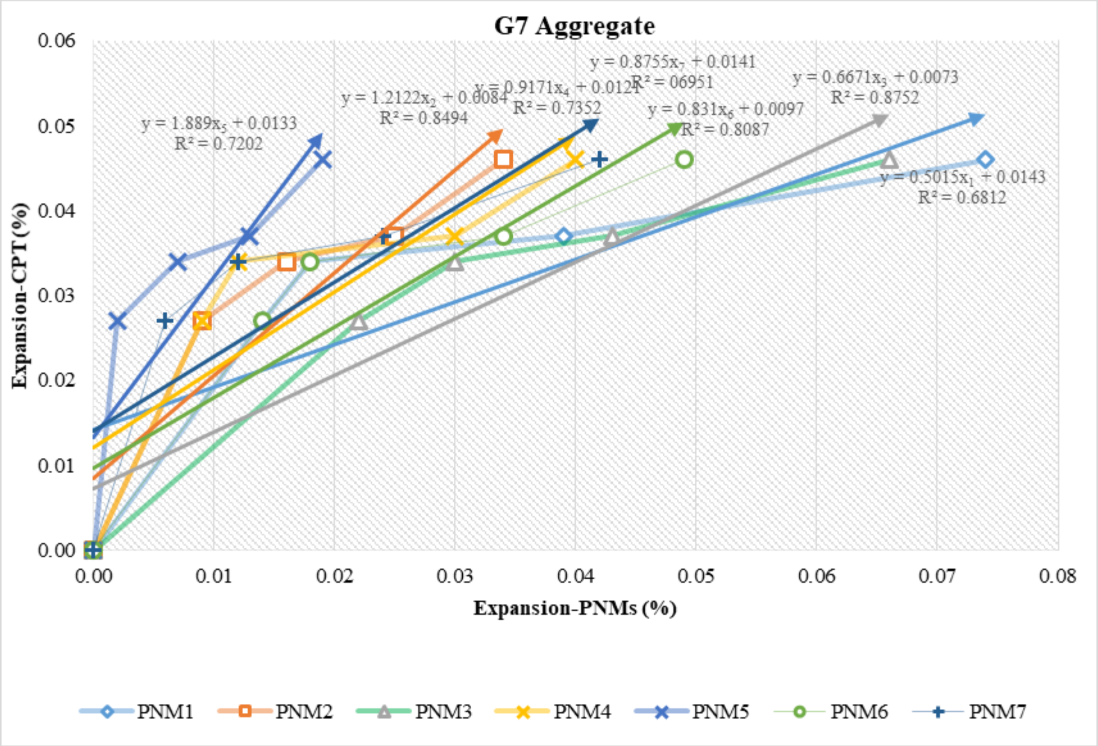


Figure 6.20 Comparison of pilot new methods data with CPT data for G7 aggregate

With regard to the parameters of regression analysis for G7 aggregate in Figure 2.21, PNM4 and PNM7 reflect good correlation in terms of “a” coefficient, (0.9171, 0.8755 in turn) but poor correlation in terms of “b” coefficient (0.0121,0.0141 in turn) and correlation coefficient (R^2=0.7352, R^2=0.6951). As considering better last data, “b” coefficient, correlation coefficient (R^2=0.8310), PNM6 comes into prominence for the best method for G7 aggregates despite of its poorer primary parameter that is “a” coefficient. PNM4 and PNM7 may be assessable as alternative methods.



Method	Regression Line Equation (Y=aX+b)	Correlation Coefficient (R^2)
PNM1	$Y=0.6414X_1+0.0067$	0.6812
PNM2	$Y=1.2122X_2+0.0084$	0.8494
PNM3	$Y=0.6671X_3+0.0073$	0.8752
PNM4	$Y=0.9171X_4+0.0121$	0.7352
PNM5	$Y=1.8890X_5+0.0133$	0.7202
PNM6	$\mathbf{Y=0.8310X_6+0.0097}$	**0.8087**
PNM7	$Y=0.8755X_7+0.0141$	0.6951

Figure 6.21 Regression analysis of pilot new methods data and CPT data for G7 aggregate

Data for G8 aggregate in Table 6.3 and in Figure 6.22 show that this type of aggregate is potentially reactive due to expansion of 0.216 % in AMBT for 14 days while it is not reactive according to CPT data that is 0.032% for 20 weeks. PNM4 gives similar result as 0.038 % for 28 days that closest value to data in CPT for 20 weeks but this harmony is not very good (Figure 6.22).

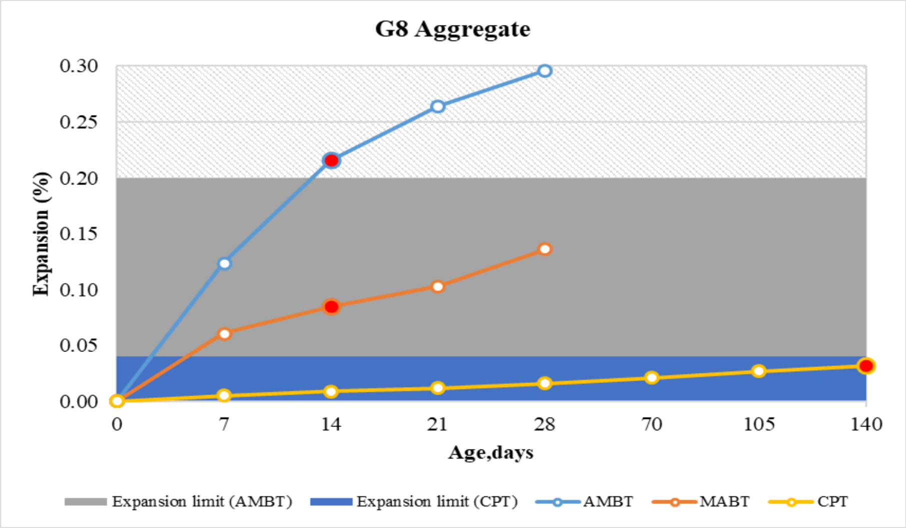


Figure 6.22 Expansion results of G8 aggregate according to the standard test methods

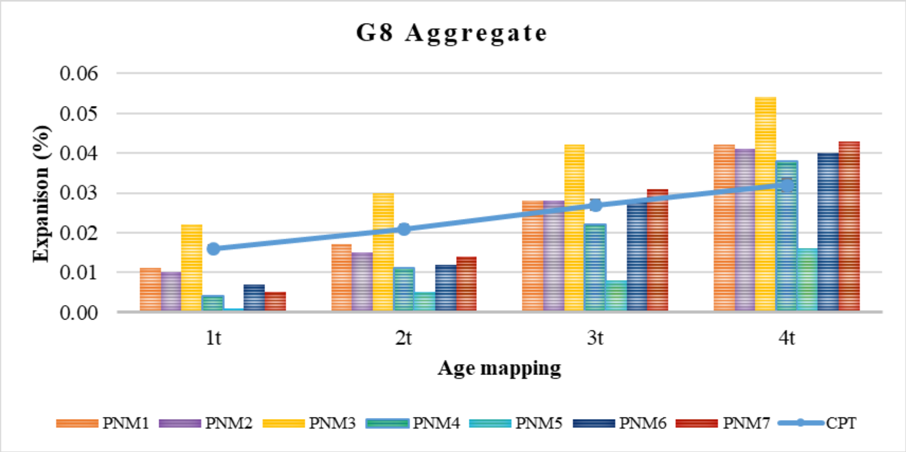
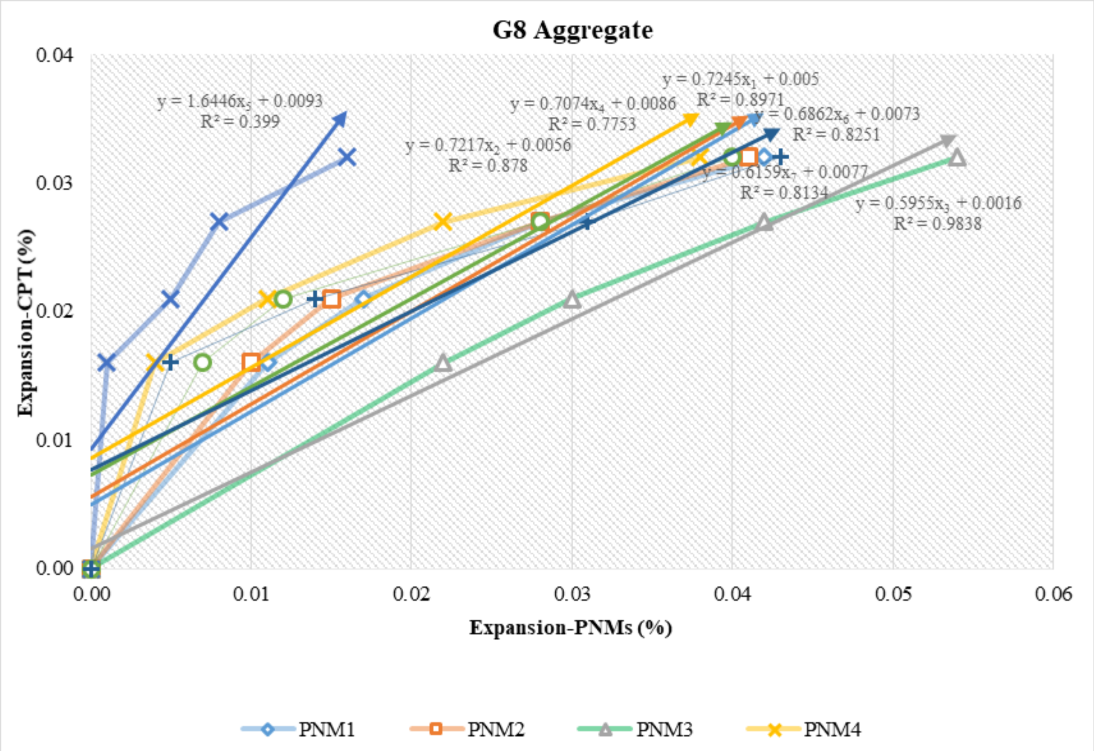


Figure 6.23 Comparison of pilot new methods data with CPT data for G8 aggregate

If viewing regression analysis parameters as seen in Figure 6.24, PNM4 has better correlation with the line equation=$0.9171X_4+0.0121$, as comparing the others. PNM6 is determined as second alternative method due to be having better value for correlation coefficient ($R^2=0.8087$) and "b" coefficient (0.0097). Nevertheless, PNM6 provides better proportional harmony in terms of last data in addition to more ideal line equation despite of worse correlation coefficient ($R^2=0.8087$). As all parameters are evaluated in order of importance, specifying PNM6 as the most ideal method for G8 aggregate is more logical in this study.

The data of preliminary experimental study demonstrates that some of the pilot new methods, which are PNM7, PNM6, PNM4, PNM2, are favourable well to predict potential alkali reactivity of aggregates because these methods lasted 28 days have given similar results to CPT data lasted 20 weeks in terms of especially last data as explained in Table 6.4. However, for analysis of data obtained from favourable PNMs, regression analysis seems giving poor correlation parameters that are "a", "b" and "R^2" coefficients. Achieving the excellent correlation is nearly impossible because time mapping applied for regression analysis cannot provide completely compatible result between PNMs data and CPT data in terms of all period. In the outcomes of preliminary experimental study, there is a remarkable point that PNM6 is the best or an alternative method for most reactive materials such as (G1, G5,G6,G7) while it cannot give mostly good correlation for non-reactive materials.



Method	Regression Line Equation (Y=aX+b)	Correlation Coefficient (R^2)
PNM1	$Y=0.6414X_1+0.0067$	0.6812
PNM2	$Y=1.2122X_2+0.0084$	0.8494
PNM3	$Y=0.6671X_3+0.0073$	0.8752
PNM4	**$Y=0.9171X_4+0.0121$**	**0.7352**
PNM5	$Y=1.889X_5+0.0133$	0.7202
PNM6	$Y=0.8310X_6+0.0097$	0.8087
PNM7	$Y=0.8755X_7+0.0141$	0.6951

Figure 6.24 Correlation between Pilot New Methods data and CPT data for G8 aggregate

Table 6.4 The outcomes of the preliminary experimental study

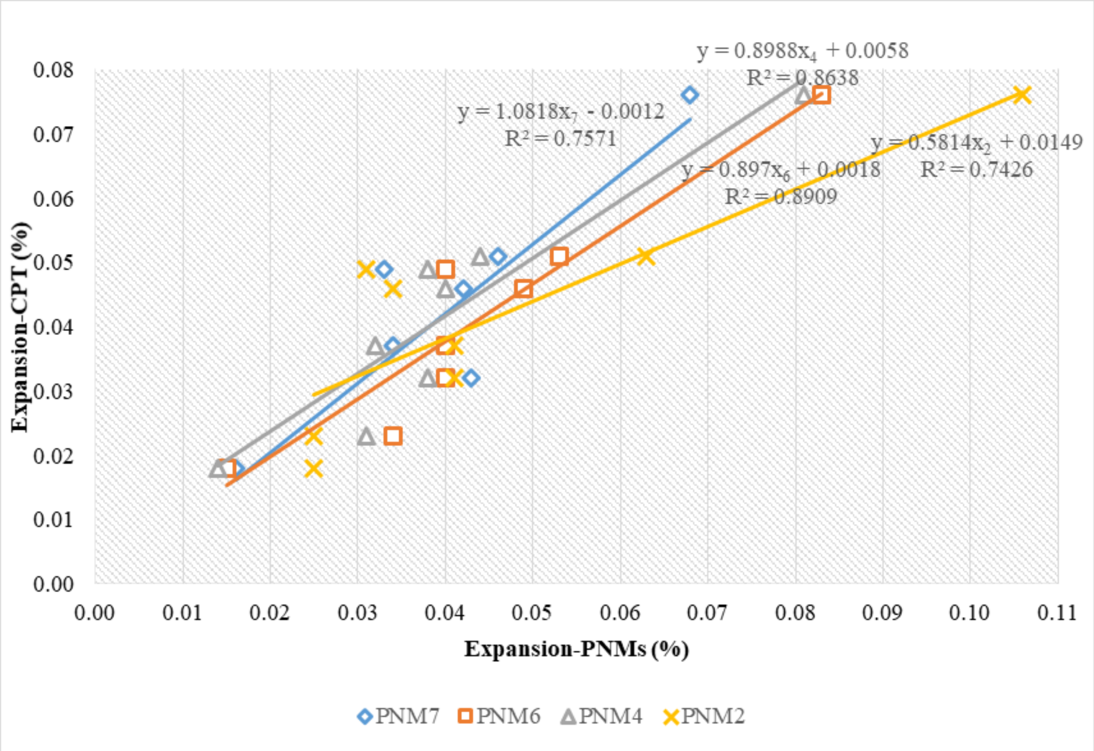
Aggregate type	Reactivity for AMBT	Reactivity for CPT	Best method	Alternative method	*Proportion harmony
G1	Reactive	Reactive	PNM7	PNM6	0.90
G2	Non-Reactive	Non-Reactive	PNM7	PNM6	0.89
G3	Suspicious	Non-Reactive	PNM2	--	1.09
G4	Reactive	Reactive	PNM7	PNM4	0.89
G5	Reactive	Reactive	PNM6	PNM4	0.82
G6	Reactive	**Reactive	PNM6	PNM7	1.03
G7	Reactive	Reactive	PNM6	PNM7	1.07
G8	Reactive	Non-Reactive	PNM4	PNM6	1.19

Notes: *(Expansion in PNM_i for 28 days / Expansion in CPT for 28 days).

**According to RILEM AC-219 AAR-4.1, if expansion percent is higher than 0.03% for 15 weeks, sample is defined as potentially reactive.

On the other hand, regression analysis enables the preliminary experimental study to decide the best ones of PNMs and then the best methods (PNM6, PNM4, PNM7, PNM2) are determined by establishing correlation between these PNMs data for 28 days and CPT data for 20 weeks. Table 6.5 gives the last results for CPT and the PNMs selected as the best for each aggregate, so calculating average deviation percent of PNMs data from CPT data may give an idea to decide the best method. Deviation values calculated by comprising data between CPT data and PNMs, the best method of ones performed in this preliminary study are observed as PNM6 prepared by soak solution consists of 0.35 N NaOH and 1% $CaCl_2$.

Similarly, correlation parameters obtained from regression analysis on last test result values (Figure 6.25) reveal that PNM6 with equation of $Y=0.8970\ X_6+0.0018$ with can be evaluated as the best method because it gives better correlation coefficient ($R^2=0.8909$) while it is 0.7571 in PNM7 with equation of $1.0818\ X_7-0.0012$. It can be said that PNM7 has better “a” coefficient but R^2 is more critical than “a” coefficient in views of analysing last test results unlike in previously analysing the test result by making time mapping.



Method	Regression Line Equation (Y=aX+b)	Correlation Coefficient (R^2)
PNM7	$Y=1.0818\ X_7-0.0012$	0.7571
PNM6	$Y=0.8970\ X_6+0.0018$	0.8909
PNM4	$Y=0.8988X_4+0.0058$	0.8638
PNM2	$Y=0.58141X_2+0.0149$	0.7426

Figure 6.25 Regression analysis of best pilot new methods data and CPT data for last values

Table 6.5 Comparison of last data of CPT and PNM7, PNM6, PNM4, PNM2

Agg. Group	CPT	PMN7		PMN6		PMN4		PMN2	
	Exp. (%)	Exp. (%)	Dev. (%)	Exp. (%)	Dev. (%)	Exp. (%)	Dev. (%)	Exp. (%)	Dev. (%)
G1	0.051	0.046	9.8	0.053	3.9	0.044	13.7	0.063	23.5
G2	0.018	0.016	11.1	0.015	16.7	0.014	22.2	0.025	38.9
G3	0.023	0.034	47.8	0.034	47.8	0.031	34.8	0.025	8.7
G4	0.076	0.068	10.5	0.083	9.2	0.081	6.6	0.106	39.5
G5	0.049	0.033	32.7	0.040	18.4	0.038	22.4	0.031	36.7
G6	0.037	0.034	8.1	0.040	8.1	0.032	13.5	0.041	10.8
G7	0.046	0.042	8.7	0.049	6.5	0.040	13.0	0.034	26.1
G8	0.032	0.043	34.4	0.040	25.0	0.038	18.8	0.041	28.1
Average Dev. (%)		20.4		17.0		18.1		26.5	

6.2 Latter Experimental Study

The principles of latter new methods to be applied on all 32 types can be specified as considering the degree of correlation between PNMs and CPT applied in the preliminary experimental study. The specifications for finalized versions of developed new methods will be mentioned in the following part.

6.2.1 Finalized Version of Developed New Methods

Based on the analysis of preliminary experimental study, the data show that PNM6 is the best of PNMs. Nevertheless, it is not said that PNM6 data provide excellent correlation with CPT data. PNM6 generally gives a slightly higher expansion rates for all aggregates except for G2 and G5 aggregates, so it is necessary to make a little correction on excessive test condition.

According to the results of this preliminary experimental study and the literature review, NaOH may cause great fluctuation in expansion percent, so it is a better way to make the ratio of NaOH stable like in PNM6 as 0.35 N. On the other hand, the effect of $CaCl_2$ on are so diverse from other chloride-based de-icers. If the percent of

$CaCl_2$ is increased from 1 to 2 like in PNM7, the expansion rates of some aggregates such as G1, G4, G5, G6 and G7 showing higher reactivity in CPT method, decrease in striking rate. On the contrary, the increase in $CaCl_2$ cause slightly higher expansion in non-reactive aggregates such as G2, G3, and G8. Therefore, the general evaluation on all test results proposes that $CaCl_2$ percent should be increased to 1.25% in one of developed new test methods to examine the effect of $CaCl_2$ on AAR expansion at a different rate.

Data of AMBT (80 ^{o}C) and MABT (60 ^{o}C) exhibits a precise deliverance that increase in storage temperature leads to dramatic jump in expansion percent for all test aggregates as seen in Table 6.3, so the storage temperature may be specified 60 ^{o}C in developed new methods like in pilot ones to prevent dramatic movement in values.

In one of developed new test methods, using a cement type having lower alkali content may be useful to observe the effect of cement type on potential alkali reactivity of aggregates in Turkey. Table 4.2 gives chemical composition of calculated Na_2Oe equivalent of Type I having higher alkali content and Type II having lower alkali content.

Usage of prisms of 50 mm x 50 mm x 200 mm and a maximum aggregate size of 16 mm can make the developed new methods easier and more practicable than the CPT method.

