Effect of Synthetic Fiber Surface Treatment on the Post-Crack Residual Strength and Toughness of Fiber Reinforced Concrete

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A Thesis

In

The Department

Of

Building, Civil, and Environmental Engineering

Presented in Partial Fulfillment of the Requirements

For the Degree of Master of Applied Science (Building Engineering) at

Concordia University

Montreal, Quebec, Canada

April 2008

Your file Votre référence
ISBN: 978-0-494-40869-8
Our file Notre référence
ISBN: 978-0-494-40869-8

Abstract

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This research involves an experimental investigation into the improvement of bonding characteristics between a mixture of polyethylene/polypropylene fibers and concrete comparing various chemical and physical surface treatments and their effect on the mechanical properties of the fiber reinforced concrete (FRC). For the chemical surface treatment, several techniques of chemical etching of the fiber's surface were used, such as two types of chromic acid solutions, potassium permanganate, and hydrogen peroxide solutions. For the physical surface treatment, UV treatment and a combination of UV and Ozone treatment were used. Non-treated and treated fibers were added at 0.32% by volume and 0.50% for the best treatment method. Compressive, flexural strength and contact angle were measured to quantify bond improvement. Among the chemical treatment techniques, chromic acid solution type B was found to be the most efficient technique versus potassium permanganate which had negative effect on the bonding strength between fibers and concrete. Investigations of physical treatment techniques showed using UV does not have a significant change on the bonding strength, but 10 minute exposure of fibers to the UV lamp in presence of ozone gave the best result in bonding of fibers. As a cumulative result, using the chemical treatment was found to be a more efficient technique rather than the physical treatment in surface modification of fibers. The contact angle was found to have no correlation to the toughness. The higher

volume of fibers gave better properties than the surface treatment techniques indicating surface treatment may not be an economical alternative.

ACKNOWLEDGMENTS

I would like to express my sincere thanks to *Dr. Dorel Feldman* for his supervision, critical review of my work, and financial support during course of this research. His assistance and valuable guidance are highly appreciated.

Special acknowledgment is due to my co-supervisor *Dr. Michelle Nokken* for her supervision, valuable help at various occasions during the course of this research, and support especially at the moments that I really needed throughout the work.

Thanks are due to *Mrs. Dorina Banu* for her encouragement and valuable help in laboratory and chemical treatment of the fibers.

Special appreciation is due to *Dr. Cameron Skinner* and *Dr. Christine E. DeWolf* for their valued recommendations on physical treatment procedure of fibers, and giving permission to use their facilities, and *Dr. Rolf Schmidt* for his valued suggestions, training to use the facilities, and helping me throughout the whole physical treatment procedures.

I am also thankful to the technical staff of the Centre for Building Studies, namely, *Mr. J. Payer*, *Mr. J. Hrib*, and *D. Roy* for their help in the fabrication of experimental set-up and the running of experiments.

Lasts but not least, I expresses my sincere gratitude to my parents for their support, encouragement, and patience not only during this work, but also throughout my whole life.

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List of Terms

aspect ratio—The ratio of length to diameter of the fiber.

balling—When fibers entangle into large clumps or balls in a mixture.

end-point deflection—The deflection value on the load-deflection curve equal to a specified proportion of span.

first crack—The point on the flexural load-deflection or tensile load-extension curve at which the form of the curve first becomes nonlinear.

first-peak load, P_1—The load value at the first point on the load-deflection curve where the slope is zero.

first-peak deflection, δ_1—The net deflection value on the load-deflection curve at first-peak load.

first-peak strength, f_1—The stress value obtained when the first-peak load is inserted in the formula for modulus of rupture.

FRC—Fiber reinforced concrete

load-deflection curve—The plot of load versus net deflection of a flexural beam specimen loaded to the end-point deflection.

monofilament—Single filament fiber typically cylindrical in cross-section.

net deflection—The deflection measured at mid-span of a flexural beam specimen exclusive of any extraneous effects due to seating or twisting the specimen on its supports or deformation of the support and loading system.

peak-lead, P_P—The maximum load on the load-deflection curve.

peak-load deflection, δ_p—The net deflection value on the load-deflection curve at peak load.

peak strength, f_P—The stress value obtained when the peak load is inserted in the formula for modulus of rupture.

residual load, $P_{75,0.75}$—The load value corresponding to a net deflection equal to 1/300 of the span using a specimen with depth of 76 mm.

residual load, $P_{75,1.5}$—The load value corresponding to a net deflection equal to 1/150 of the span using a specimen with depth of 76 mm.

residual load, $P_{75,3.0}$—The load value corresponding to a net deflection equal to 1/75 of the span using a specimen with depth of 76 mm.

residual strength, $f_{75,0.75}$—The stress value obtained when the residual load $P_{75,0.75}$ is inserted in the formula for modulus of rupture.

residual strength, $f_{75,1.5}$—The stress value obtained when the residual load $P_{75,1.5}$ is inserted in the formula for modulus of rupture.

residual strength, $f_{75,3.0}$—The stress value obtained when the residual load $P_{75,3.0}$ is inserted in the formula for modulus of rupture.

SFRC—Synthetic fiber reinforced concrete.

specimen toughness, $T_{75,3.0}$—The energy equivalent to the area under the load-deflection curve up to a net deflection of 1/75 of the span using a specimen with a depth of 76 mm.

List of Symbols

A	=	area of the cylindrical in compressive test
b	=	average width of rectangular specimens at the fracture.
d	=	average depth of rectangular specimen at the fracture.
f	=	flexural Strength
$f_{75,0.75}$	=	residual strength at span / 75
$f_{75,1.5}$	=	residual strength at span / 150
$f_{75,3.0}$	=	residual strength at span / 300
f_1	=	first-peak strength
f_P	=	peak strength
L	=	span length in flexural test
P	=	load
$P_{75,0.75}$	=	residual load at span / 75
$P_{75,1.5}$	=	residual load at span / 150
$P_{75,3.0}$	=	residual load at span / 300
P_1	=	first-peak load
P_P	=	peak load
s	=	standard deviation
$T_{75,3.0}$	=	area under load-deflection curve 0 to Span / 75
$\overline{x}$	=	mean of samples
α	=	gradient of decreasing residual strength
Θ_c	=	contact angle
δ_1	=	net deflection at first-peak Loads

δ_P = net deflection at peak and first peak load

σ = compressive strength

Chapter 1

Introduction

1.1 Background

Reinforcing brittle materials to control the post crack strength by adding a high tensile strength material as an alternative in construction has been considered since ancient times. One of the solutions to control the post crack behavior of brittle materials, such as concrete or masonry bricks, is the introduction of short fibers. Early examples of fiber reinforced materials are mud huts using baked clay reinforced with straw, and masonry mortar reinforced using animal hair. Currently, various fiber types are available for commercial usage including steel, glass, synthetic and natural fibers.

There has been interest in using fiber reinforced concrete (FRC) in the construction industry starting from the early studies of Romualdi and Batson (1963). Initially, the investigations were focused on the study of material properties and were followed by a number of applications. By innovation in the enhancement of materials, several structural applications were proposed, such as Heathrow Airport car park in London (ACI 544.4R, 1988), and the foundation slab of Potsdamer Plazt in Berlin (Falkner et al., 1997). The most recent applications are mostly considered in roads and floors concrete pavements, in the precast industry and for tunnel lining. Using fibers in floors, even in low volume fractions (<1 %), can increase the ultimate load and can be used as a replacement for conventional reinforcement. (Cominoli et al., 2006).

Fiber reinforced concrete (FRC) is concrete primarily consisting of hydraulic cements, aggregates, and discrete reinforcing fibers (ACI 544.1R, 1996). Fibers are long slender needlelike particles that are added to cement paste, mortar or concrete matrices. Fibers suitable for reinforcing concrete have been produced from steel, glass, and organic polymers (synthetic fibers). Some of them are relatively rigid like steel while others are quite flexible like glass or polypropylene, depending on the form and type (Naaman et al., 1982). The concrete matrices may be mortars, normally proportioned concrete mixtures, or mixes specifically formulated for a particular application. Generally, the length and diameter of the fibers used for FRC do not exceed 76 mm (3 in.) and 1 mm (0.04 in.), respectively (ACI 544.1R, 1996).

Brittle materials are considered to have no significant post-cracking ductility. Fibrous composites have been and are being developed to provide improved mechanical properties to otherwise brittle materials. When subjected to tension, these un-reinforced brittle matrices deform elastically. The elastic response is followed by micro-cracking, localized macro-cracking, and finally by fracture at relatively low strains. Introduction of fiber into concrete results in post-elastic property changes that range from subtle to substantial, depending upon a number of factors, including matrix strength, fiber type, fiber modulus, fiber aspect ratio, fiber strength, fiber surface bonding characteristics, fiber content, fiber orientation, and aggregate size effects (Johnston, 2001). For many practical applications, the matrix first-crack strength is not increased. In these cases, the most significant enhancement from the fibers is the post-cracking composite response. This is most commonly evaluated and controlled through toughness testing (such as measurement of the area under the load-deformation curve).

If properly engineered, one of the greatest benefits to be gained by using fiber reinforcement is improved long-term serviceability of the structure or product. Serviceability is the ability of the specific structure or part to maintain its strength and integrity and to provide its designed function over its intended service life. One aspect of serviceability that can be enhanced by the use of fibers is the control of cracking. Fibers can prevent the occurrence of large crack widths that are either unsightly or permit water and contaminants to enter, causing corrosion of reinforcing steel or potential deterioration of concrete (Shah, 1991). In addition to crack control and serviceability benefits, use of fibers at high volume percentage (5 to 10 percent or higher with special production techniques) can substantially increase the matrix tensile strength (Shah, 1991).

There are many different types of fiber shapes and materials in the building industry. Steel fibers are available in various types and since they are one the first modern fiber materials in the building industry, there are many resources and standards available regarding these kinds of fibers. On the other hand, synthetic fibers are relatively new and there is little reported research or field experiences on the some types of them. In most cases, steel fibers have higher strength than synthetic fibers but their low corrosion and alkali resistance, and most importantly the higher cost of production over synthetic fibers have to be considered.

Among of the synthetic fibers, polypropylene and polyethylene have high alkali resistance and relatively high melting point, but poor fire resistance as well as sensitivity to sunlight and oxygen. However, most significant disadvantage is the poor bond between inorganic matrix (concrete) and the organic fiber, which is the focus of this work.

1.2 Objective and Scope

The main objective of the present research is to experimentally investigate the mechanical behavior of FRC using polypropylene/polyethylene blend fiber with various surface treatments.

The specific objectives of the research are:

1. To examine the effects of different chemical and physical treatment techniques of polypropylene/polyethylene blend fibers on the flexural strength and toughness of FRC.
2. To investigate the effects of different chemical treatment techniques of polypropylene/polyethylene blend fiber on compressive strength of FRC.
3. To study the contact angle of the fibers treated with all different mentioned treatment techniques and to compare it with the flexural strength of FRC of each surface treatment.
4. To investigate the improvement of mechanical properties of the best treatment technique at a higher volume of fibers.

The experimental program consists of testing five specimens for each mixture for compressive strength for seven and twenty eight days. The flexural strength was tested for each mixture; a total of five beams were tested at twenty eight days.

Several techniques of chemical by etching functional groups of polyethylene/polypropylene fibers surface have been used in an attempt to improve the bonding of fibers and concrete. The power of the etchant and the specific changes to the surface introduced by etching determines the degree of adhesion enhancement (Silverstein and Breuer, 1993, Silverstein et al., 1994). Two types of chromic acid

solutions (solution B and solution C) as well as solutions of potassium permanganate and hydrogen peroxide were used for chemical etching/oxidation.

For the physical surface treatment two techniques of UV and a combination of UV and Ozone were used. In both cases the fibers were exposed to UV lamp alone and a UV and Ozone generator for several time periods. Single fibers which seemed to have less production surface defects were collected and exposed to the UV to measure the contact angle.

These measurements were not only used to compare the results of contact angle with the actual samples tested, but also to determine the best duration of exposure to the UV lamps in the physical treatment techniques.

1.3 Outline of Thesis

In the following chapter, based on technical literatures the use of fibers in concrete (FRC) and its benefits and effects on the compressive and flexural strength of concrete is discussed. A brief description of various fiber materials and their properties is presented. Previous research work carried out to improve the bonding between fibers and concrete, different chemical and physical surface treatment techniques, and the techniques to determine the efficiency of these techniques is reviewed.

A detailed description of the experimental program and the test methods is given in chapter 3. The properties of concrete, the fibers used as reinforcement and the flexural and compressive test methods are presented. The different chemical and physical surface treatment techniques of fibers and the technique to determine their effectiveness is discussed.

The results of this experimental program are presented and discussed in chapter 4. All flexural and compressive strength results coming from the different groups based on the treatment techniques, is discussed. Flexural strength versus displacement, and all evaluation parameters suggested by ASTM C 1609 such as test span, peak load, first-peak load, first-peak strength, and net deflection at peak load and first-peak load are presented. The final results of each treatment technique and their effects in bonding enhancement between the fibers and concrete is compared.

The final chapter includes a summary of conclusions, and also recommendations for further research in the area of bonding enhancement between fibers and concrete.

Chapter 2

Literature Review

2.1 Fiber-Reinforced Concrete

Using steel reinforcing bars or prestressing traditionally has been used to overcome the low tensile strength and low strain capacity at fracture of unreinforced concrete. Fibers of various types can also be used to improve the mechanical properties of concrete. Unlike continuous reinforcing steel which is located at specific locations in the structure to optimize performance, fibers are discontinuous and are generally distributed randomly throughout the concrete matrix.

The physical properties of fiber reinforced concrete (FRC) are highly affected by the fiber properties, volume fraction of fibers, type and orientation of fibers, and the bonding between dispersed fibers and the matrix. The general performance of FRC compared to typical plain concrete under bending is presented in Figure 2.1. It clearly can be seen that plain concrete does not have any post crack flexural strength due to the nature of the brittle materials versus FRC which exhibits resistance under bending load after the first crack occurs.

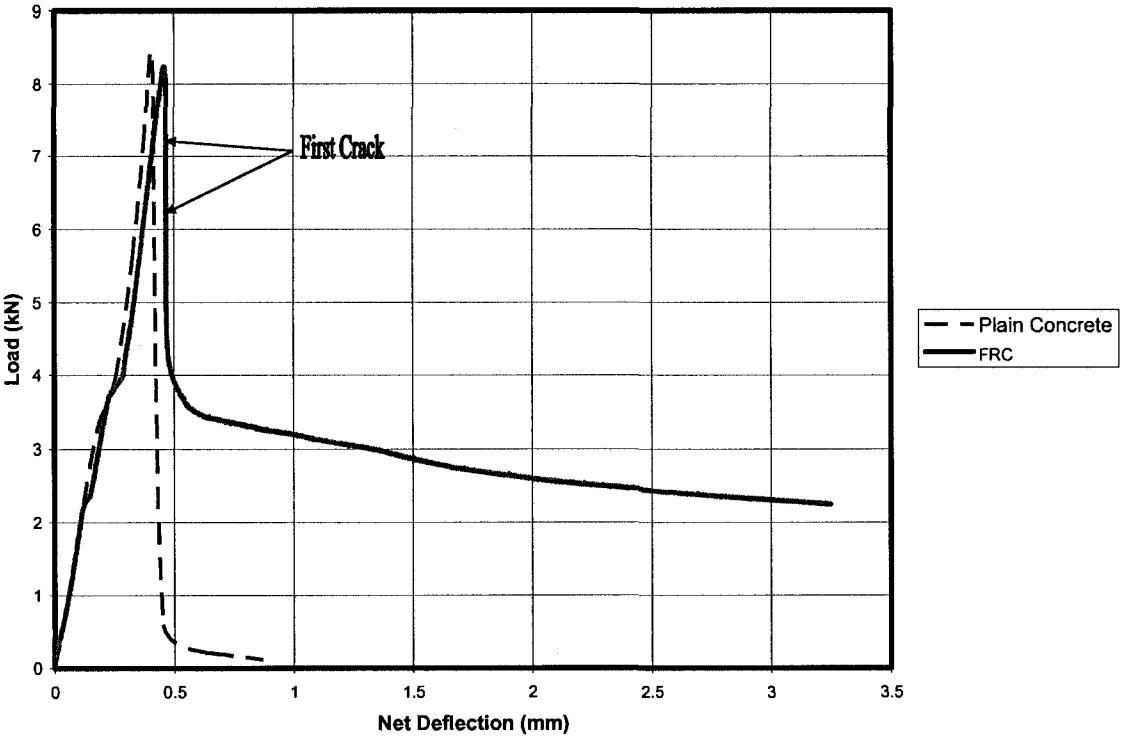


Figure 2.1—Typical Load-Deflection Curves for Matrix and Fiber Reinforced Concrete (FRC) in Bending.

The main factors governing the post crack performance of the FRC under bending are the physical properties of fiber and matrix as well as the strength of bond between the two. Bonding characteristics of FRC depend on the chemical properties of the fiber material used in concrete. These bonding characteristics will highly influence the post crack and the toughness properties of the final solid product. The higher the bonding strength between the fibers and concrete, the higher the resulting toughness and the higher post crack strength of the FRC. However, although the bonding strength of each fiber material with concrete is finite; it can be improved by surface treatment.

In this chapter, the fibers used in construction and their mechanical and physical properties are briefly reviewed. The effects of adding fibers on fresh properties of

concrete and workability of FRC are discussed. The parameters governing the compressive and flexural strength performance of FRC are analyzed. A brief review of the previous experimental studies on the different surface treatment techniques of fibers and their influence on the strength improvement of FRC is presented. Finally, the relation between the surface contact angle of fibers and the wettability of fibers is discussed.

2.2 Material and Shape of Fibers

The main aspects of fibers which are related to the improvement of flexural strength and toughness of FRC are the composition of the fibers, the shape of fibers, and finally the strength of bonding between the fibers and cement matrix. Fibers are manufactured from many materials such as metal, glass, carbon and graphite, polymer, boron, ceramic, and silicon carbide (Mallick, 1993). In the following section, the effects of fiber compositions and shape with respect to bonding with concrete and the resulting improvement of flexural strength of FRC are discussed.

2.2.1 Steel Fiber Reinforced Concrete (SFRC)

Steel fiber reinforced concrete (SFRC) refers to the combination of discontinuous discrete steel fibers with conventional concrete made of hydraulic cements containing fine or fine and coarse aggregate. The mechanical properties of the SFRC mainly depend on the shape of fibers rather than the material (Johnston, 2001). ASTM A820 (2001) classifies steel fibers based upon the method used in their manufacture, in contrast with the Japanese Society of Civil Engineering (JSCE) (ACI 544.1R, 1996) which considers the shape of fibers' cross-section in their classification.

A significant problem with using SFRC is its sensitivity to corrosion and subsequent loss of strength throughout its life time. Cracks in SFRC have been indicated to cause corrosion of fibers in laboratory and field testing when exposed to chloride environments due to fibers passing across the crack (Hoff, 1987). Appearance of the flexural or tensile cracking on SFRC can lead to catastrophic structural conditions, so that full consideration should be given to the possibility of corrosion at cracks (ACI 544.1R, 1996). Since SFRC is out of the scope of this thesis, it will not be addressed further.

2.2.2 Synthetic Fiber Reinforced Concrete (SNFRC)

Synthetic fibers are man-made fibers resulting from research and development in the petrochemical and textile industries. Synthetic fiber reinforced materials (SNFRC) utilize fibers derived from organic polymers which are available in a variety of formulations. Aramid (aromatic polyamide), a high-modulus polymeric material, was one of the first synthetic fibers used in the construction industry introduced for commercial application by late 1970s (Walton and Majumdar, 1978). The use of aramid fibers in Portland cement concrete based matrix was followed by acrylic, carbon, nylon, polyester, polyethylene, and polypropylene. For many of these fibers, there is little reported research or field experience, while others are found in commercial applications and have been the subject of extensive reporting (Bentur and Mindess, 2007). Table 2.1 summarizes the range of physical properties of selected synthetic fibers types (Cement & Concrete Institute, 1997), (ACI 544.1R, 1996).

Table 2.1—Selected Synthetic Fiber Types and Properties (Cement & Concrete Institute, 1997; ACI 544.1R, 1996).

Fiber Type[1]	Equivalent Diameter, mm × 10^{-3}	Specific Gravity	Tensile Strength, MPa	Elastic Modulus, GPa	Ultimate Elongation, Percent
Acrylic	13- 104	1.16-1.18	269- 1000	13.8-19.3	7.5-50.0
Aramid I	12	1.44	2930.273	62.0	4.4
Aramid II[2]	10	1.44	2344.218	117.2	2.5
Carbon, PAN HM[3]	8	1.6-1.7	2482-3034	379.9	0.5-0.7
Carbon, PAN HT[4]	9	1.6-1.7	3447- 3999	230.3	1.0-1.5
Carbon, pitch GP[5]	10- 13	1.6-1.7	4831- 793	27.6- 34.5	2.0-2.4
Carbon, pitch HP[6]	9- 18	1.80-2.15	1517- 3103	151.7- 482.6	0.5-1.1
Nylon[7]	23	1.14	965	5.2	20
Polyester	20	1.34-1.39	228-1103	17.2	12-150
Polyethylene[7]	25- 1016	0.92-0.96	76-586	5.0	3-80
Polypropylene[7]	-----	0.90-0.91	138-689	3.4- 4.8	15

1 Not all fiber types are currently used for commercial production of FRC.
2 High modulus.
3 Polyacrylonitrile based, high modulus.
4 Polyacrylonitrile based, high tensile strength.
5 Isotropic pitch based, general purpose.
6 Mesophase pitch based, high performance.
7 Data listed is only for fibers commercially available for FRC.

While durability in concrete in some respects relates specifically to the chemistry of each fiber type, some general physical considerations can be essential. All these polymers melt at a relatively low temperature between about 134°C for polyethylene and 257°C for polyester (ACI 544.1R, 1996), so they cannot be expected to perform under conditions where the concrete temperature approaches or exceeds these values, as in the case of fire in service.

The advantage of using synthetic fibers over SFRC is their corrosion resistance and according to ASTM C1116 (2002) their compatibility with moisture, cement alkalis, and chemical admixtures. Polypropylene and polyethylene have been reported to be very resistant to strong alkalis, while polyester was not as resistant (Lyle, 1976) (Wang et al., 1987). Since polyethylene and polypropylene are two of most common synthetic fibers used as internal reinforcement in the construction industry and are directly related to this work, the discussion will be limited to these fibers.

2.2.2.1 Polypropylene Fibers

Polypropylene fibers are produced from homopolymer polypropylene resin in a variety of shapes and sizes, and with differing properties. Polypropylene has tended to be the most widely used polymeric form of fiber reinforcement in concrete because of its excellent resistance to moisture, acids and alkalis and the economy of the raw material on a volume basis compared with steel and other alternatives (Krenchel and Jensen, 1980), (Larsen and Krenchel, 1991). Polypropylene fibers are generally used at low volume fractions, about 0.1%, to control plastic shrinkage cracking, and in larger amounts in

fibrillated form up to 0.7% to improve the hardened concrete mechanical properties (Johnston, 2001).

Fibrillated polypropylene fibers (Figure 2.2) are the most widely used in concrete. Their development was a fundamental solution to increase mechanical bonding with the concrete by separation and branching of the fibrils in the polymer strand during the mixing stage. Monofilament form of polypropylene is also available to be used in concrete, in some cases with surface treatment or surface texturing to improve bonding between fibers and concrete resulting in an enhancement in pullout resistance and overall reinforcing effectiveness (Krenchel and Shah, 1985; Portland Cement Association, 1991). Polypropylene fibers are not expected to bond chemically in concrete matrix, due to the nature of polypropylene which is hydrophobic so that there is difficulty of wetting the surface by the cement paste. However, bonding has been shown to occur by mechanical interaction (Rice et al., 1988).

Figure 2.2—Fibrillated form of polypropylene fibers (CNBM Website)

2.2.2.2 Polyethylene Fibers

There has been considerable interest in the use of polyethylene fibers in FRC (Hughes, 1984) (Bijen and Geurts, 1980) (Kobayashi and Cho, 1981) (Nakamura and Namman, 1999) due to its higher elastic modulus (Table 2.1) and better mechanical properties than polypropylene fibers. However, from an economical point of view they have relatively higher price than polypropylene fibers. High-density polyethylene in monofilament forms (40 × 0.9 mm) with wart-like surface deformations along the length of the fiber at volume fraction of 0.2-0.4% have been used in Japan (Kobayashi and Cho, 1981). These deformations are intended to improve the mechanical bonding in cement paste and mortar. It has been reported that polyethylene fibers could be easily dispersed in concrete mixtures in volume percentages of up to 4 percent using conventional mixing techniques (Kobayashi and Cho, 1981).

