EFFECT OF CARBON NANO AND MICROFIBERS ON THE MECHANICAL PROPERTIES AND DURABILITY OF CEMENT PASTES

By

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LIST OF ABBREVIATIONS

ACI- American Concrete Institute

AN- Ammonium Nitrate

ASTM- American Society for Testing and Materials

CF- Carbon Microfibers

CH- Calcium Hydroxide

CNF- Carbon Nanofibers

C_3A- Tricalcium Aluminate

C_2S- Dicalcium Silicate

C_3S- Tricalcium Silicate

CNT- Carbon Nanotubes

C-S-H- Calcium Silicate Hydrate

DI- Deionized

ICP-MS- Inductively Coupled Mass Spectrometer

PAN- Polyacrylonitrile

ML- Minimum limit

MDL- Method detection limit

MWNT- Multi-walled nanotubes

SWNT- Single-walled nanotubes

w/c- Water to cement ratio

wt%- percent by mass of cement

CHAPTER I

. INTRODUCTION

As one of the most popular materials used in the world's infrastructure it is important that cement displays exceptional strength and durability because its failure results in high financial costs and the potential loss of millions of lives. To this end several efforts to improve its properties have been and continue to be studied and implemented. The use of fiber reinforcements is one such means (Brandt, 2008); the types of reinforcements currently used include steel, glass (Proctor, 1990), cellulose (Bilba et al., 2003) and carbon fibers (Shigeyuki et al., 1986; Katz et al., 1994; Ali et al., 1972; Chen et al., 2004). The fibers are used individually or in combination.

The properties of carbon microfibers (CF) such as their size, thermal stability, high strength, elastic modulus, and apparent chemical inertness make them an especially attractive option. In fact CF reinforced cement based materials have been shown to have improved tensile and flexural properties, low drying shrinkage, high specific heat, low thermal conductivity, high electrical conductivity, high corrosion resistance and weak thermoelectric behavior (Chung, 2000). Technological advancements have led to the development of carbon fibers with better properties than the CF; these fibers are referred to as carbon nanofibers (CNF) because of their nanoscale dimensions. CNF are smaller in size, have higher strengths and elastic moduli and therefore show promise as a reinforcement material in cement. Studies on the use of CNF as reinforcement in cement are however limited; work has however been done utilizing carbon nanotubes and has shown mixed results (Markar et al., 2005; Li et al., 2005).

This work studies the effects of CNF loading on the mechanical strengths (compressive and splitting tensile strengths) and durability of cement paste with respect to decalcification; in addition it compares the effects of CNF to those of CF on these properties. The results presented here are part of an overall research program on the long term performance and durability of CNF/CF reinforced cement based materials.

Chapter 2 provides a literature review of cement based materials, CNF and CF. Information is provided on the chemistry, mechanical properties and durability of cement based materials, and the properties of pozzolanic additives, CNF and CF and their effects on cement based materials. Chapter 4 presents the experimental approach and chapter 5 describes the methods and materials used to assess the properties of different cement pastes. Results and discussion are provided in chapter 6. Chapter 7 presents the conclusions from this study and chapter 8 provides some recommendations for future work.

CHAPTER II

BACKGROUND

This section provides background information on cement based materials, including their chemistry, mechanical properties and durability. In addition information is provided on the pozzolanic additives, CNF and CF and their effects on the mechanical properties and durability of cement pastes.

Portland Cement

The typical chemical composition of type I Portland cement is provided in table 2.1.

Table 2.1 Typical Chemical Composition of Type I Portland Cement (Cemex, 2008)

Component	**Composition (%)**
Silicon Dioxide (SiO_2)	19.4
Aluminum Oxide (AL_2O_3)	5.3
Ferric Oxide (Fe_2O_3)	3.6
Calcium Oxide (CaO)	63.0
Magnesium oxide (MgO)	2.7
Sulfur Trioxide (SO_3)	3.0
Loss on Ignition (LOI)	1.5
Insoluble Residue	0.42
Alkalies (Na_2O equivalent)	0.48
Tricalcium Silicate (C_3S)	60
Dicalcium Silicate (C_2S)	10
Tricalcium Aluminate (C_3A)	8
Tertracalcium Aluminoferrite (C_4AF)	11

Portland Cement Hydration Reactions

The hardening of cement paste is due to hydration. The conditions of this hydration play an integral role in the physical and chemical properties of the hardened paste.

Upon hydration, calcium silicates (C_2S and C_3S) undergo hydrolysis producing calcium hydroxide (CH) and calcium silicate hydrates (C-S-H) (E1 and E2). The chemical composition of the C-S-H varies with the hydration conditions and the age of the cement paste (Soroka, 1979).

$$2(3CaO.SiO_2) + 6H_2O \longrightarrow 3CaO.2SiO_2.3H_2O + 3Ca(OH)_2 \quad (E1)$$

$$2(2CaO.SiO_2) + 4H_2O \longrightarrow 3CaO.2SiO_2.3H_2O + Ca(OH)_2 \quad (E2)$$

In the presence of gypsum, the hydration of tricalcium aluminate (C_3A) produces needle like crystals of a high sulfate calcium sulfoaluminate called ettringite. This ettringite continues to form until all the sulfate ions have been removed at which point further hydration of C_3A results in the conversion of the ettringite into a low sulfate sulfoaluminate referred to as monosulfate (Soroka, 1979).

Initially the ferrite reacts with gypsum and calcium hydroxide to produce needle like crystals of a solid solution consisting of high sulfate sulfoaluminate and sulfoferrite which upon removal of all the sulfate ions convert to a low sulfate alumino-ferrite solid solution in which sulfate ions are replaced by hydroxide ions (E3) (Soroka, 1979).

$$4CaO.Al_2O_3.Fe_2O_3 + CaSO_4.2H_2O + CH \longrightarrow 3CaO(Al_2O_3,Fe_2O_3).3CaSO_4.aq \quad (E3)$$

Several factors influence the rate of hydration of cement: age, cement composition, cement fineness, water to cement (w/c) ratio, temperature and the use of admixtures. The rate of hydration of the varying cement constituents differs, tricalcium aluminate hydrates fastest followed by tricalcium silicate and dicalcium silicate (Lea,1935). The hydration rate of cement

increases with its fineness. The rate of hydration and the ultimate degree of hydration decrease with decreasing w/c ratio. The rate of hydration increases with temperature up to 100ºC however the ultimate degree of hydration is not affected by temperature. Different admixtures can be used to retard or accelerate the hydration process as necessary, one such admixture is gypsum which acts as a retarder.

The structure of hardened cement paste is highly heterogeneous consisting mainly of amorphous C-S-H gel (ca. 70% by mass), CH crystals (ca. 20% by mass), unhydrated cement grains and voids containing either water or air (Birchall et al., 1978).

Mechanical Properties

The setting and hardening of cement pastes is brought about by the formation of C-S-H gel, which fills the space between cement grains.

Porosity is one important factor determining the strength of cement paste. Increased porosity leads to a decrease in the strength of cement paste. Porosity is determined by the w/c ratio and the degree of hydration. Several experimental methods have been employed in measuring the porosity of cement pastes, including water saturation method (Kim et al., 2002), water evaporation (Carde et al., 1999) method, mercury intrusion porosimetry (Care, 2008), and nitrogen adsorption (Juenger et al., 2001).

Typically the strengths of cement based materials are determined by measuring their compressive (Shigeyuki et al., 1986), splitting tensile, (Houssam et al.,1994), and flexural strengths (Houssam et al., 1994).

Durability

The durability of a cement paste can be described as its ability to resist chemical attack. This chemical attack can result in dissolution and leaching or chemical transformations. Porosity is a

major factor influencing the ability of a cement paste to resist chemical attack. The larger the porosity of the paste the more it allows the chemical attack agent to penetrate and degrade the paste. The intensity of the attack is also influenced by the specific chemical agent.

Cementitious materials are subjected to several forms of chemical attack in the environment. The main forms of environmental chemical attack are dissolution and leaching in water, acid attack, sulfate attack, and sea water attack. In the case of dissolution and leaching in water, CH present in the cement paste dissolves into the water forming an alkaline solution, this alkaline solution dissolves calcium hydrates present in the paste (Soroka, 1979). This process continues with time until all the CH is leached out as long as a continuous supply of fresh water is still available. Acid attack also dissolves cement paste. The naturally occurring acids which typically attack cementitious materials are carbonic, humic, and sulfuric acids. During acid attack, the acid reacts with the calcium hydrates to form salts. During sulfate attack the sulfates react with hydrated calcium aluminate to form ettringite resulting in an increase in volume and cracking of the cementitious matrix. In addition some sulfates react with CH to form gypsum (Baghabra Al-Amoudi, 2002). The intensity of the sulfate attack is affected by the cement type, the sulfate type, the sulfate concentration, and the quality of the cementitious material. Some of the salts present in sea water contribute to the chemical attack of cementitious materials. The magnesium chloride present in sea water reacts with CH to produce $Mg(OH)_2$ and $CaCl_2$. The sulfates present in sea water also contribute to sulfate attack of cementitious materials (Soroka, 1979).

Most of the environmental chemical attacks on cement result in the leaching of the calcium from the cement paste.

Leaching studies are therefore a good indicator of the durability of cement paste (Carde et al, 1997) and help to characterize the kinetics involved in the degradation of the material.

Pozzolanic Additives and Reinforcements

Pozzolanic Additives and Silica fume

Pozzolans are very common additives to cement pastes because they improve the strength and durability of cement. The American Concrete Institute (ACI) defines a pozzolan as a siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture react with calcium hydroxide to form compounds possessing cementitious properties (ACI Committe 116R, 1997). Silica fume is a highly reactive pozzolans used in making high strength concrete; it reacts with calcium hydroxide to produce a C-S-H gel, thereby increasing the C-S-H content of the cement paste. This increase in C-S-H gel leads to a decrease in the paste porosity (Feldman et al., 1985). There are several other popular pozzolans including fly ash, natural Pozzolans, and ground granulated blast furnace slag (Kulaa et al., 2001). These Pozzolans are used either individually or in combination.

Silica fume also known as microsilica or fumed silica are small spherical produced as a byproduct of the reduction of high purity quartz and coke in an electric arc furnace to produce silicon metal or ferrosilicon alloys (Silica Fume Association, 2008). The small size high surface area and high SiO_2 content makes silica fume a pozzolan when combined with Portland cement (Silica Fume Association, 2008). Table 2.2 lists the composition and some of the physical properties of silica fume (Jiuzhou Silicon Industries Ltd, 2008).

Table 2.2 Composition and Physical Properties of Silica Fume

Component	Composition (%)
SiO_2	94.7
Al_2O_3	0.15
Fe_2O_3	0.096
CaO	0.088
MgO	0.15
K_2O	0.91
Na_2O	0.16
Total Sulfur	0.50
Total Carbon	1.38
Ignition Loss	2.35
Water	0.75
Surface Area	Approx. 20000m^2/kg
Density	Approx. 200kg/m^3 (undensified) Approx. 400-600 kg/m^3 (densified)

Carbon Microfibers

Carbon microfibers (CF) are manufactured from pitch fibers or polymer fibers e.g. polyacrylonitrile (PAN), in either a continuous or short form. CF made from pitch are more graphitizable than those made from polymers and therefore have higher thermal conductivities and lower electrical resistivity. CF made from polymers are more widely used because they are cheaper and have better mechanical properties (Chung, 1994).

The CF are manufactured by the pyrolysis of the pitch or polymer. The PAN fibers are heated until they are turned into oxidized polyacrylonitrile fibers (OPF). The OPF is carbonized by heating progressively to higher temperatures in a nitrogen filled chamber. The final carbonization

occurs at temperatures greater than 1000ºC in order to establish strength, stiffness, electrical, and other properties (Toho Tenax America Inc, 2007). In addition, the CF are coated with a polymer in order to improve their handling characteristics and wettability (Toho Tenax America Inc, 2007).

The properties of CF are determined by their structure which is in turn determined by the production conditions. The most influencing structural features are the degree of crystallinity, the interlayer spacing**,** the crystallite sizes, the preferred orientation of the carbon layers, parallel and perpendicular to the fiber axis, the transverse and longitudinal radii of curvature of the carbon layers, the domain structure, and the volume fraction, shape and orientation of microvoids (Chung, 1994).

Carbon microfibers have been shown to be effective reinforcement in several matrices including polymers (Patton et al., 2002), metals (Lin et al., 1991), and carbons (Wang, et al., 2009).

Carbon Nanofibers

Carbon nanofibers (CNF) can be broadly defined as tubular structures with the side walls composed of angled graphitic sheets. These graphitic sheets can be arranged in various orientations producing nanofibers of various morphologies. These orientations as we will see later are determined by the conditions under which the carbon nanofibers are grown, the two main morphologies being the “herringbone (fishbone)” and the “stacked cup” (figure 2.1).

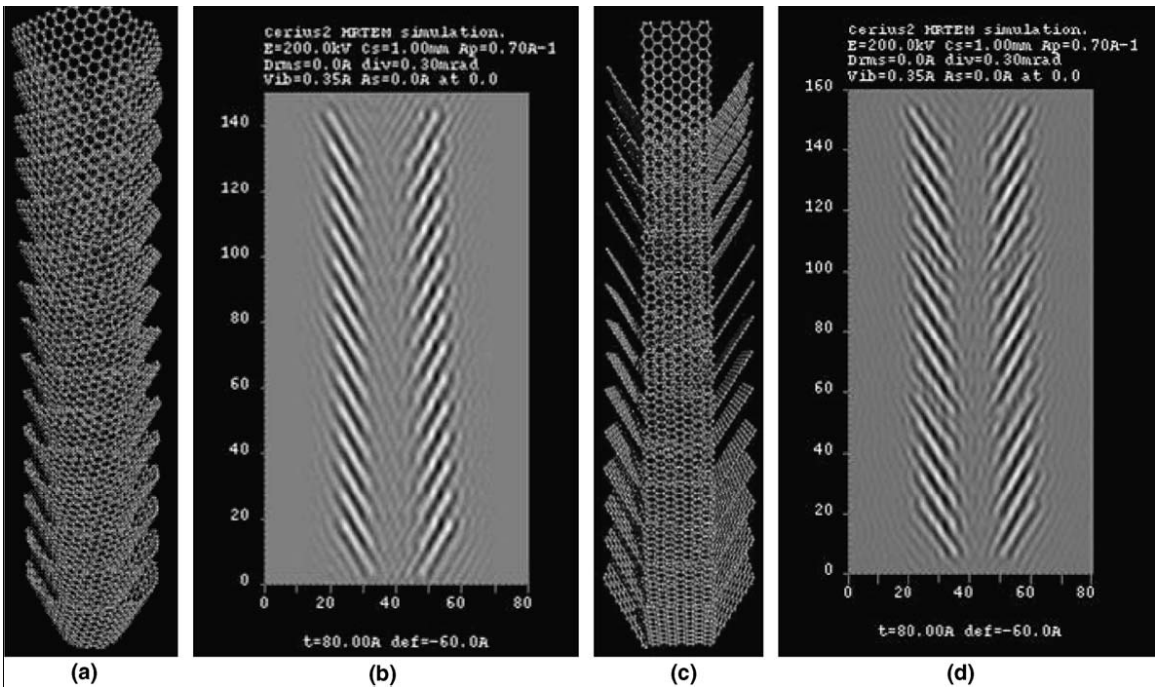

Figure 2.1 (a) and (c) Atomic models of stacked cup and herringbone carbon nanofibers, (b) and (d) their respective TEM simulated images for atomic model (Kim, 2005)

Several methods have been employed for the production of CNF. The two main methods used to produce CNF are (1) the pyrolyzing of fibers spun from an organic precursor and (2) chemical vapor deposition (CVD). In the earlier, typically fibers are produced by pyrolyzing electrospun nanofibers from polyacrylonitrile or pitch (Zussman et al., 2005). These CNF typically have diameters ranging from a few hundred nanometers to several micrometers.

Vapor grown CNF are the most popular CNF used in research because of the ability to produce them in bulk in a cost effective manner. Vapor grown CNF are produced by decomposing a hydrocarbon gas in the presence of hydrogen over a metal catalyst. The hydrocarbon gas is fed into the chamber containing the metal catalyst, which has been activated usually by a sulfur containing compound, which is maintained at a high temperature (greater than 1100˚C), under these conditions the nanofibers filaments are grown with a diameter of about 10nm. Growth stops

when the catalyst is deactivated. The filaments are then usually thickened by chemical vapor deposition of carbon.

The growth of the CNF is influenced by many factors including but not limited to the type of metal catalyst (Chambers et al., 1995; Rodriguez et al., 1995), the hydrogen source gas, the presence of additives (Kim et al., 1993), reaction temperature and reaction time.

Because of their interesting mechanical, thermal and electrical properties CNF are deemed to have great potential for composite applications. The tendency of the CNF to form millimeter sized clumps, however, poses problems in dispersion and therefore difficulties in composite preparation. One of the key features of CNF, which facilitate their use in composites, is the presence of many edges that can serve as sites for chemical and physical interactions.

Carbon Microfiber/Nanofiber Reinforced Cement-based Materials

CF have been found to have the following effects on the properties of cement based materials: increased flexural strength (Houssam at al., 1994), increased tensile strength (Ali et al., 1972), increased modulus of elasticity (Ali et al., 1972), increased air content (Pu-Woei Chen, 1993), improved freeze-thaw durability (Chen et al., 1993), decreased drying shrinkage (Chung et al., 1996), and decreased electrical resistivity (Chen et al, 2004).

In contrast, studies of CNF-cement composites are very limited; only a few studies have been conducted on carbon nanotubes (CNT)-cement composites and few baseline property measurements have been reported with mixed results. The incorporation of acid treated CNT into cement has been found to enhance the flexural and compressive strengths, failure strain and to decrease the porosity of cement (Li et al., 2005). In addition, CNT have been shown to bridge cracks and accelerate the hydration of cement (Markar et al., 2005). The structural similarities

between CNF and CNT, lower cost of CNF and some of the positive results of CNT-cement composite studies make the potential of CNF as reinforcements in cement very promising.

CHAPTER III

OBJECTIVES

This study investigated the effects of carbon nanofiber loadings and carbon fiber type (carbon nanofibers vs. carbon microfibers) on the mechanical performance and durability with respect to leaching of cement pastes.

More specifically, the objectives were to assess the cement paste performance and durability based on the following properties and characteristics: compressive strength, splitting tensile strength, water absorption capacity/ water porosity, calcium leachability in DI water and mass loss during accelerated decalcification.

CHAPTER IV

. EXPERIMENTAL APPROACH

This study investigated the effects of carbon fiber type and carbon nanofiber loading on the mechanical properties (compressive and splitting tensile strengths) and durability with respect to leaching of cement pastes.

Two different types of cement pastes were used to prepare cylindrical specimens (2x4 in). The first type of cement paste was Portland cement paste (PC), and the second type of cement paste was Portland cement with 10 wt% silica fume (SF). SF was added because it is an effective pozzolans. It reacts with the calcium hydroxide in hydrated cement paste to produce calcium silicate hydrate gel (C-S-H). The decrease calcium hydroxide and increased C-S-H gel imply a higher strength and lower porosity (Yajun et al., 2003). Each of the cement pastes were prepared with two water to cement ratios (w/c) in order to ensure adequate hydration of the cement. The PC pastes were prepared with a w/c of 0.325 and 0.435 and SF pastes were prepared with w/c of 0.365 and 0.45.

Two types of carbon fibers were used. Carbon microfibers (CF) with diameters of 6 to 7μm and lengths of 3mm and carbon nanofibers (CNF) with diameters of 100-200 nm and lengths of 30 to 100μm.

Specimens were prepared with different carbon fiber loadings. PC pastes with w/c=0.325 were prepared with fiber loadings of 0, 0.005, 0.02, 0.05, and 0.5 wt% CNF , and 0.5 wt% CF. PC

pastes with w/c=0.435 were prepared with CNF loadings of 0 and 2 wt% CNF. SF pastes with w/c=0.365 were prepared with fiber loadings of 0, 0.005, 0.02, 0.05, and 0.5 wt% CNF , and 0.5 wt% CF. SF pastes with w/c=0.45 were prepared with CNF loadings of 0 and 2 wt% CNF.

