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EFFECT OF CNF ON DRYING SHRINKAGE AND AUTOGENOUS SHRINKAGE OF CEMENT PASTE

By

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B.A. University of Tikrit, 2008

A THESIS

Submitted in Partial Fulfillment of the

Requirements for the Degree of

Master of Science

(in Civil Engineering)

The Graduate School

The University of Maine

December 2017

Advisory Committee:

Eric Landis, Professor of Civil & Environmental Engineering, Advisor

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Thesis Advisor: Prof. Eric Landis

An Abstract of the Thesis Presented

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The researchers in the concrete field have to obtain a concrete with high durability; the goal is to prolong the service life and reduce maintenance costs. Shrinkage cracks due to losing the moisture, or due to self-desiccation is a leading in reducing durability. Cellulose Nano-Fibrils (CNF) is a natural material. It is produced by isolating from a cellulosic material. CNF show high tensile strength, low density, low cost. Furthermore, CNF has ability to absorb the water. These properties give CNF potential to improve the properties and solve the problems of the conventional cement paste, for instance, shrinkage. Different (CNF) ratios were added, to evaluate the effects of CNF on drying shrinkage and autogenous shrinkage of cement paste.

Two cement paste prisms were cast and tested in accordance with ASTM C157 for each batch, with some modifications of the at curing method. The work of this study was organized into two phases. Phase one focused on free shrinkage while phase two focused on autogenous shrinkage.

In phase one, thirty-two batches were prepared. These batches were divided into four groups with water to cement ratio 35%, 40%, 45% and 50 %. Each group was further divided into CNF fraction of 0%, 0.05%, 0.1%, 0.2%, 0.5%, 1%, 1.5%, and 3%. Two cement paste prisms were cast and tested in accordance with ASTM C157 for each batch, with some modifying at curing method

In Phase two, four batches were prepared in the Concrete laboratory. Water to cement ratio was 30% for all the batches. Four different CNF ratios were used to assess the effect of the CNF on the autogenous shrinkage, the ratios of the CNF were 0 as the reference, 0.1%, 0.2% and 0.5%. Two sealed specimens were cast and were tested in according to of ASTM C157 for each batch. The flow table test was tested for all the batches in these phases.

The results of the free shrinkage (phase one) test for each batch were collected up to 90 days. When the w/c % were 40%, 45%, and 50%, the results of these groups at age 28 days showed that, the addition of the CNF with different ratios that led to increasing the free shrinkage, with the exception that at law W/C ratio and law CNF dosage, shrinkage was reduced.

The results for phase two showed that CNF had little or no effect on autogenous shrinkage. However, it must be note that extra water not considered in this study and early age effects not considered in this analy

.

DEDICATION

To those persons, whose they have given me everything, whose they have given me the right to live in this life, who they have fed me the love,

to my dear father and dear mom.

To that person, who she has been supporting and encouraging me all the time,

to the pretty Wife.

To the worthiest thing in my life, to My kids

(Ali and Yusef).

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I would like to thank the following people for their support, without whose help this work would never have been possible: Ethar Alyaseen, Hosain Haddad Kolour (he is the leader of our team), and kavya Shirisha.

I am indebted to The University of Maine and the particularly grateful for the civil engineering department.

Thanks, are for The Writing Center at the University of Maine.

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CHAPTER 1: INTRODUCTION

Concrete is and remains to be one of the most widely used construction products on the planet. due to the ease with which it can be formed into a range of sizes and shapes, its comparative durability, and its low cost. Additionally, contributing to its appeal as a construction material is the prepared availability of its most basic elements: Portland cement, aggregates, water, and admixtures. (Nmai *et al.*, 2017)

For the concrete that uses the cement paste as a binder, the cement paste is the most important part in this concrete. As other materials, cement paste has advantages, disadvantages, and problems. One of the problems of cement paste is shrinkage.

Shrinkage is a reducing of the volume of the objects because of exterior or interior conditions. This phenomenon happens in concrete and cement. Shrinkage is the contraction in concrete with the time, independent of applied loads. It is caused by loss of water through evaporation, hydration of cement and carbonation.

When concrete systems lose their water through evaporation, hydration, and carbonation, change in the volume of the system will occur. If the system is not restrained, the system will be smaller and shrink without any cracks. On the other hand, all the concrete construction and structures, such as the slab pavement, the decks of bridges, and other concrete elements, are restrained, which means tensile stresses will develop. When these stresses are greater than the tensile strength of the concrete, cracks will appear and develop.

Shrinkage causes many different cracks in the concrete surface members, including interior cracks, which all happen at a different age, from an early age to weeks or maybe months later.

These cracks reduce the performance and the durability of the members, increase the maintenance cost, causes the corrosion of the steel reinforcements, and shorten the service life, which all lead to the reduction of the sustainability of the construction.

Researchers study and reduce this phenomenon and try to explain the causes, the mechanisms, and the types.

All types of shrinkage happen in cement paste not in aggregate. However, the size, ratio, type, and modulus of elasticity of aggregate influence the magnitude of the shrinkage. Furthermore, that gives us an opportunity to see the effect of the CNF clearly due to the distribution of CNFs at the cement paste might be more homogenous than the concrete.

Cellulose Nano Fibril (CNF) is a natural material. It is Produced by isolating from a cellulosic material. Cellulose nanomaterials show high tensile strength, low density, low cost. Furthermore, the ability to absorb the water. Thus, these properties are motivating to use this material to improve the properties and solve the problems of the conventional cement paste, for instance, shrinkage. All the CNF (Cellulose nanofibril) had been used in the study was produced by Process Development Center at the University of Maine in slurry form with 3% concentration.

The objective of this study documented in this thesis was to examine the effect of small addition of CNF on the shrinkage properties of cement past. The hypotheses are that small addition of CNF will hold the water inside the cement paste structure and reducing self-

desiccation and thus, shrinkage. This hypothesis was tested in two phases; free shrinkage and autogenous shrinkage. The experimental program and results are detailed in the chapter that flow.

CHAPTER 2: BACKGROUND

Shrinkage in concrete

The shrinkage is a reduction in the volume of materials. Generally, shrinkage in concrete happened in cement paste. This phenomenon is happened because of losing the water from cement paste this phenomenon is happened because of losing the water by evaporation, hydration of cement and carbonation(Neville and Brooks, 2010). Shrinkage in cement paste happens in two stage fresh stage and hardened stage. Each type has specific reason and stage for occurring.

Types of shrinkage

1) Plastic shrinkage.

2) Chemical shrinkage.

3) Autogenous shrinkage.

4) Drying shrinkage.

5) Carbonation shrinkage

Plastic shrinkage

During the first few hours after mixing the concrete when concrete still behaves as a formable mass the plastic shrinkage commonly occurs due to loss the water from concrete surface by evaporation. That evaporation leads to cracks take place on the surface. Exposed concrete surface to undesirable, surrounding conditions (strong wind, high temperature or

low humidity) are common causes of marked plastic shrinkage in concrete and cement paste.(Mechtcherine, Reinhardt and (Eds.), 2012).

The mechanism of the plastic shrinkage

The plastic shrinkage cracks appear in fresh stage (before final sitting happen) when the evaporation ratio of water from the surface is greater than the bleeding water from the interior layer to the surface of the concrete or cement paste. This type of evaporation causes to reduce the volume. The volume reducing give rise to a shrinkage force to form at the surface. because of the low strength and stiffness of the concrete or cement paste in this stage, The cracks will appear,(Arbor, 2000)

Chemical shrinkage

Immediately, when the water adds to the cement paste or concrete the reaction will start. that means the chemical shrinkage is happening. This kind of shrinkage happens by hydration products. the compounds of cement as (C_3S, C_2S, C_3A, and C_4AF), that means chemical shrinkage will be different when the ratio of these compounds will be different in the cement. Chemical shrinkage is formed internally, inside cement paste skeleton. that means is an internal reduction. When that reduction will appear as an external reduction in cement paste skeleton, that will be called Autogenous shrinkage.

Autogenous shrinkage

Autogenous shrinkage is the other type of shrinkage, this type of shrinkage which occurs without any change in cement paste volume due to losing water or moisture by evaporation or external condition as temperature variation. Therefore, autogenous shrinkage is referred

to as self-desiccation shrinkage. This kind of shrinkage can happen in both stages of cement paste:

- fresh stage (before sitting the cement paste), in that time, the autogenous shrinkage is considered part of chemical shrinkage(Sant, Lura and Weiss, 2006).
- The hardened stage (after cement paste final sitting).

This type of shrinkage will occur, when a low water, cement ratio is used. The low water, cement ratio is less than 40 %. Others said that the effect of autogenous shrinkage appears in high-performance concrete(HPC) and ultra-high-performance concrete(UHPC).(Wu *et al.*, 2017).

Mechanism of autogenous shrinkage

The most accepted theory that explains autogenous shrinkage after the hardened stage is the capillary tension theory. The cement paste structure has a different kind of pores like gel pores. In the early age of cement paste, these pores will be formed, and they are filled with water. With time, all the compounds of cement want the water to complete the hydration processing. That means the water inside the gel pores will be consumed, Due to this case, the internal relative humidity will reduce. This phenomenon will make kind of unstable state inside the cement paste structure. The pores will transfer from saturated state to unsaturated state. that lead, to subject the gel pores to internal pressure, trying to return to equilibrium case. Finally, the volume reduction occurs called Autogenous shrinkage (Mechtcherine, Reinhardt and (Eds.), 2012)

In ordinary concrete or cement paste or when water to cement ratio more than 40%, the autogenous shrinkage does not appear (Mechtcherine, Reinhardt and (Eds.), 2012). However, if that happens, the researchers consider this type of a shrinkage as part of a

drying shrinkage (i.e. its influence is not recognized as a different type of shrinkage). On the other hand, the Autogenous shrinkage shows a distinct influence in researchers works when they work with high-performance concrete or ultra-high-performance concrete(Mechtcherine, Reinhardt and (Eds.), 2012).

In this study, the Autogenous shrinkage will be tested and measured.

Drying shrinkage

Drying shrinkage is the shrinkage that occurs attributable to the water evaporation from cement paste after final setting occurs when the unsaturated air is found in the concrete (i.e. The relative humidity less than 100%). Drying shrinkage is more pronounced than Autogenous shrinkage in normal concrete and more pronounced in normal and high water-cement ratio.

Part of drying shrinkage is reversible and the other part is irreversible if the concrete is subjected to a fully saturated condition. The irreversible part happens because new additional bonds have formed in the cement paste skeleton when the interlayer water had been evaporated, and that formation cannot be changed or reversible to the normal shape even extra water ratio was provided.

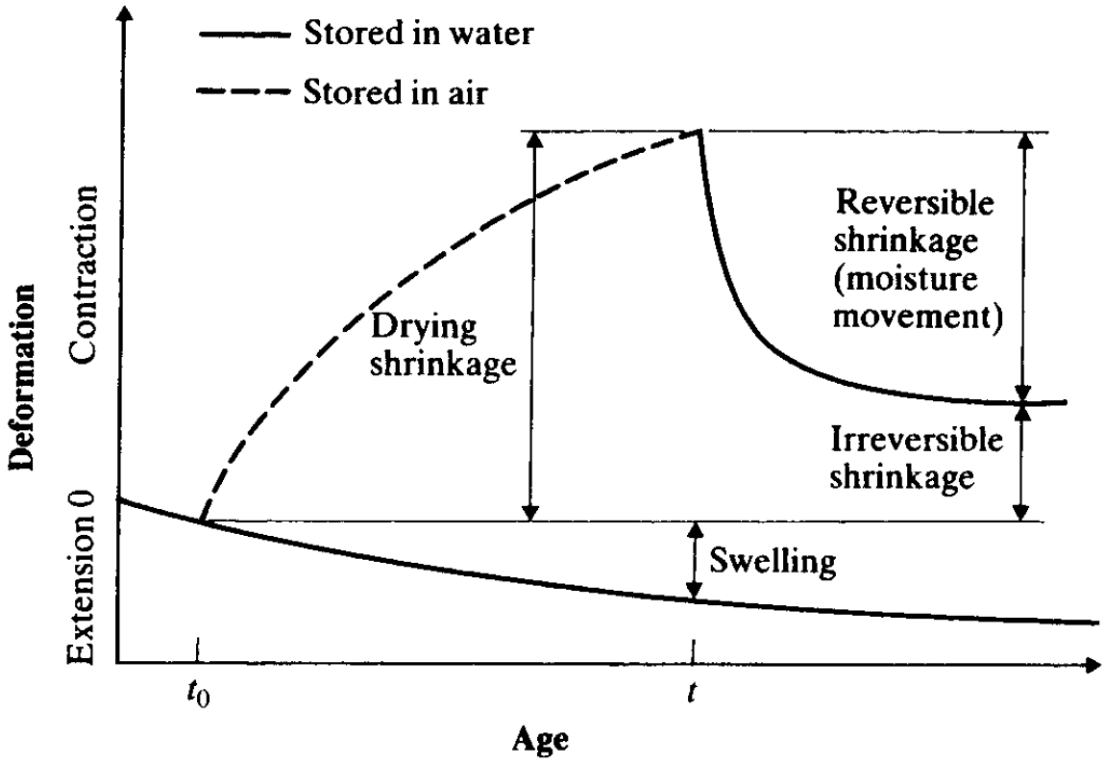

Figure 1. This graph shows the deformation Vs time, concrete has dried, then was re-saturated in water, (Neville and Brooks, 2010).

Mechanism of drying shrinkage

Drying shrinkage is considered the largest type among other types that are existed in ordinary concrete and cement paste, thus, researchers have great interest is studying this type of the shrinkage. Three mechanisms explain the moisture loss and how the volume changes. These three mechanisms are; capillary stress, disjoining pressure, and surface free energy. When the relative humidity is at the range (45 to 95) %, the capillary stress starts to develop when the meniscus is formed in the water inside the cement paste, the meniscuses are under hydrostatic tension and the curved surface is created. As a result, a

corresponding compression is created on the cement paste particles, trying to reduce the volume of the pores.(Deshpande, Darwin, and Browning, 2007).

Carbonation shrinkage

This type of shrinkage happens as a result of the carbonation process when the chemical compound in cement paste as Calcium Hydroxide Ca(OH)2 reacts with CO_2 (carbon dioxide in the atmosphere) this reaction will give out Calcium Carbonate $CaCO_3$ + H_2o. this reaction was called Carbonation shrinkage (Neville and Brooks, 2010).

Effect of superabsorbent polymers on autogenous and drying shrinkage in cement paste

Effect of superabsorbent polymer on shrinkage in cement paste is mentioned here because both superabsorbent polymers SAP and the CNF have similar behavior, they both absorb water. During the study on the effect of SAP on Autogenous shrinkage, it was found that the cement pastes had been mixed with the different quantities of the superabsorbent polymer(SAP). A suspension superabsorbent polymers have an average particle size of a diameter greater than 200µm and the isothermal condition has been assumed. The water to cement ratio was 30 %, then after the specimens were measured, they found that adding a small ratio of the superabsorbent polymer(SAP) as between (0.3%to 0.6%), in conjunction with extra water led to reducing the Autogenous shrinkage. Furthermore, adding superabsorbent polymer(SAP) led to expanding the cement paste (Mechtcherine, Reinhardt and (Eds.), 2012). The figure (3) shows the effect of the SAP on cement paste and how the small amount of SAP reduces the autogenous shrinkage.

The drying shrinkage was higher in (HPC) and (UHPC), when (SAP) and extra water is added to them. Whereas, the reference batches (i.e. without SAP and without extra water) showed less drying shrinkage. This variation in drying shrinkage seems to be smaller by using a long time wet curing. (Mechtcherine, Reinhardt and (Eds.), 2012). The figure (2) shows the effect of the SAP on cement paste and how the small amount of SAP reduces the autogenous shrinkage.

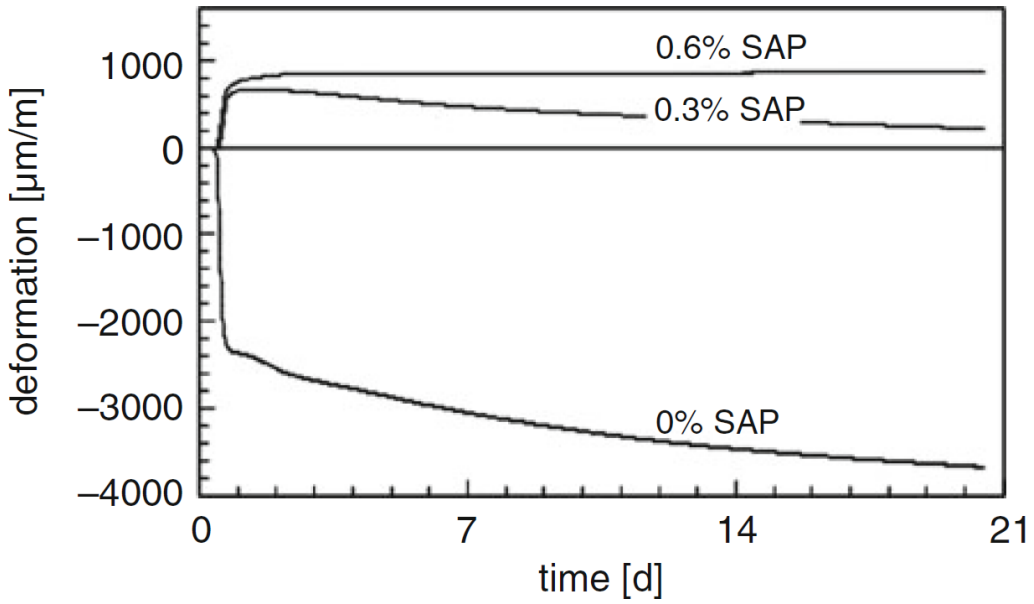

Figure 2.The graph shows the effect of (SAP) on Autogenous Shrinkage for cement pastes. (Mechtcherine, Reinhardt and (Eds.), 2012)

Methods of measuring shrinkage

In concrete structures and bridges, if the concrete is allowed to contract freely, the concrete will not be cracked. Steel reinforcement in the concrete members, fixed supports, fixed connections between the members and differential shrinkage occurs in the concrete members, all these reasons led to restraining the movement then cracks appear (Deshpande,

Darwin, and Browning, 2007). Depend on these situations. The types of measuring and evaluating shrinkage are divided into:

- free shrinkage tests (Unrestrained).
- Restrained test (ring tests)

ASTM standards

ASTM standards have been issued and developed to; measure, evaluate and explain the laboratory methods for various types of shrinkage and these are as follows:

1) ASTM C157/C157M − 08: “Standard Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete”.

In this standard, it was explained how to determine the length changes for specimens were made in the laboratory, after final setting time under specific conditions (ASTM C157, 2016). This standard has been used in the current study.