After completion of the preliminary experimental study, specifications of two new-developed test methods have been summarized as in Table 6.6. These new methods had been decided to be applied to 32 types of aggregates having different petrographic identification, mineral and chemical composition.

Table 6.6 Specifications of developed new test methods

Test Method	Test Duration	Soak Solution	Mould Dimension (mm*mm*mm)	Storage Temp.	Cem. Type	Notation
Developed New Method-1	28 days	**0.35N NaOH** **1.25% $CaCl_2$**	50x50x200	60 °C	Type I	DNM1
Developed New Method-2	28 days	**0.35N NaOH** **1% $CaCl_2$**	50x50x200	60 °C	Type II	DNM2

6.2.2 Test Results of Latter Experimental Study

In this part of the experimental study, 32 types of aggregate group were detected with DNM1 and DNM2 in addition to AMBT and CPT. Table 6.7 gives data of the experimental study averagely for 32 types of aggregates. The table consists of 7,14,21,28-day expansion rates of aggregates tested by AMBT, DNM1, DNM2 and 10,15,20-week expansion rates for only CPT method. The results are given as the average of three samples; all results can be checked from the Appendix.

Table 6.7 The results of latter experimental study

G1 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.219	0.332	0.395	0.607			
CPT	Av:	0.005	0.012	0.019	0.023	0.041	0.048	0.051
DNM1	Av:	0.016	0.028	0.041	0.047			
DNM2	Av:	0.015	0.027	0.040	0.049			
G2 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.039	0.070	0.097	0.122			
CPT	Av:	0.001	0.003	0.004	0.007	0.014	0.016	0.018
DNM1	Av:	0.004	0.008	0.011	0.016			
DNM2	Av:	0.002	0.006	0.011	0.014			
G3 aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.113	0.194	0.242	0.274			
CPT	Av:	0.004	0.007	0.009	0.011	0.017	0.021	0.023
DNM1	Av:	0.003	0.011	0.026	0.036			
DNM2	Av:	0.005	0.013	0.025	0.032			

Table 6.7 continued

G4 Aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.337	0.454	0.478	0.493			
CPT	Av:	0.019	0.030	0.035	0.040	0.047	0.061	0.076
DNM1	Av:	0.019	0.025	0.057	0.071			
DNM2	Av:	0.015	0.023	0.058	0.080			
G5 Aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.209	0.353	0.379	0.421			
CPT	Av:	0.012	0.017	0.022	0.027	0.036	0.044	0.049
DNM1	Av:	0.008	0.014	0.028	0.039			
DNM2	Av:	0.005	0.012	0.027	0.038			
G6 Aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.172	0.267	0.344	0.378			
CPT	Av:	0.012	0.018	0.020	0.022	0.026	0.033	0.037
DNM1	Av:	0.006	0.014	0.028	0.040			
DNM2	Av:	0.004	0.012	0.025	0.036			
G7 Aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.210	0.347	0.374	0.413			
CPT	Av:	0.009	0.015	0.019	0.027	0.034	0.037	0.046
DNM1	Av:	0.011	0.014	0.027	0.048			
DNM2	Av:	0.012	0.016	0.030	0.045			
G8 Aggregate	**Exp.(%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.124	0.216	0.264	0.296			
CPT	Av:	0.005	0.009	0.012	0.016	0.021	0.027	0.032
DNM1	Av:	0.007	0.011	0.030	0.042			
DNM2	Av:	0.004	0.009	0.028	0.037			
NG9 Aggregate	Exp.(%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.192	0.337	0.421	0.716			
CPT	Av:	0.004	0.006	0.007	0.007	0.011	0.013	0.016
DNM1	Av:	0.004	0.010	0.018	0.027			
DNM2	Av:	0.005	0.011	0.017	0.021			
NG10 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.028	0.163	0.461	0.801			
CPT	Av:	0.005	0.011	0.013	0.016	0.026	0.038	0.045
DNM1	Av:	0.011	0.024	0.043	0.057			
DNM2	Av:	0.008	0.022	0.039	0.052			

Table 6.7 continued

NG11 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.036	0.152	0.215	0.373			
CPT	Av:	0.003	0.005	0.008	0.008	0.010	0.013	0.014
DNM1	Av:	0.005	0.009	0.018	0.022			
DNM2	Av:	0.003	0.007	0.015	0.017			
NG12 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.005	0.005	0.010	0.026			
CPT	Av:	0.001	0.003	0.003	0.003	0.004	0.006	0.006
DNM1	Av:	0.002	0.007	0.010	0.014			
DNM2	Av:	0.002	0.004	0.008	0.011			
NG13 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.189	0.281	0.367	0.656			
CPT	Av:	0.011	0.015	0.020	0.024	0.038	0.047	0.055
DNM1	Av:	0.013	0.027	0.045	0.064			
DNM2	Av:	0.012	0.029	0.048	0.066			
NG14 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.311	0.406	0.512	0.789			
CPT	Av:	0.012	0.018	0.023	0.031	0.062	0.078	0.097
DNM1	Av:	0.011	0.032	0.068	0.080			
DNM2	Av:	0.013	0.036	0.065	0.085			
NG15 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.113	0.175	0.271	0.353			
CPT	Av:	0.004	0.007	0.009	0.011	0.017	0.021	0.022
DNM1	Av:	0.006	0.011	0.019	0.027			
DNM2	Av:	0.007	0.013	0.018	0.024			
NG16 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.027	0.074	0.117	0.135			
CPT	Av:	0.002	0.005	0.005	0.008	0.011	0.013	0.014
DNM1	Av:	0.005	0.009	0.016	0.019			
DNM2	Av:	0.003	0.007	0.014	0.017			
NG17 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.015	0.023	0.028	0.032			
CPT	Av:	0.001	0.001	0.002	0.003	0.006	0.008	0.011
DNM1	Av:	0.004	0.007	0.014	0.016			
DNM2	Av:	0.003	0.005	0.011	0.013			

Table 6.7 continued

NG18 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.072	0.165	0.234	0.287			
CPT	Av:	0.005	0.006	0.007	0.007	0.010	0.012	0.013
DNM1	Av:	0.006	0.012	0.015	0.018			
DNM2	Av:	0.007	0.011	0.013	0.017			
NG19 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.006	0.011	0.015	0.018			
CPT	Av:	0.001	0.001	0.002	0.002	0.004	0.005	0.006
DNM1	Av:	0.002	0.003	0.005	0.007			
DNM2	Av:	0.001	0.002	0.003	0.005			
NG20 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.014	0.056	0.062	0.067			
CPT	Av:	0.001	0.001	0.003	0.004	0.009	0.012	0.015
DNM1	Av:	0.003	0.007	0.017	0.021			
DNM2	Av:	0.002	0.006	0.014	0.019			
NG21 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.089	0.164	0.215	0.252			
CPT	Av:	0.002	0.007	0.011	0.015	0.023	0.027	0.030
DNM1	Av:	0.009	0.016	0.027	0.038			
DNM2	Av:	0.007	0.015	0.025	0.033			
NG22 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.023	0.089	0.126	0.136			
CPT	Av:	0.002	0.004	0.007	0.009	0.014	0.018	0.021
DNM1	Av:	0.005	0.012	0.019	0.020			
DNM2	Av:	0.004	0.011	0.015	0.018			
NG23 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.405	0.785	0.892	0.914			
CPT	Av:	0.014	0.026	0.036	0.049	0.098	0.123	0.141
DNM1	Av:	0.038	0.067	0.119	0.143			
DNM2	Av:	0.035	0.071	0.128	0.151			
NG24 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.014	0.026	0.039	0.041			
CPT	Av:	0.002	0.004	0.005	0.007	0.012	0.015	0.017
DNM1	Av:	0.006	0.011	0.019	0.021			
DNM2	Av:	0.004	0.008	0.012	0.015			

Table 6.7 continued

NG25 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.006	0.007	0.011	0.023			
CPT	Av:	0.001	0.001	0.002	0.004	0.005	0.007	0.007
DNM1	Av:	0.002	0.006	0.011	0.013			
DNM2	Av:	0.003	0.004	0.007	0.009			
NG26 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.128	0.203	0.294	0.364			
CPT	Av:	0.004	0.008	0.010	0.012	0.021	0.027	0.032
DNM1	Av:	0.005	0.010	0.018	0.025			
DNM2	Av:	0.007	0.011	0.021	0.028			
NG27 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.005	0.009	0.013	0.016			
CPT	Av:	0.000	0.001	0.001	0.002	0.003	0.004	0.004
DNM1	Av:	0.001	0.002	0.002	0.004			
DNM2	Av:	0.000	0.001	0.002	0.002			
NG28 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.289	0.501	0.741	0.814			
CPT	Av:	0.013	0.028	0.034	0.038	0.088	0.113	0.132
DNM1	Av:	0.031	0.054	0.104	0.128			
DNM2	Av:	0.027	0.056	0.113	0.140			
NG29 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.008	0.014	0.019	0.022			
CPT	Av:	0.002	0.002	0.004	0.005	0.008	0.009	0.010
DNM1	Av:	0.004	0.009	0.011	0.014			
DNM2	Av:	0.003	0.007	0.009	0.011			
NG30 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.079	0.102	0.138	0.157			
CPT	Av:	0.004	0.008	0.009	0.011	0.020	0.028	0.034
DNM1	Av:	0.008	0.014	0.025	0.036			
DNM2	Av:	0.005	0.010	0.019	0.031			
NG31 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.037	0.193	0.401	0.713			
CPT	Av:	0.007	0.015	0.021	0.026	0.034	0.045	0.053
DNM1	Av:	0.013	0.027	0.048	0.060			
DNM2	Av:	0.010	0.021	0.043	0.058			

Table 6.7 continued

NG32 Aggregate	Exp%	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	Av:	0.297	0.405	0.498	0.556			
CPT	Av:	0.017	0.025	0.032	0.037	0.047	0.053	0.058
DNM1	Av:	0.021	0.030	0.053	0.063			
DNM2	Av:	0.016	0.027	0.051	0.064			

Table 6.7 gives expansion values to determine which aggregates are reactive for both AMBT and CPT. Data obtained from CPT method is a fundamental decisive indicator to determine reactivity of aggregates, so the regression analysis will be made with CPT data for finalizing the new method. In addition, data from AMBT will be used to determine whether there is a linear relation between finalized version of developed new methods and AMBT.

6.2.3 Analysis of the Results

Figure 6.26 shows that types of G1, G4, G5, G6, G7, G8, NG9, NG13, NG14, NG23, NG26, NG28, and NG32 are potentially reactive aggregates and types of G3, NG10, NG11, NG15, NG18, NG21, and NG31 are suspicious aggregates for alkali reactivity according to AMBT. Other aggregates are observed as non-deleterious materials. On the other hand, CPT data in Figure 6.27 indicates that G1, G4, G5, G7, NG10, NG13, NG14, NG23, NG28, NG31, and NG32 are potentially reactive aggregates while the others are not reactive. Moreover, G6 aggregate may be defined as a reactive aggregate because it exceeds the expansion limit (0.030%) for 15 weeks.

NG10 aggregate gives a contradictory result because it is defined as suspicious material for AMBT but potentially reactive for CPT. Moreover, if extending test period to 28 days, expansion rate increase in huge rate, from 0.163% to 0.801%. This significant increase provides another support to the claim that the 14-day short term test sometimes gives misleading data for alkali reactivity of aggregates. As a result, AMBT applied on NG10 aggregate gives false negative results that means non-reactive or suspicious for AMBT but reactive in CPT. Like in NG10 aggregate, AMBT method applied on NG31 indicates false negative results because NG31 aggregate shows 0.193% expansion in AMBT for 14 days while it expands 0.053% in CPT. The reason for this similar behaviour between NG10 and NG31 may be having similar petrographic properties and mineralogical composition.

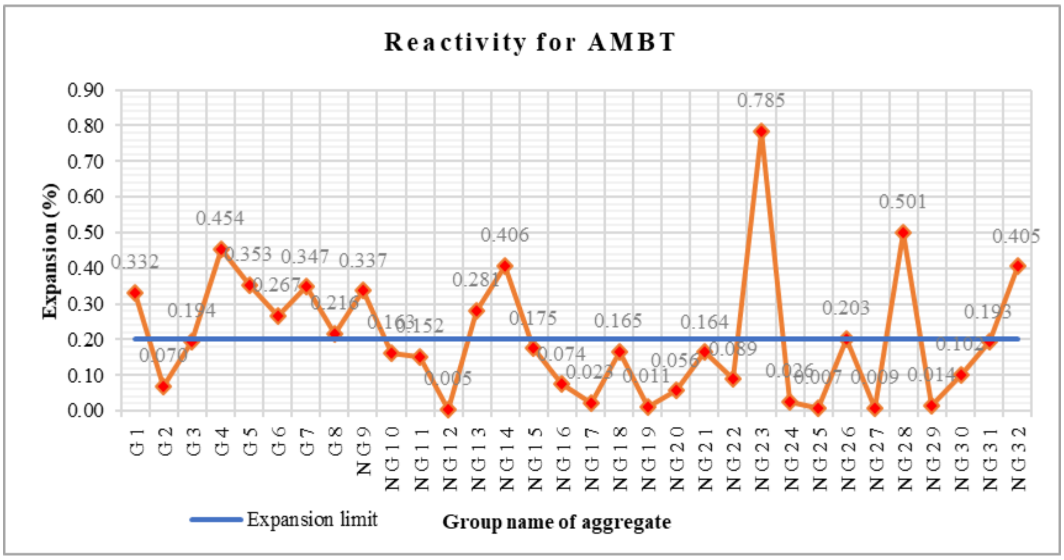


Figure 6.26 14-day expansions of all aggregates for AMBT

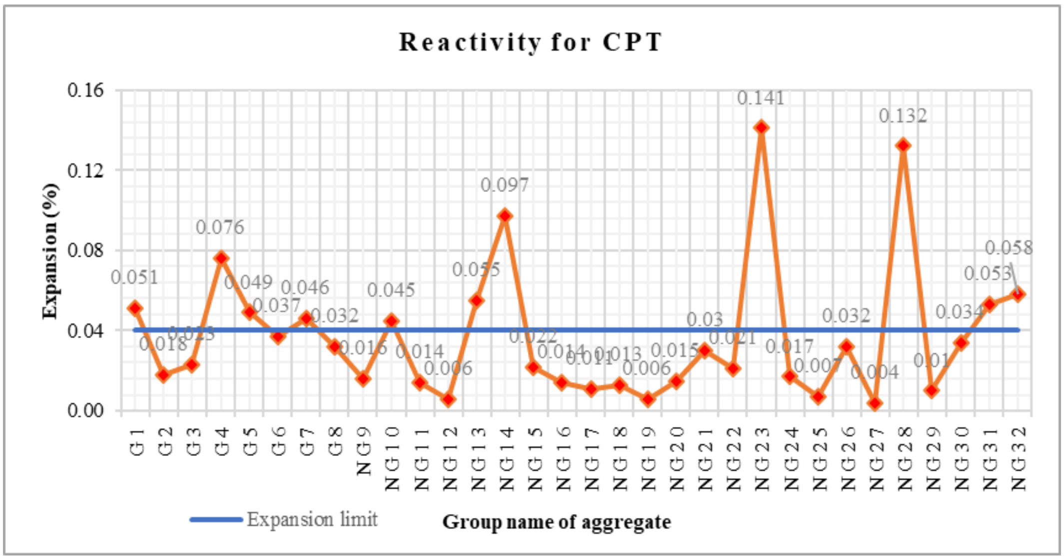


Figure 6.27 20-week expansions of all aggregates for CPT

Moreover, there are aggregates giving false positive results that means reactive in AMBT but non-reactive in CPT. For example, G8 aggregate shows 0.216% expansion for 14 days over the limit in AMBT but 0.032% expansion for 20 weeks under the limit in CPT. Like in G8 aggregate, NG9 (0.337% expansion in AMBT and 0.016% in CPT), NG26 (0.203% expansion in AMBT and 0.032% in CPT) are other examples to the aggregates giving false positive results. Common characteristic of these aggregates is to be originated from river or stream basin. The effect of petrographic properties and mineralogical composition will be evaluated in detail in the following discussion part.

The primary criteria for DNMs is the correspondence of CPT with data, but the expansion limit is set to 0.04% for 28 days as in CPT for 20 weeks. According to the results obtained from DNM1 applied on all aggregates (Figure 6.28); G1, G4, G6, NG9, NG10, NG13, NG14, NG23, NG28, NG31, and NG32 are observed as reactive or deleterious materials.

Figure 6.29 shows that G1, G4, G7, NG10, NG13, NG14, NG23, NG28, NG31, and NG32 tested by DNM2 method are observed as potentially reactive for alkali reactivity. When compared to DNM1 and DNM2, they are compatible with each other except the data obtained from G6 and G7 aggregates because their reactivity shows different behaviours in these new methods. Regression analyses for data from DNM1 and DNM2 according to that from CPT to evaluate their compliance performances will be broadly made in the discussion part.

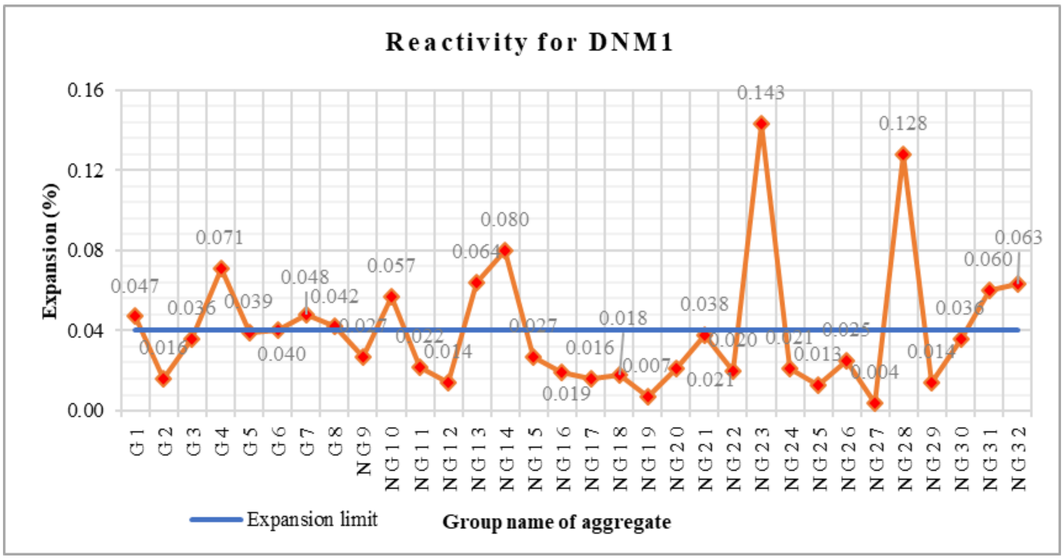


Figure 6.28 28-day expansions of all aggregates for DNM1

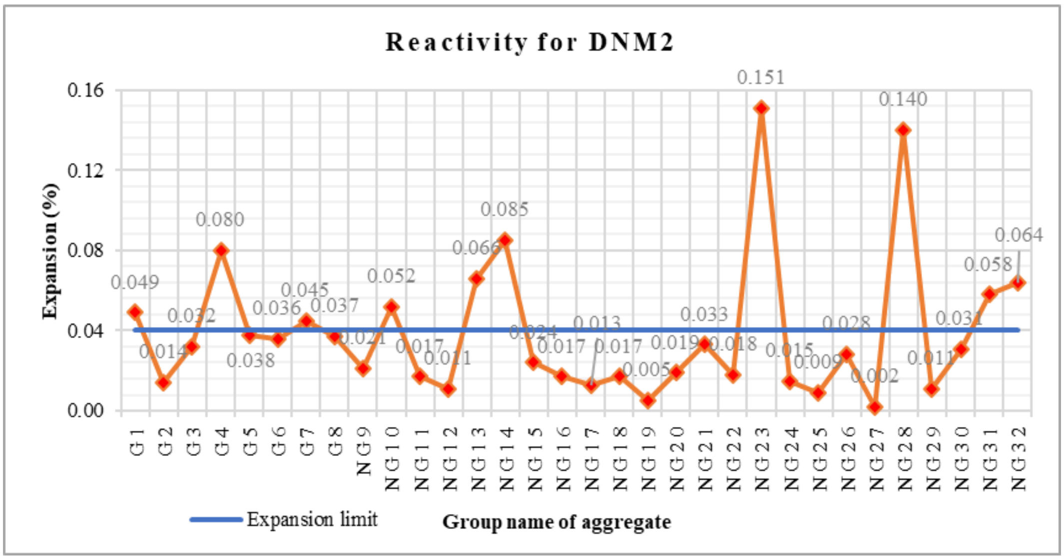


Figure 6.29 28-day expansions of all aggregates for DNM2

CHAPTER 7

DISCUSSION

This chapter consists of titles named as comparison of the standard and developed new methods, final decision for the new method, correlation of SCPT with AMBT, effect of petrographic and mineralogical properties on AAR expansion, effect of chloride based de-icers on the process of AAR, effect of temperature and cement type on AAR expansion, effect of mechanical and physical properties on ASR expansion and comparison of SCPT with field performance of aggregates.