A total of 16 paste types were studied. The mechanical properties were characterized by the compressive strength, splitting tensile strength, and compressive load displacement curves. The paste durability was characterized by the mass loss, compressive strength, and splitting tensile strength losses due to accelerated decalcification, water porosity, and the leachability by DI water.

PC and SF pastes with fiber loadings of 0, 0.5, and 2wt% were subjected to DI leaching and accelerated decalcification. The accelerated decalcification was performed by immersing the pastes in 7M ammonium nitrate solution for ca. 95 days.

The compressive and splitting tensile strength tests were performed on up to 15 replicates of each paste type at an age of 28 days and the decalcified specimens after they had been immersed in the ammonium nitrate solution for ca. 95 days.

The water porosities for PC and SF pastes with 5 different CNF loadings; 0, 0.005, 0.02, 0.05, 0.5 and 2 wt% were determined by immersion in DI water. Exposure to DI leaching for up to 3 months was performed on PC and SF pastes with CNF loadings of 0, 0.5, and 2 wt% and CF loadings of 0.5%.

CHAPTER V

. MATERIALS AND METHODS

This section covers specimen preparation, the specimen mechanical testing, the specimen decalcification, and characterization methods.

Specimen Preparation

Two carbon fiber types were used in this study: carbon nanofibers (CNF) and carbon microfibers (CF).

Carbon Nanofibers (CNF)

The CNF used were vapor grown Pyrograf III PR-19 LHT obtained from Applied Sciences Inc. (Cedarville, OH). The as grown fibers contain chemically vapor deposited carbon which was graphitized in the subsequent heat treatment at temperatures of up to 3000˚C. The fiber diameters ranged from 100 to 200nm and the lengths ranged from 30 to 100 μm. Due to the size of the CNF it is not possible to directly measure many of the properties by conventional methods. The following properties have been estimated by the manufacturer: tensile strength of 7GPa, a tensile modulus of 600GPa, a density of 1.95g/cm^3, and an electric resistivity of 55μΩ-cm (Applied Sciences Inc., 2001).

Carbon Microfibers (CF)

The CF used were carbon fibers Product 150 obtained from Toho Tenax America Inc. (Rockwood, TN). The fiber length was 3mm and the diameter ranged from 6-7μm. The CF were

produced using a polyacrylonitrile (PAN) fiber precursor. The precursor was exposed to heated air to turn it into oxidized PAN fibers, which were carbonized into carbon fibers by exposing them to progressively higher temperatures in a nitrogen-filled chamber. These fibers were coated with a polymer to promote fiber handling characteristics, wet out, and bonding (Toho Tenax America Inc). The CF have been found to have a tensile strength greater than 3450MPa, a tensile modulus greater than 207GPa, a density of 1.8g/cm^3, and an electric resistivity of 1670μΩ-cm (Toho Tenax America Inc, 2007).

Cement Paste Types

Five loadings of CNF and one loading of CF were investigated in Portland cement pastes with and without silica fume. Commercial grade type I/II Portland cement and microsilica grade 970 D densified silica fume obtained from Elkem Materials were used.

Plain Portland cement (PC) pastes were prepared with two water to cement ratios (w/c): 0.325 and 0.435. PC pastes with four CNF loadings; 0.005, 0.02, 0.05, 0.50 wt %, denoted LD1-LD4 and one CF loading 0.50 wt % were prepared with a w/c ratio of 0.325. A PC paste with a CNF loading, of 2 wt% (LD5) with a w/c of 0.435 was also prepared. The SF pastes contain 10 wt% silica fume. SF pastes were prepared with two water to cement ratios (w/c): 0.325 and 0.435. SF pastes with four CNF loadings; 0.005, 0.02, 0.05, 0.50 wt %, denoted LD1-LD4 and one CF loading 0.50 wt % were prepared with a w/c ratio of 0.365. A SF paste with a CNF loading, of 2 wt% (LD5) with a w/c of 0.45 was also prepared. Baseline pastes of each type containing no fibers at each w/c; 0.325, 0.365, 0.435, and 0.45 denoted PC-P, SF-P, PC-P2, SF-P2, respectively were also prepared. The nomenclature of the prepared specimens is summarized in table 5.1.

Table 5.1 Nomenclature of Specimen Types

Type of Paste	Type of Fiber	Fiber Loading (wt %)
PC- Plain Portland Cement	P- no fibers	LD1 (0.005 wt %)
SF- Silica Fume Portland Cement	P2- no fibers	LD2 (0.02 wt %)
	CNF- carbon nanofibers	LD3 (0.05 wt %)
	CF- carbon microfibers	LD4 (0.50 wt %)
		LD5 (2 wt%)

Cement Paste Preparation

The dry materials were placed in the mixing bowl of a Univex SRM 30+ electric mixer and mixed at low speed for about 6 minutes. The deionized water (DI) was added to the dry mix and it was mixed at high speed for 6 minutes. The pastes were cast in cylindrical molds of diameter 2" and length 4". The molds were covered and allowed to cure for a minimum of 28 days at 100% relative humidity in a Curamold concrete test cylinder curing box before further use. Several batches of each mix were prepared as necessary. The mix design is summarized in table 5.2.

Table 5.2 Mix Design

Paste Type	w/c	Specimen Type	CNF (wt %)	CF (wt %)
PC	0.325	PC-P	0	0
		PC-CNF_LD1	0.005	0
		PC-CNF_LD2	0.02	0
		PC-CNF_LD3	0.05	0
		PC-CNF_LD4	0.50	0
		PC-CF_LD4	0	0.50
	0.435	PC-P2	0	0
		PC-CNF_LD5	2	0
SF	0.365	SF-P	0	0
		SF-CNF_LD1	0.005	0
		SF-CNF_LD2	0.02	0
		SF-CNF_LD3	0.05	0
		SF-CNF_LD4	0.50	0
		SF-CF_LD4	0	0.50
	0.45	SF-P2	0	0
		SF-CNF_LD5	2	0

Mechanical Tests

Two mechanical properties were measured, compressive strength and splitting tensile strength. All tests were performed using a Super L hydraulic materials testing machine produced by Tinius Olsen Inc. (Willow Grove, PA). The mechanical properties of all specimen types at an age of 28 days and specimens that had been decalcified by NH_4NO_3 solution for 95 days were measured.

Compressive Strength

The compressive strength tests were performed according to ASTM C 39 (ASTM International, 2005). Compressive strength tests were performed on up to 15 replicates of each specimen type. Specimens with an age of 28 days were removed from the curing chamber and demolded just prior to testing. Decalcified specimens were placed in deionized water after 95 days of decalcification and removed from the DI just prior to testing. Specimens were tested while they were in a moist condition. The diameter of each specimen was measured at the top, middle and bottom of the specimen and averaged. Three length measurements were also taken and averaged. Each specimen was centered in the testing machine as shown in figure 5.1 and loaded at a position rate of 0.2 in/min (5.082 mm/min) until a load of $100lb_f$ (0.4448kN) was reached then loaded at a position rate of 0.012in/min (0.306 mm/min) until failure. The compressive strength of each specimen was computed by dividing the maximum load to failure by its average cross sectional area.

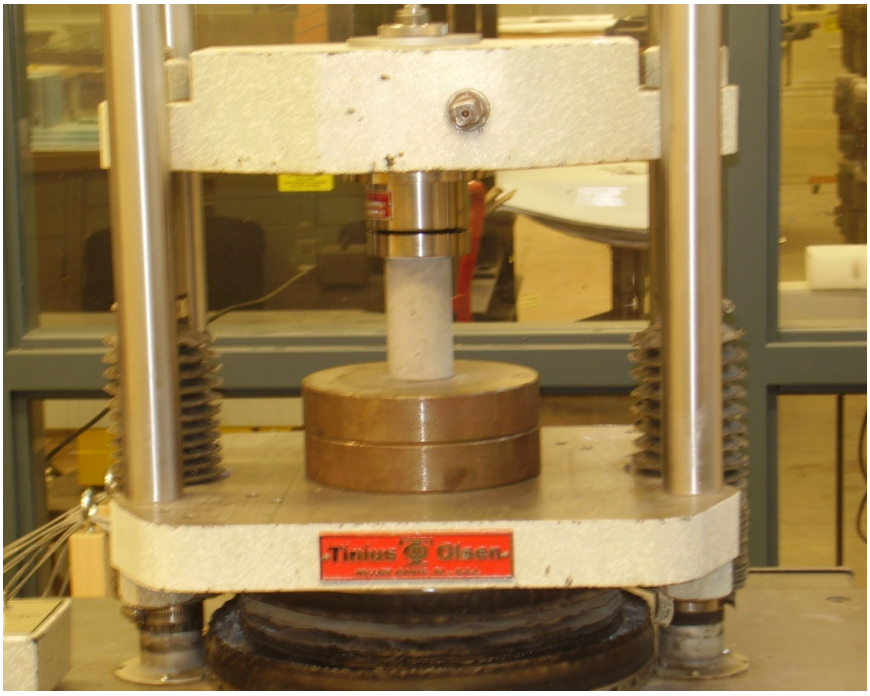

Figure 5.1 Photograph of compressive strength testing of a specimen

Splitting Tensile Strength

The splitting tensile strength tests were performed according to ASTM C 496-96 (ASTM International). Splitting tensile strength tests were performed on up to 15 replicates of each specimen type. 28 day old specimens were removed from the curing chamber and demolded just prior to testing. Decalcified specimens were placed in DI after 95 days of decalcification and removed from the DI just prior to testing. Specimens were tested while they were in a moist condition. The diameter of each specimen was measured at the top, middle and bottom of the specimen and averaged. Three length measurements were also taken and averaged. Each specimen was centered in the testing machine as shown in figure 5.2 and loaded at a load rate of 11500lbf/min (51151.8N/min) until a load of 2000lbf (8.896 kN) was reached then loaded at a position rate such that a load rate of ca. 11500lbf/min (51151.8 N/min) was maintained until

failure. The splitting tensile strength of each specimen was computed from the following formula (E5):

$T = (2 \times P)/(\pi \times L \times D)$ (E5)

Where, T = splitting tensile strength, kPa

P = maximum load to failure of specimen, kN

L = average length of specimen, mm

D = average diameter of specimen, mm

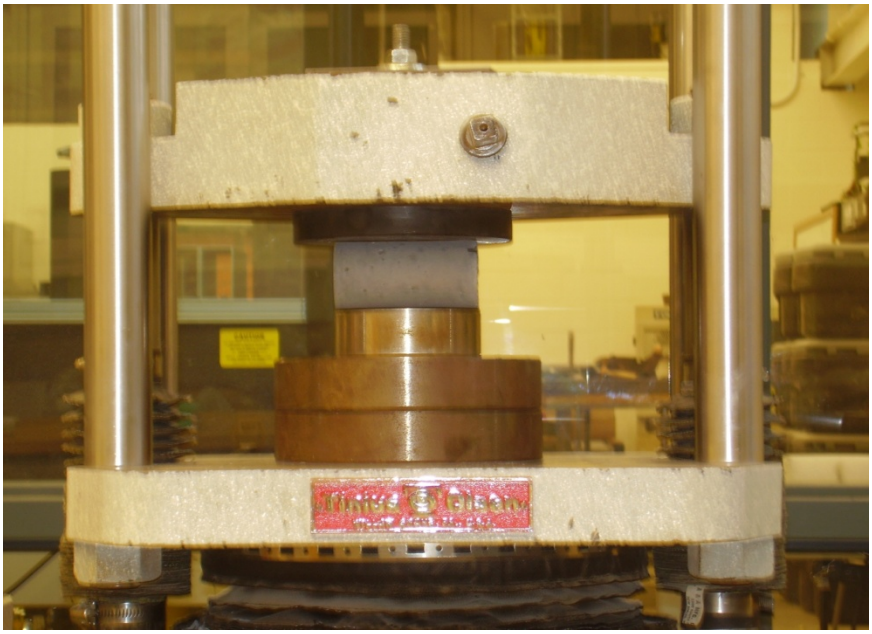

Figure 5.2 photograph of splitting tensile strength testing of a specimen

Water Absorption Capacity and Water Porosity

The water absorption capacity was determined for PC and SF specimens with CNF loadings of 0.005, 0.02, 0.05, 0.5 and 2% and their corresponding baselines. Two replicates of each specimen type at a minimum age of 28 days under went water absorption.

The specimens were removed from the curing chamber and demolded. A horizontal slice of about 1 cm thick was cut from the middle of each of the specimens for water absorption to be performed on. The specimens were dried in an oven at ca. 60ºC and weighed regularly. They were removed from the oven when their weights reached a constant value to ensure that they were completely dry. The specimens were then completely immersed in milli q water such that there was a liquid to surface area ratio of 10cm. The specimens were blotted dry and weighed at cumulative times of 0.25, 0.50, 0.75, 1.00, 1.30, 2.00, 2.50, 3.50, 5.50, 24.00 and 48.00 hours.

The water absorption capacity method was used to determine the water porosity of the specimens.

Leaching in DI Water

DI water leaching was performed on PC and SF specimens with CNF loadings of 0.5 and 2 wt %, CF loadings of 0.5 wt% and their corresponding baselines. Two replicates of each specimen at a minimum age of 28 days were used.

The DI leaching was performed according to a modified version of the Mass Transfer Rates in Monolithic Materials, MT001.1 protocol (Kosson et al., 2002). The specimens were removed from the curing chamber and demolded just prior to starting the leaching process. The diameters and lengths of each specimen were measured and recorded. Each specimen was placed in a separate container on top of a plastic mesh to ensure that the entire surface area was in contact with the DI water (Figure 5.3). 10mL of DI water was added for every cm^2 of specimen surface area. The leaching solution was exchanged with fresh DI water after each contact period. After each contact period the pH of the leachate was measured and each specimen was weighed before being placed in the fresh DI water. In addition, a 125mL sample of the leachate was collected and vacuum filtered using a 0.45µm pore size membrane and preserved for subsequent chemical analysis with 2% by volume of the sample of trace metal grade nitric acid (67-70 wt %) obtained from Fisher Scientific (Fairlawn, NJ). The chemical analysis of the sample leachate was performed using inductively coupled mass spectrometry (ICP-MS) to determine the concentrations of the following elements: aluminum, potassium, sodium, calcium, iron, and silicon.

Accelerated Decalcification Using Ammonium Nitrate (NH_4NO_3) Solution

Pastes were decalcified using NH_4NO_3 solution. NH_4NO_3 was chosen as the decalcifying agent because it increases the calcium solubility. Calcium saturation concentration increases from 0.022mol/L in water to 2.9mol/L in 6M ammonium nitrates solution (Heukamp, Ulm, & Germaine, 2001). Calcium hydroxide (CH) is leached first followed by calcium silicate hydrate (C-S-H).

$$Ca(OH)_2 + 2NH_4NO_3 \rightleftharpoons Ca^{2+} + 2OH^- + 2H^+ + 2NH_3 + 2NO_3^- \rightleftharpoons Ca(NO_3)_2 + 2NH_3\,(g) + 2H_2O \quad \text{(E6)}$$

The effects of decalcification were studied on PC and SF specimens with fiber loadings of 0.5 and 2 wt % and their corresponding baselines. After curing for a minimum of 28 days, nine replicates of each specimen type were decalcified in a 7M NH_4NO_3 solution. The specimens were placed on top of a plastic mesh in a container to ensure that the entire surface area of each specimen was in contact with the solution (Figure 5.3). The solution was added such that there was a liquid to surface area ratio of 5cm. The specimens were weighed at regular intervals over a 95 day period, and the pH monitored throughout the decalcification process. At the end of the degradation period 3 replicates of each specimen type were rinsed with DI water and cut to remove the ends which are more degraded in order to view the thickness of the degraded region (Figure 5.4). The other replicates were stored in DI water until further use. The NH_4NO_3 solution was renewed for one replicate of each of the specimens with fiber loadings of 0.5 wt % after 70 days.

The effects of accelerated decalcification were demonstrated using compressive strength, splitting tensile strength, and mass loss.

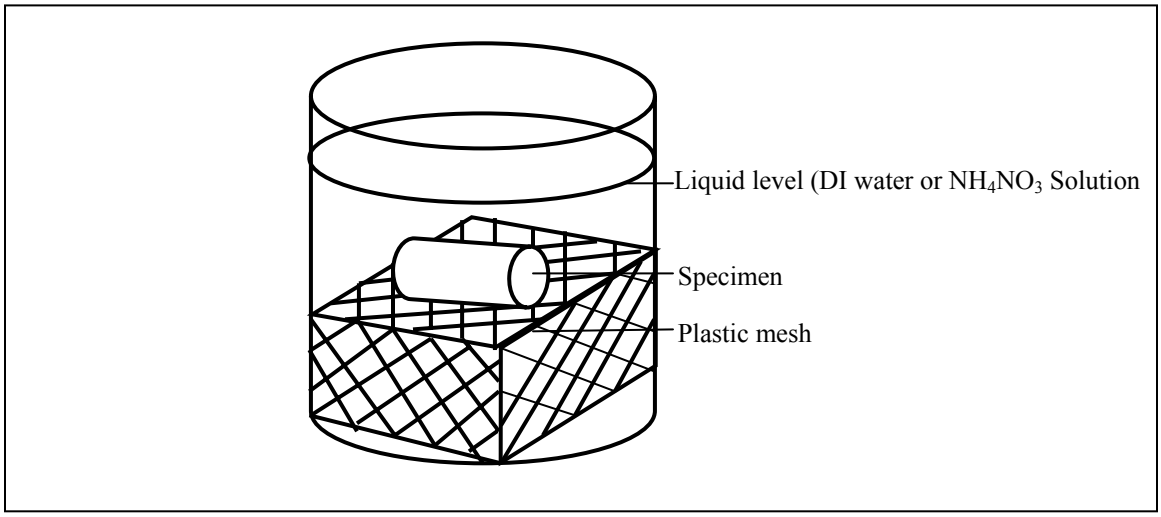

Figure 5.3 Set up for decalcification and DI leaching experiments

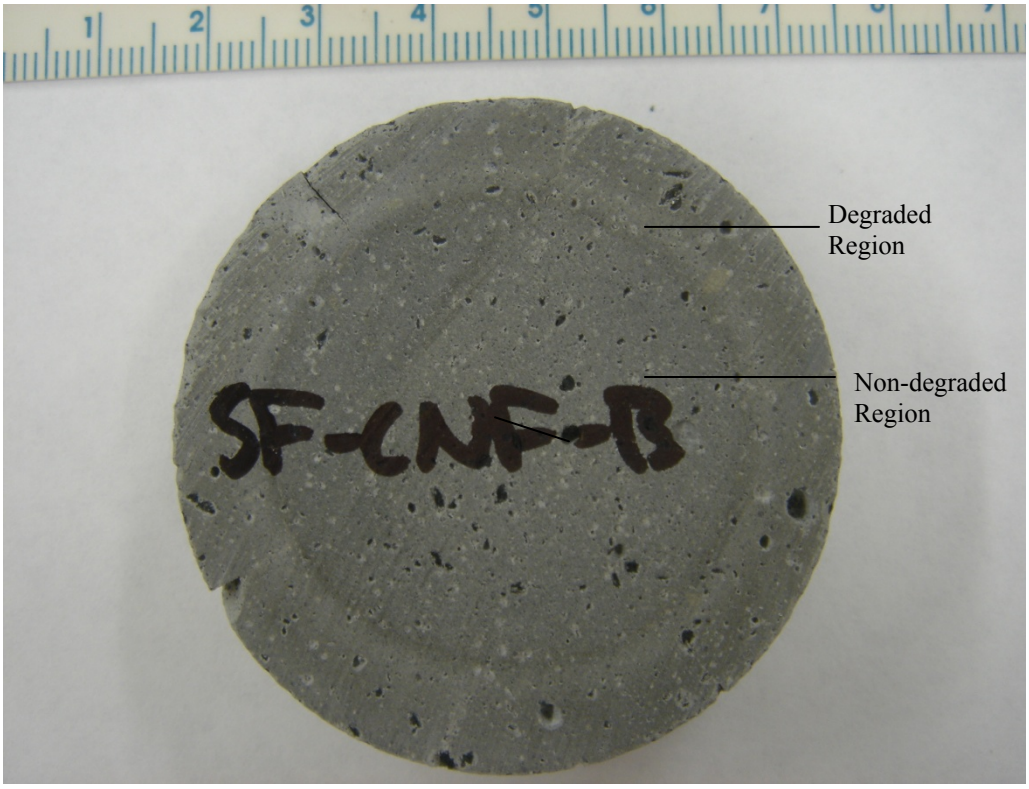

Figure 5.4 Photograph of specimen decalcified by NH_4NO_3 for 95 days showing the thickness of the degraded region.