Soroushian et al. (1992) compared the mechanical properties of polypropylene with polyethylene for different volume fractions. They found 0.025% volume fraction of polyethylene almost gives the same result as 0.1% volume fraction of polypropylene in flexural strength, demonstrating the better effectiveness of the use of polyethylene.

2.2.2.3 STRUX® 90/40 Fibers

STRUX® 90/40 is a polypropylene/polyethylene fiber blend produced by Grace Company, USA. STRUX® 90/40 is 40 mm (1.55 in.) in length with an aspect ratio of 90 that have been specifically designed to replace welded wire fabric, steel fibers, light rebar and other secondary reinforcement in slab-on-ground flooring and thin-walled precast applications. Since STRUX® 90/40 is relatively new; there are no published papers

available regarding the mechanical properties of this product other than the manufacturer's literature. Table 2.2 illustrates that the elastic modulus of STRUX® 90/40 is significantly higher than polyethylene and polypropylene, while the tensile strength is equivalent to the high range of the other two fibers. Melting point and ignition point of STRUX® 90/40 are similar to polyethylene fibers. STRUX® 90/40 is highly resistant to alkali, acid, and salt environments, and has almost the same specific gravity as polyethylene and polypropylene.

Table 2.2—Properties Comparison of STRUX® 90/40, Polypropylene, and Polyethylene Fibers.

Fiber Type	STRUX® 90/40 [1]	Polyethylene [2]	Polypropylene [2]
Specific Gravity	0.92	0.92-0.96	0.90-0.91
Absorption	None	None	None
Modulus of Elasticity, GPa	9.5	5.0	3.4- 4.8
Tensile Strength, MPa	620	76-586	138-689
Melting Point, °C	320	134	166
Ignition Point, °C	590	------	593
Alkali Resistance	High	High	High

1 Derived from Grace Company's Product Information.
2 Derived from ACI 544.1R-96.

The volume fraction of STRUX® 90/40 fibers can be varied between 0.18% to 0.7% depending on the application and desired properties, referring to the dosage table offered by the Grace Company (Appendix A). The use of STRUX® 90/40 is advantageous over

steel fibers due to the elimination of potential injuries caused by handling and placement, in addition to its corrosion resistance.

2.2.3 Shape of Fibers

It is generally known that polymeric fibers are much stronger than the bulk form of the same material, because there is less probability of internal defects, as well as the increase in mechanical properties due to the fiber crystallization attained during manufacture process. Long fibers are more effective in improving post-peak performance because of the larger bonding surface between each single fiber to the cement paste, but balling may become a problem as fiber length is increased (ACI 544.1R, 1996). Regarding the shape of fibers, they can be classified due to their diameter and character such as whiskers, wires, and single or monofilaments. Whiskers are highly crystallized fibers that are extremely strong, with very large fiber aspect ratio (length-to-diameter) ratio. In contrast, wires are large diameter fibers having small fiber aspect ratio. The shapes of fibers are chosen based in mechanical properties of each type and the intended application. Generally, whiskers are not used for reinforcement due to their poor bonding and high cost. Wires have their own applications which are out of the scope of this work.

Figure 2.3 illustrates the effect of fiber aspect ratio on tensile stress-strain improvement in the cement phase for randomly oriented multifilament glass strands. The shape of the mechanical response is similar for the varying lengths, but the longer fibers result in increased stress and strain capacity prior to fracture. Therefore, it is clear that the ductility and toughness will be increased by using longer fibers (larger aspect ratio), for the same fiber volume fraction.

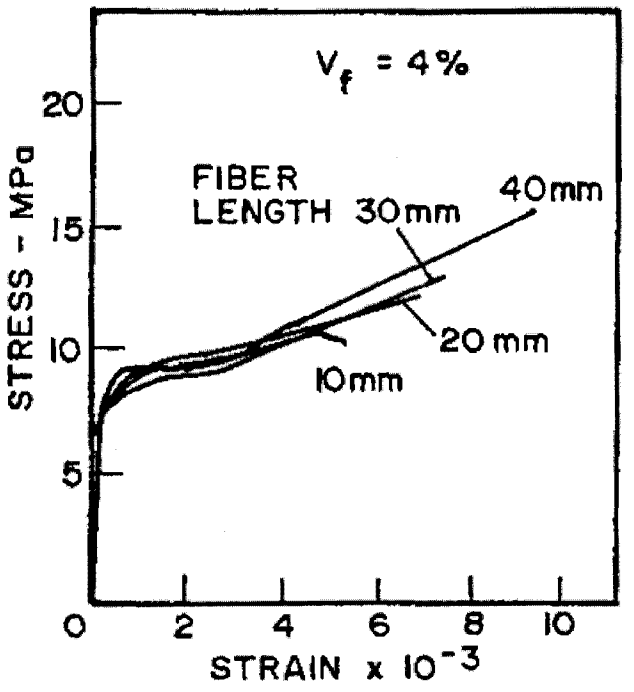


Figure 2.3—Effect of Strand Length (Aspect Ratio) on Tensile Stress-Strain Behavior for Glass Fibers in Cement Phase (Majumdar and Laws, 1991).

Feldman and Zheng (1993) have determined that:

> "In hybrid fiber-reinforced concrete based on steel and PP fibers, the stronger and stiffer steel fiber improves the ultimate strength, while the more flexible and ductile PP fiber leads to improved toughness and strain capacity in the post-crack zone."

Trottier and Mahoney (2001) developed a high tensile strength fiber that partially fibrillates during mixing with concrete, thereby increasing the bonding capacity with the matrix. The fiber is produced by extruding a mixture of polypropylene and polyethylene. An alternate to this technique is the STRUX® 90/40 fiber that is used in this work. The high tenacity of these fibers enhances the bonding with concrete and is useful in the hardened concrete to improve toughness so that sometimes they are called "structural fibers".

2.3 Fresh Properties of FRC

2.3.1 Parameters Governing the Workability of FRCs

The fluidity of concrete is reduced by adding any type of fiber (even non-water absorbent ones) because of their needlelike shape and high specific surface. The term workability is used to describe the consistency of the concrete mix. The loss of workability as a result of adding of fibers to the conventional concrete has been measured by the slump test, ASTM C 143 (2002), and this will be magnified by increasing the fiber aspect ratio, the amount of fibers added to the mixture. However, this difficulty can be solved by using vibration during placement. To have a more realistic measurement for workability of the fresh concrete using the inverted slump cone test (ASTM C 995, 2001) or the Vebe Test (BS 1881, 1983) (British Standards Institution, 1983) has been recommended (ACI 544.1R, 1996).

The main factors governing the workability and maximum fiber content possible of FRC are the fiber aspect ratio, the maximum size of the coarse aggregate, and fluidity and volume fraction of the paste phase. These factors do not include the use of chemical admixtures and the environment at the time of preparation of the concrete. Since going into depth regarding the effects of other factors (other than fibers) on workability of FRCs is beyond the scope of this work, for more information please refer to Johnson (2001).

2.3.2 Effect of Fiber Aspect Ratio on Workability

The amount of fiber added to the mixture has a dramatic effect on the workability of the concrete. The higher the fiber aspect ratio (L/D), the longer time required to vibrate

the concrete meaning the lower workability of the concrete. This phenomenon is clearly illustrated in Figure 2.4 showing the V-B time (Vibration time) of no more than 10s defining limit on fiber content for each aspect ratio beyond which workability decreases sharply for the particular mortar tested. Obviously, the desirable workability is dependent on the method of placement and more importantly the nature of application in which the concrete will be used.

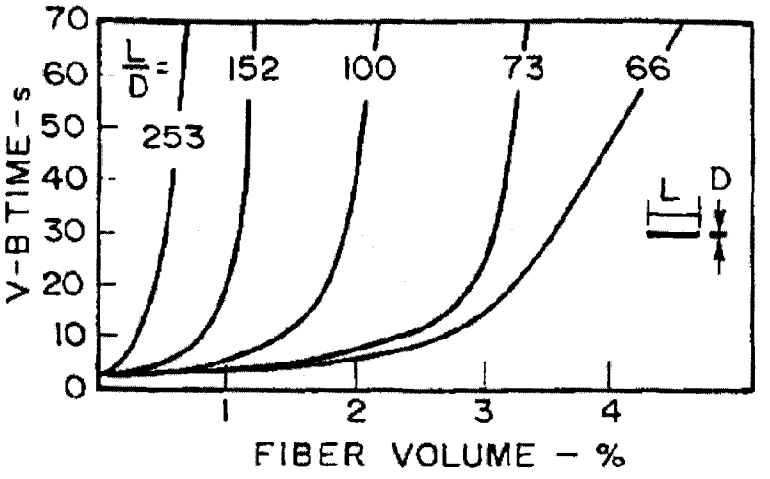


Figure 2.4—Effect of Fiber Aspect Ratio on Workability of Steel Fiber-Reinforced Mortars (Hannant, 1978).

2.3.3 Effect of Aggregate Size on Workability

The aggregate size used in the concrete mixture has direct effect on the workability of the concrete. Generally, increasing in the volume fraction and maximum size of the aggregates used in concrete decreases the volume fraction of the fluid phase so that the fibers have less space to disperse in the mixture. A 2-dimensional illustration (Figure 2.5) of course could be extended for a 3-dimensional reality has been investigated by Hannat, (1978). This figure bases on steel rigid fibers, but more or less could be considered for

the flexible fibers considering the general assumption of the availability of the free spaces. Figure 2.6 illustrates the effect of maximum aggregate size on workability of the concrete using rigid fibers; however, for flexible ones still is unknown. A V-B time of 10s, is much less for 20 mm aggregate concrete than for 10 mm aggregate concrete.

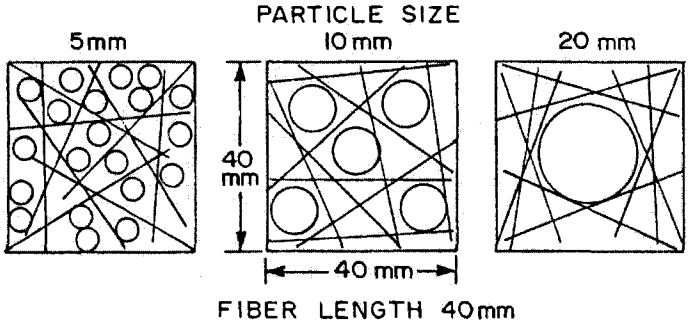


Figure 2.5—Schematic of Particle Size vs. Fiber Distribution for 40 mm Long Fibers within a 40 mm Square (Hannat, 1978).

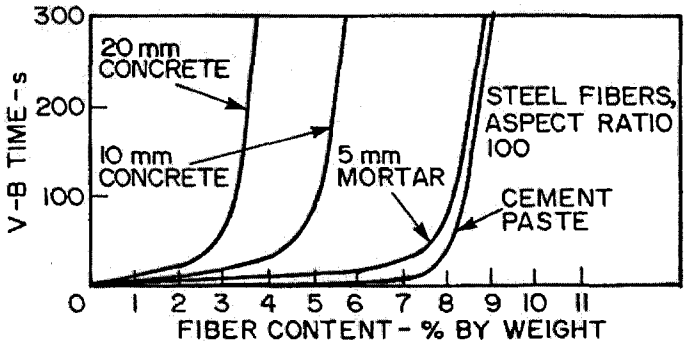


Figure 2.6—Effect of Aggregate Maximum Size on Workability for Steel Fiber of Aspect Ratio 100 (Hannat, 1978).

2.3.4 Effect of Paste Volume Fraction on Workability

The more the paste volume fraction of the concrete, the more space that the fibers can move and rotate, and the more workability for any particular fiber content (Figure 2.7) (Pieffer and Soukatchoff, 1994); moreover, the consistence of the fluid phase is important

since more viscose fluid phase might affect on the workability of the mixture negatively, thus doing a flow test has been recommended. The major keys to control the viscosity of the fluid phase of the concrete are to work on water-cement ratio of the mixture or adding superplasticizer or water- reduction admixtures.

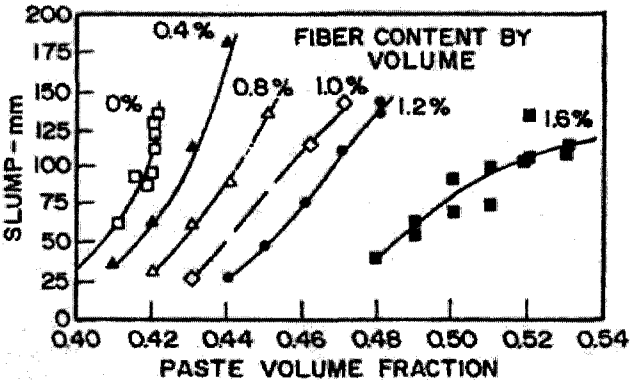


Figure 2.7—Effect of Paste Volume Fraction, on Workability of Steel Fiber-Reinforced Mortars with 30 mm Fibers (Pieffer and Soukatchoff, 1994).

2.4 Strength of FRC

The strength of FRC is directly related to the mixture proportion of the concrete as well as the fiber composition and shape, and bonding between the fibers and matrix. Since the original goal of adding fibers is not to improve the strength of the concrete but to control the cracking of FRC, the initial strength of FRC could be considered to be the same as the plain concrete. However, certain mechanical properties of FRC are affected by adding the fibers. For better understanding of the behavior of fiber reinforced concrete materials, the strength of FRC could be divided to two main points of view: compressive strength and flexural strength. Both of these characteristics of FRC are discussed separately.

2.4.1 Compressive Strength

Generally, fibers have little effect on the peak load of the compressive strength of FRC due to almost no compressive strength of fibers, since their effectiveness is in tension. However, they have direct impact on the post-peak load (Figure 2.8) (Fanella and Naaman, 1985). Since there is no special test method for the compressive strength of FRC except in Japan (JSCE SF5) (Bentur and Mindess, 2007), the same test method as compressive strength of plain concrete are generally used.

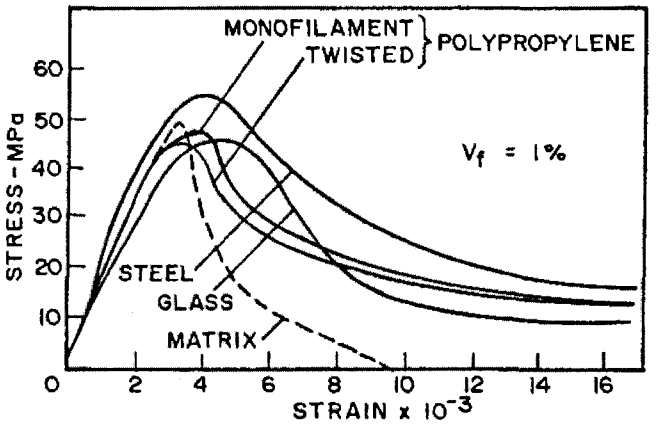


Figure 2.8—Compressive Stress-Strain Curves for Fiber-Reinforced Mortars with Various Types of Fibers (Fanella and Naaman, 1985).

Soroushian et al. (1992) have determined that the compressive strength of different volume fractions of polyethylene and polypropylene fibers decreases with increase in volume fraction as shown in figure 2.9. This figure illustrates that not only the higher volume fraction of fibers has more negative effect on the compressive strength of the FRC, but also the fiber composition has to be considered. In conclusion, the compressive strength of FRC could be considered the same as plain concrete except for minor differences due to the amount and composition of fibers, as indicated by the overlapping

confidence intervals. In this work, investigations will focus on the compressive strength of the STRUX® 90/40 fibers for 0.32 percent volume fractions.

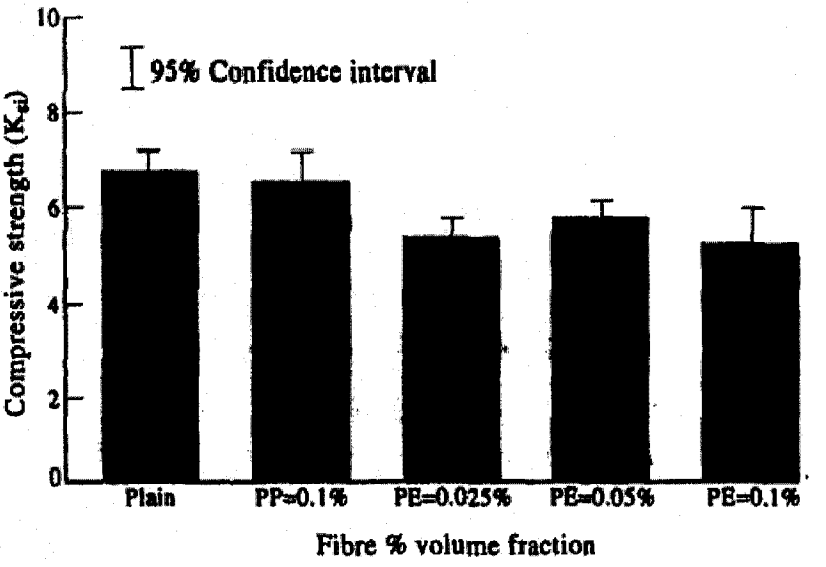


Figure 2.9—Average Values and 95% Confidence Intervals of Test Results for PE and PP Fiber-Reinforced Concrete, Compressive Strength (Soroushian et al., 1992).

2.4.2 Flexural Strength

There have been many investigations regarding the flexural strength of FRC having conflicting conclusions. Hughes and Fattuhi (1976), Hannant (1995), and Beddar (2004) found that adding fibers to concrete improves the peak flexural strength. In contrast, the design guide TR 34 (Concrete Society, 2003) suggests that adding fibers has no effect on the flexural strength of concrete, but fibers affect post crack flexural strength ratio.

Alhozaimy et al. (1996) summarizes in the following statement:

> "Contradictory test results have been reported by different investigators regarding the effects of polypropylene fibers on compressive and flexural strengths."

Soroushian et al. (1992) have determined that the flexural strength of polyethylene and polypropylene fiber reinforced concrete increases depending on the fiber composition and the volume fraction as seen in Figure 2.10. The STRUX® 90/40 fiber is relatively new so that there have been no academic investigations available concerning the flexural strength of the use of this fiber in FRC.

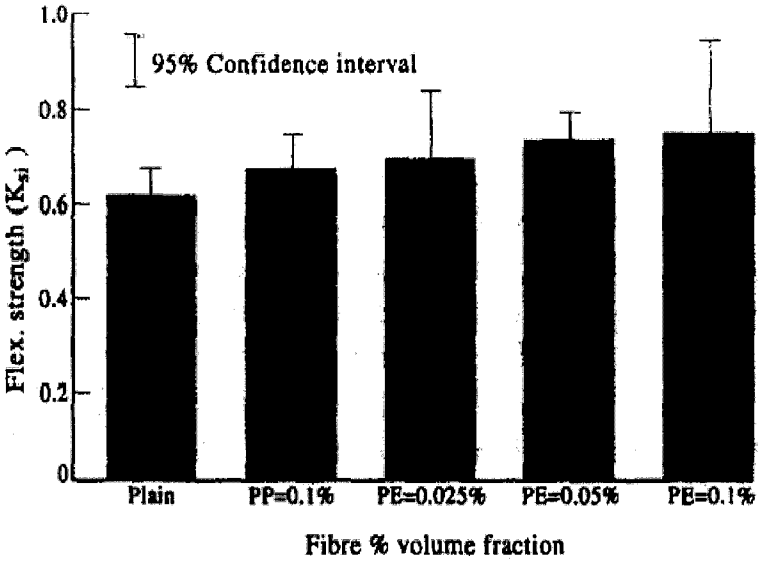


Figure 2.10—Average Values and 95% Confidence Intervals of Test Results for PE and PP Fiber-Reinforced Concrete, Flexural Strength (Soroushian et al., 1992).

To have a precise comparative investigation on the flexural performance of concrete, the geometry of specimens also has to be considered. There are two main points of view to interpreting data regarding the flexural strength of specimens. One uses methods which are derived from independency of specimens' dimensions such as ASTM C 1018 (1997), and the other which involves with the geometry of specimens similar to JSCE-SF4.

Chen et al. (1994) had a brief investigation on geometry of specimen and its effects on the toughness of the concrete. They found that the toughness of the concrete decreases

with an increase in the span-to-depth ratio of specimens. Also, the toughness will increase by increasing the width of the specimens with the same depth and span. Finally, the stress and deflection at first crack and ultimate flexural strength of the specimens are directly influenced by the specimen size. Some of their results are shown in Table 2.3.

Table 2.3—Collective Toughness Test Results from Chen et al. (1994).

Designation[a]	First Crack Strength (MPa)	First Crack Deflection (mm)	Maximum Strength (MPa)	ASTM Toughness Indices in I_{50}[b]
75:75:225	4.81	0.026	5.01	42.4
75:75:300	4.76	0.037	4.86	32.1
75:75:450	4.58	0.087	4.63	31.6
75:100:300	4.39	0.029	4.56	34.3
100:100:300	4.69	0.031	4.86	42.7
150:100:300	5.00	0.033	5.56	51.5

[a] Width:Depth:Span.
[b] According to ASTM 1018, (1989) I_{50} is equal to area under the bending load-deflection from starting point to 25.5δ (δ is the first crack deflection) divided to area under area from starting to δ. (The ASTM 1018-89 later on was changed)

2.5 Treatment Techniques of Fibers

The bonding between inorganic concrete and organic polymeric fibers has been considered due to the ordered chemical structure and lack of polar functionalities of polymeric fibers, causing poor adhesion between fibers and cement matrix (Dasgupta, S., 1990). As a result of the low compatibility of polymeric fibers with the cementitious matrix, the separation of fiber and cement matrices occurs before reaching the potential tensile strength decreasing the effectiveness of the fibers.

To improve bonding and wettability, surface photografting modification in gas-phase or liquid-phase has been widely considered (Tazuke and Kimura, 1978; Mingbo and Xingzhou, 1987; Allmer et al., 1988; Yamada et al., 1992; Feng et al., 1992; Hamilton et al., 1994). Previous investigations indicate that a pretreatment of the fibers is often necessary to improve the adhesion between polyolefin fibers and another material.

Brewis and Briggs (1985) reviewed the problem of adhesion of polyethylene and polypropylene in composites including the role of surface energy and wettability in the mechanism of adhesion. They indicated that the reason for poor adhesion of polyolefinic materials is due to the low surface energy, lack of functionality and potential weak boundary layers which they recommended that can be solved by pretreating the surface of fibers. These factors were also briefly discussed in a previous paper by same authors (Brewis and Briggs,1981).

During more recent years, various surface modification techniques to improve bonding between polymeric fibers and cement matrices have been introduced to the SNFRCs. Hild and Schwartz (1993) stated that an appropriate surface treatment technique on polyethylene fibers significantly improves the fiber/matrix bond. They found using 1 minute gas plasma treatment using argon, nitrogen, and carbon dioxide on ultra-high-strength polyethylene (UHSPE) fibers with 0.5 and 1% weight fractions has no significant differences in flexural stresses, but modest improvements of the flexural modulus and the stress-intensity factor and significant improvement in the toughness index and the fiber pull-out strength.

The treatment of fibers could be defined as all efforts that could be done on the surface of the fibers to improve the bonding between fibers and concrete. These techniques could be divided to two main groups: chemical and physical treatments.

2.5.1 Chemical Treatment of Fibers

Chemical treatment is one of the solutions to improve the bonding between polymeric fibers and cementitious material. Several techniques of fiber surface treatment have been introduced to improve the bonding properties of polyethylene fiber reinforced composite materials (Tissington et al., 1991). Chemical etching is an attempt, believed to modify the surface of the fiber by abstraction of hydrogen atoms from the polymer backbone and replacement with polar groups. This improves the wettability and the possibility of the appearance of available sites for chemical reaction with matrix (Landrock et al., 1985).

Generally, there are two main aspects of the fiber surface that directly affects the wettability, or the adhesion capability of the fiber's bonding with matrices. First is the roughness of the fiber surface which leads to increase in the apparent surface tension (Kinloch, 1987). Secondly, the introduction of polar groups containing oxygen, results in an increase of strongly hydrogen bonding at an oxidized polymer surface (Silverstein and Breuer, Polymer, 1993; Kinloch, 1987). This phenomenon will lead to enhancement of wettability of the surface fiber. Figure 2.11 represents the formation of polyethylene which could be continued by using different methods such as Ziegler and Philips process that higher polyethylene molecular weight is achieved. For more information regarding the formation of polyethylene, please refer to Feldman and Barbalata (1996). Figure 2.12 illustrates the formation of different types of polypropylene.