2) ASTMC490/C490M: "Standard Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete".

This method covers the needs of the device and tools, these are used to prepare samples for the tests. In this standard, Techniques for how to prepare and cure specimens are mentioned, conditions of testing and, procedures for calculating and reporting test results in applicable test methods(ASTM C 490, 2011). Figures (3,4 and 5), present the molds, the length comparator with specimen and reference bar.

Figure 3. The mold that was used in free shrinkage test.

Figure 4. The specimens, Concrete lab.

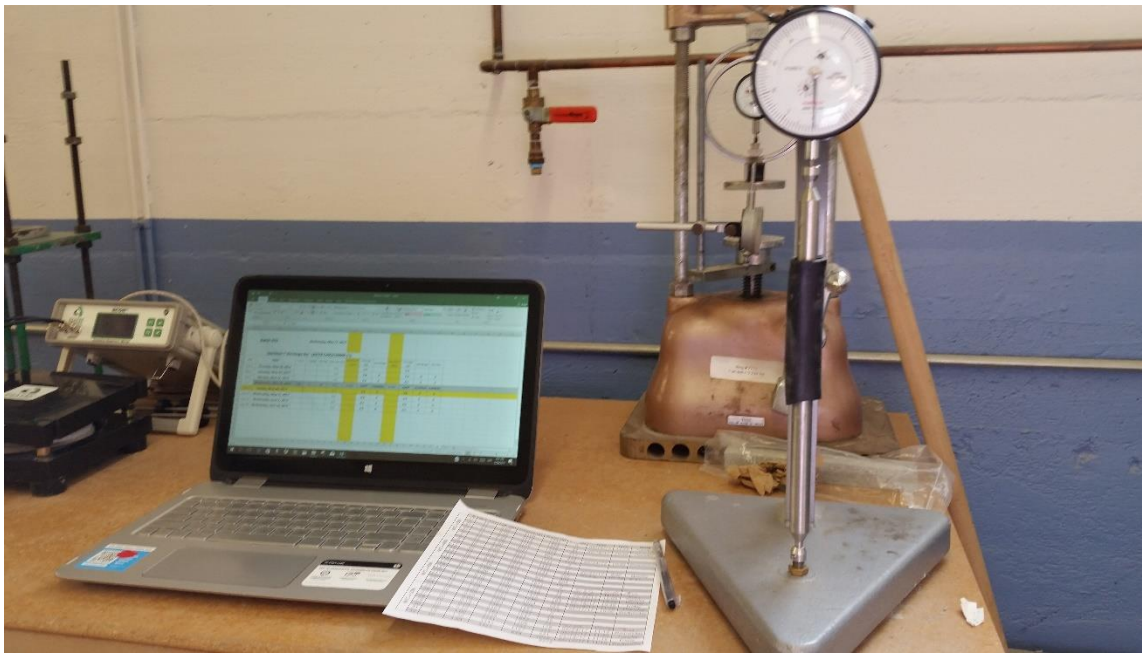

Figure 5. the length comparator with reference bar, Concrete lab.

3) ASTMC1698 − 09 (Reapproved 2014): “Standard Test Method for Autogenous Strain of Cement Paste and Mortar”.

this standard explains how to determine the length changes after final setting time under specific conditions for specimens that have been made in the laboratory.

According to this standard, the change in the length of the specimens is the Autogenous shrinkage after final setting time. In this test, the sealed specimens are used (a corrugated mold). The figure (6) presents the corrugated PE-mold and the dilatometer bench, two especially end plugs, a reference bar and the test specimen(Mass *et al.*, 2017).

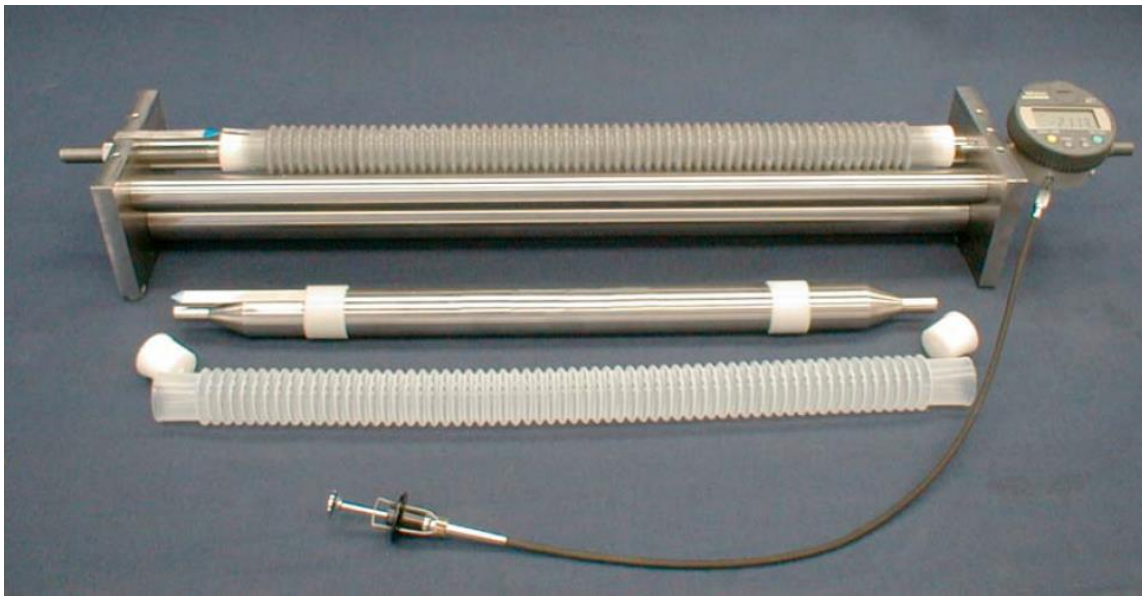

Figure 6. the corrugated PE-mold and the dilatometer bench, two especially end plugs, a reference bar and the test specimen(Mass et al., 2017)

4) ASTMC1608 – 12: “Standard Test Method for Chemical Shrinkage of Hydraulic Cement Paste”

The method for measuring and evaluating this type of shrinkage (the chemical shrinkage) was displayed in this standard. The figure below presents the illustration of Monitoring Chemical Shrinkage of Hydrating Cement Paste (Cabinets, Rooms and Statements, 2017).

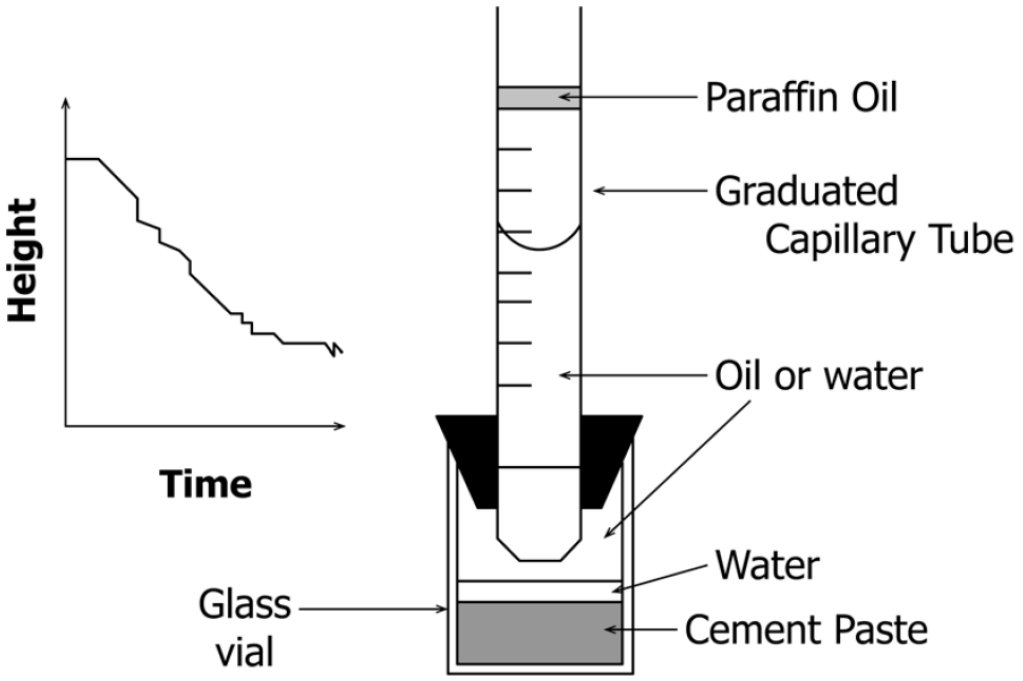

Figure 7. Chemical shrinkage measurement method. (Cabinets, Rooms and Statements, 2017)

CNF (Cellulose Nano Fibril)

At the present, Different types of fibers are used to enhance the properties of concrete or mortar or cement paste, such compressive strength, tensile strength, toughness, fatigue and other properties. The fibers as carbon fiber steel and polymer fibers was used, (Onuaguluchi, Panesar and Sain, 2014). Recently, there are some serious environmental problems that why the researchers and institutes are looking to find the green materials to enhance and develop the construction. In this study, CNF (Cellulose Nano Fibril) was used. CNF is a natural material. It is produced by isolating from a cellulosic material. Cellulose nanomaterials show high tensile strength, low density, low cost. Furthermore, the ability

to absorb the water. Thus, these properties are motivating to use this material to improve the properties and solve the problems of the conventional cement paste, for instance, shrinkage. All the CNF (Cellulose nanofibril) had been used in the study was produced by the Process Development Center at the University of Maine in slurry form with 3% concentration.

The objective of this study documented in this thesis was to examine the effect of a small addition of CNF on the shrinkage properties of cement past. The hypotheses are that small addition of CNF will hold the water inside the cement paste structure and reduce self-desiccation and thus, shrinkage. This hypothesis was tested in two phases; free shrinkage and autogenous shrinkage

CHAPTER 3: EXPERIMENTAL WORK

Objectives

Generally, this project was divided into two phases. The objectives of these works were to study the effects of adding different ratios of CNF on the drying and autogenous shrinkage in cement paste. Phase I focused on free shrinkage and phase II focused on autogenous shrinkage. For evaluation of the drying shrinkage and autogenous shrinkage, the free shrinkage test was used. Two shrinkage specimens were casted and tested for each batch in each phase. The methodology for each phase will be explained individually.

Phase 1: Free shrinkage

All the experimental work, such as mixing, casting, curing and testing were done in Civil and Environmental Engineering Departments' laboratories at the University of Maine, Orono, Maine. For this work, A series of cement paste mixtures were tested. In total, four test groups were prepared. The group (I) consisted of eight batches, and all the batches were done with the same water to cement ratio (35% W/C), but eight different ratios of cellulose nanofibrils (CNF) were used (0%, 0.05%, 0.1%, 0.2%, 0.5%, 1%, 1.5%, and 3%) from volume of the cement. The group (II) consisted of eight batches, all the batches were done with the same water to cement ratio (40% W/C), but eight different ratios of cellulose nanofibrils (CNF) were used (0%, 0.05%, 0.1%, 0.2%, 0.5%, 1%, 1.5%, and 3%) from volume of the cement. The group (III) consisted of eight batches, all the batches were done with the same water to cement ratio (45% W/C), but eight different ratios of cellulose nanofibrils (CNF) were used (0%, 0.05%, 0.1%, 0.2%, 0.5%, 1%, 1.5%, and 3%) from

volume of the cement. The group (IV) consisted of eight batches, all the batches were done with the same water to cement ratio (50% W/C), but eight different ratios of cellulose nanofibrils (CNF) were used (0%, 0.05%, 0.1%, 0.2%, 0.5%, 1%, 1.5%, and 3%) from volume of the cement. All the batches that have 0% of the CNF were considered references.

The Free shrinkage test was assumed as two types of shrinkage; Autogenous shrinkage, and dry shrinkage

Tests and the number of specimens

Group of specimens were cast and were tested for each batch, in this work. Each test had the same number of specimens and the label in each batch. The table (1), shows the details of the types of the tests that were done in this work, in addition to the number of specimens and the age of measurements.

Table 1. The types of the tests, the number of specimens and the age of measurements for each batch.

Test type	No. of specimens	measurement at age(days)	Specimen label
Compressive Cube	4	7 & 28	3,4(7days) 5,6(28days)
Shrinkage Bar	2	1,3,5,7,11,14, 2 28,90	7,8
Early Age Shrinkage (16 Batches)	1	Mixing Day	15
Notched Beam	2	28	9,10
Flow Table	1	Mixing Day	11
Rheometer (Ammeter)	1	Mixing Day	-----
Electrical Resistivity	2	1,2,3,5,7,11,1 4,21 & 28	1,2
TGA (10 Batches)	3	7 ,14 , 28	12,13,14

Materials

1) Cement:

QUIKRETE Portland Cement Type I/II (94 lb. bag) was used in all the batches.

2) Water:

Tap water from the lab was used.

3) Cellulose nanofibrils:

Cellulose nanofibrils are produced by Process Development Center (PDC) at University of Maine. The ratio of the solid was CNF=3% in this study. The concentration of the CNF was calculated by taking a sample of the CNF solution, drying it in the oven at 100 C and measuring the weight of the sample after drying. The results showed that the concentration of the CNF is 3% and this value confirms the value that was supplied by the Process Development Center.

Cement paste mix design

Excel was used to calculate the quantities of all the materials that entering and required for each batch. These materials are:

1) Portland cement.

2) The tap water (lab water).

3) The CNF solution.

The information that was inserted into the Excel spreadsheet was the density of the CNF, the density of the cement, the density of water, the ratio of solid CNF to the cement (the

Volume ratio), water to the cement ratio (the Weight ratio), a batch volume and the waste ratio.

Assumptions

There are many input data that are the same in each batch of each program such as:

- The ratio of sold CNF=3% in this study. The concentration of the CNF was calculated by taking a sample of the CNF solution and drying it in the oven at 100 C, measuring the weight of the sample after drying and calculating the CNF concentration in the solution. The results showed that the concentration of the CNF is 3% and its value is corresponding to the value that was supplied by the source (the process development center).
- The density of the CNF=1g/cc.
- The density of the cement=3.15g/cc.
- The density of water =1g/cc.
- All the water in the CNF solution was considered as a free water. In other words, the water in the CNF solution was part of the water that was calculated from the water to cement ratio.
- The batch volume(cc) plus the waste ratio=2750 cc for all batches. This value was calculated by calculating the volume of all samples for each test, the percentage of the waste was considered 25% of the total volume
- The ratio of solid CNF to the cement is a volume ratio. These values varied and each batch has specific values.

❖ The ratio of the water to the cement is a weight ratio. These values varied and each batch has specific values.

Cement Paste Mix Design

Input 1: Assumptions

CNF Density (g/cc)	CNF Solids (%)	Cement Density (g/cc)	Water Density (g/cc)
1	3	3.15	1

Input 2: Batch (Cement Paste) Specifications

CNF Solids (vol %)	W/C (%)	Batch Volume (cc)	Waste (%)
0	40	717	0

Output: Weights (gr)

Cement (gr)	Water (gr)	CNF Gel (gr)
999	400	0

Figure 8. The Excel sheet that was used to calculate the quantities of the materials for each batch. (from work documents)

Table 2. Ratios and weights of the materials in each batch.

	#	W/C (%)	CNF (%)	Cement (g)	Water (g)	CNF (g)
W/C = 35%	1*	35	0.00	4120	1442	0
	2	35	0.05	4119	1421	22
	3*	35	0.10	4118	1399	44
	4*	35	0.20	4116	1356	87
	5*	35	0.50	4110	1228	217
	6*	35	1.00	4101	1014	434
	7	35	1.50	4091	802	649
	8	35	3.00	4062	171	1290
W/C = 40%	9	40	0.00	3833	1533	0
	10	40	0.05	3832	1513	20
	11	40	0.10	3831	1493	41
	12	40	0.20	3830	1453	81
	13	40	0.50	3825	1334	202
	14	40	1.00	3816	1135	404
	15	40	1.50	3808	937	604
	16	40	3.00	3783	348	1201
W/C = 45%	17*	45	0.00	3583	1612	0
	18	45	0.05	3583	1594	19
	19*	45	0.10	3582	1575	38
	20*	45	0.20	3580	1538	76
	21*	45	0.50	3576	1426	189
	22*	45	1.00	3568	1240	378
	23	45	1.50	3561	1054	565
	24	45	3.00	3539	503	1124
W/C = 50%	25	50	0.00	3364	1682	0
	26	50	0.05	3363	1664	18
	27	50	0.10	3363	1647	36
	28	50	0.20	3361	1612	71
	29	50	0.50	3358	1506	178
	30	50	1.00	3351	1332	355
	31	50	1.50	3345	1157	531

Note: The CNF weight that looks in the table is the weight of the CNF solution (water +CNF particles).

Mixing procedure

For each batch, a dry mixer (KitchenAid (5.68 liters)) was used. For the batches without the CNF, the water was placed in the mixer bowl and after that the cement was added.

For batches with the CNF: after the weight of water and CNF were taken, they were both put together in special bowl for another mixer to mix the CNF and water for 3 minutes at 95 RPM to ensure that the dispersion of the particles of CNF in the water will occur. The mix of Water and CNF were poured in a mixing bowl, followed by adding the weighted cement. Then, the following mixing procedure was started and monitored: two minutes at speed No.1, 15 seconds rest, one minute at speed No. 2, 15 seconds rest, one minute at speed No.3, 15 seconds rest, and one minute at speed No.4. Finally, the cement paste would be ready for casting and testing. Before the mixing procedure, all molds were prepared, cleaned, oiled and were put on the vibration table.

Figure 9. KitchenAid 6 quarts (5.68 liters), Concrete lab.

Flow Table test

A flow table test was done for each batch directly after finishing the mix of the batches in the mixer. The ASTM C1437_15 titled the "standard test method for flow of hydraulic cement mortar" was used to calculate the workability. The flow table that was used is the same of the flow table that was mentioned in ASTM C230/C230M_14 titled "Standard Specification for Flow Table for Use in Tests of Hydraulic Cement".

Steps of the test

The flow table was prepared and positioned to be clean and dry, then the flow table mold was placed on the center of the flow table. A layer of the cement paste about half of the height of the mold was put in the mold. The cement paste was tamped 20 times uniformly with a steel rod over the cross-section of the layer to get a uniform filling of the mold, and then the mold was filled totally by the cement paste, and the tamping step was repeated. The surface of the cement paste was leveled with the top edge of the mold, using a straight edge blade. The top surface of the table was cleaned. The mold was taken off immediately, the table was dropped 25 times in about 20 seconds. Finally, four diameters were measured and recorded to the nearest millimeter.

Calculation

The flow was calculated by finding the average of the four diameter readings in millimeters, subtract the inside base diameter of the mold in millimeters (100mm), dividing by the inside base diameter of the mold in millimeters (100mm) and then multiplying by 100%.

