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Rapid test method for determining alkali-silica reactivity expansion in concrete and aggregate

Douglas R. Pac
University of New Hampshire, Durham

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RAPID TEST METHOD FOR DETERMINING ALKALI-SILICA
REACTIVITY EXPANSION IN CONCRETE AND AGGREGATE

BY

DOUGLAS R. PAC
B.S., Keene State College, May 2004

THESIS

Submitted to the University of New Hampshire
In Partial Fulfillment of
The Requirements for the Degree of

Master of Science
In
Civil Engineering

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Thesis Director, David L. Gress, Professor

Jo Sias Daniel, Associate Professor

Charles H. Goodspeed, Associate Professor

4/25/08

Date
DEDICATION

To my parents, whose love and devoted guidance
has made me much of who I have become
ACKNOWLEDGEMENTS

First and foremost, I’d like to acknowledge Dr. David Gress for the educational opportunities he has created for me which will become the foundation of my career to follow. He has relayed to me much of his knowledge that will serve me in all aspects of my life. I’d also like to extend my gratification to The Innovative Pavement Research Foundation for funding my research and Texas A & M for their support and cooperation throughout the study. Finally, I’d like to give thanks to Dr. Jo Sias Daniel and Dr. Charles Goodspeed for participating on my thesis committee.
# TABLE OF CONTENTS

DEDICATION ........................................................................................................................................ iii

ACKNOWLEDGEMENTS .................................................................................................................. iv

TABLE OF CONTENTS ................................................................................................................... v

LIST OF FIGURES ........................................................................................................................ vii

ABSTRACT ........................................................................................................................................... x

<table>
<thead>
<tr>
<th>CHAPTER</th>
<th>PAGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>Alkali-Silica Reaction</td>
<td>1</td>
</tr>
<tr>
<td>Current ASR Test Methods</td>
<td>4</td>
</tr>
<tr>
<td>Research Objectives</td>
<td>6</td>
</tr>
<tr>
<td>2. NEW TEST METHOD</td>
<td>8</td>
</tr>
<tr>
<td>The Dilatometer</td>
<td>8</td>
</tr>
<tr>
<td>Original Testing Procedure</td>
<td>10</td>
</tr>
<tr>
<td>Aggregate Reactivity</td>
<td>11</td>
</tr>
<tr>
<td>Advantages</td>
<td>15</td>
</tr>
<tr>
<td>3. MODIFICATIONS</td>
<td>16</td>
</tr>
<tr>
<td>Float</td>
<td>16</td>
</tr>
<tr>
<td>Oven</td>
<td>22</td>
</tr>
<tr>
<td>Automated Vibration</td>
<td>25</td>
</tr>
<tr>
<td>FIGURE</td>
<td>DESCRIPTION</td>
</tr>
<tr>
<td>--------</td>
<td>-------------</td>
</tr>
<tr>
<td>Figure 1</td>
<td>ASR Map Cracking</td>
</tr>
<tr>
<td>Figure 2</td>
<td>Length Change Measurement of an ASTM 1260 Mortar Bar</td>
</tr>
<tr>
<td>Figure 3</td>
<td>Length Change Measurement of an ASTM 1293 Concrete Prism</td>
</tr>
<tr>
<td>Figure 4</td>
<td>Dilatometers</td>
</tr>
<tr>
<td>Figure 5</td>
<td>Dilatometer Schematic</td>
</tr>
<tr>
<td>Figure 6</td>
<td>Vacuum Saturation Setup</td>
</tr>
<tr>
<td>Figure 7</td>
<td>Percent Expansion versus Time</td>
</tr>
<tr>
<td>Figure 8</td>
<td>Natural Log of Rate versus Inverse of Temperature</td>
</tr>
<tr>
<td>Figure 9</td>
<td>Original Float</td>
</tr>
<tr>
<td>Figure 10</td>
<td>Redesigned Float</td>
</tr>
<tr>
<td>Figure 11</td>
<td>Displacement versus Time with Step-Like Curve</td>
</tr>
<tr>
<td>Figure 12</td>
<td>Water Baths</td>
</tr>
<tr>
<td>Figure 13</td>
<td>Setup for the Collection of Condensate Water on Float</td>
</tr>
<tr>
<td>Figure 14</td>
<td>Insulated Tower</td>
</tr>
<tr>
<td>Figure 15</td>
<td>Constant Temperature Oven</td>
</tr>
<tr>
<td>Figure 16</td>
<td>Automated Vibration</td>
</tr>
<tr>
<td>Figure 17</td>
<td>Original Tower</td>
</tr>
<tr>
<td>Figure 18</td>
<td>Displacement versus Time – Leaking Joint</td>
</tr>
<tr>
<td>Figure 19</td>
<td>Redesigned Tower</td>
</tr>
<tr>
<td>Figure 20</td>
<td>Redesigned Tower – Technical Drawing</td>
</tr>
</tbody>
</table>
Figure 21 Solenoid Valve ................................................................. 34
Figure 22 Displacement versus Time – Water Only – 80°C – Negative Slope .......... 41
Figure 23 Displacement versus Time – Water Only - 80°C – Flat-Line ...................... 42
Figure 24 Temperature versus Time – Water Only - 80°C ..................................... 43
Figure 25 Displacement versus Time – Platte River - Water - 60°C and 80°C ............... 50
Figure 26 Displacement versus Time – Platte River – 1 N NaOH ............................. 51
Figure 27 Percent Expansion versus Time – Platte River – 1 N NaOH ....................... 52
Figure 28 Percent Expansion versus Time – Platte River
1 N NaOH – Running Average ........................................................................ 53
Figure 29 Percent Expansion versus Time – Platte River
1 N NaOH – Fitted Linearly ........................................................................... 54
Figure 30 Natural Log of Rate versus Inverse of Temperature
Platte River – 1 N NaOH ............................................................................. 55
Figure 31 Percent Expansion versus Time for ASTM C 1260
(Platte River gravel with Dragon Cement) ...................................................... 56
Figure 32 Percent Expansion versus Time – Platte River
1 N NaOH – 80 C – Early Expansion Data ..................................................... 57
Figure 33 Percent Expansion versus Time – Platte River
1 N NaOH + Supersaturated CaOH – fitted .................................................. 58
Figure 34 Percent Expansion versus Time – Platte River
.5 N NaOH – Running Average ..................................................................... 59
Figure 35 Percent Expansion versus Time – Platte River
.5 N NaOH + Supersaturated CaOH – Running Average ................................. 60
Figure 36 Displacement versus Time – Spratt Limestone
Water & 1 N NaOH - 80°C ........................................................................ 63
Figure 37 Displacement versus Time – Spratt Limestone
Water & 1 N NaOH - 70°C ........................................................................ 64
Figure 38 Displacement versus Time – Spratt Limestone
Water & 1 N NaOH - 60°C ........................................................................ 65
Figure 39  Percent Expansion versus Time – Mortar – 1 N NaOH – 80°C .......................... 70

Figure 40  Percent Expansion versus Time – Mortar
80°C – Dilatometer/1260 Comparison ................................................................. 71
ABSTRACT

RAPID TEST METHOD FOR DETERMINING ALKALI-SILICA REACTIVITY EXPANSION IN CONCRETE AND AGGREGATE

BY

Douglas R. Pac

Thesis Director: David. L. Gress

University of New Hampshire September 2008

The undesirable expansion of concrete due to a reaction between alkalis in the cement and reactive siliceous aggregates, known as alkali-silica reactivity (ASR), continues to be a world-wide problem. Current test methods for predicting ASR are either inaccurate because they are unrepresentative of field conditions or require long testing periods that become impractical for use in the field. Because of the rising interest to minimize distress caused by ASR, alternative methods must be considered.

