RESTRAINED SHRINKAGE BEHAVIOR DUE TO COMBINED AUTOGENOUS AND THERMAL EFFECTS IN MORTARS CONTAINING SUPER ABSORBENT POLYMER (SAP)

John L. Schlitter (1), T. Barrett (1) and W. Jason Weiss (1)

(1) School of Civil Engineering, Purdue University, West Lafayette, Indiana, USA

Abstract
Early age cracking has been observed to occur in concrete when it is prevented from shrinking freely. This cracking can occur more frequently in higher strength concrete due to a low water to cement ratio and an increase in the cement content. The low water to cement ratio exacerbates autogenous shrinkage while the high cement content increases both autogenous shrinkage and the potential for thermal cracking. Internal curing has been proposed as one method that can be used to reduce the potential for autogenous shrinkage cracking by providing internal reservoirs of water. Super absorbent polymers (SAP) are frequently used to absorb the water and to store that water. Several tests are frequently performed to assess the impact of materials composition on the potential for restrained shrinkage cracking [1]. The restrained ring test (ASTM C1581-09a) [2] is a simple, economical method to evaluate a concrete mixture’s susceptibility for restrained shrinkage cracking. Unfortunately, the restrained ring test only provides restraint against samples that shrink and can not be used to consider cases in which expansion occurs. Further, the conventional restrained ring test does not consider cases in which the concrete undergoes large variations in temperature since the restraint changes dimension under heating and cooling. This paper describes the use of the dual-ring test that was designed to overcome both of these limitations. In addition to assessing the autogenous shrinkage, the dual ring test can assess the influence of temperature on cracking. This paper will discuss the influence of SAP on reducing stresses that develop when autogenous shrinkage is restrained. In addition, this paper discusses the beneficial effect SAP has on reducing the potential for thermal cracking.
1. Introduction

High performance concrete (HPC) mixtures have been increasingly used based on their potential to provide a more durable product with improved service life. The use of low water-to-cement ratios mixtures (e.g., <0.40) results in a matrix with a finer pore network in HPC which in many ways is beneficial. Unfortunately, the loss of water from this fine network of pores due to self desiccation causes autogenous shrinkage [3]. This shrinkage may cause cracking when the concrete is restrained [4,5].

Shrinkage caused by self desiccation can be reduced or eliminated by supplying water to the pore network at the appropriate time. Traditional external curing provided through water ponding may not be effective in large concrete elements due to the low permeability of HPC which prohibits water from permeating through the concrete. Research has focused on the development of internal curing where water is supplied to the pores from distributed reservoirs throughout the mixture. One method of internal curing involves placing water filled porous lightweight aggregates throughout the concrete which release water from their pores when it is needed [6,7]. Alternatively, superabsorbent polymers (SAP) are being investigated as an alternative internal curing technology [8]. SAP particles have the ability to hold significant quantities of fluid to act as internal reservoirs and then later release the fluid into the concrete pore network.

2. Research Significance

Internally cured mixtures are known to cause early age expansion in addition to reducing autogenous shrinkage depending on the amount of curing water provided. Therefore, when studying the restrained behavior of internally cured mixtures, both expansion and shrinkage must be restrained. This paper examines the restrained behavior of internally cured mixtures with superabsorbent particles by using the dual restrained ring device which is capable of providing restraint for expansion and shrinkage. Further, this device has the ability of studying the restrained behavior due to changes in temperature. This paper will quantify the ability of superabsorbent particles to reduce the potential of thermal cracking.

3. Materials and Experimental Procedures

3.1 Mixture Proportions

Three mortar mixtures were used in this study to assess the effects of SAP. They are designated as M-0, M-33, and M-50 with their mixture proportions provided in Table 1. Every mixture was designed with a water-to-cement (w/c) ratio of 0.30 by mass and an aggregate volume of 55%. The proportions of SAP were designed to supply a volume of internal curing (IC) water that is equivalent to a portion of the volume of chemical shrinkage calculated with Equation (1) [6] which is generally appropriate for low w/c mixtures. The IC water does not contribute to the w/c of the mixture because it is absorbed by the SAP at the time of set. Mixtures M-33 and M-50 were proportioned to provide 33% and 50% of the chemical shrinkage volume respectively. The mixture proportions used volumetric
calculations of the cement, fine aggregate, water, SAP (and IC water held in the SAP), and high range water reducing admixture (WRA).

\[ V_{\text{Water}} = \frac{C_f \cdot CS \cdot \alpha_{\text{max}}}{\rho} \]  

(1)