7.1 Comparison of the Standard and Developed New Methods

Degree of correlation between DNMs data for 28 days and CPT data for 20 weeks are analysed to make out the best method. The data in Table 7.1 show that DNMs provide better compatible data with CPT data than PNMs. On the other hand, an excellent correlation between DNMs and CPT data cannot be entirely achieved yet in the latter study. However, some of DNM1 data and DNM2 data is almost same with CPT data such as G2, G7, NG19, NG22, NG23, NG27, NG28, NG30 in DNM1 and G1, G6, NG15, NG19, NG24, NG29 in DNM2. As the reactivity for 0.04% expansion limit of CPT is considered, DNM2 may give an insignificant misleading result for only G5 aggregate of which expansion rates are 0.049 % in CPT; 0.038 % in DNM1. Like DNM1, DMN2 ensures possibility to make good predictability for reactivity of aggregates except only for G8. For all these reasons, it is acceptable that DNMs lasted 28 days are purposive methods to evaluate potential alkali reactivity of aggregates instead of CPT methods lasted for months.

Before the final decision for the best method, firstly, last data are compared by using an assessment method named proportional harmony for DNM1 and DNM2, so preliminary evaluation can be made as seen in Table 7.3. The values of the proportional harmony (PH) are calculated by means of dividing the expansion value in DNM*i* for 28 days by the expansion value in CPT for 20 weeks for a defined aggregate. The degree of correlation obtained from proportional harmony (PH) is graded into four different groups mean that (1) if PH>1.20 or PH<0, poor correlation; (2) if PH is in the range of 1.20-0.80, moderate correlation; (3) if PH is in the range of 0.90-1.10, good correlation; (4) if PH is in the range of 0.95-1.05, excellent correlation. After the preliminary evaluations for all aggregate groups are completed one by one, general regression analysis for all aggregates will be evaluated.

G1 aggregate (a type of sandstone) observed as a reactive material for both AMBT and CPT shows a good correlation in DNM1 and an excellent correlation in DNM2 if compared with CPT data. There is no conflicting in the results of G1 aggregate because it is determined as potentially reactive for AAR. In the preliminary evaluation, DNM2 seems the best method for G1 aggregate due to providing an excellent correlation. G2 aggregate (a type of granite) shows 0.070% expansion for 14 days if tested by AMBT and 0.018% expansion for 20 weeks if tested by CPT, so the expansion results indicate that G2 aggregate is definitely non-reactive material. As a supportive, expansion rates of that tested by DNM1 and DNM2 are very low 0.016% and 0.014% in turn. The best method for G2 aggregate may be accepted as DNM1 due to better correlation with CPT. G3 aggregate is defined as a suspicious material for AAR because it shows 0.194% expansion if tested by AMBT. On the contrary, CPT applied on this polygenic river sandstone reveals that G3 aggregate is not reactive due to 0.023% expansion for 20 weeks far below the expansion limit of CPT method (0.04%). On the other hand, DNM1 and DNM2 give very close results to the expansion limit, so both new methods seem to give parallel results to those in AMBT. According to the values of proportional harmony, DNM1 and DNM2 show a poor correlation (PH=1.57 for DNM1 and PH=1.39 for DNM2) with CPT data. DNM2 may be better for G3 aggregate due to relative good correlation. G4 aggregate (siliceous river sandstone) is an important certain reactive material because it extremely exceeds the expansion limits of all methods. DNM2 method provides an excellent correlation with CPT (PH=1.05) even though appearance of a good correlation between DNM1 and CPT (PH=0.93). Other type of siliceous river sandstone named as G5 aggregate exceeds the expansion limits of AMBT and CPT but expansion percentages of G5 aggregate observed by DNM1 and DNM2 are slightly lower than 0.04% expansion limit. Unlike iin G4 aggregate, DNM1 and DNM2 applied on G5 aggregate cannot show well correlation. PH value observed in DNM1 is better than in DNM2 for G5 aggregate. DNM2 applied on G6 aggregate (recrystallized limestone) and G7 aggregate (siliceous river sandstone) shows an excellent correlation with CPT in views of the values of PH=0.97 in G6 and PH=0.98 in G7. Both G8 aggregate (meta sandstone) and NG9 (river sandstone) are determined as reactive by AMBT but they are not reactive for CPT. DNM2 shows a moderate correlation (PH=1.16) if applied on G8 but DNM1 shows a poor correlation (PH=1.31). Both new methods cannot reasonable results if applied on NG9 because very poor correlation appears in these new methods (PH=1.69 in DNM1 and PH=1.31 in DNM2). NG10 aggregate (perlite/rhyolitic glass) gives false-negative result because NG10 aggregate is observed as suspicious in AMBT (0.163% expansion) but reactive in CPT (0.045% expansion). NG10 aggregate expands greater when DNM1 and DNM2 are applied on. DNM2 provides a moderate correlation (PH=1.16) while PH=1.27 for DNM1 indicates a poor correlation. Like NG10 aggregate, NG11 aggregate (calcirudite) is defined as suspicious material according to AMBT but not reactive if tested by CPT. DNM2 may be better method for NG11 aggregate due to better PH value (1.21) in spite of a poor correlation. NG12 aggregate (dolomite) is an example for exactly non-reactive material. It has strong resistance against deleterious effect of expansion. NG12 aggregate shows very low expansion in all methods (0.005% in AMBT, 0.006% in CPT, 0.014% in DNM1 and 0.011% in DNM2). New methods applied on NG12 show very poor correlation, but this lack of harmony may be because of small numbers rather than general incompatibility.

Table 7.1 Comparison between CPT data and DNMs data for all aggregates

Aggregate Group	AMBT (%)	CPT (%)	DNM1 (%)	DNM2 (%)	PH (DNM1)	PH (DNM2)
G1	0.332 (R)	0.051 (R)	0.047	0.049	0.92***	0.96****
G2	0.070(NR)	0.018(NR)	0.016	0.014	0.89**	0.78*
G3	0.194 (S)	0.023(NR)	0.036	0.032	1.57*	1.39*
G4	0.454 (R)	0.076 (R)	0.071	0.080	0.93***	1.05****
G5	0.353 (R)	0.049 (R)	0.039	0.038	0.80**	0.78*
G6	0.267 (R)	0.037 (R)[a]	0.040	0.036	1.08***	0.97****
G7	0.347 (R)	0.046 (R)	0.048	0.045	1.04****	0.98****
G8	0.216 (R)	0.032(NR)	0.042	0.037	1.31*	1.16**
NG9	0.337 (R)	0.016(NR)	0.027	0.021	1.69*	1.31*
NG10	0.163 (S)	0.045 (R)	0.057	0.052	1.27*	1.16**
NG11	0.152 (S)	0.014(NR)	0.022	0.017	1.57*	1.21**
NG12	0.005(NR)	0.006(NR)	0.014	0.011	2.33*	1.83*
NG13	0.281 (R)	0.055 (R)	0.064	0.066	1.16**	1.20**
NG14	0.406 (R)	0.097 (R)	0.080	0.085	0.82**	0.88**
NG15	0.175 (S)	0.022 (NR)	0.027	0.024	1.23*	1.09***
NG16	0.074(NR)	0.014(NR)	0.019	0.017	1.36*	1.21*
NG17	0.023(NR)	0.011(NR)	0.016	0.013	1.45*	1.18**
NG18	0.165 (S)	0.013(NR)	0.018	0.017	1.38*	1.31*
NG19	0.011(NR)	0.006(NR)	0.007	0.005	1.17**	0.83**
NG20	0.056(NR)	0.015(NR)	0.021	0.019	1.40*	1.27*
NG21	0.164 (S)	0.030(NR)	0.038	0.033	1.27*	1.10***
NG22	0.089(NR)	0.021(NR)	0.020	0.018	0.95****	0.86**
NG23	0.785 (R)	0.141(R)	0.143	0.151	1.02****	1.06***
NG24	0.026(NR)	0.017(NR)	0.021	0.015	1.24*	0.88**
NG25	0.007(NR)	0.007(NR)	0.013	0.009	1.86*	1.29*
NG26	0.203 (R)	0.032(NR)	0.025	0.028	0.78*	0.88**
NG27	0.009(NR)	0.004(NR)	0.004	0.002	1.00****	0.50*
NG28	0.501 (R)	0.132(R)	0.128	0.140	0.97****	1.06***
NG29	0.014(NR)	0.010(NR)	0.014	0.011	1.40*	1.10***
NG30	0.102 (S)	0.034(NR)	0.036	0.031	1.06***	0.91***
NG31	0.193 (S)	0.053(R)	0.060	0.058	1.13**	1.09***
NG32	0.405 (R)	0.058(R)	0.063	0.064	1.09***	1.10***

Notes: R: Reactive; S: Suspicious; NR; Non-reactive

[a] Reactive for 15 week expansion limit (0.030 %)

PH: Proportion Harmony (Expansion in DNMi for 28 days / expansion in CPT for 20 weeks)

*****Excellent correlation (PH=0.95-1.05)*

****Good correlation (PH=0.90-1.10)*

***Moderate correlation (PH=0.80-1.20)*

** Poor correlation (PH>1.20 or PH<0.80)*

Another important point in these results, NG12 aggregate tested by DNM1 and DNM2 shows greater expansion than that tested by AMBT method. Such a result is not observed in other aggregates, so the reason will be discussed in following part as considered petrographic properties, chemical composition, mechanical and physical properties of the aggregate. For NG13 (sandstone) and NG14 (sandstone) aggregates, they can be clearly reactive due to high expansion in all methods. DNM1 is best one for NG13 with value of PH=1.16 while DNM2 is the best one for NG14 with value of PH=0.88. NG15 (sandy-limestone), NG18 (siliceous sand), NG21 (basalt), NG30 (basalt) aggregates are suspicious according to the results obtained from AMBT method while they are non-reactive for CPT. DNM2 gives better correlation for NG15, NG21 (PH=1.09 in NG15 and PH=1.10). The degree of correlation for both DNM1 and DNM2 may be seen as good because the values of proportional harmony are in the range of 0.90-1.10. DNM1 gives slightly better correlation with PH=1.06. The degree of correlation is poor for DNM1 and DNM2 applied on NG18 but DNM2 is said to have better correlation. Some of test aggregates are exactly non-reactive materials if tested by both AMBT and CPT methods, for example; NG16 (meta sandstone), NG17 (sparite), NG19 (marble), NG20 (granite), NG22 (granite), NG24 (olivine basalt), NG25 (calcitic dolomite), NG27 (marble), NG29 (half recrystallized limestone). NG16 aggregate shows steady expansion in all methods. As in some non-reactive aggregates, a poor correlation may be seen due to lower expansion rate. DMN2 can be accepted as the better method for NG16. Lower expansion rates (0.011% in AMBT and 0.006% in CPT) stand out in the results of all methods applied on NG19 but unlike in NG16, a moderate correlation can be provided by DNM1 and DNM2. The degree of correlation is at same rate for DNM1 and DNM2 because the values of PH= 1.17 in DNM1 and PH=0.83 in DNM2 are far from ideal “1” value at 0.17 ratio. Although expansion rates for NG20 (0.056% in AMBT and 0.015% in CPT) are not as low as those for NG17 and NG19, a good correlation cannot be procured by the new methods. DNM1 having PH=1.27 is better method for NG20. DNM1 provides an excellent correlation if applied on NG22 (PH=0.95) and NG27 (PH=1.00). DNM2 may be better method for NG25 and NG29 aggregates because it shows a poor correlation but better for NG25 and a good correlation for NG27 (PH=1.10). On the other hand, DNM1 shows a poor correlation for both NG25 (PH=1.86) and NG27 (PH=1.40). As the results indicate, creating a general judgment for these exactly non-reactive aggregates may not be logical because they give conflicting expansion results. NG23 (river sandstone), NG28 (quartz) and NG32 aggregates are examples to exactly reactive material due to higher expansion over the expansion limits of the methods. DNM1 applied on NG23 and NG32 which are very reactive materials (0.785%, 0.405% expansion in AMBT and 0.141%, 0.058% in CPT in turn) is found as the best methods for these river sandstones thanks to their provision of an excellent (NG23)/ a good correlation (NH32). Moreover, DNM1 applied on NG28 shows an excellent correlation with CPT by the value of PH=0.97. NG26 aggregate (limestone) gives a false positive result (reactive for AMBT but not reactive for CPT) while N31 aggregate (dacitic tuff) gives a false negative result (non-reactive or suspicious for AMBT but reactive for CPT). DNM2 shows a moderate correlation (PH=0.88) for NG26 and o good correlation (PH=1.09) for NG31, so it is the best method for these aggregates.

If you need to make a general evaluation, it can be said that DNMs does not false negative or false positive test result except in G5 and G8 aggregates like in AMBT as compared with the results of CPT. This proves another important positive side of the developed new methods. Although G5 aggregate gives false positive results, expansion rates in DNM1 and DNM2 are very slightly lower than 0.04% expansion limit (0.039% in DNM1 and 0.038% in DNM2). Therefore, G5 aggregate cannot be said to disrupt this proposition if considering experimental error margins. For the same reason, G8 aggregate shows similar behaviour with G5 aggregates even though it gives false negative result only if tested by DNM1 (0.042% expansion). Another emphasis about comparison of test methods is that there is no aggregate, which is non-reactive in AMBT but reactive in CPT or developed new methods. In other words, if an aggregate tested by AMBT method is found as non-reactive (expansion lower than 0.1% for 14 days), it can be accepted to be used in concrete structure without an application of another test method.

Table 7.2 summarized the findings of the preliminary evaluation for DNM1 and DNM2 by separating test aggregates into reactivity groups as follows according to the results obtained from the standard AMBT and CPT methods. These groups may help to establish the new method for detecting potential alkali reactivity of all test aggregates in the stage of final decision:

i. Exactly reactive in both AMBT and CPT (R).
ii. Exactly non-reactive in both AMBT and CPT (NR).
iii. Suspicious in AMBT and non-reactive in CPT (SNR).
iv. Suspicious in AMBT and reactive in CPT (SR).
v. Reactive in AMBT and non-reactive in CPT (RNR).
vi. Non-reactive in AMBT and reactive in CPT (NRR).

Table 7.2 Reactivity groups of aggregates and best method for them

Reactivity group	Aggregate	Explanations for the best method
R	G1, G4, G5, G6, G7, NG13, NG14, NG23, NG28, NG32	DNM1 is the best method for G5, NG13, NG23, NG28 and NG32 aggregates while DNM2 is the best method for G1, G4, G6, G7 and NG14 aggregates
NR	G2, NG12, NG16, NG17, NG19, NG20, NG22, NG24, NG25, NG27, NG29	DNM1 is the best method for G2, NG19, NG22, NG27 aggregates while DNM2 is the best method for NG12, NG16, NG17, NG19, NG20, NG24 and NG25 and NG29 aggregates
SNR	G3, NG15, NG18, NG21, NG30	DNM1 is the best method for NG30 aggregate while DNM2 is the best method for G3, NG15, NG18 and NG21 aggregates
SR	NG10, NG31	DNM2 is the best method for both NG10 and NG31
RNR	G8, NG9, NG26	DNM2 is the best method for all aggregates (G8, NG9 and NG26) classified in this reactivity group
NRR	None	None

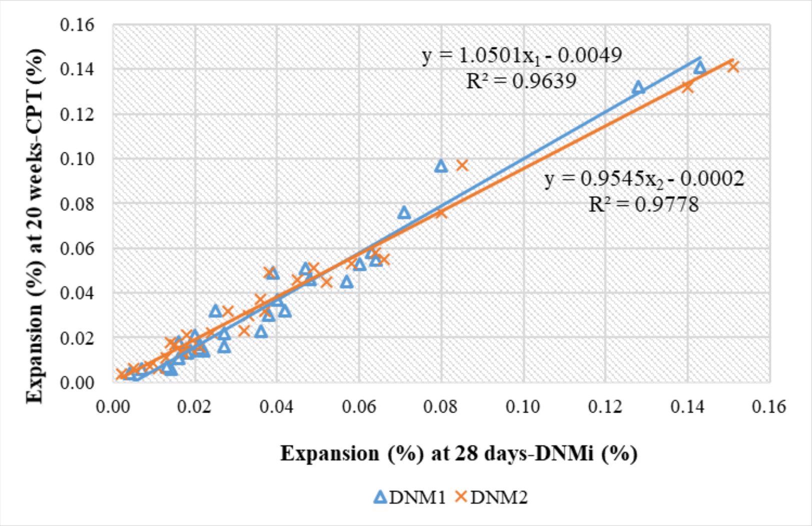
DNM2 pronouncedly comes into prominence to be best method for the aggregates defined in reactivity groups named as SNR, SR and RNR. As for R group, DNM1 give better correlation for 5 types of aggregate like as DNM2, so no method can stand out for this group. On the other hand, DNM2 is best method for NR group because it provides better correlation for 8 types of aggregate while DNM1 does for 5 types of aggregates. According to the preliminary evaluation, DNM2 seems to be the best method for this experimental thesis study but it is not reasonable to give a definite judgment at this stage. Therefore, a regression analysis for data of DNM1 and DNM2 by comparing them with data of CPT needs to be made to reach the final decision.

7.2 Final Decision for the New Method

Regression analysis on DNMs and CPT data will clarify whether DNMs can provide good correlation and which method is the best in addition to the preliminary evaluation mentioned in the previous part.

Figure 7.1 shows correlation parameters obtained by regression analysis on DNMs data and CPT data for all aggregates. When the parameters of regression line equation for DNMs are examined, the target is to provide the ideal equation (Y=X) that means coefficients of “a” and “b” must be “1” and ”0” in turn and correlation coefficient (R^2) must be “1”. Coefficients of “a” for the line equations of DNMs are nearly identical to “1”, but it is slightly better (a=0.9545) in DNM2. Moreover, DNM2 provides better correlation coefficient (R^2=0.9778) than that of DNM1 (R^2=0.9639). Although DNM2 is better in terms of these parameters, the values are very close to each other. On the other hand, “b” coefficient of line equation for DNM2 (b=0.0005) is almost value of the ideal equation (b=0). Consequently, DNM2 can be accepted the best method to detect aggregates for alkali reactivity as considering both the preliminary evaluation and the regression analysis made to compare last data of DNMs and CPT. Finally, this developed new test has been entitled as **“short-term concrete prism test method to assess aggregates for potential alkali reactivity (SCPT)”**.

Latifee has designed a new method named as miniature concrete prism test (MCPT) and compared the results of this method with CPT (ASTM C 1293) taking 1 years [Latifee, 2013]. DNM2 entitled as SCPT developed in this experimental study has some critical advantages if compared it with MCPT. DNM2 lasts for 28 days while MCPT takes 56 days. One of main targets of this thesis study is to design a new method not exceeding 30 days due to construction market condition. When Latifee made a regression analysis for MCPT and CPT, the line equation and correlation coefficient are found as $Y=1.276X_1-0.0091$ and ($R^2=0.968$). DNM2 is clearly observed to provide better parameters in view of the values of a=0.9545, b=0.0002 and $R^2=0.9778$.