A key factor in the prediction of ASR in concrete over time is the reactivity of the aggregate. In the present study, an attempt was made to develop a method for determining volumetric expansion of aggregate as a result of ASR. Since ASR is a thermally activated process, expansion rates at different temperatures can be used to determine the material's activation energy. This new method can categorize aggregates based on their reactivity over a short period of time and provides a unique parameter to evaluate ASR.¹
Results have indicated that the new method is effective in rapidly determining the volumetric expansion and activation energy in siliceous aggregates in sodium hydroxide. However, thus far the method is incapable of producing accurate results for non-siliceous aggregates, mortar, and concrete. In order to get accurate results for these materials a modified version of the testing method must be developed.
CHAPTER 1

INTRODUCTION

Alkali-Silica Reaction

Alkali-silica reaction (ASR) was first discovered by Tom Stanton in the 1940's when unknown cracking of concrete buildings, bridges, and roadways were being investigated in California. These failures were a result of cracking throughout the entire structure and seen at the surface was map cracking with gel exuding from the cracks. When investigated it was found that the failures were a result of internal expansion caused by a chemical reaction between the alkalis in the cement and a reactive form of silica in the aggregate. ASR has since been observed in many other countries and has become one of the leading causes for concrete deterioration.

The expansion that occurs from ASR is a result of the following mechanism: Gel is formed due to a reaction between the hydroxyl ions in the alkaline cement pore solution and the reactive silica in the aggregates (i.e. chert, opal, and quartzite). Basically, the high pH (13.2 +/-), from the presence of Na\(^+\), K\(^+\), Ca\(^{2+}\), and OH\(^-\) in the pore solution, decomposes the reactive silica to produce a gel. When the gel begins to absorb available water through osmosis it expands resulting in an expansive pressure. If the expansive pressure exceeds the tensile strength of the concrete, cracks will begin to form and eventually radiate from the aggregate particles into the surrounding cement mortar.
The formation of cracks allows more water to enter the system enabling further ASR growth. If the concrete is unrestrained (no reinforcement) typical “map cracking” will occur as presented in figure 1.4

The conditions required to initiate ASR expansion are sufficient alkali and reactive silica content, calcium, and a sufficient amount of moisture (≥ 85 percent relative humidity). Unless all of these conditions are present ASR expansion will not occur. Conditions that effect ASR expansion are temperature and aggregate particle size. ASR is a thermally activated process so the rate of reaction increases due to high reaction kinetics at higher temperatures. Expansions will occur at low temperatures but not as rapidly. ASR expansion will also increase when smaller aggregate particles are used due to an increase in surface area.

Figure 1: ASR Map Cracking4

It can take as little as months or up to decades for ASR cracks to manifest on the outside surface of a concrete structure depending on the reactivity of the aggregate, alkali loadings, and the surrounding environment. There have been no reported cases of
structural collapse as a result of ASR but it should be noted that ASR cracking will eventually lead to further deterioration by freeze/thaw mechanism, deicing salt intrusion (steel corrosion), or chloride and alkali intrusion in marine environments.5

The ideal solution to prevent ASR from occurring is to use a non-reactive aggregate. Most aggregates, however, contain silica (SiO₂), but fortunately siliceous aggregates are only reactive if they have a poor crystalline structure. The amorphous composition results in an aggregate with poor bonding characteristics that is easily broken down by the high pH pore fluid in the concrete. There are sources in the U.S. that contain non-reactive aggregates but they are typically in remote locations forcing contractors to use local materials with higher reactivity in order to save money. If a reactive aggregate must be used, the mix must be mitigated to prevent ASR. This can also be difficult unless an economical source of low alkali cement is available. In 1990 the clean air act was passed to reduce emission levels. As a result, alkalis are collected during cement production and reintroduced into the cement kiln. This caused a general increase in alkali content and made it more difficult to find low alkali cements.5 If low alkali cement cannot be obtained then the mix must be mitigated with a class F fly ash, ground granulated blast furnace slag (GGBFS), lithium nitrate, or silica fume. Partial replacement of the cement with mineral admixtures reduces the pH of the pore solution reducing the generation of ASR gel. The only other alternative in preventing deleterious expansion is to control the presence of moisture which can be difficult and sometimes impossible depending upon the environment of the construction.
Preventing concrete from developing ASR can be physically and economically difficult so it is important to identify a mixture’s potential reactivity so that it can be mitigated, if needed, in a cost effective manner.

**Current ASR Test Methods**

Currently, the most commonly used test to determine ASR expansion is the ASTM C 1260 – Potential alkali reactivity of aggregates (mortar bar method) because results can be determined within as little as 16 days.\(^6\) The 1260 is an accelerated test that predicts whether a particular concrete mixture is going to be reactive and cause detrimental issues when used in the field. Results are determined by measuring the percent linear expansion of a 1”x1”x10” mortar bar that is immersed in 80°C (176°F) 1 N Sodium Hydroxide (NaOH) as presented in figure 2. The high testing temperature along with the high level of alkalinity (Ph = 14) of the testing solution accelerates the creation of ASR gel in the mortar bar creating rapid expansion. If the mortar bar has a percent linear expansion that exceeds specification, which is typically .1 percent at 14 days, then the aggregate being tested must be further evaluated and mitigated if necessary before use.

The downside to the quick testing time of ASTM C 1260 is that the results are not representative of actual concrete field performance. The high test temperature and alkalinity level far exceed the conditions of field concrete. So, doubts remain as to whether the accelerated results from ASTM C 1260 are at all relatable to expansions under field conditions.
Figure 2: Length Change Measurement of an ASTM C 1260 Mortar Bar

An alternative test to predict ASR expansion is the ASTM C 1293 – Standard Test Method for Concrete Aggregates by Determination of Length Change of Concrete Due to Alkali-Silica Reaction. Recently, it has been suggested to be the most effective test in predicting the field performance of a particular concrete mixture. ASTM C 1293, like ASTM 1260, determines the percent linear expansion of a concrete sample but the testing environment, in this case, is much more representative of field conditions. The procedure requires that a 3 inch concrete prism be tested at 38°C (100°F) in 100 percent relative humidity for a year. If the sample is mitigated an additional year of testing is required. A length change measurement of a concrete prism tested according to ASTM C 1293 is presented in figure 3.
The obvious downside to the representative test results ASTM C 1293 provides is the impracticality of the testing period.