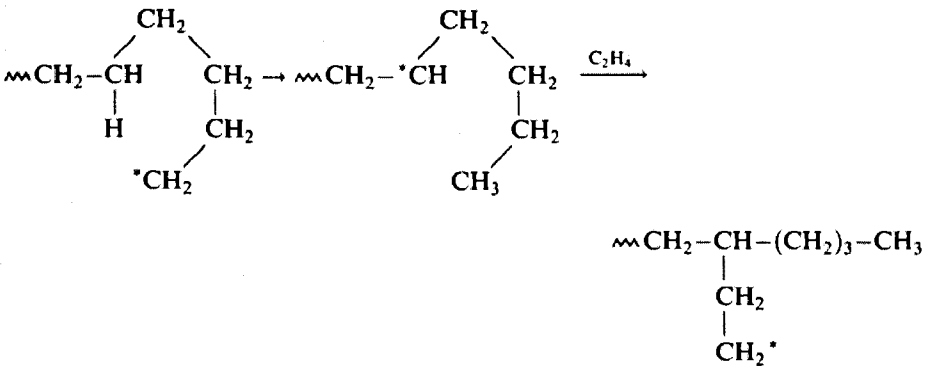
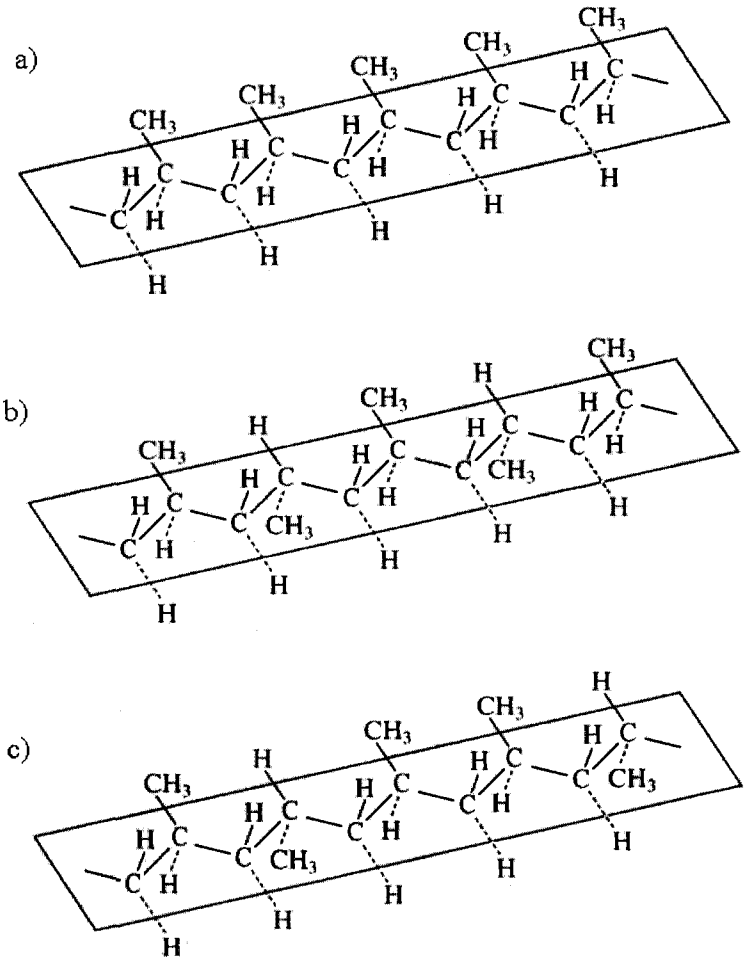


Figure 2.11—Formation of Polyethylene (Feldman and Barbalata, 1996)



a) Isotactic Polypropylene
b) Syndiotactic Polypropylene
c) Atactic Polypropylene

Figure 2.12—Formation of Different Types of Polypropylene (Feldman and Barbalata, 1996)

M. S. Silverstein et al. (1994) indicate that:

"One of the more significant changes in the fiber's surface chemistry is the introduction of carbon-oxygen bonds. These polar groups increase surface tension, enhance wetting, and present possible sites for chemical reactions with an epoxy resin yielding enhanced adhesion."

Generally, the higher wettability results in a greater chance of chemical interaction between the fibers and the matrix so that stronger bonding is achieved. A study on effects of surface modification by chromic acid, potassium permanganate ($KMnO_4$) and hydrogen peroxide (H_2O_2) etching indicate that surface morphology and in failure mechanism for the etched fibers apparently changed (Silverstein and Breuer, 1993).

Silverstein and Breuer (1993) studied the wettability and flotation of etched ultra high molecular weight polyethylene (UHMW-PE) fibers. They found the apparent surface tension of the rough and oxygen-rich chromic acid etched and $KMnO_4$ etched fibers was greater than those of the H_2O_2 etched and as-received fibers reflecting a low surface oxygen content and a smooth surface, respectively. The results are presented in Figure 2.13.

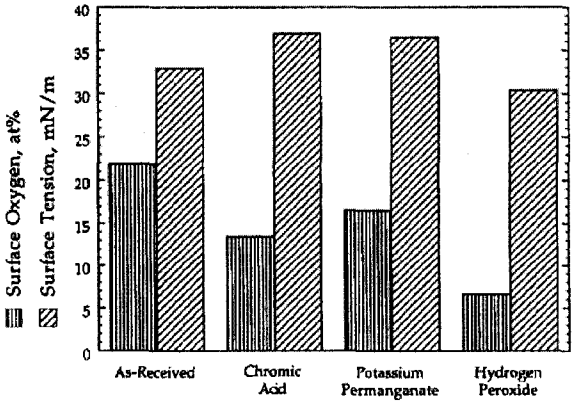


Figure 2.13—Apparent Surface Tension and Surface Oxygen Content for As-Received and Etched Fibers (Silverstein and Breuer, 1993).

In another investigation using epoxy droplets Silverstein and Breuer (1993) found chromic acid was the most powerful of the etchants investigated removing the weak boundary layer and oxidizing the polyethylene fibers, whereas, potassium permanganate and hydrogen peroxide etched fibers are weakly bonded to epoxy.

In this thesis, an investigation on bonding characteristics of chemically treated fibers to the cement using two type of chromic acid solution group (B & C), potassium permanganate, and hydrogen peroxide solutions will be performed. Chromic acid solution B, refers to potassium dichromate, is commonly used for improving the adhesion, dyeing and printing properties of polyolefine (Landrock, 1985). Solution C is based on sodium dichromate dehydrate which is a well-known oxidizing agent in organic chemistry.

2.5.2 Physical Treatment of Fibers

Another solution to enhance the bonding strength between fibers and cement is physical surface treatment of the fibers. Many surface treatment techniques of polyethylene (PE) and polypropylene (PP) have been introduced such as using corona-discharge treatment (Carley and Kitze, 1980; Spell and Christenson, 1979; Owens, 1975), activated gas plasma or glow discharge (Hall et al. 1969; Baszkin et al., 1976; Hollaham and Bell, 1974; Boenig, 1982), but none of these techniques has been accepted as a successful technique because of their technical or economical problems. Physical surface treatment could be described as all dry technique without involving wet chemical solutions. Surface treatment of polypropylene and polyethylene fibers by ozone has been considered as an efficient, economical, and potentially practical technique (Dasgupta, 1990). Because of the lack of fundamental information regarding to the mechanism of

ozone reaction and its control, this process has not yet been considered as a commercial technique. One of the scopes of this thesis is to focus on the results in this work that might contribute towards this objective.

Silverstein et al. (1994) found that the introduction of carbon-oxygen bonds (polar groups) in the fiber's surface present possible sites for chemical reactions so that enhance adhesion (Section 2.5.1), therefore the appearance of carbonyl (Figure 2.14, a) and carboxyl (Figure 2.14, b) on polyethylene and polypropylene fiber's surface could be advantageous to improve the bonding between fibers and concrete.

Figure 2.14—The Structure of Carbonyl (a) and Carboxyl (b).

Dasgupta (1990) investigated various levels of ozone surface treatment of Pulpex polypropylene and polyethylene pulp fibers and flakes to determine the difference in the amounts of ozone in and out in a closed system which is considered as the ozone uptake of the fibers or flakes. Then he used infrared spectrometry to determine the level of carbonyl and carboxyl generated by reaction of these polymers with ozone. The analytical results of carbonyl and carboxyl groups of polypropylene shown in Figures 2.15 and 2.16 demonstrate a linear relationship between the generation of carbonyl

groups in the polymer and the ozone uptake the fiber within the level of treatment made. It was found that PE generates higher levels of carbonyl groups than PP.

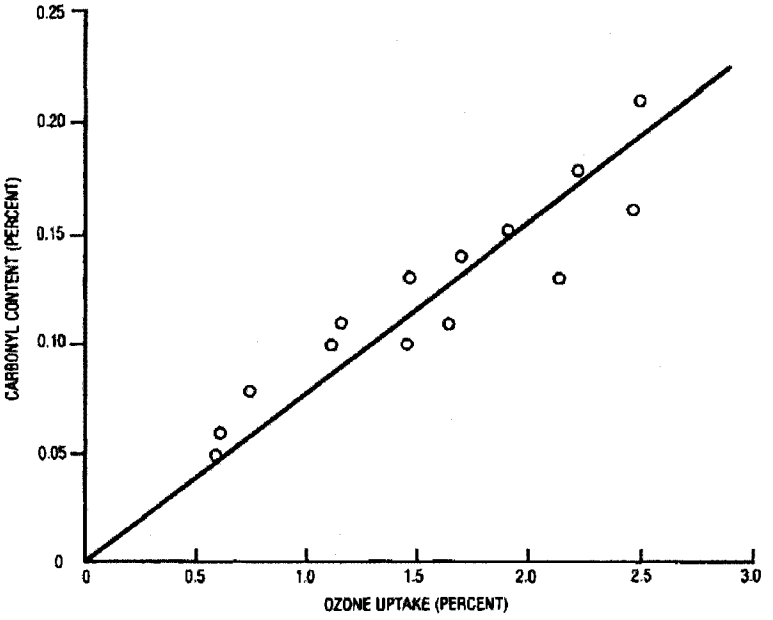


Figure 2.15—Carbonyl Contents of Ozonized Pulpex Polypropylene Pulp at Various Levels of Treatment. (Dasgupta, 1990)

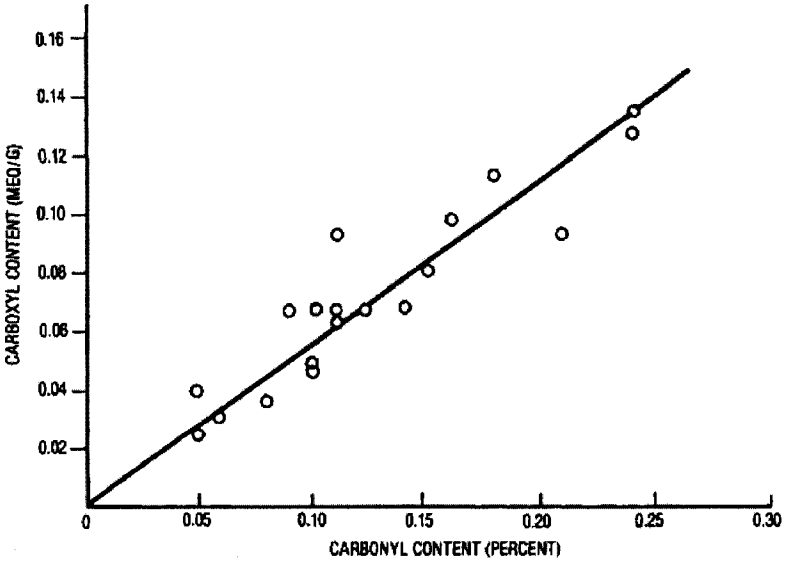


Figure 2.16—Carboxyl Contents of Ozonized Pulpex Polypropylene Pulp at Various Levels of Treatment (Dasgupta, 1990)

Figure 2.15 demonstrates the effectiveness of the ozone surface treatment of polypropylene and polyethylene. In Dasgupta's study, the level of treatment was defined as the ozone uptake (the difference in the amounts of ozone in and out) from a closed system. Then the procedure was followed by measuring the generated of carboxyl versus carbonyl in percent (Figure 2.16). In this work, the level of treatment was measured based on the duration of exposing to ozone in a steady rate.

Chtourou et al. (1993) used the same technique as Dasgupta to determine the ozone uptake from polyethylene pulp fiber by ozone treatment. They compared the amount of ozone in the outlet gas which left the reaction flask by means of the outlet tube with and without fiber showing the outlet ozone flow rate (mg/min) decreases, not only when reaction time increases, but also when fibers are present in the reaction flask (Figure 2.17).

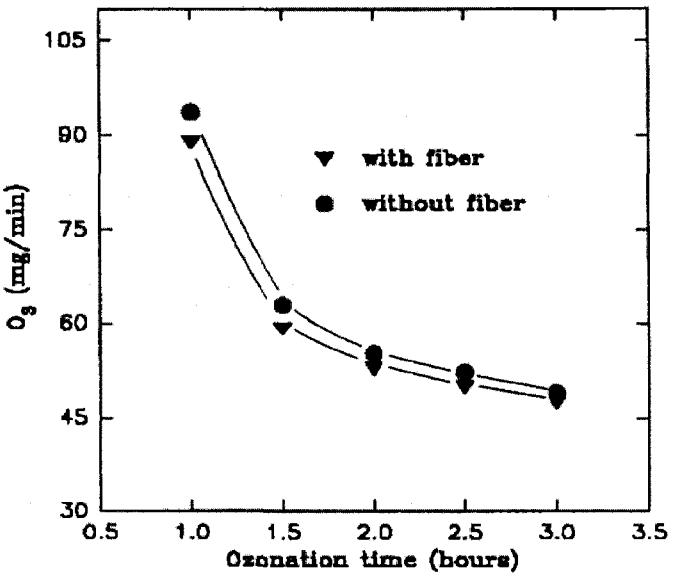


Figure 2.17—Outlet Ozone Flow Rate (mg/min), Determined Through $KI/Na_2S_2O_3$ Solutions, as a Function of Ozonation Time (Chtourou et al., 1993).

They concluded that ozone uptake for surface oxidation of the PE pulp fiber did not change with ozonation time, but the effect of this ozone uptake on the fiber was directly related to the time of reaction (Figure 2.18).

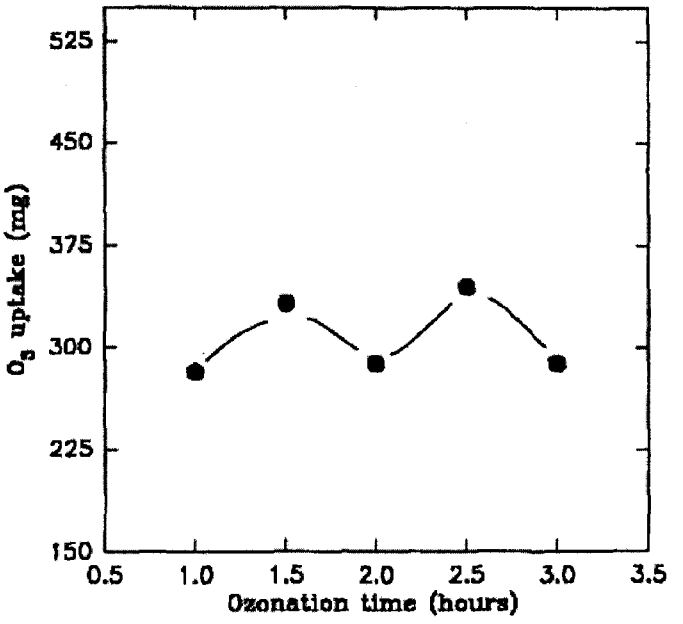


Figure 2.18—Total Ozone Uptake (mg), Used for the Uurface Oxidation of 25 g of PE Pulp Fiber, as a Function of the Ozonation Time (Chtourou et al., 1993).

Another technique of surface treatment is ultraviolet light/ozone (UVO_3) which has been developed in Canada and Japan in past decade (Foerch et al., 1990) (Yoshikawa and Kojima, 1992) (Yoshikawa et al., 1994) (Mcintyre and Walzak, 1995). The procedure of using this technique is to expose the polymer surface to UV in a definite flow of ozone. Mcintyre and Walzak (1995) reported the appearance of oxidation groups such as –C—O— and –C=O groups in the surface of polyolefin reacted by UVO_3 and a reduction of the contact angle to water of PP of 30°. Also, Yoshikawa et al. (1994) stated that the adhesion of PE fiber to epoxy resin matrix improved by using this technique.

Gongjian et al. (1996) investigated the use of UV light/ozone (UVO_3) treatment technique to modify the surface of polyethylene (PE) and polypropylene (PP). They exposed polyethylene and propylene sheets to UVO_3 for various times. Then they measured the contact angle to water of PE and PP sheets for each UVO_3 treatment time. Figure 2.19 illustrates the change of contact angle versus treatment time.

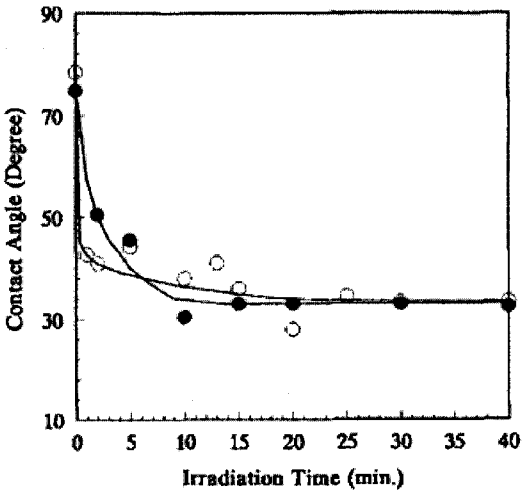


Figure 2.19—Changes of Contact Angle with Irradiation Time of PE and PP Sheets. UVO_3 Treatment Conditions: Temperature: 40°C; $\mathbf{O_2}$ Flow rate: 3 L/min. (●): PP Sheets; (○): PE Sheets (Gongjian et al., 1996).

The figure shows the time of exposure to UVO_3 has a direct effect on the contact angle of samples which reduced by increasing the treatment time. It can be seen that the rate of decreasing the contact angle of PE is greater than PP meaning that PE becomes hydrophilic more rapidly. However, both fiber types experienced insignificant contact angle reduction after 10 minutes of exposure. Also, they measured the change of tensile shear adhesive strength of PE and PP versus the UVO_3 treatment time (Figure 2.20).

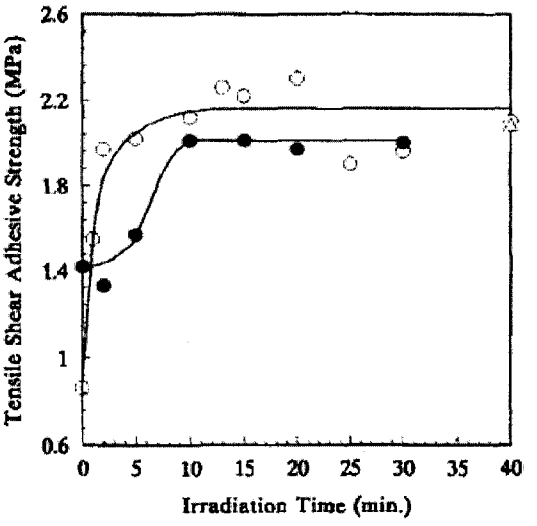


Figure 2.20—Changes in Tensile Shear Adhesive Strength with Irradiation Time of PE and PP Sheets. UVO_3 Treatment Conditions: Temperature: 40°C; O_2 Flow Rate: 3 L/min. (●): PP Sheets; (o): PE Sheets (Gongjian et al., 1996).

The results indicate the surface treatment with UVO_3 is an effective technique in adhesive strength improvement of PE and PP, considering the rate of adhesive strength greatly changes within first 10 min of exposing to the UNO and then it continues in a slower manner until there will be no more changes in the enhancement.

There has been more investigation on using ozone treatment on carbon fibers by Fu et al. (1996) who used ozone treatment involving exposure to O_3 gas (0.3 vol. %, in air) for 10 min at 160°C for carbon fiber. They reported that the tensile strength, modulus and ductility of carbon fiber reinforced cement paste were increased.

2.6 Contact Angle (Θ_c) Measurements

One of the major aspects which have direct effect on the strength of adhesion between fibers and matrix is the wettability of fibers. The reason of poor bonding strength between polypropylene fibers and cementitious matrix is widely accepted because of the

low wettability of polypropylene (Bentur and Mindess, 1990) (Hannat, 1978) (Addis, 1994) (Currie and Gradiner, 1989). To measure the wettability of fibers, the contact angle method has been commonly used to determine polymer surface tension and interfacial tension (Kinloch , 1987). This method includes the measurement of contact angle between a droplet of adhesion, which is water in our work, and a flat horizontal surface of polymeric fibers (2-dimension) (Figure 2.21).

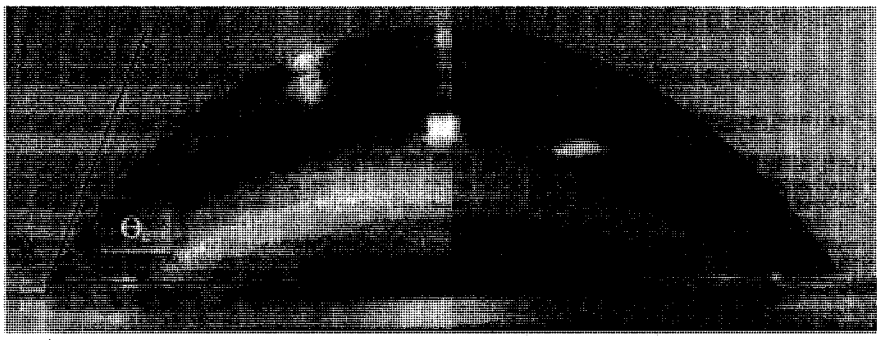


Figure 2.21—Image From a Contact Angle Device.

The difference of contact angles between the droplet and solid's surface (Figure 2.21) could be due to the surface energy of materials; the lower θ_c, the lower surface energy of adhesion or higher surface energy of the fibers results the better wettability of fibers. There has been a number of investigations that used the contact angle technique to determine the wettability of fibers such as on Ultra High Molecular Weight Polyethylene (Nardin, 1987) and Carbon (Gilbert et al., 1990), to verify the effectiveness of the surface treatment techniques (in the most surface treatment literature mentioned before).

2.7 Summary

It can be concluded from the literature review that using polyethylene and polypropylene as reinforcement is so effective to improve the post-crack strength besides their advantages such as corrosion resistance as well as their compatibility with moisture and cement alkalis environment of concrete (FRC). The main problem of using them is their poor adhesion to the cement matrix because of their ordered chemical structure and lack of polar groups on their surface.

Various surface treatment techniques have been introduced during past few decades struggling to improve the bonding between polymeric fibers and concrete. These efforts could be divided to two main groups: chemical and physical treatment techniques. Some of the results showed the enhancement of bonding between fibers and concrete in contrast some of them did not. Not only the amount of improvement is important but also that cost and probability of using these techniques in industrial scale has to be considered.

The experimental program of the present investigation into different surface treatment techniques of fibers in bonding improvement between fibers and concrete is presented in the following chapter. The results of this experimental work are discussed in chapter 4.

Chapter 3

Experimental Program

3.1 General

The experimental program was planned to study on the bonding characteristics of fibers introduced to concrete mixtures (FRC) and a solution to improve the bonding between the polyethylene/propylene blend fibers and concrete. The concrete mixing was conducted in the Structural Laboratory of Concordia University, followed by mechanical testing in the Building Materials Laboratory. Physical treatments and contact angle measurements were carried out in the Chemistry Department of Concordia University at Loyola Campus.

The steps of this research program were to:

1. determine the best proportion of aggregates, cement, and water to achieve the desired target compressive strength (30 MPa) meanwhile the proper workability of the concrete (slump).
2. examine the compressive and flexural strength of the plain concrete (without fibers).
3. investigate the improvement of the mechanical properties of the concrete by introduction of polyethylene/propylene blend fibers without using surface treatment to later on compare the effectiveness of the different treatment techniques.

4. study the effects of different chemical and physical treatment techniques on the improvement of bonding between fibers and concrete, meanwhile on the change of their contact angle between a droplet of water and the surface of fibers.

