

# RECLAMATION

*Managing Water in the West*

## Preliminary Chemical Shrinkage Analysis of Nano Silica Cementitious Binders

***Science & Technology Program***

MERL Research Report No. MERL-2013-53

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Bureau of Reclamation  
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**BUREAU OF RECLAMATION**  
**Science & Technology Program**  
**Materials Engineering and Research Laboratory, 86-68180**

# **Chemical Shrinkage Analysis of Nano Silica Cementitious Binders**

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# Contents

|                          |           |
|--------------------------|-----------|
| <b>Background .....</b>  | <b>1</b>  |
| <b>Introduction.....</b> | <b>2</b>  |
| <b>Conclusions.....</b>  | <b>4</b>  |
| <b>Test Results.....</b> | <b>5</b>  |
| Regime 1 .....           | 7         |
| Regime 2 .....           | 9         |
| Regime 3 .....           | 11        |
| Regime 4 .....           | 13        |
| <b>References.....</b>   | <b>15</b> |

## List of Figures

|                                          |    |
|------------------------------------------|----|
| Figure 1. Automated setup.....           | 6  |
| Figure 2. Regime 1, gradation 1.....     | 8  |
| Figure 3. Regime 1, gradation 2.....     | 8  |
| Figure 4. Regime 1, gradation 3.....     | 8  |
| Figure 5. Regime 2, gradation 1.....     | 10 |
| Figure 6. Regime 2, gradation 2.....     | 10 |
| Figure 7. Regime 2, gradation 3.....     | 10 |
| Figure 8. Regime 3, gradation 1.....     | 12 |
| Figure 9. Regime 3, gradation 2.....     | 12 |
| Figure 10. Regime 3, gradation 3.....    | 12 |
| Figure 11. Regime 4, slag mixes.....     | 14 |
| Figure 12. Regime 4, fly ash mixes. .... | 14 |

## List of Tables

|                                                                          |    |
|--------------------------------------------------------------------------|----|
| Table 1. Testing Matrix Description.....                                 | 3  |
| Table 2. Description of the Four NS Replacements for Each Gradation..... | 3  |
| Table 3. Regime 1 mix design. ....                                       | 7  |
| Table 4. Regime 2 mix design. ....                                       | 9  |
| Table 5. Regime 3 mix design. ....                                       | 11 |
| Table 6. Regime 4 mix design. ....                                       | 13 |

# Background

The Bureau of Reclamation's Materials Engineering and Research Laboratory (MERL) requested research money to performing chemical shrinkage testing for nano-silica (NS) paste mixes.

The strength and durability of concrete is dependent on the engineering properties of the hydrated cement matrix and its resilience to chemical degradation over time. With the advent of emerging technologies, recycled materials have lead their way into concrete to make a more sustainable product. Silica fume, or micro silica, is a smoke byproduct from furnaces used to produce silicon metal or ferrosilicon alloys. Micro silica particles are in the 0.1  $\mu\text{m}$  range, 100 times smaller than cement particles. This pozzolan is already used as a partial replacement for cement in concrete for increased strength and abrasion resistance.

Recent advances in nanotechnology have resulted in the manufacturing of colloidal silica in a laboratory possible. This colloidal silica is popular in the electronics and coatings industry. A nano-meter (nm) silica particle is 1000 times smaller than the micro silica particle currently used in concrete. Jon Belkowitz, from Intelligent Concrete, is currently pursuing a Ph D in Mechanical Engineering at Steven's Institute of Technology. He has previously tested concrete with NS particles for strength, shrinkage, and thermal gravimetric analysis. Through the use of nano-materials, an increase can be observed in engineering properties of concrete by almost 30%. In addition it can significantly reduce the chemical degradation of concrete while reducing the total amount of cementitious materials needed for a given concrete strength.

The shrinkage performance of cementitious binders made with NS, compared to other traditional concreting technologies, is unknown at this time. Shrinkage in the concrete matrix is a main contributor of cracks and therefore reduced durability. Evaluation is needed before the use of NS will be considered effective for shrinkage reduction. This research aims to test the effects of different sizes and percentages of NS in cement binders to evaluate chemical shrinkage.

# Introduction

The investigation was performed by personnel of the Materials Engineering and Research Laboratory (MERL). ASTM C 1608 Method A, “Standard Test Method for Chemical Shrinkage of Hydraulic Cement Paste<sup>1</sup>” was used to compare various cementitious binders according to the test matrix developed. Approximately 50 samples were tested. Three different NS gradations were used throughout experimentation in a combination of four regimes. Each gradation is a narrow distribution with spherical particle diameters ranging: 1) Gradation 1 (G1) 3-6 nm, 2) Gradation 2 (G2) 15-17 nm and 3) Gradation 3 (G3) 45-47 nm.

For a basis of comparison, control mixtures were analyzed before the experiments were carried out using the NS. Four separate regimes were used to look at the interaction and effects of NS dosage on various cementitious materials combinations. The program was designated to identify the size effects of NS on the chemical shrinkage of the concrete binding matrix utilizing specific replacements of cement with common secondary cementitious materials (SCMs). Four percentage replacements were selected based on surface area calculations of the NS particles in relation to the particle size of Ordinary Portland Cement (OPC) to match a proportional volume of NS required due to voids in the given mix. Regime 4 tested only control mixtures (no NS) consisting of OPC with high volume replacements of SCMs (Class F fly ash and Slag). The mixtures were proportioned to discern differences in shrinkage reduction specific to the different gradations in order to obtain the optimal replacement dosage of NS.

A description of the test matrix is provided below in Table 1 and can be seen in completion in Appendix A. The four percent replacements of NS were identified with letters A through D. A description of the replacements for each gradation category is provided in Table 2. For each sample test, the regime number, gradation range, and surface area percentage replacement letter are used for identification. For example, R2-1B would represent regime 2, gradation 1, and NS replacement percentage B. Regimes 1 through 3 also have a control mix where no NS replacement occurred. This is designated by regime number and “control”, for example, R1-CONTROL. All regime 4 samples were cement and SCM only mixes, no NS. The percentage of the SCM used is designated in the name followed by either FA for class F fly ash or SLAG for slag. An example of regime 4 is R4-40FA, where 40FA is fly ash replacement of cement at 40% of the total cementitious.

This report gives a brief summary of the test results and draws some very limited conclusions. Further analysis of the data and NS interactions are expected to be discussed further in a journal article or subsequent report.

**Table 1. Testing Matrix Description**

|                                                            |                                                                                                                                                |
|------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------|
| <i>Regime 1</i><br>OPC Replaced by NS                      | CONTROL - OPC Only<br>G1 – 4 different NS % Replacements<br>G2 – 4 different NS % Replacements<br>G3 – 4 different NS % Replacements           |
| <i>Regime 2</i><br>OPC Replaced by 20% Pozz - Class F + NS | CONTROL - OPC + Class F Only<br>G1 – 4 different NS % Replacements<br>G2 – 4 different NS % Replacements<br>G3 – 4 different NS % Replacements |
| <i>Regime 3</i><br>OPC Replaced by 20% Slag + NS           | CONTROL - OPC + SLAG Only<br>G1 – 4 different NS % Replacements<br>G2 – 4 different NS % Replacements<br>G3 – 4 different NS % Replacements    |
| <i>Regime 4</i><br>OPC Replaced by Pozz - Type F or Slag   | OPC + 30 Class F Only<br>OPC + 40 Class F Only<br>OPC + 50 Class F Only<br>OPC + 30 Slag Only<br>OPC + 40 Slag Only<br>OPC + 50 Slag Only      |

**Table 2. Description of the Four NS Replacements for Each Gradation**

| Replacement | G1 = 3-6 nm | G2 = 15-17 nm | G3 = 45-47 nm |
|-------------|-------------|---------------|---------------|
| A           | 0.0087%     | 0.0218%       | 0.0546%       |
| B           | 0.0874%     | 0.2184%       | 0.5460%       |
| C           | 0.8736%     | 2.1840%       | 5.4600%       |
| D           | 1.7472%     | 4.3680%       | 10.9200%      |

# Conclusions

- Chemical shrinkage is affected by adding NS to cement paste mixes.
- Regime 1 used NS to replace OPC in multiple amounts and gradations to test for chemical shrinkage. Each NS mix had a lower chemical shrinkage than the control mix without NS. Shrinkage reduction with the addition on the NS ranged from 2 to 46% of the OPC Control. As the dosage of NS increased the chemical shrinkage did not necessarily decrease.
- The 20% class F replacement control mix had approximately 20% reduction in shrinkage from the OPC control. An additional 10 to 45% more shrinkage reduction was seen from NS replacement on top of the initial 20% reduction from the ash. In Regime 2, each NS mix performed better than the 20% class F replacement control mix in terms of chemical shrinkage. Each gradation and percent replacement combination performed about the same and shrinkage results did not differ greatly from one to another.
- The 20% slag replacement control mix had approximately 50% reduction in shrinkage from the OPC control. Regime 3 is the only set of mixes where the 20% slag control mix had less shrinkage than the mixes with the NS addition. This was exaggerated more in gradations 1 and 3, but was inconsistent at best. It appears that for mixtures with slag, NS does not further decrease the shrinkage, and could in some cases actually reduce the overall shrinkage benefits of the slag.
- Regime 4 used slag and class F fly ash to replace OPC by 30%, 40%, and 50%.
  - Increasing fly ash and slag both decrease the chemical shrinkage for these tests. The 20% fly ash replacement control mix had approximately 20% reduction in shrinkage from the OPC control, whereas at high volumes of class F fly ash, a 40-50% reduction in shrinkage from OPC can be realized.
  - The 20% slag replacement control mix had approximately 50% reduction in shrinkage from the OPC control, whereas at high volumes of slag an additional benefit is not realized.
- Slag and high volume class F fly ash mixes had the overall highest reduction in shrinkage from the OPC control mix. However, for specific gradation and replacement combinations the 20% fly ash mixes with NS could also achieve this reduction in shrinkage at comparable levels.

# Test Results

Chemical shrinkage results are presented from graphs after performing ASTM C1608. All paste mixes were mixed with de-aired water and the required cement/slag/fly ash/NS. The paste was then carefully placed in a vial that was roughly 50 mm in height and 25 mm in diameter. The paste is placed at a height of no more than 20 mm in the vial. De-aired water is carefully placed above the sample with a ~3 ml disposable syringe. Then a rubber stopper, through which an inverted glass pipette passes (1 ml), is placed carefully into the top of the vial. Additional de-aired water is added to the top of the pipette so it is close to full and a few drops of paraffin oil are added to the top of the pipette to prevent evaporation. The vial/pipette combination is placed in a water bath in an appropriate holder immediately after casting and addition of water to the vial. The level of de-aired water in the pipette is measured for a period of at least 24 hours and chemical shrinkage is reported as a normalized value of ml of water absorbed to grams of cement in the vial for this 24 hour period<sup>2</sup>.