**Table 1 – SSD Mixture Proportions (kg/m³)**

<table>
<thead>
<tr>
<th>Material</th>
<th>M-0</th>
<th>M-33</th>
<th>M-50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement (Type I)</td>
<td>728</td>
<td>707</td>
<td>696</td>
</tr>
<tr>
<td>Bulk Water</td>
<td>218</td>
<td>212</td>
<td>209</td>
</tr>
<tr>
<td>IC Water*</td>
<td>0</td>
<td>12.44</td>
<td>18.57</td>
</tr>
<tr>
<td>Fine Aggregate</td>
<td>1440</td>
<td>1440</td>
<td>1440</td>
</tr>
<tr>
<td>SAP</td>
<td>0.00</td>
<td>0.69</td>
<td>1.03</td>
</tr>
</tbody>
</table>

* Water absorbed by SAP at time of set

### 3.2 Materials

Type I ordinary portland cement (ASTM C 150-05) [2] was used with a Blaine fineness of 370 m²/kg, and an estimated Bogue composition of a 56 % C₃S, 16 % C₂S, 12 % C₃A and 7 % C₄AF. River sand was used with a fineness modulus of 2.71 and an oven dry specific gravity of 2.58. A high range water reducing admixture (HRWRA) Glenium 3030 was added at 1 gram per 100 grams of cement. Tap water (23°C ± 1°C) was used as the mix water. A Super Absorbent Polymer (SAP) was used for internal curing [8].

### 3.3 Mixing Procedure

The mixing procedure followed ASTM C 192-07 [2] where the oven dried fine aggregate, cement, and SAP were placed in the lightly dampened mixer and mixed for 15 seconds to achieve an even distribution of SAP. The mix water containing the HRWRA was then added and the time of water to cement contact was noted. The batch was mixed for 3 minutes, rested for 3 minutes while the sides of the mixer were scraped, and then mixed for a final 2 minutes.

### 3.4 Absorption Test for SAP

Determining the absorption value of the SAP is challenging because it is difficult to distinguish between SAP gel and unabsorbed fluid. There are various methods that have been utilized to determine the absorption capacity of SAP [1]. This study utilized a modified version of the teabag test due to its relative simplicity and repeatability. A coffee filter was used to submerge a known weight of SAP in simulated pore solution for approximately one minute to allow all of the SAP particles to become saturated. A small rod was used to slightly agitate the SAP to ensure all particles were exposed to the fluid. The filter and SAP were then allowed to drip dry, and weight measurements were taken at 2, 5, 15, and 30 minutes. The test was repeated for two weights (0.25g and 0.50g) of SAP. A simulated pore solution (Table 2) was used to better represent the fluid the SAP would encounter in the cementitious paste.

### 3.5 Unrestrained Linear Autogenous Deformation – Corrugated Tube Test

The corrugated tube test ASTM C 1698-09 [2] was used to measure the linear autogenous deformation of the three mortar mixtures in the first seven days [9]. Each mortar mixture was
cast into three 30mm diameter corrugated polyethylene tubes. Each tube was filled, vibrated, tapped, then capped and placed in steel racks within a controlled humidity and temperature chamber. Linear Variable Differential Transformer (LVDT’s) displacement transducers (0.5 mm) measured the deformation at each end of the tube once every five minutes. The measurements were zeroed at the time of set as assessed using ASTM C403.

Table 2 – Simulated Pore Solution

| Amount (g/g water) |  
|-------------------|---|
| NaOH              | 0.010 |
| KOH               | 1.884 |
| Ca(OH)$_2$        | 0.022 |
| K$_2$SO$_4$       | 4.844 |
| Na$_2$SO$_4$      | 1.404 |
| CaSO$_4$          | 0.370 |

3.6 Split Tensile, Compressive Strength, and Elastic Modulus Tests
A set of 100mm x 200mm cylinders were cast for each of the three mortar mixtures in order to determine the development of split tensile capacity and elastic modulus during the first seven days. The cylinders were cast in three lifts, rodded 25 times and vibrated after each lift. Using a hydraulic compression machine with a 3000 kN capacity, the split tensile capacity and elastic modulus were measured at ages 1, 3, 5, and 7 days after mixing. The split tensile test was carried out in accordance with ASTM C 496/C 496M-04 where cylinders were loaded perpendicular to their length with a load distribution bar until splitting was observed. Three cylinders were split tensile tested for each mixture at each age.

The elastic modulus was measured in accordance with ASTM C 469-02 where a compressometer was affixed to the test cylinder which was loaded to 40% of the ultimate load. The compressometer utilized a displacement transducer to measure the change in length of the cylinder due to the applied compression and provided continuous measurement during the loading procedure. Two cylinders were tested for elastic modulus for each mixture at each age. No cylinder was tested for elastic modulus at more than one age.