The experimental program consisted of testing a total of 154 cylindrical specimens for the compression test and 112 rectangular specimens for the flexural test. All cylindrical specimens had the same dimension, $d = 102$ mm and $h = 204$ mm and beams had cross section dimension, $b = 76$ mm and length, $l = 305$ mm. Initially several specimens were prepared and tested to design the best mixing proportion of the concrete. At the next step, the cylinders and beams were divided into several groups, based on the fiber dosage and the treatment technique of the fibers. Table 3.1 illustrates the abbreviations of non-fiber, untreated and treated fiber properties of the prepared samples that were used.

Table 3.1—Abbreviation and Properties of Groups.

Abbreviation	Dosage of Fibers, %	Property
PC	0	Plain concrete without fiber
NT	0.32	FRC without treatment, middle dosage
CAB	0.32	FRC treated with Chromic Acid Sol., Type B
CAC	0.32	FRC treated with Chromic Acid Sol. Type C
PP	0.32	FRC treated with, Potassium Permanganate Permanganate
HP	0.32	FRC treated with Hydrogen Peroxide
UV-30	0.32	FRC treated with 30 min. UV
UVO_3-5	0.32	FRC treated with 5 min. UV and Ozone
UVO_3-10	0.32	FRC treated with 10 min. UV and Ozone
UVO_3-40	0.32	FRC treated with 40 min. UV and Ozone
UVO_3-90	0.32	FRC treated with 90 min. UV and Ozone
NT (HD)	0.50	FRC without treatment, High Dosage
CAB (HD)	0.50	FRC treated with Chromic Acid Sol. Type B, High Dosage

3.2 Materials

3.2.1 Aggregates

In civil engineering, the term of aggregate means a mass of crushed stone, gravel, or sand, predominantly composed of individual particles, but in some cases including clays and silts (Mamlouk and Zaniewski, 2006). In this work, use of the term aggregate will be limited to gravel coarse aggregate and sand referred to respectively as coarse and fine aggregate. About 70 % of the concrete specimens' volume in this thesis is made up of aggregates. Using the aggregates as filler not only reduces the amount of cement paste so that a concrete mixture is more economic, but also improves the quality of the concrete due to greater volume stability than hardened cement paste, under the condition that the aggregate volume does not exceed a certain amount.

The size of aggregates used in civil engineering ranges between five microns to one hundred fifty millimeters. The size of aggregates generally is determined by sieve analysis (ASTM C136, 2005). The particles larger than the openings in each sieve are retained by the sieve, and the smaller ones pass trough. According to ASTM C 125 (2003) coarse aggregates are defined as particles retained on the 4.75-mm, and fine aggregates as those passing the 4.75-mm sieve.

The shapes of particles are divided to two distinguished shapes; angular shapes which are the particles from crushed rocks and rounded particles due to weathering in transporting in water. Angular shape articles generally produce higher stability. The sizes of fine and course aggregates used in this work are according to the ASTM classifications (ASTM C136, 2005) and the shapes of the course aggregate particles are angular have

particle size range from 2.5 to 10 mm. The aggregates were provided by Cement St-Laurent.

3.2.2 Portland Cement

There are different types of Portland cement with distinguished properties, and they are chosen based on the concrete application. Generally, choosing the type of cement is related to the required rate of strength gain, the heat generation due to the hydration of cement and the required resistance to sulfate. Table 3.2 describes the five standard types of Portland cement specified by ASTM C150 (2002) and CSA A23.1.

Table 3.2—Standard Types of Portland Cement Specified by ASTM C150 and CSA.

ASTM Type	CSA Type	Name	Application
I	GU	Normal, General Use	General concrete work when the special properties of other types are not needed. Suitable for floors, reinforced concrete structure, pavements, etc.
II	MS, MH	Moderate Sulfate Resistance, Moderate Heat of Hydration	Protection against moderate sulfate exposure, 0.1-0.2% weight water soluble sulfate in soil or 150-1500 ppm sulfate in water (sea water). Can be specified with a moderate heat of hydration, making it suitable for large piers, heavy abutments, and retaining walls. The moderate heat of hydration is also beneficial when placing concrete in warm weather.
III	HE	High Early Strength	Used for fast-track construction when forms need to be removed as son as possible or structures needs to be put in service as soon as possible. In cold weather, reduces time required for controlled curing.
IV	LH	Low Heat of Hydration	Used when mass of structure, such as large dams, requires careful control of the heat of hydration.
V	HS	High Sulfate Resistance	Protection from severe sulfate exposure, 0.2-2.0% weight water soluble sulfate in soil or 1500-10,800 ppm sulfate in water

In this work, Type GU hydraulic cement produced by Cement St-Laurent, Québec, Canada was used.

3.2.3 STRUX® 90/40 Fibers

STRUX® 90/40 is a polypropylene/polyethylene blend fiber produced by Grace Company, USA (Figure 3.1). STRUX® 90/40 is 40 mm in length with an aspect ratio of 90 that has specifically been designed to replace welded wire fabric, steel fibers, light rebar and other secondary reinforcement in slab-on-ground flooring and thin-walled precast applications.

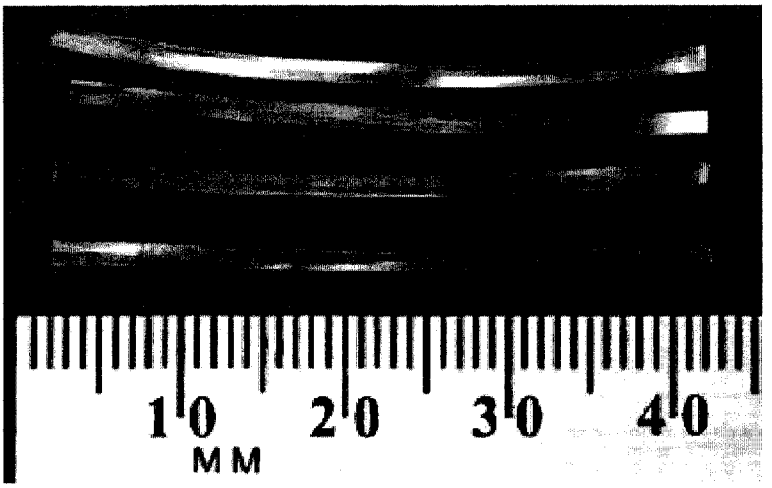


Figure 3.1—Image from STRUX® 90/40 Fibers.

Since STRUX® is relatively new product; there are no academic papers available regarding the mechanical properties of this product. Table 3.3 illustrate that the elastic modulus and tensile strength of STRUX® are relatively high compared to polyethylene and polypropylene. The melting point and ignition point of STRUX® is close to

polyethylene fibers. STRUX® is highly resistant to alkali, acid, and salt environments, while having almost the same specific gravity as polyethylene and polypropylene.

Table 3.3—Properties Comparison of STRUX® 90/40, Polypropylene (PP), and Polyethylene (PE) Fibers.

Fiber Type*	STRUX 90/40	Polyethylene	Polypropylene
Specific Gravity	0.92	0.92-0.96	0.90-0.91
Absorption	None	None	None
Modulus of Elasticity, GPa	9.5	5.0	3.4- 4.8
Tensile Strength, MPa	620	76-586	138-689
Melting Point, °C	320	134	166
Ignition Point, °C	590	------	593
Alkali Resistance	High	High	High

* The information is derived from ACI 544.1R-96, and Grace Company's Product Information

The volume fraction of FRC using STRUX® could be varied between 0.18% to 0.70% depending on the application and desired properties referring to the dosage table offered by the Grace Company. The use of STRUX® 90/40 is advantageous over the steel fibers due to the elimination of potential injuries caused by handling and placement, besides its corrosion resistance. For more information regarding to design specification of STRUX® 90/40 please refer to product information on Grace Company website.

3.2.4 ADVA 140® Admixture

Admixtures are ingredients other than Portland cement, water, aggregates and fiber reinforcement that may be added to concrete to impart specific qualities to either the plastic (fresh) or hardened concrete (ASTM C494, 2005; ACI 116R, 2000). The Portland

Cement Association (PCA) identifies four major reasons for using admixtures (Kosmatka et al., 2002):

1. to reduce the cost of concrete construction
2. to achieve certain properties in concrete more effectively than by other means
3. to ensure quality of concrete during the stages of mixing, transporting, placing, and curing in adverse weather conditions
4. to overcome certain emergencies during concrete operations

Admixtures could be considered such as air entrainers, water reducers, retarders, and accelerators. The admixture used in this work, ADVA 140®, is a high range water reducing admixture recommended by the fiber manufacturer (Grace Company). The general purpose of using these admixtures is to gain workability without sacrificing the quality of the concrete, since exceeding required amount of water for the hydration cement will detriment the mechanical properties of the concrete.

3.3 Determination of the Mixing Proportion

The proportioning of concrete mixes directly affects workability and the strength of the concrete. The PCA (Portland Cement Association) specifies three qualities required of properly proportioned concrete mixtures (Kosmatka et al., 2002):

1. acceptable workability of freshly mixed concrete
2. durability, strength, and uniform appearance of hardened concrete
3. economy

To determine the best mixing proportion of the concrete all of the above specifications have to be carefully considered.

3.3.1 Moisture Content of Aggregates

Concrete mixture design is based on aggregate in the saturated surface dry condition. Considering the moisture content of aggregates to design an exact proportioning of concrete mixes and adding a proper amount of mixing water is essential. A high moisture content of aggregates results in a higher total amount of specified water to the designed mixing proportion causing higher than desired workability but more importantly lower designed strength and more concrete shrinkage (Mamlouk and Zaniewski, 2006; ACI 224R, 2001); On the other hand, lower moisture content can cause lower than desired workability and less chance of proper distribution of fibers to the concrete (Johnston, 2001). All the effects of moisture content are not limited to those were mentioned above, for more information refer to Mamlouk and Zaniewski (2006).

The determination of the moisture content of aggregates, fine and coarse, is essential to have consistent slump and mechanical properties. ASTM C 127 (2004) and C 128 (2004) suggested that the coarse and fine aggregates must be oven dry at 110 ± 5°C for sufficient time to reach a constant dry mass. Then the moisture content of the aggregates can be determined by comparing the mass in naturally moist condition to oven dried condition. According to the standard, for the nominal maximum size of coarse aggregates used in this experiment (less than 12.5 mm) the minimum mass of test sample for the oven drying is 2 kg and for the fine aggregates is 1 kg. The mass should be reported to the nearest 0.01kg.

In this work, the moisture content of both fine and course aggregates were considered and evaluated according to ASTM standards, respectively C 128 (2004) and C 127 (2004). First a proper mixing proportion to reach the desired slump and compressive

strength was determined. Then the moisture contents of aggregates in that mixing proportion that was used were determined. Since the moisture content of the aggregates varied due to the settling of the water to the bottom of the storage barrels, the moisture contents was evaluated every single time before making each set of samples. Then the mixing proportion was adjusted to the originally determined mixing proportion.

3.3.2 Design of the Mixing Proportion

The design of the best mixing proportion is directly affected by all parameters previously mentioned in section 2.3.The initial mixing proportion used in this work was suggested by St. Lawrence Company which was on the basis of satisfaction of the required workability of the concrete and a water/cement ratio of 0.5 (Table 3.4). Meanwhile, the target compressive strength of concrete in this work was specified to reach 30 MPa at the age of 28 days.

Table 3.4—Mixing Proportion of Concrete Suggested by St. Lawrence Company.

Concrete Materials	**kg/m^3**
Coarse aggregate	1140
Fine Aggregate	760
Cement	345
Water	173

After testing several specimens using the above mixing proportion, the compressive strength was achieved about 12 MPa which did not satisfy the initial specified strength that was assigned for this work. This could be due all environmental conditions of the

structural lab and the materials that were used, since as mentioned previously many factors might affect the strength of concrete. At the next step, the mixing proportion was changed to the following with a water/cement ratio of 0.66 (Table 3.5) which was recommended by Grace Company.

Table 3.5—Mixing Proportion of Concrete Suggested by Grace Company.

Concrete Materials	**kg/m^3**
Coarse aggregate	878
Fine Aggregate	1074
Cement	270
Water	178

A number of compressive strength tests of this mixing proportion showed compressive strength results about 18 MPa which still was too far from 30 MPa. The procedure to find the best mixing proportion was continued by changing the water/cement ratio from 0.66 to 0.59 while adding 60 g more cement to the mixing proportion than the amount suggested by Grace. The mixing proportion used is illustrated in Table 3.6. By using this mixing proportion, a compressive strength of 29.7 MPa was reached at 28 days which was completely satisfactory for this work. All detailed compressive strength results regarding to the steps of determination the mixing proportion are presented in Appendix B.

Table 3.6—Mixing Proportion of Concrete Used in This Work.

Concrete Materials	**kg/m^3**
Coarse aggregate	878
Fine Aggregate	1074
Cement	330
Water	195

The slump test was performed to evaluate the workability of the concrete produced by using this mixing proportion. The slump of concrete was measured in preparation of concrete for all groups in two steps as recommended by Grace Company: first after preparation of plain concrete, and then after adding the fibers and mixing for five minutes. The results showed the slump was relatively high (about 160 mm) before adding the fibers, but once the fibers (at 0.32% dosage) were introduced to the concrete the slump was decreased by about 60 mm which was desirable according to ASTM C 143 (2002). For the samples made with 0.50% dosage of fibers, the slump again decreased to about 40 mm. The results of slump test proved the higher the fiber content, the lower the slump of concrete which the reason of this criterion was briefly discussed in previous sections. The concrete specifications for the mixing proportion are illustrated in Table 3.7.

Table 3.7—Specification of Typical Plain Concrete Used in This Work.

Plain Concrete Group (PC)		Values
Water/Cement Ratio		0.59
Slump, mm		60
Maximum Load, kN	7 Days	216
	28 Days	268
Compressive Strength, MPa	7 Days	24.0
	28 Days	29.7

3.4 Fiber Surface Treatment Procedures

3.4.1 Chemical Treatment

The fibers were separated manually and introduced into the respective solutions, where they were left for various times and temperatures as noted below. After treatment, the fibers were washed ten times in 2.5 L tap water and thereafter dried at 45°C for 24 h. The procedures were as following:

1. Chromic acid solution (solution B) :

   It was prepared by mixing potassium dichromate, sulfuric acid and distilled water in a 37.5:750:60 mass ratio. The fibers were immersed in 470 ml solution and the treatment was done at 70°C for 1min.

2. Chromic acid solution (solution C):

   It was prepared by mixing sodium dichromate dehydrate, sulfuric acid and water in a 100:134: 427 mass ratio and the treatment was done at 55°C for 18 h.

3. Potassium permanganate solution:

   It was prepared by mixing potassium permanganate, nitric acid and water in a 15.8:1.58:625 mass ratios (actually mixing 500ml aqueous 0.2M potassium permanganate solution with 125 ml aqueous 0.2M nitric acid solution. The fibers were immersed in 625 ml solution and kept at room temperature for 24 h.

4. Hydrogen peroxide solution:

The concentration of hydrogen peroxide in water was 30%. The fibers were immersed in 600 ml solution and kept at room temperature for 24 h.

3.4.2 Physical Treatment

The physical surface treatment of fibers in this work was divided to two main techniques: 1) UV, 2) UV and Ozone.

To perform the UV treatment, a clear 175 watt UV mercury lamp from General Electric Company (GE) was used. A number of individual fibers (5 to 10) which had less production defects on their surface were collected and placed on an aluminum plate. Then the area between the lamp and the plate was covered by aluminum sheets. The reason for doing this was to have a better exposure of UV to all different sides of fibers. The fibers were exposed to the UV lamp for 10, 30, 60, 90, and 120 minutes to determine the best duration of UV treatment by using the contact angle measurement method, as mentioned in section 3.6.3. To prepare enough fibers to make a complete set of samples, the fibers divided into several (3-4) batches, and then each batch was exposed to the UV lamp for the specified time. The reason was due to the limitation of available surface area under the lamp and to have more effective treatment the fibers would not be overlapped.

For the UV and Ozone treatment technique, a UVO_3 Cleaner, Model 342 from Jelight Company, INC. (CA, USA) was used. All procedures for this treatment technique were the same as that mentioned for UV treatment, but the duration of exposure was 5, 10, 15, 20, 30, 40, 60, 90 minutes. Since the UVO_3 cleaner used in this technique was a closed system with reflective internal surface, there was no need to cover around the fibers and lamp with an aluminum sheet.

3.5 Sampling, Making and Curing Test Specimens

The mechanical properties of concrete are greatly affected by the condition of making and curing procedures as well as sampling of the specimens. ASTM C 192 (2005) describes the standard requirements for preparation of materials, mixing concrete, and making and curing concrete specimens under the laboratory conditions. Regarding to this standard the following is a highlight of specifications that had to be considered:

Both cylindrical and rectangular molds have to be made of steel, cast iron, or other nonabsorbent material, nonreactive with concrete containing portland or other hydraulic cements; moreover, molds have to hold their dimensions and shape under all conditions of use.

A suitable sealant has to be used to prevent escaping moisture from joints of the molds.

Using proper vibrator is essential to prevent the appearance of bubbles in fresh concrete resulting lower strength of final product. To this purpose, using external table is permitted with a minimum frequency of 3600 vibrators per minute.

The number of layers for sampling the specimens is 2 for layers for cylindrical molds up to 225 mm diameter, and 1 layer for rectangular molds up to 200 mm depth, using an external vibrator.

Tests ages often used are 7 and 28 days for compressive strength tests, versus 14 and 28 for flexural strength tests.

For more specifications regarding to the temperature, cement, aggregates, mixing conditions and procedures refer to ASTM standards.

All of above specifications were followed for sampling, making and curing of test specimens in this work except for the external vibrator. ASTM C 192 (2005) indicates that the duration of vibration depends on the workability of the concrete and effectiveness of the vibrator, should be between 5 to 10 seconds. Since the frequency of the external table vibrator used in this work could not be controlled, the proper duration of vibration was determined as soon as appearance of the smooth surface of the concrete and ceasing the break of large bubbles through the top surface. This method is also acceptable according to the mentioned ASTM standard.

3.5.1 Procedure of Making Plain Concrete

The procedure to make samples form plain concrete was as follows:

1) Inside of the mixer was wetted,

2) Coarse aggregate and 30% of mixing water were added to the mixer,

3) The mixer was started and then fine aggregate was added,

4) Cement and remaining water was added to mixer while the mixer was running,

5) The concrete was mixed for 3 minutes, followed by a 3 minutes rest and then 2 minutes final mixing.

3.5.2 Procedure of Making Fiber Reinforced Concrete

This procedure was performed according to the Grace Company recommendation:

1) Inside of the mixer was wetted,

2) Stone and sand (coarse and fine aggregates) were added to mixer and mixed for 30 seconds,

3) 75% of the mixing water accompanied by the superplasticizer was added to mixer and mixed for 30 seconds,

4) The mixer was switched off for 2 minutes,

5) The cement and rest of water were added after restarting the mixer,

6) The concrete was mixed for 3-4 minutes followed by 2 minutes rest followed by 2 minutes final mixing,

7) The first slump test was performed,

8) The concrete was put back to the mixer and the fibers were added while the mixer was running,

9) The fiber reinforced concrete was mixed for 5 minutes,

10) The second slump was measured.

3.5.3 Sampling, Curing and Specifications of Test Specimens

The concrete used for casting both cylindrical and beams molds were from the same batch for testing each group. The specimens were prepared within 15 minutes and stored with a plastic cover in a moist environment. After removal from the molds at 24 hours, the specimens were submerged in lime-saturated water until testing. The typical mixing proportion was given in section 3.3.2 with the water/cement ratio of 0.59. The designed compressive strength of the concrete ranged from 23 MPa to 24 MPa at the age of 7 days and from 29 MPa to 30 MPa at age of 28 days.

The initial slump of the mix (before adding the fibers) was 160±20 mm and the second slump (after introduction of fibers) was 60±20 mm. The superplasticizer was used in this work was ADVA 140® which is a high range water reducing admixture. The required

amount of ADVA 140® added to the concrete recommended by Grace Company was from 400 to 600 ml per 100 kg of concrete In this work 500 ml per 100 kg of cement was chosen.

3.6 Design of Test Specimens

The first step of the experimental program was the determination of the geometry of specimens for compressive and flexural strength. In general, standards were used when possible or dimensions were based on previous investigations in this field. Some constraints were introduced due the difficulty of handling larger specimens and the capacity of equipment had to be considered. The following is the design of the geometry of specimens for each of tests.

3.6.1 Geometry of Specimens for Compression Test

The most used shape of specimens for compression test referring to the North American standards and previous studies has been cylinders. ASTM C 192 (2005) suggests that the diameter of cylindrical specimen should be at least three times the nominal maximum size of the coarse aggregate in the concrete. The size of aggregates used in this work ranged between 2.5 to 10 mm therefore according to the ASTM standard the diameter of cylindrical molds has to be greater than 30 mm. The size of specimens that was chosen for this work was 102 mm in diameter and 204 mm in height, a commonly used size in practice. The ASTM standard specified a correction coefficient for specimens which have less than 1.8 length/diameter ratio, but since the length to diameter ratio of the specimen used in this work is 2; there is no need for use of the

coefficient. The number of layers to make specimens for this size of mold is specified 2 with equal depth, and could be consolidated either by rodding or vibration. In this work an external vibrator table was used.

3.6.2 Geometry of Specimens for Flexural Test

Rectangular specimens are the most widely used shape to test the flexural strength of concrete. The ASTM C 192 (2005) also has to be applied so that the minimum cross-sectional dimension of the rectangular section for this work has to be 30 mm. Additionally, ASTM C 1609 (2005) suggests the length of specimen for the flexural strength test has to be at least 50 mm greater than three times of the depth. Also, the length of the test specimen shall not be more than two times the depth greater than the span. In addition, for specimens containing fibers, the depth and width of the specimen should be three times the fiber length (40 mm in this work).

In this work, rectangular shaped metal molds *(76×76×305 mm)* were used which satisfied the length requirements, but not the requirements for the depth and width of specimen due to the limited capacity of mixer and testing machine.

3.7 Test Set-up and Instruments

In this work, three types of tests were performed:

1. Compression test
2. Flexural test
3. Contact angle measurement

The purpose of the first test was to determine the compressive strength of the concrete, to design a proper mixture proportion, and moreover to investigate the effect of adding different types of surface treated fibers. The flexural test is the most relevant test to the objective of this work which is to evaluate the effectiveness of different fiber surface treatment techniques by examining the load-deflection curve for the specimens, and finally to study the ultimate (peak load) and residual strength at any ratio of net deflection. The last test's purpose was to determine the effectiveness of each surface treatment technique by determining the fiber's surface (wettability) and specify the best duration of treatment for the physical treatment techniques (UV and UVO_3).

3.7.1 Set-up for Compression Test

This test method was performed according to ASTM C39 (2001), Standard Test Method for Compressive Strength of Cylindrical Concrete Specimen, which consists of applying a compressive axial load to molded cylinders until failure occurs; however in this work the application of load (in some cases) was continued to evaluate the compressive strength behavior for different fiber surface treatment techniques after reaching the failure point (from the load versus the travel of testing machine's platens). Two Tinius Olsen Testing Machine (PA, USA) with different capacities of 300 and 600 kN were used, depending on the ultimate compressive strength of specimens.

All specimens were tested as soon as removed from the moist storage at the ages of 7 and 28 days. After the specimens were cleaned by a moist towel, they were placed hardened face up between two load bearing caps (Figures 3.2 and 3.3). The purpose of using those caps was to distribute the load equally all over the surface of specimens, since

the face that the specimen is casted usually does not have a completely smooth surface. The caps included a hard thick plastic sheet inside which could be replaced after specific number of uses.

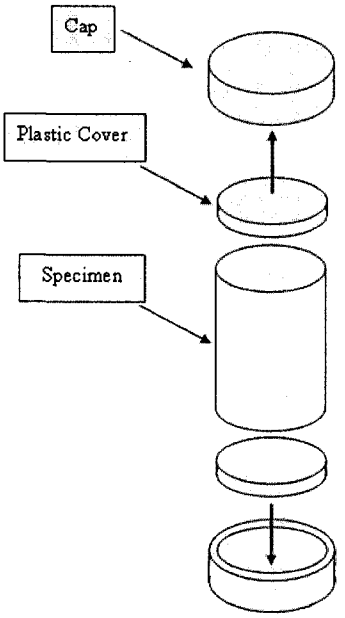


Figure 3.2—Schematic Diagram for Compression Tests.