The water level in the pipette was obtained from a webcam that took pictures at specific intervals during the 24 hour test. These pictures were then run through a program that recorded fluid displacement. This can be seen in the figure below. Some of the tests did not have a successful output from the program and these results were read manually from the pictures taken. This automated setup was built at MERL and based on previous models developed by Ecole Polytechnique in Lausanne, Switzerland and the University of Texas Austin.



**Figure 1. Automated setup.**

## Regime 1

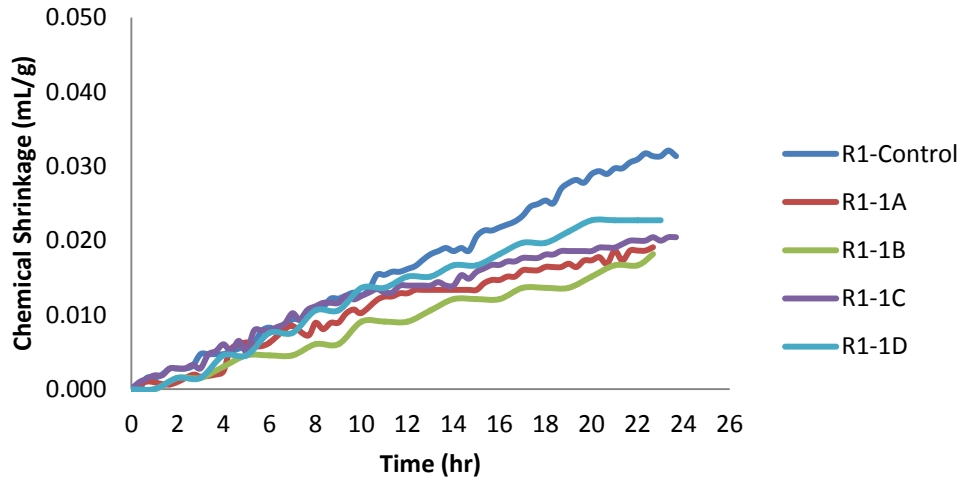
Thirteen mixes were made with 2 samples per mix. This regime had mixes that replaced OPC with NS. The table below shows the mix design used for this regime, including the identification, gradation, cement, NS, and water.

**Table 3. Regime 1 mix design.**

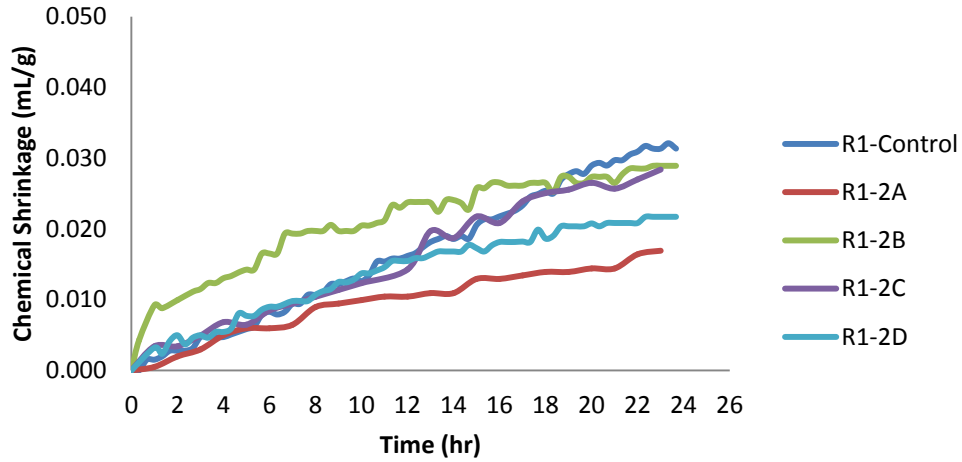
| Mix # | Identification/Gradation |         | Cement | NS    | Water |
|-------|--------------------------|---------|--------|-------|-------|
| 1     | R1-                      | CONTROL | 350.0  | 0     | 140   |
| 2     | R1-                      | 1A      | 350.0  | 0.20  | 139.8 |
| 3     | R1-                      | 1B      | 349.7  | 2.04  | 138.4 |
| 4     | R1-                      | 1C      | 346.9  | 20.38 | 124.2 |
| 5     | R1-                      | 1D      | 343.9  | 40.77 | 108.5 |
| 6     | R1-                      | 2A      | 349.9  | 0.16  | 139.9 |
| 7     | R1-                      | 2B      | 349.2  | 1.61  | 139.4 |
| 8     | R1-                      | 2C      | 324.0  | 16.09 | 134.3 |
| 9     | R1-                      | 2D      | 333.9  | 32.19 | 128.5 |
| 10    | R1-                      | 3A      | 349.8  | 0.38  | 139.9 |
| 11    | R1-                      | 3B      | 348.1  | 3.82  | 138.6 |
| 12    | R1-                      | 3C      | 330.9  | 38.22 | 126.4 |
| 13    | R1-                      | 3D      | 311.8  | 76.44 | 112.7 |

Figures 2-4 show the chemical shrinkage results for Regime 1.

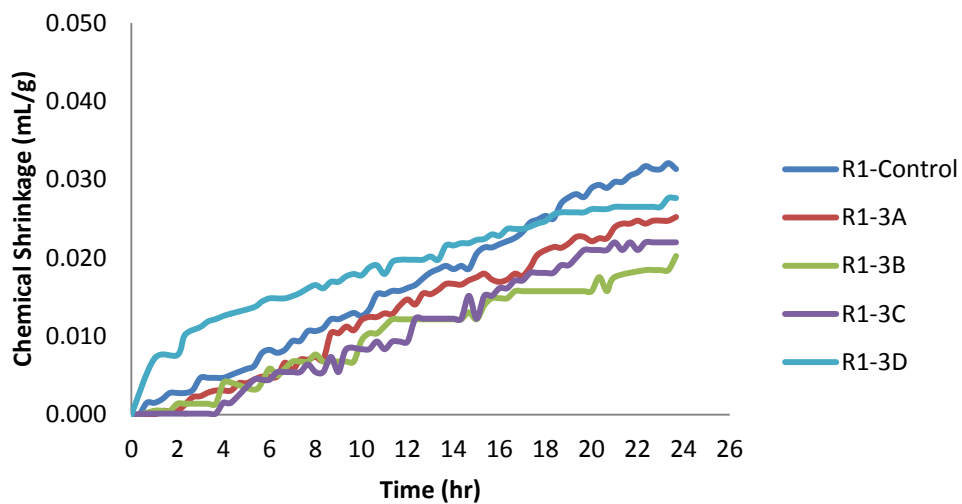
At the 24 hour readings for each mix, the control mix had more shrinkage versus the NS mixes. Shrinkage reduction with the addition on the NS ranged from 2 to 46% of the OPC Control. Gradation 2 at the lowest NS replacement rate had the highest shrinkage reduction at about 46%. As the dosage of NS increased the chemical shrinkage did not necessarily decrease.



**Figure 2. Regime 1, gradation 1.**



**Figure 3. Regime 1, gradation 2.**



**Figure 4. Regime 1, gradation 3.**

## Regime 2

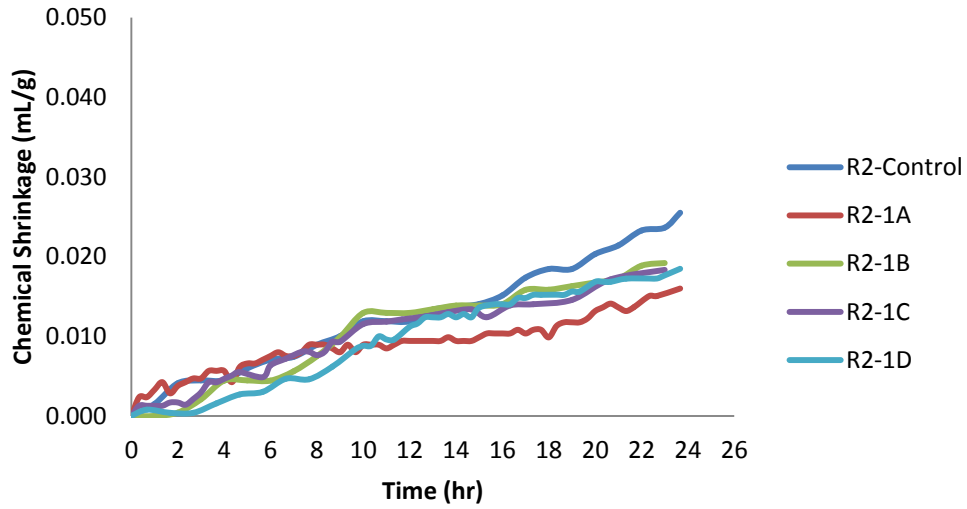
Thirteen mixes were made with 2 samples per mix. This regime had mixes that replaced OPC with 20% class F fly ash and NS. The table below shows the mix design used for this regime, including the identification, gradation, cement, fly ash, NS, and water.