Method	Regression Line Equation (Y=aX+b)	Correlation Coefficient (R^2)
DNM1	$Y=1.0501X_1-0.0049$	0.9639
DNM2	**$Y=0.9545X_2-0.0002$**	**0.9778**

Figure 7.1 Regression analysis of DNMs data and CPT data for all aggregates

7.3 Correlation of SCPT with AMBT

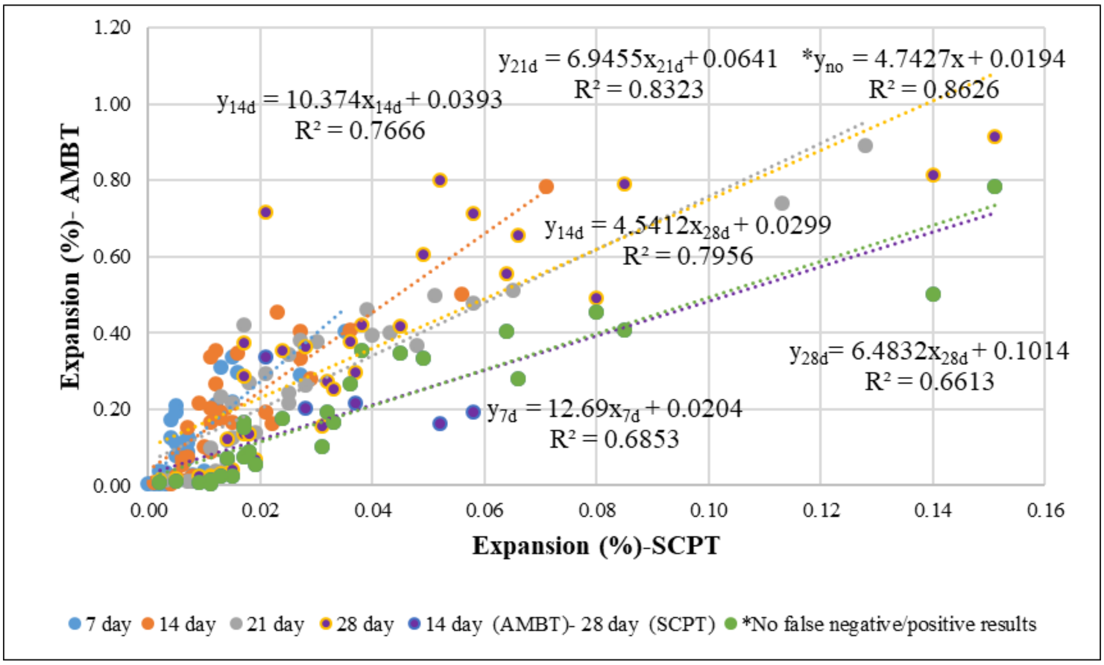
AMBT is not used to make final decision for the best method even though it is a guiding standard test method. The reasons for this are discussed in detail in previous chapters. However, an analysis for correlation of SCPT with AMBT may be useful for crosschecking SCPT data. In this part, time mapping does not need because data of both methods is for 28 days. Expansion rates for 7, 14, 21, 28 days are separately compared to search whether there is a linear relation between SCPT and AMBT. In addition, 14-day expansion rates in AMBT and 28-day expansion rates in SCPT which are completion time of tests are compared. Figure 7.2 shows correlation parameters of regression analysis made by comparing data of SCPT and AMBT.

For correlation of SCPT with AMBT, ideal regression line equation is logical as follow:

Y=Xi that is a=5; b=0 and $R^2= |1|$ (ideal equation)

Based on expansion limits of both test methods (0.2/0.04%), “a” coefficient may be desired as “5.0” in the ideal equation.

With examination of all regression line equations, none of them indicates good correlation. Data obtained from AMBT and SCPT are in different number ranges and rate of expansion increase with time varies substantially due to different exposure condition. Moreover, AMBT may show false negative/positive results. For all mentioned reasons, any reliable numerical relationship between both methods may become impossible. If considered correlation parameters, data from 14-day expansion rates of AMBT and 28-day expansion rates of SCPT shows relatively better correlation. Related line equation of Y_{14d} =4.5412X_{28d}-0.0299 has closest “a” coefficient to ideal equation (a=5.0) even though it cannot provide best “b” coefficient and correlation coefficient (R^2). On the other hand, same comparison is made by extracting false negative/positive results in AMBT. That is not an appropriate comparison way but line equation of *$Y_{14d(no)}$ =4.7427X_{28d}-0.0194 obtained by this way gives the best correlation parameters in terms of “a=4.74”, “b=0.0194” and (R^2=0.86). Herewith, false negative/positive result can be demonstrated as an important disadvantage of AMBT.



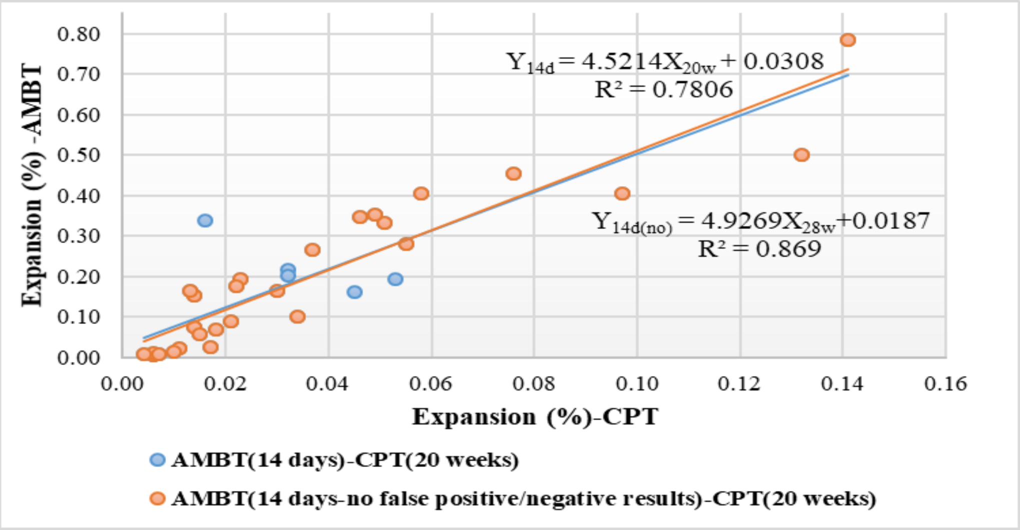
Method/day (y axis)	**Method/day (x axis)**	**Regression Line Equation (Y=aX+b)**	**Correlation Coefficient (R^2)**
AMBT/7	SCPT/7	$Y_{7d}=12.690X_{7d}-0.0204$	0.6853
AMBT/14	SCPT/14	$Y_{14d}=10.374X_{14d}-0.0393$	0.7666
AMBT/21	SCPT/21	$Y_{21d}=6.9455X_{21d}-0.0641$	0.8323
AMBT/28	SCPT/28	$Y_{28d}=6.4832X_{28d}-0.1014$	0.6613
AMBT/14	SCPT/28	$Y_{14d}=4.5412X_{28d}-0.0299$	0.7956
*AMBT/14	SCPT/28	$*Y_{14d(no)}=4.7427X_{28d}-0.0194$	0.8626

*Notes:*Expansion rates of aggregates giving false negative/positive results in AMBT for 14 days is extracted from data of regression analysis.*

Figure 7.2 Correlation of SCPT with AMBT

7.4 Correlation of CPT with AMBT

An analysis for correlation of CPT with AMBT may be useful to compare the performances of SCPT and CPT in view of compatibility with AMBT. In this part, time mapping explained in 6.1.2 needs to be performed because data of CPT and AMBT are for 20 weeks and 28 days in order. Moreover, a regression analysis is made by extracting expansion data of aggregates giving false negative/positive results in AMBT for 14 days.



Method/day (y axis)	**Method/day (x axis)**	**Regression Line Equation (Y=aX+b)**	**Correlation Coefficient (R^2)**
AMBT/14	CPT/28	$Y_{14d}=4.5214X_{28d}-0.0308$	0.7806
AMBT/14 (no false)	CPT/28	$Y_{14d(no)}=4.9269X_{28d}-0.0187$	0.8690

Figure 7.3 Correlation of CPT with AMBT

For correlation of CPT with AMBT, the ideal regression line equation is as described in the previous part due to same expansion limits. Correlation parameters obtained from the regression analysis show that SCPT ($Y_{14d}=4.5412X_{28d}-0.0299$, $R^2=0.7956$) gives better, but not significantly, correlation with AMBT than CPT ($Y_{14d}=4.5214X_{28d}-0.0308$, $R^2=0.7806$) as seen in Figure 7.3. On the other hand, CPT method certainly gives better correlation than SCPT if false negative/positive results in AMBT are neglected. The regression analysis made by this way supports this conclusion because the line equation of CPT method ($Y_{14d(no)}$=4.9269X_{28d}-0.0187, R^2=0.8690) gives better values for all correlation parameters. For example, coefficient of “a=4.9269” is very close to 5.0 in the ideal equation.

7.5 Effect of Petrographic and Mineralogical Properties on AAR expansion

Most test aggregates used for this experimental study are also examined by qualitative mineralogical and petrographic analysis. These aggregates, especially defined with the abbreviation “NG”, have TSE certification. Therefore, the test reports including results of qualitative mineralogical and petrographic analysis could be requested from the companies exploiting aggregate quarries. These reports belong to General Directorate of Mineral Research and Exploration (MTA), Istanbul Technical Unıversity (ITU), Bulent Ecevıt University, Dokuz Eylul University and other test laboratories. Some aggregates defined as basalt were examined for also chemical composition in TSE Chemistry Laboratory. On the other hand, there is no available test report for some river basin sandstones because these types of aggregates are not legally used in construction works. No company has operation licence for exploiting these sand quarries, so the river sandstones, examined in especially preliminary experimental study, were randomly taken their basins. Table 7.3 shows petrographic identification, macroscopic examination, origin, texture, mineralogical/ chemical composition of most aggregates used in this experimental study due to related expressions in the test reports.

First of all, a superficial evaluation will be done on especially aggregate types giving abnormal expansion result by using Table 7.4 that gives susceptible rock types and reactive minerals for AAR. These reactive minerals and rocks are listed according to the degree of reactivity. After that, the effect of petrographic and mineralogical properties on AAR expansion are evaluated by grouping the aggregates into their petrographic identifications.

Table 7.3 Petrographic and mineralogical properties of the aggregates

Agg.	Petrographic identification	Macroscopic examination	Texture	Mineralogical composition
G2	[3,8] Granite	Fine-medium grain	All crystalline euhedral granular	-Plagioclase, orthoclase, quartz, biotite, hornblend, opaque -Plagioclase (43-44%)/main mineral -Orthoclase (26-27%) -Quartz (14-15 %) -Biotite (8-9 %) -Hornblende (5-6%) Main chemical composition: SiO_2 (~65%), Al_2O_3(~14%), Fe_2O_3 (~4), Na_2O (~3%), K_2O (~3%)
G6	[1]Recrystallized limestone	Fine grain	Micro-meso crystalline	-Diabase (~30 %) -Basaltic andesite (~20 %) -Limestone (~25 %) -Granit (~10 %) -Quartz (~5 %)
NG9	[4] River Sandstone	Fine-medium grain,reddy (radyolarite) dark gray, (oolitic)	Oolitic, phaneritic, granodioritic	-Limestone, andesite, granodiorite, sandstone, conglomerate and quartzite -No main mineral
NG10	[5] Perlite/ rhyolitic glass	Volcanic glass, glassy glossy, fine-medium grain, gray,	Perlitic	-Phenocrystalline and microcrystals (17%) -Plagioclase, sanidine, quartz, biotite, volcanic glass
NG11	[1,3] Calcirudite	Fine-medium grain	Detrial	-Quartz (90-95 %)/main mineral -Muscovite/Illite (1-2 %) -Chlorite (0.5-1 %) -Plagioclase (1-2 %)
NG12	[1] Dolimite	Fine grain, light gray, massif	Micro-meso crystalline	-Dolomite (main mineral) -Calcite (minor) -Quartz (minor)
NG13	[1] River Sandstone/ Sedimentary Rocks	Fine-free grain, medium rounded, gravel size,yellowish gray	Detrial	Main Mineral: Quartz, alkali feldspar, mica, rock pieces (chert)
NG15	[1] Sandy Limestone	Medium-coarse-fine grain,Very pale orange, massif, detrail	Detrial, fine grained	-Carbonate grains (~35%) -Fossil grain and shell (~55%) -Intraclast (~10%)
NG16	[1] Meta Sandstone	Fine grain, greenish gray, massif	Detrial	Main minerals: quartz, feldspar, rock pieces (chert), muscovite, chlorite; cement (minor)

Table 7.3 continued

NG17	(6) Sparite	coarse grain, gray, stiff	Hard, tight and compact	Sparite (55%) Calcite (40%) Micrite (5%)
NG18	(3) Siliceous Sand	semi-cornered, gravel to silt size, detrial	No textural maturity	-Metamorphic (52%) sedimentary (48%) -Limestone (29%), marble (12%), muscovite quartz shale (22 %), quartzite (9%), litarenitic sandstone (19%), feldspar (9%) -Mineralogical composition; quartz (33%), calcite (41%), feldspar (12%), muscovite (1%), sericite (1%), sandstone item (12 %)
NG19	(1) Marble	Fine grain, medium,light gray, massif	Granoblastic	Carbonate groups (dolomite, calcite)
NG20	(4) Granite	Fine-medium grain	Holocrystal-line granular	Plagioclase, orthoclase, quartz, hornblende, chlorite, biotite, epidote, titanite Main minerals: Plagioclase (39%) Orthoclase (34-35%) -Quartz (7-8 %) -Biotite (5-6 %) -Hornblende (4-5%) -Chlorite (1-2%) Main chemical composition: SiO_2 (~61%), Al_2O_3(~16%), Fe_2O_3 (~6), Na_2O (~3%), K_2O (~6%)
NG21	(1,8) Basalt	Fine-medium-coarse grain	Porphyritic	Main mineral composition: Fenocrystallines (plagioclase, pyroxene, olivine group minerals) and paste Main chemical composition: SiO_2 (61.5%), Al_2O_3(17.3%), CaO (7.2%), Na_2O (3.4%), K_2O (1.4 %)
NG22	(3) Granite	Fine grain	Holocrystal-line granular	Plagioclase, orthoclase, quartz, biotite, hornblende Main minerals: Plagioclase (40-41%) Orthoclase (26%) -Quartz (24 %) -Biotite (5-5.5 %) -Hornblende (1.5-2%) Main chemical composition: SiO_2 (~72%), Al_2O_3(~14%), Fe_2O_3 (~2), Na_2O (~4%), K_2O (~3%)

Table 7.3 continued

NG24	(2,8) Olivine Basalt	Fine-medium-coarse grain	Volcanic porphyritic	Main mineral compositions: Fenocrystallines (15-20 %); olivine (major), pyroxene (minor) Paste; plagioclase (55-60 %) Main chemical composition: SiO_2 (50.8%), Al_2O_3(17.5%), CaO (9.2%), Na_2O (3.9%), K_2O (1.8 %)
NG25	(1)Calcitic Dolomite	Fine-medium grain, Light brownish gray, massif	Detrial	Main mineral: Carbonate groups (dolomite, calcite)
NG26	(7) Limestone (large of quartz, andesite, granite)	Fine grain	Detrial	-Diabase (~35 %) -Basaltic andesite (~28 %) -Limestone (~21 %) -Granit (~8 %) -Quartz (~4 %) -Marl (~1.5 %) -Radyolarite (~1 %)
NG27	(1) Marble	Fine grain, white, massif	Granoblastic	Carbonate groups (dolomite, calcite)
NG28	(2)Quartz	Fine-medium-coarse grain	Detrial	-Quartz sand (~97%) -Clay minerals (~2%) -Feldspar (~1%) Main chemical composition: SiO_2 (59.7%), Al_2O_3(15.4%), Na_2O (2.9%), K_2O (2.3 %)
NG29	(2)Half-Recrystallized Limestone	Fine grain, gray, fracture	Rhom-bohedral	-Sparitic-secondary calcite (30-32 %) -Primary calcite (64-66%) -Iron oxide yield (2-4%)
NG30	(2)Basalt	Fine-medium-coarse grain	-Volcanic porphyritic/ glomera-porphyritic	-Fenocrystallines (~50%); plagioclase, pyroxene(major), olivine, opaque (minor) -Matrix (~50%); plagioclase microlite, pyroxene (major), volcanic glass (minor) -Main chemical composition: SiO_2 (61.4 %), Al_2O_3(17.1%), CaO (6.9%), Na_2O (3.2%), K_2O (1.3 %)
NG31	(1)Dacitic Tuff	Fine grain, medium light gray, detrial	Hyalopilitic	Quartz, feldspar group minerals (plagioclase, alkali feldspar), mica group mineral (biotite), pumice, volcanic glass

Notes: Test reports belong to; (1)General Directorate of Mineral Research and Exploration (MTA), (2)İstanbul Technical Unıversity (ITU), (3)Dokuz Eylul University (DEU),(4) Bulent Ecevıt University (BEU),(5)Bilgi Ground Laboratory, (7) Kahramanmaras University,(8) Turkish Standard Institute Chemistry Laboratory.

As making general assessment about effect of petrographic and mineralogical properties for 32 types of aggregate on potential alkali reactivity, most river sandstones from Kizilirmak, Sakarya, Gediz, Bartın, and Dicle are clearly seen as potentially very reactive (Table 4.1). The group names of the aggregates expanding extremely are G1, G4, G5, G6, G7, NG13, NG14, NG23, NG32 (exactly reactive-R) as tested by all methods. Sandstones named as G1, G2, G4 G5, G7, NG13, NG14, NG23, NG32 are examples to said exactly reactive. The reason for the high reactivity is most likely related to the amount of silica mineral in the sandstones that is known as the most prominent factor affecting AAR. Accordingly, Table 7.4 shows that amorphous silica is the most reactive mineral. Only two of exactly reactive aggregates (R) named as G6 (limestone) and NG28 (quartz) are not sandstone but like other sandstones, G6 and NG28 aggregates are originated from river or stream basin. The expansion results of exactly reactive aggregates (R) may be observed compatible with their petrographic properties and mineral composition. Increasing concentration of amorphous silica in sandstones and quartz mineral in NG28 aggregate can cause AAR expansion in high rate because Table 7.4 indicates that amorphous silica and quartz are susceptible minerals for AAR.

In this experimental study, petrographic identification of G2, NG20 and NG22 aggregates are defined as “granite”. However, they have different mineral and chemical compositions (Table 7.3). 14-day expansions of G2, NG20, NG22 are, in order, 0.070%, 0.056% and 0.089% for AMBT. Their 20-week expansion rates are 0.018%, 0.015% and 0.021% for CPT. The granites determined as non-reactive show parallel expansion as considering expansion limits of AMBT and CPT. The highest expansion are respectively observed in NG22, NG20 and G2 aggregates in both AMBT and CPT. Noteworthy point on main mineral composition of these aggregates is that quartz ratio in NG22 is quite higher than the others. In addition, the chemical composition analysis shows percent of high purity (SiO_2) silica sand in NG22 (approximately 72% that is highest among these granites). Both two highest percent seems as an important factor for higher expansion for the granites due to alkali aggregate reaction. Stanton stated that a quartz under pressure and cracking with the effect of metamorphism might cause alkali aggregate reaction although there are no quartz polymorphs that may trigger the reaction according to petrographic investigations on granites [Stanton, 1940].

Table 7.4 AAR-susceptible rocks and minerals within aggregates [FHWA, 2010]

Silica minerals according to the decrease in reactivity	**Rocks according to the decrease in reactivity**
Amorphous silica	Volcanic glasses with their tuff
Unstable crystalline silica	Opal such as shales, sandstones, silicified carbonate rocks, diatomite
Chert	Metaquartzite- metaphorical sandstones
Chalcedony	Granitic gneisses
Other cryptocrystalline forms of silica	Deformed granitic gneisses
Metaphorically separated and degraded quartz	Metaphorical rocks including other silica
Deformed quartz	Siliceous - micaceous schists and phyllites
Half-crystallized quartz	well-crystallized volcanic rocks such as latites, andesite, perlite, some basalt, dacites
Pure quartz	Pegmatitic volcanic rocks
	Rocks not including silica

Three types of aggregates (NG21, NG24 and NG30) defined as “basalt” were tested in this study. Measured expansion rates for 14 days are 0.164% in NG21, 0.026% in NG24 and 0.102% in NG30 in AMBT while they are ,in order, 0.030%, 0.017%, 0.034% in CPT. Interesting point in expansion percent of these basalts is that all of them are observed as non-reactive material if tested by CPT even though they are seen as suspicious material on AMBT. Petrographic test reports of these basalts show that mineralogical compositions of NG21 and N30 aggregates are similar to each other while ratio of high purity (SiO_2) silica sand in NG21 and NG30 aggregates is 61-62% higher than that of NG 24, nearly 51%. However, NG30 aggregate shows considerably more expansion than NG21 aggregate if tested by both AMBT and CPT in spite of similar mineral composition, macroscopic and textural structure. This contradictory result may be seen as a proof to inadequacy of petrographic examination for determining potential alkali reactivity of aggregates. In AAR test performed on basalt aggregates, basalt representing acid-median character and basaltic andesite with a matrix formed by wholly volcanic glass are potentially low reactive constituents according to the research and observations made in this study. On the other hand, investigation of volcanic glass with optical microscope has limitations due to very fine grained matrix of the rock.