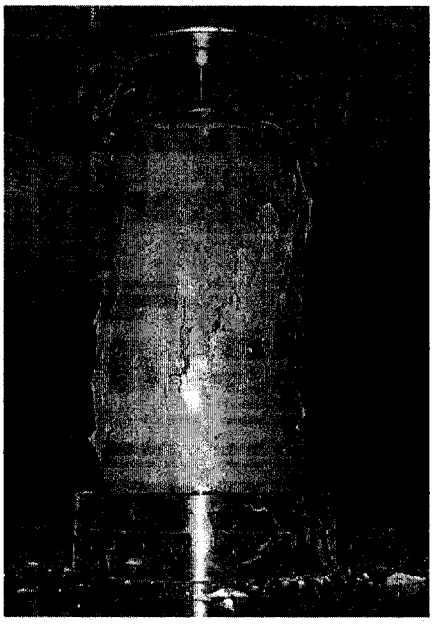


Figure 3.3—Image from the Set-up of Specimens for Compression Test.

ASTM C39 (2001) specifies the moving head travel of the testing machine shall travel at a rate of approximately 1 mm/min, but in this work the rate of movement were visually controlled in a continuous rate considering with no shock applied to the specimens during the test (the testing machines used do not have the capability of rate control). After the specimens failed the testing machine was continued to apply load until the end capacity of the machine's displacement plotter.

This work initially began by taking compression test as mentioned in section 3.3.2 to determine the best mixing proportion to reach desired compressive strength meanwhile proper workability. For testing the compressive strength of each group of samples 10 cylindrical specimens with 102 mm in diameter and 204 mm in height were prepared to be tested at ages of 7 and 28 days (i.e. 5 samples for each of these times). After testing several specimens coming from various mixing proportions a proper mixing proportion was designed. The compressive strength was calculated by dividing the maximum load carried by the specimen by cross-section area (ASTM C 39, 2004).

The performance of compression test was continued up to certain point which results clearly showed there were not significant changes in the compressive strength between the samples of concrete made by various fiber surface treated techniques and the sample which did not have any fibers. From that point the study program was limited to investigate on only flexural strength of samples however one cylindrical specimen from each batch was prepared and tested for the quality control of the concrete.

3.7.2 Set-up for Flexural Test

The investigation of the effects of fiber surface treatment technique was based on the flexural strength of specimens. Generally, there two main flexural test methods: center point loading and third-point loading. The main difference between these two method is in the center point loading the load applies in on a point at the middle of the specimens (span), but third point loading includes a load cell at top which applies the load in two different point of specimen (Figure 3.4 and 3.5).

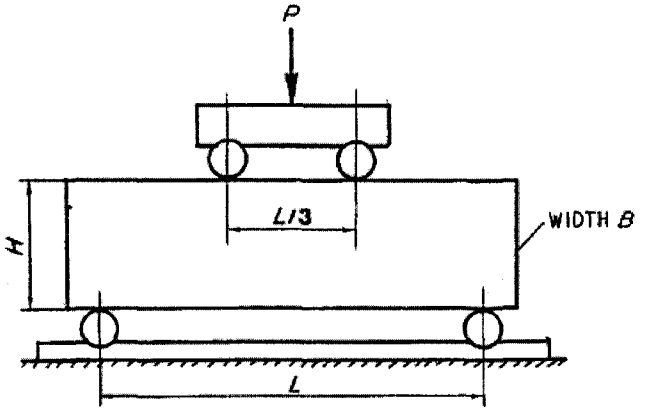


Figure 3.4—Schematic Diagram for flexural tests, Third-Point Loading (Wang, et al., 1987).

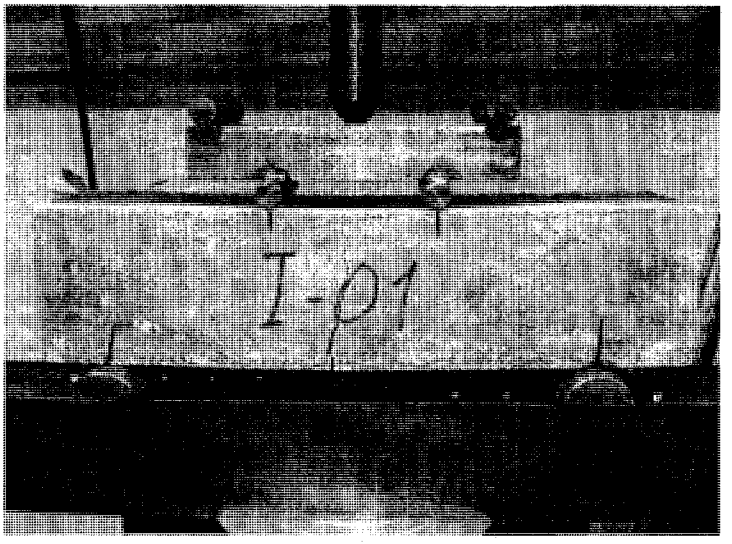


Figure 3.5—Image from the Set-up of Specimens for Flexural Test, Third-Point Loading.

In this work third-point loading method was preferred which ensure that forces applied to the beam will be vertical to face of the specimen and applied without eccentricity (ASTM C 78, 2002). Figure 3.6 illustrates how the loads apply in third-point loading method to the specimens. It also ensures that a constant moment occurs in the middle third of the specimen for more accurate calculation of stresses. In this method, parameters such as first-peak, peak and residual loads at specific deflections are derived from the load-deflection curve to evaluate the flexural performance of fiber reinforced concrete.

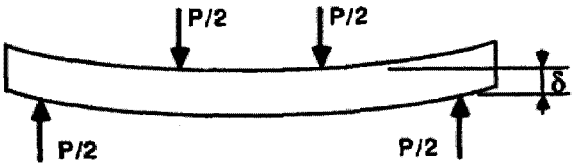


Figure 3.6—Typical Load-Deflection Curve of a Flexural Member (Chanvillard et al., 1990)

In this work, an Instron (Model 1125) manufactured by Electromechanical Test System (MA, USA) with a machine capacity of 100 kN was used to perform the flexural test. This testing machine is capable to apply load at a constant specified rate (by ASTM C 1609, 2005) and to generate the load versus net deflection curve accurately.

The flexural test was the main method that was used in this work to study the bonding strength between concrete and fibers with various surface treatment techniques applied to them. The test initially was started by using 3 rectangular shaped beams with depth and width of 76 mm and the length of 305 mm for testing the flexural strength of plain concrete. Later on, the number of molds was increased to 5 beams in the case of testing surface treated fibers to increase the accuracy of results.

ASTM C 1609 (2005) indicates that rate of applying the load in flexural test has significant effect on evaluating the flexural strength of concrete. In this standard, the rate of load shall be constant within the range 0.05 to 0.10 mm/min until the specified end-point is reached for a 350 by100 by 100 mm specimen size. The specified end-point is defined as 1/150 of the test span (L) while the reading point for after the first crack occurs is 1/600 of the test span. Since the dimensions of specimen used in this work were smaller than those used in the standard, the low range (0.05 mm/min) was chosen for the rate of applying load. As the length of specimen has to be at least 50 mm greater than the three times the depth (25 mm from each side of the seaports), for a 350×100×100 mm specimen size this value comes to 2 mm ([350-50]/150). Figures 3.7 and 3.8 illustrate the parameters that have to be calculated, specified by ASTM C 1609 (2005).

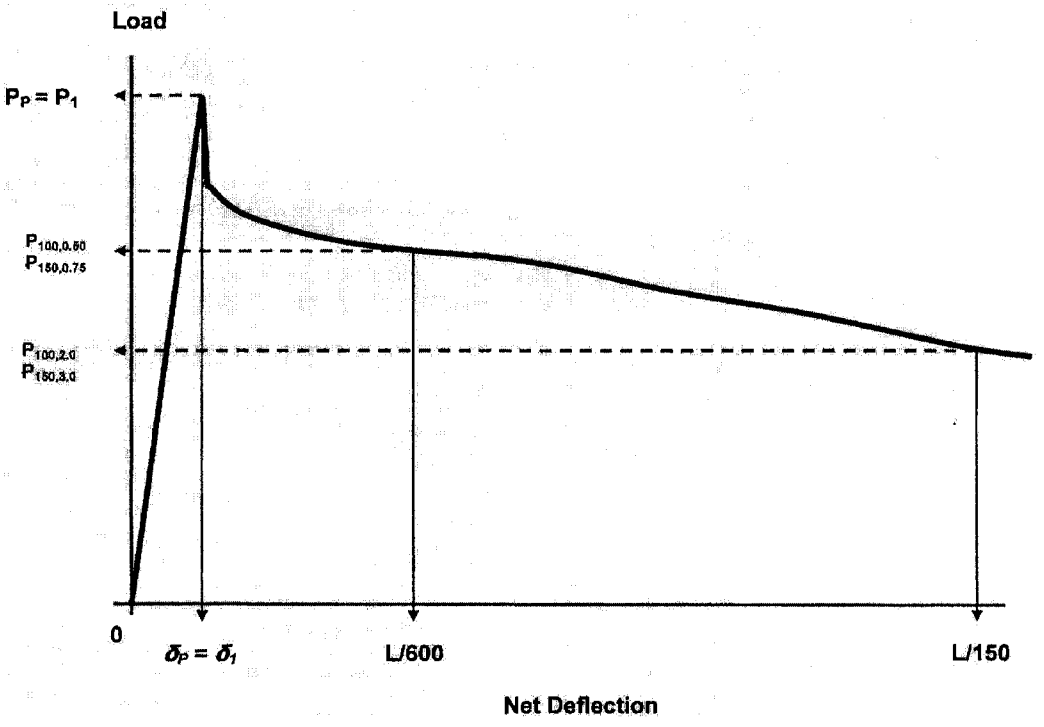


Figure 3.7—Parameter Calculation for First-Peak load equal to Peak Load (Not to Scale) (ASTM C 1609, 2005).

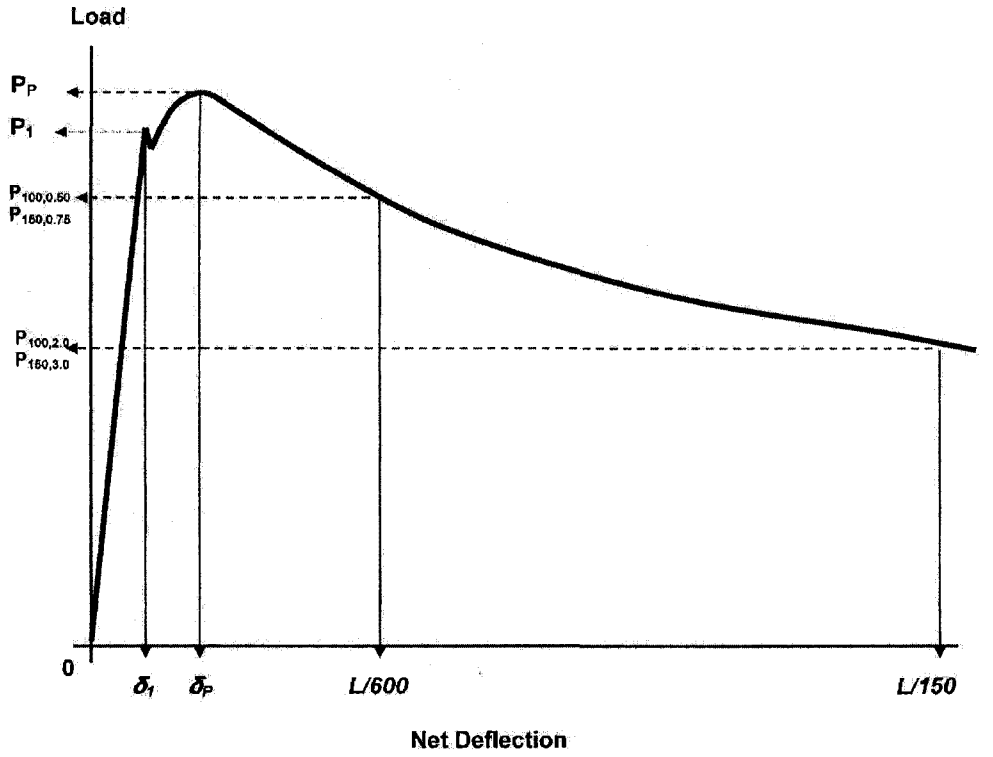


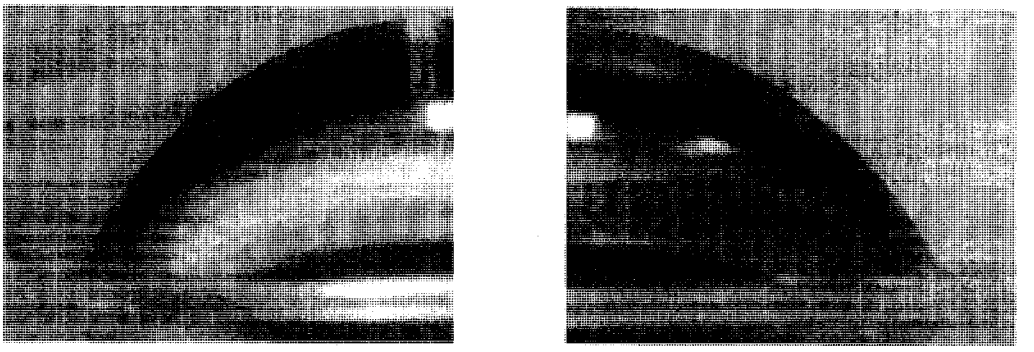
Figure 3.8—Parameter Calculation when Peak Load is Greater than First-Peak load (Not to Scale) (ASTM C 1609, 2005).

3.7.3 Contact Angle Measurement

Contact angle measurement is a method to evaluate the wettability of fiber's surface by determination of the angle between the surface of the fiber and a water droplet. This test was performed to determine the best duration of treatment for the physical surface treatment techniques (UV and UVO_3), and moreover to compare the effectiveness of all fiber surface treatment techniques presented in this work. Decreasing contact angle illustrates better wettability of fibers so that the more effective the surface treatment technique.

A PAT-1 surface tensiometer manufactured by Sinterface Technologies, (Berlin, Germany) accompanied with a digital camera was chosen to perform this test. The use of

the equipment was kindly provided by Dr. Rolf Schmidt, the Chemistry Department. This method included putting a droplet of water on the surface of fiber which had been fixed on a plate. Then the digital camera was fixed in a plane at the same level of the plate to take a picture. To reach more accurate results, this could be done in two steps meaning that pictures were taken both from left and right side of the droplet (Figure 3.9). The last step is to measure the angle between each droplet and fiber's surface by using UTHSCSA Image Tool, Version 3.00 software.



(a) (b)

Figure 3.9—Image From the Right Side (a) and Left Side (b) of a Water Droplet.

Chapter 4

Experimental Results

4.1 General

In the previous chapter, an experimental program to investigate the effect of fiber surface treatment on the bonding strength between concrete and fibers was described. Ten cylindrical specimens were prepared and tested to determine the compressive strength (for 7 and 28 days). Five rectangular specimens for all surface treatment techniques used in this work were prepared and tested to study the flexural strength of FRC and bonding strength of fibers in concrete. The contact angle measurement of water droplet on the surface of fibers and "Gradient of Decreasing Residual Strength" were introduced to evaluate the effectiveness of the various fiber surface treatment techniques. In the following sections, the results of the experimental investigation are reported and discussed. The variables in the tests are the technique of fiber surface treatment and the dosage of fiber added to the concrete. The procedures to test the compressive and flexural strength are according to the ASTM standards and the contact angle measurement is a common method to evaluate the wettability of fibers in FRC. "Gradient of Decreasing Residual Strength" is a developed method that is introduced in this work. All results from the contact angle measurement and "Gradient of Decreasing Residual Strength" are

compared to those obtained from the flexural strength tests. The Coefficient of Variation for all the compressive and flexural strength tests was calculated and compared to those specified by ASTM standards to validate the results.

4.2 Plain Concrete versus Fiber Reinforced Concrete

4.2.1 Mixing Proportion

The mixing proportion for plain concrete which was determined by modification of mixing proportion recommended by Grace Company, previously mentioned in section 3.3.2, is shown in Table 4.1.

Table 4.1—Mixing Proportion of Plain Concrete.

Concrete Materials	**kg/m^3**
Coarse aggregate	878
Aggregate	1074
Cement	330
Water	195

To study the effectiveness of different fiber surface treatment techniques on the bonding strength of fibers and concrete there was need to know the bonding strength without using any surface treatment techniques. This test was performed in two stages using the middle and high dosage of fibers as recommended by the fiber manufacturer. The middle dosage was the base amount of fibers that used in this work for investigation on bonding strength. The high dosage of untreated fiber reinforced concrete was tested to compare with the high dosage of the treatment technique that gave the best results among

of all surface treatment techniques. The middle and high dosages were 0.32% and 0.50% fibers by volume, meaning respectively 3.1 and 4.75 grams added to the concrete. The typical mixture proportion for fiber reinforced concrete used in this work is presented in Table 4.2.

Table 4.2— Typical Mixture Proportion for Fiber Reinforced Concrete Used in this Work.

Concrete Materials		**kg/m^3**
Coarse aggregate		878
Aggregate		1074
Cement		330
Water		195
Fiber	Middle dosage	3.10
	High Dosage	4.75
ADVA 140®, 500ml/100kg of Cement		

As previously discussed in section 3.3.1, adding fibers significantly affects the workability or the slump of concrete. After preparation of each mixture, the slump test was measured according to ASTM C 143 (2002) to indicate the workability of plain concrete and fiber reinforced concrete. The results (Table 4.3) proves the effectiveness of using the superplasticizer (ADVA 140®) since after introduction of fibers to concrete the slump remained similar to the mixture without fibers (about 60 mm).

Table 4.3—1st and 2nd Slump Values for Typical Plain and Fiber Reinforced Concrete Mixture Proportion.

Group	Slump, mm	
	1st	2nd
Plain Concrete (PC)	60	–
Non-Treated, Middle Dosage (NT)	165	65

4.2.2 Compressive Strength

In the following sections, initially the procedure to calculate the compressive strength is mentioned. Then the calculation of Coefficient of Variation which was applied to all group test results and their acceptable ranges according to ASTM C 39 (2004) and C 1609 (2005) respectively for plain and fiber reinforced concrete will be presented and compared. The detailed results of all compression tests are presented in Appendix B.

4.2.2.1 Calculation of Compressive Strength

The steps to calculate the compressive strength were as following:

1) The values derived from the testing machine were converted from lb to N:

$$\text{P, Load (lb)} \times 4.448222 = \text{Load (N)} \tag{4.1}$$

2) Cross-section area of the cylindrical specimens were calculated:

$$A = \pi \times (D/2)^2 = \pi \times (0.102/2)^2 = 0.008171 \text{ m}^2 \tag{4.2}$$

3) The compressive strength was calculated by division of load to area:

$$\sigma = P/A \text{ (N/ m}^2 \text{ or Pa)} /1{,}000{,}000 = \sigma \text{ (MPa)} \tag{4.3}$$

4) The results were reported to the nearest 0.1 MPa (ASTM C 39, 2004).

5) Standard Deviation and Coefficient of Variation (COV): The Coefficient of Variation (COV) had to be calculated and compared to those specified by ASTMC 39 standard and to make sure the tests had been performed properly. Standard Deviation and Coefficient of Variation was calculated as following:

$$s = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}(x_i - \overline{x})^2}, \tag{4.4}$$

where:

s = Standard Deviation

N = Number of Samples

$x_i = \{x_1, x_2, \ldots, x_n\}$

$\overline{x}$ = The mean of samples that was calculated from formula 4.5.

$$\overline{x} = \frac{1}{N}\sum_{i=1}^{N} x_i = \frac{x_1 + x_2 + \cdots + x_N}{N} \tag{4.5}$$

Then the Coefficient of Variation was calculated as following:

Coefficient of Variation (COV) = Standard Deviation (s) / Mean of samples ($\overline{x}$) × 100 % (4.6)

Table 4.4 illustrates the acceptable values of Coefficient of Variations for compressive strength of concrete specified by ASTM C 39 (2004). The Coefficient of

Version (COV) for the specimen size used in this work at laboratory condition has to be 3.2% or less.

Table 4.4—Acceptable Values of Coefficient of Variation for Compressive Strength (ASTM C 39, 2004).

Size of Specimen	Conditions	Coefficient of Variation	Acceptable Range of Individual Cylinder Strengths	
			2 cylinders	3 cylinders
150 by 300 mm	Laboratory conditions	2.4 %	6.6 %	7.8 %
	Field conditions	2.9 %	8.0 %	9.5 %
100 by 200 mm	Laboratory conditions	3.2 %	9.0 %	10.6 %

4.2.2.2 Compressive Strength Results

Table 4.5 compares the compressive strength of plain and fiber reinforced concrete. According to ASTM C 39 (2004) the maximum load and compressive strength for both 7 and 28 days has to be reported.

Table 4.5—Compressive Strength Test Results for Mixture Proportion of Plain and Fiber Reinforced Concrete.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa (COV %)
PC	216	24.0	268	29.7 (0.5)
NT	196	21.7	239	26.6 (2.0)

It can be clearly seen that the compressive strength of concrete decreased by introduction of fibers to concrete. These results agree with the findings of Soroushian et al. (1992), section 2.4.1, that the compressive strength decreases with increase in volume

fraction of fibers.

4.2.3 Flexural Strength

In all cases for fiber reinforced concrete mixtures in this work, the results showed that First-Peak loads were equal to peak load so that Figure 3.7 which previously presented in section 3.7.2 was mainly considered. A span of 228.6 mm (9 in), which satisfied the minimum length specified by the standard (228.6 + 50 = 278.6 mm < 305 mm), was chosen for the flexural tests in this work. After taking the first flexural test, it was seen that the L/600 (span/600) parameter occurred before the peak load (Figure 4.1). It was due to the smaller dimensions of specimen used in this work (305×76×76 mm) than the specimen dimensions (350×100×100 mm) used in the standard.