**Table 4. Regime 2 mix design.**

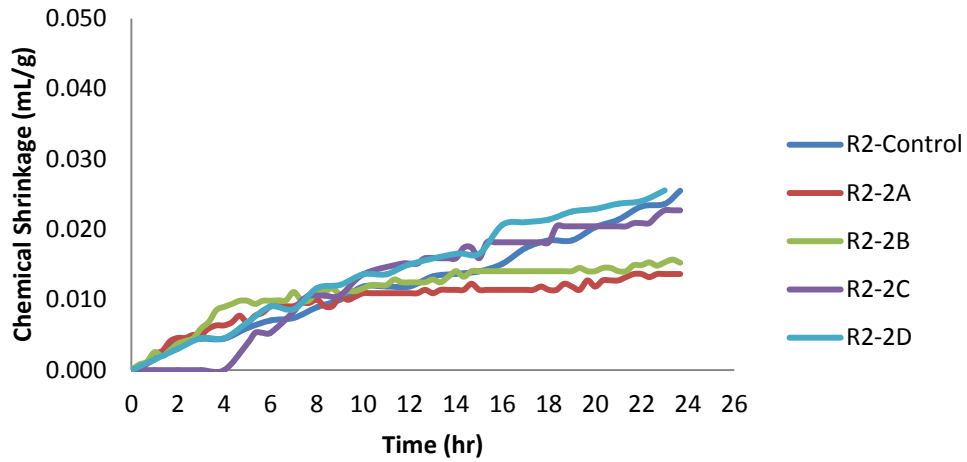
| Mix # | Identification/Gradation | Cement | Fly Ash | NS    | Water |
|-------|--------------------------|--------|---------|-------|-------|
| 1     | R2- CONTROL              | 280.0  | 70.0    | 0.00  | 140.0 |
| 2     | R2- 1A                   | 280.0  | 70.0    | 0.20  | 139.8 |
| 3     | R2- 1B                   | 280.0  | 69.7    | 2.04  | 138.4 |
| 4     | R2- 1C                   | 280.0  | 66.9    | 20.38 | 124.2 |
| 5     | R2- 1D                   | 280.0  | 63.9    | 40.77 | 108.5 |
| 6     | R2- 2A                   | 280.0  | 69.9    | 0.16  | 139.9 |
| 7     | R2- 2B                   | 280.0  | 69.2    | 1.61  | 139.4 |
| 8     | R2- 2C                   | 280.0  | 62.0    | 16.09 | 134.3 |
| 9     | R2- 2D                   | 280.0  | 53.9    | 32.19 | 128.5 |
| 10    | R2- 3A                   | 280.0  | 69.8    | 0.38  | 139.9 |
| 11    | R2- 3B                   | 280.0  | 68.1    | 3.82  | 138.6 |
| 12    | R2- 3C                   | 280.0  | 50.9    | 38.22 | 126.4 |
| 13    | R2- 3D                   | 280.0  | 31.8    | 76.44 | 112.7 |

Figures 5-7 show the chemical shrinkage results for Regime 2.

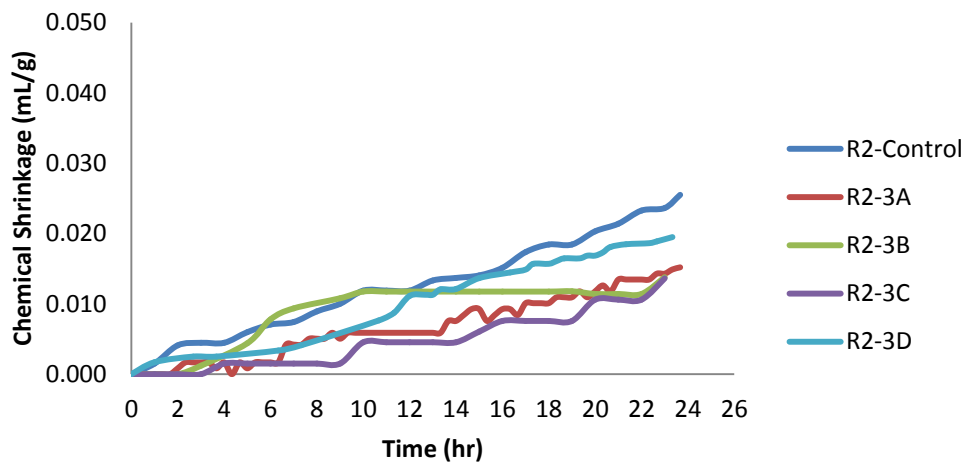
The 20% class F replacement control mix had approximately 20% reduction in shrinkage from the OPC control. An additional 10 to 45% shrinkage reduction was seen from NS replacement on top of the initial 20% reduction from the ash. In Regime 2, each NS mix performed better than the 20% class F replacement control mix in terms of chemical shrinkage. Each gradation and percent replacement combination performed about the same and shrinkage results did not differ greatly from one to another.



**Figure 5. Regime 2, gradation 1.**



**Figure 6. Regime 2, gradation 2.**



**Figure 7. Regime 2, gradation 3.**

### Regime 3

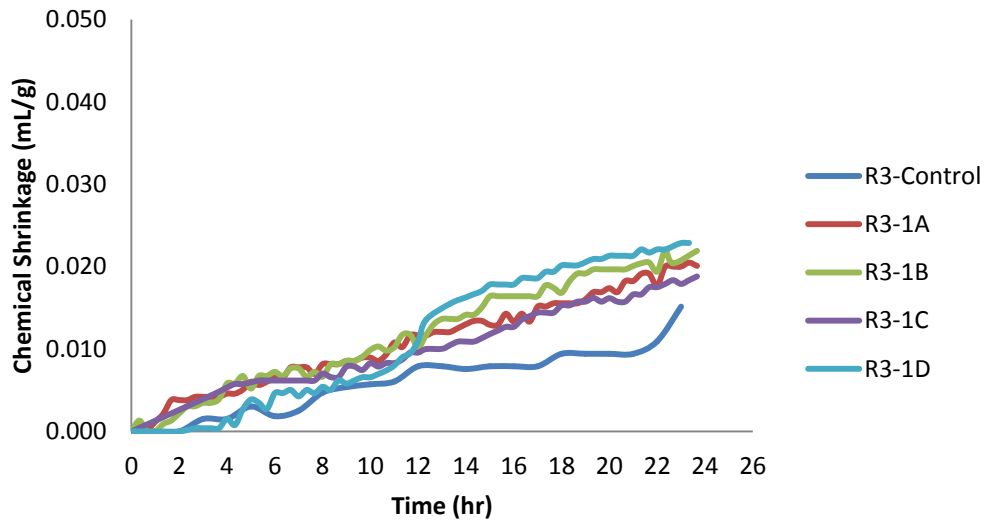
Thirteen mixes were made with 2 samples per mix. This regime had mixes that replaced OPC with 20% slag and NS. The table below shows the mix design used for this regime, including the identification, gradation, cement, fly ash, NS, and water.

**Table 5. Regime 3 mix design.**

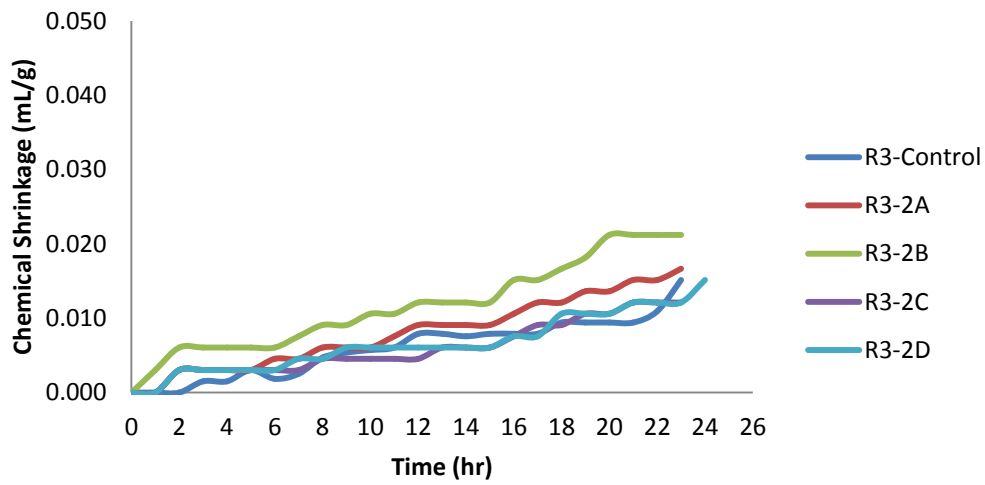
| Mix # | Identification/Gradation | Cement | Slag | NS    | Water |
|-------|--------------------------|--------|------|-------|-------|
| 1     | R3- CONTROL              | 280.0  | 70.0 | 0     | 140.0 |
| 2     | R3- 1A                   | 280.0  | 70.0 | 0.15  | 139.9 |
| 3     | R3- 1B                   | 280.0  | 69.8 | 1.47  | 138.9 |
| 4     | R3- 1C                   | 280.0  | 67.8 | 14.66 | 128.7 |
| 5     | R3- 1D                   | 280.0  | 65.6 | 29.32 | 117.3 |
| 6     | R3- 2A                   | 280.0  | 69.9 | 0.12  | 140.0 |
| 7     | R3- 2B                   | 280.0  | 69.4 | 1.16  | 139.6 |
| 8     | R3- 2C                   | 280.0  | 64.2 | 11.57 | 135.9 |
| 9     | R3- 2D                   | 280.0  | 58.4 | 23.14 | 131.7 |
| 10    | R3- 3A                   | 280.0  | 69.9 | 0.27  | 139.9 |
| 11    | R3- 3B                   | 280.0  | 68.6 | 2.75  | 139.0 |
| 12    | R3- 3C                   | 280.0  | 56.3 | 27.48 | 130.2 |
| 13    | R3- 3D                   | 280.0  | 42.5 | 54.97 | 120.4 |

Figures 8 -10 show the chemical shrinkage results for Regime 3.

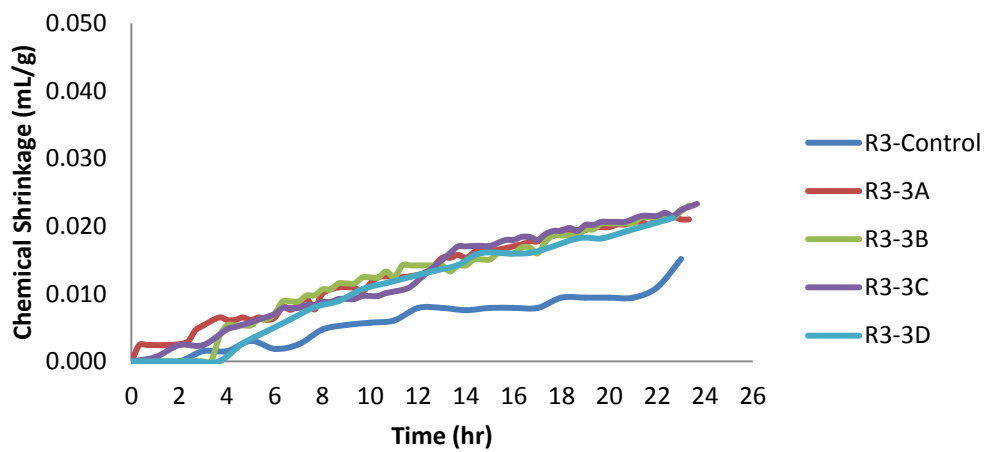
The 20% slag replacement control mix had approximately 50% reduction in shrinkage from the OPC control. Regime 3 is the only set of mixes where the 20% slag control mix had less shrinkage than the mixes with the NS addition. This was exaggerated more in gradations 1 and 3, but was inconsistent at best. It appears that for mixtures with slag, NS does not further decrease the shrinkage, and could in some cases actually reduce the overall shrinkage benefits of the slag.