Figure (10) shows the steps of the test and also all the results will be presented in chapter 3 in tables and graphs.

A

B

C

D

E

F

Figure 10. Steps of flow table test.

Preparing and casting the specimens

After mixing the mixture and after the flow table sample was taken, the cement paste mixture was ready for casting free shrinkage molds. Two cold-rolled steel molds of dimensions 1" x 1" x 11.25" were used for molding the specimens. These molds were produced by the Humboldt Manufacturing Company. Two specimens were prepared for each batch.

ASTM C157/C157M-08 was used for casting the specimens. The cement paste was placed in the mold in two layers. After placing the first layer, the cement paste was filled under the gage studs by using a straight edge blade. The first layer was then vibrated using the vibrating table. The vibration time and frequency varied according to the stiffness of the mixture (the workability of the batch). The first layer was vibrated until flat and separated uniformly, usually for 30 to 45 seconds. If the cement paste was too stiff to vibrate, the molds were compacted and filled by hand. The second layer of cement paste was then placed, and the cement paste was placed in the corners of the molds with a straight edge blade. The second layer was also vibrated. The surplus cement paste was then removed and the cement paste surface was leveled. The exterior edges of the molds were then cleaned to remove the extra cement paste. Figure (11) shows the mold on the vibrating table.

Figure 11. Two cold-rolled steel molds of dimensions 1" x 1" x 11.25"

Curing

Directly after casting, vibrating and cleaning the molds, all the molds were put in suitable zipper bags to ensure that no extra moisture goes in or out of the specimens during the curing time. Three or Four (3 × 6 in.) concrete cylinders were then placed in the zipper bags around the mold to ensure that the walls of the plastic bags will do not touch the top surface of the molds. All the molds were put on suitable, leveled racks in the wet room to ensure that all the batches had the same conditions during curing, the team members were careful to keep the same curing time and the same conditions during curing to get the best results. After 24 +/-1h from adding the water, the specimens were de-molded, named and also the ends were marked.

Drying

The air storage method was used to store the specimens. After de-molding the specimens and throughout data collection period, a plastic chamber was used to store the specimens. The plastic chamber is a container made by wood structure and covered by a plastic sheet. The plastic chamber is located in the Concrete laboratory at the University of Maine. Six wood racks are located inside the plastic chamber in layers with about a 2" gap between the layers. The specimens were organized on wood racks inside of the plastic chamber with about 1" gap between the specimens to ensure that air circulation is occurring and also all the specimens were at the same conditions. To meet ASTM C157 standard requirements, an adhesive clear tape was used to wrap the surface of all the racks to ensure that the wood surface does not absorb moisture from the specimens. Figure (12) illustrates the plastic chamber and method of arrangement of the specimens.

Figure 12. Plastic chamber and method of arrangement of specimens.

The measurement and data collection

After de-molding the specimens, the initial reading of the length of the specimen was taken. The reading had been measured by using a length comparator. The used length comparator was produced by Humboldt Company under the name "length comparator, Dial Indicator H-3250". The figure (13) shows the length comparator. This device meets the requirements of ASTM c157 and ASTM c490. The specimens have been named and marked for the upper and lower ends to ensure that all specimens are placed in the same direction and at

the same position in the length comparator device at each measurement of each age was taken.

A reference bar was used to set up a reference reading, prior to that, the specimens were measured in each batch and of each age. Prior to start taking the measurements, the length comparator was installed to adjust and simplify the reading of the reference bar and thus to reduce the calculations and simplify them. These measuring steps were applied as per the users' manual of the Length Comparator device. The reference reading was set on 0.2 in, prior to measure the change in the specimen's length.

The initial reading of the specimen's length was taken directly after de-mold the specimens, that means after 1 day from adding water to the cement of each batch. The readings were taken at ages; 3 days, 5 days, 7 days,11 days,14 days, 21 days, 28 days and 90 days, respectively. At each reading, the temperature and relative humidity were recorded. All the readings are reported in tables as shown in the Appendix(A).

Figure 13. The length comparator.

Calculation

the functions are mentioned in the ASTM C157 standard “Standard Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete” and ASTM C490 standard "Standard Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete" was used to calculate the free shrinkage in each batch of each age.

$$free\ shrinkage = \left(\frac{(CDR\ x - ICDR) * 25.4}{G}\right) * 100\%$$

Where;

free shrinkage = The shrinkage that is occurring at any age%.

CRD x = difference between the length comparator reading of the specimen and the reference bar at any age (in).

ICRD = difference between the length comparator reading of the specimen and the reference bar at 1-day age (initial reading) (in).

G = the distance between the gage studs

Phase 2: Autogenous shrinkage test

Further investigation was required, to understand the Autogenous shrinkage attributable to the effect of low water cement ratio.

The hypotheses of the work of phase 2

- In the group 1, the CNF helps to reduce the self-desiccation phenomena, thus reduce or prevent the autogenous shrinkage to occur.
- The autogenous shrinkage behavior is similar to SAP's behavior, where Adding a small amount of the SAP, has led to reduce or prevent the autogenous shrinkage to occur (Mechtcherine, Reinhardt and (Eds.), 2012).

Therefore, four batches containing the same w/c%=30% were mixed with different CNF ratio to assess the effect of the CNF on the autogenous shrinkage only, and that will be discussed in detail later in this chapter.

Methodology

- Four batches were prepared in the Concrete laboratory in the Civil and Environment Engineering Department at the University of Maine. All of the specimens had the same water to cement ratio with different ratios of the CNF. The ratios of the CNF were 0 as the reference, 0.1%, 0.2% and 0.5%.
- Two specimens for each batch had been cast, and the same molds used in phase 1 were used.
- The instrument used in this work was the same instrument that was used to measure the free shrinkage that was mentioned in the ASTM C157 standard, which was adopted in the previous work but used to sealed specimens (the method of sealing the molds and specimens will be clarified). This is stated in ASTM C1698:

  *"*Measurements with the corrugated mold system are in good agreement with unrestrained length change measurements obtained using Test Method C157/C157M with sealed specimens however, Test Method C157/C157M does not allow measurement of the shrinkage occurring before 24 h''

- The autogenous shrinkage in this work was calculated after 24 hours from adding water to the cement, which means measuring the autogenous shrinkage in the hardened stage.

Materials

1) Cement:

The cement used was QUIKRETE Portland Cement Type I/II (94 lb. bag) - commercial grade.

2) Water:

The water used was tap water from the lab.

3) Cellulose nanofibers:

Cellulose nanofibers produced by the Process Development Center (PDC) at the University of Maine. The ratio of solid CNF is3%. In this study, the concentration of the CNF was calculated by taking a sample of the CNF solution, drying it in the oven at 100º C, and measuring the weight of the sample after drying and calculating the CNF concentration in the solution. The results showed that the concentration of the CNF is 3%, and its value is corresponding to the value that was supplied by the Process Development Center.

Cement paste mix design

An Excel spreadsheet program was designed to calculate the quantities of materials that were required for each mix.

The Excel spreadsheet program used to calculate the quantities of materials for the previous work is the same Excel spreadsheet program used to calculate the quantities of materials for this work.

For all batches: The batch volume(cc) plus the waste ratio equals 1000 cc. This value was calculated by calculating the size of all samples for one batch. The percentage of the waste was considered 25% from the total volume.

All the batches have the same water/cement ratio and different CNF ratios. Table (3) shows the weights and ratios of all the materials involved in the mixing for all batches.

Table 3. Weights and ratios of all the materials involved in the mixing for all batches of the autogenous shrinkage tests.

Batch #	W/C (%)	CNF (%)	Cement (g)	Water (g)	CNF (g)
A	**30**	**0.00**	**2024**	**607**	**0**
B	**30**	**0.10**	**2023**	**586**	**21**
C	**30**	**0.20**	**2022**	**565**	**43**
D	**30**	**0.50**	**2019**	**502**	**107**

Mixing procedure

The mixing procedure used was the same mixing procedure used in the previous work, as well as the same mixers. In this work, the batch size was reduced to 1250 cc because in this work it was designed only to know the effect of the CNF on the autogenous shrinkage, and the size of the batch was calculated on this basis.

Flow table test

The instrument, the measurement, and the calculation used in this work were the same as the instrument, the measurement, and calculation which had been used in the previous work.

Preparing the molds

The molds used in this work were the same as those used in the previous work. Some modifications had been made to the molds such as:

- 1"*1" plastic parts had been manufactured with a hole in the middle. Each piece had been fixed at the end of each mold in order to ensure the sealing of the specimens and not to lose moisture from the ends during the collection data period. The figure (14), shows the shape of the plastic piece and the place of installation.

Figure 14. The shape of the plastic piece and the place of installation.

❖ A plastic cover had been manufactured to ensure that moisture and water were not lost from the mold during the curing period in the wet room. The figure (15) shows the details of the plastic cover. The figure (16) shows where the plastic cover was installed after the completion of the casting process.

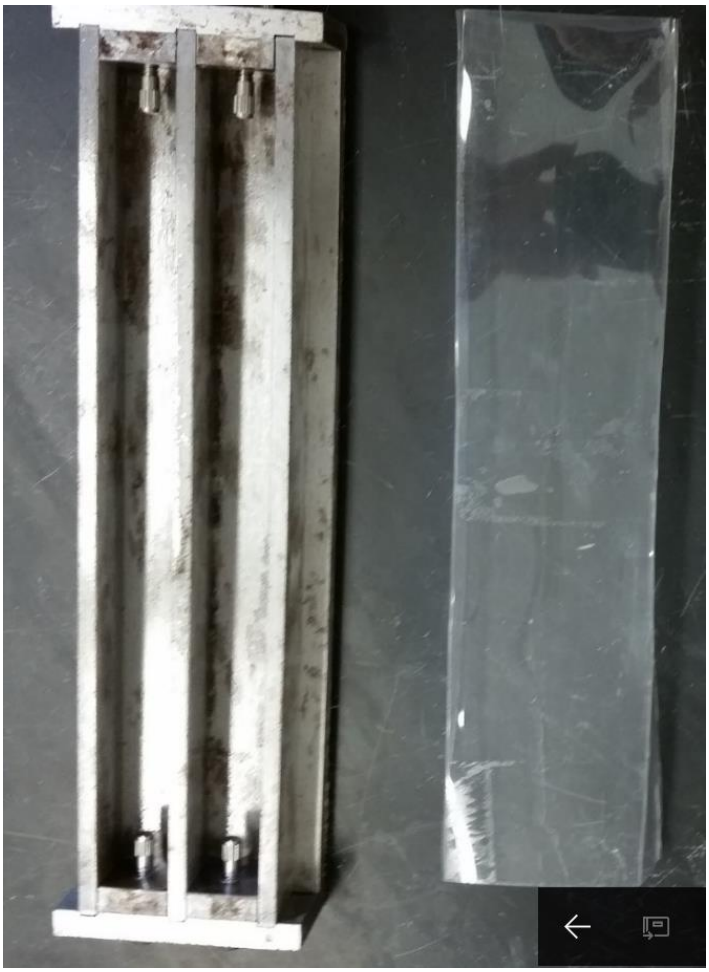

Figure 15. The shape of the plastic cover and the mold before casting.

Casting the specimens

After preparing the molds and assembling them, installing the gage studs at the ends, and installing the plastic pieces in the place at the ends (as explained previously), the molds are placed on the vibration table. After completion of the mixing process and of the Flow Table

test, the casting process begins. ASTM C157/C157M − 08 had been used for casting specimens. The cement paste is placed in the mold in two layers. After placing the first layer, the cement paste was filled under the gage studs by using a straight edge blade. The first layer was then vibrated using the vibrating table. The vibration time and frequency was varied according to the stiffness of the mixture (a workability of the batch). Usually, the first layer was vibrated for 30 to 45 seconds. Hands were used to compact and fill the molds when the cement paste was too hard to compact and vibrate by using the vibrating table. The second layer of the cement paste was then placed, and the cement paste was placed in the corners of the molds by a straight edge blade. The second layer was also vibrated. The surplus cement paste was then removed, and the cement paste surface was leveled. The exterior edges of the molds were then cleaned to remove the extra cement paste. The location of the plastic pieces had been noted at the ends of the mold. Suitable food wrap sheets had been used to wrap the molds, then the plastic cover had been taken and placed on top. Finally, the adhesive tape was used to secure the plastic wrap on the mold and help to prevent losing the moisture. The figure (16), shows the molds after the casting and the wrapping.

Figure 16. The molds after the casting and the wrapping.

De-mold and seal the specimens

After 24 hours, add cement to the water. The preparation was done to open the molds and seal the specimens.

Sealed steps

Step No.1: After opening the mold and cleaning the specimens immediately, a food wrap plastic sheet had been used to wrap the specimens. The same quantity of plastic wrap was used for each specimen to ensure that all specimens were under the same conditions. Each specimen was wrapped 5 times as shown in figure (17).

Figure 17. Sealed the specimen, step No.1.

Step No.2: A shipping bubble plastic sheet had been used to wrap the specimen. The same quantity had been used for each specimen to ensure that all specimens were under the same conditions. Each specimen was wrapped 1 time, as shown in figure (18).

Step No.3: The numbering and naming of each specimen was done. The arrow sign signifies the upper end of the specimen. The position of the specimen in the length comparator should be in the same direction at each reading, as shown in figure (18).

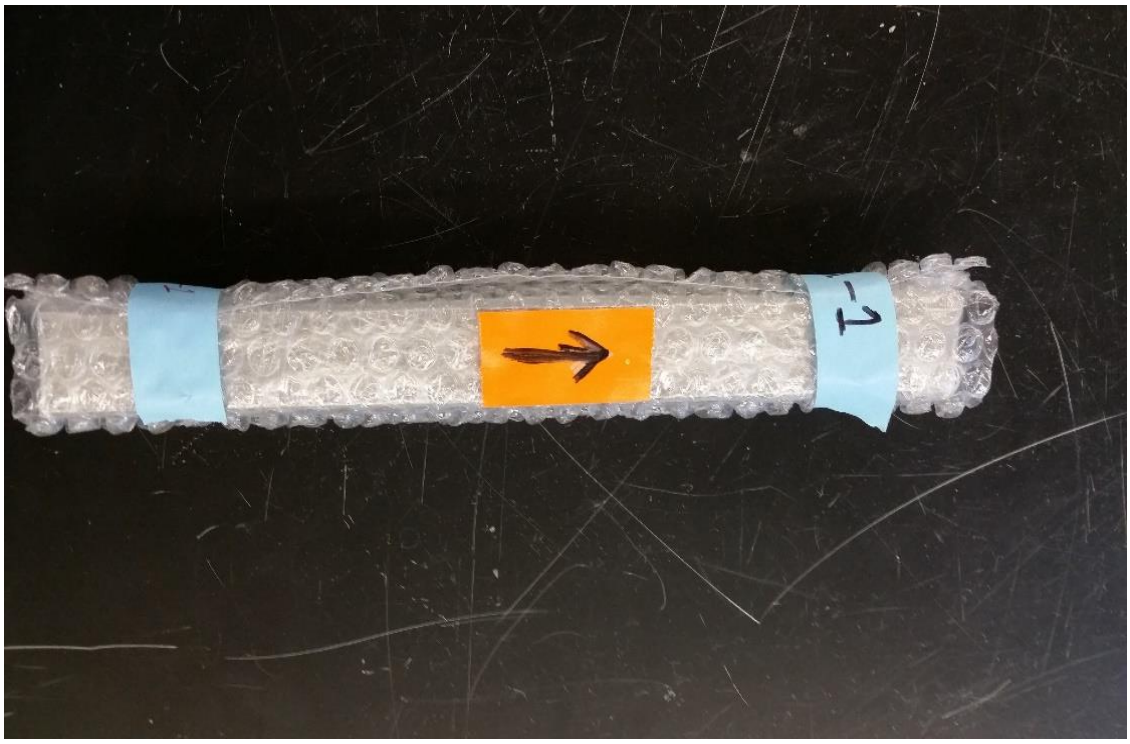

Figure 18. *Sealed the specimen*, step No.2 and step No.3.

Step No.4: The shipping bubble plastic sheet was used to wrap the two specimens for each batch. To ensure the thermal insulation, all the batches were under the same conditions, as shown in figure (19).

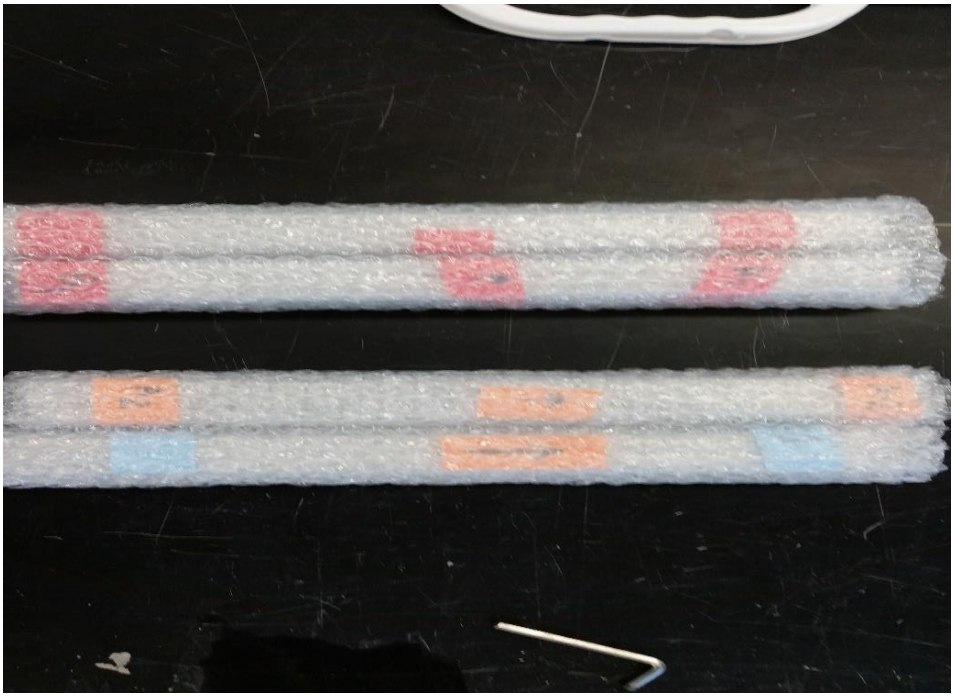

Figure 19. *Sealed the specimen*, step No.4.

5) The specimens of each two batches were stored using a Cool and Hot bag.

6) Finally, all the specimens were placed in the plastic chamber.

The initial reading had been taken after the completion of the third step above, as well as the weight of each specimen. The weight was taken in this test to ascertain the type of shrinkage. The figure (20), shows the specimen in the device.