Limestones in Turkey are known as non-reactive materials for AAR if removed from the quarries. On the other hand, G6, NG15, NG26, NG29 aggregates are limestone as a petrographic definition. These four limestones show different behaviour for the process of alkali aggregate reactivity. G6 aggregate, exactly reactive, consists of some reactive materials such as basaltic andesite (20% by weight) and quartz (20% by weight) as seen in Table 7.4. NG15 aggregate (sandy limestone) is suspicious for AMBT and non-reactive for CPT even though it contains large amounts of reactive carbonate grains (35% by weight). NG26 aggregate (limestone) that is an example to the conflict between AMBT and CPT methods seems to be reactive as tested by AMBT but not reactive for CPT. Reactive materials in NG26 are basaltic andesite (28%) and quartz (4%). G6, NG15 and NG26 are natural aggregates originated from a river while NG29 is mined from a stone quarry. NG29 (half-recrystallized limestone) aggregate is exactly non-reactive material (NR) because it shows very low expansion when tested by all methods. The reason for this may be due to too much calcite in its mineral composition. Calcite in NG29 is one of most known non-reactive rocks. NG29 includes calcite in two different form (1) sparitic-secondary (30-32%); (2) primary calcite (64-66%). NG10 (perlite) and NG31 (dacitic tuff) are the lightest aggregates used in this experimental study as considering the parameters of particle densities (Table 4.1). In addition to the river sandstone, NG10 (perlite/rhyolitic glass) is potentially reactive according to CPT data in spite of being suspicious material for AMBT. This perlite consists of phenocrystalline and microcrystals (17%), plagioclase, sanidine, quartz, biotite, volcanic glass. Table 7.4 indicates that two of them, quartz and volcanic glass, are AAR- susceptible materials. Like NG10, NG31 shows lower expansion than the limit of AMBT but higher expansion than that of CPT. Its mineral composition composes of quartz, volcanic glass (reactive), plagioclase, biotite, pumice. The striking point in the expansion process of NG10 and NG31 is a flash increase in the expansion. 14-day expansions of NG10 and NG31 are, in order, 0.163% and 0.193% while their 28-day of expansions are 0.801% and 0.713% as tested by AMBT method (Table 6.7). Similarly, if tested by CPT method, the flash increase is clearly observed from 1-week to 20-week expansion for both aggregates. Both of NG10 and NG20 consist of quartz and volcanic glass which are defined as a reactive material. The aggregates including quartz do not show unusual increase in expansion regardless of whether they are reactive or not. For example, NG11 (calcirudite) aggregate including large amount of quartz as a main mineral in 90-95% expands gradually. Therefore, the reason for this flash increase in the expansion rates of NG10 and NG31 may be probably due to volcanic glass. Moreover, NG11 aggregate is a logical example to that petrographic examination cannot always give precise result for reactivity of aggregates because it is suspicious for AMBT but non-reactive for CPT in a manner that will not create doubt due to very low expansion for 20 weeks (0.014%) although NG11 aggregate is mostly composed of quartz.

NG12 (dolomite) and NG25 (calcitic dolomite) aggregates show similar expansion behaviour. Both are exactly reactive for all methods because they hardly expand even though very heavy exposure conditions are applied to them in terms of temperature, alkali content and relative humidity. Dolomite is a non-reactive mineral and the main mineral of both types of aggregate. These aggregates also consist of calcite in minor amounts that is non-reactive mineral. Mineral composition may be seen as a crucial factor to affect directly reactivity of both dolomite aggregates. NG17 (sparite), NG19 (marble), NG27 (marble) aggregates are exactly non-reactive aggregates. Table 7.3 shows that NG17 consists of sparite (55%), calcite (40%) and micrite (5%) while NG19 and NG27 aggregates is composed of mostly calcite and dolomite. All mentioned minerals are not included in Table 7.3 showing susceptible rocks and minerals for AAR, so it is natural that these aggregates are found as non-reactive material. In short, petrographic and mineralogical properties of aggregates seem to influence on AAR expansion rate even though it cannot give the exact result. Extra tests must be applied to determine whether aggregate is reactive or not before used in a concrete structure.

7.6 Effect of Chloride Based De-Icers on the Process of AAR

In this experimental study, sodium chloride (NaCl), calcium chloride ($CaCl_2$), magnesium chloride ($MgCl_2$) are used as additive chemical materials to soak solution with sodium hydroxide (NaOH) to regulate the process of alkali aggregate reactivity. The specimens tested by PNM2, PNM3, PNM4 are stored at fixed 0.3NaOH for all methods with extra 2% NaCl in PNM2, 2% $CaCl_2$ in PNM3, 2% $MgCl_2$ in PNM4. The results obtained from this experimental thesis study show that these chloride based de-icers affects AAR expansion in different rates. Expansive enhancer effects in order of the de-icers are as follow (1) NaCl> (2) $CaCl_2$ >(3) $MgCl_2$. Moreover, these results prove the statement of that Sirivivatnanon divalent or cations like as calcium (Ca^{2+}) don't cause expansion in concrete as much as monovalent cations like as sodium and potassium (Na^+, K^+) do [Sirivivatnanon et al, 1987].

Furthermore, the only difference between PNM6 (0.35 N NaOH+ 1% $CaCl_2$) and PNM7 (0.35 N NaOH+ 2% $CaCl_2$) is the percent of $CaCl_2$ in their soak solution. The expansion rates for PNM6 and PNM7 show clearly that increasing $CaCl_2$ ratio causes striking decrease in reactive aggregates while it causes increase in non-reactive aggregates. On the other hand, DNM1 and DNM2 were conducted with 1% $CaCl_2$ and 1.25% $CaCl_2$ but making evaluation is not logical if considering the effect of different cement types.

7.7 Effect of Temperature on AAR Expansion

Effect of temperature can be evaluated by the results of the preliminary experimental study because different temperatures were tested only in the preliminary study. The mortar bars were prepared for the standard accelerated mortar bar method (AMBT) and stored 80 ºC. In addition, mortar bars prepared by the same mixture in AMBT were exposed to temperature of 60 ºC for modified accelerated mortar bar method

(MABT). Expansion rates of almost all aggregates decrease dramatically at especially first days that is an appropriate result to the literature.

Figure 7.4 shows expansion rates of mortar bars prepared for both methods. Expansion rates are comparatively given at 7, 14, 21, 28 days separately. As seen in this figure, fifty percent of error bars are added to expansion data of mortar bars tested by AMBT (80 ºC). This error ratio is very high tolerance, but expansion rates of mortar bars tested at 60 ºC (MABT) are generally lower in such this large proportion at all days. Consequently, temperature has a significant effect that can change AAR expansion dramatically.

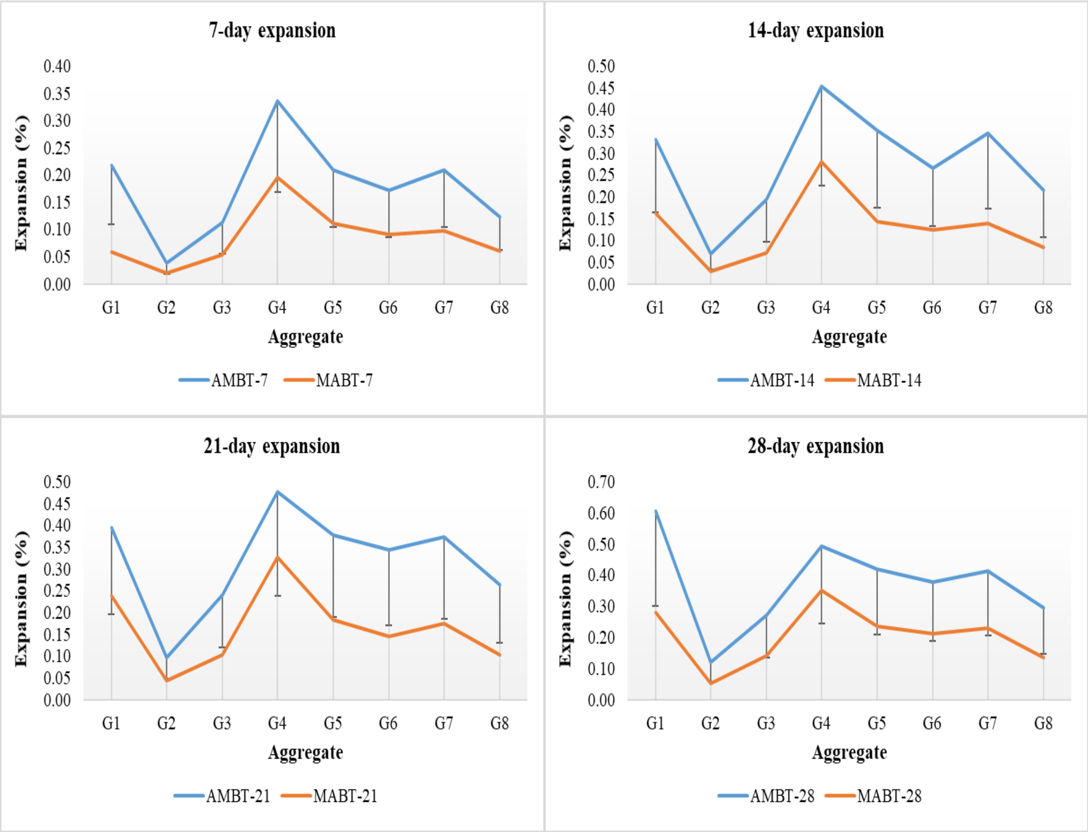


Figure 7.4 Expansion rates of mortar bars at AMBT (80 ºC) and MABT (60 ºC)

7.8 Effect of Cement Type on AAR Expansion

Researches have stated that the type of cement may affect the expansion of mortar bars or concrete prisms in terms of alkali content if sufficient alkali is not present. Two types of CEM I 42.5 R specified by related standards “ASTM C150 or TS EN 197-1” were used to develop the new method in this study. Table 4.2 shows that Type I cement has higher alkali content with Na_2O_{eq}=1.23 while Na_2O_{eq} of Type II cement is 0.94. The effect of cement type can be only explained by comparing PNM6 performed only in the preliminary experimental study and DNM2 because these two methods were performed by the same soak solution but different types of cement. Concrete prisms tested by DNM2 were prepared with type II cement having lower alkali cement. Probably, that’s why they expand in higher rates.

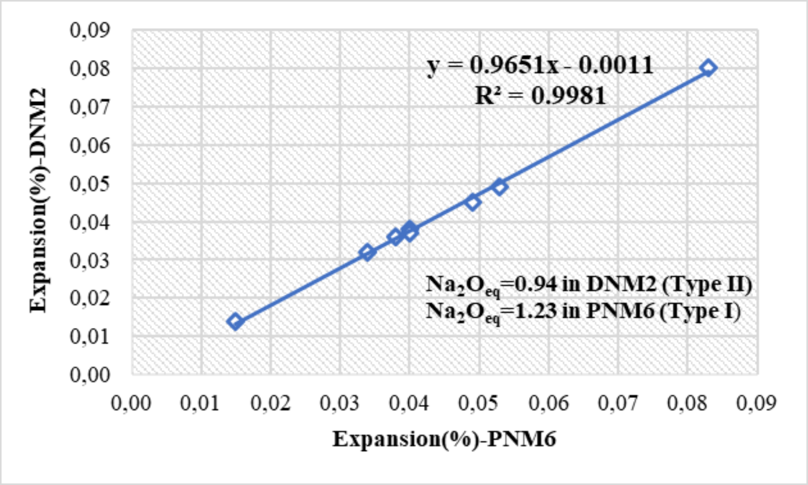


Figure 7.5 Comparison of expansion rates according to cement type

A regression analysis has been made by comparison of expansion rates observed in concrete prisms tested by DNM2 and PNM6 (Figure 7.5). Correlation parameters obtained by the regression analysis indicates that line equation of Y=aXi+b may be converted to Y=aXi because “b=0.0011” coefficient is so small value that it can be neglected. Also, correlation coefficient (R^2=0.9981) can be acceptable as “1.0” that means excellent correlation. Eventually, it is possible to establish a formula for effect of cement type on AAR expansion as considering equivalent sodium oxide (Na_2O_{eq}). Y means expansion rate in prisms tested by DNM2 while X means that by PNM6.

Assume that Y=0.9651X for expansion rate, Φ_{eq}= the ratio of equivalent sodium oxide (Na_2O_{eq}) = Y (Na_2O_{eq})/ X (Na_2O_{eq})

Given: Na_2O_{eq}=0.94 (DNM2) and Na_2O_{eq}=1.23 (PNM6) → Φ_{eq}=0.94/1.23=0.7642

Substitute Φ_{eq} to the line equation of $Y= \Phi_{eq}\,(0.9651/0.7642)X$ → $Y=1.26\Phi_{eq}\,X$

Established formula of **$Y=1.26\Phi_{eq}X$** that seems to be acceptable for this experimental study.

7.9 Effect of Mechanical and Physical Properties on ASR Expansion

The parameters of mechanical and physical properties of aggregate are ρ_a, ρ_{rd}, ρ_{ssd} for the particle density, Los Angeles coefficient for fragmentation (LA), water content (W_C) and water absorption (WA_{24}). The results for these parameters are given in Table 4.1. Except water absorption (WA_{24}), other parameters cannot be said to have a consistent effect on AAR expansion of test aggregates according to the results obtained from this experimental study. The aggregates showing extreme expansion such as G4, NG14, NG23, NG28 and NG32 have higher absorption capacity. However, NG10 and NG31 aggregates, which are the lightest, can absorb water in the highest rate among all test aggregates due to their high porosity although they don't expand at a remarkable rate. As for exactly non-reactive aggregates such as NG17, NG25, NG27 and NG29, their water absorption capacities are comparatively lower. For example, NG29 aggregate absorbs water in 0.9% by fine form and 0.4% by coarse form for 24 hours while NG23 does in 1.8% by fine form and 0.6% by coarse form. Hence, water absorption may be one of possible factor affecting AAR process because it can be related to the porosity ratio affecting the resistance of aggregates to chemical reaction. On the other hand, it may be not logical to claim conclusively that water absorption has a direct effect on AAR expansion in views of all expansion result.

7.10 Comparison of SCPT with Field Performance of Aggregates

The best way to examine potential alkali-reactivity of aggregates is the evaluation of past field performance. For this purpose, the following should be investigated in existing concrete structures:

i. Cement amount of concrete, alkali amount of cement and water-cement ratio.
ii. The annual moisture rate of the area where the concrete structure is located.
iii. Whether the concrete in question is more than 15 years old.
iv. Whether the external factors that will affect the future structure are more severe than the existing structure.
v. Whether mineral or chemical additives are used in the existing concrete structure [Farny & Kosmatka, 1997].

If there is not enough information about the field performance of the aggregate or if there is a change in the material to be used in the new building, laboratory tests should be performed to determine the potential reactivity. In fact, whether the existing aggregate stock is the same as the aggregate used in the field concrete should be determined by petrographic analysis. Petrographic analysis of concrete cores taken from existing concrete also provides information on severity of the damage, if any. However, necessary permissions for taking concrete coring could not be taken because it is highly probable that structural and visual problems may arise from the existing old buildings including test aggregates used in this study. Therefore, it was not possible to evaluate the field performance of these aggregates in detail.

In a concrete structure, formation of AAR does not depend solely on the reactivity of the aggregate as mentioned in previous parts. Type of cement, degree of temperature, relative humidity, many external factors, mineral/chemical admixtures in concrete etc. have important roles on process of AAR. In this study, a superficial research can be conducted on the field performances of some test aggregates to check the reliability of the new developed method (SCPT). The suppliers declared the concrete structures consisting of these aggregates so that their field performances could be started to be observed. Table 7.5 gives field performances of some test aggregates with pictures of the existing structures.

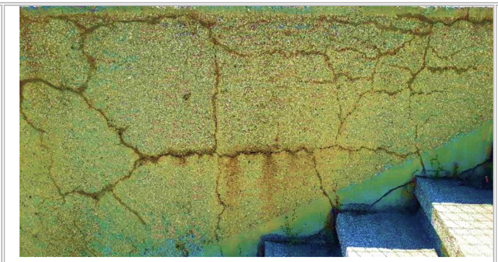
ASR and ACR are known as most common types of alkali-aggregate reaction. Visual symptoms indicating damages induced by both reaction usually resembles each other. Map-cracking, displacement, pop-outs, exudations are major symptoms of ASR and ACR.

In comparison of SCPT with field performance, non-reactive aggregates for all test methods and field performance are excluded. Concrete structures consisting of suspicious and reactive test aggregates were observed in view of visual manifestations from formation of AAR damage. Achieving a detailed quantitative or qualitative comparison may not be possible due to lack of information on constituent of the existing concretes and different environment conditions (relative humidity, temperature). Moreover, AAR is a time dependent reaction, so age of concrete structure is very important for severity of deterioration. For all these reasons, it is inevitable that there is some contradictory result even though the results obtained from the new method (SCPT) are mostly consistent with field performance of aggregates. For example, NG28 (quartz) aggregate is very reactive in SCPT but no visual symptoms for AAR damage were observed in the concrete structures with this aggregate. However, an intense map cracking can be clearly seen on the concrete prism (Table 7.5) prepared as test specimens with NG28 aggregate. Moreover, NG10 (perlite) and NG31 (tuff) aggregates do not show reactivity in the field even if they are reactive in SCPT.

Table 7.5 Comparison of SCPT with field performance of aggregates

Group name	SCPT (%)	Field Performance	Structure affected by AAR	Age of structure
G1	0.049 (Reactive)	Reactive	 *A concrete curb stone in Çankırı*	10 years
G4	0.080 (Reactive in high level)	Reactive	 *A ground concrete in a high school in Çankırı*	10 years
G5	0.038 (Non-reactive)	Non-reactive	No visual of AAR-damage observed	--
G6	0.036 (Non-reactive)	Non-reactive	No visual of AAR-damage observed	--
G7	0.045 (Reactive)	Reactive	 *A manhole cover concrete in Kastamonu*	15 years
G8	0.037 (Non-reactive)	Non-reactive	No visual of AAR-damage observed	
NG10	0.052 (Reactive)	Non-reactive	No visual of AAR-damage observed	
NG13	0.066 (Reactive)	Reactive	 *Concrete pavement cover block in Bartın*	15 years

Table 7.5 continued

NG14	0.085	Reactive	 *A ladder wall in Diyarbakır*	15 years
NG23	0.151 (Reactive in very high level)	Reactive	 *A ground concrete in a high school in Bilecik*	10 years
NG28	0.140	Non-reactive	No visual of AAR-damage observed  *Test Specimens prepared by NG28 aggregate tested by SCPT*	---
NG31	0.058	Non-reactive	No visual of AAR-damage observed	
NG32	0.064	Reactive	 *A wall concrete in a car park in Samsun*	20 years

CHAPTER 8

SUMMARY, CONCLUSIONS, LIMITATIONS AND RECOMMENDATIONS

8.1 Summary

The results of this experimental study prove that establishing an alternative new method to available standard test methods may be possible to improve predictability for potential alkali reactivity of aggregates. In preliminary experimental study, performances of seven different new methods were evaluated on 8 types of aggregates to provide best correlation with CPT data. After completion of preliminary study, two new methods in addition to seven methods were applied on new 24 types of aggregates (totally 32) by modifying best preliminary methods to improve correlation. As results of the studies, a new method has been finalized as considering the degree of correlation and entitled as “short-term concrete prism test method to assess aggregates for potential alkali reactivity (SCPT)”. Comparison of SCPT versus CPT data gives the best linear equation that is Y(CPT data)=0.9545X (SCPT data)+0.0002. The specifications of the finalized new method are given as below:

i. Test period: 28 days.
ii. Soak solution: 0.35N NaOH+ 1% $CaCl_2$.
iii. Storage temperature: 60 °C.
iv. Expansion limit: 0.04%.
v. Dimension of test mould: 50 mm x 50 mm x 200 mm concrete prism.
vi. Water/cement ratio: 0.50.
vii. Maximum aggregate size: 16 mm.

viii. Aggregate proportion: nearly 72% of total concrete weight (45% fine, 55% coarse).

ix. Alkali equivalent (Na_2Oe eq.) of cement: 0.90-0.95%.