**Figure 8. Regime 3, gradation 1.**



**Figure 9. Regime 3, gradation 2.**



**Figure 10. Regime 3, gradation 3.**

## Regime 4

Six mixes were made with 2 samples per mix. This regime had mixes that replaced OPC with class F fly ash and slag both with percentage replacements of 30%, 40%, and 50%. The table below shows the mix design used for this regime, including the identification, gradation, cement, slag, NS, and water.

**Table 6. Regime 4 mix design.**

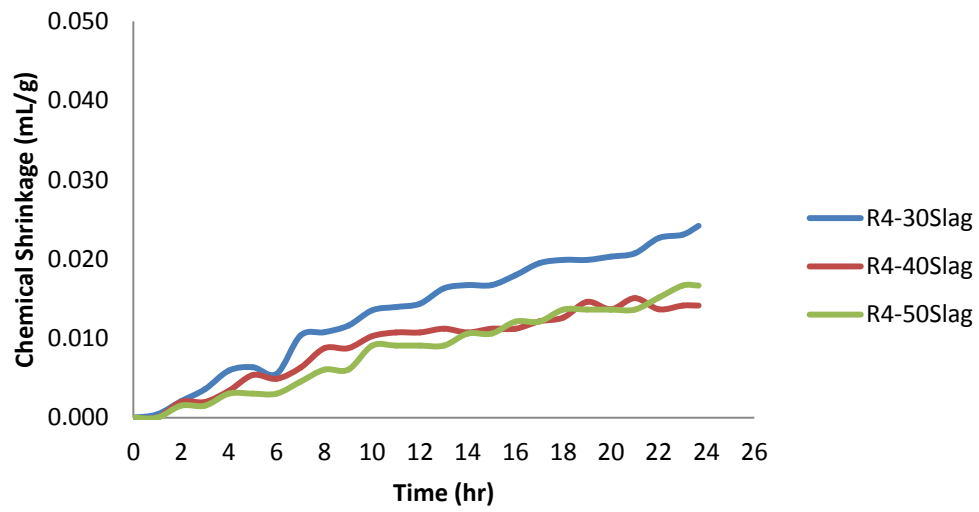
| Mix # | Identification/Gradation | Cement | Fly Ash | Slag |
|-------|--------------------------|--------|---------|------|
| 1     | R4- 30FA                 | 245    | 105     | 0    |
| 2     | R4- 40FA                 | 210    | 140     | 0    |
| 3     | R4- 50FA                 | 175    | 175     | 0    |
| 7     | R4- 30SLAG               | 245    | 0       | 105  |
| 8     | R4- 40SLAG               | 210    | 0       | 140  |
| 9     | R4- 50SLAG               | 175    | 0       | 175  |

Figures 11 and 12 show regime 4 chemical shrinkage results.

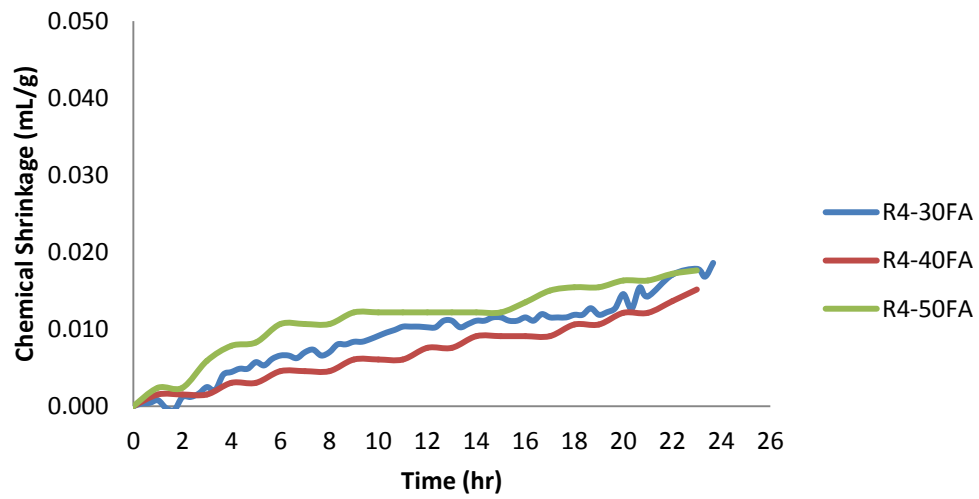
Increasing fly ash and slag both decrease the chemical shrinkage for these tests. The 20% fly ash replacement control mix had approximately 20% reduction in shrinkage from the OPC control, whereas at high volumes of class F fly ash a 40-50% reduction in shrinkage from OPC can be realized.

The 20% slag replacement control mix had approximately 50% reduction in shrinkage from the OPC control, whereas at high volumes of slag an additional benefit is not realized.

Slag and high volume class F fly ash mixes had the overall highest reduction in shrinkage from the OPC control mix. However, for specific gradation and replacement combinations the 20% fly ash mixes with NS could also achieve these reductions in shrinkage at comparable levels.



**Figure 11. Regime 4, slag mixes.**



**Figure 12. Regime 4, fly ash mixes.**

# References

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<sup>1</sup> ASTM, American Society for Testing and Materials, West Conshohocken, PA, 2013.

<sup>2</sup> Ideker, J.H., “Early-Age Behavior of Calcium Aluminate Cement Systems,”  
Dissertation. The University of Texas at Austin, May 2008.

*Preliminary Chemical Shrinkage Analysis of  
Nano Silica Cementitious Binders  
MERL Research Report No. MERL-2013-53*

***Appendix***

**Regime 1****OPC Replaced by NS**

R1- CONTROL  
R1- 1A  
R1- 1B  
R1- 1C  
R1- 1D  
R1- 2A  
R1- 2B  
R1- 2C  
R1- 2D  
R1- 4A  
R1- 4B  
R1- 4C  
R1- 4D

**Regime 2****OPC Replaced by 20% Pozz - Class F + NS**

R2- CONTROL  
R2- 1A  
R2- 1B  
R2- 1C  
R2- 1D  
R2- 2A  
R2- 2B  
R2- 2C  
R2- 2D  
R2- 4A  
R2- 4B  
R2- 4C  
R2- 4D

**Regime 3****OPC Replaced by 20% Slag + NS**

R3- CONTROL  
R3- 1A  
R3- 1B  
R3- 1C  
R3- 1D  
R3- 2A  
R3- 2B  
R3- 2C  
R3- 2D  
R3- 4A  
R3- 4B  
R3- 4C  
R3- 4D

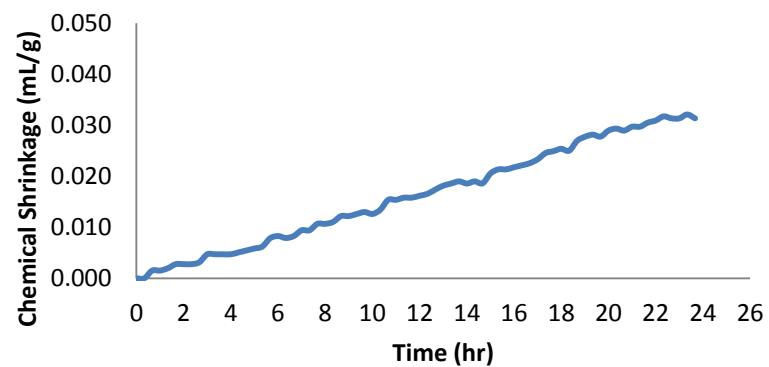
**Regime 4****OPC Replaced by Pozz - Type F or Slag**

R4- 30FA  
R4- 40FA  
R4- 50FA  
R4- 30SLAG  
R4- 40SLAG  
R4- 50SLAG

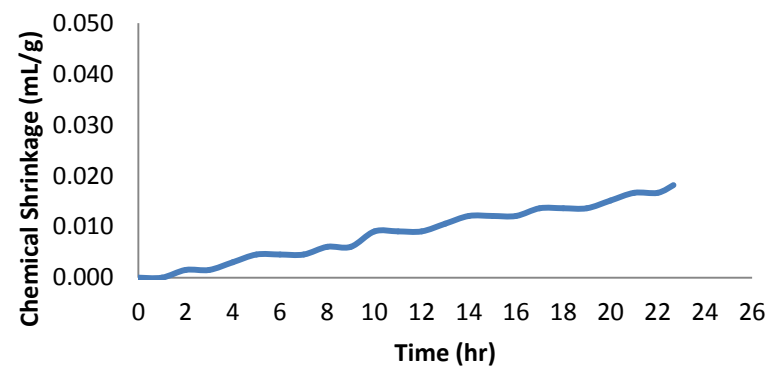
**TESTING RESULTS SUMMARY**

| ASTM C1608 Method A |                                                    | ASTM C1608 Method A |                                                    |
|---------------------|----------------------------------------------------|---------------------|----------------------------------------------------|
| Test ID             | Chemical Shrinkage,<br>(ml of water)/(grams of cm) | Test ID             | Chemical Shrinkage,<br>(ml of water)/(grams of cm) |
| R1-Control          | 0.031                                              | R4-30FA             | 0.019                                              |
| R1-1A               | 0.019                                              | R4-40FA             | 0.015                                              |
| R1-1B               | 0.018                                              | R4-50FA             | 0.018                                              |
| R1-1C               | 0.022                                              | R4-30Slag           | 0.024                                              |
| R1-1D               | 0.026                                              | R4-40Slag           | 0.014                                              |
| R1-2A               | 0.017                                              | R4-50Slag           | 0.017                                              |
| R1-2B               | 0.031                                              |                     |                                                    |
| R1-2C               | 0.029                                              |                     |                                                    |
| R1-2D               | 0.022                                              |                     |                                                    |
| R1-3A               | 0.026                                              |                     |                                                    |
| R1-3B               | 0.020                                              |                     |                                                    |
| R1-3C               | 0.022                                              |                     |                                                    |
| R1-3D               | 0.028                                              |                     |                                                    |
| R2-Control          | 0.026                                              |                     |                                                    |
| R2-1A               | 0.016                                              |                     |                                                    |
| R2-1B               | 0.020                                              |                     |                                                    |
| R2-1C               | 0.019                                              |                     |                                                    |
| R2-1D               | 0.018                                              |                     |                                                    |
| R2-2A               | 0.014                                              |                     |                                                    |
| R2-2B               | 0.016                                              |                     |                                                    |
| R2-2C               | 0.023                                              |                     |                                                    |
| R2-2D               | 0.026                                              |                     |                                                    |
| R2-3A               | 0.016                                              |                     |                                                    |
| R2-3B               | 0.014                                              |                     |                                                    |
| R2-3C               | 0.014                                              |                     |                                                    |
| R2-3D               | 0.020                                              |                     |                                                    |
| R3-Control          | 0.015                                              |                     |                                                    |
| R3-1A               | 0.020                                              |                     |                                                    |
| R3-1B               | 0.020                                              |                     |                                                    |
| R3-1C               | 0.019                                              |                     |                                                    |
| R3-1D               | 0.023                                              |                     |                                                    |
| R3-2A               | 0.017                                              |                     |                                                    |
| R3-2B               | 0.023                                              |                     |                                                    |
| R3-2C               | 0.014                                              |                     |                                                    |
| R3-2C               | 0.012                                              |                     |                                                    |
| R3-2D               | 0.015                                              |                     |                                                    |
| R3-3A               | 0.021                                              |                     |                                                    |
| R3-3B               | 0.023                                              |                     |                                                    |
| R3-3C               | 0.023                                              |                     |                                                    |
| R3-3D               | 0.022                                              |                     |                                                    |