Figure 3: Length Change Measurement of an ASTM C 1293 Concrete Prism

Research Objectives

The ultimate goal of this research was to produce an alternative testing method for predicting concrete ASR performance over a short period of time that is representative of field conditions. This was done by combining laboratory measured material properties and exposure conditions (aggregate reactivity, temperature, alkalinity, humidity, and age of concrete) through a kinetics-based mathematical formulation.°

To accomplish the objectives, a mathematical model was developed and put into use to determine the effect of each parameter. The main focus of this particular study was to develop a method for determining one of the key parameters mentioned above;
aggregate reactivity. This was done by using dilatometry to measure volumetric ASR expansion of an aggregate sample at three test temperatures to predict the materials activation energy.
CHAPTER 2

NEW TEST METHOD

The Dilatometer

The Dilatometer was originally designed by the Texas Transportation Institute at Texas A&M University for determining the coefficient of thermal expansion (CoTE) of aggregates (figure 4). The apparatus was created because the low durability of concrete is typically attributed to the difference in thermal properties between the aggregate and the mortar. It was recognized by Texas A & M that the device could also be used for determining volumetric expansion of concrete and aggregate due to ASR. 

Figure 4: Dilatometers
A cross section of the original dilatometer can be seen in figure 5. The apparatus consists of a stainless steel pot where the aggregate sample is placed, a brass lid with a hollow stainless steel tower and a thermocouple port, a float, a linear variable differential transducer (LVDT), and a data acquisition system. The inside of the brass lid was designed at an angle to allow entrapped air to move easily along the surface during its removal process.

Figure 5: Dilatometer Schematic
Original Testing Procedure

The original testing procedure consists of the following steps\textsuperscript{10}:

1. The stainless steel container is filled 80 percent full of oven-dried aggregate.

2. The brass lid is secured tightly to ensure no leaking.

3. NaOH is added at room temperature until the fluid level enters the tower.

4. A vacuum (3 Pa) is applied for 45 minutes to remove entrapped air between aggregate particles. The container is also placed on a vibrating table to facilitate the removal of entrapped air. A BOC Edwards RV8 Rotary Vane Pump was used to apply the 3 Pa vacuum (figure 6).

5. The dilatometer is then brought up to the test temperature and the LVDT core is attached to the float and inserted in the tower.

6. The LVDT is secured and as ASR expansion initiates the fluid level changes and an electrical signal from the LVDT is generated. The signal is acquired and recorded by the data acquisition system (LabVIEW) as a displacement in inches. The LVDT was a Schaevitz Model HCA 1000, with a range in displacement of 2 inches.

7. Temperature inside the dilatometer was also recorded by the data acquisition system through the thermocouple port in the brass lid. A 1/8 inch, type T Omega thermocouple was used.

Note: The LabVIEW program used for this research can be seen in appendix B and appendix C.
Aggregate Reactivity

Because ASR is a thermally activated process, the Arrhenius function was used to determine the aggregates ASR activation energy. The activation energy is defined as the energy that must be overcome in order for a chemical reaction to occur.

To determine the aggregates ASR activation energy, tests are performed at three different temperatures, typically, 60°C, 70°C, and 80°C. A plot is constructed of percent volumetric expansion versus time as presented in figure 7. As temperature increases so
does the rate of ASR expansion because the reaction process in thermally activated. Expansion data for this method can be produced in as little as 3 days. Once the rate of expansion is determined at each temperature a plot is constructed of the natural log of the rate verses the inverse of temperature in Kelvin as presented in figure 8. The data are fitted linearly and the slope of this line is equivalent to the activation energy divided by the universal gas constant (8.314 J/mol·K).
Figure 8: Natural Log of Rate versus Inverse of Temperature
Advantages

The foremost benefit of the dilatometer is that it has the potential to categorize aggregates based on their reactivity over a short period of time, thus providing a unique parameter to evaluate ASR. This parameter in combination with concrete material properties and exposure conditions allows for a rapid assessment of ASR reactivity.

This method can also characterize an aggregate's susceptibility to produce ASR gel as a function of aggregate size and alkalinity because both coarse and fine aggregates can be tested in the device and the levels of alkalinity of the test solution can be adjusted. The established relationship between alkalinity and aggregate size will make it possible to adjust the activation energy based on exposure the aggregate will receive under field conditions.

The new method also continuously determines volumetric expansions of the material being tested opposed to the standard linear testing currently being used such as ASTM C 1260 and ASTM C 1293. Experience with these tests has clearly indicated that continuous expansion data rather than a single value of expansion would be the appropriate criteria to assess ASR potential. Furthermore, additional time required to manually collect data are eliminated by the automated data collection system and reduces the potential for human error.

The dilatometer also allows for aggregate and recycled concrete aggregate (RCA) to be tested alone. This allows individual materials to be tested for ASR potential without having to cast a concrete sample. This reduces testing time and categorizes the materials independent of the cement mixture that will be used.
CHAPTER 3

MODIFICATIONS

Although the new test method works conceptually, mechanical flaws initially hindered the device from producing usable ASR expansion data. Throughout this study many modifications have been made to eliminate error associated with the original dilatometer design and testing procedure.

Float

The original float design can be seen in figure 9. The float was constructed of High Density Polyethylene (HDPE) with one closed end. The float was machined to 1 inch in diameter and had a height of 2 1/8 inch with a 1/8 inch thick threaded lid to seal the unclosed end. Teflon tape was used to help seal the lid. A 1/8 inch threaded stainless steel rod was used to attach the core of the LVDT to the float's lid.

Testing with the original float instantly became problematic. Initial ASR testing gave flat-line results when expansion was expected. This occurred because the float would stick due to frictional force between the LVDT core and shell. Because the float was designed to have a low buoyant force it was unable to overcome the frictional force created when the core came in contact with the sides with the LVDT. Once stuck the float would remain immovable throughout the testing.
Additional problems began when the joint between the lid and the base of the float began to take in fluid even with the use of Teflon tape. The loss of fluid volume along with the decrease in buoyancy of the float counteracted any expansion occurring as a result of ASR. The loss typically exceeded ASR expansion and gave an accumulated result that showed negative expansion. A marine sealant was used to permanently seal this joint and a new issue arose.

The harsh testing environment (1 N NaOH at 60°-80° Celsius) and the pressure buildup from a sealed system undergoing a significant change in temperature caused the HDPE float to creep into a barrel shape which, again, created issues in accurately determining a sample’s expansion over time. The only practical solution was to create a
float that had sufficient buoyancy, was leak proof, and volumetrically stable under the testing conditions.

After performing basic buoyancy calculations it was decided that thin-walled (.02 inches) stainless steel tubing with a 1 inch outside diameter would be used for the new design. Disks with the same thickness and diameter as the tubing were used to seal both ends (figure 10). The float's length was increased from the original 2 ¼ inches to 5 inches due to the higher density of the steel. The float was designed to have a 3/8 inch freeboard in 80 C NaOH making it more buoyant than the original float. A 3/8 inch connection thread for 4-40 rod was welded on top of the float to minimize poor alignment of the threaded rod reducing frictional forces between the LVDT core and shell.

Figure 10: Redesign Float
Many approaches were taken to permanently seal the stainless steel float. Attempts were made at silver soldering the float but a seal could not be achieved because the pressures that were created from the extreme temperature destroyed the final weld before it could cool. Marine sealants wouldn't work because the NaOH test solution destroyed the material's bond at the high testing temperature. Eventually, it was determined that the only method that worked was TIG (Tungsten Inert Gas) welding the stainless disks to the tubing. Even this method only had a 50 percent success rate. Much time was spent welding the floats and then testing each float to see if it was leak proof. In order to determine if a float was sealed it was placed in a desiccator and submerged under water with the use of a weight. When negative pressure was applied to the desiccator bubbles would appear wherever there had been a leak as air was pulled from within the float. At this point the floats were marked and then re-welded. This cycle continued until there was a sealed float for each dilatometer. Throughout the ASR testing the floats were weighed before and after every test to ensure that they did not take in any testing solution.