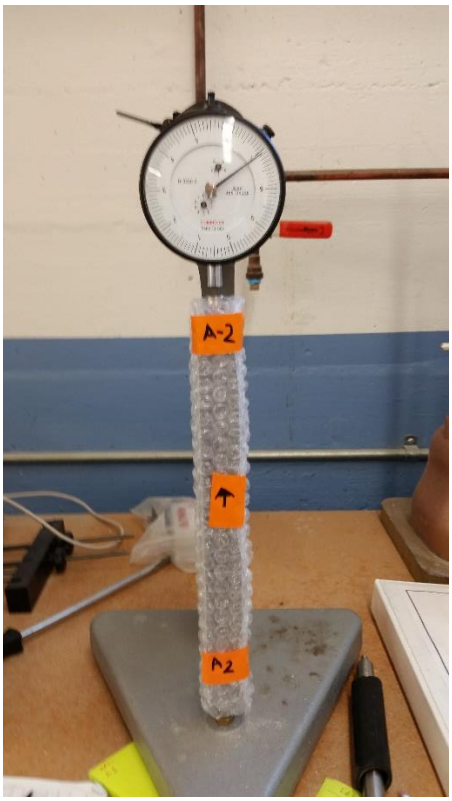

Figure 20. The specimen in the Length Comparator.

Measurement and data collection

The initial reading was taken after the completion of the third step of sealing, as well as the weight of each specimen. The reading had been measured by using a length comparator. The length comparator is the same that had been used in the previous work (phase 1, Summer 2017), as well as the same method of measuring, but the specimen ages were different. At age 2days, 3 days, 4 days, 5 days, 7 days, 9 days, 11 days, 14 days, 21 days and 28 days, the readings were taken sequentially. At each measurement, the temperature and relative humidity were recorded. All the readings were recorded and tabulated in tables. All the tables were placed in the Appendix(C).

The calculation

The functions are mentioned in the ASTM C157 standard and ASTM cC490 standard and were used to calculate the autogenous shrinkage in each batch at each age.

$$The\ Autogenous\ shrinkage\ \% = \left(\frac{(CDR\ x - ICDR) * 25.4}{G}\right) * 100\%$$

Where:

$The\ Autogenous\ shrinkage\ \%$ = The Autogenous shrinkage that is occurring at any age,

CRD x = difference between the comparator reading of the specimen and the reference bar at any age.

ICRD = difference between the comparator reading of the specimen and the reference bar at 1 day age (initial reading).

G = the distance between the gage studs (254mm)

CHAPTER 4: RESULTS

Introduction

As mentioned in chapter two, the goal for this study was to find the effect on shrinkage for the CNF. In this chapter, the results of the free shrinkage tests (phase 1), and autogenous shrinkage tests (phase 2) are presented. Additionally, the results of the flow table tests for each batch are presented. All the results in this chapter represent the results of the average of two specimens because of two specimens casted and tested for each batch.

Free shrinkage was assumed as two types of shrinkage; autogenous shrinkage, and dry shrinkage. Free shrinkage tests (phase 1), included series of cement paste batches, which were tested. In total, four test programs were done. All the programs had 8 batches with different cellulose nanofibrils (CNF) contents. They were 0 as a reference, 0.05 %, 0.1%, 0.2%, 0.5%, 1%, 1.5%, and 3% from the cement volume. The water cement ratios for each program were; 0.35% for group (1), 0.4% for group (2), 0.45% for group (3), and 0.5% for group (4).

For autogenous shrinkage tests (phase 2), four batches were prepared in the concrete lab in the Civil and Environmental Engineering Department at the University of Maine. All of them had the same water to cement ratio with different ratios of the CNF. The ratios of the CNF were 0 as the reference, 0.1%, 0.2%, and 0.5%.

All the results for each specimen and for each batch are presented in individual tables in the Appendix (A and B).

Flow table test

A flow table test was done for each batch directly after finishing the mix of the batches in the mixer. The ASTM C1437_15 titled the "standard test method for flow of hydraulic cement mortar" was used to calculate the workability. The flow table that was used is the same of the flow table that was mentioned in ASTM C230/C230M-14 titled "Standard Specification for flow-table for Use in Tests of Hydraulic Cement". The tables (4, 5, 6, and 7) shows: four columns of dimeters in (mm), the average of these dimeters and the Flow table (%). The calculation of Flow table test was mentioned and explained in chapter three. The figure (21) shows the relation between flow table vs. CNF %, each curve present specific W/C ratio.

Table 4. The results for flow table test, group 1, W/C=35%.

#	W/C %	CNF	1st D (mm)	2nd D (mm)	3rd D (mm)	4thD (mm)	the average	flow (%)
1	35	0	210	210	210	210	210	110.0
2	35	0.05	200	200	200	200	200	100.0
3	35	0.1	200	200	200	200	200	100.0
4	35	0.2	190	190	195	195	192.5	92.5
5	35	0.5	165	165	165	165	165	65.0
6	35	1	140	140	140	145	141.25	41.3
7	35	1.5	130	130	130	130	130	30.0
8	35	3	115	115	115	110	113.75	13.8

Table 5. The results for flow table test, group 2, W/C=40%.

#	W/C %	CNF	1st D (mm)	2nd D (mm)	3rd D (mm)	4thD (mm)	the average	flow (%)
9	40	0	240	240	235	235	230	235.0
10	40	0.05	240	240	240	240	245	241.3
11	40	0.1	225	225	225	225	225	225.0
12	40	0.2	215	215	215	215	215	215.0
13	40	0.5	190	190	195	195	190	192.5
14	40	1	165	165	165	165	160	163.8
15	40	1.5	140	140	140	145	145	142.5
16	40	3	120	120	120	120	120	120.0

Table 6. The results for flow table test, group 3, W/C=45%.

#	W/C %	CNF	1st D (mm)	2nd D (mm)	3rd D (mm)	4thD (mm)	the average	flow (%)
17	45	0	falling after 5 drop, high workablety					
18	45	0.05	falling after 14 drop, high workablety					
19	45	0.1	250	250	250	250	250	250.0
20	45	0.2	245	245	245	245	245	245.0
21	45	0.5	210	210	210	210	210	210.0
22	45	1	175	175	175	175	175	175.0
23	45	1.5	155	155	155	155	150	153.8
24	45	3	130	130	130	130	130	130.0

Table 7. The results for flow table test, group 4, W/C=50%.

#	W/C %	CNF	1st D (mm)	2nd D (mm)	3rd D (mm)	4thD (mm)	the average	flow (%)
25	50	0	falling after 1 drop, high workablety					
26	50	0.05	falling after 6 drop, high workablety					
27	50	0.1	falling after 7 drop, high workablety					
28	50	0.2	250	250	250	250	250	150.00
29	50	0.5	230	230	230	230	225	128.75
30	50	1	185	185	185	185	185	85.00
31	50	1.5	165	165	165	165	165	65.00
32	50	3	130	130	130	130	130	30.00

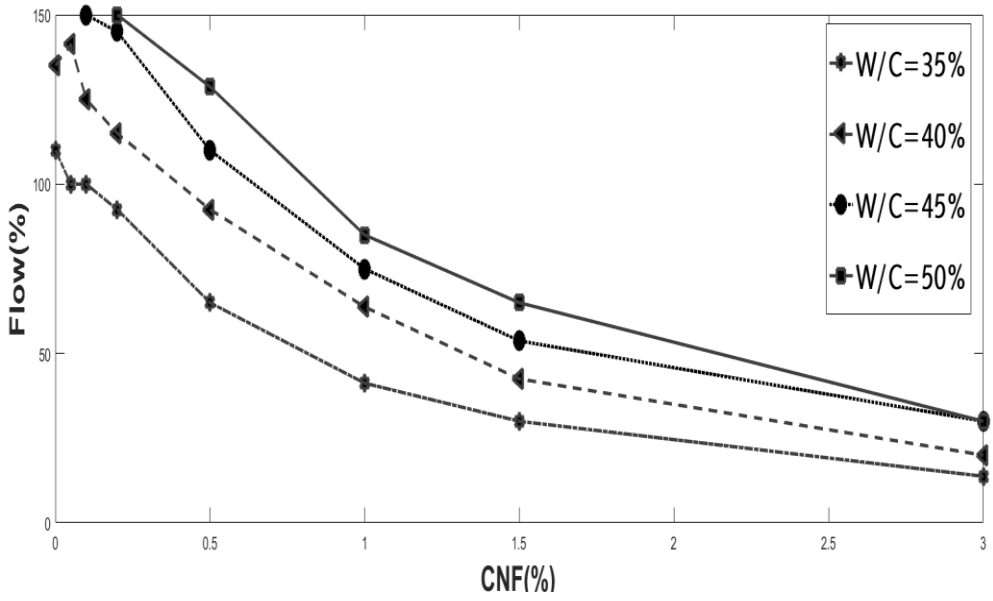

Figure 21. Shows the relation between flow table vs. CNF %, each curve present specific W/C ratio

Phase 1: Free shrinkage test

The comparison among the results of batches for group 1

The different ratios of the CNF had been used to evaluate the free shrinkage in cement paste with a constant water to cement ratio that was 35%. The figures (22), and table (8) are show the results for each proportion of the CNF. The figure presents eight curves, and each curve shows the average result of the free shrinkage for one batch. The curves and the table show that, when the CNF was added in low ratios such as 0.05%, 0.1%, 0.2% and 0.5%, the free shrinkage decreased by 12%, 10.5%, 10% and 8%, respectively. These results were at age 28 days. However, the increase in the proportion of cellulose over of 0.5% led to increasing the value of the free shrinkage.

At the age of 90 days, the same effect occurred but at different rates, as adding 0.05%, 0.1%, and 0.2% gave positive results where the free shrinkage was decreased by 7%, 13.5%, and 5%, respectively. The increase in the proportion of the CNF over 0.5% led to increasing the value of the free shrinkage. That means adding small ratio from the CNF gives positive results for reducing the value of free shrinkage.

Table 8. The average results of the free shrinkage along the ages up to 90 days, for each batch in the group (1), W/C ratio is 35%.

age(days)	The CNF Ratio%							
	0	0.05	0.1	0.2	0.5	1	1.5	3
3	-0.16	-0.10	-0.12	-0.10	-0.12	-0.15	-0.14	-0.14
5	-0.23	-0.15	-0.17	-0.16	-0.19	-0.19	-0.20	-0.23
7	-0.25	-0.20	-0.21	-0.21	-0.21	-0.26	-0.25	-0.27
11	-0.28	-0.22	-0.24	-0.22	-0.28	-0.29	-0.28	-0.28
14	-0.31	-0.27	-0.25	-0.29	-0.28	-0.29	-0.30	-0.31
21	-0.33	-0.28	-0.27	-0.29	-0.30	-0.34	-0.34	-0.34
28	-0.34	-0.30	-0.31	-0.31	-0.31	-0.35	-0.33	-0.35
90	-0.39	-0.36	-0.34	-0.37	-0.39	-0.41	-0.39	-0.41

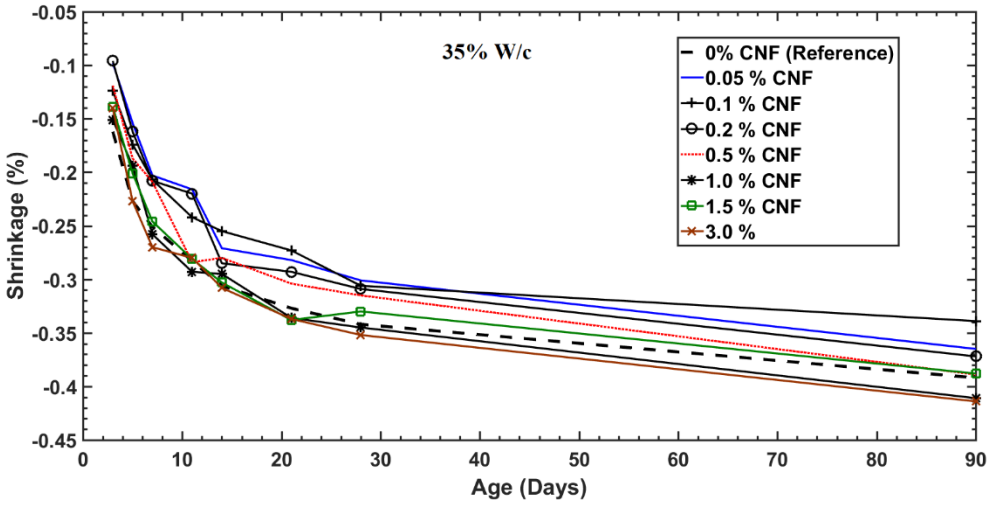

Figure 22. The curves of group (1).

The comparison among the results of batches for group 2

The different ratio of the CNF used to evaluate the free shrinkage in cement paste with constant water to cement ratio was 40%. The Figure (23) and the table (9) show the results for each proportion of the CNF. The figure presents eight curves, and each curve presents the average results of the free shrinkage for one batch. The curves show, at age 28 days, when the CNF was added the free shrinkage increased.

At the age of 90 days, when the CNF was added in low ratios such as 0.05%, 0.1%, and 0.2%, the free shrinkage decreased by 3%, 2.5%, and 3%, respectively. On the other hand, increasing the proportion of the CNF over 0.2%, led to increasing the value of the free shrinkage. That means adding the CNF does not give helpful results for reducing the value of free shrinkage either short term or long term.

Table 9. The average results of the free shrinkage along the ages up to 90 days, for each batch in the group (2), W/C ratio is 40%.

age (days)	The CNF Ratio(%)							
	0	0.05	0.1	0.2	0.5	1	1.5	3
3	-0.07	-0.08	-0.09	-0.08	-0.09	-0.10	-0.11	-0.14
5	-0.13	-0.15	-0.14	-0.14	-0.15	-0.18	-0.18	-0.20
7	-0.16	-0.19	-0.18	-0.18	-0.19	-0.21	-0.22	-0.26
11	-0.21	-0.22	-0.22	-0.23	-0.24	-0.28	-0.27	-0.30
14	-0.23	-0.24	-0.24	-0.25	-0.27	-0.28	-0.30	-0.31
21	-0.27	-0.28	-0.26	-0.30	-0.28	-0.31	-0.31	-0.35
28	-0.29	-0.30	-0.29	-0.30	-0.31	-0.33	-0.34	-0.37
90	-0.37	-0.35	-0.36	-0.36	-0.39	-0.40	-0.42	-0.45

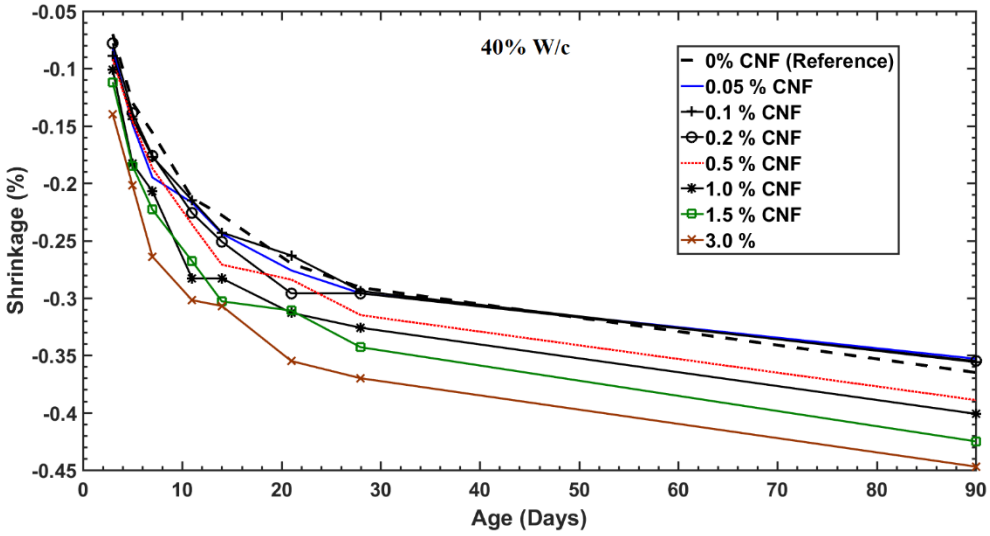

Figure 23. The curves of group (2).

The comparison among the results of batches for group (3)

The different ratio of the CNF used to evaluate the free shrinkage in cement paste with constant water to cement ratio was 45%. The figure (24) and the table (10) show the result for each proportion of the CNF. The figure presents eight curves, and each curve presents the average results of the free shrinkage for one batch. The curves show, at age 28 days, when the CNF was added the free shrinkage increased, except with the ratio of 0.1%. When this ratio was added, the free shrinkage was reduced to 5.5%

At the age of 90 days, the curves show there was no benefit when the CNF was added in low ratios such as 0.05%, 0.1%, and 0.2%.

Over 0.2% ratio of the CNF had been added, and that led the free shrinkage to increase, except for the ratio of 1%. When this ratio was added, the free shrinkage was reduced to

about 2.5%. That means adding the CNF does not give helpful results for reducing the value of free shrinkage either short term or long term.

Table 10. The average results of the free shrinkage along the ages up to 90 days, for each batch in the group (3), W/C ratio is 45%.

age(days)	The CNF Ratio(%)							
	0	0.05	0.1	0.2	0.5	1	1.5	3
3	-0.07	-0.06	-0.06	-0.06	-0.09	-0.09	-0.10	-0.10
5	-0.13	-0.12	-0.12	-0.12	-0.17	-0.17	-0.17	-0.17
7	-0.16	-0.16	-0.16	-0.15	-0.20	-0.20	-0.21	-0.23
11	-0.21	-0.21	-0.21	-0.20	-0.23	-0.23	-0.23	-0.29
14	-0.24	-0.24	-0.23	-0.23	-0.24	-0.23	-0.26	-0.29
21	-0.29	-0.29	-0.25	-0.25	-0.28	-0.26	-0.28	-0.33
28	-0.29	-0.29	-0.28	-0.29	-0.31	-0.29	-0.31	-0.37
90	-0.35	-0.35	-0.35	-0.35	-0.36	-0.34	-0.37	-0.45

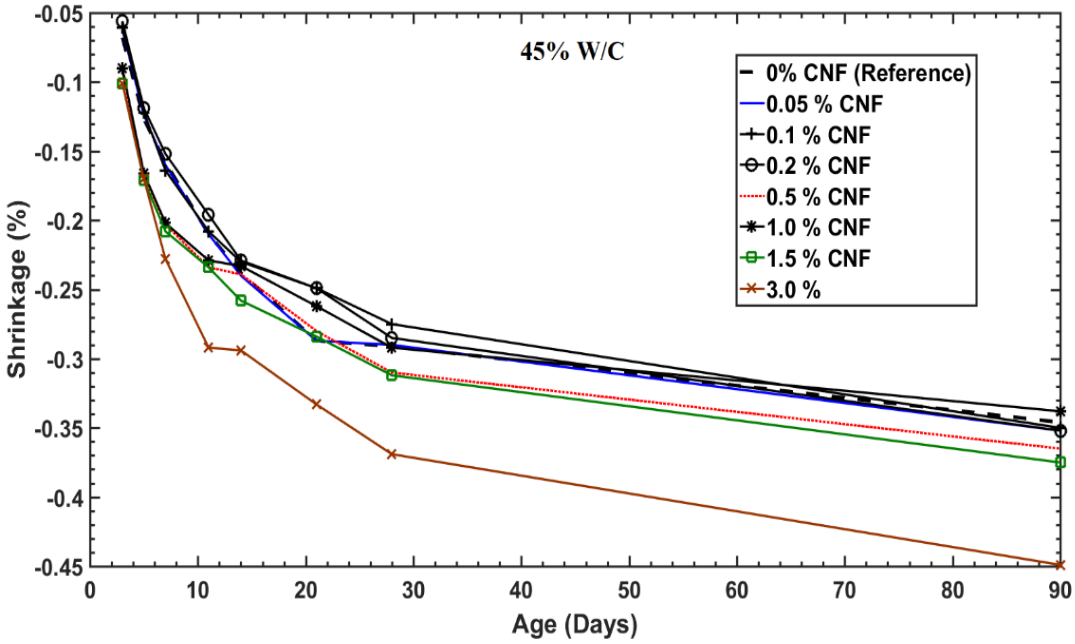

Figure 24.The curves of group (3).