8.2 Conclusions

Based on the results obtained from this experimental study, the conclusions can be mentioned as follows:

1) SCPT method has been regulated to provide a good correlation with CPT method because it represents the most similar exposure conditions that can be seen in the field. The specifications of SCPT method can provide some advantages to eliminate problems experienced in standard mortar bar and concrete prism test method. Firstly, SCPT takes 28 days unlike long-term CPT methods taking months even years. Secondly, excessive exposure conditions in AMBT are alleviated thanks to usage of soak solution consists of 0.35N NaOH+ 1% $CaCl_2$ instead of 1N NaOH and taking down storage temperature from 80 ºC to 60 ºC. Moreover, alkali leaching experienced in CPT methods can be prevented by SCPT due to storage in a soak solution. Thirdly, preparation of concrete mixture is made easier by usage of 50 mm x 50 mm x 200 mm narrowed concrete prisms instead of 75 mm x 75 mm x 300 mm. Fourthly, that 16 mm maximum size of coarse aggregate instead of 22.4 mm increase workability of concrete. Moreover, modified size distribution of aggregate in the concrete mixture can help increasing workability. Lastly, SCPT examines fine and coarse aggregate together, so excessive crushing of aggregates in the mortar bar test can be precluded.

2) AMBT may give false negative/positive test results for some aggregates. On the other hand, SCPT data shows excellent compatibility with CPT data on determining reactivity of the test aggregates. Except G5 aggregate, all aggregates tested by both SCPT and CPT methods shows similar degree of

reactivity. G5 aggregate tested by SCPT expands in 0.038% that is slightly lower than 0.04% expansion limit while it is observed as potentially reactive material if tested by CPT. However, this expansion rate can be acceptable within usual margin of error from uncertainty of measurement in the experiment studies. In this manner, one drawback of the available test methods has been also eliminated thanks to the developed new method (SCPT).

3) Regression analysis made for determining degree of correlation between AMBT and CPT data proves that there is no good correlation between each other. The reason for this incompatibility may be due to false positive/ negative expansion results from AMBT. For example, the test results exhibit that aggregate types of G1, G4, G5, G6, G7, G8, NG9, NG13, NG14, NG23, NG26, NG28, NG32 are potentially reactive aggregates and also types of G3, NG10, NG11, NG15, NG18, NG21, NG30, NG31 are suspicious aggregates for alkali reactivity in AMBT. However, types of only G1, G4, G5, G6, G7, NG10, NG13, NG14, NG23, NG28, NG31, NG32 are potentially reactive aggregates if tested by CPT. Moreover, NG10 gives a contradictory result because it is defined as suspicious material for AMBT but potentially reactive for CPT (false negative result). On the other hand, G8, NG9, NG26 are found as potentially reactive materials for AMBT method although they are not reactive according to the results from CPT method. Like as mentioned in the literature review, the 14-day short-term test may give unreliable result due to excessive test condition. However, AMBT method can provide good correlation CPT if false negative/positive results in AMBT are extracted from the comparison analysis. This reveals that the major disadvantage of AMBT is false negative/positive results. SCPT gives better correlation with AMBT than CPT for all test results but poor correlation for extracted false negative/positive results.

4) Sodium chloride (NaCl) has been determined as the most effective chloride based de-icers on alkali aggregate reaction in concrete because of accelerating the reaction. Concrete prism specimens exposed to a soak solution with calcium chloride ($CaCl_2$) instead of NaCl shows less expansion in comparison to specimens exposed to NaCl de-icer solution. Moreover, it is observed that magnesium chloride ($MgCl_2$) does not contribute to alkali aggregate reaction as much as other chlorides.

5) The concrete prism specimens tested in the developed new test methods expand in a sharp increase after especially 14 days if soak solution of mentioned methods consist of sodium hydroxide (NaOH) with $CaCl_2$. Furthermore, only difference between PNM6 (0.35N NaOH+ 1% $CaCl_2$) and PNM7 (0.35N NaOH+ 2% $CaCl_2$) is the percent of $CaCl_2$ in their soak solutions. The expansion rates for PNM6 and PNM7 show clearly that increasing $CaCl_2$ ratio causes striking decrease in reactive aggregates while it causes increase in non-reactive aggregates. Therefore, $CaCl_2$ should be examined in detail in views of AAR expansion mechanism.

6) As making non-detailed evaluation on petrographic and mineralogical properties of tested aggregates, it can be observed that reactive mineral or rocks like as sandstone, quartz, volcanic glass and high purity (SiO_2) silica sand have an effect on expansion rate from AAR. Additionally, volcanic glass has been found to cause a flash increase in the expansions of NG10 and NG31. Petrographic and mineralogical properties of aggregates seems having an effect on potential alkali reactivity according to the results obtained from this experimental study even though it cannot give exact results. Thus, extra tests must be applied to determine whether aggregate is reactive or not before used in a concrete structure.

7) The results of modified accelerated mortar bar test method (60 °C) show that storage temperature is too important accelerator factor for expansion from

AAR in especially very reactive aggregates because dramatic decline in expansion percent in mortar bar specimen appears as storage temperature is reduced from 80 ºC to 60 ºC.

8) The Cement type having a higher alkali content increases expansion as two types of cement with different alkali content are compared. In this experimental study, regression analysis has been made by comparison of expansion rates observed in samples tested by DNM2 and PNM6. As taking equivalent sodium oxide of the cements into consideration, a formula has been established for the effect of cement type according to only expansion rates of preliminary test aggregates named as G*i*. This formula is as follow:

   $$“Y=1.26\Phi eqX”$$

   where; Y,X= expansion (%)

   Φeq= the ratio of equivalent sodium oxide = Y (Na_2O_{eq})/ X (Na_2O_{eq}).

9) Except water absorption (WA_{24}), other parameters of mechanical and physical properties of test aggregates cannot be said to have a consistent effect on AAR expansion. Water absorption may be seen as one of possible factor affecting AAR process due to its relation to the porosity ratio affecting the resistance of aggregates to chemical reaction.

10) In comparison of SCPT with field performance, it may not be possible to obtain an excellent compatibility between them because lack of information on constituent of the existing concretes and different environment conditions (relative humidity, temperature). However, SCPT gives generally compatible results with field performance of aggregates in view of visual manifestations formed by AAR on existing concrete structure.

8.3 Limitations

The main aim of this experimental study is to overcome drawbacks of standard AAR test methods. Development of SCPT can be considered to have minimized these drawbacks. However, SCPT has a few limitations as below:

1) $CaCl_2$, which is the fundamental material of the soak solution prepared for SCPT, may react with some constituents and cause formation of unexpected products due to the unclear points of the role of calcium in the AAR mechanism. Therefore, it may be necessary to make detailed petrographical examination on the aggregates and investigate behaviour of the constituents of these aggregates when they react with calcium.

2) SCPT is an accelerated concrete prism test method by increasing test conditions of the standard CPT methods. Concrete prism samples with some type of aggregates may expand slowly, so expansion process of these samples cannot be observed during the 28-day test period. That's why false results can be given by SCPT.

8.4 Recommendations

Considering the results obtained from the study for development of a new method, the following works can be recommended for further researches:

1) Performance of SCPT should be checked by long-term concrete prism test method such as ASTM C1293 taking 1or 2 years.

2) Test aggregates used in this study should be followed for their field performances to make precise control of performance of SCPT. Firstly, history of their field performances should be observed in detail. Secondly, future construction works built by using these aggregates should be followed for even years

3) $CaCl_2$ (calcium chloride) should be examined in detail in view of the mechanism of alkali aggregate expansion as considering the role of calcium on the gel expansion.

4) Whether a significant correlation between the rates CaO/SiO_2 and expansion of AAR products is provided or not should be investigated.

5) SCPT should be performed to detect potential alkali reactivity of the reinforced concrete specimens.

6) Mitigation strategies of AAR on reinforced concrete can be examined in depth with SCPT.

7) In order to increase the alkalinity, the use of potassium hydroxide instead of sodium hydroxide needs to be investigated to examine its effect on the structure of AAR products.

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APPENDICES

Table A.1 Raw data of the preliminary experimental study

G1 aggregate	Exp (%)	7 d	14 d	21 d	28 d	10 w	15 w	20 w
AMBT	1	0.238	0.360	0.431	0.654			
	2	0.208	0.304	0.369	0.565			
	3	0.211	0.332	0.385	0.601			
	Av:	**0.219**	**0.332**	**0.395**	**0.607**			
MABT	1	0.059	0.165	0.240	0.286			
	2	0.058	0.165	0.238	0.283			
	3	0.059	0.162	0.235	0.278			
	Av:	**0.059**	**0.164**	**0.238**	**0.282**			
CPT	1	0.005	0.012	0.020	0.025	0.041	0.048	0.052
	2	0.005	0.012	0.021	0.022	0.042	0.048	0.051
	3	0.005	0.011	0.017	0.021	0.040	0.047	0.051
	Av:	**0.005**	**0.012**	**0.019**	**0.023**	**0.041**	**0.048**	**0.051**
PNM1	1	0.024	0.047	0.056	0.018			
	2	0.025	0.049	0.061	0.020			
	3	0.030	0.056	0.062	0.021			
	Av:	**0.026**	**0.051**	**0.060**	**0.068**			
PNM2	1	0.018	0.035	0.053	0.063			
	2	0.016	0.031	0.052	0.060			
	3	0.021	0.037	0.057	0.065			
	Av:	**0.018**	**0.034**	**0.054**	**0.063**			
PNM3	1	0.028	0.081	0.136	0.163			
	2	0.025	0.079	0.131	0.159			
	3	0.031	0.084	0.128	0.156			
	Av:	**0.028**	**0.081**	**0.132**	**0.159**			
PNM4	1	0.006	0.023	0.035	0.045			
	2	0.008	0.024	0.037	0.048			
	3	0.011	0.027	0.040	0.039			
	Av:	**0.008**	**0.025**	**0.037**	**0.044**			

Table A.1 continued

PNM5	1	0.008	0.014	0.026	0.031			
	2	0.006	0.010	0.017	0.022			
	3	0.005	0.008	0.014	0.020			
	Av:	**0.008**	**0.011**	**0.019**	**0.024**			
PNM6	1	0.012	0.024	0.041	0.053			
	2	0.012	0.025	0.042	0.052			
	3	0.013	0.027	0.043	0.054			
	Av:	**0.012**	**0.025**	**0.042**	**0.053**			
PNM7	1	0.013	0.021	0.039	0.046			
	2	0.012	0.022	0.038	0.046			
	3	0.013	0.025	0.040	0.045			
	Av:	**0.013**	**0.023**	**0.039**	**0.046**			
G2 aggregate	**Exp (%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.040	0.070	0.096	0.123			
	2	0.037	0.068	0.093	0.112			
	3	0.041	0.071	0.102	0.131			
	Av:	**0.039**	**0.070**	**0.097**	**0.122**			
MABT	1	0.023	0.028	0.041	0.058			
	2	0.021	0.030	0.048	0.054			
	3	0.019	0.032	0.046	0.053			
	Av:	**0.021**	**0.030**	**0.045**	**0.055**			
CPT	1	0.001	0.003	0.004	0.007	0.015	0.017	0.019
	2	0.000	0.002	0.005	0.007	0.013	0.014	0.016
	3	0.001	0.003	0.006	0.008	0.015	0.016	0.019
	Av:	**0.001**	**0.003**	**0.004**	**0.007**	**0.014**	**0.016**	**0.018**
PNM1	1	0.011	0.015	0.021	0.029			
	2	0.015	0.019	0.025	0.035			
	3	0.009	0.013	0.019	0.025			
	Av:	**0.012**	**0.016**	**0.022**	**0.030**			

Table A.1 continued

PNM2	1	0.003	0.004	0.016	0.024			
	2	0.005	0.007	0.020	0.027			
	3	0.003	0.005	0.017	0.025			
	Av:	**0.004**	**0.005**	**0.018**	**0.025**			
PNM3	1	0.017	0.022	0.027	0.035			
	2	0.017	0.024	0.032	0.036			
	3	0.019	0.025	0.031	0.036			
	Av:	**0.018**	**0.024**	**0.030**	**0.036**			
PNM4	1	0.002	0.006	0.012	0.013			
	2	0.001	0.006	0.008	0.014			
	3	0.002	0.006	0.012	0.016			
	Av:	**0.002**	**0.005**	**0.011**	**0.014**			
PNM5	1	0.002	0.003	0.007	0.011			
	2	0.001	0.002	0.006	0.009			
	3	0.002	0.002	0.007	0.007			
	Av:	**0.002**	**0.003**	**0.007**	**0.009**			
PNM6	1	0.004	0.008	0.014	0.016			
	2	0.004	0.007	0.011	0.013			
	3	0.005	0.006	0.011	0.015			
	Av:	**0.004**	**0.007**	**0.012**	**0.015**			
PNM7	1	0.002	0.007	0.012	0.016			
	2	0.003	0.009	0.011	0.014			
	3	0.002	0.008	0.014	0.017			
	Av:	**0.002**	**0.008**	**0.012**	**0.016**			
G3 aggregate	**Exp (%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.125	0.205	0.249	0.289			
	2	0.104	0.184	0.238	0.270			
	3	0.110	0.194	0.238	0.262			
	Av:	**0.113**	**0.194**	**0.242**	**0.274**			

Table A.1 continued

MABT	1	0.045	0.058	0.102	0.133			
	2	0.054	0.072	0.098	0.144			
	3	0.065	0.090	0.112	0.146			
	Av:	**0.055**	**0.073**	**0.104**	**0.141**			
CPT	1	0.004	0.008	0.009	0.010	0.017	0.020	0.022
	2	0.003	0.008	0.008	0.011	0.017	0.020	0.020
	3	0.004	0.006	0.008	0.011	0.018	0.023	0.026
	Av:	**0.004**	**0.007**	**0.009**	**0.011**	**0.017**	**0.021**	**0.023**
PNM1	1	0.005	0.015	0.019	0.028			
	2	0.006	0.015	0.025	0.032			
	3	0.006	0.015	0.024	0.032			
	Av:	**0.006**	**0.015**	**0.023**	**0.031**			
PNM2	1	0.003	0.011	0.015	0.026			
	2	0.003	0.010	0.018	0.025			
	3	0.006	0.010	0.022	0.025			
	Av:	**0.004**	**0.011**	**0.018**	**0.025**			
PNM3	1	0.018	0.031	0.044	0.055			
	2	0.018	0.022	0.038	0.050			
	3	0.020	0.036	0.044	0.056			
	Av:	**0.019**	**0.030**	**0.042**	**0.054**			
PNM4	1	0.001	0.011	0.018	0.030			
	2	0.002	0.008	0.018	0.030			
	3	0.002	0.009	0.020	0.031			
	Av:	**0.002**	**0.009**	**0.019**	**0.031**			
PNM5	1	0.002	0.005	0.009	0.011			
	2	0.001	0.005	0.009	0.012			
	3	0.001	0.006	0.012	0.015			
	Av:	**0.001**	**0.006**	**0.010**	**0.013**			
PNM6	1	0.004	0.011	0.023	0.035			
	2	0.003	0.010	0.020	0.032			
	3	0.002	0.010	0.022	0.036			
	Av:	**0.003**	**0.010**	**0.022**	**0.034**			

Table A.1 continued

PNM7	1	0.005	0.010	0.024	0.034			
	2	0.005	0.010	0.022	0.033			
	3	0.005	0.012	0.025	0.035			
	Av:	**0.005**	**0.011**	**0.024**	**0.034**			
G4 aggregate	**Exp (%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.334	0.437	0.461	0.482			
	2	0.356	0.467	0.496	0.504			
	3	0.322	0.459	0.478	0.494			
	Av:	**0.337**	**0.454**	**0.478**	**0.493**			
MABT	1	0.183	0.276	0.311	0.344			
	2	0.201	0.301	0.345	0.354			
	3	0.204	0.268	0.329	0.361			
	Av:	**0.196**	**0.282**	**0.328**	**0.353**			
CPT	1	0.019	0.029	0.034	0.040	0.046	0.058	0.074
	2	0.021	0.031	0.034	0.038	0.046	0.063	0.078
	3	0.018	0.031	0.036	0.043	0.048	0.063	0.075
	Av:	**0.019**	**0.030**	**0.035**	**0.040**	**0.047**	**0.061**	**0.076**
PNM1	1	0.021	0.048	0.081	0.110			
	2	0.020	0.049	0.085	0.115			
	3	0.021	0.048	0.082	0.110			
	Av:	**0.021**	**0.048**	**0.083**	**0.112**			
PNM2	1	0.018	0.021	0.069	0.112			
	2	0.018	0.020	0.066	0.103			
	3	0.019	0.020	0.065	0.104			
	Av:	**0.018**	**0.021**	**0.067**	**0.106**			
PNM3	1	0.029	0.072	0.117	0.180			
	2	0.026	0.071	0.120	0.182			
	3	0.030	0.080	0.127	0.191			
	Av:	**0.028**	**0.074**	**0.121**	**0.184**			

Table A.1 continued

PNM4	1	0.016	0.024	0.057	0.079			
	2	0.015	0.024	0.060	0.083			
	3	0.018	0.025	0.058	0.080			
	Av:	**0.016**	**0.025**	**0.058**	**0.081**			
PNM5	1	0.007	0.011	0.018	0.028			
	2	0.007	0.010	0.018	0.030			
	3	0.007	0.011	0.018	0.031			
	Av:	**0.004**	**0.011**	**0.018**	**0.030**			
PNM6	1	0.019	0.027	0.060	0.082			
	2	0.016	0.028	0.062	0.085			
	3	0.021	0.026	0.060	0.081			
	Av:	**0.019**	**0.027**	**0.061**	**0.083**			
PNM7	1	0.015	0.025	0.051	0.069			
	2	0.016	0.021	0.047	0.064			
	3	0.021	0.023	0.053	0.071			
	Av:	**0.017**	**0.023**	**0.050**	**0.068**			
G5 aggregate	**Exp (%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.206	0.351	0.378	0.415			
	2	0.202	0.343	0.369	0.408			
	3	0.219	0.365	0.391	0.442			
	Av:	**0.209**	**0.353**	**0.379**	**0.421**			
MABT	1	0.109	0.154	0.208	0.254			
	2	0.128	0.146	0.169	0.245			
	3	0.099	0.134	0.178	0.214			
	Av:	**0.112**	**0.145**	**0.185**	**0.238**			
CPT	1	0.011	0.018	0.022	0.030	0.037	0.040	0.048
	2	0.011	0.014	0.020	0.024	0.032	0.038	0.046
	3	0.015	0.020	0.025	0.028	0.039	0.046	0.052
	Av:	**0.012**	**0.017**	**0.022**	**0.027**	**0.036**	**0.044**	**0.049**

Table A.1 continued

PNM1	1	0.018	0.022	0.045	0.072			
	2	0.017	0.021	0.040	0.070			
	3	0.013	0.019	0.040	0.067			
	Av:	**0.016**	**0.021**	**0.042**	**0.070**			
PNM2	1	0.008	0.016	0.029	0.035			
	2	0.005	0.011	0.019	0.026			
	3	0.009	0.015	0.026	0.033			
	Av:	**0.007**	**0.014**	**0.025**	**0.031**			
PNM3	1	0.022	0.030	0.042	0.066			
	2	0.020	0.029	0.040	0.060			
	3	0.017	0.025	0.036	0.057			
	Av:	**0.020**	**0.028**	**0.039**	**0.061**			
PNM4	1	0.010	0.011	0.025	0.035			
	2	0.009	0.011	0.027	0.038			
	3	0.009	0.012	0.033	0.041			
	Av:	**0.009**	**0.011**	**0.028**	**0.038**			
PNM5	1	0.003	0.008	0.013	0.020			
	2	0.002	0.006	0.013	0.019			
	3	0.004	0.009	0.017	0.023			
	Av:	**0.003**	**0.007**	**0.014**	**0.021**			
PNM6	1	0.011	0.013	0.027	0.037			
	2	0.008	0.014	0.030	0.043			
	3	0.001	0.011	0.032	0.040			
	Av:	**0.007**	**0.013**	**0.030**	**0.040**			
PNM7	1	0.010	0.015	0.025	0.036			
	2	0.010	0.014	0.024	0.034			
	3	0.007	0.010	0.019	0.030			
	Av:	**0.009**	**0.013**	**0.023**	**0.033**			