The redesigned float was successful in eliminating the defects found in the original float except that the float continued to occasionally stick during testing. In order to increase the float's buoyancy its length was increased and due to the tall, cylindrical, nature of the float it had the tendency to want to turn over and float on its side. Because it was restricted to do so, a greater lateral force was produced than with the previous float. Although its buoyancy was increased so was the friction between the LVDT core and shell. Poor welding only made the sticking matters worse. TIG welding the thin material was a very difficult and precise operation and as a result there typically was an
uneven distribution of weld around the circumference of the float. The off centered weight distribution created further float alignment issues. Grinding the excess weld was an option but it was very difficult to do so without destroying the seal.

The importance of having a float that was volumetrically stable and leak proof outweighed the float's ability to create smooth displacement curves. A temporary solution for the “sticking” problem was simply to tap the side of the LVDT which would cause enough disturbances to free the float. Step-like curves were produced because the float would remain stuck over periods of time and then become freed due to tapping (figure 11). Although the solution was not ideal it was temporarily satisfactory.
Figure 11: Displacement versus Time with Step-Like Curve
Oven

Initial procedures tested the dilatometer in a constant temperature water bath. The test was performed at 60° C, 70° C, and 80° C and a picture of the initial setup is presented in figure 12.

![Water Baths](image)

**Figure 12: Water Baths**

It became evident, through testing, that condensation water was collecting on the inner walls of the dilatometer tower. This was caused by the extreme temperature differential (maximum of ~60°C) between the base of the dilatometer in the 80° C water bath and the top of the LVDT at room temperature.

Condensation became a problem when tapping was performed to free the float. The following describes the mechanism involved: 1) Water evaporates and a vapor is created within the dilatometer tower at the high test temperature resulting in an initial decrease in fluid level. 2) Water vapor condenses on tower walls from extreme temperature differential. 3) System eventually comes to equilibrium and fluid level
remains unchanged. 4) LVDT is tapped to free float and causes condensate water to flow back into its initial source resulting in an increase in fluid level. 5) Simultaneously, condensate water is deposited onto the float causing a loss in buoyancy and a negative float displacement. 6) When the amount of water on top of the float reaches its capacity, surface tension is overwhelmed and fluid flows over the sides of the float increasing float displacement. The collection of condensate water on top of the float was observed and measured using the set up in figure 13.

The result was that there were three unknown displacements occurring when condensation is present; increase from ASR, decrease from loss of float buoyancy, and increase from condensation entering the fluid level. Not being able to quantify one volume change from the other made it impossible to determine ASR expansion.

Figure 13: Setup for the Collection of Condensate Water on Float

Many attempts were made at insulating the dilatometer tower to eliminate the severe temperature differential and end condensation but insulating only got the LVDT
and tower within 5°C of the testing temperature (figure 14). This was a large enough
difference in temperature to allow the condensation issue to remain. The only practical
solution was to discontinue the use of the water bath and move the entire device into a
constant temperature oven as presented in figure 15.

Figure 14: Insulated Tower

This transition proved to be effective in eliminating condensation but manual
tapping could no longer be performed to free the float because opening the oven door
created a significant drop in temperature which was instantly observed as a loss in
volume from thermal contraction of the fluid, aggregate, and metal container. This fluid
level drop gave a false displacement reading when the float was freed. The float would
typically remain stuck after the oven door was closed and temperature stabilized and the true fluid level could not be observed.

![Figure 15: Constant Temperature Oven](image)

**Automated Vibration**

An automated vibration system was installed to resolve the floats "sticking" dilemma without ever having to open the oven door. A D to A board was added to the National Instrument data acquisition system and LabVIEW was programmed to signal a switch to turn on a 12 volt vibrator for five seconds in between every minute of data collection. The vibrator was secured to the LVDT with zip ties and when activated caused the float to spin and free up (figure 16). With the new vibration system the float
could effectively be freed in between every recorded data point giving a minute by minute update on the float’s position. Graphs of the float’s position went from step-like functions to smooth curves making analysis less complicated.

A 12 VDC ceramic permanent magnet motor was acquired to construct the vibrator. The motor was 1.25 inches in diameter and 1.875 inches in length and the motors shaft protruded a half of an inch from the top of the vibrator and rotated at 3000 RMP. A metal disk that was a half inch in diameter and a quarter inch in thickness was drilled and pressed at a 3/16” offset to the motors shaft. The slight offset and the high revolution speed of the motor created a vibration effect that was suitable for freeing the float.

A check was made to ensure that the motors magnet did not disrupt the LVDT’s displacement readings. This was done by using a micrometer to accurately control the position of the LVDT rod as data were acquired. The vibrator was applied to the LVDT and activated. It was found that displacement was only affected when the vibrator’s position on the vertical axis if the LVDT changed during data collection. The vibrator was secured to the LVDT with zip ties to prevent any vertical movement throughout testing.
The original tower was designed with three portholes, which can be seen on the left side of the tower in figure 17. They were installed so that the fluid level could be controlled at the beginning of a test. The threaded plugs could simply be removed and then reinserted after the fluid reached the test temperature. This would ensure that the floats initial position was sufficient for the material being tested.
The joint that can be seen above the portholes was not in the original design. It was created so that the float could be inserted after vacuum saturation was performed. The original procedure required the float to be inserted through the bottom of the tower and then held in place as the brass lid was fastened to the base. Fluid was then pored in through the top of the tower and vacuum saturation was performed with the float in the tower. The testing procedure became more practical after the new joint was created and testing became more efficient and repeatable.

Although the new joint facilitated testing an unexpected issue arose. A loss of fluid level was observed during testing due to water vapor escaping through the created
joint as well as through the portholes and the joint where the LVDT was fastened to the
tower with set screws. This fluid loss, once again, counteracted ASR expansion and
made it difficult to get accurate results. The loss from evaporation was greater than the
expansion from ASR resulting in an overall decrease in displacement (figure 18).

To remediate the issue a nonpermanent sealant (Plumber’s Goop ®) was used
before every test to seal the leaking joints but careful attention had to be given to sealing
the final joint. Unlike the rest of the joints this one had to be sealed after the dilatometer
came up to testing temperature. This was to ensure that there would be no complications
with pressure build up as the sealed system was heated to its testing temperature. For
example, if the joint was sealed at room temperature, immediately before a test began at a
higher temperature, the build up in pressure would destroy the still curing sealant. This
would allow the escape of water vapor to persist complicating accurate data collection.
And if the final joint was given time to set up before bringing the dilatometer to testing
temperature the system would be able to withhold the pressure and further issues would
arise. For instance, the dissolution of the materials being tested (i.e. silica, calcium, etc.)
is a function of pressure as well as temperature and pH. Typically, as pressure increases
so does the rate of dissolution. Dissolution becomes problematic for the testing at hand
because of the related volume changes as will be discussed in more detail in chapter 4.
Additional pressure related issues exist if air remains in the system because the solubility
of air, like the solids being tested, is affected by pressure. As air goes in and out of the
solution from pressure changes, volume changes occur and make the collection of ASR
expansion data an impossibility. The new procedure of sealing the final joint at the test
temperature became necessary due to the importance of having a sealed system
unaccompanied by pressure. The new procedure worked effectively but it became very
time consuming between the time spent waiting to perform the final seal and the time
required to strip the dilatometer of old Goop and reseal before every test.