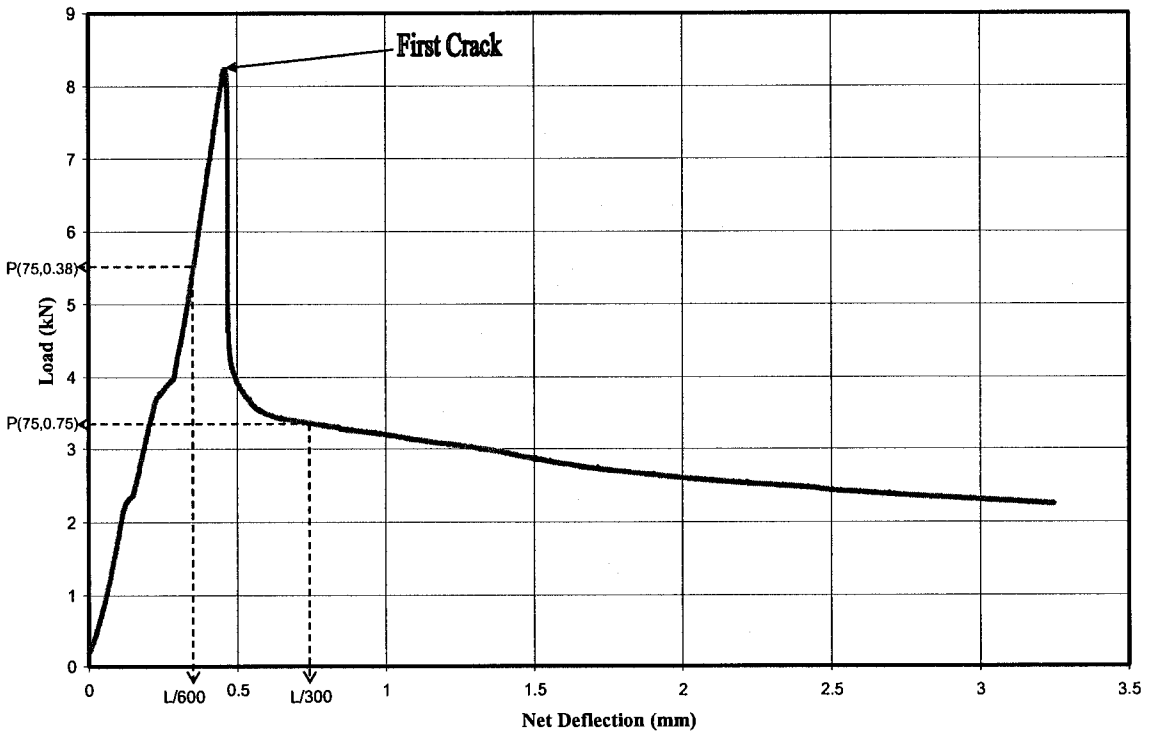
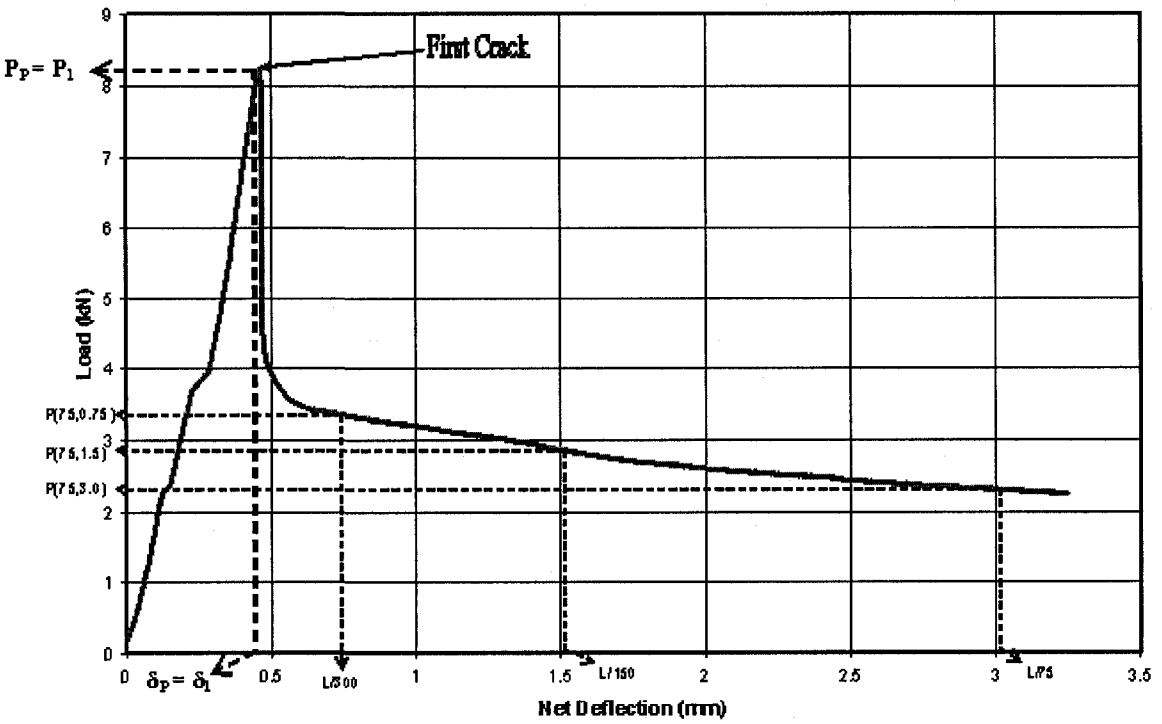


Figure 4.1—The L/600 Residual Strength Typically Seen in the Flexural Test in This Work (Load-Deflection Curve).

To achieve proper results to study the bonding strength of specimens the above parameters were modified to L/300 (span/300) for the residual strength after the appearance of the first crack and L/150 (span/150) as specified by the standard. Additionally, a parameter L/75 (span/75) was defined in this work in order to more fully characterize the residual strength at the End-Point of 3 mm (Figure 4.2).



L = Test Span
$P_P = P_1$ = Peak Load = First-Peak Load
$\delta_P = \delta_1$ = Net Deflection at Peak and First-Peak Loads
$P_{75,0.75}$ = Residual Load at Span / 300
$P_{75,1.5}$ = Residual Load at Span / 150
$P_{75,3.0}$ = Residual Load at Span / 75

Figure 4.2—Example of Parameter Determination Used in this Work.

4.2.3.1 Calculation of Flexural Strength

The steps to calculate the flexural strength parameters were as following:

1) The span of testing machine was measured to the nearest 2 mm (ASTM C 1609, 2005).

2) The load derived from testing machine for Peak Load, Pp, or Firs-Peak Load, P_1, and the Residual Load at Spans L/300, L/150, and L/75 ($P_{75,0.75}$, $P_{75,1.5}$, $P_{75,3.0}$) were collected.

3) Peak Strength, fp, or First-Peak Strength, f_1, and Residual Strength at Spans L/300, L/150 and L/75 ($f_{75,0.75}$, $f_{75,1.5}$, $f_{75,3.0}$) were calculated by following formula (ASTM C 1609, 2005):

$$f = PL/bd^2 \tag{4.7}$$

where:

f = the strength, MPa,

P = the load, N,

L = the span length, mm,

b = the average width of the specimen, mm,

d = the average depth of the specimen, mm at the fracture.

4) loads and strengths were reported to the nearest respectively 1 N and 0.05 MPa respectively.

5) the toughness (T, Area under L-D Curve 0 to Span L/ 75) was measured, with aid of software available in the equipment, and reported to the nearest 0.1 Joule.

6) the calculation of standard deviation and Coefficient of Variation (COV) were previously mentioned in section 4.2.2.1. The acceptable values of Coefficient of Variations for flexural strength of fiber reinforced concrete specified by ASTM C 1609 (2005) are presented in Table 4.6. The dimensions of specimens used for flexural test in the standard (102×102×356 mm) were not the same as those we used in this work, but their COV ranges was used for L/300 (after the first crack occurs) and L/150 (the End-Point residual strength recorded in the standard) of the flexural results of this work.

Table 4.6—Acceptable Values of Coefficient of Variation for Flexural Strength (ASTM C 1609, 2005).

Parameters	COV, % (ASTM C 1609)
P_p, N	7.7
f_p, MPa	8.3
δ_1, mm	20.0
$P_{100,0.50}$, N	17.6
$P_{100,2.0}$, N	15.2
$f_{100,0.50}$, MPa	17.8
$f_{100,2.0}$, MPa	16.4
$T_{100,2.0}$, Joule	16.0

4.2.3.2 Flexural Strength Results

The flexural test specifications were previously mentioned in section 3.7.2. The flexural strength test was performed on five beams. In the case of varied results a minimum of three results, which were close, were collected as representative for flexural performance of that group. The results of the flexural strength test for plain and non-

treated fiber reinforced concrete are presented in Table 4.7. All detailed results and calculations of flexural tests including 5 beams are presented in Appendix B.

Table 4.7—Flexural Strength Test Results for Mixing Proportion of Plain and Non-Treated Fiber Reinforced Concrete.

Parameters	PC (COV %)	NT (COV %)	Acceptable COV % (ASTM C 1609)
P_p, N	9,604 (1.7)	8,199 (1.3)	7.7
f_P, MPa	4.96 (1.7)	4.24 (1.3)	8.3
δ_P, mm	0.46 (4.6)	0.45 (4.4)	20.0
$P_{75,0.75}$, N	0	2,528 (4.5)	17.6
$P_{75,1.5}$, N	0	2,435 (4.9)	15.2
$P_{75,3.0}$, N	0	2,067	–
$f_{75,0.75}$, MPa	0	1.30 (4.5)	17.8
$f_{75,1.5}$, MPa	0	1.26 (4.8)	16.4
$f_{75,3.0}$, MPa	0	1.06	–
$T_{75,3.0}$, Joule	1.9 (3.3)	7.7 (4.7)	16.0

The results showed there was no post crack flexural strength for the specimens prepared without using any fibers. It can be seen that all COV results for the tests are within the range that specified by ASTM standards, so that the results are acceptable.

The results of all other groups (prepared with different surface treatment techniques) will be presented with same order in following sections. After presenting the test results, all groups will be compared and discussed.

4.3 Chemical Treatment Results

The first and second slump results taken during the preparation of concrete for chemical surface treatment groups are presented in Table 4.8. The design slump was 65+/-5mm; the results were 65+/- 10mm.

Table 4.8—1st and 2nd Slump Values for Chemical Fiber Surface Treated Groups.

Group	Slump, mm	
	1st	2nd
Chromic Acid Sol., Type B (CAB)	185	65
Chromic Acid Sol., Type C (CAC)	150	55
Potassium Permanganate (PP)	185	75
Hydrogen Peroxide (HP)	155	65

4.3.1 Compressive Strength Results

The compressive strength results of chemical surface treated groups in addition to the results of plain and untreated fiber reinforced concrete brought from section 4.2.2.2 are presented in Table 4.9.

Table 4.9—Compressive Strength Test Results for Plain, Fiber Reinforced Concrete and Chemical Surface Treated Groups.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa (COV %)
PC	216	24.0	268	29.7 (0.5)
NT	196	21.7	239	26.6 (2.0)
CAB	198	22.0	250	27.7 (2.4)
CAC	203	22.5	256	28.4 (1.7)
PP	197	21.8	250	27.9 (2.2)
HP	201	22.3	249	27.6 (2.0)

It can be seen that the compressive strength results do not vary significantly with the various treatment techniques investigated. These results agree with the findings of Soroushian et al. (1992), section 2.4.1, that the peak stress capacity is similar regardless of fiber content.

4.3.2 Flexural Strength Results

The flexural strength results of chemical surface treated groups are presented in Table 4.10. Coefficient of Variations for all parameters is pointed out in the smaller font and could be compared to those values in last column which are acceptable standard ranges.

Table 4.10—Flexural Strength Test Results for Chemical Surface Treated Fiber Reinforced Concrete, 28 Days.

Parameters	CAB (COV %)	CAC (COV %)	PP (COV %)	HP (COV %)	Acceptable COV % (ASTM C 1609)
P_p, N	8,609 (3.5)	9,175 (4.4)	8,589 (2.2)	8,669 (1.0)	7.7
f_P, MPa	4.45 (3.5)	4.74 (4.4)	4.44 (2.2)	4.48 (1.0)	8.3
δ_P, mm	0.44 (1.3)	0.45 (6.5)	0.45 (4.9)	0.46 (3.3)	20.0
$P_{75,0.75}$, N	3,401 (3.2)	3,068 (7.5)	2,226 (9.4)	2,798 (4.5)	17.6
$P_{75,1.5}$, N	3,241 (3.2)	2,846 (9.1)	2,174 (10.2)	2,659 (1.9)	15.2
$P_{75,3.0}$, N	2,752	2,513	1,882	2,216	–
$f_{75,0.75}$, MPa	1.76 (3.1)	1.59 (5.6)	1.15 (9.3)	1.45 (4.6)	17.8
$f_{75,1.5}$, MPa	1.67 (3.3)	1.47 (9.1)	1.12 (10.1)	1.38 (1.8)	16.4
$f_{75,3.0}$, MPa	1.42	1.30	0.97	1.14	–
$T_{75,3.0}$, Joule	9.8 (0.6)	9.0 (7.4)	7.1 (8.0)	8.3 (1.8)	16.0

The results showed the peak load was not significantly changed by introduction of different chemical surface treatment techniques while residual strength at span / 75

($f_{75,3.0}$) was clearly affected by the surface treatment technique. Group CAB (treated by Chromic Acid Solution, Type B) gave the best end-point residual strength results among of all chemical surface treatment techniques; in contrast the PP group (treated by Potassium Permanganate) had a negative effect in the bonding strength of fibers.

4.3.3 Contact Angle Measurement

The contact angle was calculated from the results derived from 8 to 12 water droplet on the surface of fibers. Two pictures from right and left side of each droplet were taken (previously mentioned in section 3.7.3) meaning a total of 16 to 24 images were taken. Then the angle between the droplet and surface of the fibers were measured with aid of software available in the equipment used.

4.3.3.1 Calculation of Contact Angle Measurement (Θ_C)

The average of all contact angles was calculated as following which represented the contact angle of that specific surface treatment technique:

$$\overline{x} = \frac{1}{N}\sum_{i=1}^{N} x_i = \frac{x_1 + x_2 + \cdots + x_N}{N} \tag{4.8}$$

The detailed results of contact angle measurements for all fiber surface treated techniques are presented in Appendix B.

4.3.3.2 Contact Angle Measurement Results

The results of contact angle measurement for chemical surface treatment techniques are presented and compared to the untreated one are illustrated in Table 4.11.

Figure 4.11—Contact Angle Measurement of Untreated and Chemical Surface Treated of Fibers.

Group	Contact Angle, Degree
NT	94.01
CAB	69.37
CAC	69.14
PP	74.12
HP	85.16

It can be clearly seen that the contact angle is significantly affected by applying chemical surface treatment on fibers. The lowest values were obtained on those fibers treated by chromic acid solution either type B or C, concluding chromic acid is an effective solution used for the chemical treatment to improve the wettability of fibers. The contact angle of fibers treated by potassium permanganate was lower than those treated by hydrogen peroxide.

4.4 Physical Treatment Results

The first step for the physical surface treatment technique was to find the best duration of treatment. To do this, the contact angle measurement method was used. Initially fibers

were treated for several durations and then the contact angles were measured. From the results of contact angle, the best duration of exposure to the lamps was chosen. As previously concluded in section 4.3.1, there was not major change in the compressive strength by using different techniques of surface treatment. Therefore, the compressive strength was limited to the quality control of concrete (only 1 specimen per group).

4.4.1 UV

The procedures of UV surface treatment and specification of the UV lamp were previously mentioned in section 3.4.2. In the following sections, initially the duration of treatment will be determined by using the contact angle measurement method. Then slump, and compressive strength (quality control) results will be presented and finally the flexural strength test results will be discussed. All detailed results can be found in Appendix B.

4.4.1.1 Contact Angle Measurement Results

To find the best time of treatment by UV, the fibers were exposed to the UV lamp for 10, 30, 60, 90 and 120 minutes. The results are presented below (Table 4.12):

Figure 4.12—Contact Angle Values for UV Surface Treated Fibers.

Group	Contact Angle, Degree
UV-10	93.11
UV-30	92.29
UV-60	93.00
UV-90	92.89
UV-120	92.19

The results of contact angle measurement showed that the contact angle at 30 minutes was slightly lower, but after that point there was no significant change in the contact angle (Figure 4.3). The contact angles form 30 to 120 minutes treatments were little changed while the duration of treatment was one fourth. In comparison with no treatment, the contact angle decreased by less than 2 degrees for 30 minutes treatment. The 30 minutes UV surface treatment was chosen to examine. The results for UV surface treatment (UV-30) are presented below.

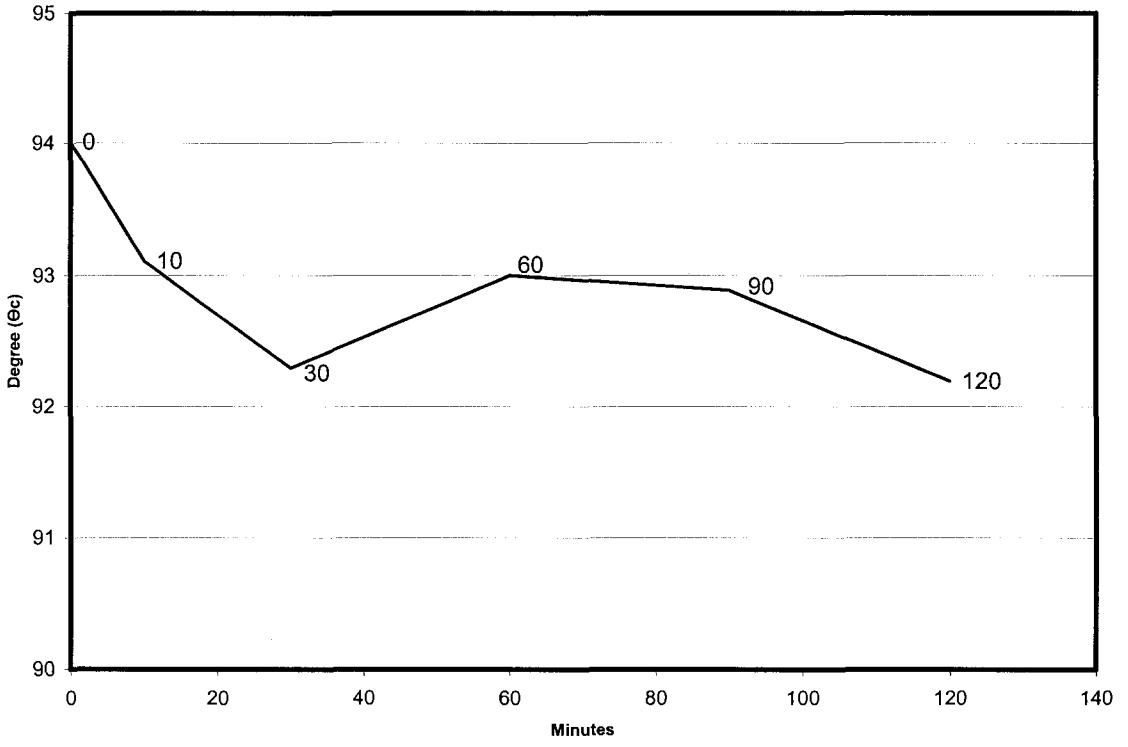


Figure 4.3—Contact Angle Changes versus Duration of UV Treatment.

4.4.1.2 Slump and Quality Control

The slump and compressive strength results for the quality control of fiber reinforced concrete with fiber treated by UV for 30 minutes are respectively shown in Tables 4.13, and 4.14.

Table 4.13—1st and 2nd Slump Values of UV Treated Group for 30 Minutes (UV-30).

Group	Slump, mm	
	1st	2nd
Treated by UV for 30 Minutes (UV-30)	165	55

Table 4.14—Quality control of UV Treated Group for 30 Minutes (UV-30), 28 Days.

Group	Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa
UV30	256	28.4

4.4.1.3 Flexural Strength Results

The results of flexural strength test and the group made by untreated fibers (NT) brought from section 4.2.3.2 are presented in Table 4.15.

Table 4.15—Flexural Strength Test Results of UV Treated Group for 30 Minutes (UV-30), and Non-Treated Group (NT), 28 Days.

Parameters	UV-30 (COV %)	NT (COV %)	Acceptable COV % (ASTM C 1609)
P_p, N	8,834 (5.7)	8,199 (1.3)	7.7
f_P, MPa	4.56 (5.7)	4.24 (1.3)	8.3
δ_P, mm	0.43 (3.5)	0.45 (4.4)	20.0
$P_{75,0.75}$, N	2,574 (9.8)	2,528 (4.5)	17.6
$P_{75,1.5}$, N	2,432 (8.7)	2,435 (4.9)	15.2
$P_{75,3.0}$, N	2,057	2,067	–
$f_{75,0.75}$, MPa	1.33 (10.1)	1.30 (4.5)	17.8
$f_{75,1.5}$, MPa	1.26 (8.8)	1.26 (4.8)	16.4
$f_{75,3.0}$, MPa	1.06	1.06	–
$T_{75,3.0}$, Joule	7.9 (6.0)	7.7 (4.7)	16.0

The results for 30 minutes UV surface treatment and their comparison to non treated one, showed no significant change in residual strength at span / 75 ($f_{75,3.0}$). It could be concluded that UV surface treatment was an ineffective technique to improve the bonding strength of fiber in fiber reinforced concrete. At this point, the investigation on bonding strength of UV treatment technique was suspended.

4.4.2 UV and Ozone (UVO_3)

The results for using only UV surface treatment showed there was not any improvement in the bonding strength between fibers and concrete. The experimental program was continued by study using combined UV and Ozone fiber surface treatment technique. In the same manner as the UV treatment technique, the duration of treatment will be determined followed by slump, compressive and flexural test results. The detailed results are presented in Appendix B.

4.4.2.1 Contact Angle Measurement Results

To find the best time of treatment by UV and Ozone the fibers were treated for 5, 10, 15, 20, 30, 40, 60 and 90 minutes. The results are presented in Table 4.16.

Figure 4.16— Contact Angle Values for UV and Ozone Surface Treated Fibers.

Group	Contact Angle, Degree
UVO_3-5	69.64
UVO_3-10	69.81
UVO_3-15	68.73
UVO_3-20	65.72
UVO_3-30	63.43
UVO_3-40	63.42
UVO_3-60	63.82
UVO_3-90	60.40

Figure 4.4 illustrates the contact angle change versus the treatment duration. The results of contact angle measurement showed that the contact angle for only using 5 minutes UV and Ozone surface treatment was significantly decreased. Then the values continued to drop in a slower manner up to 90 minutes treatment. To investigate on effectiveness of the UV and ozone technique, three durations of exposure were chosen: 5, 40 and 90 minutes.

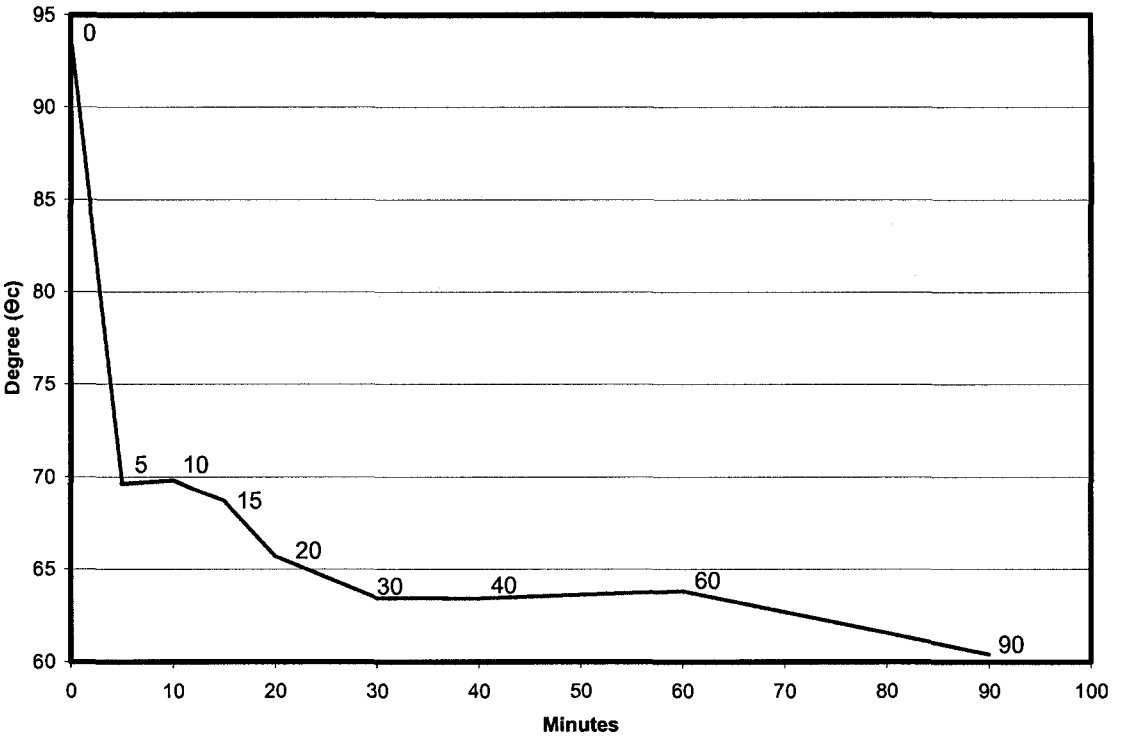


Figure 4.4—Contact Angle Changes versus Duration of UV and Ozone Treatment.

4.4.2.2 Slump and Quality Control

The slump and compressive strength results for the quality control of fiber reinforced concrete with fiber treated by UV and Ozone for 5, 40 and 90 minutes are respectively shown in Table 4.17, and 4.18.

Table 4.17—1st and 2nd Slump Values for Fiber Surface Treated by UV and Ozone.

Group	Slump, mm	
	1st	2nd
UV and Ozone for 5 Minutes (UVO_3-5)	255	165
UV and Ozone for 40 Minutes (UVO_3-40)	205	115
UV and Ozone for 90 Minutes (UVO_3-90)	195	85

Table 4.18—Quality control of UV and Ozone Treated Groups, 28 Days.

Group	Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa
UVO_3-5	216	23.9
UVO_3-40	219	24.3
UVO_3-90	218	24.2

The compressive strength results for the quality control of UV and Ozone treated groups were dramatically lower than those results for chemical surface treated techniques (about 28 MPa); meanwhile the slumps was much higher (about 100 mm).

4.4.2.3 Flexural Strength Results

The results of flexural strength test of UV and Ozone groups for various treatment durations are presented in Table 4.19.

Table 4.19—Flexural Strength Test Results of UV and Ozone Treated Groups, 28 Days.