3.7 Restrained Shrinkage – Dual Ring Test
The restrained shrinkage behavior of the mortar mixtures were characterized with a newly developed dual ring restraining device [10] as shown in Figure 1. This device is based upon the standardized single ring test (ASTM C1581-09) where an annulus of mortar is cast around a metal ring that provides restraint [11]. The standard ring test is limited to restraining specimens that shrink. As its name implies, the dual ring test incorporates a second ring located on the outer edge of the sample in order to provide restraint against specimens that expand in addition to shrinking. This capability is significant when studying shrinkage reducing technologies such as internal curing [12] as they may exhibit early age expansion.
The dual ring test also has the ability of maintaining a stable degree of restraint while varying temperature. This is significant because it enables the study of thermally induced volume changes in addition to volume change from autogenous shrinkage. Contrary to the standard restraining ring test which is fabricated from conventional ASTM A53 Grade B steel, the dual ring was fabricated from Invar 36 which has an extremely low coefficient of thermal expansion. When the temperature is varied, the dual restraining rings remain volumetrically stable while the specimen shrinks or expands thereby allowing the residual stresses to be measured.

The restraining rings are instrumented with four equally spaced CEA-00 strain gages in order to measure the ring strain which is then used to calculate the residual stress in the specimen [13-15]. Converting ring strain to specimen residual stress is based upon principles of force equilibrium and displacement compatibility. The residual stress is calculated with Equation (2) where \( R_{IC}, R_{II}, R_{IC}, R_{OC}, R_{OO} \) represent the ring geometry, \( (\varepsilon_{IN}) \) and \( (\varepsilon_{OUT}) \) represent the average strain output from the four gauges on each ring, and \( E_{INVAR} \) represents the modulus of Invar which is 141 GPa (20450 ksi). The simplified equation for the dual ring geometry used in this study is provided in Equation (3).

\[
\sigma_{R}(R_{IC}) = -\varepsilon_{IN}E_{INVAR}\left(\frac{R_{IC}^2 - R_{II}^2}{2R_{IC}^2}\right)\left[\frac{R_{OC}^2 + R_{IC}^2}{R_{OC}^2 - R_{IC}^2}\right] - \varepsilon_{OUT}E_{INVAR}\left(\frac{R_{OO}^2 - R_{OC}^2}{2R_{OC}^2}\right)\left[\frac{2R_{OC}^2}{R_{OC}^2 - R_{IC}^2}\right]
\] (2)

\[
\sigma_{R}(R_{IC}) = -0.53\varepsilon_{IN}E_{INVAR} - 0.58\varepsilon_{OUT}E_{INVAR}
\] (3)

The temperature of the dual ring test is controlled by placing the specimen and restraining rings inside a highly insulated chamber with a coil of copper tubing in which ethylene glycol–water mixture is pumped by a water bath. In this study, the water bath temperature was held at a constant 23°C for a select amount of time and then lowered at a rate of 2°C per hour. Multiple test series were performed on mixtures M-0 and M-33 where the temperature was lowered at various times within the first five days. This approach investigated if the specimen cracked, its cracking capacity, and the amount of temperature changed required to induce cracking. It should be noted that it is assumed the specimen does not undergo drying shrinkage as it is sealed inside the insulation chamber throughout the test duration.
4. Results and Discussion

4.1 Results of Absorption Test
Figure 2 shows the results of the absorption test. Two trials were performed using different amounts of dry SAP. The absorption value converged to 18 grams of water per 1 gram of dry SAP. This absorption value was then used in Equation (1) to determine the amount of SAP required to match the specified portion of chemical shrinkage within the mortar.

4.2 Results of Unrestrained Linear Autogenous Deformation Test
The results of the unrestrained linear autogenous deformation can be seen in Figure 3. Mixtures with SAP have less linear shrinkage than the control M-0 mixture. Mixture M-33 reduced the shrinkage by 50%. Mixture M-50 expanded rapidly until 24 hrs, where it reached a plateau until 48 hrs then continued to expand at a constant rate through the end of the test.