To permanently solve the leaking issue a new tower was designed that eliminated
the unnecessary joints that existed in the original design and all required joints where
sealed with either an o-ring or permanent marine polyurethane sealant (3M - 5200). This
eliminated the use of Goop and made the testing more efficient and consistent. Through
testing, the new tower was found to be airtight and was consistently effective in
eliminating fluid loss as a result of water vapor escaping the system. Proof on
determining if the system was airtight will be discussed in chapter 4. A picture of the
redesigned tower can be seen in figure 19 and a technical drawing can be seen in figure
20.
Figure 18: Displacement versus Time - Leaking Joint
Figure 19: Redesigned Tower
Figure 20: Redesigned Tower – Technical Drawing
Solenoid

With the use of the new tower the system was found to be airtight. For this reason, the tower was additionally machined to accommodate a 12 volt solenoid valve above the fluid level for the purpose of releasing unwanted pressure buildup as was previously discussed (figure 21). The LabVIEW program was modified so that the user could activate a switch that opened and closed the valve without having to open the oven door. If a manual valve had been used a pressure free system could never be achieved due to the temperature drop that immediately occurs when the oven door is opened.

Figure 21: Solenoid Valve
The testing procedure established after the addition of the solenoid was to close the valve when the dilatometer came up to the testing temperature. This procedure included that the system stabilized overnight before the valve was closed. Having a pressure free system that was also sealed eliminated error and reduced variation in data.
CHAPTER 4

RESULTS

Introduction

A plan was proposed to prove that the new testing procedure and redesigned apparatus were capable of accurately recording ASR expansion with minimal error. The first step was to prove that the system was airtight to ensure that there was no counteracting negative expansion as a result of fluid loss through evaporation or leaking. This was done by performing a test with only water in the dilatometer. A flat line in fluid level displacement after temperature equilibrium suggests no loss of fluid, thus validating that all joints were perfectly sealed.

Once a flat line with water only was achieved the effectiveness of the vacuum saturation technique could be determined. This was performed by testing with water and aggregate. If, after vacuuming, a flat line could again be achieved then the vacuum saturation method would be deemed acceptable. However, if a negative slope was produced it could be concluded that the method of vacuuming was not sufficient in saturating the aggregate and new method or a calibration curve would have to be used.
Finally, testing with NaOH could be performed to produce expansion data and aggregate activation energies. This would be done using various aggregate types, solution normalities, aggregate sizes, and temperatures.

**Water-Only Testing**

Many of the modifications that were performed on the original dilatometer were a result of producing negative slopes (fluid loss) when running water-only tests as presented in figure 22. The loss in fluid level displacement for this particular test was only .01 inches over 50 hours which is equivalent to a volumetric loss of .24 mL. Although this seems to be a particularly small volume loss, when compared to gel created over a 50 hour time period for a typical reactive aggregate it becomes significant and the counteracting effect complicates an accurate recording of expansion.

To put this in perspective, an aggregate that produces an expansion of .1 percent over 14 days under ASTM C 1260 testing conditions (minimum expansion that is considered reactive) would only create .05 mL of gel. This is approximately one fifth of the volume that was lost in the water-only test in figure 22. Not to mention, the gel that is created during the ASTM C 1260 test occurs over 14 days while the fluid loss in the water test is over 50 hours. So, it can be seen that it is essential for fluid loss to be minimized and ideally eliminated.

In order for fluid loss to be eliminated the source of the leaks had to be pinpointed. This was done by performing pressure tests similar to those described when testing for leaks in the stainless floats. After fluid loss was observed in a water-only test the dilatometer would be submerged in a bucket of water and positive air pressure would
be applied to the system until air bubbles were seen in the water bucket escaping from the dilatometer. The leaking joint/joints were then modified until they were sealed as previously discussed. As these alterations were made negative slopes decreased until a flat line was achieved (figure 23). The data fluctuation which looks like noise is a result of the automated vibration causing the float to reorient with each vibration. Electronic noise within the LVDT's would be smaller in magnitude than the fluctuation seen in figure 23. The magnitude of the fluctuation is a function of the applied voltage and it should be noted that the actual curve of fluid level displacement would lie along the center point of the fluctuation span. The slight and gradual change in fluid level displacement over the 60 hour period is related to a change in temperature inside the dilatometer (figure 24). The oven is capable of keeping the temperature of the dilatometer within a tenth of a degree Celsius but this is a large enough temperature change (given the volume of the fluid being tested and the high coefficient of thermal expansion of water) to create the change in fluid level displacement as seen in figure 23. The small overall decrease in fluid level should not be confused with fluid escaping the system but should be attributed to the change in temperature over the testing period. Thermal expansion calculations were performed to verify that the minor loss in displacement is entirely related to the change in oven temperature over the testing period (appendix A). With temperature change taken into consideration there was no loss in fluid for this test proving that the system was perfectly sealed.

In order to ensure that the system could be sealed consistently, water-only tests were run on multiple dilatometers and it was found that repeatable displacement data could be produce that represented a minimal amount of error.
Recording the weight of the dilatometer before and after the test supplied additional proof that the system wasn’t leaking. When the system was leaking, the recorded volumetric water loss was equivalent to the weight loss suggesting that the loss in volume was a direct result of fluid loss and that there was no additional phenomenon occurring. When flat lines in displacement were recorded, after the modifications had been made the weight also remained the same. Thus, there were two forms of evidence proving that the system had been sealed.

Sealing the dilatometer to be within an acceptable percentage of error was a difficult task given the original design and the small ultimate ASR expansion that would be recorded in a typical test. The quantity of ASR gel that could be generated over the short testing period for a reactive siliceous aggregate could be as small as .004 cubic inches at the 60°C testing temperature. This will be discussed in the section on aggregate testing. A fluid loss of .0002 cubic inches would result in a five percent error of the total ASR expansion at the low test temperature. That is equivalent to a less than a single droplet of water. At higher temperatures greater quantities of gel are created allowing for greater fluid loss with the same percentage of error. Based on the expansion data for a reactive aggregate at 80°C there could be a fluid loss of .005 cubic inches which is equivalent to 6 drops of fluid loss and still be within 5 percent error. It can be seen from the displacement curve for the water-only test and the thermal expansion calculation that the dilatometer was effectively sealed with 0 percent error.