Analytical Method (ICP-MS)

A Perkin-Elmer ELAN DRC III inductively-coupled mass spectrometer (ICP-MS) was used to perform chemical analysis of the DI leaching leachate samples.

A 7 point calibration with a blank was performed. The calibration concentrations were 10, 25, 50, 100, 250, and 500µg/L. The correlation coefficients of curve was verified to be at least 0.995. An initial check standard (ICV) of 50µg/L and an initial check blank of 1% nitric acid were then run. The analysis of the samples was then performed. Continuous check blank (CCB) and continuous check verification (CCV) were performed at intervals of 12-20 samples during sample analysis.

The CCB was 1% nitric acid and the CCV was about 50µg/L. A spike analysis per 10-20 samples was performed. The spike concentration was 500µg/L at 10x dilution. All samples were diluted at 10x. Table 5.3 provides the minimum level (ML) and method detection limit (MDL) for the elements analyzed.

Table 5.3 MDL and ML of Elements Analyzed by ICP-MS

Element	MDL (µg/L)	ML (µg/L)
Sodium	0.11	0.20
Potassium	0.19	0.50
Aluminum	0.13	0.20
Silicon	0.19	0.50
Iron	0.16	0.50
Calcium	0.20	0.50

CHAPTER VI

RESULTS AND DISCUSSION

Mechanical Properties

The effects of CNF loading and fiber type (CNF vs. CF) on the compressive strength, splitting tensile strength and compressive load displacement curves are discussed in the following sections.

Effect of CNF Loading

Portland cement pastes (PC pastes) and portland cement pastes with silica fume (SF pastes) prepared with 6 different CNF loadings (0, 0.005, 0.02, 0.05, 0.5, and 2wt %) were tested.

Compressive Strength

Compressive strength at 28 days of the PC and SF pastes with varying CNF loadings are shown in figures 6.1 and 6.2, respectively.

The following conclusions were made:

- CNF loadings from 0.005 to 0.50 wt % had no significant impact on the compressive strength of the PC pastes at w/c=0.325.
- A CNF loading of 2 wt % resulted in a decrease of the compressive strength of the PC pastes at w/c=0.435.

- CNF loadings up to 2 wt % had no significant impact on the compressive strength of the SF pastes.

- The CNF loading had no apparent effect on the variability of the compressive strength within each specimen type for both pastes.

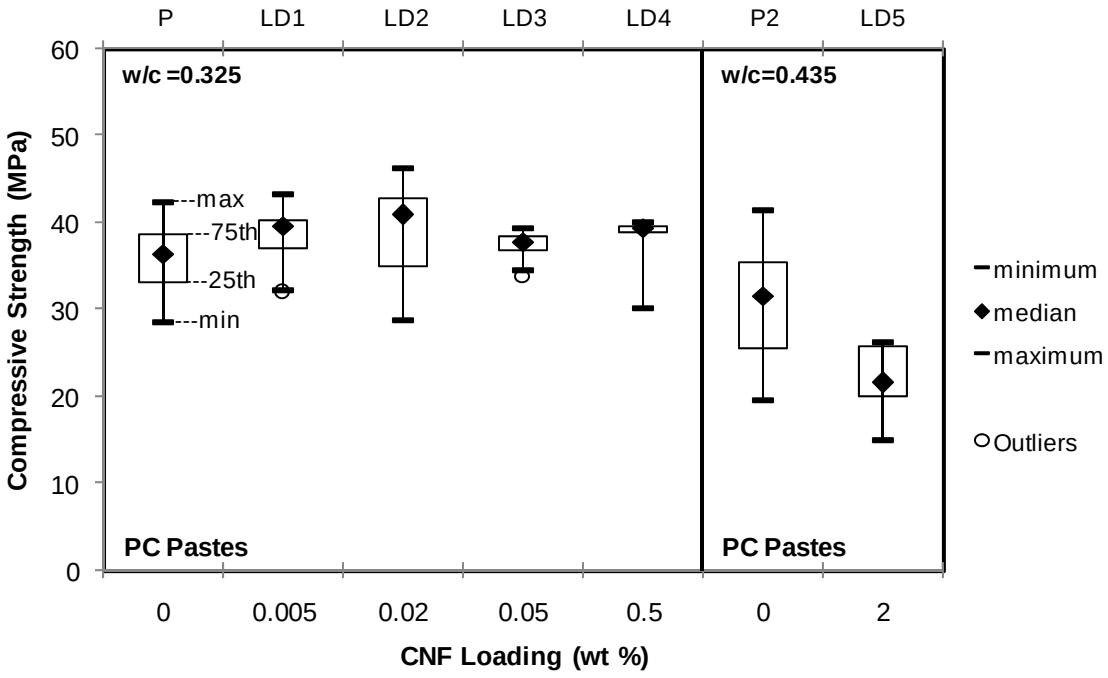

Figure 6.1 Effect of CNF loading on the compressive strength of PC pastes at 28 days

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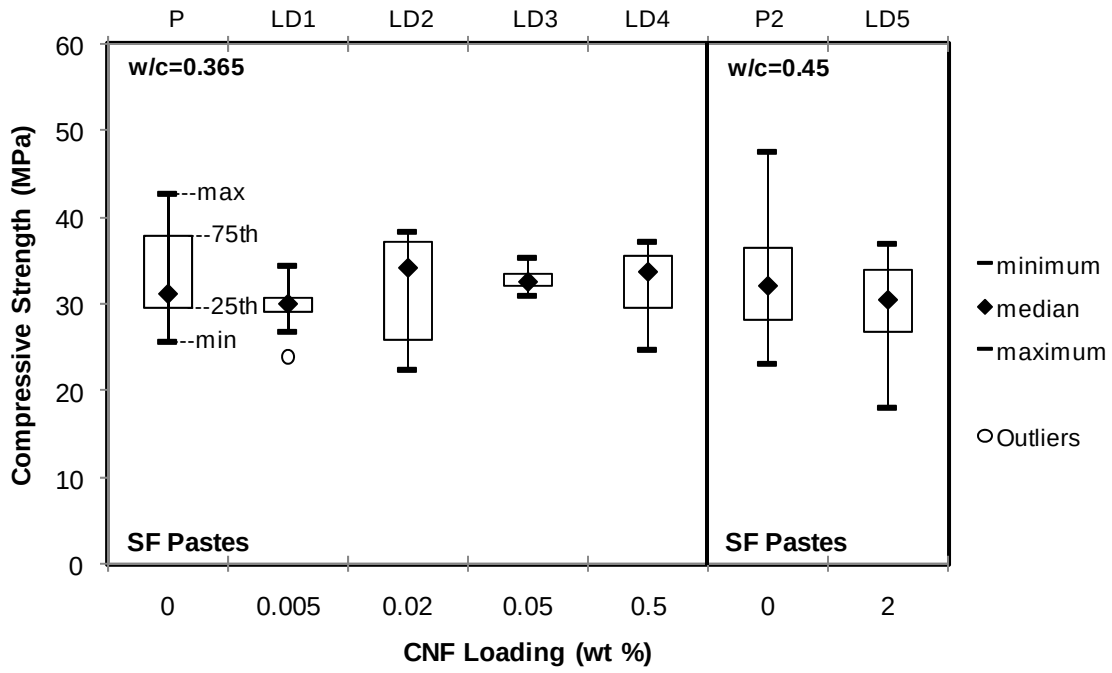

Figure 6.2 Effect of CNF loading on the compressive strength of SF pastes at 28 days

Splitting Tensile Strength

Splitting tensile strength at 28 days of the PC and SF pastes with varying CNF loadings are shown in figure 6.3 and 6.4 respectively:

The following conclusions were made:

- CNF loading of up to 2 wt % had no significant impact on the splitting tensile strength of the PC and SF pastes.
- CNF loading had no significant effect on the variability of the splitting tensile strength within each specimen type for both pastes.

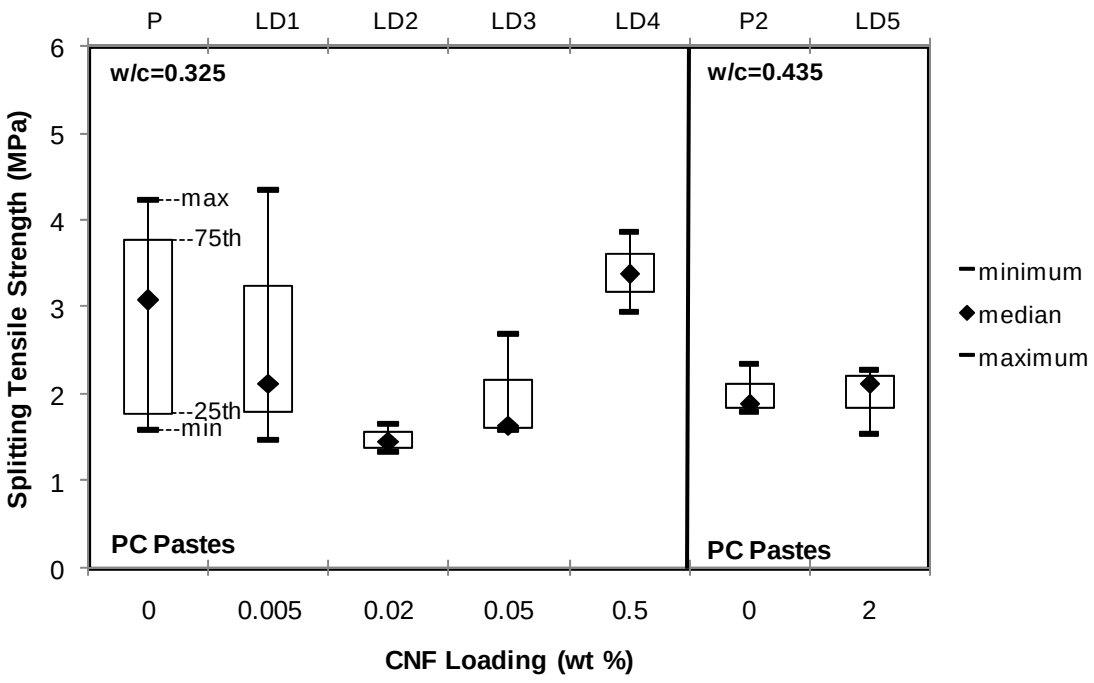

Figure 6.3 Effect of CNF loading on the splitting tensile strength of PC pastes at 28 days

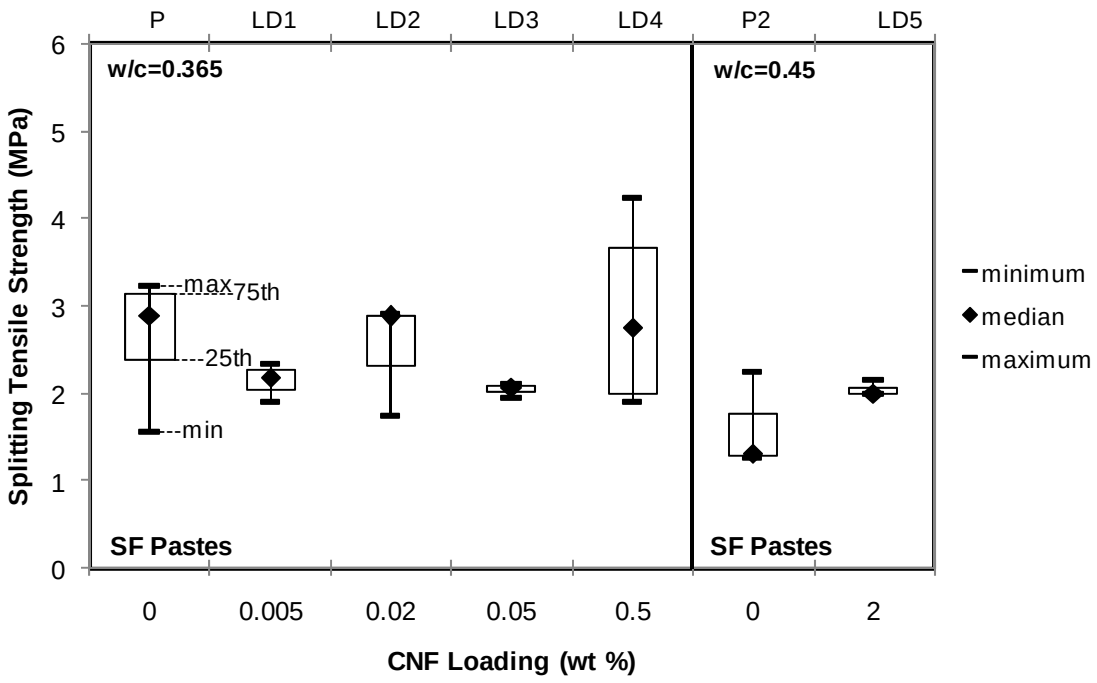

Figure 6.4 Effect of CNF loading on the splitting tensile strength of SF pastes at 28 days

Compressive Load Displacement Curves

The load displacement curves for compressive strength tests of PC and SF pastes with various CNF loadings are shown in figure 6.5. The slopes of the curves prior to failure were studied and listed in table 6.1.

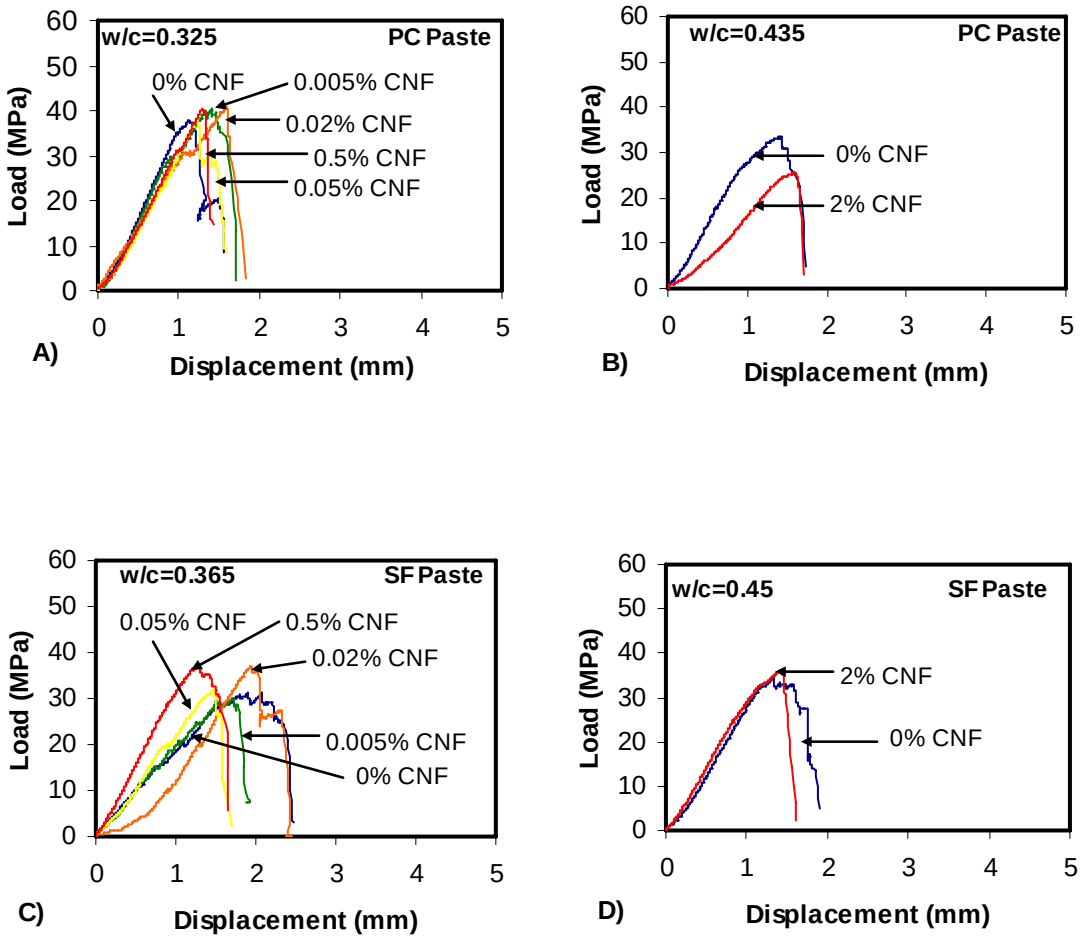

Figure 6.5 Effect of CNF loading on the compressive load displacement curves of PC and SF pastes A) PC pastes at w/c=0.325, B) PC pastes at w/c=0.435, C) SF pastes at w/c=0.365, and D) SF pastes at w/c=0.45

Table 6.1 Effect of CNF loading on the slope of the compressive load displacement curves of PC and SF pastes prior to failure

			Slope (MPa/mm)				
Paste Type	**w/c**	**CNF (wt%)**	**Mean**	**Standard Deviation**	**Minimum**	**Median**	**Maximum**
PC	0.325	0	27.4	9.6	12.2	23.4	40.6
		0.005	32.2	4.9	27.3	30.8	40.0
		0.02	30.2	1.9	27.7	30.9	32.3
		0.05	35.9	3.8	32.8	35.2	42.4
		0.50	34.1	4.8	25.8	33.9	39.6
	0.435	0	32.4	10.1	17.0	31.4	47.9
		2	19.0	3.5	13.3	17.8	25.2
SF	0.365	0	19.2	7.1	7.7	17.6	31.0
		0.005	21.8	2.2	19.4	22.2	24.8
		0.02	23.9	3.6	18.1	24.2	27.6
		0.05	24.1	3.6	19.0	24.7	28.9
		0.50	29.0	6.6	17.0	32.9	35.1
	0.45	0	28.0	3.7	21.7	28.5	33.4
		2	29.0	4.3	21.0	31.3	32.8

The following conclusions were drawn based on these results:

- For the PC pastes, CNF loadings up to 0.5 wt % had no significant impact on the slopes of the compressive load displacement curves, which indicated that low CNF loading had no significant impact on the ductility of the pastes. In contrast, for the 2 wt % CNF loading a decrease in the slope was observed indicating an increase in the ductility.

- No effect of CNF loading on the load-displacement curves could be observed for the SF pastes.

Conclusions

CNF loadings up to 0.5 wt % had no significant effect on the compressive strength of the PC pastes. In contrast CNF loading of 2 wt % resulted in a decrease in the compressive strength of

the PC pastes. CNF loadings up to 2 wt % had no significant effect on the compressive strength of the SF pastes. No effect of CNF loading could be observed on the splitting tensile strength of both PC and SF pastes. A CNF loading of 2 wt % modified the deformation characteristics of the PC paste for the sample tested.

Effect of Fiber Type (CNF vs. CF)

In order to determine the effect of fiber type (CNF vs. CF) on the mechanical properties of PC and SF pastes two types of PC and SF pastes were prepared, one with 0.5 wt% CNF and the other with 0.5 wt % CF. The effect of fiber type on the compressive strengths, splitting tensile strengths, and compressive load displacement curves was evaluated.

Compressive Strength

Figure 6.6 (A) shows the results of compressive strength tests on 3 types of PC pastes: PC pastes with no fibers, PC pastes reinforced with 0.5 wt % CNF and PC pastes reinforced with 0.5 wt % CF. Figure 6.6 (B) shows the results of compressive strength tests on 3 types of SF pastes: SF pastes with no fibers, SF pastes reinforced with 0.5 wt % CNF and SF pastes reinforced with 0.5 wt % CF.