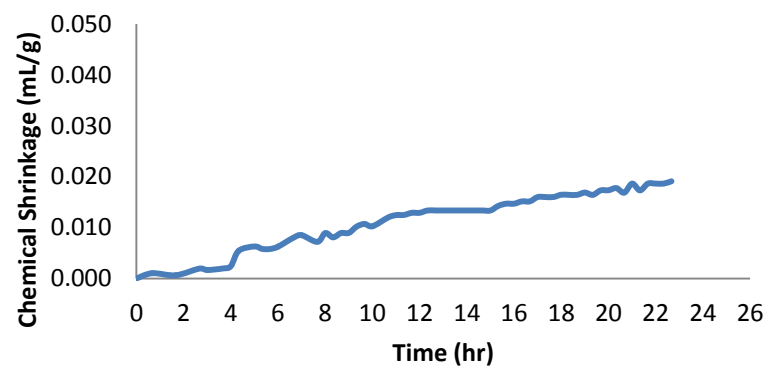
### R1-Control



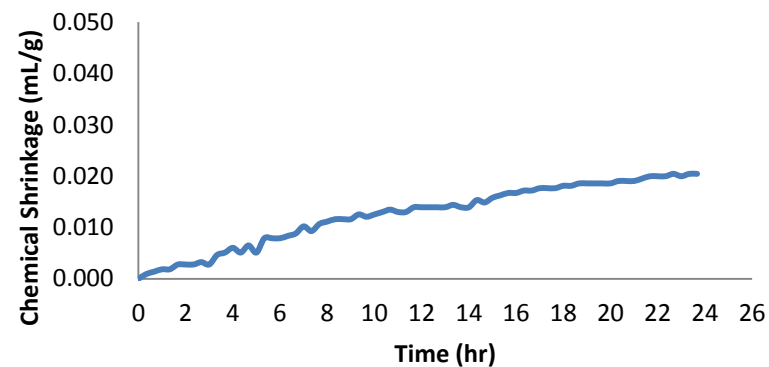
### R1-1B



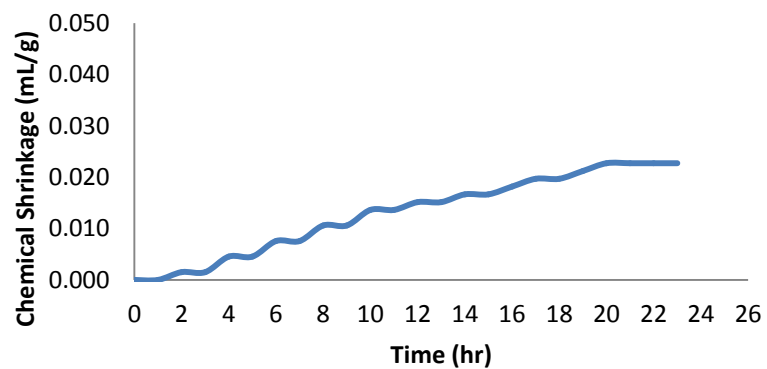
### R1-1A



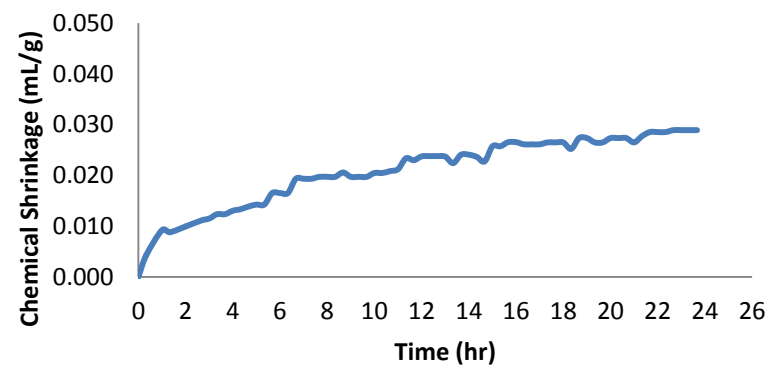
### R1-1C



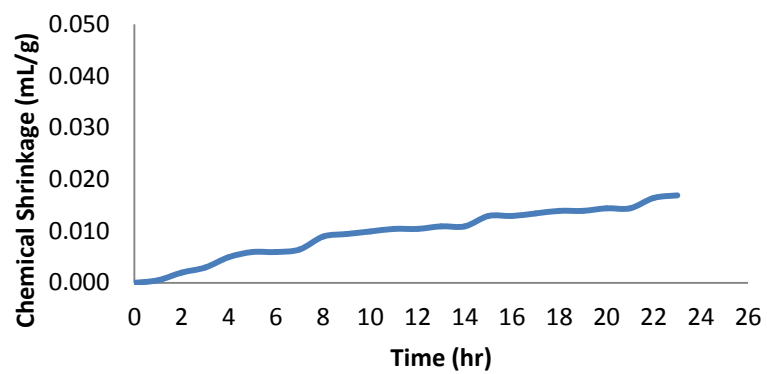
### R1-1D



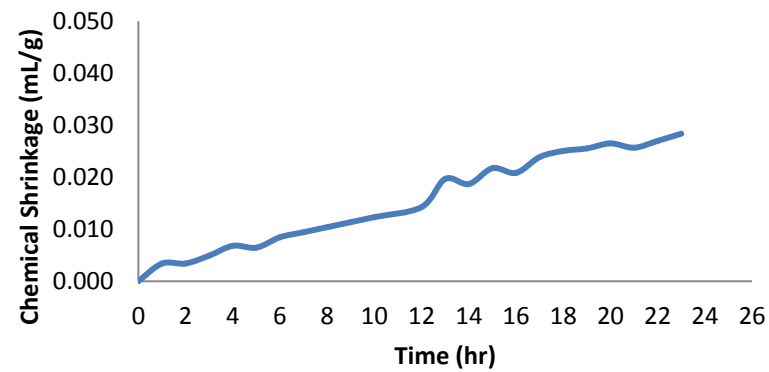
### R1-2B



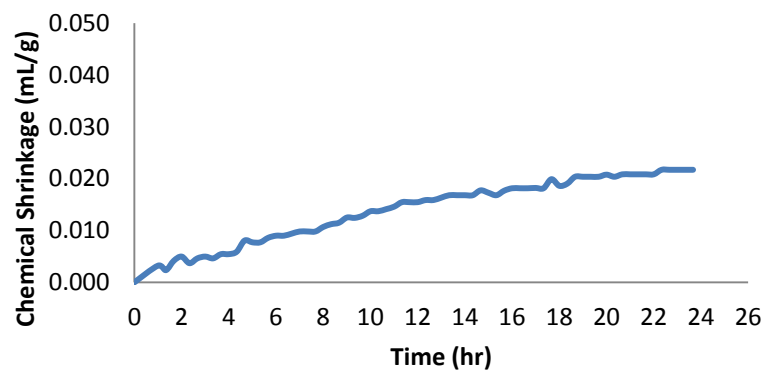
### R1-2A



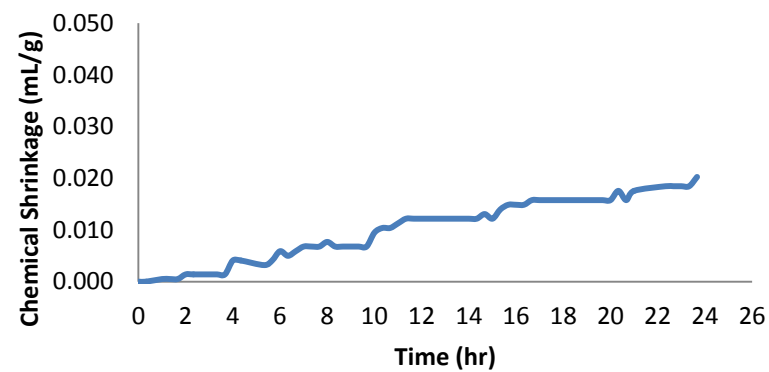
### R1-2C



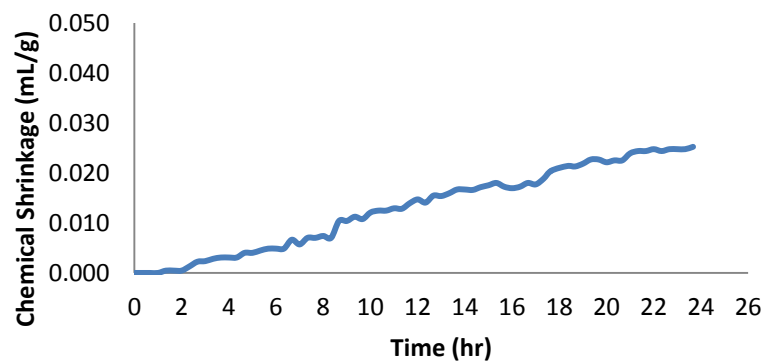
**R1-2D**



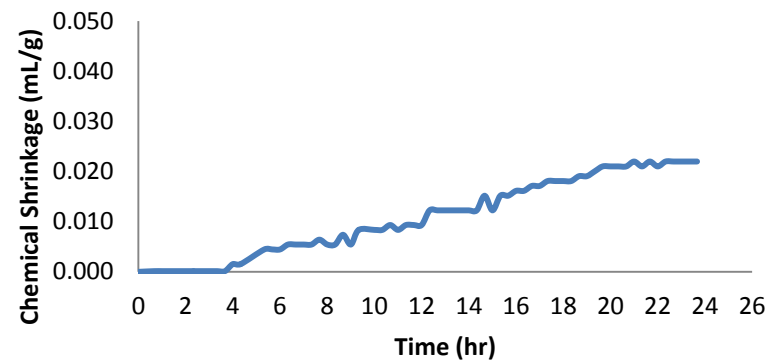
**R1-3B**



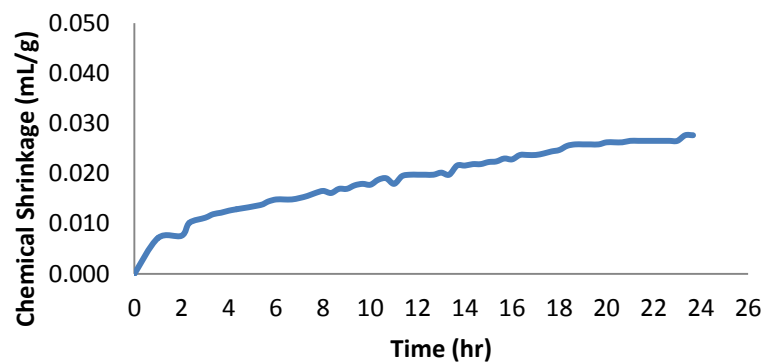