The comparison among the results of batches for group 4

The different ratio of the CNF used to evaluate the free shrinkage in cement paste with constant water to cement ratio was 50%. The figure (25) and table (11) show the results for each proportion of the CNF. The figure presents eight curves, and each curve presents the average results of the free shrinkage for one batch. The curves show, at age 28 days, when the CNF was added the free shrinkage increased.

At the age of 90 days, the curves show, when the CNF was added, the free shrinkage was increased, except for the ratio 0.2%. When this ratio was added, the free shrinkage had reduced to about 7%. That means adding the CNF does not give helpful results for reducing the value of free shrinkage either short term or long term.

Table 11. The average results of the free shrinkage along the ages up to 90 days, for each batch in the group (4), W/C ratio is 50%.

Age (days)	The CNF Ratio%							
	0	0.05	0.1	0.2	0.5	1	1.5	3
3	-0.04	-0.03	-0.03	-0.05	-0.05	-0.08	-0.09	-0.12
5	-0.09	-0.09	-0.09	-0.11	-0.11	-0.19	-0.16	-0.20
7	-0.14	-0.15	-0.14	-0.13	-0.16	-0.22	-0.20	-0.24
11	-0.20	-0.20	-0.21	-0.19	-0.21	-0.28	-0.26	-0.29
14	-0.21	-0.25	-0.22	-0.23	-0.24	-0.31	-0.28	-0.33
21	-0.26	-0.27	-0.26	-0.28	-0.29	-0.31	-0.33	-0.37
28	-0.28	-0.30	-0.30	-0.29	-0.29	-0.33	-0.35	-0.40
90	-0.37	-0.38	-0.37	-0.34	-0.36	-0.39	-0.39	-0.43

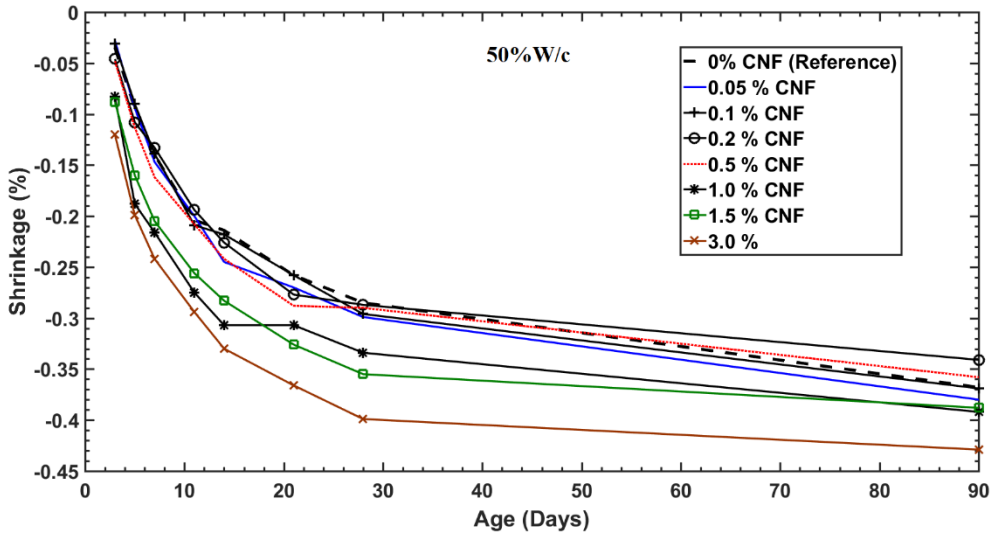

Figure 25. The curves of group (3).

Comparison among the references

In this comparison, the results for the reference batch for each group was collected and presented in one table and one graph. The reference batch is when the ratio of the CNF was 0%. The reference batches were batch No.1, batch No.9, batchNo.17, and batch No.25. The table (12) and figure (26), shown below, present that the highest free shrinkage occurred with the lowest water cement ratio, and the lowest water to cement ratio was 35%.

Table 12. The average results of the free shrinkage along the ages up to 90 days, for the reference batch of each group.

The age(days)	The water to cement ratio(%)			
	35%	0.4	0.45	0.5
3	-0.16	-0.07	-0.07	-0.04
5	-0.23	-0.13	-0.13	-0.09
7	-0.25	-0.16	-0.16	-0.14
11	-0.28	-0.21	-0.21	-0.20
14	-0.31	-0.23	-0.24	-0.21
21	-0.33	-0.27	-0.29	-0.26
28	-0.34	-0.29	-0.29	-0.28
90	-0.39	-0.37	-0.35	-0.37

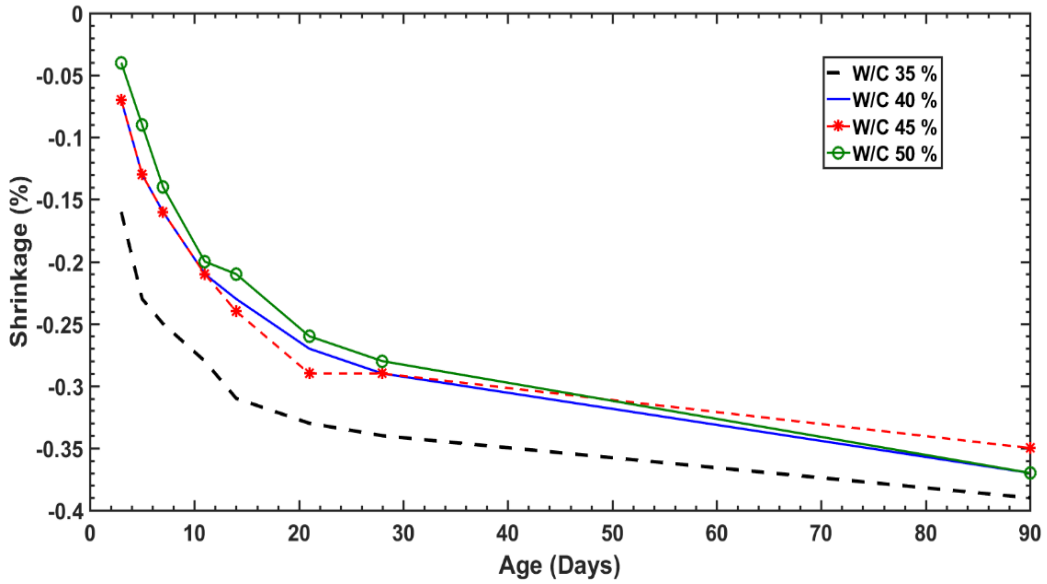

Figure 26. The free shrinkage (%) Vs. age (days), for the reference batch of each group.

Comparison between the batches that have a similar workability

The results of the batches that have a similar flow table test result (i.e. similar workability) were shown in this comparison. These batches were: batch No.1, batch No.12, and batch No.21. Batch No.1 was considered as a reference. The table (13) presents brief information for each batch. The flow table test was used to present the workability.

Working with the CNF shows that when adding CNF to cement paste batch, it leads to reducing the workability of this batch. The comparison between two batches shows that they both had the similar workability. The batches with CNF developed free shrinkage less than the reference, although both of these batches with the CNF were prepared with the higher ratio of water than the reference batch.

The figure (27) shows the curves of the free shrinkage for batches No.1 and No.12. The Figure (28) shows the curves of the free shrinkage for batches No.1 and No.21.

Table 13. Brief information for the batches that have similar workability.

Batch No.	#1	#12	#21
Group No.	1	2	3
CNF(%)	0	0.2	0.5
w/c(%)	35	40	45
The Workabiliy	110%	115%	110%
The free shrinkage resulte			
age(days)	**#1**	**#12**	**#21**
3	-0.16	-0.08	-0.09
5	-0.23	-0.14	-0.17
7	-0.25	-0.18	-0.20
11	-0.28	-0.23	-0.23
14	-0.31	-0.25	-0.24
21	-0.33	-0.30	-0.28
28	-0.34	-0.30	-0.31
90	-0.39	-0.36	-0.37

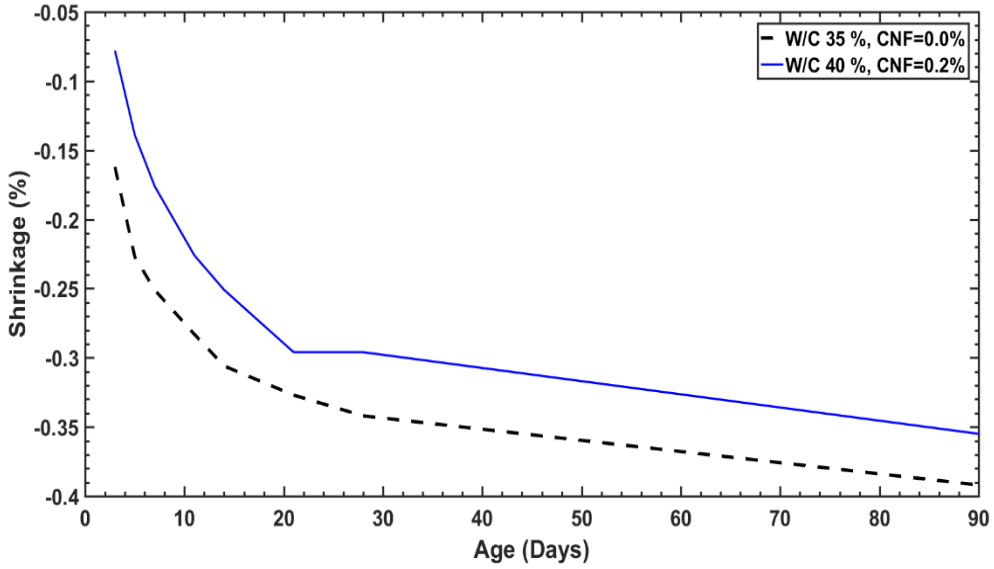

Figure 27. The figure shows two batches, they both have the same workability, with different W/C%, with different CNF%.

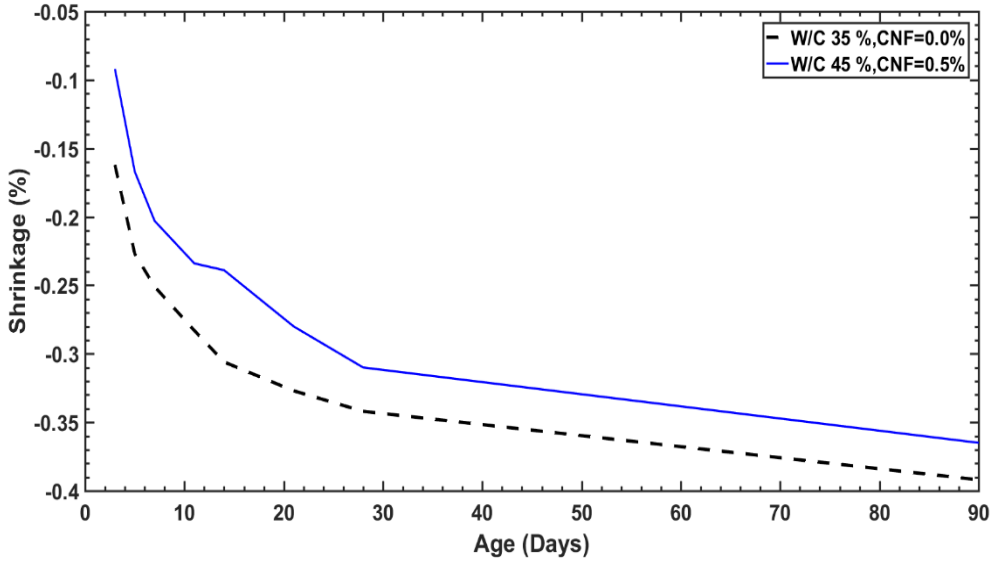

Figure 28. The figure shows two batches, they both have the same workability, with different W/C%, with different CNF%.

Phase 2: Autogenous shrinkage test

The comparison among the results of the batches for phase 2

A different ratio of the CNF was used to evaluate the Autogenous shrinkage in cement paste (as explained previously), and the water to cement ratio that was 30%. These batches were batch A, batch B, batch C, and batch D. Batch A was considered as a reference. The table (14) presents brief information for each batch. The flow table test was used to present the workability. The figure (29) presents four curves, and each curve shows the average result of the Autogenous shrinkage for each batch (%) vs. age (days).

Table 14. Brief information and results, of Autogenous Shrinkage tests.

Batch label	A	B	C	D
CNF(%)	0	0.1	0.2	0.5
w/c(%)	30	30	30	30
The Workabiliy	94%	89%	70%	48%
The results of the Autogenous shrinkage tests				
Age (days)	A	B	C	D
2	-0.04	-0.04	-0.04	-0.04
3	-0.06	-0.06	-0.07	-0.06
4	-0.07	-0.07	-0.08	-0.07
5	-0.08	-0.08	-0.08	-0.08
7	-0.09	-0.08	-0.1	-0.09
9	-0.1	-0.09	-0.11	-0.1
11	-0.11	-0.1	-0.11	-0.1
14	-0.1	-0.1	-0.11	-0.1
21	-0.11	-0.1	-0.12	-0.11
28	-0.11	-0.1	-0.12	-0.11

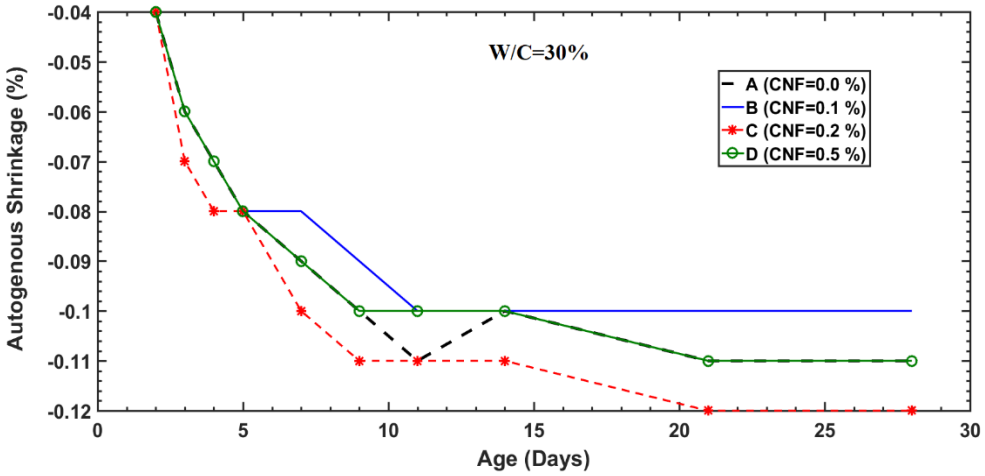

Figure 29. The average result of the autogenous shrinkage for each batch (%) Vs. age(days).

CHAPTER 5: SUMMARY AND CONCLUSION

The summary of the results for the groups

In general, the addition of the CNF to the cement paste led to reducing the workability (the flow) of cement paste, which is according to the Flow table results for all groups. In other words, the group of batches has the same water to cement ratio with different CNF ratios, the batch that has the lowest CNF ratio will have the highest flow. While the batch that has highest CNF% will have the lowest flow. This behavior is due to the ability of the CNF to absorb part of the mixing water in different proportions, these proportions have not been accurately identified. The tables (4, 5, 6, and 7) show the results for the flow table tests, the figure (30) shows the relation between flow table vs. CNF %, each curve present specific W/C ratio.

Phase 1: The free shrinkage tests

At age 28 days, the results showed that the group two, when the water to cement ratio was 40%. The increase in the CNF ratio that was added to batches in the same group led to increasing the free shrinkage. The rate of the increment of the free shrinkage was 27% when 3% of the CNF was added.

The group three, when the water to cement ratio was 45%. The increase in the CNF ratio that was added to batches in the same group led to increasing the free shrinkage. The rate of the increase of the free shrinkage was 26% when 3% of the CNF was added.

The group four, when the water to cement ratio was 50%. The increase in the CNF ratio that was added to batches in the same group led to increasing the free shrinkage. The rate of the increment of the free shrinkage was 40% when 3% of the CNF was added.

For the group one, when the water to cement ratio was 30% and a small quantity of the CNF such as 0.05% was added, a13% reduce in the free shrinkage occurred, when a comparison took place with the reference batch for this group. Using a small rate of the CNF such as (0.05%, 0.1%, 0.2% and 0.5) has helped to decrease the free shrinkage in this group. However, no benefit was noticed from adding other rates such as (1%, 1.5%, and 3%).

The free shrinkage was assumed as two types of shrinkage; autogenous shrinkage, and dry shrinkage. The results show that there is not a distinguished autogenous shrinkage in ordinary cement paste or in the normal concrete, on another hand, there is a distinguished autogenous shrinkage with the high-performance concrete or low water-cement ratio (i.e. when the water to the cement ratio less than 40%). In ordinary concrete and with the concrete that has more than 40 % water to cement ratio the self-desiccation phenomena was not noticed(Wu *et al.*, 2017). The autogenous shrinkage does not appear clearly to the specimens of the batches with w/c ratio greater than 40%, hence, the free shrinkage is drying shrinkage.

The batches that had water to cement ratio as 40%, 45% and 50%, with the addition of CNF ratios, the resulting drying shrinkage has increased accordingly. Interpretation of results is more CNF means more voids filled with water, hence, more water will be exposed to evaporate, finally greater drying shrinkage to occur.