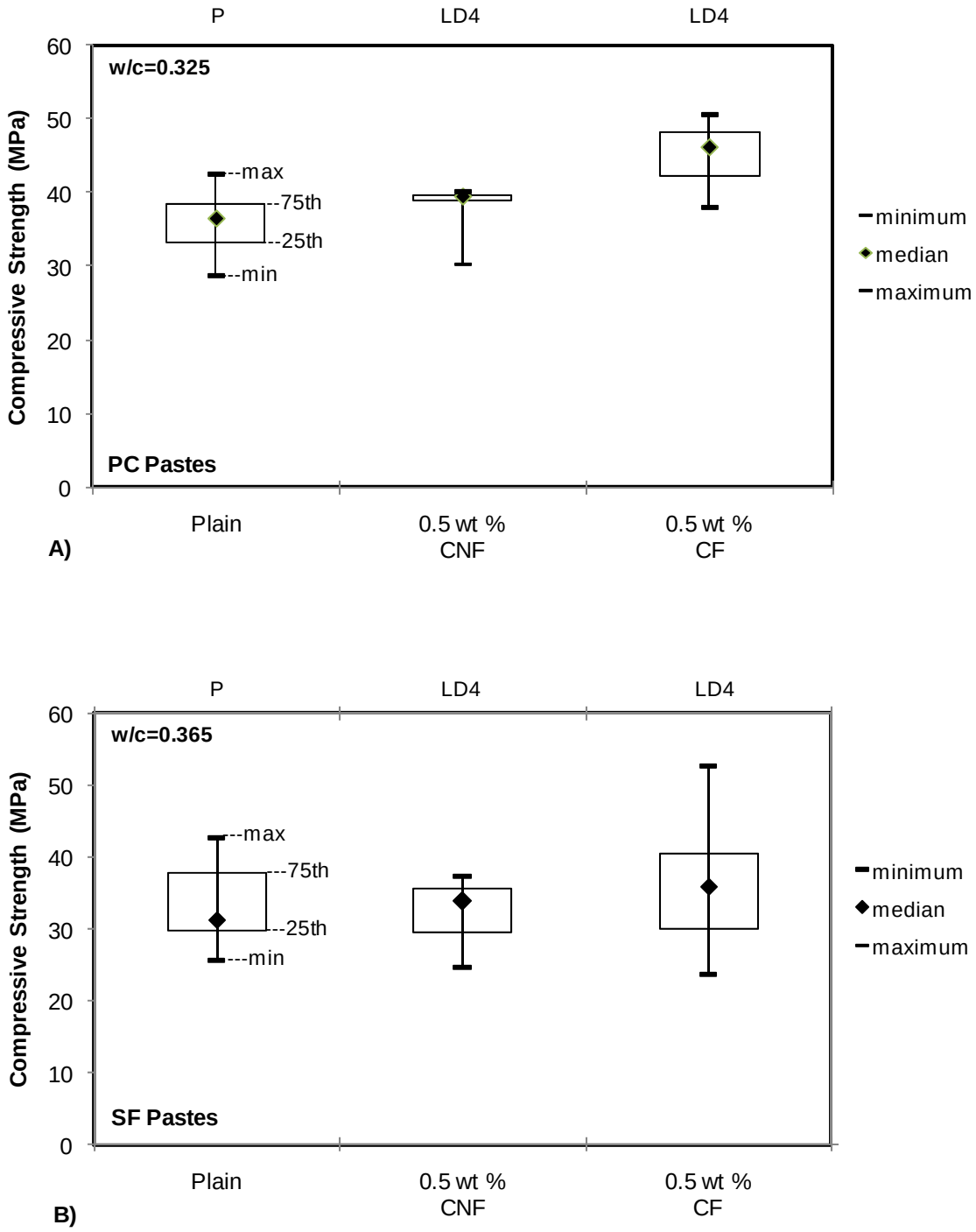

Figure 6.6 Effect of fiber type (CNF vs. CF) on the compressive strength of A) PC pastes, and B) SF pastes

The following conclusions were made:

- 0.5 wt% CNF addition had no significant effect on the compressive strength of the PC and SF pastes.
- In contrast, 0.5 wt% CF loading resulted in a 21% increase in the median compressive strength of the PC paste.

Splitting Tensile Strength

Figure 6.7 (a) shows the results of splitting tensile strength tests on 3 types of PC pastes; PC pastes with no fibers, PC pastes reinforced with 0.5 wt % CNF, and PC pastes reinforced with 0.5 wt % CF. Figure 6.7 (b) shows the results of splitting tensile strength tests on 3 types of SF pastes; SF pastes with no fibers, SF pastes reinforced with 0.5 wt % CNF, and SF pastes reinforced with 0.5 wt % CF.

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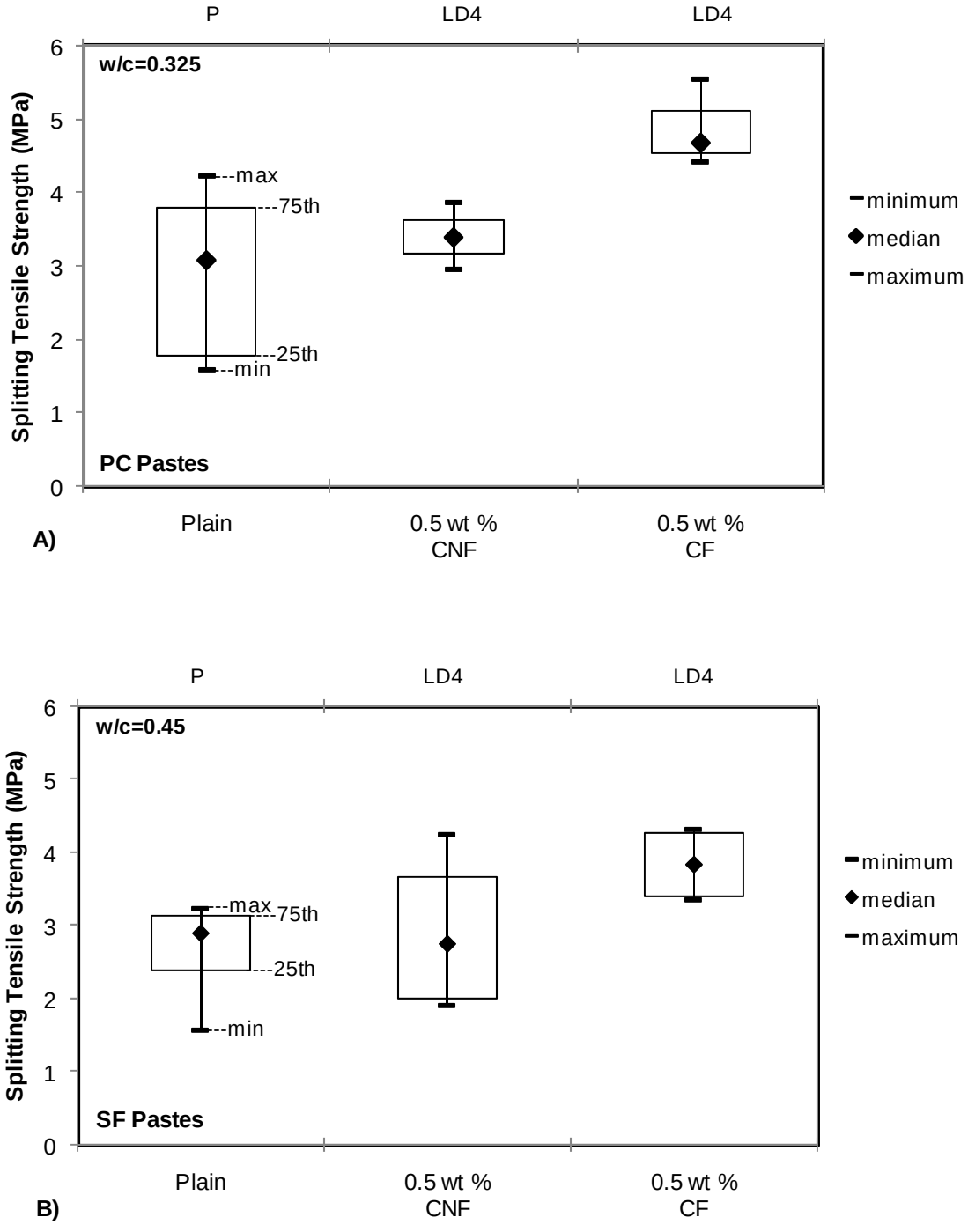

Figure 6.7 Effects of fiber type (CNF vs. CF) on the splitting tensile strength of A) PC pastes, and B) SF pastes

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The following conclusions were drawn based on the results shown in figure 6.7:

- Addition of 0.5 wt% CNF had no significant effect on the splitting tensile strength of both PC and SF pastes.
- In contrast, addition of 0.5 wt % CF yielded a 52% increase in the median splitting tensile strength of PC paste and a 32% increase in the median splitting tensile strength of SF paste.

Compressive Load Displacement Curves

Figure 6.8 (A) shows the compressive load displacement curves of PC pastes with no fibers, PC pastes reinforced with 0.5 wt % CNF and PC pastes reinforced with 0.5 wt % CF. Figure 6.8 (B) shows the compressive load displacement curves of SF pastes with no fibers, SF pastes reinforced with 0.5 wt % CNF and SF pastes reinforced with 0.5 wt % CF. The slopes prior to failure of the curves are listed in tables 6.2.

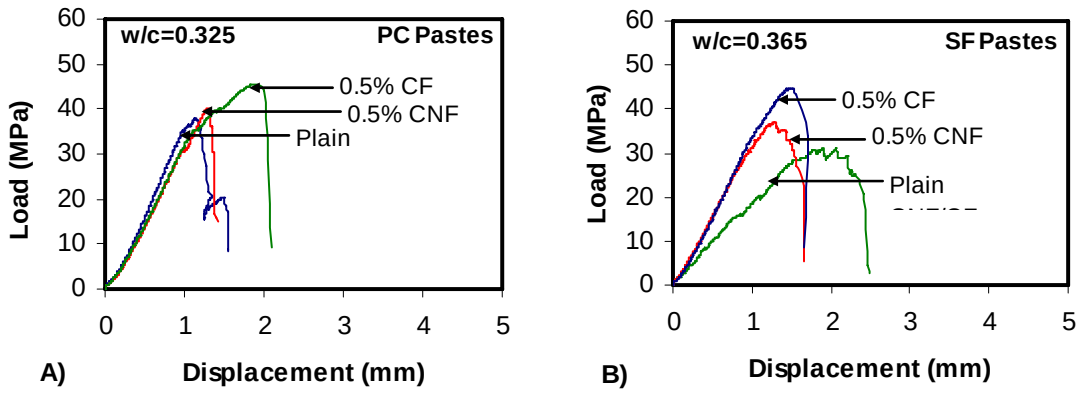

Figure 6.8 Effect of fiber type (CNF vs. CF) on the load displacement curves of: A) PC pastes and B) SF pastes.

Table 6.2 Effect of fiber type (CNF vs CF) on the slope of the compressive load displacement curves of PC and SF pastes prior to failure

				Slope (MPa/mm)				
Paste Type	**w/c**	**Fiber Type**	**Fiber Loading (wt%)**	**Mean**	**Standard Deviation**	**Minimum**	**Median**	**Maximum**
PC	0.325		0	27.4	9.6	12.2	23.4	40.6
		CNF	0.50	34.1	4.8	25.8	33.9	39.6
		CF	0.50	23.5	6.3	15.0	22.9	35.2
SF	0.365		0	19.2	7.1	7.7	17.6	31.0
		CNF	0.50	29.0	6.6	17.0	32.9	35.1
		CF	0.50	24.6	7.9	16.6	20.1	37.3

The following conclusions were drawn:

- Addition of with 0.5 wt % CNF and 0.5 wt % CF had no significant effect on the slopes of the compressive load displacement curves of both PC and SF pastes.

Conclusions

A CF loading of 0.5 wt % yielded a 21% increase in the compressive strengths of PC pastes and increases in the splitting tensile strengths of PC and SF pastes of 52% and 32% respectively. In contrast, a CNF loading of 0.5 wt % had no significant effect on the compressive and splitting tensile strengths of PC and SF pastes. Neither a CNF loading of 0.5 wt % nor a CF loading of 0.5 wt % had a significant effect on the slopes of the compressive load displacement curves of PC and SF pastes.

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Durability

The durability of PC and SF pastes with CNF and CF loadings of 0.5 and 2 wt % and their corresponding baselines was studied by analyzing their leaching kinetics in DI water and the

effects of accelerated decalcification by a 7M NH_4NO_3 solution on their mass loss, their water absorption capacities, and their compressive strengths.

Water Absorption Capacity and Water Porosity

The results of water absorption tests on PC and SF pastes reinforced with various CNF loadings are shown in figure 6.11. The water porosity at 48 hours of PC and SF pastes reinforced with CNF are shown in table 6.3.

The results of the water absorption tests confirmed the known fact that an increase in the water to cement ratio yields an increase in the porosity of cement pastes (Lea, 1937). Water absorption tests found that CNF loadings up to 0.5 wt % had no significant effect on the water porosity at 48 hours of the PC and SF pastes. A decrease in the water porosity at 48 hours of the PC and SF pastes occurred for the higher CNF loading of 2 wt %.

Table 6.3 Water Porosity

Paste Type	w/c	CNF (wt %)	Average Water Porosity (%)	Standard Deviation
PC	0.325	0	25.6	0.6
		0.005	23.3	1.2
		0.02	22.9	0.1
		0.05	24.0	0.2
		0.50	23.1	0
	0.435	0	31.1	0.1
		2	27.6	0.5
SF	0.365	0	22.1	0.1
		0.005	25.6	0.1
		0.02	24.9	0.6
		0.05	25.0	0.2
		0.50	24.0	0.1
	0.45	0	30.6	0.2
		2	28.5	0.3

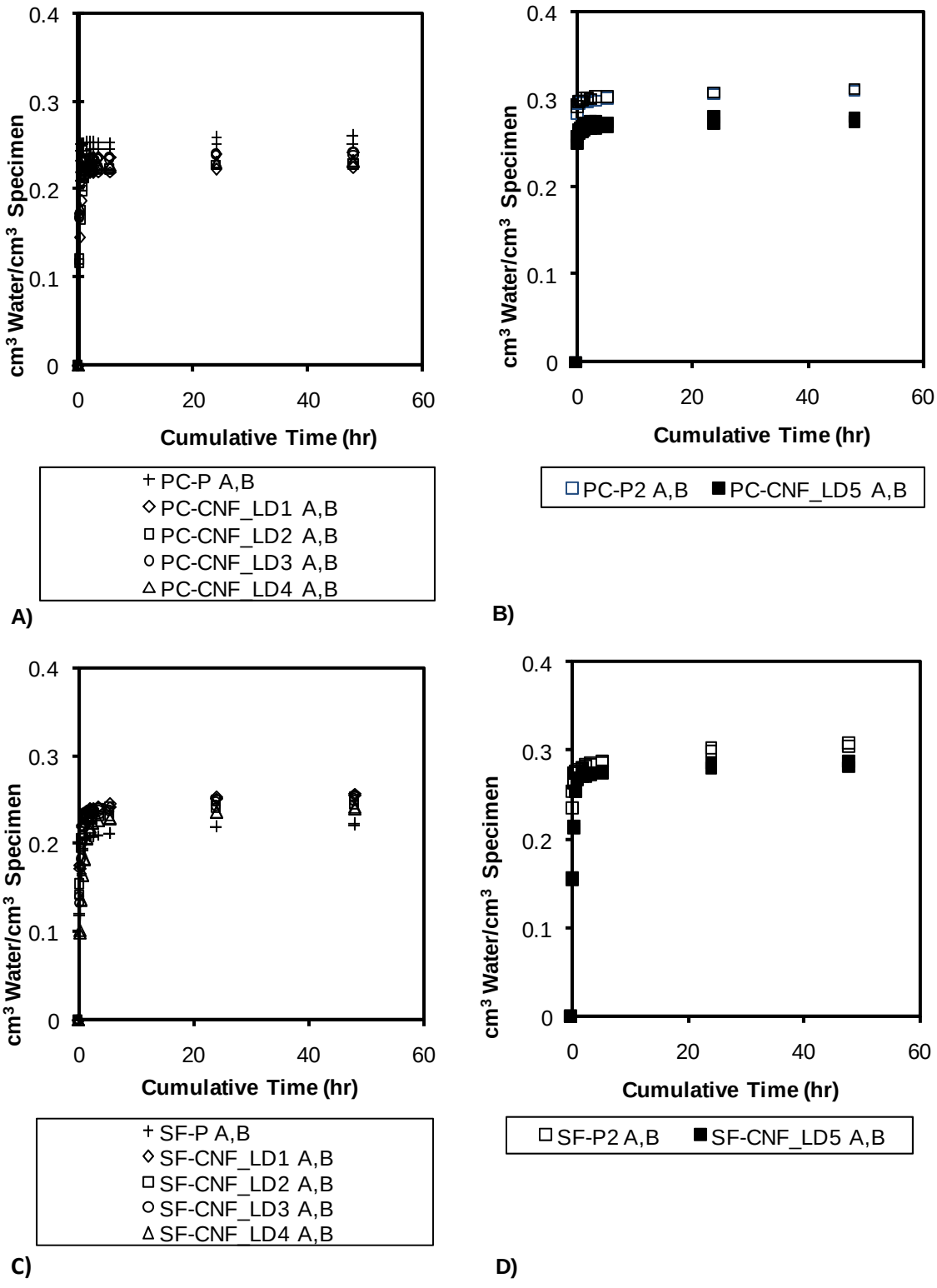


Figure 6.9 Effect of CNF loading on the water absorption capacities of: A) PC pastes at w/c=0.325, B) PC pastes at w/c=0.435, C) SF pastes at w/c=0.365, and D) SF pastes at w/c=0.45.

The following conclusions were drawn:

- CNF loadings from 0.005 to 0.5 wt % had no significant effect on the water porosity of PC paste at w/c=0.325.
- A CNF loading of 2 wt % yielded a decrease of about 12% in the water porosity of PC paste at w/c=0.435.
- CNF loadings from 0.005 to 0.5 wt % had no significant effect on the water porosity of SF paste at w/c=0.365.
- A CNF loading of 2 wt % yielded a decrease of about 7% in the water porosity of SF paste at w/c=0.365.