Parameters	UVO_3-5 (COV %)	UVO_3-40 (COV %)	UVO_3-90 (COV %)	Acceptable COV % (ASTM C 1609)
P_p, N	8,097 (5.0)	8,076 (1.9)	8,086 (3.9)	7.7
f_P, MPa	4.18 (5.0)	4.17 (2.0)	4.18 (3.8)	8.3
δ_P, mm	0.47 (5.1)	0.47 (8.7)	0.45 (5.5)	20.0
$P_{75,0.75}$, N	2,591 (8.7)	2,910 (13.7)	2,207 (4.3)	17.6
$P_{75,1.5}$, N	2,301 (5.6)	2,586 (11.9)	2,038 (3.5)	15.2
$P_{75,3.0}$, N	1,939	2,203	1,763	–
$f_{75,0.75}$, MPa	1.34 (8.8)	1.50 (13.9)	1.14 (4.2)	17.8
$f_{75,1.5}$, MPa	1.19 (5.6)	1.34 (12.2)	1.05 (3.8)	16.4
$f_{75,3.0}$, MPa	1.00	1.14	0.91	–
$T_{75,3.0}$, Joule	7.4 (4.4)	8.2 (8.5)	6.7 (3.5)	16.0

The compressive strength tests taken for the quality control of concrete for UVO_3-5, UVO_3-40 and UVO_3-90 groups were about 24 MPa which were much lower than the other previous group compressive strength results. Also the slump results showed much higher than before.

The flexural strength results showed the best bonding strength was for UVO_3-40 (40 min. treatment) group, however for UVO_3-90 (90 min. treatment) adversely affected the bonding strength of fibers. This could be because of curved shape of fibers after 90 minutes treatment. The UV and Ozone treatment generator not only generates UV and Ozone, but also generates heat. That heat might cause the deformation or degradation of fibers after 90 minutes. Figure 4.6 illustrates the shape of fibers after 90 minutes

treatment; however for lower durations of treatment relatively no deformation of fibers was apparent (Figure 4.5).

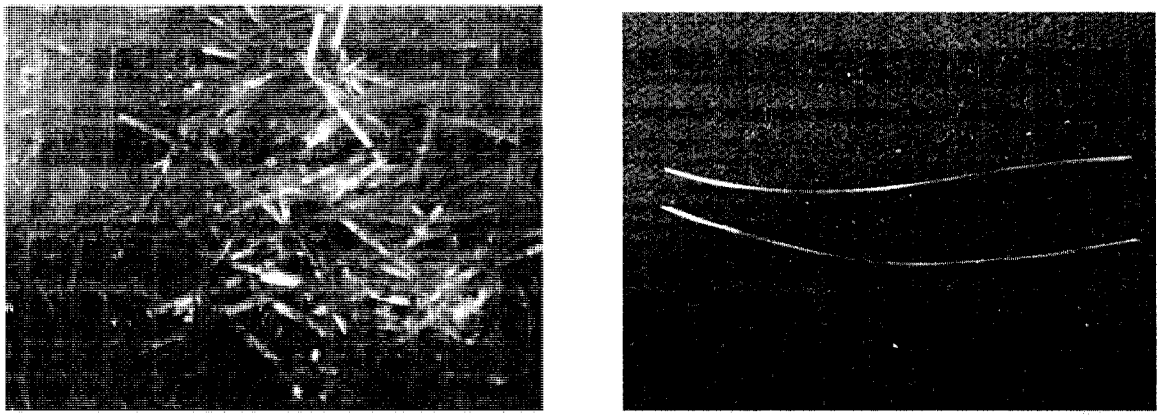


Figure 4.5—Images from Fibers Treated for 5 Minutes UV and Ozone.

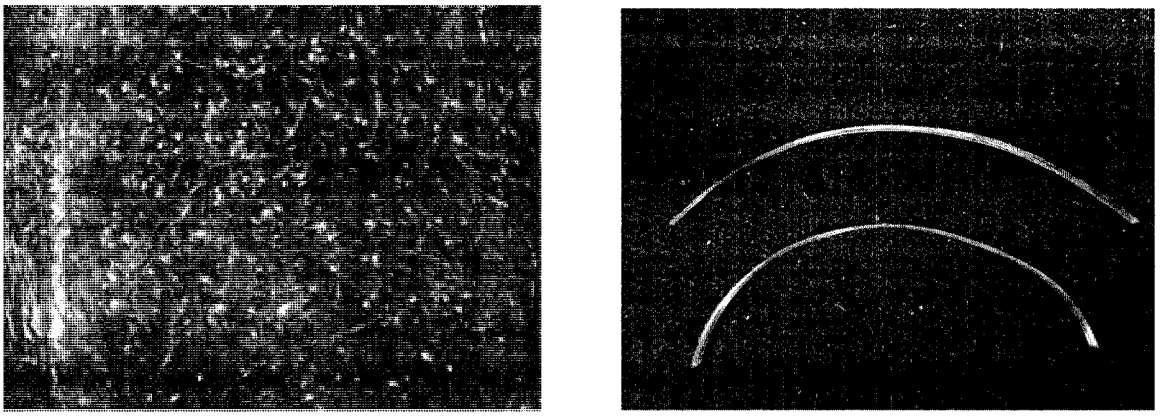


Figure 4.6—Images from Fibers Treated for 90 Minutes UV and Ozone.

Because of unacceptable compressive strength results for UVO_3-5, UVO_3-40 and UVO_3-90 groups the investigation on bonding strength for UV and Ozone treatment technique was repeated with a new batch of cement. Since the 90 minutes UV and Ozone affected the bonding the fibers negatively (from the results of previous section), the duration of treatment for performing the tests were modified to 5, 10, and 40 minutes. To make sure that the new cement had the same chemical properties as that cement that

previously used a set of samples without surface treatment of fibers were prepared and again tested. The results are presented in Tables from 4.20 to 4.22.

Table 4.20—Comparison of 1st and 2nd Slump Values between Previous Non-Treated Group and Repeated One.

Group	Slump, mm	
	1st	2nd
Non-Treated, Middle Dosage (NT)	165	65
Non-Treated, Middle Dosage (NT, Repeated)	150	55

Table 4.21— Comparison of Quality Control between Previous Non-Treated Group and Repeated One, 28 Days.

Group	Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa
NT	239	26.6
NT (R)	257	27.5

Table 4.22—Comparison of Flexural Strength Test Results between Previous Non-Treated Group and Repeated One, 28 Days.

Parameters	NT-R (COV %)	NT (COV %)	Acceptable COV % (ASTM C 1609)
P_p, N	8,551 (1.9)	8,199 (1.3)	7.7
f_P, MPa	4.42 (1.9)	4.24 (1.3)	8.3
δ_P, mm	0.46 (1.3)	0.45 (4.4)	20.0
$P_{75,0.75}$, N	2,575 (3.5)	2,528 (4.5)	17.6
$P_{75,1.5}$, N	2,443 (3.9)	2,435 (4.9)	15.2
$P_{75,3.0}$, N	2,065	2,067	–
$f_{75,0.75}$, MPa	1.33 (3.3)	1.30 (4.5)	17.8
$f_{75,1.5}$, MPa	1.26 (4.0)	1.26 (4.8)	16.4
$f_{75,3.0}$, MPa	1.07	1.06	–
$T_{75,3.0}$, Joule	7.8 (3.2)	7.7 (4.7)	16.0

Table 4.22 illustrates that new results are almost the same as those previously taken. So the experimental program was continued with using the new cement batch.

4.4.2.4 Slump and Quality Control for Repeated Experiments

The repeated slump and compressive strength results for the quality control of fiber reinforced concrete with fibers treated by UV and Ozone for 5 and 40 minutes, and for the new treatment duration of 10 minutes are presented in Tables 4.23 and 4.24.

Table 4.23—1st and 2nd Slump Values for Repeated 5 and 40 and New 10 Minutes UV and Ozone Treatment.

Group	Slump, mm	
	1st	2nd
Treated by UV and Ozone for 5 Minutes (UVO_3-5-R)	145	45
Treated by UV and Ozone for 10 Minutes (UVO_3-10)	150	50
Treated by UV and Ozone for 40 Minutes (UVO_3-40-R)	160	50

Table 4.24—Quality Control for Repeated 5 and 40 and New 10 Minutes UV and Ozone Treatment, 28 Days.

Group	Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa
UVO_3-5-R	255	28.2
UVO_3-10	254	28.1
UVO_3-40-R	267	29.6

4.4.2.5 Flexural Strength Results for Repeated Experiments

The results of flexural strength test for Repeated 5 and 40 and New 10 minutes UV and Ozone treatment are presented in Table 4.25.

Table 4.25—Flexural Strength Test Results for Repeated 5 and 40 and New 10 Minutes UV and Ozone Treatment, 28 Days.

Parameters	UVO_3-5-R (COV %)	UVO_3-10 (COV %)	UVO_3-40-R (COV %)	Acceptable COV % (ASTM C 1609)
P_p, N	8,816 (1.5)	9,109 (6.2)	8,589 (4.5)	7.7
f_P, MPa	4.55 (1.5)	4.71 (6.2)	4.44 (4.6)	8.3
δ_P, mm	0.50 (1.2)	0.50 (9.2)	0.52 (8.6)	20.0
$P_{75,0.75}$, N	2,587 (7.4)	2,552 (5.0)	2,370 (12.3)	17.6
$P_{75,1.5}$, N	2,409 (7.3)	2,464 (8.1)	2,352 (13.1)	15.2
$P_{75,3.0}$, N	2,066	2,183	2,142	—
$f_{75,0.75}$, MPa	1.34 (7.3)	1.32 (4.9)	1.23 (12.2)	17.8
$f_{75,1.5}$, MPa	1.24 (7.2)	1.27 (8.1)	1.22 (13.4)	16.4
$f_{75,3.0}$, MPa	1.07	1.13	1.11	—
$T_{75,3.0}$, Joule	7.8 (5.9)	8.0 (5.6)	7.5 (9.5)	16.0

The flexural strength results showed fiber reinforced concrete made with 10 minutes UV and Ozone fiber treatment had the highest bonding strength among of all physical surface treatment techniques. The 40 minutes UV and Ozone treated group gave lower residual strength at span / 75 ($f_{75,3.0}$) than the 10 minutes treated group, while the contact angle of fibers used for this group was also much lower. Also the coefficient of various (COV) values for 40 minutes treatment was relatively higher than two other groups. This

could be due to the moderate deformation of fibers due to the heat generated by the UV lamp; which might affect the area of bonding between the fibers and concrete.

4.5 High Dosage of Fibers Results

Table 4.26 compares all results came from the previous sections. It can be seen that the chemical surface treatment using chromic acid solution, Type B was the most effective technique in improvement of bonding strength between the fibers and concrete. Group CAB was chosen to investigate fiber reinforced concrete made with higher fiber dosage (0.50 %).

Table 4.26—Comparison of Flexural Test Results for All Surface Treatment Techniques.

Group	$P_{p,\ N}$	$f_{P,\ MPa}$	$P_{75,0.75,\ N}$	$P_{75,1.5,\ N}$	$P_{75,\ 3.0,\ N}$	$f_{75,0.75,\ MPa}$	$f_{75,1.5,\ MPa}$	$f_{75,3.0,\ MPa}$
NT	8,199	4.24	2,528	2,435	2,067	1.30	1.26	1.06
CAB	8,609	4.45	3,401	3,241	2,752	1.76	1.67	1.42
CAC	9,175	4.74	3,068	2,846	2,513	1.59	1.47	1.30
PP	8,589	4.44	2,226	2,174	1,882	1.15	1.12	0.97
HP	8,669	4.48	2,798	2,659	2,216	1.45	1.38	1.14
UV-30	8,834	4.56	2,574	2,432	2,057	1.33	1.26	1.06
UVO_3-5	8,816	4.55	2,587	2,409	2,066	1.34	1.24	1.07
UVO_3-10	9,109	4.71	2,552	2,464	2,183	1.32	1.27	1.13
UVO_3-40	8,589	4.44	2,370	2,352	2,142	1.23	1.22	1.11

Two set of samples were prepared with the higher dosage of untreated and chromic acid Solution B treated fibers. The results are presented in the following sections.

4.5.1 Slump and Quality Control

The slump and compressive strength results for the quality control of fiber reinforced concrete prepared with the higher fiber dosage are presented in Tables 4.27, and 4.28.

Table 4.27—1st and 2nd Slump Values for Non-Treated, High Dosage and Chromic Acid Solution, Type B Treated, High Dosage.

Group	Slump, mm	
	1st	2nd
Non-Treated, High Dosage (NT, HD)	180	35
Treated by Chromic Acid Sol., Type B (CAB, HD)	175	25

Table 4.28—Quality Control for Non-Treated, High Dosage and Chromic Acid Solution, Type B Treated, High Dosage, 28 Days.

Group	Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa
NT (HD)	236	26.1
CAB (HD)	239	26.5

It can be clearly seen that the slump for high dosage groups are lower than middle dosage ones (Table 4.9). These results agree with the findings of Pieffer and Soukatchoff (1994), previously mentioned in section 2.3.4., the increased volume of fibers results in decreased workability. Also, the compressive strength for high dosage groups was slightly lower than middle range one that agrees with the finding of Soroushian et al. (1992), section 2.4.1.

4.5.2 Flexural Strength Results for High Dosage of Fibers

The results of flexural strength tests for mixtures containing high dosages of fibers are presented in Table 4.29.

Table 4.29—Flexural Strength Test Results for Non-Treated, High Dosage and Chromic Acid Solution Type B Treated, High Dosage, 28 Days.

Parameters	NT (HD) (COV %)	CAB (HD) (COV %)	Acceptable COV % (ASTM C 1609)
P_p, N	8,428 (1.2)	9,220 (3.8)	7.7
f_P, MPa	4.35 (1.2)	4.76 (3.9)	8.3
δ_P, mm	0.46 (6.7)	0.52 (4.5)	20.0
$P_{75,0.75}$, N	4,059 (4.6)	3,990 (4.5)	17.6
$P_{75,1.5}$, N	3,987 (5.1)	3,863 (4.3)	15.2
$P_{75,3.0}$, N	3,298	3,544	—
$f_{75,0.75}$, MPa	2.10 (4.5)	2.06 (4.5)	17.8
$f_{75,1.5}$, MPa	2.06 (5.1)	2.00 (4.3)	16.4
$f_{75,3.0}$, MPa	1.70	1.83	—
$T_{75,3.0}$, Joule	11.5 (3.7)	11.4 (3.9)	16.0

The results of flexural strength test for high dosage fibers showed the residual strength of the non treated mixture after the first crack at span / 300 and span / 150 ($f_{75,0.75}$ and $f_{75,1.5}$) was higher than the chromic acid solution treated while the rate of loss of the residual strength was much higher. This phenomenon was named "Gradient of Decreasing Residual Strength" which will be introduced and discussed in section 4.6.6. As a results, the residual strength of the untreated group at the end-point (span / 75, $f_{75,3.0}$) was lower than that for chromic acid solution treated group.

4.6 Comparison of Groups' Results

4.6.1 Compressive Strength

In this section, the compressive strength results brought from different groups is compared and discussed. Figures 4.7 and 4.8 compare the compressive strength from samples made by plain concrete and those which were prepared with fibers. The graphs derived from the load-displacement showed little differences in maximum compressive strength but higher in post crack strength of specimens with fibers. This could be concluded from the graphs that plain concrete had no post crack compressive strength after the failure occurs due to the brittle characteristic of concrete, but for the FRC the specimen still had compressive strength even up to 9 mm displacement of the platens.

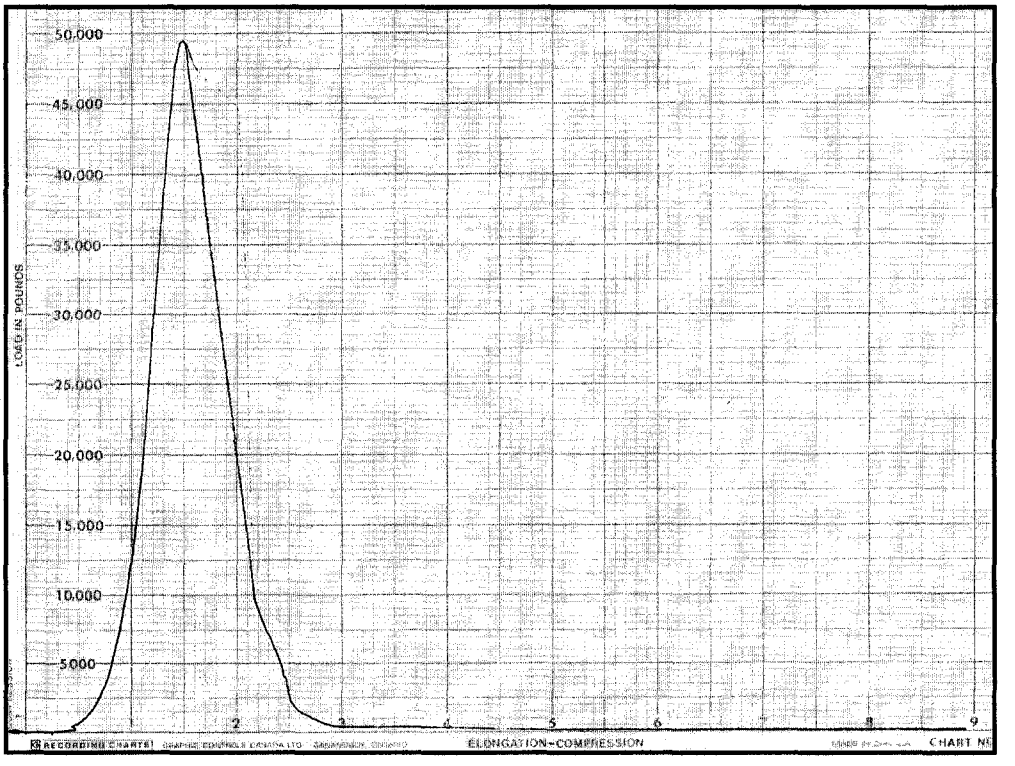


Figure 4.7—Typical Load-Deflection Curve for Compressive Strength of Plain Concrete.

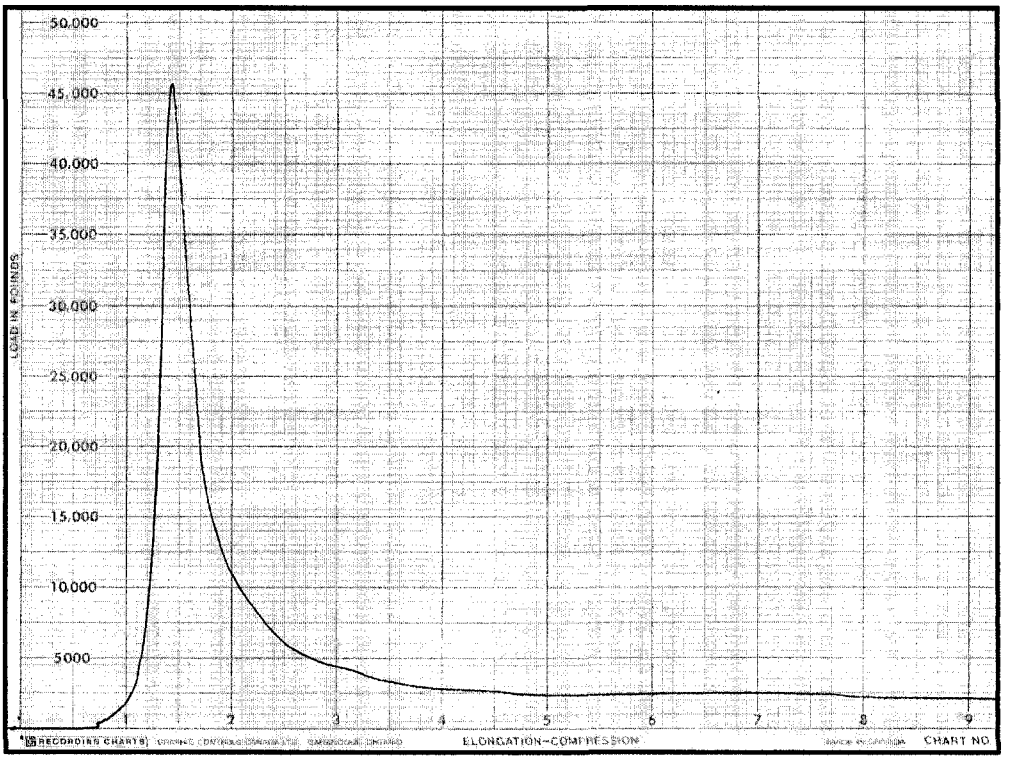


Figure 4.8— Typical Load-Deflection Curve for Compressive Strength of Fiber Reinforced Concrete.

Figure 4.9 compares the average compressive strength of different groups for 7 and 28 days age. It can be clearly seen that the compressive strength of concrete decreased by introduction of fibers to concrete. Moreover, there was very little change in compressive strength by using different fiber surface treatment techniques.

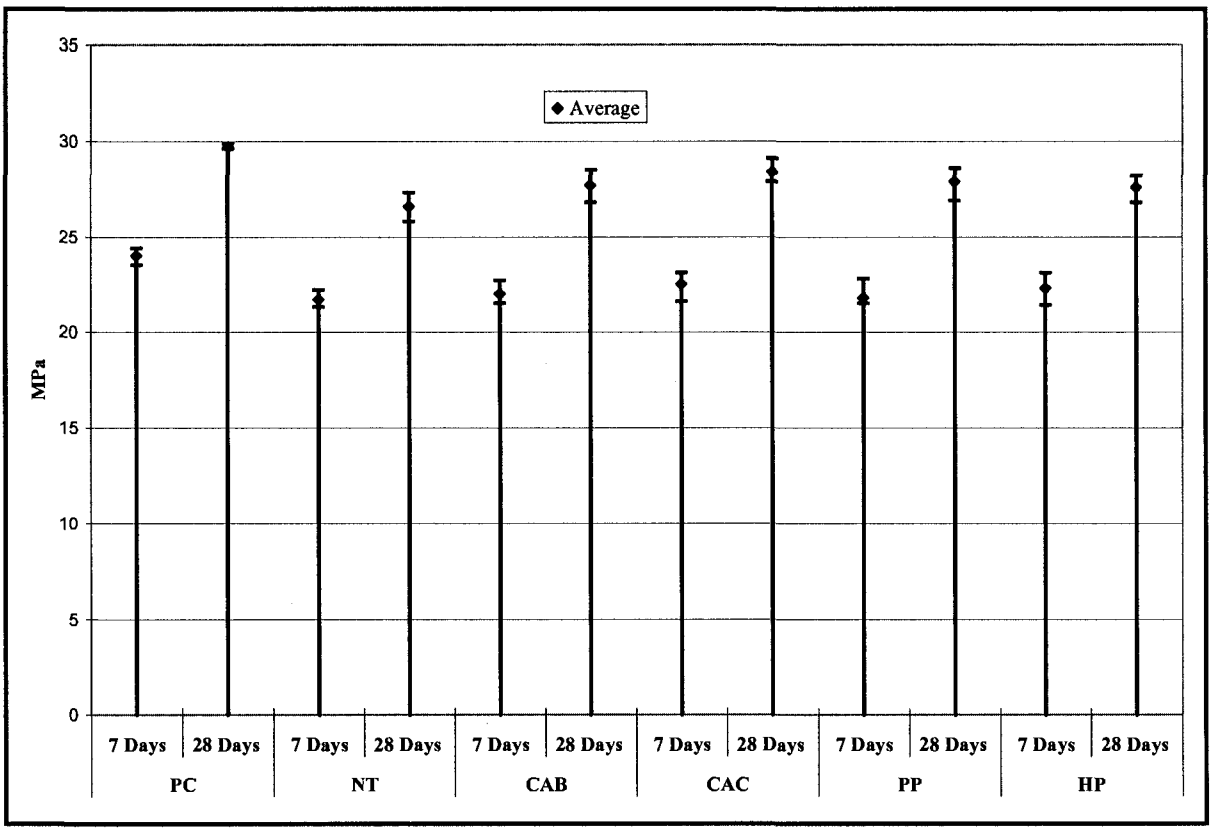


Figure 4.9—Comparison of Compressive Strength of Plain Concrete and Chemical Fiber Surface Treated Groups, for 7 and 28 Days.

4.6.2 Flexural Strength

The average peak load of the flexural tests for all groups is presented in Figure 4.10. The figure shows the peak load changes slightly by using different fibers surface treatment techniques, but the highest peak load was observed for the plain concrete. As a conclusion, there is no improvement in peak load by using surface treatment techniques. The pick load for high dosage group were slightly higher than middle dosage ones.