**R1-3A**



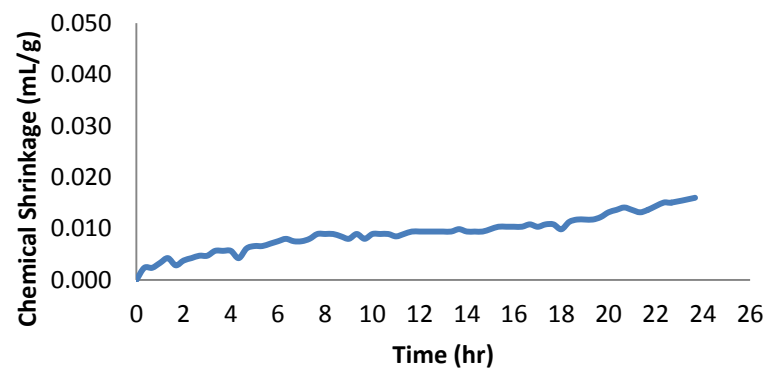
**R1-3C**



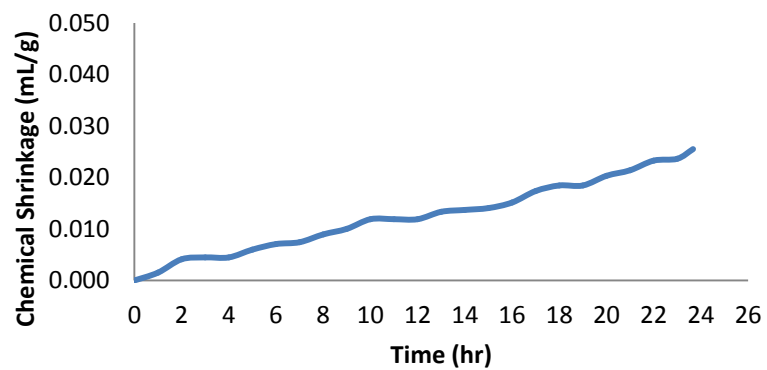
**R1-3D**



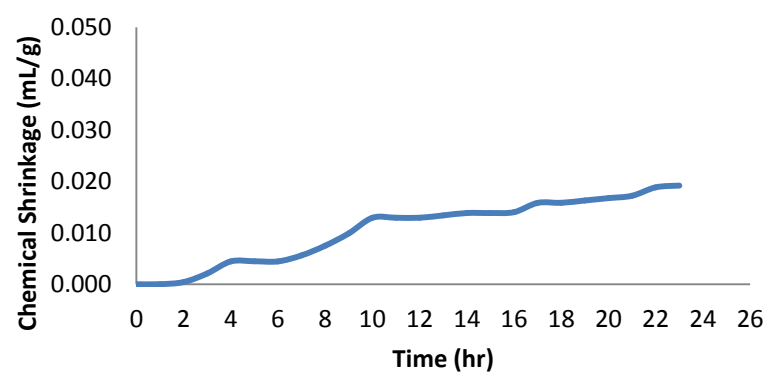
**R2-1A**



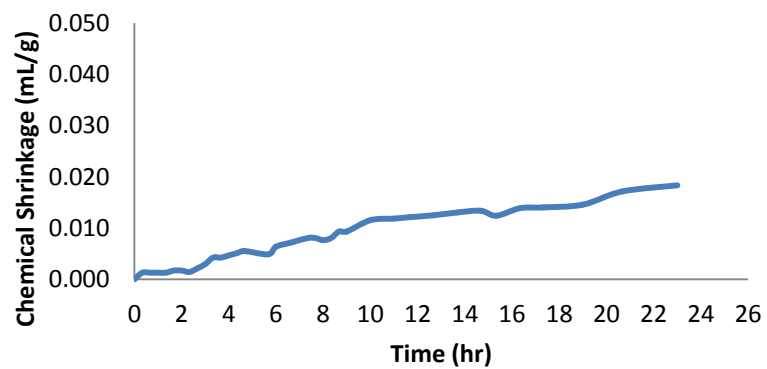
**R2-Control**



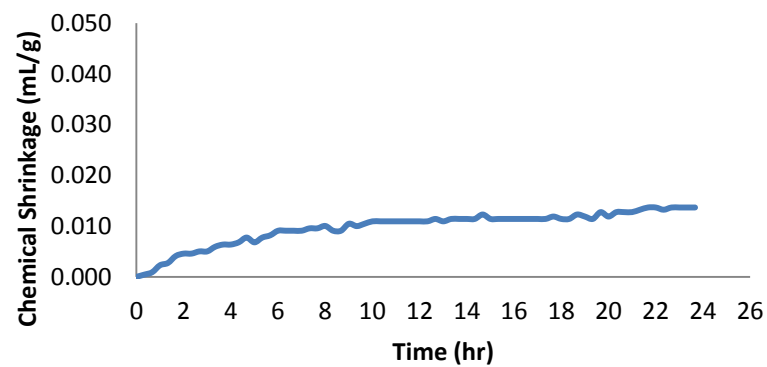
**R2-1B**



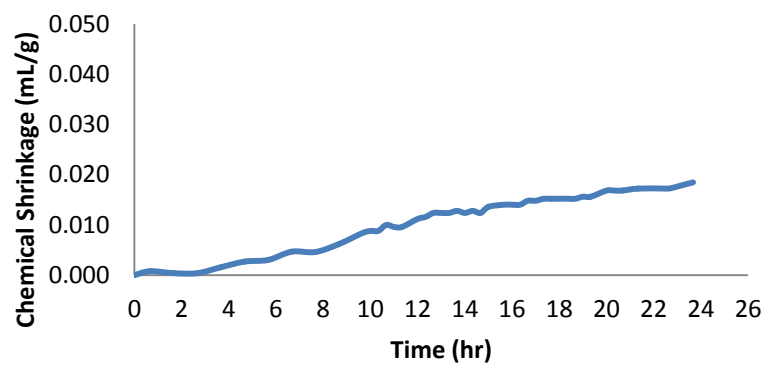
### R2-1C



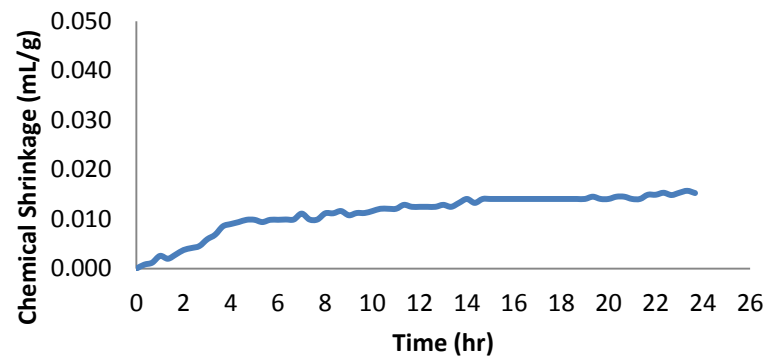
### R2-2A



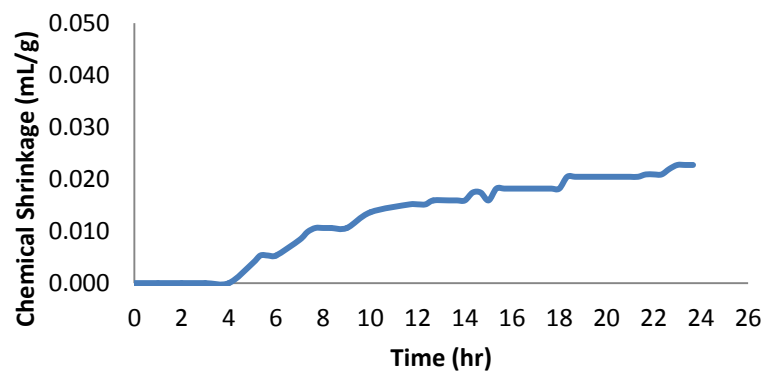
### R2-1D



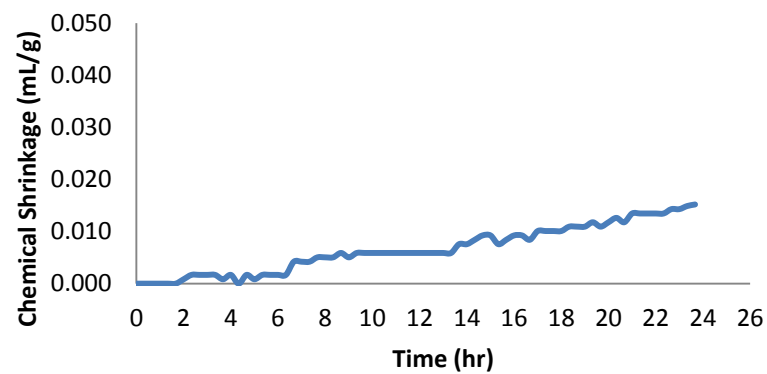
### R2-2B



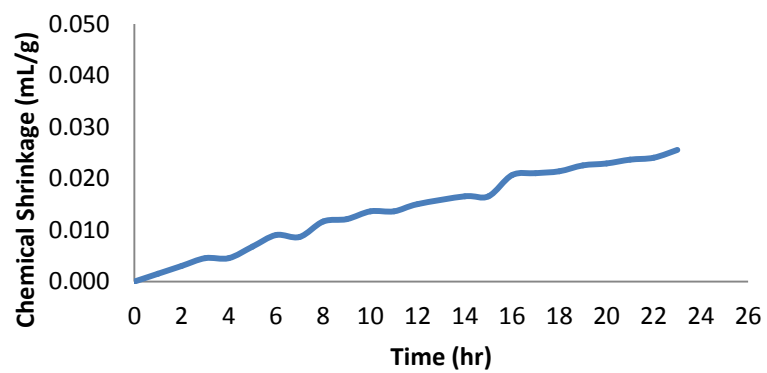
**R2-2C**



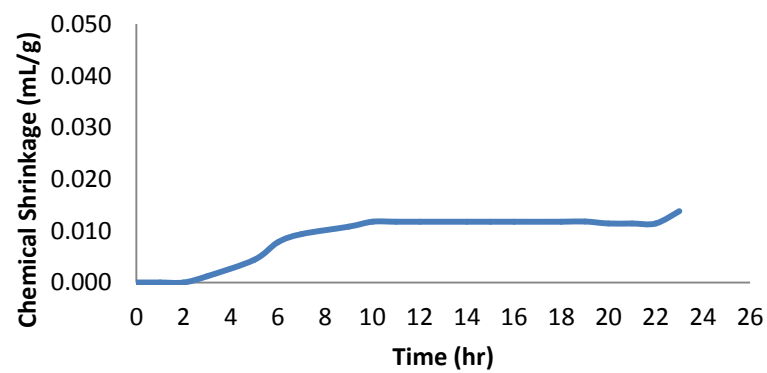