Kinetics of degradation through leaching

The effects of DI leaching on the release flux of calcium from PC and SF pastes reinforced with 0.5 wt % and 2 wt% CNF and 0.5 wt% CF are shown in figure 6.10 and 6.11, respectively. The following conclusions were drawn:

- A CNF loading of 0.5 wt% and a CF loading of 0.5 wt% had no significant effect on the flux of calcium from the PC and SF pastes.
- A CNF loading of 2 wt% had no significant effect on the flux of calcium for the PC paste with w/c=0.435.
- 2 wt% CNF loading slightly decreased the release flux of calcium for the SF paste with w/c=0.45

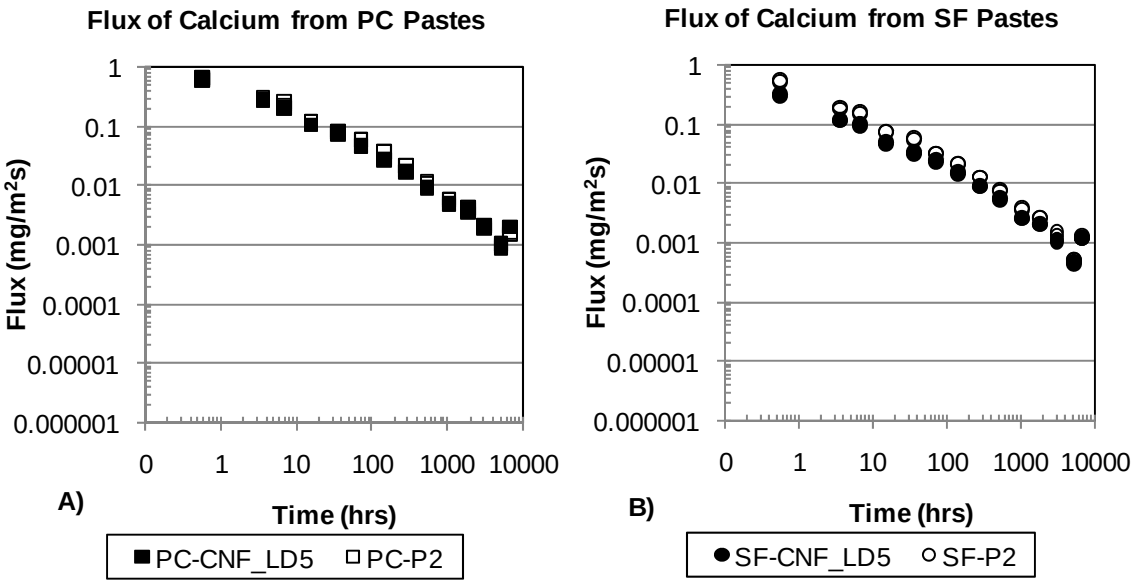


Figure 6.10 Flux of calcium from cement pastes during leaching with DI water: A) PC pastes with w/c=0.435, B) SF pastes with w/c=0.45

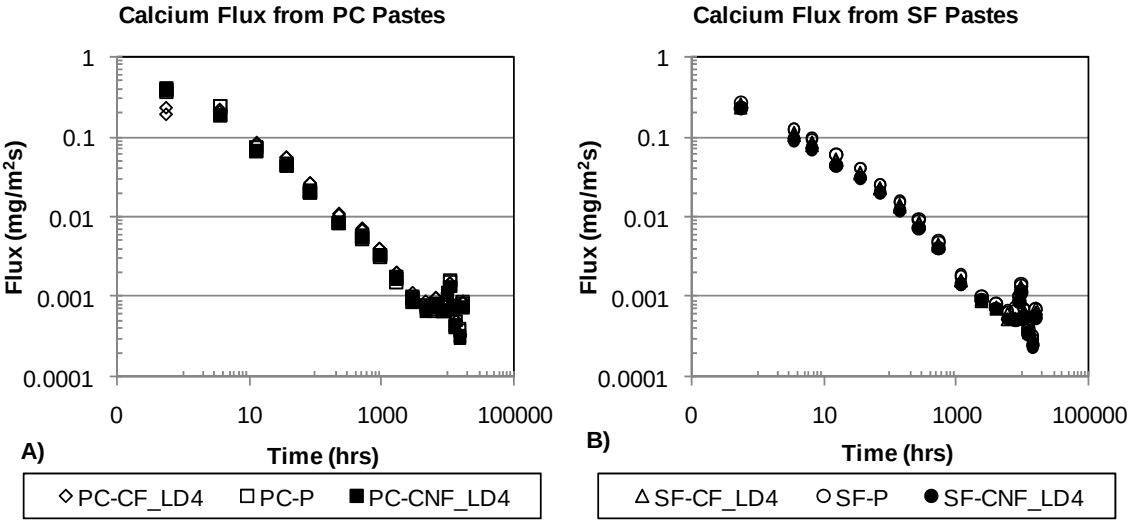


Figure 6.11 Flux of calcium from cement pastes leached with DI water: A) PC pastes at w/c=0.325, B) SF pastes at w/c=0.365

Accelerated Decalcification using NH_4NO_3 solution

Mass Loss as a function of time

The percent mass loss with time due to decalcification with NH_4NO_3 is shown in figure 6.11 for PC pastes with no fibers, and PC pastes reinforced with 0.5 wt% CNF and PC pastes reinforced with 0.5 wt% CF (figure 6.11 A) and SF pastes with no fibers, SF pastes reinforced with 0.5 wt% CNF and SF pastes reinforced with 0.5 wt% CF (figure 6.11 B). The following conclusions were drawn:

- After 95 days of decalcification there was no significant difference in mass loss between PC pastes reinforced with 0.5 wt% CNF, CF, and the PC pastes with no fibers.
- After 95 days of decalcification the % mass loss of SF pastes reinforced with 0.5 wt% CNF was 9% lower than that of the SF pastes with no fibers at w/c=0.365.

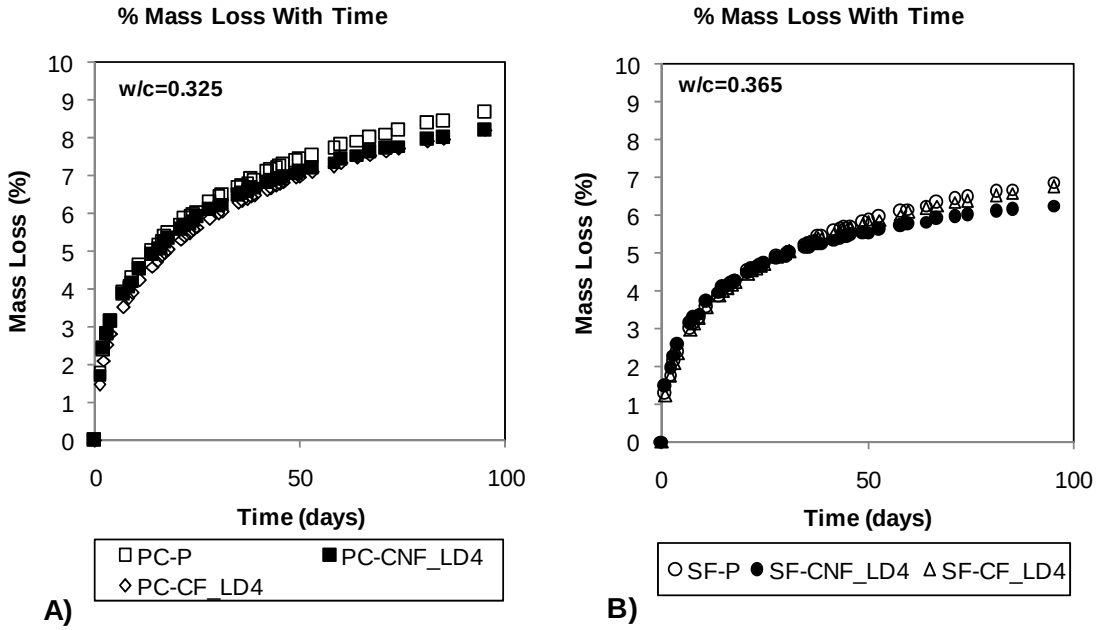


Figure 6.12 Percent mass loss of cement pastes as a function of time during decalcification with NH4NO3 solution a) PC pastes at w/c=0.325 b) SF pastes at w/c=0.365

The percent mass loss with time due to decalcification with NH_4NO_3 for PC pastes with no fibers and PC pastes reinforced with 2 wt% CNF are shown in figure 6.13 A and SF pastes with no fibers and SF pastes reinforced with 2 wt% CNF are shown in figure 6.13 B. The following conclusions were drawn based on these results:

- After 95 days of decalcification the % mass loss of PC pastes reinforced with 2 wt% CNF was 23% lower than that of the PC pastes with no fibers at w/c=0.435.
- After 95 days of decalcification the % mass loss of SF pastes reinforced with 2 wt% CNF was 20% lower than that of the PC pastes with no fibers at w/c=0.45.

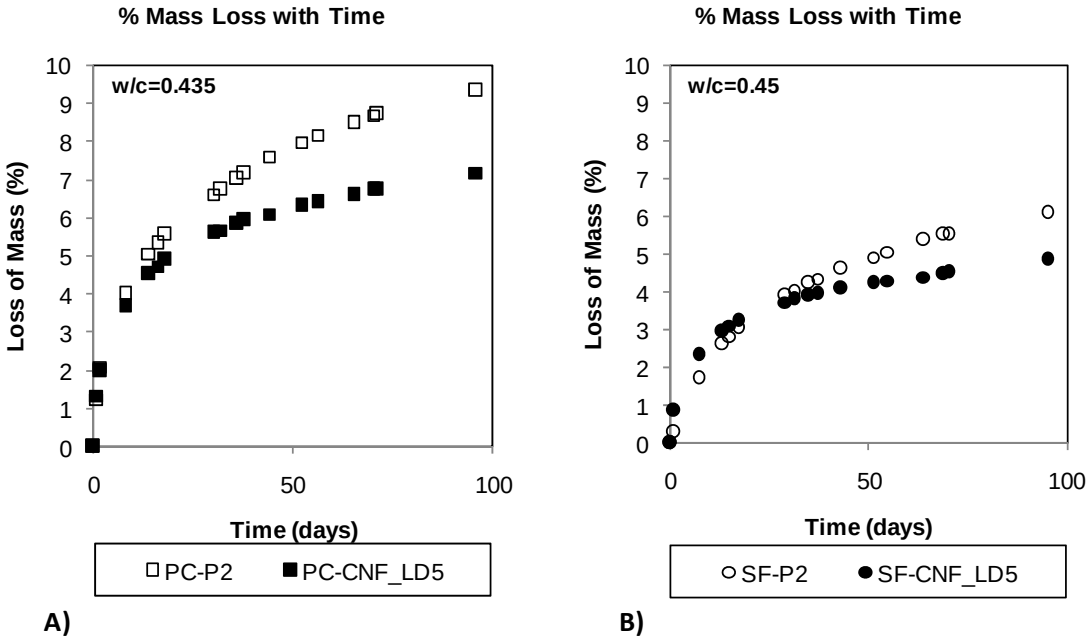


Figure 6.13 Percent mass loss of cement pastes as a function of time during decalcification with NH4NO3 solution a) PC pastes at w/c=0.435 b) SF pastes at w/c=0.45

The average % mass loss of the PC and SF pastes cement specimens after NH_4NO_3 degradation for 95 days are shown in table 6.5.

Table 6.4 Average % mass loss of the PC and SF cement specimens after NH_4NO_3 degradation for 95 days

Paste Type	Average % Mass Loss	Standard Deviation
PC-P	8.7	0.17
PC-CNF_LD4	8.2	0.11
PC-CF_LD4	8.2	0.14
PC-P2	9.3	0.53
PC-CNF_LD5	7.2	0.11
SF-P	6.9	0.20
SF-CNF_LD4	6.3	0.15
SF-CF_LD4	6.8	0.13
SF-P2	6.1	0.13
SF-CNF_LD5	4.9	0.12

Conclusions

There was no significant difference in the % mass loss after 95 days of decalcification of PC and SF pastes reinforced with 0.5 wt% CNF or CF and PC and SF pastes with no fibers. In contrast, the mass loss in PC and SF pastes reinforced with 2 wt% CNF there was 23% and 20% less mass loss respectively than pastes with no fibers.

Effect of Decalcification on the Mechanical Properties

Compressive Strength

The results of compressive strength tests on PC Pastes with and without 0.5 wt% CF and CNF which were degraded using NH_4NO_3 for ca. 95 days are shown in figure 6.14. The following conclusions were drawn:

- After decalcification there was no significant difference in the compressive strengths between the plain PC pastes, PC pastes reinforced with 0.5 wt % CNF and PC pastes reinforced with 0.5 wt % CF at w/c=0.325.
- After decalcification there was no significant difference in the compressive strengths between the PC pastes reinforced with 2 wt % CNF and plain PC pastes at w/c=0.435.

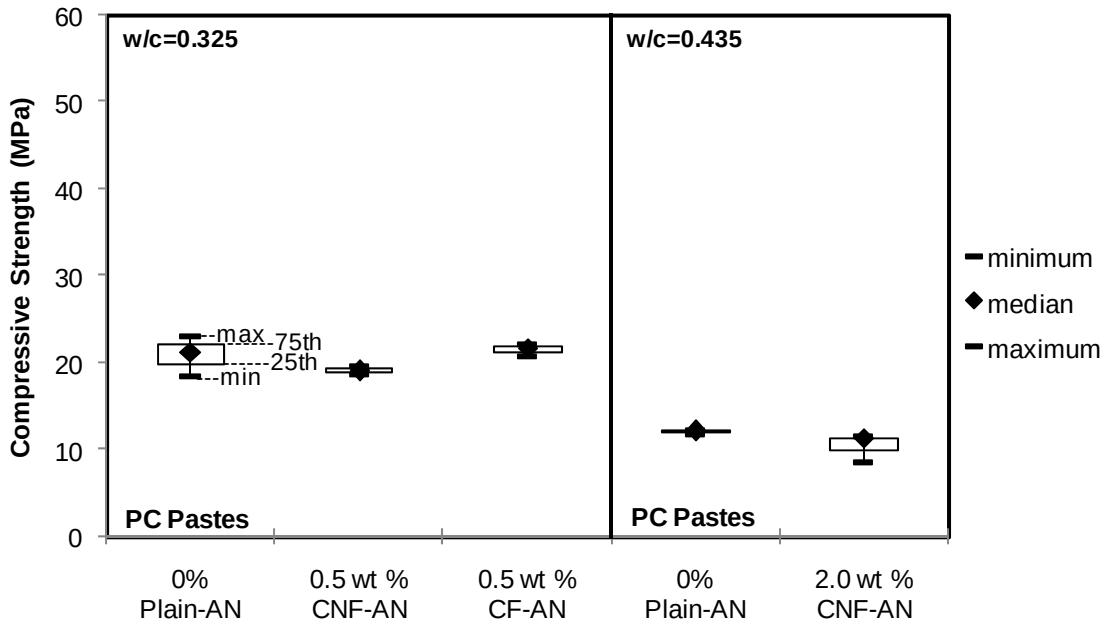


Figure 6.14 Compressive strength of NH4NO3 degraded PC pastes

Figure 6.15A shows the results of compressive strength tests on two types of PC pastes at w/c=0.325: PC paste with no fibers and PC paste with 0.5 wt % CNF. Figure 6.13B shows the results of compressive strength tests on two types of PC pastes at w/c=0.435: PC paste with no fibers and PC paste with 2 wt % CNF. The pastes were tested after curing for 28 days and after accelerated decalcification for ca. 95 days. The following conclusions were drawn:

- Exposure to NH_4NO_3 for ca. 95 days yielded a 51% decrease in the median compressive strengths of the PC pastes reinforced with 0.5 wt % CNF and a 42% decrease in the median compressive strength of PC pastes with no fibers.

- Exposure to NH_4NO_3 yielded a 62% decrease in the median compressive strength of the plain PC pastes while a 48% decrease for PC pastes reinforced with 2 wt% CNF.

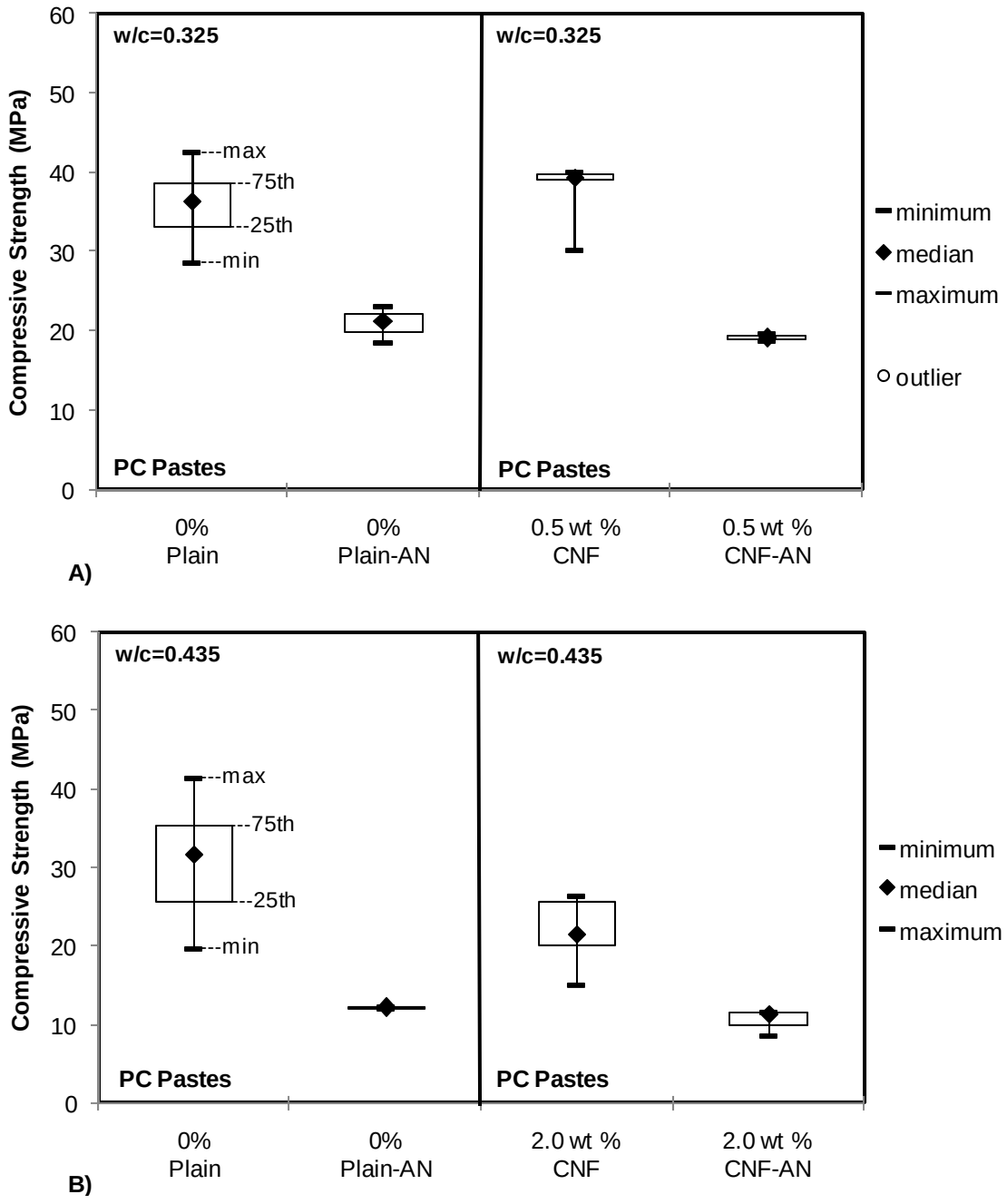


Figure 6.15 Effect of CNF on the NH_4NO_3 degradation of PC pastes: A) 0.5 wt% CNF, B) 2 wt% CNF

Figure 6.16 shows the results of compressive strength on two types of PC pastes at w/c=0.325; PC paste with no fibers and PC paste with 0.5 wt % CF. The pastes were tested after curing for about 28 days and after exposure to NH_4NO_3 for 95 days. The following conclusions were drawn:

- Decalcification yielded a 53% decrease in the median compressive strength of PC paste reinforced with 0.5 wt % CF at w/c=0.325.

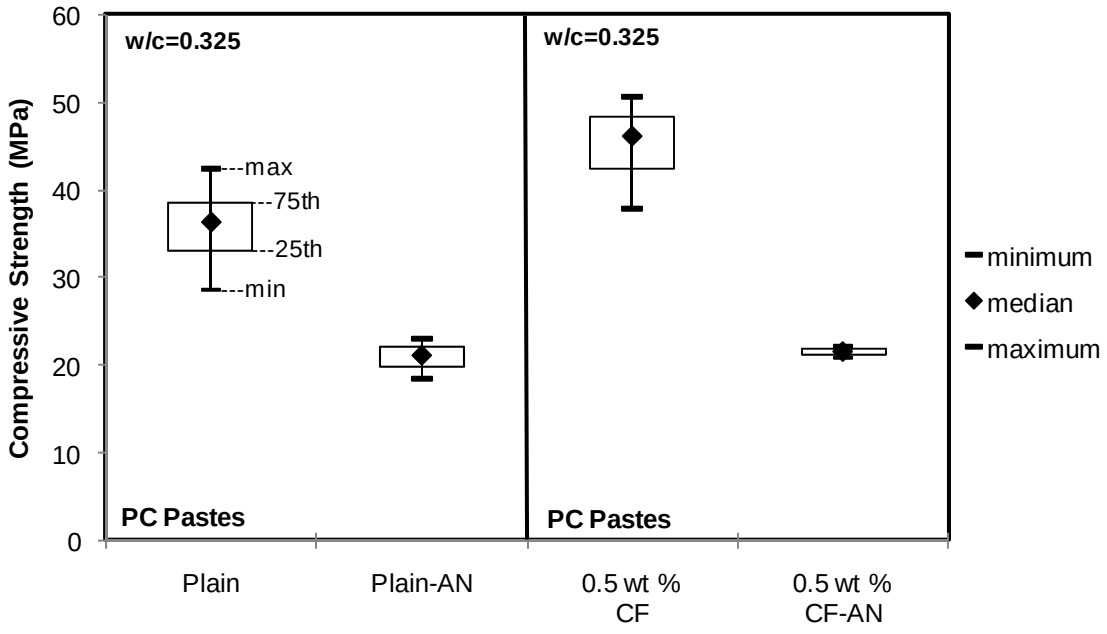


Figure 6.16 Effect of 0.5 wt % CF reinforcement on the compressive strength of decalcified PC pastes

The results of compressive strength tests on SF cement pastes, which were decalcified using NH_4NO_3 for 95 days are shown in figure 6.17. The following conclusions were drawn:

- After decalcification there was no significant difference in the compressive strengths of plain SF pastes, SF pastes reinforced with 0.5 wt % CNF and SF pastes reinforced with 0.5 wt % CF at w/c=0.365.