4.3 Results of Split Tensile, Modulus Testing, and Compressive Strength
The results of the split tensile testing are shown in Figure 4. The observed tensile capacity of the SAP mixture is slightly less than the plain mixture. SAP mixture M-33 was on average 1.2 MPa (174 psi) or 20% lower than the plain mixture M-0. This can be explained by the fact that the SAP particles can act like low strength voids in the system [16]. The seven day modulus history of mixtures M-0 and M-33 is presented in Figure 5. A 5% to 10% reduction in modulus was observed for the SAP mixture M-33. As the data indicates, the modulus was reduced at early ages creating a more compliant sample. Figure 6 shows the compressive strength history of M-0 and M-33. A 10% to 20% reduction in compressive strength was observed for the SAP mixture M-33.
4.4 Results of Dual Ring Test

The residual stress from the series of five M-0 specimens tested in the dual ring is presented in Figure 7. Four specimens were subjected to a temperature profile consisting of constant temperature 23°C± 0.2 °C, and then reduced at a constant rate of 1°C/h beginning at either 24, 36, 48, or 72 hours. One M-0 specimen was held at a constant temperature until it cracked. The residual stress that developed prior to reducing temperature was due almost entirely to autogenous shrinkage. When the temperature was reduced, the residual stress increased due to the thermal volume change of the specimen. Cracking of the specimen can be seen by the sudden release of residual stress (i.e., a sudden decrease in stress). The temperature reduction required to crack the four M-0 specimens was between -10°C and -15°C.

The residual stress series from the M-33 SAP mixture is presented in Figure 8. The residual stress observed during the constant temperature period was reduced by 30% when compared to the plain M-0 mixture. This was expected based on the reduced free shrinkage observed in the corrugated tube results. Three of the four M-33 specimens cracked, and they required twice the amount of temperature reduction to crack as compared to the M-0 mixture. Further, the M-33 specimens cracked at higher stresses than the plain M-0 mixture. This appears to
contradict the split tensile results which had shown the SAP mixture was weaker in tension. This is likely due to the potential for microcracking which could reduce the strength of a concrete under sustained load. In addition, there may be an effect of stiffness and time dependant loading contributing to cracking performance. The M-33 SAP mixture is more compliant and is subjected to lower sustained levels of residual stress prior to lowering the temperature; therefore it is less likely to develop cracks.

The temperature reduction series can be used to show how near the sample is to cracking. The reserve cracking capacity of the specimen can be defined as the magnitude of stress required by the temperature reduction to crack the sample. A reserve capacity value was measured every time the specimen cracked. The summary of these values is plotted in Figure 9 and it can be seen that the M-33 mixture has approximately twice the reserve strength when compared with the plain M-0 mixture. Therefore, the SAP mixture would be expected to be far less sensitive to restrained shrinkage cracking.

![Figure 8 – M-33 Residual Stress Series](image1)

![Figure 9 – Reserve Strength](image2)

A single M-50 specimen was tested with a temperature reduction occurring at 72 hours. The residual stress from this specimen is plotted along with the 72 hour M-0 and M-33 mixtures in Figure 10. The expansion of the M-50 mixture observed in the corrugated tube test was observed to produce a compressive residual stress in the dual ring that peaked at 13 hours and completely dissipated by 24 hours. No strain developed in the inner ring during the first 24 hours. It is likely that this reduction in stress is due to stress relaxation while the modulus of elasticity is low and the creep is high in the sample at early age. From 24 hours until 72 hours the M-50 mixture developed 50% less residual stress due to autogenous shrinkage than the M-33 mixture. While the reduced residual stress is typically desired, its benefit may not be realized. The reserve cracking capacity of the M-50 mixture was observed to equal that of the M-33 mixture at 72 hours (3.5 MPa). Although the M-50 mixture developed less residual stress due to autogenous shrinkage, its cracking capacity was also reduced. This suggests that increased SAP dosages would not likely benefit the cracking performance of the mixture. Further trials at different SAP dosages would be required to optimize the cracking capacity.
5. Conclusions

The use of superabsorbent polymers (SAP) as an internal curing agent in concrete has shown positive results to reduce early age cracking. Although up to 20% reduction in compressive and tensile strength were observed in specimens containing SAP, the early age restrained shrinkage cracking resistance improved. This behavior can be attributed to reduced autogenous shrinkage, reduced modulus of elasticity and possibly an increase in creep. The dual ring provides a unique method to assess the restrained shrinkage performance of introducing SAP to cementitious mixtures. It has produced initial data indicating that relatively modest dosages of SAP may provide measurable benefits.

Acknowledgements

The authors gratefully acknowledge the donation of the SAP material provided by Dr. Ole Mejlhede Jensen. This work was conducted in the Charles Pankow Concrete Materials Laboratory at Purdue University. As such, the authors gratefully acknowledge the support which has made this laboratory and its operation possible.

Disclaimer

Certain commercial equipment, instruments, or materials are identified in this report in order to foster understanding. Such identification is not intended to imply recommendation or endorsement by Purdue University, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

References