**R2-3A**



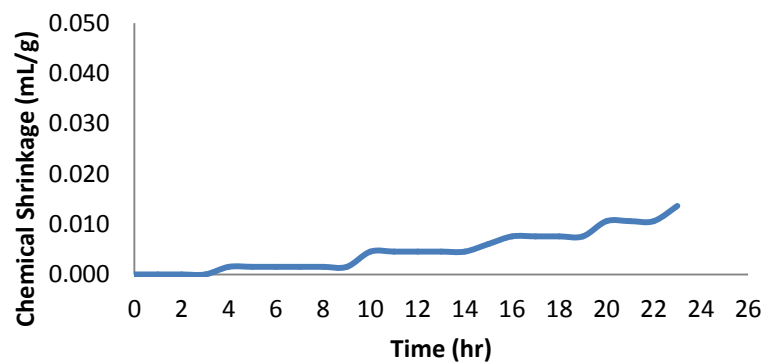
**R2-2D**



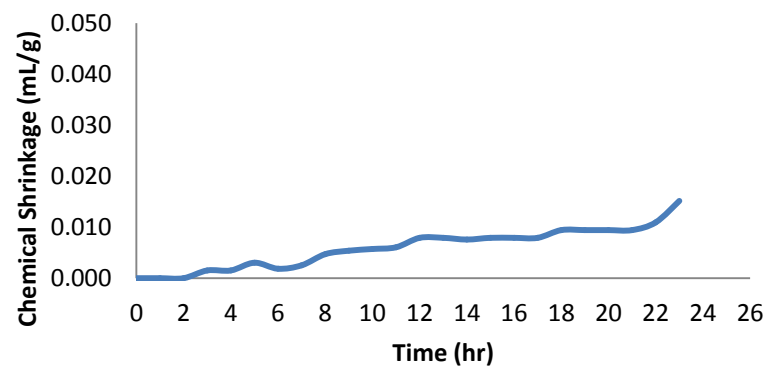
**R2-3B**



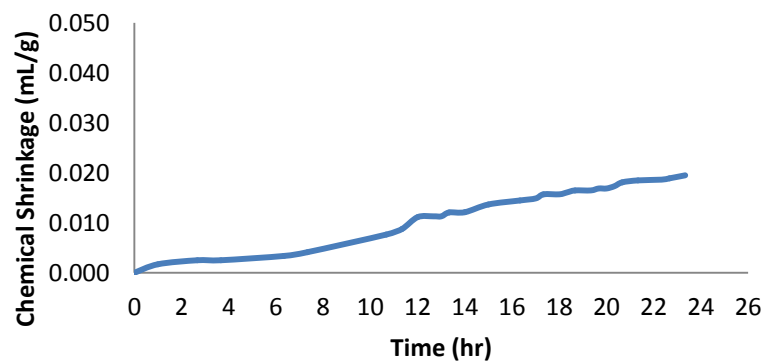
### R2-3C



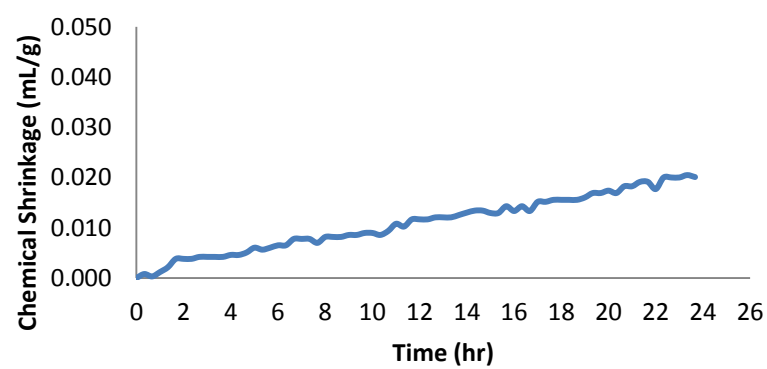
### R3-Control



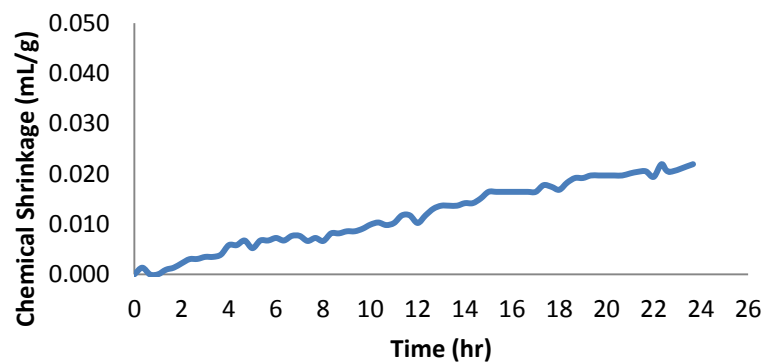
### R2-3D



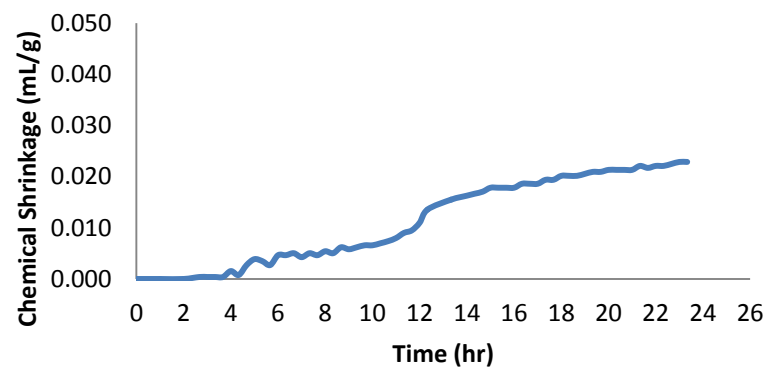
### R3-1A



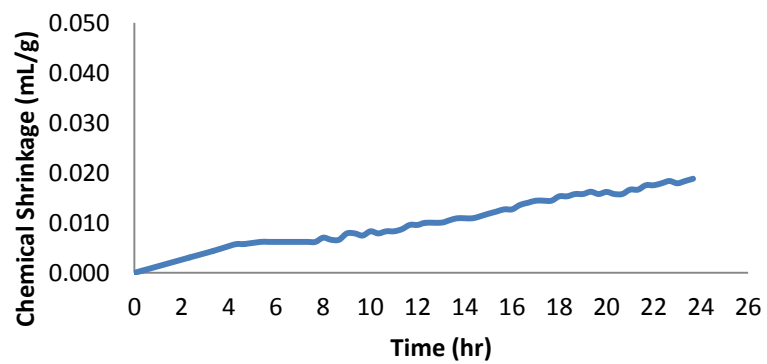
**R3-1B**



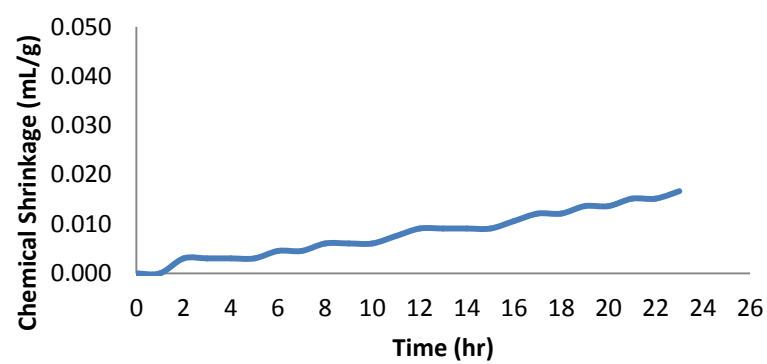
**R3-1D**



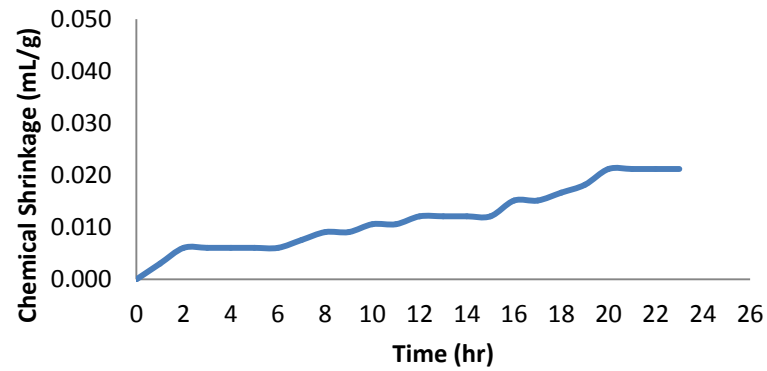
**R3-1C**



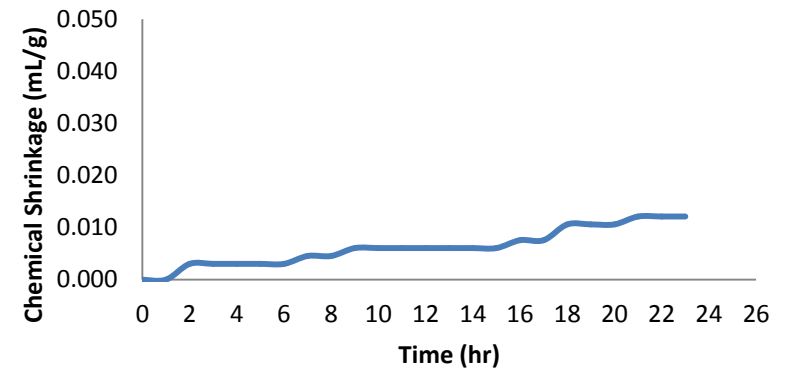