- After decalcification there was no significant difference in the compressive strengths of SF pastes reinforced with 2 wt % CNF and plain SF pastes at w/c=0.45.

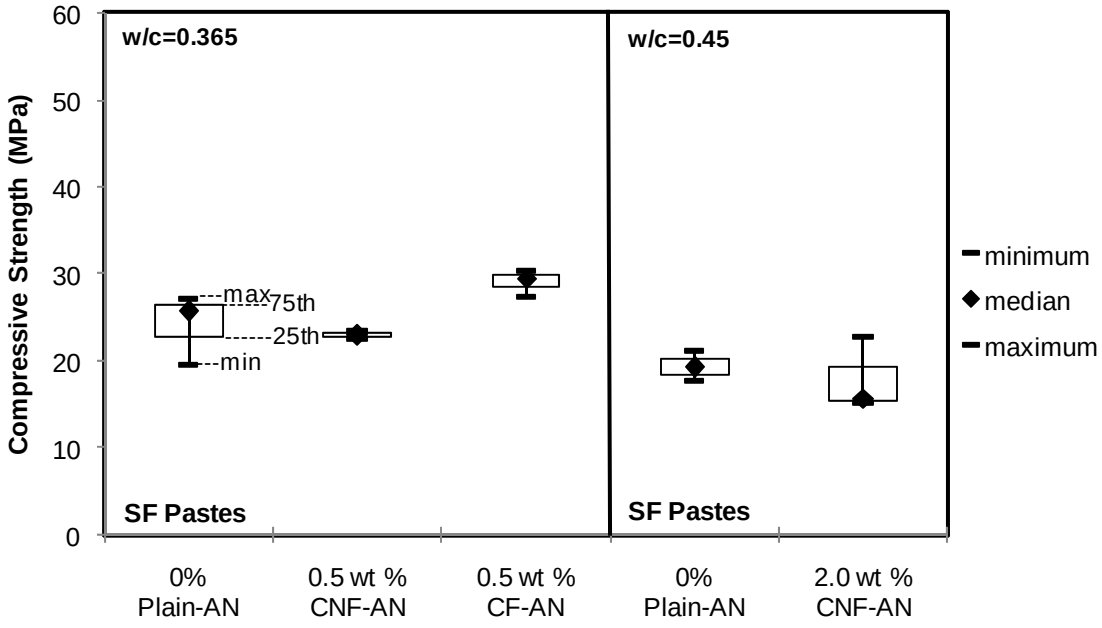
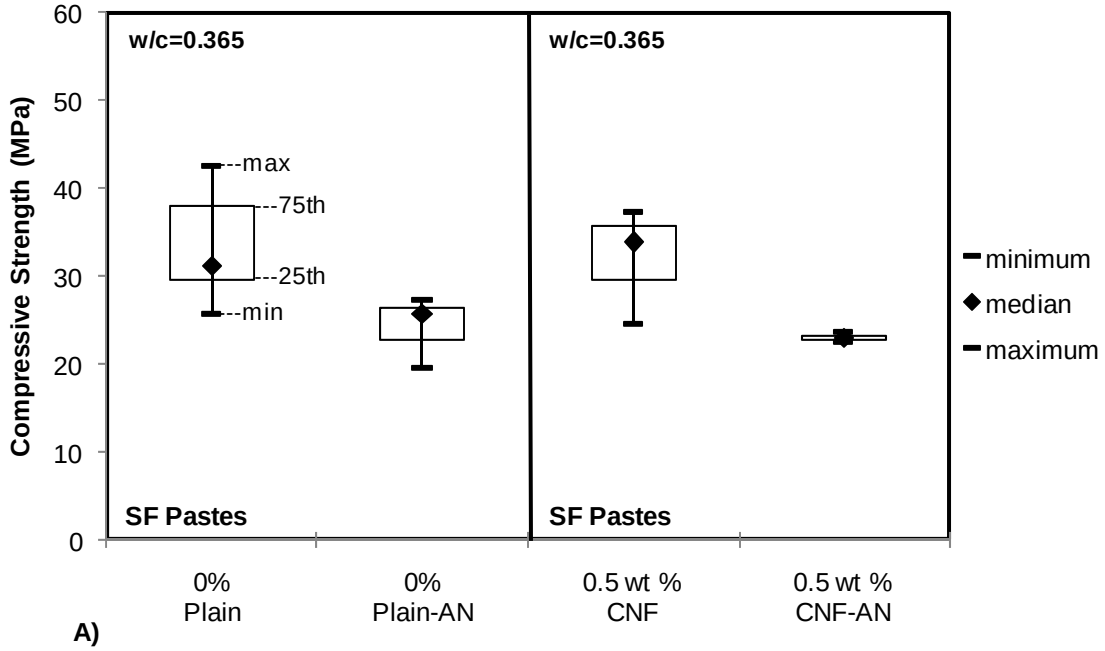


Figure 6.17 Compressive strengths of NH_4NO_3 degraded SF pastes

Figure 6.18(A) shows the results of compressive strength tests on two types of SF pastes at w/c=0.325: SF paste with no fibers and SF paste with 0.5 wt % CNF. Figure 6.18(B) shows the results of compressive strength tests on two types of SF pastes at w/c=0.435: SF paste with no

fibers and SF paste with 2 wt % CNF. The pastes were tested after curing for a minimum of 28 days and after exposure to NH_4NO_3 for 95 days. The following conclusions were drawn:

- Decalcification yielded a 18% decrease in the median compressive strengths of the plain SF pastes with at w/c=0.365.
- Decalcification yielded a 40% decrease in the median compressive strengths of plain SF pastes at w/c=0.45.
- Decalcification yielded a 48% decrease in the median compressive strengths of SF pastes reinforced with 2 wt % CNF at w/c=0.45.



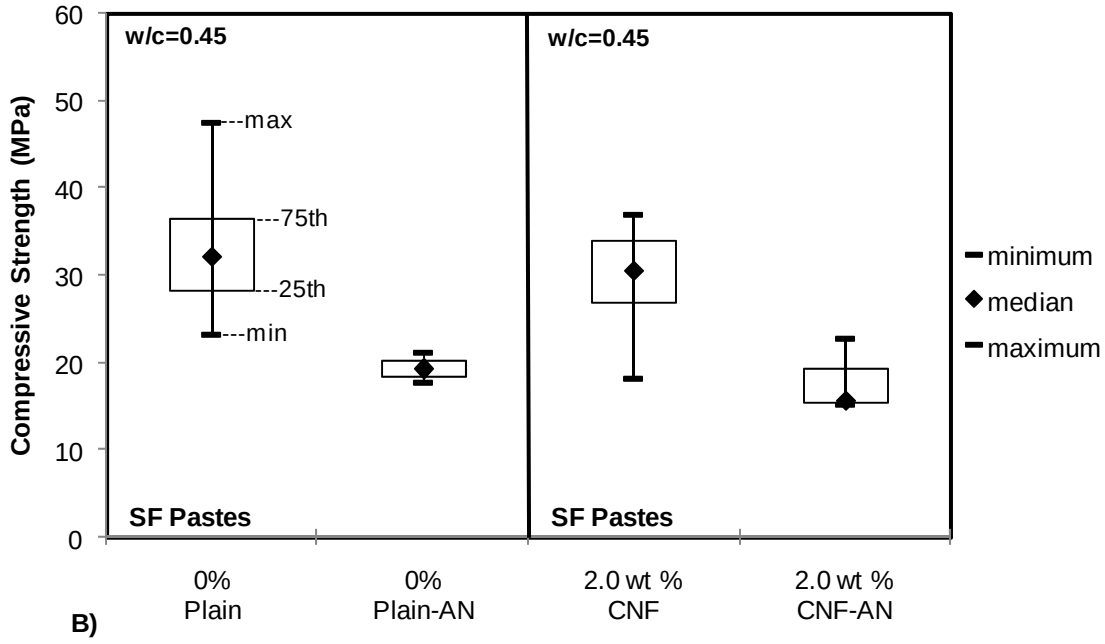


Figure 6.18 Effect of fiber reinforcement on the compressive strength of NH_4NO_3 degraded SF pastes 95 day exposure: A) 0.5 wt% CNF, B) 2 wt% CNF

Figure 6.19 shows the results of compressive strength on two types of SF pastes at w/c=0.365: SF paste with no fibers and SF paste with 0.5 wt % CF. The pastes were tested after curing for a minimum of 28 days and after accelerated decalcification for 95 days. The following conclusions were drawn:

- There was no change in the compressive strengths of SF pastes reinforced with 0.5 wt % CF and SF pastes with no fibers at w/c=0.365 after decalcification.

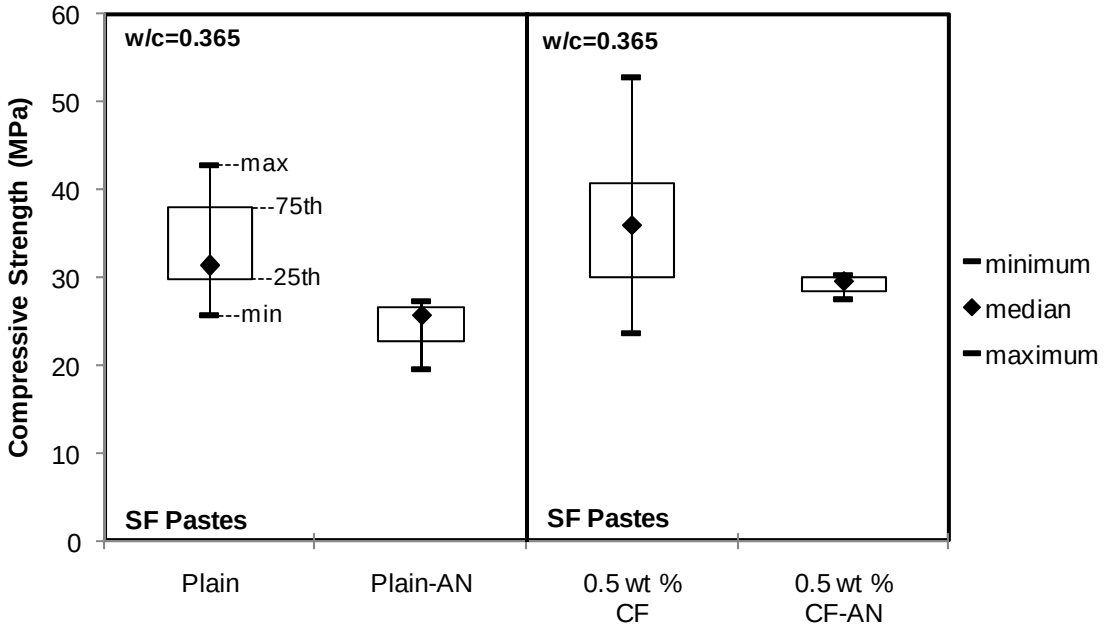


Figure 6.19 Effects of 0.5 wt % CF reinforcement on the compressive strength of NH_4NO_3 degraded SF pastes 95 day exposure

Compressive Load Displacement Curves

Figure 6.26 shows the effect of NH_4NO_3 degradation on the compressive load displacement curves of PC pastes. The slopes prior to failure are summarized in tables 6.9 and 6.10, respectively. The following conclusions were drawn based on these results:

- After decalcification the median slope of the compressive load displacement curves of PC pastes reinforced with 2 wt % CNF was 30% lower than that of plain PC pastes at w/c=0.435.
- Decalcification yielded a 44% decrease in the median slope of the compressive load displacement curves of PC pastes reinforced with 0.5 wt % CNF at w/c=0.325.
- Decalcification yielded a 50% decrease in the median slope of the compressive load displacement curves of plain PC pastes w/c=0.435.
- Decalcification yielded a 50% decrease in the median slope of the compressive load displacement curves of PC pastes reinforced with 2 wt % CNF at w/c=0.435.

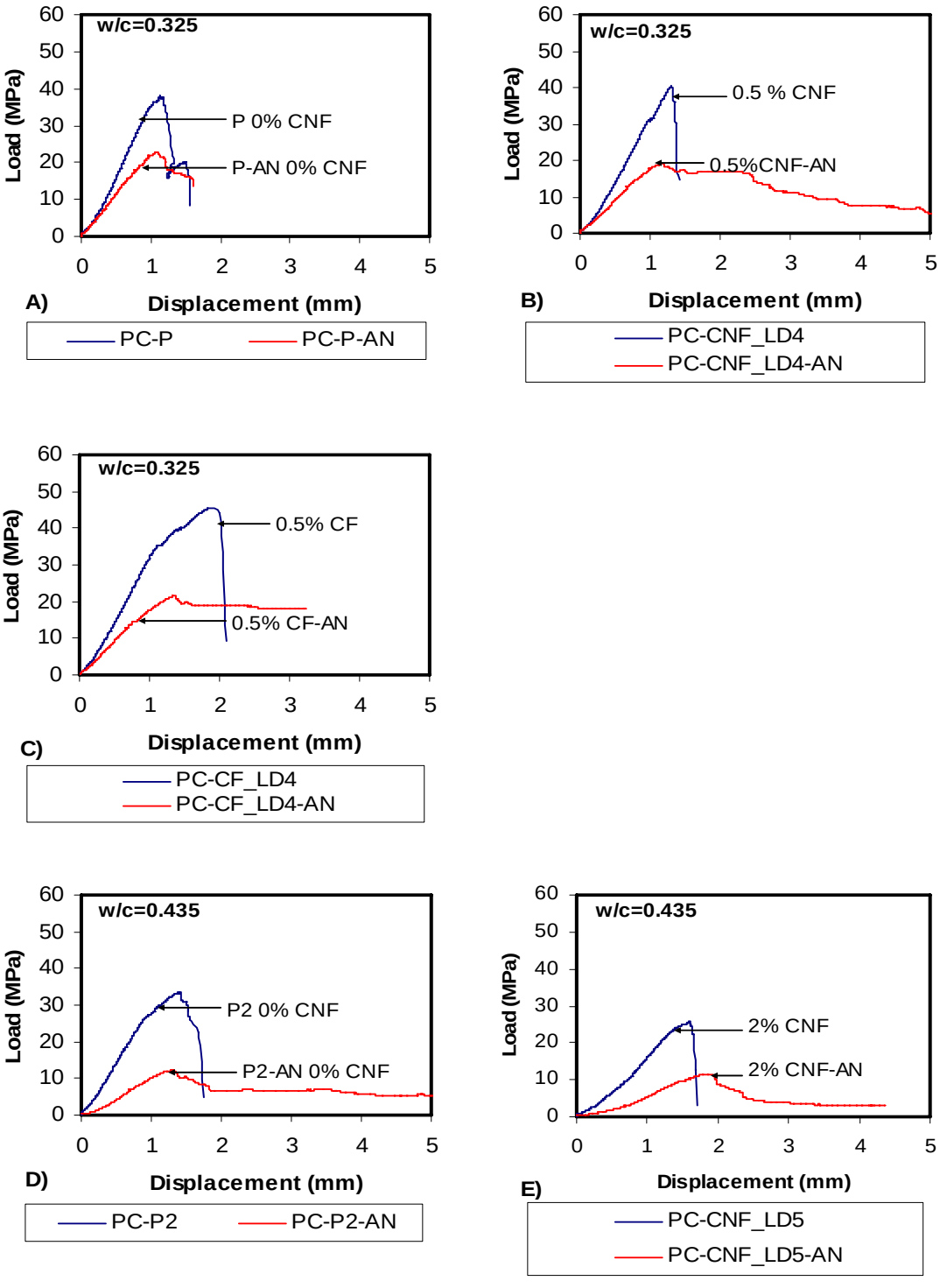


Figure 6.20 Effects of 95 day exposure to NH_4NO_3 on the compressive load displacement curves of PC pastes

Table 6.5 Effect of NH_4NO_3 degradation on the slope of the compressive load displacement curves of PC pastes prior to failure

				Slope (MPa/mm)				
Paste Type	**w/c**	**Specimen Type**	**Fiber (wt%)**	**Mean**	**Standard Deviation**	**Minimum**	**Median**	**Maximum**
PC	0.325	PC-P	0	27.4	9.6	12.2	23.4	40.6
		PC-P-AN	0	22.3	1.8	20.9	21.8	24.3
		PC-CNF_LD4	0.50	34.1	4.4	25.8	33.9	39.6
		PC-CNF_LD4-AN	0.50	20.7	4.8	17.4	19.0	25.7
		PC-CF_LD4	0.50	23.5	6.3	15.0	22.9	35.2
		PC-CF_LD4-AN	0.50	19.4	0.4	19.2	19.3	19.8
	0.435	PC-P2	0	32.4	10.1	17.0	31.4	47.9
		PC-P2-AN	0	13.0	0.5	12.6	12.8	13.5
		PC-CNF_LD5	2	19.0	3.5	13.3	17.8	25.2
		PC-CNF_LD5-AN	2	8.1	1.9	5.9	9.0	9.4

Figure 6.27 shows the effect of NH_4NO_3 degradation on the load displacement curves of SF pastes which are summarized in tables 6.9 and 6.10 respectively. The following conclusions were drawn based on these results:

- Decalcification had no significant effect on the slopes of the load displacement curves of plain SF pastes at w/c=0.365. In contrast, decalcification yielded a decrease in the slopes of SF pastes reinforced with fibers and plain SF pastes at w/c=0.45.

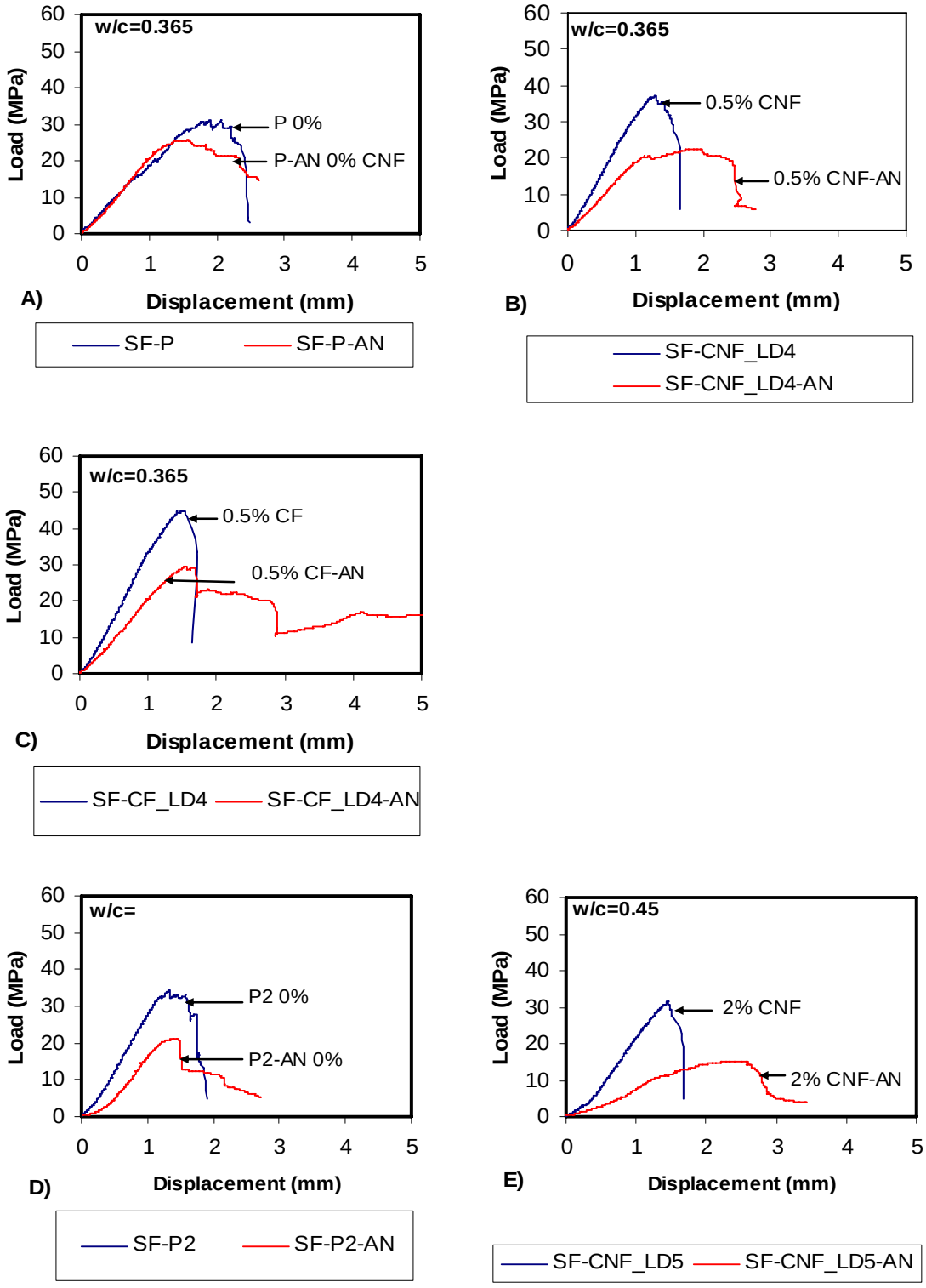


Figure 6.21 Effect of NH_4NO_3 degradation on the load displacement curves SF pastes.