Table A.1 continued

G6 aggregate	**Exp (%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	
AMBT	1	0.171	0.263	0.336	0.374			
	2	0.164	0.285	0.352	0.394			
	3	0.181	0.254	0.345	0.365			
	Av:	**0.172**	**0.267**	**0.344**	**0.378**			
MABT	1	0.075	0.132	0.152	0.203			
	2	0.094	0.116	0.154	0.224			
	3	0.105	0.125	0.132	0.215			
	Av:	**0.091**	**0.124**	**0.146**	**0.214**			
CPT	1	0.011	0.016	0.019	0.014	0.028	0.035	0.040
	2	0.015	0.020	0.021	0.015	0.026	0.032	0.036
	3	0.009	0.018	0.020	0.014	0.025	0.032	0.035
	Av:	**0.012**	**0.018**	**0.020**	**0.022**	**0.026**	**0.033**	**0.037**
PNM1	1	0.014	0.022	0.036	0.052			
	2	0.018	0.027	0.040	0.059			
	3	0.015	0.023	0.034	0.053			
	Av:	**0.016**	**0.024**	**0.037**	**0.055**			
PNM2	1	0.005	0.018	0.022	0.040			
	2	0.003	0.015	0.018	0.038			
	3	0.002	0.015	0.021	0.044			
	Av:	**0.003**	**0.016**	**0.020**	**0.041**			
PNM3	1	0.016	0.027	0.042	0.068			
	2	0.025	0.039	0.061	0.087			
	3	0.018	0.030	0.048	0.074			
	Av:	**0.020**	**0.032**	**0.050**	**0.076**			
PNM4	1	0.001	0.010	0.019	0.030			
	2	0.004	0.010	0.021	0.031			
	3	0.005	0.011	0.023	0.034			
	Av:	**0.003**	**0.010**	**0.021**	**0.032**			
PNM5	1	0.001	0.005	0.015	0.019			
	2	0.003	0.010	0.022	0.026			
	3	0.001	0.006	0.018	0.022			
	Av:	**0.002**	**0.007**	**0.018**	**0.022**			

Table A.1 continued

PNM6	1	0.003	0.012	0.022	0.038			
	2	0.007	0.013	0.025	0.037			
	3	0.007	0.014	0.024	0.040			
	Av:	**0.006**	**0.013**	**0.024**	**0.038**			
PNM7	1	0.002	0.014	0.024	0.035			
	2	0.002	0.014	0.026	0.034			
	3	0.003	0.014	0.023	0.033			
	Av:	**0.002**	**0.014**	**0.024**	**0.034**			
G7 aggregate	**Exp (%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	
AMBT	1	0.212	0.345	0.369	0.410			
	2	0.200	0.335	0.361	0.407			
	3	0.219	0.360	0.391	0.422			
	Av:	**0.210**	**0.347**	**0.374**	**0.413**			
MABT	1	0.094	0.133	0.169	0.229			
	2	0.108	0.148	0.183	0.244			
	3	0.093	0.135	0.173	0.224			
	Av:	**0.098**	**0.139**	**0.175**	**0.232**			
CPT	1	0.010	0.016	0.020	0.028	0.035	0.037	0.048
	2	0.010	0.013	0.017	0.026	0.031	0.035	0.044
	3	0.008	0.015	0.020	0.027	0.036	0.040	0.047
	Av:	**0.009**	**0.015**	**0.019**	**0.027**	**0.034**	**0.037**	**0.046**
PNM1	1	0.016	0.020	0.037	0.073			
	2	0.015	0.018	0.038	0.075			
	3	0.011	0.017	0.041	0.074			
	Av:	**0.014**	**0.018**	**0.039**	**0.074**			
PNM2	1	0.009	0.018	0.031	0.038			
	2	0.006	0.014	0.022	0.029			
	3	0.011	0.017	0.023	0.035			
	Av:	**0.009**	**0.016**	**0.025**	**0.034**			

Table A.1 continued

PNM3	1	0.026	0.034	0.048	0.071			
	2	0.022	0.028	0.041	0.066			
	3	0.019	0.027	0.039	0.060			
	Av:	**0.022**	**0.030**	**0.043**	**0.066**			
PNM4	1	0.011	0.012	0.026	0.037			
	2	0.008	0.013	0.028	0.040			
	3	0.007	0.012	0.035	0.043			
	Av:	**0.009**	**0.012**	**0.030**	**0.040**			
PNM5	1	0.001	0.007	0.012	0.017			
	2	0.002	0.006	0.014	0.021			
	3	0.002	0.008	0.014	0.018			
	Av:	**0.002**	**0.007**	**0.013**	**0.019**			
PNM6	1	0.015	0.018	0.032	0.048			
	2	0.015	0.017	0.033	0.049			
	3	0.011	0.019	0.038	0.050			
	Av:	**0.014**	**0.018**	**0.034**	**0.049**			
PNM7	1	0.005	0.011	0.023	0.039			
	2	0.006	0.012	0.024	0.044			
	3	0.008	0.014	0.026	0.043			
	Av:	**0.006**	**0.012**	**0.024**	**0.042**			
G8 aggregate	**Exp (%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.118	0.207	0.254	0.290			
	2	0.124	0.217	0.261	0.294			
	3	0.131	0.225	0.276	0.303			
	Av:	**0.124**	**0.216**	**0.264**	**0.296**			
MABT	1	0.064	0.088	0.106	0.140			
	2	0.061	0.085	0.108	0.138			
	3	0.059	0.081	0.096	0.131			
	Av:	**0.061**	**0.085**	**0.103**	**0.136**			

Table A.1 continued

CPT	1	0.005	0.009	0.012	0.015	0.020	0.025	0.031
	2	0.005	0.008	0.012	0.015	0.021	0.026	0.032
	3	0.005	0.009	0.013	0.017	0.023	0.029	0.033
	Av:	**0.005**	**0.009**	**0.012**	**0.016**	**0.021**	**0.027**	**0.032**
PNM1	1	0.008	0.016	0.026	0.041			
	2	0.012	0.018	0.028	0.043			
	3	0.013	0.018	0.031	0.042			
	Av:	**0.011**	**0.017**	**0.028**	**0.042**			
PNM2	1	0.010	0.014	0.026	0.036			
	2	0.011	0.016	0.027	0.042			
	3	0.009	0.016	0.031	0.045			
	Av:	**0.010**	**0.015**	**0.028**	**0.041**			
PNM3	1	0.024	0.034	0.048	0.059			
	2	0.020	0.031	0.045	0.055			
	3	0.022	0.036	0.049	0.061			
	Av:	**0.022**	**0.030**	**0.042**	**0.054**			
PNM4	1	0.003	0.012	0.024	0.038			
	2	0.004	0.010	0.020	0.034			
	3	0.004	0.010	0.023	0.041			
	Av:	**0.004**	**0.011**	**0.022**	**0.038**			
PNM5	1	0.001	0.005	0.007	0.013			
	2	0.001	0.005	0.007	0.015			
	3	0.001	0.004	0.010	0.019			
	Av:	**0.001**	**0.005**	**0.008**	**0.016**			
PNM6	1	0.008	0.012	0.027	0.041			
	2	0.008	0.014	0.027	0.037			
	3	0.006	0.011	0.030	0.042			
	Av:	**0.007**	**0.012**	**0.028**	**0.040**			
PNM7	1	0.006	0.015	0.033	0.045			
	2	0.005	0.016	0.030	0.043			
	3	0.003	0.012	0.029	0.040			
	Av:	**0.005**	**0.014**	**0.031**	**0.043**			

Table A.2 Raw data of the latter experimental study

G1 aggregate	**Exp (%)**	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.238	0.360	0.431	0.654			
	2	0.208	0.304	0.369	0.565			
	3	0.211	0.332	0.385	0.601			
	Av:	**0.219**	**0.332**	**0.395**	**0.607**			
CPT	1	0.005	0.012	0.020	0.025	0.041	0.048	0.052
	2	0.005	0.012	0.021	0.022	0.042	0.048	0.051
	3	0.005	0.011	0.017	0.021	0.040	0.047	0.051
	Av:	**0.005**	**0.012**	**0.019**	**0.023**	**0.041**	**0.048**	**0.051**
DNM1	1	0.014	0.027	0.039	0.045			
	2	0.018	0.031	0.045	0.051			
	3	0.016	0.027	0.038	0.048			
	Av:	**0.016**	**0.028**	**0.041**	**0.047**			
DNM2	1	0.014	0.026	0.035	0.046			
	2	0.014	0.029	0.044	0.052			
	3	0.017	0.026	0.040	0.050			
	Av:	**0.015**	**0.027**	**0.040**	**0.049**			
G2 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.040	0.070	0.096	0.123			
	2	0.037	0.068	0.093	0.112			
	3	0.041	0.071	0.102	0.131			
	Av:	**0.039**	**0.070**	**0.097**	**0.122**			
CPT	1	0.001	0.003	0.004	0.007	0.015	0.017	0.019
	2	0.000	0.002	0.005	0.007	0.013	0.014	0.016
	3	0.001	0.003	0.006	0.008	0.015	0.016	0.019
	Av:	**0.001**	**0.003**	**0.004**	**0.007**	**0.014**	**0.016**	**0.018**
DNM1	1	0.003	0.007	0.010	0.016			
	2	0.004	0.007	0.011	0.015			
	3	0.004	0.010	0.012	0.016			
	Av:	**0.004**	**0.008**	**0.011**	**0.016**			

Table A.2 continued

DNM2	1	0.002	0.005	0.009	0.012			
	2	0.002	0.005	0.010	0.013			
	3	0.003	0.008	0.013	0.017			
	Av:	**0.002**	**0.006**	**0.011**	**0.014**			
G3 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.125	0.205	0.249	0.289			
	2	0.104	0.184	0.238	0.270			
	3	0.110	0.194	0.238	0.262			
	Av:	**0.113**	**0.194**	**0.242**	**0.274**			
CPT	1	0.004	0.008	0.009	0.010	0.017	0.020	0.022
	2	0.003	0.008	0.008	0.011	0.017	0.020	0.020
	3	0.004	0.006	0.008	0.011	0.018	0.023	0.026
	Av:	**0.004**	**0.007**	**0.009**	**0.011**	**0.017**	**0.021**	**0.023**
DNM1	1	0.002	0.012	0.029	0.041			
	2	0.003	0.011	0.027	0.037			
	3	0.003	0.009	0.023	0.031			
	Av:	**0.003**	**0.011**	**0.026**	**0.036**			
DNM2	1	0.006	0.012	0.025	0.029			
	2	0.006	0.015	0.027	0.034			
	3	0.004	0.011	0.024	0.032			
	Av:	**0.005**	**0.013**	**0.025**	**0.032**			
G4 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.334	0.437	0.461	0.482			
	2	0.356	0.467	0.496	0.504			
	3	0.322	0.459	0.478	0.494			
	Av:	**0.337**	**0.454**	**0.478**	**0.493**			
CPT	1	0.019	0.029	0.034	0.040	0.046	0.058	0.074
	2	0.021	0.031	0.034	0.038	0.046	0.063	0.078
	3	0.018	0.031	0.036	0.043	0.048	0.063	0.075
	Av:	**0.019**	**0.030**	**0.035**	**0.040**	**0.047**	**0.061**	**0.076**

Table A.2 continued

DNM1	1	0.020	0.025	0.058	0.072			
	2	0.020	0.025	0.058	0.071			
	3	0.018	0.024	0.055	0.071			
	Av:	**0.019**	**0.025**	**0.057**	**0.071**			
DNM2	1	0.015	0.024	0.060	0.080			
	2	0.013	0.023	0.056	0.079			
	3	0.016	0.023	0.057	0.080			
	Av:	**0.015**	**0.023**	**0.058**	**0.080**			
G5 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.206	0.351	0.378	0.415			
	2	0.202	0.343	0.369	0.408			
	3	0.219	0.365	0.391	0.442			
	Av:	**0.209**	**0.353**	**0.379**	**0.421**			
CPT	1	0.011	0.018	0.022	0.030	0.037	0.040	0.048
	2	0.011	0.014	0.020	0.024	0.032	0.038	0.046
	3	0.015	0.020	0.025	0.028	0.039	0.046	0.052
	Av:	**0.012**	**0.017**	**0.022**	**0.027**	**0.036**	**0.044**	**0.049**
DNM1	1	0.007	0.014	0.028	0.038			
	2	0.008	0.012	0.025	0.037			
	3	0.010	0.015	0.030	0.042			
	Av:	**0.008**	**0.014**	**0.028**	**0.039**			
DNM2	1	0.005	0.011	0.025	0.038			
	2	0.006	0.011	0.027	0.035			
	3	0.007	0.013	0.030	0.042			
	Av:	**0.005**	**0.012**	**0.027**	**0.038**			
G6 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.171	0.263	0.336	0.374			
	2	0.164	0.285	0.352	0.394			
	3	0.181	0.254	0.345	0.365			
	Av:	**0.172**	**0.267**	**0.344**	**0.378**			

Table A.2 continued

CPT	1	0.011	0.016	0.019	0.014	0.028	0.035	0.040
	2	0.015	0.020	0.021	0.015	0.026	0.032	0.036
	3	0.009	0.018	0.020	0.014	0.025	0.032	0.035
	Av:	**0.012**	**0.018**	**0.020**	**0.022**	**0.026**	**0.033**	**0.037**
DNM1	1	0.005	0.011	0.025	0.038			
	2	0.006	0.011	0.027	0.035			
	3	0.007	0.013	0.030	0.042			
	Av:	**0.006**	**0.014**	**0.028**	**0.040**			
DNM2	1	0.004	0.012	0.026	0.037			
	2	0.004	0.012	0.026	0.038			
	3	0.004	0.011	0.023	0.033			
	Av:	**0.004**	**0.012**	**0.025**	**0.036**			
G7 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.212	0.345	0.369	0.410			
	2	0.200	0.335	0.361	0.407			
	3	0.219	0.360	0.391	0.422			
	Av:	**0.210**	**0.347**	**0.374**	**0.413**			
CPT	1	0.010	0.016	0.020	0.028	0.035	0.037	0.048
	2	0.010	0.013	0.017	0.026	0.031	0.035	0.044
	3	0.008	0.015	0.020	0.027	0.036	0.040	0.047
	Av:	**0.009**	**0.015**	**0.019**	**0.027**	**0.034**	**0.037**	**0.046**
DNM1	1	0.012	0.016	0.030	0.052			
	2	0.009	0.012	0.025	0.046			
	3	0.012	0.015	0.026	0.047			
	Av:	**0.011**	**0.014**	**0.027**	**0.048**			
DNM2	1	0.012	0.014	0.027	0.044			
	2	0.012	0.015	0.028	0.044			
	3	0.013	0.019	0.034	0.046			
	Av:	**0.012**	**0.016**	**0.030**	**0.045**			

Table A.2 continued

G8 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.118	0.207	0.254	0.290			
	2	0.124	0.217	0.261	0.294			
	3	0.131	0.225	0.276	0.303			
	Av:	**0.124**	**0.216**	**0.264**	**0.296**			
CPT	1	0.005	0.009	0.012	0.015	0.020	0.025	0.031
	2	0.005	0.008	0.012	0.015	0.021	0.026	0.032
	3	0.005	0.009	0.013	0.017	0.023	0.029	0.033
	Av:	**0.005**	**0.009**	**0.012**	**0.016**	**0.021**	**0.027**	**0.032**
DNM1	1	0.007	0.010	0.029	0.043			
	2	0.005	0.010	0.029	0.040			
	3	0.008	0.013	0.032	0.043			
	Av:	**0.007**	**0.011**	**0.030**	**0.042**			
DNM2	1	0.004	0.010	0.031	0.045			
	2	0.004	0.010	0.030	0.043			
	3	0.003	0.009	0.030	0.046			
	Av:	**0.004**	**0.009**	**0.030**	**0.045**			
NG9 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.207	0.354	0.453	0.763			
	2	0.181	0.322	0.395	0.687			
	3	0.189	0.336	0.416	0.698			
	Av:	**0.192**	**0.337**	**0.421**	**0.716**			
CPT	1	0.005	0.006	0.008	0.008	0.012	0.013	0.017
	2	0.004	0.007	0.008	0.008	0.012	0.014	0.016
	3	0.003	0.005	0.006	0.006	0.010	0.012	0.016
	Av:	**0.004**	**0.006**	**0.007**	**0.007**	**0.011**	**0.013**	**0.016**
DNM1	1	0.005	0.011	0.018	0.028			
	2	0.005	0.011	0.020	0.029			
	3	0.003	0.009	0.017	0.024			
	Av:	**0.004**	**0.010**	**0.018**	**0.027**			

Table A.2 continued

DNM2	1	0.005	0.011	0.017	0.019			
	2	0.005	0.012	0.019	0.024			
	3	0.004	0.010	0.016	0.021			
	Av:	**0.005**	**0.011**	**0.017**	**0.021**			
NG10 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.038	0.180	0.529	0.876			
	2	0.022	0.148	0.408	0.749			
	3	0.025	0.162	0.446	0.779			
	Av:	**0.028**	**0.163**	**0.461**	**0.801**			
CPT	1	0.006	0.012	0.014	0.017	0.028	0.040	0.047
	2	0.005	0.012	0.015	0.017	0.027	0.040	0.048
	3	0.004	0.010	0.011	0.015	0.024	0.035	0.041
	Av:	**0.005**	**0.011**	**0.013**	**0.016**	**0.026**	**0.038**	**0.045**
DNM1	1	0.012	0.026	0.045	0.060			
	2	0.012	0.025	0.045	0.060			
	3	0.010	0.022	0.040	0.053			
	Av:	**0.011**	**0.024**	**0.043**	**0.058**			
DNM2	1	0.008	0.023	0.041	0.050			
	2	0.008	0.023	0.040	0.055			
	3	0.007	0.021	0.037	0.052			
	Av:	**0.008**	**0.022**	**0.039**	**0.052**			
NG11 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.042	0.160	0.226	0.391			
	2	0.032	0.146	0.205	0.358			
	3	0.034	0.151	0.213	0.370			
	Av:	**0.036**	**0.152**	**0.215**	**0.373**			
CPT	1	0.004	0.006	0.009	0.009	0.012	0.015	0.016
	2	0.003	0.006	0.010	0.009	0.011	0.015	0.017
	3	0.002	0.004	0.006	0.007	0.008	0.010	0.010
	Av:	**0.003**	**0.005**	**0.008**	**0.008**	**0.010**	**0.013**	**0.014**

Table A.2 continued

DNM1	1	0.006	0.011	0.019	0.024			
	2	0.006	0.010	0.020	0.024			
	3	0.004	0.007	0.016	0.019			
	Av:	**0.005**	**0.009**	**0.018**	**0.022**			
DNM2	1	0.003	0.008	0.017	0.015			
	2	0.003	0.008	0.016	0.019			
	3	0.002	0.006	0.013	0.017			
	Av:	**0.003**	**0.007**	**0.015**	**0.017**			
NG12 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.006	0.006	0.011	0.026			
	2	0.005	0.006	0.011	0.028			
	3	0.004	0.004	0.009	0.025			
	Av:	**0.005**	**0.005**	**0.010**	**0.026**			
CPT	1	0.002	0.004	0.004	0.004	0.005	0.007	0.008
	2	0.001	0.004	0.005	0.004	0.006	0.006	0.007
	3	0.000	0.002	0.001	0.002	0.002	0.005	0.004
	Av:	**0.001**	**0.003**	**0.003**	**0.003**	**0.004**	**0.006**	**0.006**
DNM1	1	0.002	0.008	0.010	0.016			
	2	0.003	0.008	0.011	0.015			
	3	0.002	0.006	0.010	0.012			
	Av:	**0.002**	**0.007**	**0.010**	**0.014**			
DNM2	1	0.002	0.005	0.009	0.010			
	2	0.002	0.005	0.009	0.013			
	3	0.001	0.003	0.007	0.011			
	Av:	**0.002**	**0.004**	**0.008**	**0.011**			
NG13 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.197	0.293	0.381	0.674			
	2	0.187	0.276	0.358	0.643			
	3	0.184	0.275	0.361	0.652			
	Av:	**0.189**	**0.281**	**0.367**	**0.656**			