Sealing the system was an extremely important step because expansion data prior to having a perfect seal would contain unknown rates of fluid loss making analysis impossible. For future work, the dilatometer should be redesigned to ensure an effective
seal in a more practical manner given the importance of the issue. This should be done by permanently sealing unnecessary joints by welding opposed to using a marine sealant which will eventually lose its bond in the harsh testing environment. Additionally, better sealing mechanisms will be required where joints are needed. For instance, the use of a larger o-ring where the brass lid meets the stainless steel base of the dilatometer would make the sealing that particular joint more effective. Additionally, latches may be an alternative to the existing threads. This would eliminate the possibility of the o-ring bunching up and would apply an evenly distributed pressure along the o-ring. The tests should also be performed at higher temperatures (70°C, 75°C, and 80°C) to create larger ASR expansion and minimize the percentage of error if any exists.
Figure 22: Displacement versus time at 80°C - Negligible Slope
Figure 23: Displacement versus Time - Water Only - 80°C - Flat-Line
Figure 24: Temperature versus Time – Water Only - 80°C

$\Delta T_{\text{over 60 hours}} = 0.032^\circ\text{C}$
Aggregate Testing

Platte River

The first aggregate tested is from the Platte River in Nebraska. The natural particle distribution of this aggregate ranges from dust to a 3/8 inch particle. The siliceous aggregate is mined from the Platte river glacial deposits. The aggregate is composed of gneiss, granite, and pegmatite which account for the reactivity of the gravel. Among the minor rocks present, some of the volcanic porphyries contain minerals that also react with alkali and form ASR gel.\textsuperscript{11}

Tests were conducted using the Platte River gravel following the procedure mentioned in chapter 2 except water was used instead of sodium hydroxide to prevent ASR expansion from occurring. Without a high alkaline solution (NaOH) a reaction can not be initiated and expansion would not occur. After the samples were immersed in water they received a negative pressure of 3 Pa for 45 minutes on a vibrating table. The concept is that any entrapped air should be removed from the intense vibration while under vacuum. If the technique was not effective and air remained in the system, this would create the possibility of air bubbles leaving the system during a test creating a measured loss in fluid displacement. This would offset ASR expansion and complicate the collection of accurate data.

The tests were run at 60°C and 80°C to ensure that entrapped air could be removed independent of temperature. After temperature came to equilibrium fluid displacement was recorded for 90 hours and a flat-line was achieved for both tests indicating that the technique was effective in removing air and saturating the material (figure 25).
Now that the dilatometer was proven to be sealed and the vacuum saturation technique was found to be effective, testing with NaOH could take place and activation energies could be determined.

Platte River was tested again in a 1 N NaOH solution at three temperatures; 60°C, 70°C, and 80°C and the results are presented in figure 26. These data represent the fluid level displacement after the system had come to temperature equilibrium and the solenoid valve had been closed. Thus, the displacement seen is directly a function of ASR gel generation over a four day period. Displacement from thermal expansion and evaporation loss (from the solenoid being left open to release pressure) was recorded overnight (~12 hours) but was not included when determining the aggregate’s activation energy. As expected, expansion increased as temperature increased. This suggests that the increase in temperature caused more ASR due to high reaction kinetics. Two tests were run at 80°C and it was verified by the overlapping data that repeatability was possible using different dilatometers.

The fluid level displacement was mathematically manipulated and plotted as a percent volumetric expansion of the initial volume of the aggregate (figure 27). To do this the change in fluid level was simply multiplied by the inside area of the tower to get the volume change. To determine the aggregate’s initial volume, a specific gravity test was performed on the Platte River aggregate ($G_s = 2.47$). Because the same weight distribution of aggregate particles is used for each test the specific gravity could be divided into the total weight to get the absolute volume. It can be seen in figure 27 that there was a total percent volume increase of approximately .005 percent, .04 percent, and .11 percent for 60°C, 70°C, and 80°C respectively over 90 hours. This graph was
reproduced to eliminate the fluctuation caused by the automated vibration by taking a running average of the expansion data. The data were plotted in figure 28 with the y-axis ranging from -.1 to .15 percent and the x-axis ranging from 0 to 100 hours. This scale and technique was used when analyzing other Platte River ASR expansion data for ease of comparison.

Determining the rate of expansion for this particular material was slightly complicated due to the nonlinearity of the curves. Because the rate was constantly changing over the testing period the curves were fitted linearly at three different time periods; 0-30 hours, 0-60 hours, and 0-90 hours (figure 29). The slopes were determined and a plot of the natural log of rate versus the inverse in temperature was constructed to determine the activation energy for each time period (figure 30). A linear curve was fit to the data ($R^2 > .9$) and it can be seen that the three curves have relatively parallel slopes independent of the testing period. The activation energies ranged from 156 kJ/mol – 167 kJ/mol. By taking the average it can be concluded that approximately 162 kJ/mol are required to initiate alkali silica reaction for the Platte River in a 1 N NaOH solution.

A standard ASTM C 1260 was run on the Platte River to determine if the material was considered reactive under the accelerated mortar bar criteria. This served as a good reference for correlating activation energies to reactivity. Figure 31 shows that the material expanded .295 percent of its initial length over a 14 day period which is well above the .1 percent criteria that classifies an aggregate as potentially reactive. Further aggregate testing beyond this research will have to be performed in order to make a more advanced correlation between activation energy and aggregate reactivity. These data
suggest that any aggregate with an activation energy under 162 kJ/mol could be considered reactive. This is a very conservative suggestion considering that the Platte River created expansion data that was almost three times the minimum criteria for reactivity in the ASTM C 1260. An aggregate with an ASTM C 1260 expansion of .1 percent at 14 days may be a good aggregate for determining the maximum energy that will be needed to initiate potentially deleterious ASR.

Because the testing procedure requires that the system stabilizes over night, approximately 15 hours of initial ASR expansion is lost. The procedure was designed in this manner so that the person running the test would not have to remain in the lab throughout the five hour duration it takes for the temperature to come to equilibrium. Instead, the tester could come into the lab the following morning, close the valve, and initiate the collection of ASR expansion data. The problem, however, is that approximately 10 hours of early expansion data is lost and it was predicted that much of the ASR expansion occurs in this early time period. To determine the amount of gel that is created during this time period, a test was performed where the valve was closed immediately after the temperature came to equilibrium. A test was run simultaneously using the standard procedure for comparison purposes. Platte River aggregate was tested in a 1 N NaOH solution and the results are presented in figure 32. It can be seen that the prediction was correct. Approximately one-third of the expansion occurs during the first 10 hours. The curve for the standard procedure was shifted up to make a comparison between the two easier. It can be seem that the curves are very similar aside from the 10 hours of lost data. Future work will require that the LabVIEW program is modified to automatically close the solenoid valve after temperature becomes constant so that the
most of the early expansion data are not lost. Some data (approximately the first 5 hours) will be lost as temperature changing.

Although the chemical reaction taken place with the Platte River in the dilatometer testing has been referred to as ASR its true reaction is slightly different because it lacks the presence of calcium. True ASR, as explained earlier, requires sufficient alkali, reactive silica, calcium, and a sufficient amount of moisture (≥ 85 percent R.H.) and unless all of the conditions are present ASR expansion will not occur. It is observed that the gel being generated, even without calcium, has expansive properties but it has no true field significance. In order to produce practical results the test was rerun with a 1 normal NaOH solution that was supersaturated with CaOH. The gel that was created in this case was the same as ASR gel that would be created in concrete. The true data were curve fitted and reproduced in figure 33 to eliminate the noise that is created from the automated vibration. Due to the abundant amount of calcium more gel was created at the higher temperatures over the same time period as in the previous test and gel continues to be generated after the other gel formation had ceased. The same procedure was used to determine the activation energy and it was found that the activation energy was decreased to 138 kJ/mol. This suggests that when calcium is present less energy is needed to create expansive gel.