**R3-2A**



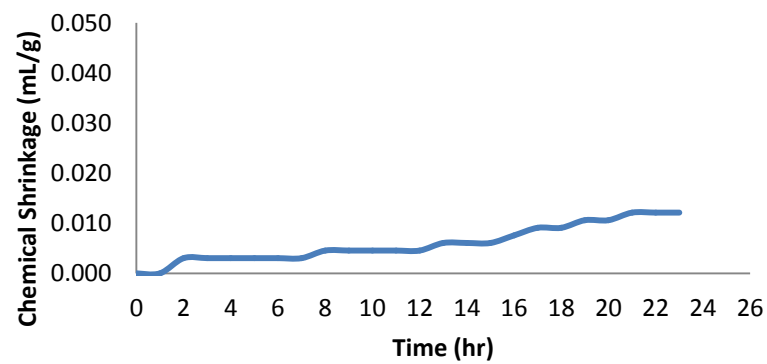
**R3-2B**



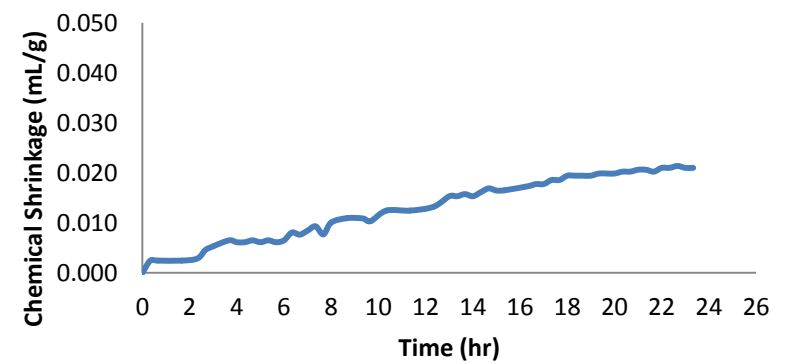
**R3-2D**



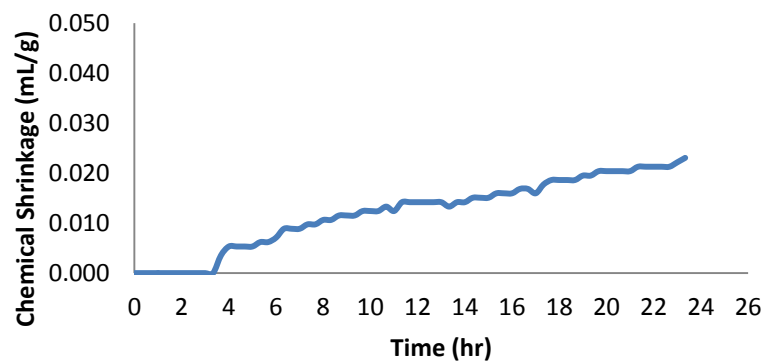
**R3-2C**



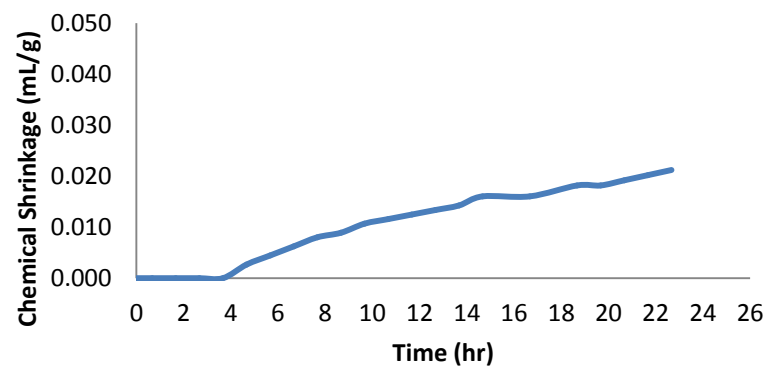
**R3-3A**



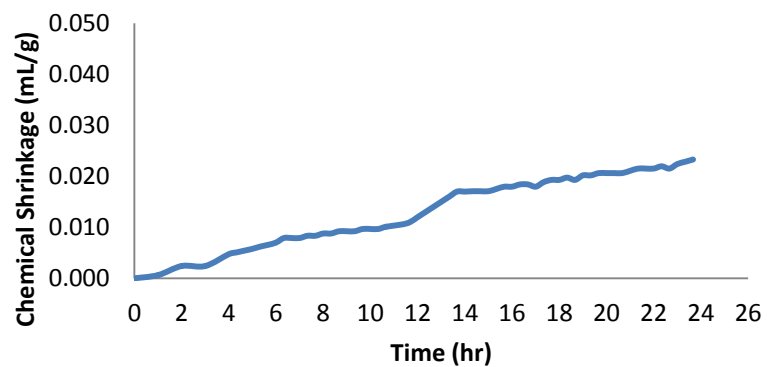
**R3-3B**



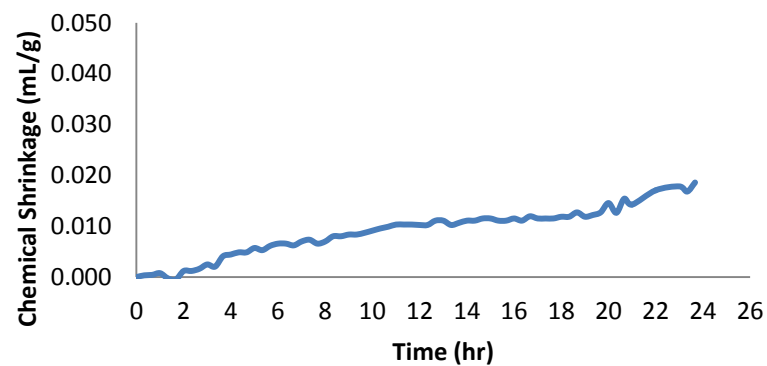
**R3-3D**



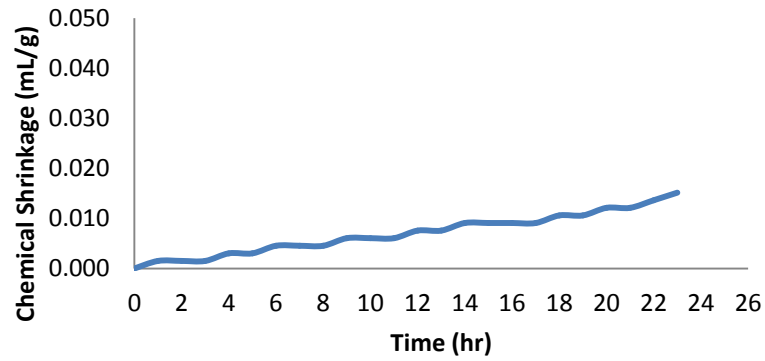
**R3-3C**



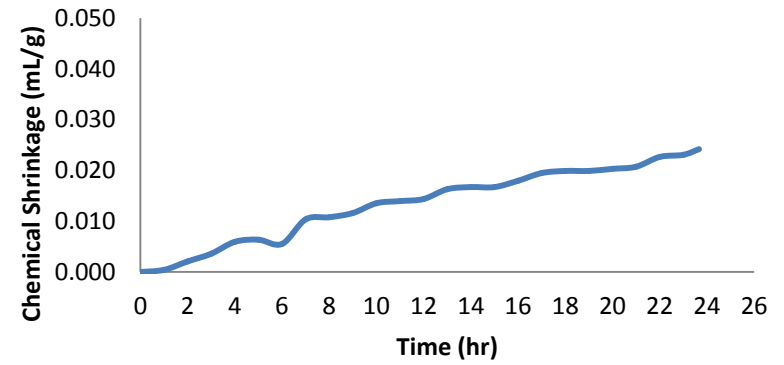
**R4-30FA**



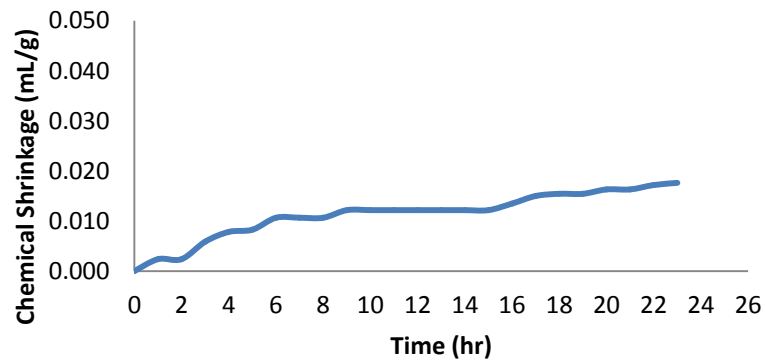
**R4-40FA**



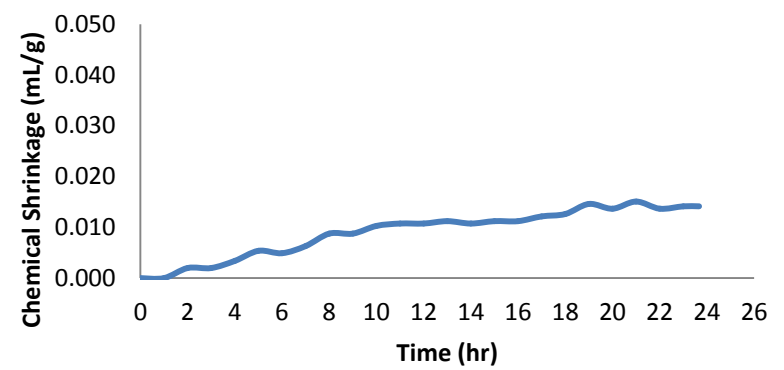
**R4-30Slag**



**R4-50FA**



**R4-40Slag**



## R4-50Slag