Table 6.6 Effects of NH_4NO_3 degradation on the slope of the compressive load displacement curves of SF pastes prior to failure

				Slope (MPa/mm)				
Paste Type	**w/c**	**Specimen Type**	**CNF (wt %)**	**Mean**	**Standard Deviation**	**Minimum**	**Median**	**Maximum**
SF	0.365	SF-P	0	19.2	7.1	7.7	17.6	31.0
		SF-P-AN	0	19.4	4.9	13.9	21.8	22.7
		SF-CNF_LD4	0.50	29.0	6.6	17.0	32.9	35.1
		SF-CNF_LD4-AN	0.50	19.6	0.8	18.8	19.7	20.3
		SF-CF_LD4	0.50	24.6	7.9	16.6	20.1	37.3
		SF-CF_LD4-AN	0.50	21.3	1.7	19.3	22.3	22.3
	0.45	SF-P2	0	28.0	3.7	21.7	28.5	33.4
		SF-P2-AN	0	17.9	3.7	15.4	16.2	22.2
		SF-CNF_LD5	2	29.0	4.3	21.0	31.3	32.8
		SF-CNF_LD5-AN	2	13.5	3.1	10.8	12.8	16.9

CHAPTER VII

CONCLUSIONS

CNF loadings up to 2 wt % had no significant effect on the mechanical properties of PC and SF pastes, except in the case of PC pastes reinforced with 2 wt% CNF where there was a decrease in the compressive strength. Addition of 0.5 wt% CF impacted the mechanical properties of PC and SF pastes by increasing their compressive and splitting tensile strengths.

A CNF loading of 0.5 wt% and a CF loading of 0.5 wt% had no significant effect on the mass loss of the PC paste due to decalcification. The higher CNF loading of 2 wt% seemed to increase the durability of the PC pastes. This increase in the durability was characterized by a lower water porosity, a lower mass loss and a lower loss of compressive strength due to exposure to ammonium nitrate solution.

Due to the heterogeneous nature of cement pastes there is a high level of variability in mechanical test results. It is therefore necessary to have a large number of replicates (greater than 5) for each test in order to draw accurate conclusions from the results obtained.

CHAPTER VIII

FURTHER WORK

A CNF loading of 2 wt% showed the most potential for improving the durability of PC pastes. This CNF loading was also found to decrease the compressive strength of PC pastes. This lower compressive strength could possibly be attributed to the presence of large clumps of fibers visible in the paste. This hypothesis should be investigation by studying the level of fiber dispersion within the paste and effective means of improving that dispersion.

Additional investigations are also necessary to conclusively determine the effect of CNF loading on the durability of PC and SF pastes. This investigation should include study of the pastes microstructure using scanning electron microscopy, and a more detailed look at the porosity and pore size distribution using mercury intrusion porosimetry and BET porosimetry.

Appendix

Compressive Strength Data

Baseline

PC-P; w/c=0.325

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	15090
3.7	2.0	14660
3.7	2.0	17440
3.8	2.0	12880
3.8	2.0	14010
3.8	2.0	19030
3.8	2.0	16680
3.9	2.0	17610
3.8	2.0	17590
3.8	2.0	13160
3.7	2.0	17000
3.8	2.0	17270
3.8	2.0	15380
3.7	2.0	16320
3.8	2.0	15110

PC-P2; w/c=0.435

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.6	2.0	16080
3.5	2.0	8670
3.4	2.0	18100
3.5	2.0	18450
3.4	2.0	14330
3.7	2.0	14000
3.8	2.0	15160
3.8	2.0	10780
3.7	2.0	13680
3.8	2.0	9270

PC-CNF; w/c=0.325; 0.005 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	18120
3.8	2.0	14330
3.8	2.0	17810
3.8	2.0	16610
3.9	2.0	19460

PC-CNF; w/c=0.325; 0.02 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	12900
3.9	2.0	20800
3.8	2.0	19240
3.8	2.0	18400
3.8	2.0	15690

PC-CNF; w/c=0.325; 0.05 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	12900
3.9	2.0	20800
3.8	2.0	19240
3.8	2.0	18400
3.8	2.0	15690

PC-CNF; w/c=0.325; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	18090
3.7	2.0	17690
3.8	2.0	17360
3.8	2.0	17750
4.0	2.0	13550
3.8	2.0	17840
3.9	2.0	5350
3.9	2.0	17740

PC-CNF; w/c=0.435; 2 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	9300
3.7	2.0	6780
3.8	2.0	11460
3.8	2.0	9660
3.7	2.0	11830
3.8	2.0	9740
4.0	2.0	8840
3.7	2.0	11600
3.9	2.0	11590
3.9	2.0	8170

PC-CF; w/c=0.325; 0.5 wt% CF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	22400
3.7	2.0	22800
3.7	2.0	19540
3.9	2.0	21000
3.9	2.0	17760
3.8	2.0	20400
3.8	2.0	16980
3.8	2.0	21500

SF-P; w/c=0.365

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	18430
3.6	2.0	19140
3.7	2.0	14020
3.8	2.0	16990
4.0	2.0	14550
3.9	2.0	11540
4.0	2.0	14010
3.8	2.0	12760
3.9	2.0	13310

SF-P2; w/c=0.45

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	21400
3.8	2.0	10430
3.8	2.0	16720
3.8	2.0	14510
3.7	2.0	17670
3.8	2.0	14390
3.8	2.0	13410
3.8	2.0	12360
3.8	2.0	15540
3.8	2.0	12330

SF-CNF; w/c=0.365; 0.005 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	13760
3.9	2.0	13100
3.9	2.0	15520
3.9	2.0	13490
3.9	2.0	10660

SF-CNF; w/c=0.365; 0.02 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	16670
3.9	2.0	11660
3.9	2.0	15350
3.9	2.0	17110
3.9	2.0	10090

SF-CNF; w/c=0.365; 0.05 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	15850
3.8	2.0	14380
3.9	2.0	13880
3.9	2.0	14670
3.9	2.0	15050

SF-CNF; w/c=0.365; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	12180
3.7	2.0	15950
3.6	2.0	15890
3.8	2.0	16660
3.8	2.0	16170
3.8	2.0	14500
3.9	2.0	11100
3.9	2.0	13670

SF-CNF; w/c=0.45; 2 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	13780
3.8	2.0	16610
3.8	2.0	10750
3.8	2.0	12940
3.7	2.0	11830
3.8	2.0	13670
3.8	2.0	15700
3.9	2.0	14170
3.7	2.0	16210
3.9	2.0	8170

SF-CF; w/c=0.365; 0.5 wt% CF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	19550
3.7	2.0	16440
3.7	2.0	23700
3.7	2.0	18350
3.9	2.0	13360
3.9	2.0	15910
3.9	2.0	17790
3.8	2.0	13490
3.8	2.0	10660

Ammonium Nitrate Solution Degraded Specimens (95 days exposure)

PC-P; w/c=0.325

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	8280
3.8	2.0	9510
3.8	2.0	10310

PC-P2; w/c=0.435

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.6	2.0	5330
3.7	2.0	5440
3.7	2.0	5450

PC-CNF; w/c=0.325; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	8570
3.8	2.0	8780
3.9	2.0	8360

PC-CNF; w/c=0.435; 2 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	5200
3.7	2.0	3860
3.8	2.0	5080

PC-CF; w/c=0.325; 0.5 wt% CF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	9510
3.7	2.0	9750
3.7	2.0	10030

SF-P; w/c=0.365

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	8790
3.8	2.0	12240
3.7	2.0	11580

SF-P2; w/c=0.45

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	8670
3.8	2.0	7970
3.7	2.0	9530

SF-CNF; w/c=0.365; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	10800
3.9	2.0	10260
3.8	2.0	10280

SF-CNF; w/c=0.45; 2 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	6830
3.9	2.0	7060
4.0	2.0	10240

SF-CF; w/c=0.365; 0.5 wt% CF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	13260
3.8	2.0	12350
3.8	2.0	13620

Splitting Tensile Strength Data

Baseline

PC-P; w/c=0.325

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
4.0	2.0	7200
4.0	2.0	5980
4.0	2.0	6790
4.0	2.0	5230
4.0	2.0	7710
3.8	2.0	3070
3.8	2.0	2930
3.7	2.0	2660

PC-P2; w/c=0.435

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	3040
3.5	2.0	3720
3.6	2.0	3090

PC-CNF; w/c=0.325; 0.005 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
4.0	2.0	7790
3.8	2.0	2510
3.9	2.0	3730

PC-CNF; w/c=0.325; 0.02 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	2560
3.8	2.0	2320
3.8	2.0	2890

PC-CNF; w/c=0.325; 0.05 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	2730
3.9	2.0	4710
3.9	2.0	2890

PC-CNF; w/c=0.325; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	6470
3.7	2.0	4990
3.7	2.0	5690

PC-CNF; w/c=0.435; 2 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	3540
3.8	2.0	3930
3.8	2.0	2660

PC-CF; w/c=0.325; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	9300
3.8	2.0	8040
3.7	2.0	7470

SF-P; w/c=0.365

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	5240
3.7	2.0	5360
3.6	2.0	3750
3.6	2.0	5110
3.9	2.0	3720
3.9	2.0	4680
3.9	2.0	2770

SF-P2; w/c=0.45

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	2160
3.8	2.0	3830
3.7	2.0	2170

SF-CNF; w/c=0.365; 0.005 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	3380
3.9	2.0	3840
3.9	2.0	4100

SF-CNF; w/c=0.365; 0.02 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	5080
3.9	2.0	5060
3.9	2.0	3090

SF-CNF; w/c=0.365; 0.05 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	3650
3.8	2.0	3650
3.9	2.0	3460

SF-CNF; w/c=0.365; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	3250
3.8	2.0	3570
3.7	2.0	5850
3.7	2.0	7050

SF-CNF; w/c=0.45; 2 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	3750
3.9	2.0	3480
3.9	2.0	3510

SF-CF; w/c=0.365; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.6	2.0	5640
3.8	2.0	7360
3.6	2.0	5430
3.8	2.0	7240

Ammonium Nitrate Solution Degraded Specimens (95 days exposure)

PC-P; w/c=0.325

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	5520
3.8	2.0	6430
3.8	2.0	7730

PC-P2; w/c=0.435

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.6	2.0	3080
3.6	2.0	1798

PC-CNF; w/c=0.325; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	5570
3.9	2.0	5650
3,8	2.0	2720

PC-CNF; w/c=0.435; 2 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.7	2.0	2510
3.9	2.0	2210
3.8	2.0	2270

PC-CF; w/c=0.325; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	5730
4.0	2.0	5370
3.8	2.0	5940

SF-P; w/c=0.365

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	3840
3.9	2.0	4150
3.8	2.0	3930

SF-P2; w/c=0.45

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	2780
3.8	2.0	2590
3.9	2.0	2920

SF-CNF; w/c=0.365; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.8	2.0	4040
3.8	2.0	3830
3.7	2.0	3870

SF-CNF; w/c=0.45; 2 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	2590
3.8	2.0	60600
3.9	2.0	3700

SF-CF; w/c=0.325; 0.5 wt% CNF

Average Height (in)	Average Diameter (in)	Ultimate Strength (lb)
3.9	2.0	5230
3.8	2.0	3930
3.9	2.0	4570

Leaching with DI Water Data

PC-P; w/c=0.325

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	10.1	10.3	0.706	0.633	12.8	11.7	0.0449	0.0398	0.0956	0.0787	0.118	0.1233	28.7	27.2
2	3.00	9.8	9.8	0.28	0.286	4.8	5.13	0.091	0.0893	0.196	0.192	0.0892	0.0902	25	24.1
3	18.57	10.3	10.3	0.795	0.869	14.7	16.3	0.344	0.374	0.972	0.859	0.127	0.117	47.1	47
4	26.75	10.3	10.2	0.695	0.715	13.1	13.5	0.467	0.444	1.88	1.7	0.133	0.128	42.2	39.5
5	70.50	10.5	10.4	1.12	1.14	21.1	21.3	0.607	0.646	3.36	3.47	0.195	0.139	47.8	50.2
6	238.00	11.3	11.4	2.22	2.26	43.6	43.7	1.04	1.06	4.1	3.91	0.228	0.212	66.6	68.5
7	313.50	11.4	11.4	1.82	1.77	34.7	33.8	1.02	1.03	4.58	4.39	0.189	0.176	56.1	58.6
8	519.00	11.0	11.0	2.19	2.11	40.8	39.4	1.22	1.19	4.38	4.34	0.181	0.168	56.9	56.2
9	1019.50	11.1	11.1	2.61	2.59	47.2	46.6	1.44	1.43	3.84	3.65	0.17	0.165	54.2	58.7
10	1464.00	11.5	11.5	2.88	2.8	50.4	49.8	1.58	1.54	3.65	3.74	0.15	0.1252	50.3	48.1
11	1848.00	11.0	11.1	2.87	2.79	49.9	49.6	1.57	1.54	3.65	3.77	0.153	0.1207	50.1	48.2
12	2040.00	11.2	11.3	2.01	1.96	33.4	32.7	1.48	1.43	3.85	3.89	0.132	0.125	45.5	48.8
13	1680.33	11.5	11.3	1.49	1.48	23.1	23.2	1.21	1.18	4.23	3.95	0.168	0.173	37.6	38.2
14	1009.17	11.2	11.2	0.799	0.779	12.4	12.4	0.992	0.972	4.53	4.58	0.315	0.345	37.8	38.6
15	693.50	11.5	11.4	0.751	0.745	11.5	11.1	1.13	1.04	4.25	4.35	0.158	0.162	37.3	37.1
16	1323.75	11.2	11.2	0.726	0.721	11.1	11.4	1.01	1.15	4.13	4.16	0.154	0.146	33.5	34.6
17	1796.83	11.1	11.3	0.67	0.662	10.1	10.2	1.01	1.07	3.52	3.46	0.147	0.155	31.4	30.5
18	2238.92	11.5	11.4	0.58	0.54	10	10.2	1.08	0.98	3.48	3.71	0.102	0.0936	29	29.8
19	738.50	10.9	10.9	0.405	0.382	5.4	5.77	0.92	0.895	3.74	4.03	0.082	0.0795	21.2	21.5

PC-P2; w/c=0.435

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	11.2	11.2	1.79	1.81	20.7	21.1	0.0325	0.036	0.121	0.136	0.165	0.175	47.4	46.6
2	3.00	11.0	11.1	0.59	0.579	7.06	7.02	0.0875	0.0855	0.173	0.188	0.119	0.125	28.5	29.9
3	3.00	11.0	11.0	0.455	0.444	5.61	5.67	0.104	0.0978	0.189	0.193	0.0901	0.0942	22.7	24.6
4	16.00	11.3	11.4	1.56	1.52	18.4	18.6	0.254	0.239	0.458	0.445	0.276	0.273	65.1	63.9
5	24.32	11.3	11.3	1.44	1.46	16.8	17.4	0.327	0.338	0.526	0.535	0.351	0.345	66	63.6
6	48.00	11.5	11.5	1.87	1.93	22.1	23.4	0.557	0.574	0.906	0.911	0.349	0.344	90.1	92.6
7	96.00	11.5	11.5	2.48	2.56	30.7	31.6	0.807	0.797	1.09	1.08	0.406	0.402	114	113
8	172.17	11.7	11.7	3.06	3.28	37.8	39.2	0.927	0.948	1.29	1.27	0.441	0.44	123	120.8
9	335.83	12.1	12.0	4.07	4.31	50.8	55.2	1.36	1.43	1.03	0.921	0.402	0.374	129	122
10	721.12	11.6	11.5	5.58	6.07	70.1	76.1	1.83	1.86	0.755	0.778	0.406	0.388	140	133
11	815.88	11.4	11.6	4.54	4.93	32.3	35	1.58	1.61	0.954	0.982	0.293	0.287	113	110
12	1705.50	11.5	11.5	7.1	7.59	49.1	52.4	2.02	2.06	0.875	0.97	0.301	0.286	119	112
13	2330.50	11.5	11.4	6.05	6.22	44.2	45	1.7	1.75	0.71	0.694	0.328	0.358	77.3	78.9
14	738.83	10.9	11.1	1.41	1.54	15.1	15.7	0.986	1.02	1.36	1.36	0.19	0.198	37.9	35.6

PC-CNF_LD4, w/c=0.325; 0.5 wt% CNF

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	10.5	10.4	0.874	0.897	16.6	16.8	0.0338	0.0414	0.000095	0.000095	0.0866	0.0933	28.9	30.2
2	3.00	9.7	10.1	0.288	0.307	5.23	5.38	0.0677	0.0783	0.0626	0.0797	0.0447	0.0557	18.8	18.9
3	18.57	10.6	10.5	0.806	0.841	14.9	15.8	0.348	0.366	0.764	0.776	0.219	0.18	41.9	41.9
4	26.75	10.0	10.0	0.675	0.699	12.7	13.1	0.498	0.502	1.65	1.72	0.149	0.131	39.4	40.1
5	70.50	10.2	10.2	0.991	1.03	19.3	19.5	0.648	0.645	2.72	2.71	0.175	0.199	48.7	48
6	238.00	11.4	11.3	1.92	1.97	37.9	38.8	1.06	1.05	3.89	3.81	0.242	0.297	68.8	67.8
7	313.50	11.3	11.4	1.55	1.62	30.3	31.5	1.03	1.07	4.53	4.47	0.192	0.203	60.4	62.9
8	519.00	11.0	11.1	1.9	1.99	36.7	38.2	1.18	1.23	4.6	4.58	0.189	0.192	59.2	60.9
9	1019.50	11.0	11.1	2.38	2.43	44.6	46.1	1.38	1.41	4.17	3.74	0.187	0.186	58	62.3
10	1464.00	11.5	11.5	2.75	2.78	50.1	51.3	1.45	1.53	4.41	3.93	0.12	0.144	43.1	49.4
11	1848.00	11.2	11.3	2.75	2.77	49.9	50.5	1.45	1.53	4.37	3.93	0.127	0.132	43.4	48
12	2040.00	11.3	11.4	1.91	1.92	33.4	33.8	1.45	1.47	4.06	3.76	0.144	0.181	51	58.8
13	1680.33	11.2	11.3	1.43	1.41	23.5	23.1	1.51	1.16	3.99	3.79	0.183	0.179	39.6	39.1
14	1009.17	11.2	11.2	0.75	0.774	12.3	12.4	0.945	0.92	4.38	4.41	0.232	0.227	37.4	38.4
15	693.50	11.4	11.4	0.697	0.7	11	11	1.14	1.13	4.26	4.14	0.176	0.188	32.5	31.9
16	1323.75	11.1	11.3	0.692	0.7	11	10.8	1.11	1.1	4.17	4.18	0.159	0.162	32.6	33.7
17	1796.83	11.1	11.0	0.63	0.64	10.6	10.6	1.04	1.01	4.27	4.23	0.119	0.11	27.2	26
18	2238.92	11.1	11.0	0.56	0.57	9	9.6	0.993	1.04	4.21	3.99	0.0835	0.09	23.8	24.9
19	738.50	10.8	10.9	0.34	0.346	5.32	5.43	0.691	0.722	4.34	4.24	0.0404	0.0466	18.4	19.4