It is anticipated that ASR activation energy is a function of alkalinity and that a relationship could be established that will enable mathematical modeling of ASR susceptibility under alkali levels that concrete is likely to be exposed to in field conditions. In order to establish a correlation, the previous two tests were reproduced at half the sodium hydroxide normality. Future research would require additional alkali
levels (.25 N and 2 N) to establish a more accurate correlation but under time constraints only one level of alkalinity was evaluated. The percent expansion results are presented in figures 34 and 35. It can be seen that halving the alkali level produced unusual results at the lower temperatures. ASR expansion has a "pessimum" effect that is dependent on several variables; alkali content, calcium content, temperature, etc. The contractions suggest that the threshold for producing ASR expansion had not been passed at the lower temperature and alkali content over the testing time. Furthermore, an alternative chemical reaction causing shrinkage had taken place. Future investigation must take place to identify the exact cause of the results just mentioned. Testing at higher temperatures and higher normalities may be a solution for establishing a correlation between activation energy and alkalinity but at this time a correlation could not be made.
Figure 25: Displacement versus Time - Platte River - Water - 60°C and 80°C
Figure 26: Displacement versus Time – Platte River – 1 N NaOH
Figure 27: Percent Expansion versus Time – Platte River – 1 N NaOH
Figure 28: Percent Expansion versus Time - Platte River - 1 N NaOH - Running Average
Figure 29: Percent Expansion versus Time – Platte River – 1 N NaOH – Fitted Linearly
Figure 30: Natural Log of Rate versus Inverse of Temperature – Platte River – 1 N NaOH
Figure 31: Percent Expansion versus Time for ASTM C 1260 (Platte River gravel with Dragon Cement)
Figure 32: Percent Expansion versus Time – Platte River – 1 N NaOH – 80 C – Early Expansion Data
Figure 33: Percent Expansion versus Time – Platte River – 1 N NaOH + Supersaturated CaOH - fitted
Figure 34: Percent Expansion versus Time – Platte River - .5 N NaOH – Running Average
Figure 35: Percent Expansion versus Time – Platte River - 5 N NaOH + Supersaturated CaOH – Running Average
Spratt Limestone

Spratt Limestone was the second material to be tested because of its established history of being alkali silica reactive in field structures and it has also served as a reference aggregate in many ASR studies. This aggregate is obtained from Spratt quarry in Ontario Province of Canada. It is a crushed limestone with a particle distribution ranging from #8 to a ¾ inch particle. The aggregate consists of calcite with minor amounts of dolomite with about 10 percent insoluble residue. The reactive component of the rock is reported to consist of 3 percent to 4 percent of microscopic chalcedony and black chert, which is finely dispersed in the matrix.¹²

A similar testing plan was proposed with the Spratt aggregate as was the Platte River. The material was first tested with water to determine if the vacuum saturation technique was adequate in saturating the new aggregate and unexpected results arose. Instead of a flat-line, there was an initial contraction in overall volume followed by volume expansion throughout the remainder of the test. This effect was staggered between the different test temperatures. There was a longer period of decline for the lower temperatures but in all cases the volume eventually expanded. When Spratt was tested in 1 N NaOH very similar curves were produce as when it was tested in water. Figures 36 - 38 show displacement versus time results for Spratt tested in water and NaOH tests for the three different test temperatures. The similar volume increases for water and NaOH could not be a result of ASR gel generation as the rates should be much higher in the high alkali (pH = 14) solution. The results suggested that there was some other phenomenon occurring.
Theoretically, the results indicate that the volume changes could be a result of the dissolution of calcium carbonate as explained by Holmes-Farley in the following statement.

When calcium and carbonate ions are dissolved in water, a number of the water molecules become tightly attached to the ions. Overall, the volume occupied by undissolved calcium carbonate and water is larger than the volume occupied by calcium and carbonate ions dissolved in this same amount of water. This volume change is primarily due to the increased "density" that can be attained by water molecules around ions compared to pure water.\(^3\)

This would explain the initial decrease in volume as seen in the curves. The following increase in volume may be a result of the formation of calcium bicarbonate but further investigation would have to take place to determine the validity of this assumption. The preliminary findings do however suggest that calcium carbonates cannot be tested using the dilatometer method unless an alternative testing method can be established.

62
Figure 36: Displacement versus Time — Spratt Limestone — Water & 1 N NaOH - 80°C
Figure 37 - Displacement versus Time - Spratt Limestone - Water & 1 N NaOH - 70°C
Figure 38 - Displacement versus Time - Spratt Limestone – 1 N NaOH - 60°C
Mortar Testing

Mortar bars were prepared as specified by the ASTM C 1260 method to be tested in the dilatometer. Platte River was used as the aggregate because it was expected that a siliceous aggregate would give better results than an aggregate composed of calcium carbonate (Spratt Limestone). This would eliminate the complex volume changes that were found when testing Spratt alone. The same grading was used as in a standard ASTM C 1260 which ranges from the #100 sieve to the #8 sieve. Cement acquired from Texas A & M was used for all mortar bars to eliminate variation in data. The ASTM C 1260 water to cement ratio and NaOH to mortar bar ratio were also used. The mortar bars were cast as specified by the ASTM C 1260 but the curing process was modified. The standard ASTM C 1260 curing procedure states that the mortar bars should cure for 1 day in 100 percent relative humidity at room temperature before the samples are removed from their molds and tested for ASR expansion. The modified curing process followed the standard except the mortar bars were given an additional 5 days of curing in saturated lime water (CaOH) before they were tested in the dilatometer. The extended curing time was given to the mortar bars to minimize the effect of hydration volume loss. In addition, the extended curing in lime water also would ensure that the mortar was fully saturated before initiating the test. This would eliminate the effect of testing solution entering air voids in the mortar and producing a contraction in volume.

Tests were performed on the mortar bars at 80°C in three separate solutions; 1 N NaOH, supersaturated CaOH, and water. The expansion data are presented in figure 39. It was expected that there would be a contraction in volume when the mortar bars were tested in water which could be explained by the volume contraction from the dissolution
of calcium as was also seen when Spratt was tested in water. It was also anticipated that the mortar volume change when tested in supersaturated CaOH would remain unchanged because there would be no available “space” for calcium to dissolve. The result however was a contraction in volume that was roughly two-thirds the contraction as seen in the water test. The difference between the curves would represent the contraction due to calcium dissolution. The results further suggest that there is at least one other variable causing contraction that would eventually have to be identified in order to get accurate results when testing mortar for ASR expansion. It is unlikely that the contraction in volume is related to saturation due to the 5 day presoak period. The volume change may be due to cement hydration and if proven to be true the mortar would have to be fully hydrated before testing for ASR could initiate.

There was essentially no change in volume for the mortar tested in 1 N NaOH and it should be noted that the spikes in the data can be ignored as they were a result of noise created in the electronics. The flat-line in this case does not indicate that ASR is not occurring. The generation of gel is occurring but graphically it is unapparent because other factors are offsetting the expansion (dissolution, hydration, etc.). The exact cause of the contractions must be fully identified so that they can be eliminated if mortar testing is to work in the dilatometer. For the time being, subtraction of the water curve from the NaOH curve would give a rough approximation of the volume of ASR gel being produced. It would not give an exact result because the factors causing contraction in the water test would be of different magnitudes in the NaOH test.