For the group one, when W/C ratio was 0.35 (i.e. the W/C ratio less than 0.40 %), that means autogenous shrinkage had happened. when 0.05%, 0.1%, or 0.2% of the CNF were used, immediately the free shrinkage decreased. That mean a small quantities of The CNF help to prevent or reduce the autogenous shrinkage. (Jensen and Hansen, 2002) said add small quantities of Superabsorbent polymer (SAP) to the cement paste leads to a reduction of the autogenous shrinkage, furthermore that, sometimes lead to an expansion. As long as CNF has the same behavior of the superabsorbent polymer (SAP). The behavior is absorbent water in the beginning and release it later. By this mechanism, Finally, using a small amount of CNF help to reduce or prevent self-desiccation phenomena and reduce the autogenous shrinkage. Depend on this phenomenon and these results, the work of phase 2 was designed.

Phase 2: The autogenous shrinkage tests

Four batches (A, B, C, and D) were prepared in the Concrete laboratory in the Civil and Environmental Engineering Department at the University of Maine, all the batches had the same water to cement ratio with different rates of the CNF. The rates of the CNF were (0 as a reference ,0.1%, 0.2% and 0.5%). Table (14) and figure (29) show the information and the results of the tests. ASTM C157 was used to measure the autogenous shrinkage. the sealed specimens were used, to ensure the shrinkage that occurred was autogenous shrinkage. Specimens being sealed with the procedure that explained in chapter three, under the title (sealed steps). The measurements were started at age was one day (24 hours) when the 1 (in) *1(in)*11.25(in) specimens were produced, these specimens were wrapped and they were stored in hot and cool bags. The length change of two specimens for each batch was reported, the results showed that there was no effect on the Autogenous

shrinkage of the CNF, after using different ratios of the CNF. The reasons will be explained and Identified:

- The CNF was used, it is a solution. this solution contains a concentration of solid CNF and water. In the hypotheses of design for the cement paste for these work (phase 1and phase 2), assumed that all the water in the CNF solution was considered as a free water. In other words, the water in the CNF solution was part of the water that was calculated from the water to cement ratio. After doing this series of experiments and batches for phase 1 and phase 2, this assumption is not acceptable, because of the increase in the proportion of CNF reduce the value of the flow (the workability) of the cement paste, which means that the CNF trapped the molecules of the water in the solution and may be holding additional water from the water mixture.
- Three batches: batch No.1, batch No.12 and batch No.21 were showed in the table (13), the figure (27), and the figure (28). all the batches had the similar flow table value (the same workability). The batch No.1 was a reference batch for the other two batches. It was noted that the beaches had CNF developed free shrinkage less than the reference batch, while the water to cement ratio for these batches was higher than the water to cement ratio for the reference batch, so an extra water should be considered to investigate and determine the effect of CNF on autogenous shrinkage. This behavior is similar to the behavior of the SAP (the extra water must be added to utilize its properties in Autogenous shrinkage).

Conclusions

- The water in the CNF solution cannot be considered as free water, because of CNF was noted that it has ability to absorb more water that it has. The addition of the CNF to cement paste batch that leads to increase in the drying shrinkage for this batch.
- Using the ASTM C157 for calculating and measuring the Autogenous shrinkage but sealed specimens should be used in this test.
- Adding a small ratio of the CNF as 0.05 %, 0.1% and 0.2% with extra water to cement paste batch that leads to decrease in autogenous shrinkage for this batch.
- In the free shrinkage test, when W/C ratio was 35%, and low CNF ratio was used, CNF helps to reduce the self-desiccation phenomena, thus reduce or prevent the autogenous shrinkage to occur The hypothesis of that is CNF help in internal curing however when a high ratio of CNF was used led to increasing the shrinkage. The Interpretation of that is more CNF means more voids filled with water, hence, more water will be exposed to evaporate, finally greater drying shrinkage to occur

Recommendation

Working to find the percentage of water that was held by CNF. Working to find the effect of CNF plus extra water on autogenous shrinkage. Working to find the effect of CNF plus extra water on Autogenous shrinkage in the early age of cement paste.

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APPENDIX

APPENDIX (A). FREE SHRINKAGE TESTS

In these tables, all the results for shrinkage specimens for each batch at each age also the temperature and Relative humidity were recorded for each age for phase one (free shrinkage tests).

Table 15. Group (1), when W/C ratio (35%) and CNF ratio (0%).

Batch #1				**casting date:**	**6/20/17**							
Age (days)	**Date**	**Time**	**T (F)**	**RH (%)**	**Ref. Bar (in)**	**Spe. No.7(in)**	**Change (in)**	**shrinkage**	**Spe. No.8(in)**	**Change (in)**	**shrinkage**	**Ave.Shr. (%)**
1	6/21/17	9:45			0.2	0.2014	0.0014		0.1963	-0.0037		
3	6/23/17	5:00	72	63	0.2	0.1853	-0.0147	-0.16	0.1805	-0.0195	-0.16	-0.16
5	6/25/17	11:15	73	62	0.2	0.179	-0.021	-0.23	0.1741	-0.0259	-0.23	-0.23
7	6/27/17	10:48	73	55	0.2	0.1767	-0.0233	-0.25	0.1716	-0.0284	-0.25	-0.25
11	7/1/17	5:15	71	65	0.2	0.1735	-0.0265	-0.28	0.1685	-0.0315	-0.28	-0.28
14	7/4/17	1:20	75	61	0.2	0.1712	-0.0288	-0.31	0.1662	-0.0338	-0.31	-0.31
21	7/11/17	12:10	75	60	0.2	0.1692	-0.0308	-0.33	0.1641	-0.0359	-0.33	-0.33
28	7/18/17	12:30	75	61	0.2	0.1676	-0.0324	-0.34	0.1627	-0.0373	-0.34	-0.34
90	9/18/17	3:00	71	65	0.2	0.163	-0.037	-0.39	0.1575	-0.0425	-0.39	-0.39

Table 16. Group (1), when W/C ratio (35%) and CNF ratio (0.05%).

Batch #2					**Casting date:**	**6/2/17**						
Age (days)	**Date**	**Time**	**T (F)**	**RH (%)**	**Ref. Bar (in)**	**Spe. No.7(in)**	**Change ()**	**shrinkage (%)**	**Spe. No.8(in)**	**Change ()**	**shrinkage (%)**	**Ave.Shr. (%)**
1	**6/3/17**	11:00			0.2	0.1816	-0.0184		0.1844	-0.0156		
3	**6/5/17**	9:15	67	60	0.2	0.172	-0.028	-0.10	0.1748	-0.0252	-0.10	-0.10
5	**6/7/17**	11:50	69	59	0.2	0.1664	-0.0336	-0.15	0.1692	-0.0308	-0.15	-0.15
7	**6/9/17**	12:00	69	57	0.2	0.1616	-0.0384	-0.20	0.1645	-0.0355	-0.20	-0.20
11	**6/13/17**	12:30	77	70	0.2	0.1605	-0.0395	-0.21	0.1629	-0.0371	-0.22	-0.22
14	**6/16/17**	9:40	70	52	0.2	0.154	-0.046	-0.28	0.1587	-0.0413	-0.26	-0.27
21	**6/23/17**	5:15	72	63	0.2	0.1539	-0.0461	-0.28	0.1565	-0.0435	-0.28	-0.28
28	**6/30/17**	2:00	71	63	0.2	0.1519	-0.0481	-0.30	0.1548	-0.0452	-0.30	-0.30
90	**8/31/17**	1:00	72	55	0.2	0.1459	-0.0541	-0.36	0.1483	-0.0517	-0.37	-0.36

Table 17.Group (1), when W/C ratio (35%) and CNF ratio (0.1%).

Batch #3					casting date: 5/18/17							
Age (days)	Date	Time	T (deg)	RH ()	Ref. Bar (in)	Spe. No.7(in)	Change ()	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/19/17	9:30			0.2	0.1652	-0.0348		0.1758	-0.0242		
3	5/21/17	10:00	71	44	0.2	0.1527	-0.0473	-0.13	0.1639	-0.0361	-0.12	-0.12
5	5/23/17	2:15	72	50	0.2	0.1481	-0.0519	-0.17	0.1587	-0.0413	-0.17	-0.17
7	5/25/17	9:45	70	51	0.2	0.1443	-0.0557	-0.21	0.1559	-0.0441	-0.20	-0.21
11	5/29/17	11:50	69	54	0.2	0.1408	-0.0592	-0.25	0.1525	-0.0475	-0.24	-0.24
14	6/1/17	11:20	68	62	0.2	0.1396	-0.0604	-0.26	0.1513	-0.0487	-0.25	-0.25
21	6/8/17	10:50	71	60	0.2	0.1384	-0.0616	-0.27	0.1488	-0.0512	-0.27	-0.27
28	6/15/17	9:25	71	45	0.2	0.1349	-0.0651	-0.31	0.1458	-0.0542	-0.30	-0.31
90	8/16/17	11:45	73	64	0.2	0.1316	-0.0684	-0.34	0.1426	-0.0574	-0.34	-0.34

Table 18. Group (1), when W/C ratio (35%) and CNF ratio (0.2%).

Batch #4					casting date: 6/2/17							
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/3/17	11:00			0.2	0.1771	-0.0229		0.1795	-0.0205		
3	6/5/17	9:20	67	60	0.2	0.168	-0.032	-0.09	0.1698	-0.0302	-0.10	-0.10
5	6/7/17	11:50	69	59	0.2	0.1612	-0.0388	-0.16	0.1635	-0.0365	-0.16	-0.16
7	6/9/17	11:10	69	57	0.2	0.1565	-0.0435	-0.21	0.1592	-0.0408	-0.21	-0.21
11	6/13/17	12:35	77	70	0.2	0.1555	-0.0445	-0.22	0.1578	-0.0422	-0.22	-0.22
14	6/16/17	9:45	70	52	0.2	0.149	-0.051	-0.29	0.1514	-0.0486	-0.29	-0.29
21	6/23/17	5:25	72	63	0.2	0.1483	-0.0517	-0.29	0.1507	-0.0493	-0.29	-0.29
28	6/30/17	2:05	71	63	0.2	0.1468	-0.0532	-0.31	0.149	-0.051	-0.31	-0.31
90	8/31/17	1:10	72	55	0.2	0.1408	-0.0592	-0.37	0.1425	-0.0575	-0.38	-0.37

Table 19. Group (1), when W/C ratio (35%) and CNF ratio (0.5%).

Batch #5					casting date:	6/5/17						
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/6/17	9:30			0.2	0.1892	-0.0108		0.1926	-0.0074		
3	6/8/17	10:55	71	60	0.2	0.1774	-0.0226	-0.12	0.181	-0.019	-0.12	-0.12
5	6/10/17	11:25	70	54	0.2	0.1706	-0.0294	-0.19	0.1743	-0.0257	-0.19	-0.19
7	6/12/17	11:30	75	68	0.2	0.1684	-0.0316	-0.21	0.1722	-0.0278	-0.21	-0.21
11	6/16/17	9:45	70	52	0.2	0.161	-0.039	-0.29	0.1649	-0.0351	-0.28	-0.28
14	6/19/17	9:45	71	67	0.2	0.1615	-0.0385	-0.28	0.1652	-0.0348	-0.28	-0.28
21	6/26/17	11:25	73	62	0.2	0.1592	-0.0408	-0.30	0.1627	-0.0373	-0.30	-0.30
28	7/3/17	11:20	73	66	0.2	0.1581	-0.0419	-0.32	0.1617	-0.0383	-0.31	-0.31
90	9/3/17	11:30	70	56	0.2	0.1509	-0.0491	-0.39	0.1544	-0.0456	-0.39	-0.39

Table 20. Group (1), when W/C ratio (35%) and CNF ratio (1%).

Batch #6					casting date:	6/7/17						
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/8/17	10:25			0.2	0.191	-0.009					
3	6/10/17	11:25	70	54	0.2	0.1761	-0.0239	-0.15				-0.15
5	6/12/17	11:30	75	68	0.2	0.1719	-0.0281	-0.19				-0.19
7	6/14/17	11:35	73	45	0.2	0.1656	-0.0344	-0.26				-0.26
11	6/18/17	12:10	70	62	0.2	0.1622	-0.0378	-0.29				-0.29
14	6/21/17	10:05	72	67	0.2	0.162	-0.038	-0.29				-0.29
21	6/28/17	11:40	72	59	0.2	0.1579	-0.0421	-0.34				-0.34
28	7/5/17	1:30	75	61	0.2	0.157	-0.043	-0.35				-0.35
90	9/5/17	11:30	70	56	0.2	0.1505	-0.0495	-0.41				-0.41

Table 21.Group (1), when W/C ratio (35%) and CNF ratio (1.5%).

Batch #7			casting date:	5/24/17								
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/25/17	9:30			0.2	0.1956	-0.0044		0.1677	-0.0323		
3	5/27/17	11:15	67	54	0.2	0.1814	-0.0186	-0.14	0.1545	-0.0455	-0.13	-0.14
5	5/29/17	11:55	69	54	0.2	0.1756	-0.0244	-0.20	0.1482	-0.0518	-0.20	-0.20
7	5/31/17	10:10	68	59	0.2	0.171	-0.029	-0.25	0.1438	-0.0562	-0.24	-0.25
11	6/4/17	11:45	68	60	0.2	0.1675	-0.0325	-0.29	0.1405	-0.0595	-0.28	-0.28
14	6/7/17	11:55	69	59	0.2	0.1657	-0.0343	-0.30	0.138	-0.062	-0.30	-0.30
21	6/14/17	11:35	73	45	0.2	0.1621	-0.0379	-0.34	0.1347	-0.0653	-0.34	-0.34
28	6/21/17	10:05	72	67	0.2	0.1629	-0.0371	-0.33	0.1355	-0.0645	-0.33	-0.33
90	8/22/17	1:45	75	65	0.2	0.1572	-0.0428	-0.39	0.1298	-0.0702	-0.39	-0.39

Table 22. Group (1), when W/C ratio (35%) and CNF ratio (3%).

Batch #8			casting date:	6/9/17								
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/10/17	11.15			0.2	0.1998	-0.0002		0.2005	0.0005		
3	6/12/17	11:35	75	68	0.2	0.1861	-0.0139	-0.14	0.1866	-0.0134	-0.14	-0.14
5	6/14/17	11:35	73	45	0.2	0.1777	-0.0223	-0.22	0.178	-0.022	-0.23	-0.23
7	6/16/17	9:50	70	52	0.2	0.1733	-0.0267	-0.27	0.1738	-0.0262	-0.27	-0.27
11	6/20/17	11.25	72	67	0.2	0.1725	-0.0275	-0.28	0.1726	-0.0274	-0.28	-0.28
14	6/23/17	5:30	72	63	0.2	0.1696	-0.0304	-0.31	0.17	-0.03	-0.31	-0.31
21	6/30/17	2:10	71	63	0.2	0.1669	-0.0331	-0.33	0.1671	-0.0329	-0.34	-0.34
28	7/7/17	5:30	75	62	0.2	0.1654	-0.0346	-0.35	0.1656	-0.0344	-0.35	-0.35
90	9/7/17	9:00	70	62	0.2	0.1594	-0.0406	-0.41	0.1595	-0.0405	-0.42	-0.41

Table 23.Group (2), when W/C ratio (40%) and CNF ratio (0.00%).

Batch #9				**casting date:**	**5/29/17**							
Age (days)	**Date**	**Time**	**T (F)**	**RH (%)**	**Ref. Bar (in)**	**Spe. No.7(in)**	**Change (in)**	**shrinkage (%)**	**Spe. No.8(in)**	**Change (in)**	**shrinkage (%)**	**Ave.Shr. (%)**
1	5/30/17	10:40			0.2	0.1933	-0.0067		0.1882	-0.0118		
3	6/1/17	11:30	68	62	0.2	0.1861	-0.0139	-0.07	0.1817	-0.0183	-0.07	-0.07
5	6/3/17	11:45	68	60	0.2	0.1805	-0.0195	-0.13	0.1755	-0.0245	-0.13	-0.13
7	6/5/17	9:25	67	60	0.2	0.1782	-0.0218	-0.15	0.1725	-0.0275	-0.16	-0.16
11	6/9/17	11:10	69	57	0.2	0.1713	-0.0287	-0.22	0.1683	-0.0317	-0.20	-0.21
14	6/12/17	11:35	75	68	0.2	0.1705	-0.0295	-0.23	0.1662	-0.0338	-0.22	-0.23
21	6/19/17	9:45	71	67	0.2	0.1665	-0.0335	-0.27	0.1619	-0.0381	-0.27	-0.27
28	6/26/17	11:30	73	62	0.2	0.1642	-0.0358	-0.30	0.1601	-0.0399	-0.29	-0.29
90	8/27/17	2:00	72	52	0.2	0.1574	-0.0426	-0.36	0.1522	-0.0478	-0.37	-0.37

Table 24. Group (2), when W/C ratio (40%) and CNF ratio (0.05%).

Batch #10				**casting date:**	**6/9/17**							
Age (days)	**Date**	**Time**	**T (F)**	**RH (%)**	**Ref. Bar (in)**	**Spe. No.7(in)**	**Change (in)**	**shrinkage (%)**	**Spe. No.8(in)**	**Change (in)**	**shrinkage (%)**	**Ave.Shr. (%)**
1	6/10/17	11:20			0.2	0.1762	-0.0238		0.183	-0.017		
3	6/12/17	11:40	75	68	0.2	0.168	-0.032	-0.08	0.1748	-0.0252	-0.08	-0.08
5	6/14/17	11:40	73	45	0.2	0.161	-0.039	-0.15	0.1688	-0.0312	-0.14	-0.15
7	6/16/17	9:55	70	52	0.2	0.157	-0.043	-0.20	0.1639	-0.0361	-0.19	-0.19
11	6/20/17	11:25	72	67	0.2	0.1547	-0.0453	-0.22	0.1617	-0.0383	-0.22	-0.22
14	6/23/17	5:30	72	63	0.2	0.152	-0.048	-0.25	0.1591	-0.0409	-0.24	-0.24
21	6/30/17	2:15	71	63	0.2	0.1489	-0.0511	-0.28	0.156	-0.044	-0.27	-0.28
28	7/7/17	5:30	75	62	0.2	0.1467	-0.0533	-0.30	0.1542	-0.0458	-0.29	-0.30
90	9/7/17	9:00	70	62	0.2	0.1413	-0.0587	-0.35	0.1484	-0.0516	-0.35	-0.35

Table 25. Group (2), when W/C ratio (40%) and CNF ratio (0.1%).