PC-CNF_LD5; w/c=0.435; 2 wt% CNF

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	11.3	11.3	2.34	2.48	28.9	29.5	0.0485	0.0501	0.18	0.172	0.178	0.184	45.9	48
2	3.00	11.0	11.1	0.775	0.812	9.37	9.51	0.138	0.136	0.176	0.164	0.111	0.121	28	29.4
3	3.00	10.9	11.0	0.575	0.597	7	7.27	0.135	0.13	0.2	0.204	0.0778	0.0824	20.8	21.7
4	16.00	11.4	11.4	1.93	2.02	22.7	23.4	0.306	0.298	0.501	0.492	0.388	0.377	58	60.7
5	24.32	11.4	11.4	1.77	1.83	21.1	21.6	0.395	0.409	0.883	0.992	0.384	0.374	63.4	65.2
6	48.00	11.5	11.5	2.32	2.41	28.7	29.1	0.565	0.577	1.31	1.29	0.365	0.369	75	77.4
7	96.00	11.4	11.5	3.16	3.24	39.1	39.8	0.816	0.828	1.49	1.47	0.339	0.346	90.3	92.2
8	172.17	11.7	11.7	3.9	4.2	48.2	48.5	0.987	0.992	1.61	1.61	0.377	0.381	102	102
9	335.83	11.9	11.9	4.8	5.36	61.5	66.1	1.27	1.3	1.3	1.29	0.394	0.418	108	109
10	721.12	11.5	11.5	6.53	6.86	82.1	86.9	1.71	1.77	0.967	0.941	0.335	0.336	121	118
11	815.88	11.3	11.0	5.24	5.49	37	38.9	1.56	1.58	1.16	1.17	0.27	0.256	104	99.6
12	1705.50	11.4	11.4	7.9	8.26	53	55.3	2.07	2.11	0.983	1.02	0.326	0.293	120	111
13	2330.50	11.4	11.4	7.92	8.25	42	43.3	1.79	1.76	0.819	0.853	0.318	0.299	73.4	70.6
14	738.83	11.2	11.2	1.58	1.71	12.9	13.8	1.07	1.11	1.53	1.49	0.179	0.2	49.9	51.1

PC-CF_LD4; w/c=0.325; 0.5 wt% CF

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	10.0	10.1	0.44	0.50	8.32	9.81	0.02	0.02	0.00	0.00	0.04	0.02	14.10	17.20
2	3.00	9.7	9.7	0.29	0.27	4.54	4.86	0.09	0.09	0.09	0.08	0.09	0.07	22.20	21.00
3	18.57	10.1	10.5	0.75	0.77	13.20	14.10	0.31	0.33	0.74	0.82	0.33	0.26	53.20	51.40
4	26.75	10.1	10.1	0.63	0.65	11.40	12.20	0.44	0.45	1.56	1.45	0.22	0.21	50.50	50.60
5	70.50	10.3	10.3	0.95	1.01	17.50	18.40	0.65	0.63	2.80	2.58	0.24	0.25	63.40	63.60
6	238.00	11.4	11.4	1.84	1.96	33.50	35.90	1.01	1.04	3.60	3.42	0.28	0.28	85.60	89.30
7	313.50	11.4	11.4	1.52	1.60	27.20	29.30	1.00	1.04	4.25	3.97	0.24	0.24	73.30	77.20
8	519.00	11.0	10.9	1.88	2.00	33.60	35.90	1.18	1.18	4.22	4.22	0.26	0.26	71.40	71.20
9	1019.50	11.1	11.1	2.38	2.50	42.10	44.60	1.40	1.41	3.56	4.63	0.22	0.21	70.40	70.60
10	1464.00	11.5	11.5	2.77	2.94	47.70	51.70	1.49	1.57	3.94	3.91	0.15	0.15	49.00	56.40
11	1848.00	11.1	11.2	2.77	2.93	47.80	51.50	1.48	1.56	3.98	3.99	0.17	0.15	48.60	55.90
12	2040.00	11.3	11.3	1.92	2.01	31.70	33.40	1.51	1.52	3.32	3.46	0.21	0.20	68.60	68.00
13	1680.33	11.2	11.4	1.44	1.44	23.80	23.40	1.19	1.23	4.32	3.68	0.21	0.21	41.50	42.30
14	1009.17	11.2	11.2	0.75	0.78	12.40	12.60	0.98	0.96	4.32	4.40	0.26	0.27	37.70	38.80
15	693.50	11.4	11.4	0.72	0.73	11.40	11.50	1.12	1.12	4.12	3.94	0.18	0.20	35.00	35.30
16	1323.75	11.2	11.2	0.71	0.70	11.10	11.30	1.10	1.02	4.12	3.17	0.17	0.18	32.80	33.40
17	1796.83	11.2	11.2	0.65	0.63	10.10	10.50	1.09	1.14	4.32	4.30	0.16	0.14	30.70	30.60
18	2238.92	11.3	11.3	0.56	0.54	10.70	10.80	1.08	1.00	3.91	3.48	0.09	0.10	24.80	25.90
19	738.50	10.8	10.9	0.44	0.46	6.00	6.29	0.82	0.86	4.21	3.86	0.07	0.06	21.30	22.20

SF-P; w/c=0.365

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	10.7	10.6	0.0211	0.0231	0.252	0.229	0.0494	0.0484	0.000095	0.000095	0.0469	0.0384	19.5	16.9
2	3.00	10.3	10.4	0.0217	0.0221	0.103	0.0901	0.0542	0.0578	0.0918	0.0833	0.0263	0.0235	12.3	12.3
3	3.00	10.3	10.2	0.0246	0.0235	0.0797	0.0679	0.0514	0.0503	0.148	0.138	0.0173	0.0149	9.7	9.31
4	16.00	10.9	10.9	0.134	0.1519	0.563	0.546	0.224	0.213	1.69	1.62	0.112	0.0833	31.9	31.7
5	24.32	11.0	11.0	0.14	0.1595	0.77	0.712	0.256	0.259	2.45	2.51	0.0947	0.0802	33	33.1
6	48.00	11.1	11.1	0.192	0.2	1.63	1.57	0.373	0.393	3.76	3.89	0.112	0.0978	40.3	41.1
7	96.00	11.2	11.2	0.329	0.341	2.8	2.93	0.54	0.57	5.23	5.13	0.134	0.128	49.1	49.7
8	181.50	11.9	11.9	0.446	0.473	3.81	4.08	0.699	0.734	6.01	5.84	0.151	0.149	55.9	57.4
9	361.50	10.8	10.9	0.503	0.535	4.42	4.72	0.81	0.829	6.63	6.48	0.16	0.154	59.3	60.6
10	1019.50	10.8	10.9	0.786	0.79	6.2	8.24	0.928	0.943	6.28	6.14	0.157	0.162	63	62.6
11	1464.00	11.3	11.3	0.608	0.654	5.83	6.23	0.779	0.775	7.19	7.17	0.133	0.121	51.5	49.1
12	1848.00	11.1	11.1	0.616	0.646	5.83	6.2	0.786	0.773	7.23	7.08	0.122	0.116	51.8	49.8
13	2040.00	10.9	10.9	0.59	0.598	4.05	4.17	0.728	0.664	8.07	8.31	0.115	0.0923	47.6	42.8
14	1680.33	11.1	11.1	0.36	0.399	3.62	3.96	0.462	0.464	7.1	7.33	0.13	0.136	39.9	41.3
15	1009.17	11.1	11.1	0.217	0.256	2.1	2.32	0.547	0.53	6.82	6.9	0.247	0.234	35.6	34.7
16	693.50	11.2	11.3	0.205	0.223	2.1	2.24	0.509	0.495	6.61	6.67	0.159	0.168	33.2	34.3
17	1323.75	11.0	11.1	0.198	0.188	2.29	2.21	0.486	0.491	6.49	6.9	0.124	0.119	32.4	31.6
18	1796.83	10.8	10.8	0.202	0.211	2.3	2.26	0.503	0.498	6.21	6.57	0.0911	0.0913	24.1	24.5
19	2238.92	10.8	10.9	0.206	0.193	2.54	2.62	0.496	0.516	6.21	6.03	0.0729	0.0793	22.5	23.8
20	738.50	10.6	10.6	0.166	0.171	1.66	1.8	0.434	0.43	6.22	6.15	0.0529	0.0426	17.9	17.1

SF-P2; w/c=0.45

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	11.2	11.2	0.0307	0.0282	9.66E-05	9.66E-05	0.0417	0.0409	0.054	0.0584	0.146	0.135	40.6	40
2	3.00	10.9	10.9	0.0217	0.0189	9.66E-05	9.66E-05	0.0832	0.0813	0.16	0.1714	0.0779	0.076	19.3	19.1
3	3.00	10.8	10.8	0.0077	0.00817	9.66E-05	9.66E-05	0.0949	0.0936	0.256	0.263	0.0615	0.0618	15.8	15.9
4	16.00	11.2	11.2	0.0316	0.0311	0.0674	0.0569	0.252	0.273	0.764	0.792	0.143	0.14	38.9	40.5
5	24.32	11.3	11.3	0.0624	0.0614	0.254	0.233	0.381	0.378	1.59	1.61	0.213	0.206	46.9	46.7
6	48.00	11.3	11.4	0.125	0.117	0.674	0.63	0.502	0.518	1.89	1.91	0.246	0.23	52.7	52.1
7	96.00	11.3	11.3	0.236	0.248	1.52	1.4	0.777	0.762	2.61	2.66	0.25	0.262	67.9	71
8	172.17	11.5	11.5	0.362	0.38	2.44	2.31	0.916	0.927	2.5	2.53	0.272	0.285	75.1	76.7
9	335.83	11.7	11.7	0.588	0.583	4.05	3.84	1.25	1.18	2.52	2.29	0.244	0.314	86.4	86.5
10	721.12	11.4	11.4	0.816	0.894	5.67	5.63	1.39	1.38	2.32	2.33	0.266	0.244	93.3	88.6
11	815.88	11.3	11.4	0.732	0.755	5.11	5.14	1.16	1.16	2.83	2.74	0.171	0.174	71.4	72.1
12	1705.50	11.1	11.1	1.09	1.16	7.65	8.1	1.44	1.37	2.56	2.91	0.231	0.203	86.9	75.3
13	2330.50	11.1	11.0	0.99	1.03	7.29	7.54	0.982	0.993	2.44	2.54	0.162	0.162	40.1	39.7
14	738.83	11.1	11.0	0.295	0.29	2.5	2.35	0.827	0.775	2.98	3.13	0.147	0.131	32.9	30.6

SF-CNF_LD4; w/c=0.365; 0.5 wt% CNF

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	10.7	10.7	0.0699	0.0612	0.797	0.785	0.058	0.0632	0.000095	0.000095	0.0854	0.0826	17.6	17.3
2	3.00	10.4	10.4	0.0515	0.0445	0.109	0.0935	0.08	0.0713	0.0811	0.0746	0.048	0.0453	9.91	9.21
3	3.00	10.3	10.3	0.0282	0.0284	0.0426	0.0371	0.0953	0.0723	0.172	0.163	0.0533	0.0518	7.63	7.18
4	16.00	10.9	10.8	0.0929	0.0845	0.599	0.53	0.243	0.231	1.59	1.57	0.185	0.109	24.8	24.2
5	24.32	10.9	10.9	0.13	0.117	1.09	1.04	0.278	0.257	2.8	2.87	0.12	0.104	26.3	25.2
6	48.00	11.0	11.0	0.264	0.241	2.5	2.23	0.415	0.391	4.31	4.23	0.152	0.141	33.7	33.9
7	96.00	11.1	11.1	0.394	0.376	3.92	3.38	0.569	0.553	5.46	5.42	0.17	0.158	39.9	41.1
8	181.50	11.9	11.8	0.476	0.466	4.55	4.45	0.712	0.684	6.2	6.33	0.18	0.185	45.4	46.7
9	361.50	10.8	10.9	0.501	0.494	4.75	4.68	0.791	0.781	6.72	6.94	0.195	0.244	49.4	51.1
10	1019.50	10.9	10.9	0.652	0.656	6.36	6.32	0.837	0.815	6.9	7.05	0.205	0.203	51.9	51.3
11	1464.00	11.4	11.3	0.636	0.634	5.99	5.96	0.743	0.725	7.59	7.65	0.184	0.186	45.7	46.1
12	1848.00	11.1	11.1	0.628	0.625	5.99	6.01	0.748	0.73	7.52	7.67	0.192	0.182	46.1	46.3
13	2040.00	11.0	11.1	0.516	0.498	4.92	4.48	0.695	0.637	8.39	8.71	0.147	0.141	37.4	37.3
14	1680.33	11.1	11.1	0.367	0.361	3.99	3.82	0.448	0.441	7.04	7.08	0.122	0.118	29.4	29.6
15	1009.17	11.1	11.1	0.239	0.236	2.32	2.3	0.491	0.482	6.7	6.78	0.246	0.233	30.6	31.3
16	693.50	22.2	11.3	0.24	0.236	2.3	2.27	0.475	0.461	6.81	6.89	0.207	0.197	28.6	27.3
17	1323.75	10.7	10.7	0.202	0.16	2.21	2.13	0.41	0.395	6.73	6.74	0.106	0.113	26.2	24.4
18	1796.83	10.6	10.7	0.202	0.197	2.1	2.04	0.387	0.376	6.73	6.72	0.0753	0.0721	22.2	21
19	2238.92	10.5	10.5	0.22	0.216	2.68	2.74	0.347	0.372	6.94	6.9	0.0539	0.0596	19.2	18.4
20	738.50	10.4	10.6	0.162	0.148	1.7	1.52	0.314	0.344	6.79	6.6	0.0288	0.0228	13.8	15

SF-CNF_LD5; w/c=0.45; 2 wt% CNF

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	11.0	11.0	0.109	0.117	0.724	0.754	0.0933	0.0945	9.30E-05	9.30E-05	0.0988	0.094	24.7	23.6
2	3.00	10.7	10.7	0.0436	0.038	0.116	0.127	0.0977	0.0994	9.30E-05	9.30E-05	0.0456	0.0482	12.3	12.2
3	3.00	10.6	10.6	0.0262	0.0214	0.03	0.0265	0.118	0.125	0.0624	0.0579	0.0411	0.045	10.2	9.94
4	16.00	11.1	11.0	0.08	0.0733	0.482	0.451	0.298	0.294	0.739	0.716	0.0993	0.0909	27.2	27
5	24.32	11.1	11.0	0.0974	0.0904	0.47	0.44	0.373	0.36	1.03	1.01	0.118	0.125	27.9	27.7
6	48.00	11.2	11.2	0.181	0.165	1.15	1.094	0.47	0.46	2.19	2.07	0.162	0.159	41.5	40.8
7	96.00	11.2	11.2	0.318	0.309	2.26	2.1	0.604	0.612	3.11	3.14	0.188	0.198	50.6	50.9
8	172.17	11.4	11.4	0.429	0.446	3.09	3.13	0.695	0.699	3.51	3.49	0.195	0.201	54.3	54.5
9	335.83	11.6	11.6	0.663	0.665	5.67	5.63	0.867	0.856	3.95	3.89	0.198	0.179	63.9	63.8
10	721.12	11.3	11.3	0.798	0.779	5.55	5.6	0.926	0.954	3.91	3.74	0.204	0.18	62.7	68.5
11	815.88	11.3	11.3	0.67	0.647	4.55	4.63	0.842	0.866	4.07	3.7	0.121	0.133	57.5	59.3
12	1705.50	10.8	10.8	0.94	0.927	6.78	6.64	0.937	0.999	4.34	3.87	0.152	0.168	59.2	68.7
13	2330.50	11.0	11.0	0.88	0.94	6.04	6.06	0.757	0.758	3.79	3.65	0.127	0.134	37	36.9
14	738.83	11.0	11.0	0.278	0.271	2.09	2.02	1.07	1.11	4.52	4.33	0.0979	0.111	30.7	32.5

SF-CF_LD4; w/c=0.365; 0.5 wt% CF

				Concentration (mg/L)											
		pH		Sodium		Potassium		Aluminum		Silicon		Iron		Calcium	
Extract #	Duration (hrs)	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	2.18	10.7	10.7	0.0279	0.0263	0.375	0.317	0.0555	0.0635	0.000095	0.000095	0.0502	0.0308	17.4	18.8
2	3.00	10.4	10.5	0.0266	0.0264	0.0889	0.0797	0.0601	0.0672	0.152	0.148	0.0229	0.0259	10.8	11.4
3	3.00	10.2	10.3	0.0209	0.0254	0.05462	0.05605	0.0538	0.061	0.161	0.153	0.015	0.0178	8.1	8.65
4	16.00	10.9	10.9	0.0857	0.0763	0.421	0.401	0.222	0.247	1.62	1.59	0.0666	0.0583	27.5	28.5
5	24.32	10.9	10.0	0.0937	0.0838	0.611	0.585	0.265	0.275	2.73	2.77	0.11	0.0935	28.9	29.5
6	48.00	11.1	11.1	0.184	0.182	1.46	1.46	0.423	0.41	4.29	4.24	0.123	0.1134	37.1	37.5
7	96.00	11.2	11.1	0.318	0.333	2.81	3.08	0.575	0.603	5.25	5.56	0.121	0.118	44.2	46.2
8	181.50	11.9	11.9	0.435	0.453	3.85	4.15	0.728	0.749	5.96	6.21	0.148	0.141	50.8	52.7
9	361.50	10.8	10.8	0.467	0.5	4.29	4.67	0.807	0.832	6.46	6.82	0.149	0.148	53.9	56.3
10	1019.50	10.9	10.9	0.627	0.639	5.89	6.3	0.879	0.904	6.41	6.71	0.177	0.16	56.4	56
11	1464.00	11.3	11.3	0.726	0.746	5.49	5.91	0.739	0.746	7.37	7.9	0.128	0.127	46.6	44.5
12	1848.00	11.2	11.1	0.726	0.741	5.49	5.99	0.738	0.763	7.38	7.87	0.119	0.114	47	45.4
13	2040.00	10.9	11.0	0.414	0.435	3.82	4.19	0.6	0.683	8.76	8.93	0.0764	0.064	36.6	43.6
14	1680.33	11.0	11.0	0.334	0.373	3.47	3.67	0.432	0.445	7.36	7.68	0.123	0.113	30.9	30.7
15	1009.17	11.1	11.1	0.223	0.258	2.07	2.27	0.486	0.483	7.24	7.27	0.211	0.215	32.4	33.2
16	693.50	11.1	11.3	0.195	0.191	2.04	2.08	0.468	0.449	7.19	6.93	0.14	0.146	31.9	30.7
17	1323.75	11.1	11.1	0.188	0.185	2	1.95	0.484	0.476	6.79	6.7	0.114	0.107	29.6	29.1
18	1796.83	10.6	10.6	0.186	0.187	2.05	2.08	0.475	0.462	6.79	6.83	0.0798	0.0822	22.2	23.8
19	2238.92	10.8	10.7	0.177	0.16	2.48	2.3	0.478	0.49	6.81	6.74	0.0678	0.0795	22.2	23.7
20	738.50	10.5	10.7	0.158	0.186	1.72	1.74	0.328	0.33	6.59	6.31	0.0355	0.0432	16.2	18.4

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