Run alongside the ASTM C 1260-like dilatometer testing was a standard ASTM C 1260 and a modified ASTM C 1260 that would be used for comparison. The modified
ASTM C 1260 mortar bars received the same curing as the dilatometer test and the percent linear expansion was recorded each day alongside the continuous data collection of the dilatometer test. This was done so that the percent linear expansion could be compared to the percent volumetric expansion of the dilatometer test. The linear result would simply be multiplied by three to produce a volumetric result that could be used for comparison. The results from this test would give the actual volumetric expansions from ASR unaffected by factors in the dilatometer test that caused contraction. The modified 1260 was run in water and NaOH just like the dilatometer testing.

Figure 40 shows a compilation of the various mortar testing. It can be seen that the modified 1260 in water (3) flat lined proving that the mortar bars in the dilatometer test are not contracting but instead the contraction is from an overall volume loss of the fluid/solid combination. It can also be seen that the modified 1260 in NaOH (2) expanded .1 percent less than the standard 1260 (1). This is due to the lower porosity of the modified samples as a result of the longer curing process they received. Because the modified samples were given 5 additional days to hydrate it became more difficult for the NaOH solution to penetrate the less porous matrix resulting in less ASR expansion. It should also be noted that the curve of the modified 1260 in NaOH (2) would give the best comparison to the dilatometer testing in NaOH (5) because they had identical preparation. If the factors causing contraction in the dilatometer test could be eliminated than the two curves would ideally overlap. The dilatometer calibrated curve (6) was produced by subtracting the dilatometer – NaOH curve (5) from the dilatometer – water curve (4). As mentioned earlier, this curve would give an approximation of ASR expansion in the dilatometer by calibrating for the factors causing contraction.
curve, however, would not be exact because the factors causing contraction in the water test would be of different magnitudes in the NaOH test. Because of this it can be seen that the dilatometer calibrated curve (6) is slightly larger in expansion than the 1260 modified curve (2). This may be because there would be less dissolution of calcium in NaOH than water.

Approximations for ASR mortar expansion in the dilatometer can be established at this point but the exact cause for volume contractions will have to be identified in order to get precise results.
Figure 39: Percent Expansion versus Time – Mortar – 1 N NaOH - 80°C
Figure 40: Percent Expansion versus Time – Mortar – 80°C – Dilatometer/1260 Comparisons
CONCLUSIONS & FUTURE WORK

These conclusions are based on the results of this thesis and may or may not apply to similar materials. The conclusions are as follows.

Conclusions

- The dilatometer functions mechanically after making the following modifications:
  - Float redesign.
  - Application of external vibration.
  - Oven testing.
  - Dilatometer tower redesign.
  - Solenoid application.
- The dilatometer can be sealed to within an acceptable margin of error.
- Volume remains unchanged when siliceous aggregate is tested in water.

Therefore, the vacuum saturation procedure is effective in removing entrapped air from test samples and the dissolution of silica does not affect the volume over a four day testing period.
- Activation energies can be determined for siliceous aggregates in a 1 N NaOH solution.
- Lowering the alkali level becomes problematic at lower test temperatures (volume contractions).
• Calcium carbonates produce volume contraction followed by undetermined volume expansions when tested in water. Therefore, calcium carbonates cannot be tested for ASR in the dilatometer until volume expansions and contractions can be explained and eliminated.

• Mortar produces volume contractions that are partially related to calcium dissolution and partially related to some other undetermined factor/factors (possibly hydration) when tested in water. Therefore, mortar can not accurately be tested for ASR in the dilatometer until all volume contractions can be explained and eliminated.

• Less energy is needed to produce expansive gel when calcium is present in a 1 N NaOH solution.

Future Work

• There should be further redesign made to the dilatometer to eliminate any unnecessary joints either by constructing a solid cast apparatus or by welding unneeded joints. Currently, some joints are sealed with a marine sealant which will eventually lose its bond due to the harsh testing environment. A permanent seal would ensure that statistically similar test results could be attained with the use of different Dilatometers and by different users. To further ensure this matter, necessary joints (particularly the joint where the brass lid fastens to the base) should be equipped with better sealing mechanisms. The use of larger o-rings or latches that could be attached to the lid for extensive tightening may work better and more consistently in sealing the system. The device needs to be redesigned.
so that each user can efficiently and effectively seal the system to prevent fluctuation in data.

- A linear bearing LVDT should be used to eliminate float sticking without the use of an automated vibration system. This will reduce the amount of hardware and software setup for new users and will eliminate data fluctuation caused by vibration.

- LabVIEW should be programmed to automatically close the solenoid valve after the test has run for a given period and the temperature has reached equilibrium. The current procedure specifies that the valve be left open overnight to allow temperature to come to equilibrium. However, it takes much less time for temperature to stabilize resulting in the loss of valuable ASR data. Programming the valve to close earlier would minimize the amount of loss data and early trends could be established.

- Test various siliceous aggregates with different ASTM C 1260 expansions to correlate aggregate activation energy.

- Investigate causes for volume expansion of calcium carbonate in water.

- Investigate causes for volume contraction of early age mortar in water aside from calcium dissolution.

- Testing should be performed at higher temperatures (70°C, 75°C, 80°C) to increase ASR expansion rates. This should especially be done when testing in low alkali solutions to eliminate volume contraction at the low temperatures.

- Test siliceous aggregate at different alkali levels to establish trend between ASR activation energy and alkalinity.
• Develop a universal testing procedure, apparatus design, and data acquisition program that different users can use to establish comparisons.

• Eventually test concrete and recycled concrete aggregate (RCA) for ASR in the dilatometer.

• Test fully hydrated mortar or concrete in supersaturated CaOH to see if a flat line can be achieved.
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APPENDIX A: Thermal Expansion Calculation

\[ \Delta T_{\text{OVER 60 HOURS}} = 0.032^\circ \text{C (FROM FIGURE 30)} \]
\[ \partial_T = 6.2082 \times 10^{-4} @ 80^\circ \text{C} \]

\[ \Delta V = (V_0)(\Delta T)(\partial_T) \]
\[ = (160 \text{ IN}^3)(0.032^\circ \text{C})(6.2082 \times 10^{-4}) \]
\[ = 0.0031 \text{ IN}^3 \]

CHANGE IN VOL = (\pi)(TOWER RADIUS)^2(DISPLACEMENT)
\[ \Delta v = (\pi)(R^2)(D) \]

\[ D = \Delta v/((\pi)(R^2)) \]
\[ = 0.0031/(\pi)(0.6875^2) \]
\[ = 0.0021 \text{ INCHES} \]

THE CALCULATED FLUID LOSS AS A RESULT OF THERMAL CONTRACTION WAS EQUAL TO 0.0021 INCHES.

THE ACTUAL FLUID LOSS OVER THE TEST (AS SEEN IN FIGURE 19) WAS EQUAL TO 0.0021 INCHES.

THERE WAS NO LOSS OF FLUID AS A RESULT OF LEAKING JOINTS. SYSTEM WAS PERFECTLY SEALED.
APPENDIX B: LabVIEW Front Panel

Displacement

Temperature

Sample rate (Minutes)
1

Vibrator