Batch #11			casting date: 5/22/17									
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/23/17	9:30			0.2	0.1865	-0.0135		0.1908	-0.0092		
3	5/25/17	9:45	70	51	0.2	0.1775	-0.0225	-0.09	0.1822	-0.0178	-0.09	-0.09
5	5/27/17	11:20	67	54	0.2	0.172	-0.028	-0.15	0.177	-0.023	-0.14	-0.14
7	5/29/17	12:00	69	54	0.2	0.1686	-0.0314	-0.18	0.1738	-0.0262	-0.17	-0.18
11	6/2/17	11:40	70	56	0.2	0.1651	-0.0349	-0.22	0.1698	-0.0302	-0.21	-0.22
14	6/5/17	9:30	67	60	0.2	0.1623	-0.0377	-0.25	0.1671	-0.0329	-0.24	-0.24
21	6/12/17	11:40	75	68	0.2	0.1606	-0.0394	-0.26	0.165	-0.035	-0.26	-0.26
28	6/19/17	9:50	71	67	0.2	0.1576	-0.0424	-0.29	0.1619	-0.0381	-0.29	-0.29
90	8/20/17	11:25	73	63	0.2	0.1514	-0.0486	-0.36	0.1558	-0.0442	-0.36	-0.36

Table 26. Group (2), when W/C ratio (40%) and CNF ratio (0.2%).

Batch #12			casting date: 5/26/17									
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/27/17	10:45			0.2	0.1764	-0.0236		0.1858	-0.0142		
3	5/29/17	12:00	69	54	0.2	0.1686	-0.0314	-0.08	0.1782	-0.0218	-0.08	-0.08
5	5/31/17	10:10	68	59	0.2	0.1626	-0.0374	-0.14	0.1723	-0.0277	-0.14	-0.14
7	6/2/17	11:40	70	56	0.2	0.1593	-0.0407	-0.17	0.1682	-0.0318	-0.18	-0.18
11	6/6/17	10:10	67	56	0.2	0.1542	-0.0458	-0.23	0.1635	-0.0365	-0.23	-0.23
14	6/9/17	11:15	69	57	0.2	0.1517	-0.0483	-0.25	0.161	-0.039	-0.25	-0.25
21	6/16/17	11:05	70	52	0.2	0.1473	-0.0527	-0.30	0.1567	-0.0433	-0.30	-0.30
28	6/23/17	5:30	72	63	0.2	0.1472	-0.0528	-0.30	0.1567	-0.0433	-0.30	-0.30
90	8/24/17	5:10	73	55	0.2	0.1415	-0.0585	-0.35	0.1508	-0.0492	-0.36	-0.36

Table 27.Group (2), when W/C ratio (40%) and CNF ratio (0.5%).

Batch #13			casting date: 5/31/17									
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/1/17	10:00			0.2	0.1901	-0.0099		0.1913	-0.0087		
3	6/3/17	11:55	68	60	0.2	0.181	-0.019	-0.09	0.1822	-0.0178	-0.09	-0.09
5	6/5/17	9:30	67	60	0.2	0.1757	-0.0243	-0.15	0.177	-0.023	-0.15	-0.15
7	6/7/17	12:00	69	59	0.2	0.1715	-0.0285	-0.19	0.1731	-0.0269	-0.18	-0.19
11	6/11/17	10:30	71	64	0.2	0.1668	-0.0332	-0.24	0.1682	-0.0318	-0.23	-0.24
14	6/14/17	11:40	73	45	0.2	0.1631	-0.0369	-0.27	0.1649	-0.0351	-0.27	-0.27
21	6/21/17	10:10	72	67	0.2	0.1618	-0.0382	-0.29	0.1637	-0.0363	-0.28	-0.28
28	6/28/17	11:50	72	59	0.2	0.1588	-0.0412	-0.32	0.1606	-0.0394	-0.31	-0.31
90	8/29/17	9:00	72	52	0.2	0.152	-0.048	-0.39	0.1529	-0.0471	-0.39	-0.39

Table 28. Group (2), when W/C ratio (40%) and CNF ratio (1%).

Batch #14			casting date: 6/5/17									
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/6/17	9:35			0.2	0.1786	-0.0214		0.188	-0.012		
3	6/8/17	10:55	71	60	0.2	0.1684	-0.0316	-0.10	0.1783	-0.0217	-0.10	-0.10
5	6/10/17	11:25	70	54	0.2	0.1611	-0.0389	-0.18	0.1695	-0.0305	-0.19	-0.18
7	6/12/17	11:45	75	68	0.2	0.1588	-0.0412	-0.20	0.167	-0.033	-0.21	-0.21
11	6/16/17	11:10	70	52	0.2	0.151	-0.049	-0.28	0.1599	-0.0401	-0.29	-0.28
14	6/19/17	9:55	71	67	0.2	0.151	-0.049	-0.28	0.1598	-0.0402	-0.29	-0.28
21	6/26/17	11:35	73	62	0.2	0.148	-0.052	-0.31	0.157	-0.043	-0.31	-0.31
28	7/3/17	11:25	73	66	0.2	0.1469	-0.0531	-0.32	0.1556	-0.0444	-0.33	-0.33
90	9/3/17	11:30	70	56	0.2	0.1391	-0.0609	-0.40	0.1485	-0.0515	-0.40	-0.40

Table 29. Group (2), when W/C ratio (40%) and CNF ratio (1.5%).

Batch #15				casting date: 5/31/17								
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/1/17	10:00			0.2	0.1838	-0.0162		0.192	-0.008		
3	6/3/17	11:55	68	60	0.2	0.1725	-0.0275	-0.11	0.1812	-0.0188	-0.11	-0.11
5	6/5/17	6:30	68	57	0.2	0.1655	-0.0345	-0.19	0.1739	-0.0261	-0.18	-0.18
7	6/7/17	12:05	69	59	0.2	0.1619	-0.0381	-0.22	0.1701	-0.0299	-0.22	-0.22
11	6/11/17	10:35	71	64	0.2	0.1573	-0.0427	-0.27	0.1658	-0.0342	-0.27	-0.27
14	6/14/17	11:40	73	45	0.2	0.154	-0.046	-0.30	0.1622	-0.0378	-0.30	-0.30
21	6/21/17	10:15	72	67	0.2	0.153	-0.047	-0.31	0.1615	-0.0385	-0.31	-0.31
28	6/28/17	11:50	72	59	0.2	0.1499	-0.0501	-0.34	0.1583	-0.0417	-0.34	-0.34
90	8/29/17	9:00	72	52	0.2	0.142	-0.058	-0.42	0.1502	-0.0498	-0.42	-0.42

Table 30. Group (2), when W/C ratio (40%) and CNF ratio (3%).

Batch #16				casting date: 6/7/17								
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/8/17	10:30			0.2	0.1992	-0.0008		0.1993	-0.0007		
3	6/10/17	11:30	70	54	0.2	0.1856	-0.0144	-0.14	0.1854	-0.0146	-0.14	-0.14
5	6/12/17	11:50	75	68	0.2	0.1795	-0.0205	-0.20	0.1793	-0.0207	-0.20	-0.20
7	6/14/17	11:45	73	45	0.2	0.1733	-0.0267	-0.26	0.1732	-0.0268	-0.27	-0.26
11	6/18/17	12:00	70	62	0.2	0.1695	-0.0305	-0.30	0.1695	-0.0305	-0.30	-0.30
14	6/21/17	10:20	72	67	0.2	0.1691	-0.0309	-0.31	0.169	-0.031	-0.31	-0.31
21	6/28/17	12:10	72	59	0.2	0.1644	-0.0356	-0.35	0.1643	-0.0357	-0.36	-0.35
28	7/5/17	1:30	75	61	0.2	0.1628	-0.0372	-0.37	0.1629	-0.0371	-0.37	-0.37
90	9/5/17	11:30	70	56	0.2	0.1552	-0.0448	-0.45	0.1553	-0.0447	-0.45	-0.45

Table 31. Group (3), when W/C ratio (45%) and CNF ratio (0.00%).

Batch #17				casting date: 5/25/17								
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/26/17	9:30			0.2	0.19	-0.01		0.1998	-0.0002		
3	5/28/17	4:50	69	55	0.2	0.1834	-0.0166	-0.07	0.193	-0.007	-0.07	-0.07
5	5/30/17	12:50	69	55	0.2	0.1774	-0.0226	-0.13	0.1874	-0.0126	-0.13	-0.13
7	6/1/17	11:30	68	62	0.2	0.1744	-0.0256	-0.16	0.1842	-0.0158	-0.16	-0.16
11	6/5/17	6:35	68	57	0.2	0.1692	-0.0308	-0.21	0.1793	-0.0207	-0.21	-0.21
14	6/8/17	11:00	71	60	0.2	0.1665	-0.0335	-0.24	0.1764	-0.0236	-0.24	-0.24
21	6/15/17	9:30	71	45	0.2	0.1618	-0.0382	-0.29	0.1715	-0.0285	-0.29	-0.29
28	6/22/17	10:15	73	64	0.2	0.1613	-0.0387	-0.29	0.1712	-0.0288	-0.29	-0.29
90	8/23/17	4:00	75	63	0.2	0.1558	-0.0442	-0.35	0.1658	-0.0342	-0.35	-0.35

Table 32. Group (3), when W/C ratio (45%) and CNF ratio (0.05%).

Batch #18				casting date: 5/26/17								
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/27/17	11:00			0.2	0.1878	-0.0122		0.1824	-0.0176		
3	5/29/17	12:00	69	54	0.2	0.182	-0.018	-0.06	0.176	-0.024	-0.07	-0.06
5	5/31/17	10:15	68	59	0.2	0.1761	-0.0239	-0.12	0.1697	-0.0303	-0.13	-0.12
7	6/2/17	11:45	69	56	0.2	0.1725	-0.0275	-0.16	0.1663	-0.0337	-0.16	-0.16
11	6/6/17	10:15	67	56	0.2	0.1678	-0.0322	-0.20	0.1611	-0.0389	-0.22	-0.21
14	6/9/17	11:20	69	57	0.2	0.1645	-0.0355	-0.24	0.1584	-0.0416	-0.24	-0.24
21	6/16/17	11:15	70	52	0.2	0.1601	-0.0399	-0.28	0.1537	-0.0463	-0.29	-0.29
28	6/23/17	5:35	72	63	0.2	0.1599	-0.0401	-0.28	0.1532	-0.0468	-0.30	-0.29
90	8/24/17	5:15	73	55	0.2	0.1536	-0.0464	-0.35	0.1473	-0.0527	-0.36	-0.35

Table 33. Group (3), when W/C ratio (45%) and CNF ratio (0.1%).

Batch #19			casting date: 5/23/17									
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/24/17	9:00			0.2	0.1968	-0.0032		0.1615	-0.0385		
3	5/26/17	10:00	69	54	0.2	0.1899	-0.0101	-0.07	0.1563	-0.0437	-0.05	-0.06
5	5/28/17	4:50	69	55	0.2	0.1839	-0.0161	-0.13	0.1503	-0.0497	-0.11	-0.12
7	5/30/17	12:50	69	55	0.2	0.1798	-0.0202	-0.17	0.1463	-0.0537	-0.15	-0.16
11	6/3/17	12:00	68	60	0.2	0.1758	-0.0242	-0.21	0.1416	-0.0584	-0.20	-0.21
14	6/6/17	10:15	67	56	0.2	0.1732	-0.0268	-0.24	0.1398	-0.0602	-0.22	-0.23
21	6/13/17	12:40	77	70	0.2	0.1715	-0.0285	-0.26	0.1378	-0.0622	-0.24	-0.25
28	6/20/17	11:30	72	67	0.2	0.1688	-0.0312	-0.28	0.1353	-0.0647	-0.27	-0.28
90	8/21/17	11:30	73	63	0.2	0.1615	-0.0385	-0.36	0.128	-0.072	-0.34	-0.35

Table 34. Group (3), when W/C ratio (45%) and CNF ratio (0.2%).

Batch #20			casting date: 5/22/17									
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/23/17	9:30			0.2	0.1936	-0.0064		0.1937	-0.0063		
3	5/25/17	9:50	70	51	0.2	0.1878	-0.0122	-0.06	0.1884	-0.0116	-0.05	-0.06
5	5/27/17	11:20	67	54	0.2	0.1819	-0.0181	-0.12	0.182	-0.018	-0.12	-0.12
7	5/29/17	12:05	69	54	0.2	0.1792	-0.0208	-0.15	0.1782	-0.0218	-0.16	-0.15
11	6/2/17	11:45	70	56	0.2	0.1748	-0.0252	-0.19	0.174	-0.026	-0.20	-0.20
14	6/5/17	6:40	68	57	0.2	0.1713	-0.0287	-0.23	0.171	-0.029	-0.23	-0.23
21	6/12/17	11:55	75	68	0.2	0.1695	-0.0305	-0.24	0.1688	-0.0312	-0.25	-0.25
28	6/19/17	10:00	71	67	0.2	0.1655	-0.0345	-0.29	0.1656	-0.0344	-0.29	-0.29
90	8/20/17	11:30	70	63	0.2	0.159	-0.041	-0.35	0.159	-0.041	-0.35	-0.35

Table 35. Group (3), when W/C ratio (45%) and CNF ratio (0.5%).

B#21				casting date: 5/19/17								
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/20/17	8:30			0.2	0.1928	-0.0072		0.1726	-0.0274		
3	5/22/17	12:00	72	45	0.2	0.1839	-0.0161	-0.09	0.1634	-0.0366	-0.09	-0.09
5	5/24/17	1:30	71	50	0.2	0.1767	-0.0233	-0.16	0.1559	-0.0441	-0.17	-0.17
7	5/26/17	10:10	69	54	0.2	0.1731	-0.0269	-0.20	0.1523	-0.0477	-0.21	-0.20
11	5/30/17	12:55	69	55	0.2	0.1698	-0.0302	-0.23	0.1495	-0.0505	-0.23	-0.23
14	6/2/17	11:50	70	56	0.2	0.1694	-0.0306	-0.24	0.1489	-0.0511	-0.24	-0.24
21	6/9/17	11:20	69	57	0.2	0.1653	-0.0347	-0.28	0.1449	-0.0551	-0.28	-0.28
28	6/16/17	11:15	70	52	0.2	0.1624	-0.0376	-0.31	0.1419	-0.0581	-0.31	-0.31
90	8/17/17	12:00	72	54	0.2	0.157	-0.043	-0.36	0.1366	-0.0634	-0.37	-0.36

Table 36. Group (3), when W/C ratio (45%) and CNF ratio (1%).

Batch #22				casting date: 5/18/17								
Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/19/17	10:00			0.2	0.1905	-0.0095		0.1764	-0.0236		
3	5/21/17	10:00	71	44	0.2	0.1814	-0.0186	-0.09	0.1678	-0.0322	-0.09	-0.09
5	5/23/17	2:30	72	50	0.2	0.174	-0.026	-0.17	0.1603	-0.0397	-0.16	-0.17
7	5/25/17	10:00	70	51	0.2	0.1704	-0.0296	-0.20	0.1567	-0.0433	-0.20	-0.20
11	5/29/17	12:10	69	54	0.2	0.1679	-0.0321	-0.23	0.1539	-0.0461	-0.23	-0.23
14	6/1/17	11:40	68	62	0.2	0.1674	-0.0326	-0.23	0.1537	-0.0463	-0.23	-0.23
21	6/8/17	11:10	71	60	0.2	0.1649	-0.0351	-0.26	0.1505	-0.0495	-0.26	-0.26
28	6/15/17	9:35	71	45	0.2	0.1619	-0.0381	-0.29	0.1475	-0.0525	-0.29	-0.29
90	8/16/17	11:50	73	64	0.2	0.1573	-0.0427	-0.34	0.1431	-0.0569	-0.34	-0.34

Table 37. Group (3), when W/C ratio (45%) and CNF ratio (1.5%).

Batch #23 **casting date: 5/17/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/18/17	9:30			0.2	0.1847	-0.0153		0.1547	-0.0453		
3	5/20/17	9:00	73	44	0.2	0.1748	-0.0252	-0.10	0.1448	-0.0552	-0.10	-0.10
5	5/22/17	11:30	72	45	0.2	0.1678	-0.0322	-0.17	0.1379	-0.0621	-0.17	-0.17
7	5/24/17	1:30	71	50	0.2	0.1643	-0.0357	-0.21	0.1341	-0.0659	-0.21	-0.21
11	5/28/17	5:00	69	55	0.2	0.1616	-0.0384	-0.23	0.1317	-0.0683	-0.23	-0.23
14	5/31/17	10:20	68	59	0.2	0.1595	-0.0405	-0.26	0.1292	-0.0708	-0.26	-0.26
21	6/7/17	12:05	69	59	0.2	0.1567	-0.0433	-0.28	0.1268	-0.0732	-0.28	-0.28
28	6/14/17	11:45	73	45	0.2	0.1541	-0.0459	-0.31	0.1238	-0.0762	-0.31	-0.31
90	8/15/17	12:37	75	53	0.2	0.1477	-0.0523	-0.38	0.1179	-0.0821	-0.37	-0.37

Table 38. Group (3), when W/C ratio (45%) and CNF ratio (0.05%).

Batch #24 **casting date: 5/30/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/31/17	10:00			0.2	0.1959	-0.0041		0.1945	-0.0055		
3	6/2/17	11:55	70	56	0.2	0.186	-0.014	-0.10	0.1845	-0.0155	-0.10	-0.10
5	6/4/17	11:45	68	60	0.2	0.1787	-0.0213	-0.17	0.1783	-0.0217	-0.16	-0.17
7	6/6/17	10:20	67	56	0.2	0.1737	-0.0263	-0.23	0.1719	-0.0281	-0.23	-0.23
11	6/10/17	11:45	70	54	0.2	0.1672	-0.0328	-0.29	0.1658	-0.0342	-0.29	-0.29
14	6/13/17	12:40	77	70	0.2	0.1671	-0.0329	-0.29	0.1655	-0.0345	-0.29	-0.29
21	6/20/17	11:30	72	67	0.2	0.1632	-0.0368	-0.33	0.1616	-0.0384	-0.33	-0.33
28	6/27/17	5:45	73	55	0.2	0.1597	-0.0403	-0.37	0.1581	-0.0419	-0.37	-0.37
90	8/28/17	2:00	72	52	0.2	0.1516	-0.0484	-0.45	0.1505	-0.0495	-0.45	-0.45

Table 39. Group (4), when W/C ratio (50%) and CNF ratio (0.00%).