Table A.2 continued

CPT	1	0.012	0.016	0.021	0.025	0.041	0.051	0.058
	2	0.011	0.016	0.021	0.026	0.040	0.047	0.056
	3	0.010	0.013	0.018	0.022	0.034	0.042	0.052
	Av:	**0.011**	**0.015**	**0.020**	**0.024**	**0.038**	**0.047**	**0.055**
DNM1	1	0.016	0.031	0.046	0.066			
	2	0.015	0.027	0.047	0.065			
	3	0.009	0.022	0.041	0.062			
	Av:	**0.013**	**0.027**	**0.045**	**0.064**			
DNM2	1	0.015	0.033	0.050	0.068			
	2	0.014	0.029	0.049	0.067			
	3	0.008	0.026	0.046	0.064			
	Av:	**0.012**	**0.029**	**0.048**	**0.066**			
NG14 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.320	0.419	0.526	0.808			
	2	0.309	0.402	0.503	0.777			
	3	0.305	0.398	0.506	0.781			
	Av:	**0.311**	**0.406**	**0.512**	**0.789**			
CPT	1	0.013	0.019	0.024	0.034	0.066	0.081	0.101
	2	0.012	0.019	0.024	0.033	0.062	0.079	0.097
	3	0.011	0.016	0.021	0.027	0.057	0.075	0.094
	Av:	**0.012**	**0.018**	**0.023**	**0.031**	**0.062**	**0.078**	**0.097**
DNM1	1	0.014	0.036	0.069	0.082			
	2	0.013	0.032	0.070	0.081			
	3	0.007	0.027	0.064	0.077			
	Av:	**0.011**	**0.032**	**0.068**	**0.080**			
DNM2	1	0.016	0.040	0.067	0.087			
	2	0.015	0.036	0.066	0.085			
	3	0.009	0.033	0.063	0.083			
	Av:	**0.013**	**0.036**	**0.065**	**0.085**			

Table A.2 continued

NG15 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.116	0.184	0.281	0.365			
	2	0.111	0.170	0.266	0.345			
	3	0.112	0.171	0.267	0.349			
	Av:	**0.113**	**0.175**	**0.271**	**0.353**			
CPT	1	0.005	0.008	0.010	0.012	0.020	0.025	0.024
	2	0.004	0.008	0.010	0.013	0.019	0.021	0.023
	3	0.003	0.005	0.007	0.009	0.013	0.016	0.019
	Av:	**0.004**	**0.007**	**0.009**	**0.011**	**0.017**	**0.021**	**0.022**
DNM1	1	0.007	0.013	0.020	0.029			
	2	0.007	0.011	0.021	0.028			
	3	0.005	0.010	0.017	0.025			
	Av:	**0.006**	**0.011**	**0.019**	**0.027**			
DNM2	1	0.008	0.017	0.020	0.026			
	2	0.008	0.013	0.019	0.025			
	3	0.006	0.010	0.016	0.022			
	Av:	**0.007**	**0.013**	**0.018**	**0.024**			
NG16 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.030	0.079	0.123	0.141			
	2	0.025	0.070	0.115	0.132			
	3	0.026	0.072	0.114	0.133			
	Av:	**0.027**	**0.074**	**0.117**	**0.135**			
CPT	1	0.003	0.006	0.006	0.009	0.012	0.015	0.016
	2	0.002	0.006	0.006	0.010	0.013	0.013	0.015
	3	0.002	0.004	0.003	0.006	0.009	0.011	0.011
	Av:	**0.002**	**0.005**	**0.005**	**0.008**	**0.011**	**0.013**	**0.014**
DNM1	1	0.006	0.010	0.017	0.021			
	2	0.006	0.009	0.018	0.020			
	3	0.004	0.008	0.014	0.017			
	Av:	**0.005**	**0.009**	**0.016**	**0.019**			

Table A.2 continued

DNM2	1	0.004	0.011	0.016	0.019			
	2	0.004	0.007	0.015	0.018			
	3	0.002	0.004	0.012	0.015			
	Av:	**0.003**	**0.007**	**0.014**	**0.017**			
NG17 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.017	0.025	0.030	0.037			
	2	0.013	0.023	0.027	0.029			
	3	0.014	0.022	0.026	0.030			
	Av:	**0.015**	**0.023**	**0.028**	**0.032**			
CPT	1	0.002	0.002	0.003	0.004	0.007	0.010	0.012
	2	0.001	0.002	0.003	0.005	0.008	0.008	0.012
	3	0.001	0.000	0.000	0.001	0.004	0.006	0.009
	Av:	**0.001**	**0.001**	**0.002**	**0.003**	**0.006**	**0.008**	**0.011**
DNM1	1	0.005	0.008	0.015	0.018			
	2	0.005	0.007	0.016	0.017			
	3	0.003	0.006	0.012	0.014			
	Av:	**0.004**	**0.007**	**0.014**	**0.016**			
DNM2	1	0.004	0.009	0.013	0.015			
	2	0.004	0.005	0.012	0.014			
	3	0.002	0.002	0.009	0.011			
	Av:	**0.003**	**0.005**	**0.011**	**0.013**			
NG18 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.075	0.170	0.242	0.299			
	2	0.070	0.161	0.227	0.279			
	3	0.071	0.164	0.232	0.284			
	Av:	**0.072**	**0.165**	**0.234**	**0.287**			
CPT	1	0.006	0.007	0.008	0.008	0.011	0.014	0.014
	2	0.005	0.007	0.008	0.009	0.012	0.012	0.014
	3	0.005	0.005	0.005	0.005	0.008	0.010	0.011
	Av:	**0.005**	**0.006**	**0.007**	**0.007**	**0.010**	**0.012**	**0.013**

Table A.2 continued

DNM1	1	0.007	0.013	0.016	0.019			
	2	0.007	0.012	0.017	0.019			
	3	0.005	0.011	0.013	0.016			
	Av:	**0.006**	**0.012**	**0.015**	**0.018**			
DNM2	1	0.008	0.015	0.015	0.018			
	2	0.008	0.011	0.014	0.018			
	3	0.006	0.008	0.011	0.016			
	Av:	**0.007**	**0.011**	**0.013**	**0.017**			
NG19 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.006	0.011	0.016	0.020			
	2	0.005	0.010	0.013	0.017			
	3	0.006	0.011	0.015	0.017			
	Av:	**0.006**	**0.011**	**0.015**	**0.018**			
CPT	1	0.002	0.002	0.003	0.002	0.005	0.006	0.007
	2	0.001	0.002	0.003	0.003	0.006	0.005	0.007
	3	0.001	0.000	0.000	0.002	0.002	0.005	0.005
	Av:	**0.001**	**0.001**	**0.002**	**0.002**	**0.004**	**0.005**	**0.006**
DNM1	1	0.003	0.004	0.006	0.008			
	2	0.003	0.003	0.006	0.008			
	3	0.001	0.002	0.004	0.006			
	Av:	**0.002**	**0.003**	**0.005**	**0.007**			
DNM2	1	0.002	0.006	0.003	0.006			
	2	0.002	0.002	0.003	0.005			
	3	0.000	0.001	0.002	0.004			
	Av:	**0.001**	**0.002**	**0.003**	**0.005**			
NG20 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.013	0.057	0.065	0.067			
	2	0.014	0.054	0.060	0.063			
	3	0.014	0.058	0.062	0.064			
	Av:	**0.014**	**0.056**	**0.062**	**0.065**			

Table A.2 continued

CPT	1	0.002	0.000	0.004	0.004	0.008	0.013	0.016
	2	0.001	0.002	0.004	0.005	0.010	0.012	0.016
	3	0.001	0.000	0.002	0.004	0.008	0.012	0.014
	Av:	**0.001**	**0.001**	**0.003**	**0.004**	**0.009**	**0.012**	**0.015**
DNM1	1	0.002	0.008	0.016	0.020			
	2	0.004	0.007	0.018	0.022			
	3	0.002	0.006	0.016	0.020			
	Av:	**0.003**	**0.007**	**0.017**	**0.021**			
DNM2	1	0.003	0.006	0.015	0.017			
	2	0.003	0.005	0.013	0.021			
	3	0.001	0.007	0.013	0.018			
	Av:	**0.002**	**0.006**	**0.014**	**0.019**			
NG21 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.088	0.161	0.210	0.245			
	2	0.089	0.164	0.217	0.258			
	3	0.089	0.166	0.215	0.254			
	Av:	**0.089**	**0.164**	**0.214**	**0.252**			
CPT	1	0.003	0.006	0.012	0.015	0.022	0.028	0.028
	2	0.002	0.008	0.012	0.016	0.024	0.027	0.031
	3	0.002	0.006	0.010	0.015	0.022	0.027	0.031
	Av:	**0.002**	**0.007**	**0.011**	**0.015**	**0.023**	**0.027**	**0.030**
DNM1	1	0.008	0.017	0.026	0.037			
	2	0.010	0.016	0.028	0.039			
	3	0.008	0.015	0.026	0.037			
	Av:	**0.009**	**0.016**	**0.027**	**0.038**			
DNM2	1	0.008	0.015	0.026	0.032			
	2	0.008	0.014	0.024	0.035			
	3	0.006	0.016	0.024	0.032			
	Av:	**0.007**	**0.015**	**0.025**	**0.033**			

Table A.2 continued

NG22 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.022	0.086	0.121	0.130			
	2	0.023	0.089	0.129	0.140			
	3	0.023	0.091	0.125	0.138			
	Av:	**0.023**	**0.089**	**0.125**	**0.136**			
CPT	1	0.003	0.003	0.008	0.009	0.013	0.019	0.019
	2	0.002	0.005	0.008	0.010	0.015	0.018	0.021
	3	0.002	0.003	0.006	0.009	0.013	0.017	0.022
	Av:	**0.002**	**0.004**	**0.007**	**0.009**	**0.014**	**0.018**	**0.021**
DNM1	1	0.004	0.013	0.018	0.019			
	2	0.006	0.012	0.020	0.021			
	3	0.004	0.011	0.018	0.019			
	Av:	**0.005**	**0.012**	**0.019**	**0.020**			
DNM2	1	0.005	0.011	0.016	0.017			
	2	0.005	0.010	0.014	0.020			
	3	0.003	0.012	0.014	0.018			
	Av:	**0.004**	**0.011**	**0.015**	**0.018**			
NG23 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.397	0.777	0.882	0.901			
	2	0.454	0.835	0.940	0.969			
	3	0.365	0.742	0.854	0.873			
	Av:	**0.405**	**0.785**	**0.892**	**0.914**			
CPT	1	0.015	0.025	0.038	0.052	0.090	0.112	0.127
	2	0.012	0.028	0.037	0.050	0.108	0.135	0.159
	3	0.014	0.025	0.034	0.046	0.095	0.122	0.136
	Av:	**0.014**	**0.026**	**0.036**	**0.049**	**0.098**	**0.123**	**0.141**
DNM1	1	0.041	0.059	0.108	0.129			
	2	0.039	0.077	0.131	0.161			
	3	0.035	0.064	0.118	0.138			
	Av:	**0.038**	**0.067**	**0.119**	**0.143**			

Table A.2 continued

DNM2	1	0.038	0.069	0.118	0.137			
	2	0.036	0.077	0.140	0.169			
	3	0.031	0.068	0.127	0.146			
	Av:	**0.035**	**0.071**	**0.128**	**0.151**			
NG24 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.013	0.025	0.037	0.038			
	2	0.014	0.026	0.042	0.042			
	3	0.014	0.028	0.038	0.043			
	Av:	**0.014**	**0.026**	**0.039**	**0.041**			
CPT	1	0.003	0.003	0.006	0.007	0.011	0.016	0.015
	2	0.002	0.005	0.006	0.008	0.013	0.015	0.017
	3	0.002	0.003	0.004	0.007	0.011	0.014	0.018
	Av:	**0.002**	**0.004**	**0.005**	**0.007**	**0.012**	**0.015**	**0.017**
DNM1	1	0.005	0.012	0.018	0.020			
	2	0.007	0.011	0.020	0.022			
	3	0.005	0.010	0.018	0.020			
	Av:	**0.006**	**0.011**	**0.019**	**0.021**			
DNM2	1	0.005	0.008	0.013	0.014			
	2	0.005	0.007	0.011	0.017			
	3	0.003	0.009	0.011	0.015			
	Av:	**0.004**	**0.008**	**0.012**	**0.015**			
NG25 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.005	0.006	0.010	0.023			
	2	0.006	0.007	0.013	0.024			
	3	0.006	0.008	0.010	0.021			
	Av:	**0.006**	**0.007**	**0.011**	**0.023**			
CPT	1	0.002	0.000	0.003	0.004	0.004	0.007	0.006
	2	0.001	0.002	0.003	0.005	0.006	0.007	0.007
	3	0.001	0.000	0.001	0.004	0.004	0.006	0.008
	Av:	**0.001**	**0.001**	**0.002**	**0.004**	**0.005**	**0.007**	**0.007**

Table A.2 continued

DNM1	1	0.001	0.007	0.010	0.012			
	2	0.003	0.006	0.012	0.014			
	3	0.001	0.005	0.010	0.012			
	Av:	**0.002**	**0.006**	**0.011**	**0.013**			
DNM2	1	0.004	0.004	0.008	0.008			
	2	0.004	0.003	0.006	0.011			
	3	0.002	0.005	0.006	0.009			
	Av:	**0.003**	**0.004**	**0.007**	**0.009**			
NG26 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.122	0.195	0.290	0.356			
	2	0.129	0.206	0.304	0.384			
	3	0.132	0.208	0.287	0.351			
	Av:	**0.128**	**0.203**	**0.294**	**0.364**			
CPT	1	0.005	0.007	0.011	0.012	0.020	0.029	0.033
	2	0.004	0.009	0.011	0.013	0.022	0.026	0.032
	3	0.004	0.007	0.009	0.012	0.020	0.027	0.030
	Av:	**0.004**	**0.008**	**0.010**	**0.012**	**0.021**	**0.027**	**0.032**
DNM1	1	0.004	0.011	0.017	0.024			
	2	0.006	0.010	0.019	0.026			
	3	0.004	0.009	0.017	0.024			
	Av:	**0.005**	**0.010**	**0.018**	**0.025**			
DNM2	1	0.008	0.011	0.022	0.027			
	2	0.008	0.010	0.020	0.030			
	3	0.006	0.012	0.020	0.028			
	Av:	**0.007**	**0.011**	**0.021**	**0.028**			
NG27 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.006	0.008	0.014	0.016			
	2	0.005	0.010	0.014	0.017			
	3	0.005	0.008	0.012	0.016			
	Av:	**0.005**	**0.009**	**0.013**	**0.016**			

Table A.2 continued

CPT	1	0.001	0.001	0.001	0.002	0.003	0.005	0.005
	2	0.000	0.001	0.001	0.003	0.004	0.004	0.004
	3	0.000	0.000	0.001	0.002	0.003	0.004	0.004
	Av:	**0.000**	**0.001**	**0.001**	**0.002**	**0.003**	**0.004**	**0.004**
DNM1	1	0.001	0.002	0.003	0.003			
	2	0.001	0.003	0.002	0.004			
	3	0.001	0.002	0.002	0.004			
	Av:	**0.001**	**0.002**	**0.002**	**0.004**			
DNM2	1	0.000	0.001	0.002	0.002			
	2	0.001	0.001	0.002	0.003			
	3	0.000	0.002	0.001	0.002			
	Av:	**0.000**	**0.001**	**0.002**	**0.002**			
NG28 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.272	0.479	0.707	0.782			
	2	0.303	0.521	0.760	0.836			
	3	0.291	0.503	0.755	0.825			
	Av:	**0.289**	**0.501**	**0.741**	**0.814**			
CPT	1	0.014	0.027	0.036	0.033	0.080	0.105	0.124
	2	0.014	0.031	0.035	0.042	0.094	0.119	0.138
	3	0.012	0.027	0.032	0.040	0.090	0.115	0.134
	Av:	**0.013**	**0.028**	**0.034**	**0.038**	**0.088**	**0.113**	**0.132**
DNM1	1	0.030	0.052	0.101	0.121			
	2	0.034	0.055	0.109	0.134			
	3	0.030	0.056	0.103	0.130			
	Av:	**0.031**	**0.054**	**0.104**	**0.128**			
DNM2	1	0.030	0.057	0.118	0.146			
	2	0.026	0.054	0.110	0.133			
	3	0.026	0.058	0.112	0.142			
	Av:	**0.027**	**0.056**	**0.113**	**0.140**			

Table A.2 continued

NG29 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.007	0.013	0.017	0.021			
	2	0.009	0.014	0.020	0.024			
	3	0.008	0.015	0.019	0.020			
	Av:	**0.008**	**0.014**	**0.019**	**0.022**			
CPT	1	0.002	0.002	0.003	0.004	0.007	0.008	0.009
	2	0.003	0.002	0.005	0.005	0.008	0.010	0.010
	3	0.002	0.003	0.004	0.006	0.008	0.009	0.011
	Av:	**0.002**	**0.002**	**0.004**	**0.005**	**0.008**	**0.009**	**0.010**
DNM1	1	0.004	0.009	0.010	0.014			
	2	0.005	0.009	0.012	0.016			
	3	0.004	0.010	0.011	0.015			
	Av:	**0.004**	**0.009**	**0.011**	**0.015**			
DNM2	1	0.004	0.007	0.010	0.012			
	2	0.003	0.007	0.009	0.011			
	3	0.003	0.008	0.009	0.012			
	Av:	**0.003**	**0.007**	**0.009**	**0.012**			
NG30 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.076	0.097	0.131	0.149			
	2	0.084	0.107	0.144	0.167			
	3	0.078	0.103	0.138	0.155			
	Av:	**0.079**	**0.102**	**0.138**	**0.157**			
CPT	1	0.004	0.008	0.008	0.010	0.019	0.026	0.031
	2	0.005	0.008	0.010	0.011	0.020	0.029	0.034
	3	0.004	0.009	0.009	0.012	0.020	0.028	0.036
	Av:	**0.004**	**0.008**	**0.009**	**0.011**	**0.020**	**0.028**	**0.034**
DNM1	1	0.008	0.014	0.024	0.036			
	2	0.009	0.014	0.026	0.038			
	3	0.008	0.015	0.025	0.037			
	Av:	**0.008**	**0.014**	**0.025**	**0.037**			

Table A.2 continued

DNM2	1	0.006	0.010	0.020	0.033			
	2	0.005	0.010	0.019	0.031			
	3	0.005	0.011	0.019	0.030			
	Av:	**0.005**	**0.010**	**0.019**	**0.031**			
NG31 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.034	0.185	0.390	0.695			
	2	0.042	0.201	0.418	0.738			
	3	0.036	0.194	0.396	0.706			
	Av:	**0.037**	**0.193**	**0.401**	**0.713**			
CPT	1	0.007	0.015	0.020	0.025	0.033	0.043	0.050
	2	0.008	0.015	0.022	0.026	0.034	0.046	0.053
	3	0.007	0.016	0.021	0.027	0.034	0.045	0.055
	Av:	**0.007**	**0.015**	**0.021**	**0.026**	**0.034**	**0.045**	**0.053**
DNM1	1	0.013	0.027	0.047	0.060			
	2	0.014	0.027	0.049	0.062			
	3	0.013	0.028	0.048	0.061			
	Av:	**0.013**	**0.027**	**0.048**	**0.061**			
DNM2	1	0.011	0.021	0.044	0.060			
	2	0.010	0.021	0.043	0.058			
	3	0.010	0.022	0.043	0.057			
	Av:	**0.010**	**0.021**	**0.043**	**0.058**			
NG32 aggregate	Exp (%)	**7 d**	**14 d**	**21 d**	**28 d**	**10 w**	**15 w**	**20 w**
AMBT	1	0.290	0.397	0.487	0.538			
	2	0.304	0.413	0.515	0.581			
	3	0.296	0.406	0.493	0.549			
	Av:	**0.297**	**0.405**	**0.498**	**0.556**			
CPT	1	0.017	0.025	0.031	0.036	0.046	0.051	0.055
	2	0.018	0.025	0.033	0.037	0.047	0.054	0.058
	3	0.017	0.026	0.032	0.038	0.047	0.053	0.060
	Av:	**0.017**	**0.025**	**0.032**	**0.037**	**0.047**	**0.053**	**0.058**

Table A.2 continued

DNM1	1	0.021	0.030	0.052	0.063			
	2	0.022	0.030	0.054	0.065			
	3	0.021	0.031	0.053	0.064			
	Av:	**0.021**	**0.030**	**0.053**	**0.064**			
DNM2	1	0.017	0.027	0.052	0.066			
	2	0.016	0.027	0.051	0.064			
	3	0.016	0.028	0.051	0.063			
	Av:	**0.016**	**0.027**	**0.051**	**0.064**			

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Turkish Standards Institute	Certification/ Laboratory	Civil Engineer, MSc	2010-present

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