Batch #25 **casting date: 5/29/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/30/17	10:45			0.2	0.1837	-0.0163		0.1947	-0.0053		
3	6/1/17	11:40	68	62	0.2	0.1804	-0.0196	-0.03	0.1911	-0.0089	-0.04	-0.04
5	6/3/17	12:00	68	60	0.2	0.1744	-0.0256	-0.09	0.1855	-0.0145	-0.09	-0.09
7	6/5/17	6:40	68	57	0.2	0.1695	-0.0305	-0.14	0.1814	-0.0186	-0.14	-0.14
11	6/9/17	11:20	69	57	0.2	0.1634	-0.0366	-0.21	0.1749	-0.0251	-0.20	-0.20
14	6/12/17	11:55	75	68	0.2	0.1622	-0.0378	-0.22	0.174	-0.026	-0.21	-0.21
21	6/19/17	10:05	71	67	0.2	0.1578	-0.0422	-0.26	0.1699	-0.0301	-0.25	-0.26
28	6/26/17	11:40	73	62	0.2	0.1555	-0.0445	-0.29	0.1668	-0.0332	-0.28	-0.28
90	8/27/17	2:00	72	52	0.2	0.1477	-0.0523	-0.37	0.1583	-0.0417	-0.37	-0.37

Table 40. Group (4), when W/C ratio (50%) and CNF ratio (0.05%).

Batch #26 **casting date: 6/1/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	6/2/17	9:30			0.2	0.191	-0.009		0.1892	-0.0108		
3	6/4/17	11:55	68	60	0.2	0.1884	-0.0116	-0.03	0.1863	-0.0137	-0.03	-0.03
5	6/6/17	10:21	67	56	0.2	0.1817	-0.0183	-0.09	0.18	-0.02	-0.09	-0.09
7	6/8/17	11:10	71	60	0.2	0.1767	-0.0233	-0.15	0.1745	-0.0255	-0.15	-0.15
11	6/12/17	11:55	75	68	0.2	0.1716	-0.0284	-0.20	0.1693	-0.0307	-0.20	-0.20
14	6/15/17	9:35	71	41	0.2	0.1668	-0.0332	-0.25	0.1651	-0.0349	-0.24	-0.25
21	6/22/17	10:20	73	64	0.2	0.1646	-0.0354	-0.27	0.1625	-0.0375	-0.27	-0.27
28	6/29/17	12:10	72	61	0.2	0.1616	-0.0384	-0.30	0.1598	-0.0402	-0.30	-0.30
90	8/30/17	1:00	72	55	0.2	0.1519	-0.0481	-0.40	0.1535	-0.0465	-0.36	-0.38

Table 41. Group (4), when W/C ratio (50%) and CNF ratio (0.1%).

Batch #27 **casting date: 5/30/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/31/17	10:00			0.2	0.1856	-0.0144		0.1862	-0.0138		
3	6/2/17	12:00	70	56	0.2	0.1823	-0.0177	-0.03	0.1833	-0.0167	-0.03	-0.03
5	6/4/17	12:00	68	60	0.2	0.1767	-0.0233	-0.09	0.1773	-0.0227	-0.09	-0.09
7	6/6/17	10:25	67	56	0.2	0.172	-0.028	-0.14	0.1725	-0.0275	-0.14	-0.14
11	6/10/17	12:00	70	54	0.2	0.165	-0.035	-0.21	0.1656	-0.0344	-0.21	-0.21
14	6/13/17	12:40	77	70	0.2	0.1642	-0.0358	-0.22	0.1647	-0.0353	-0.22	-0.22
21	6/20/17	11:35	72	67	0.2	0.1602	-0.0398	-0.26	0.1608	-0.0392	-0.26	-0.26
28	6/27/17	5:50	73	55	0.2	0.1565	-0.0435	-0.30	0.157	-0.043	-0.30	-0.30
90	8/28/17	2:00	72	52	0.2	0.1492	-0.0508	-0.37	0.1499	-0.0501	-0.37	-0.37

Table 42. Group (4), when W/C ratio (50%) and CNF ratio (0.2%).

Batch #28 **casting date: 5/25/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/26/17	9:45			0.2	0.1622	-0.0378		0.1843	-0.0157		
3	5/28/17	5:00	69	55	0.2	0.1575	-0.0425	-0.05	0.1799	-0.0201	-0.04	-0.05
5	5/30/17	1:00	69	55	0.2	0.1513	-0.0487	-0.11	0.174	-0.026	-0.10	-0.11
7	6/1/17	11:45	68	62	0.2	0.1491	-0.0509	-0.13	0.1712	-0.0288	-0.13	-0.13
11	6/5/17	6:45	68	57	0.2	0.1432	-0.0568	-0.19	0.1652	-0.0348	-0.19	-0.19
14	6/8/17	11:15	71	60	0.2	0.1401	-0.0599	-0.22	0.1619	-0.0381	-0.23	-0.23
21	6/15/17	9:40	71	45	0.2	0.135	-0.065	-0.28	0.157	-0.043	-0.28	-0.28
28	6/22/17	10:25	73	64	0.2	0.134	-0.066	-0.29	0.1561	-0.0439	-0.29	-0.29
90	8/23/17	4:05	75	63	0.2	0.1289	-0.0711	-0.34	0.1505	-0.0495	-0.34	-0.34

Table 43. Group (4), when W/C ratio (50%) and CNF ratio (0.5%).

Batch #29 **casting date: 5/24/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/25/17	9:30			0.2	0.1785	-0.0215		0.1799	-0.0201		
3	5/27/17	11:25	67	54	0.2	0.174	-0.026	-0.05	0.175	-0.025	-0.05	-0.05
5	5/29/17	12:15	69	54	0.2	0.1682	-0.0318	-0.10	0.168	-0.032	-0.12	-0.11
7	5/31/17	10:20	68	59	0.2	0.163	-0.037	-0.16	0.1635	-0.0365	-0.17	-0.16
11	6/4/17	12:05	68	60	0.2	0.159	-0.041	-0.20	0.1585	-0.0415	-0.22	-0.21
14	6/7/17	12:10	69	59	0.2	0.1555	-0.0445	-0.23	0.1552	-0.0448	-0.25	-0.24
21	6/14/17	11:50	73	45	0.2	0.151	-0.049	-0.28	0.1507	-0.0493	-0.30	-0.29
28	6/21/17	10:20	72	67	0.2	0.1508	-0.0492	-0.28	0.1506	-0.0494	-0.30	-0.29
90	8/22/17	1:50	75	65	0.2	0.144	-0.056	-0.35	0.144	-0.056	-0.36	-0.36

Table 44. Group (4), when W/C ratio (50%) and CNF ratio (1%).

Batch #30 **casting date: 5/23/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/24/17				0.2	0.1777	-0.0223		0.1805	-0.0195		
3	5/26/17	10:20	69	54	0.2	0.1695	-0.0305	-0.08	0.1727	-0.0273	-0.08	-0.08
5	5/28/17	5:05	69	55	0.2	0.1592	-0.0408	-0.19	0.1645	-0.0355	-0.16	-0.18
7	5/30/17	1:05	69	55	0.2	0.1564	-0.0436	-0.22	0.1598	-0.0402	-0.21	-0.21
11	6/3/17	12:05	68	62	0.2	0.1506	-0.0494	-0.28	0.1539	-0.0461	-0.27	-0.27
14	6/6/17	10:30	67	56	0.2	0.1475	-0.0525	-0.31				
21	6/13/17	12:45	77	70	0.2	0.1475	-0.0525	-0.31				
28	6/20/17	11:40	72	67	0.2	0.1448	-0.0552	-0.33				
90	8/21/17	11:30	73	63	0.2	0.1391	-0.0609	-0.39				

Table 45. Group (4), when W/C ratio (50%) and CNF ratio (1.5%).

Batch #31 **casting date: 5/19/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/20/17	9:00			0.2	0.193	-0.007		0.1899	-0.0101		
3	5/22/17	12:00	72	45	0.2	0.1847	-0.0153	-0.08	0.1809	-0.0191	-0.09	-0.09
5	5/24/17	1:30	71	50	0.2	0.1774	-0.0226	-0.16	0.174	-0.026	-0.16	-0.16
7	5/26/17	10:30	69	54	0.2	0.1732	-0.0268	-0.20	0.1694	-0.0306	-0.21	-0.20
11	5/30/17	1:10	69	55	0.2	0.1682	-0.0318	-0.25	0.1644	-0.0356	-0.26	-0.26
14	6/2/17	12:00	70	56	0.2	0.1654	-0.0346	-0.28	0.1618	-0.0382	-0.29	-0.28
21	6/9/17	11:25	69	57	0.2	0.1608	-0.0392	-0.33	0.158	-0.042	-0.32	-0.33
28	6/16/17	11:20	70	52	0.2	0.1587	-0.0413	-0.35	0.1544	-0.0456	-0.36	-0.35
90	8/17/17	12:05	72	54	0.2	0.1549	-0.0451	-0.39	0.1517	-0.0483	-0.39	-0.39

Table 46. Group (4), when W/C ratio (50%) and CNF ratio (3%).

Batch #32 **casting date: 5/17/17**

Age (days)	Date	Time	T (F)	RH (%)	Ref. Bar (in)	Spe. No.7(in)	Change (in)	shrinkage (%)	Spe. No.8(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	5/18/17	9:30			0.2	0.1877	-0.0123		0.166	-0.034		
3	5/20/17	9:00	73	44	0.2	0.1755	-0.0245	-0.12	0.1545	-0.0455	-0.12	-0.12
5	5/22/17	11:30	72	45	0.2	0.1676	-0.0324	-0.20	0.1469	-0.0531	-0.19	-0.20
7	5/24/17	1:30	71	50	0.2	0.163	-0.037	-0.25	0.143	-0.057	-0.23	-0.24
11	5/28/17	5:10	69	55	0.2	0.1582	-0.0418	-0.30	0.1376	-0.0624	-0.29	-0.29
14	5/31/17	10:25	68	59	0.2	0.155	-0.045	-0.33	0.1337	-0.0663	-0.33	-0.33
21	6/7/17	12:15	69	59	0.2	0.1512	-0.0488	-0.37	0.1305	-0.0695	-0.36	-0.37
28	6/14/17	11:50	73	45	0.2	0.1482	-0.0518	-0.40	0.127	-0.073	-0.40	-0.40
90	8/15/17	12:44	75	53	0.2	0.1452	-0.0548	-0.43	0.124	-0.076	-0.43	-0.43

APPENDIX (B). AUTOGENOUS SHRINKAGE TESTS

In these tables, all the results for shrinkage specimens for each batch at each age, the weights of the specimens at age 1 day and age 28 day were recorded, also the temperature were recorded for each age for phase two (Autogenous shrinkage tests).

Table 47. Autogenous shrinkage results for batch (A), when W/C ratio (30%), and CNF ratio is 0.00 %.

Batch :A

casting day	w/c%	CNF%
9/20/17	30	0

Specimens 1&2, (ASTM C157), sealed specemens

	The weight of the specimens (g)	
	after 1 day	after 28 days
Spe. No.1	392.3	392
Spe. No.2	407.7	407.4

Age	Date	Time	T (deg)	Ref. Bar (in)	Specimen _1 (in)	Change ()	shrinkage	Specimen _2(in)	Change ()	shrinkage	Ave.Shr.
1	9/21/17	12:30		0.2	0.1777	-0.0223		0.1571	-0.0429		
2	9/22/17	1:00		0.2	0.1736	-0.0264	-0.04	0.1528	-0.0472	-0.04	-0.04
3	9/23/17	1:30	69	0.2	0.1715	-0.0285	-0.06	0.1508	-0.0492	-0.06	-0.06
4	9/24/17	2:00	72	0.2	0.1705	-0.0295	-0.07	0.1498	-0.0502	-0.07	-0.07
5	9/25/17	3:00	72	0.2	0.1699	-0.0301	-0.08	0.1492	-0.0508	-0.08	-0.08
7	9/27/17	3:30	73	0.2	0.1688	-0.0312	-0.09	0.148	-0.052	-0.09	-0.09
9	9/29/17	2:00	69	0.2	0.1678	-0.0322	-0.10	0.1468	-0.0532	-0.10	-0.10
11	10/1/17	2:30	63	0.2	0.1667	-0.0333	-0.11	0.1458	-0.0542	-0.11	-0.11
14	10/4/17	2:00	70	0.2	0.1676	-0.0324	-0.10	0.1466	-0.0534	-0.11	-0.10
21	10/11/17	2:00	70	0.2	0.1667	-0.0333	-0.11	0.146	-0.054	-0.11	-0.11
28	10/18/17	12:00	70	0.2	0.1664	-0.0336	-0.11	0.1456	-0.0544	-0.12	-0.11

Table 48. Autogenous shrinkage results for batch (B), when W/C ratio (30%), and CNF ratio is 0.1 %.

Batch :B	casting day	w/c%	CNF%
	9/20/17	30	0.1

Specimens 1&2 - (ASTM C157), sealed specemens

	The weight of the specimens (g)	
	after 1 day	after 28 days
Spe. No.1	402.7	402.4
Spe. No.2	388.9	388.6

Age (days)	Date	Time	T (F)	Ref. Bar (in)	Spe. No.1(in)	Change (in)	shrinkage (%)	Spe. No.2(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	9/21/17	12:45		0.2	0.146	-0.054		0.1805	-0.0195		
2	9/22/17	1:30		0.2	0.1415	-0.0585	-0.05	0.1761	-0.0239	-0.04	-0.04
3	9/23/17	1:30	69	0.2	0.1397	-0.0603	-0.06	0.1742	-0.0258	-0.06	-0.06
4	9/24/17	2:00	72	0.2	0.139	-0.061	-0.07	0.1733	-0.0267	-0.07	-0.07
5	9/25/17	3:00	72	0.2	0.1385	-0.0615	-0.07	0.1727	-0.0273	-0.08	-0.08
7	9/27/17	3:30	73	0.2	0.1377	-0.0623	-0.08	0.172	-0.028	-0.09	-0.08
9	9/29/17	2:00	69	0.2	0.1367	-0.0633	-0.09	0.1709	-0.0291	-0.10	-0.09
11	10/1/17	2:30	63	0.2	0.1358	-0.0642	-0.10	0.17	-0.03	-0.11	-0.10
14	10/4/17	2:00	70	0.2	0.1365	-0.0635	-0.09	0.1707	-0.0293	-0.10	-0.10
21	10/11/17	2:00	70	0.2	0.1359	-0.0641	-0.10	0.1702	-0.0298	-0.10	-0.10
28	10/18/17	12:00	70	0.2	0.1358	-0.0642	-0.10	0.1698	-0.0302	-0.11	-0.10

Table 49. Autogenous shrinkage results for batch (C), when W/C ratio (30%), and CNF ratio is 0.2%.

Batch :C	casting day	w/c%	CNF%
	9/22/17	30	0.2

Specimens 1&2, (ASTM C157), sealed specemens

	The weight of the specimens (g)	
	after 1 day	after 28 days
Spe. No.1	395.4	395
Spe. No.2	399.9	399.6

Age (days)	Date	Time	T (F)	Ref. Bar (in)	Spe. No.1(in)	Change (in)	shrinkage (%)	Spe. No.2(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	9/23/17	2:10		0.2	0.1988	-0.0012		0.1971	-0.0029		
2	9/24/17	2:00	72	0.2	0.1948	-0.0052	-0.04	0.193	-0.007	-0.04	-0.04
3	9/25/17	3:00	72	0.2	0.1921	-0.0079	-0.07	0.1908	-0.0092	-0.06	-0.07
4	9/26/17	3:00	73	0.2	0.1911	-0.0089	-0.08	0.1895	-0.0105	-0.08	-0.08
5	9/27/17	3:30	73	0.2	0.1902	-0.0098	-0.09	0.1888	-0.0112	-0.08	-0.08
7	9/29/17	2:00	69	0.2	0.1888	-0.0112	-0.10	0.1872	-0.0128	-0.10	-0.10
9	10/1/17	2:30	63	0.2	0.1878	-0.0122	-0.11	0.1863	-0.0137	-0.11	-0.11
11	10/3/17	11:00	67	0.2	0.1882	-0.0118	-0.11	0.1867	-0.0133	-0.10	-0.11
14	10/6/17	2:00	70	0.2	0.1879	-0.0121	-0.11	0.1867	-0.0133	-0.10	-0.11
21	10/13/17	1:00	70	0.2	0.187	-0.013	-0.12	0.1856	-0.0144	-0.12	-0.12
28	10/20/17	1:30	70	0.2	0.1865	-0.0135	-0.12	0.1853	-0.0147	-0.12	-0.12

Table 50.Autogenous shrinkage results for batch (D), when W/C ratio (30%), and CNF ratio is 0.5%.

Batch :D	casting day	w/c%	CNF%
	9/22/17	30	0.5

Specimens 1&2, (ASTM C157), sealed specemens

	The weight of the specimens (g)	
	after 1 day	after 28 days
Spe. No.1	400.1	
Spe. No.2	386.5	

Age (days)	Date	Time	T (F)	Ref. Bar (in)	Spe. No.1(in)	Change (in)	shrinkage (%)	Spe. No.2(in)	Change (in)	shrinkage (%)	Ave.Shr. (%)
1	9/23/17	3:00		0.2	0.1671	-0.0329		0.1468	-0.0532		
2	9/24/17	2:00	72	0.2	0.1632	-0.0368	-0.04	0.1428	-0.0572	-0.04	-0.04
3	9/25/17	3:00	72	0.2	0.1611	-0.0389	-0.06	0.1408	-0.0592	-0.06	-0.06
4	9/26/17	3:00	73	0.2	0.16	-0.04	-0.07	0.1397	-0.0603	-0.07	-0.07
5	9/27/17	3:30	73	0.2	0.1593	-0.0407	-0.08	0.1388	-0.0612	-0.08	-0.08
7	9/29/17	2:00	69	0.2	0.1578	-0.0422	-0.09	0.1375	-0.0625	-0.09	-0.09
9	10/1/17	2:30	63	0.2	0.1567	-0.0433	-0.10	0.1364	-0.0636	-0.10	-0.10
11	10/3/17	11:00	67	0.2	0.1572	-0.0428	-0.10	0.1368	-0.0632	-0.10	-0.10
14	10/6/17	2:00	70	0.2	0.157	-0.043	-0.10	0.1367	-0.0633	-0.10	-0.10
21	10/13/17	1:00	70	0.2	0.156	-0.044	-0.11	0.1358	-0.0642	-0.11	-0.11
28	10/20/17	1:30	70	0.2	0.156	-0.044	-0.11	0.1356	-0.0644	-0.11	-0.11

BIOGRAPHY OF THE AUTHOR

Mohammed Ahmed was born in Al-Anbar, Iraq on November 11, 1986. He graduated from high school in 2004. At 2008, He graduated from the Civil Engineering Department, the University of Tikrit, with a Bachelor's degree. He had worked as a site engineer and office engineer in several local and international companies in Iraq. At 2012, he was nominated for a scholarship by the Iraqi government (the prime minister office). Mohammed Ahmed is a candidate for the Master of Science degree in Civil Engineering from the University of Maine in December 2017.