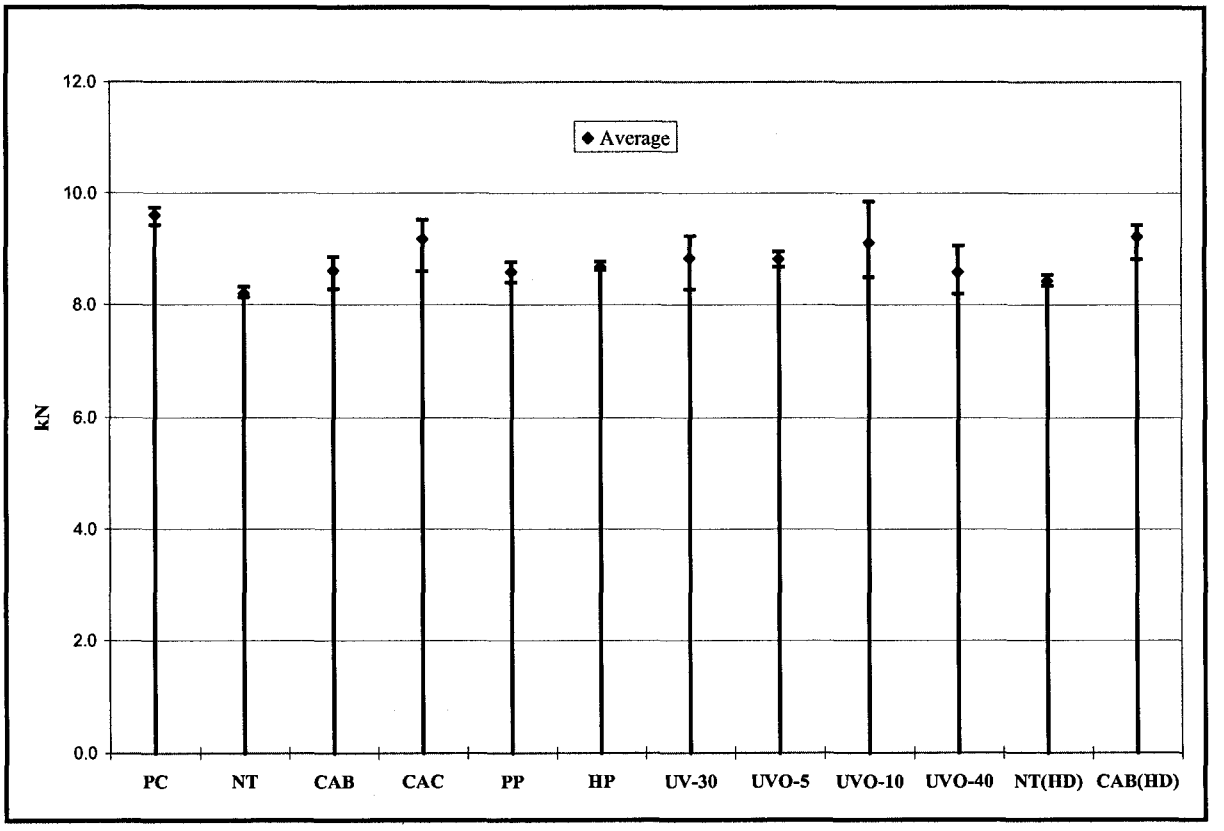


Figure 4.10—Comparison of Peak Load of Flexural Strength for 28 Days.

4.6.3 Residual Strength at Span / 75 ($f_{75,3.0}$)

Figure 4.11 illustrates the residual strength at span / 75 ($f_{75,3.0}$, the last point that the flexural strength was recorded) for all groups used in this work. As it was expected, plain concrete does not have any post crack strength. The results show significant change in residual strength at L / 75 by using different fiber surface treatment techniques. In all cases, the residual strength was improved from slight to considerable except for group PP (fiber surface treated by Potassium Permanganate) which had negative effect. The highest residual strength is for group CAB (HD), high dosage fiber surface treated with Chromic Acid Solution Type B) concludes the amount of fibers added to concrete has greater effect on improvement of residual strength than using fiber surface treatment techniques.

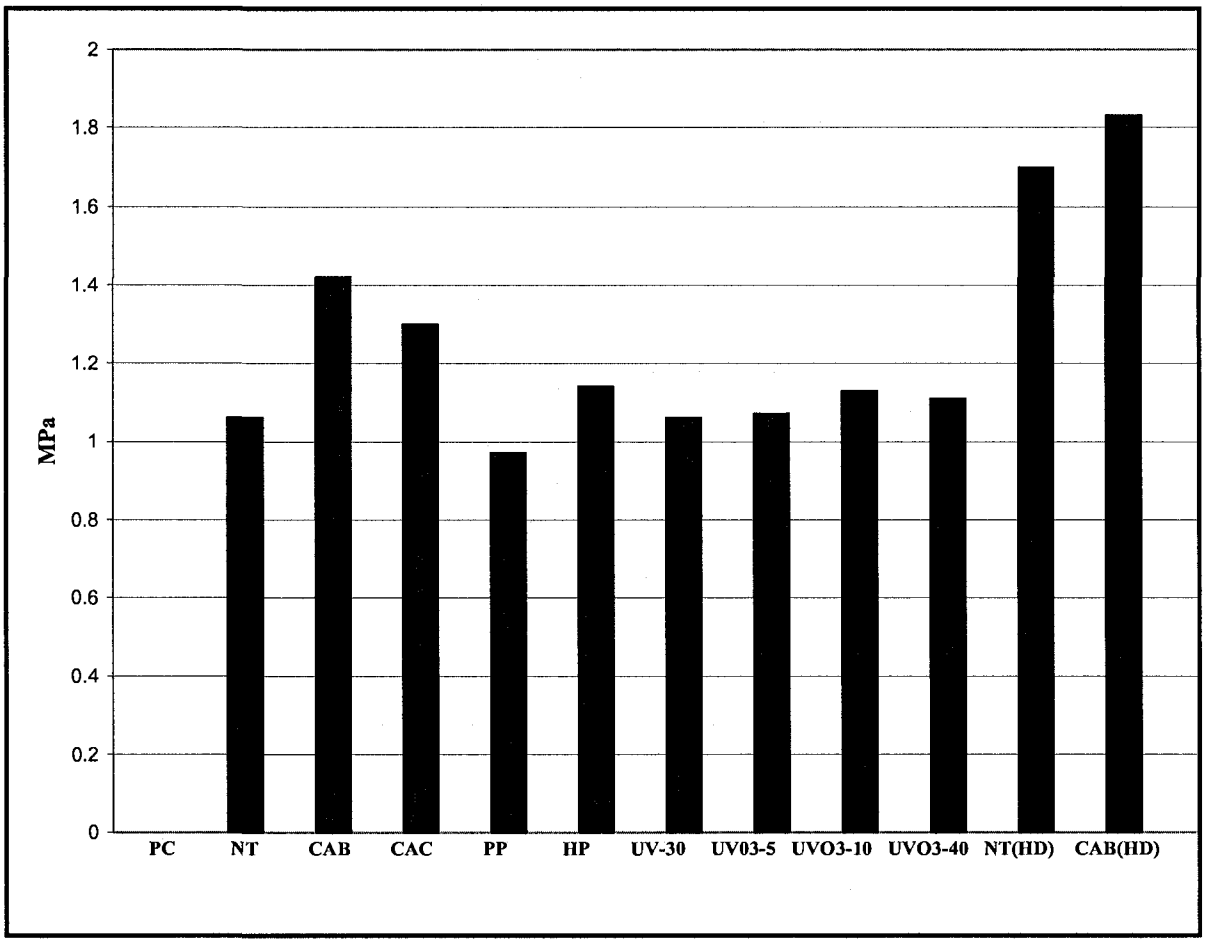


Figure 4.11—Comparison of Residual Strength at Span / 75 ($f_{75,3.0}$), for 28 Days.

4.6.4 Contact Angle

The contact angles for all different groups are compared in Figure 4.12. It can be seen that all fiber surface treatment techniques improved the contact angle between the surface of fibers and water droplet except for group UV-30 (30 min. UV fiber surface treatment) which there was almost no change in the contact angle. It can be concluded that UV surface treatment (without ozone) has no effect on the contact angle of STRUX® 90/40

fibers. The highest improvement for contact angles belongs to UVO_3-40 group (40 min. UV and Ozone fiber surface treatment), however the deformation of fibers because of the heat generation of the UV lamp, as previously mentioned in section 4.4.2.5, gave lower residual strength at span / 75 than UVO_3-10 group (Figure 4.10).

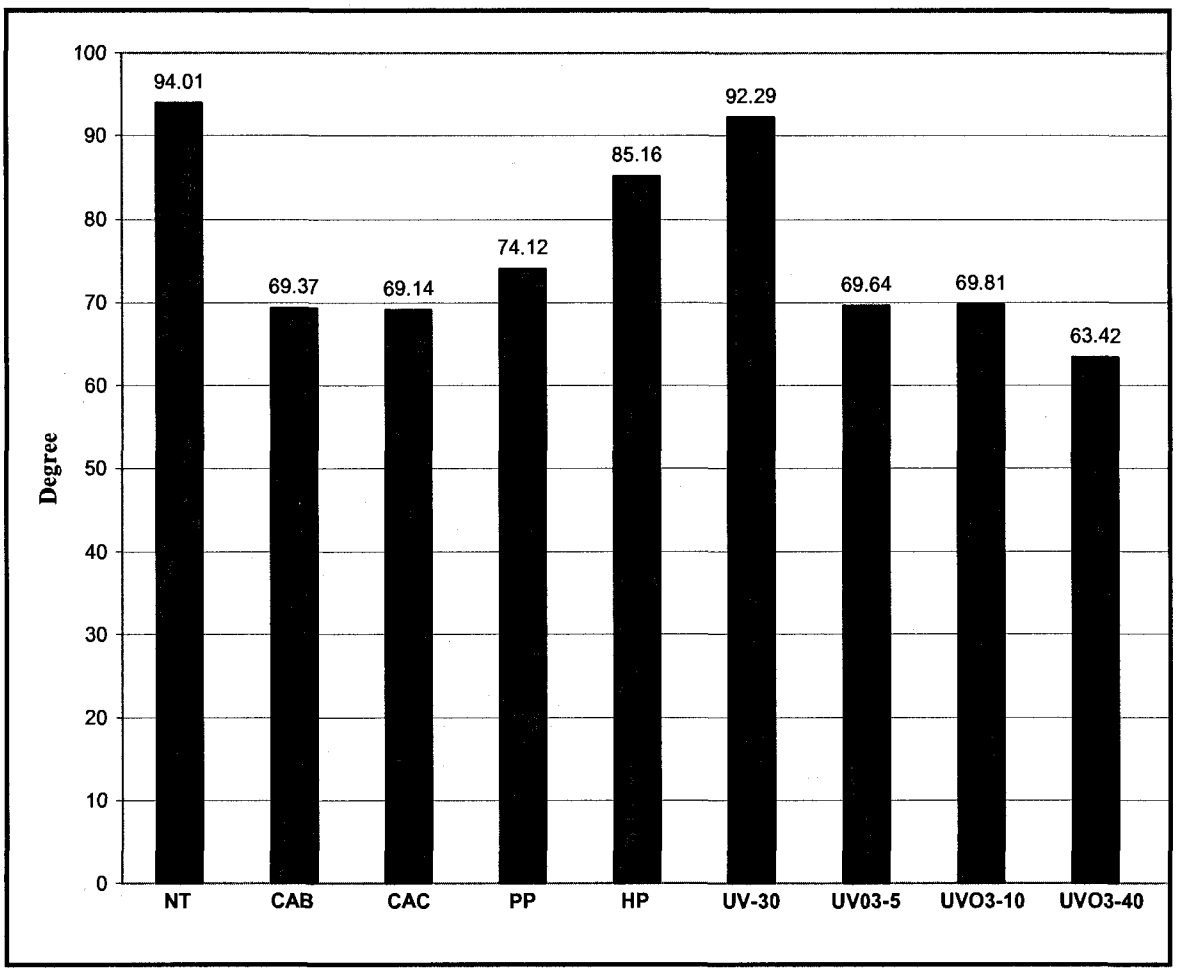


Figure 4.12—Comparison of Contact Angle for Different Surface Treatment Techniques.

4.6.5 Toughness ($T_{75,3.0}$)

The comparison of the toughness ($T_{75,3.0}$) for different fiber surface treatment techniques (Figure 4.13) shows the group which was prepared with the high dosage of fibers has greater toughness than middle dosage ones. For the physical fiber surface treated groups, there was very little change in toughness, but for the chemical fiber surface treated group, the differences are much more apparent in the same order of their residual strength.

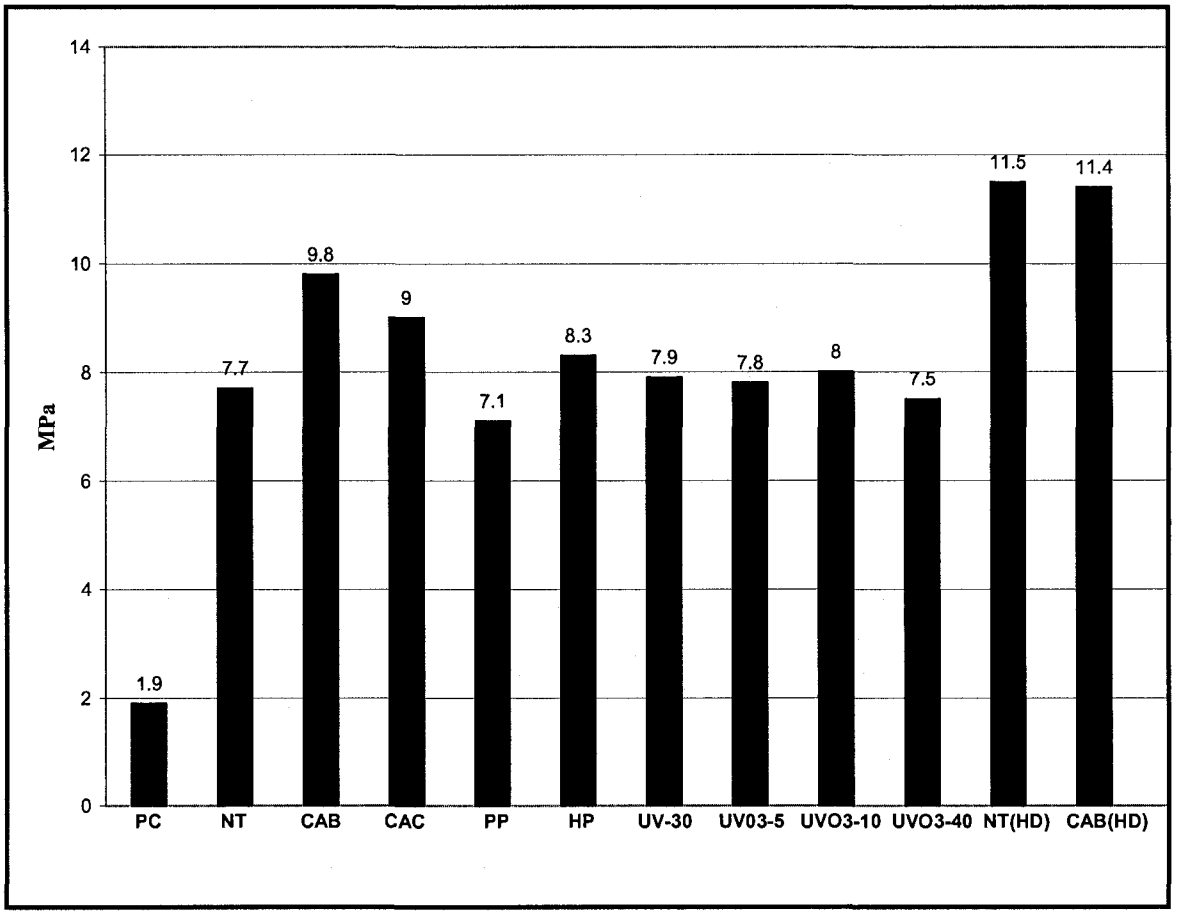


Figure 4.13—Comparison of Toughness ($T_{75,3.0}$) for Different Groups.

4.6.6 Gradient of Decreasing Residual Strength

In this work, a new method of investigation on of the effectiveness of each fiber surface treatment technique was presented which has been named "Gradient of Decreasing Residual Strength". Fiber reinforced concrete is a non homogeneous material in which fibers are randomly distributed within the concrete matrix. Since the number of fibers per unit area of concrete is unknown, the number of fibers in the failure area of one specimen might be completely different from another specimen, causing totally different flexural strength results. Gradient of Decreasing Residual Strength could be defined as the capability of FRC to maintain the residual strength after the first crack occurs. The lower the degree shows the higher capability of remaining residual strength meaning higher bonding strength. Gradient of Decreasing Residual Strength was calculated from the load deflection curve from a point after the first crack (L / 300) to the end of curve (L / 75). Figure 4.14 illustrates the points that Gradient of Decreasing Residual Strength is calculated.

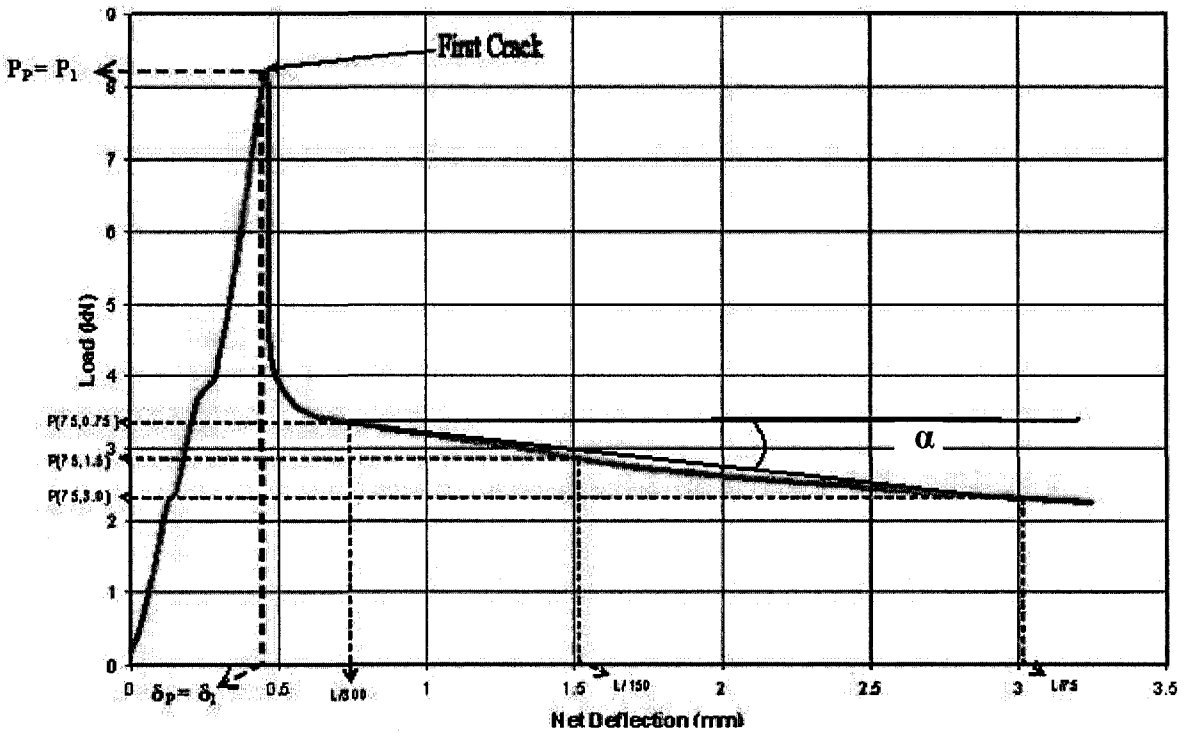


Figure 4.14— Gradient of Decreasing Residual Strength (α) From the Load-Deflection Curve.

Figure 4.15 compares the Gradient of Decreasing Residual Strength for different fiber surface treatment techniques. The gradient for UV and Ozone fiber surface treatment technique is decreased by increasing the duration of treatment which illustrates the rate of decrease in the residual strength in UVO_3 surface treatment technique is directly related to the duration of treatment. The lowest gradient among of chemical surface treated techniques belongs to group PP which had lowest residual strength (even lower than untreated ones). The reason could be that the flexural strength of this group was already low that the deflection does not have that much effect on the rate of loss of the residual strength. Generally, for the chemical surface treatment techniques, it can be seen the groups which have higher residual and toughness than the non-treated group, the rate of loss of the residual strength is higher.

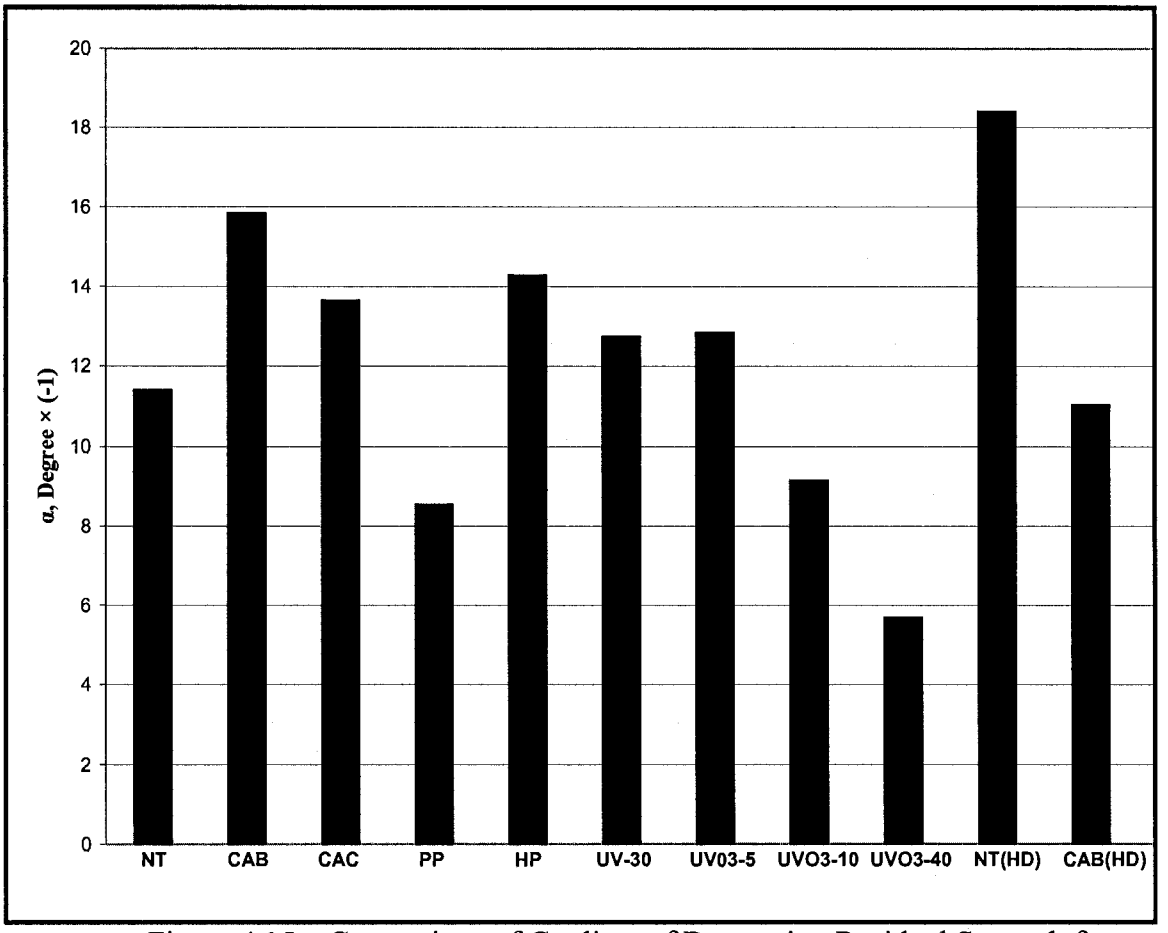


Figure 4.15—Comparison of Gradient of Decreasing Residual Strength for Different Groups.

The greatest result that could be concluded from this method of evaluation is that the bonding strength appeared in the differences between the NT and CAB, high dosage groups. These groups have almost the same toughness, but it can be seen that the CAB (HD) group (treated with Chromic Acid Solution, Type B) has much lower gradient than the non-treated one, while has relatively high residual strength. This proves the effectiveness of the surface treatment technique; although they have the same toughness.

4.6.7 Toughness ($T_{75,3.0}$) versus Contact Angle

The toughness versus contact angle is illustrated in Figure 4.16. R-Squared line of the graph is shown to evaluate the effectiveness of fiber surface treatment techniques on the toughness of fiber reinforced concrete. The UVO_3-40 group's result was eliminated due to the physical deformation of fibers which had effect on the flexural performance.

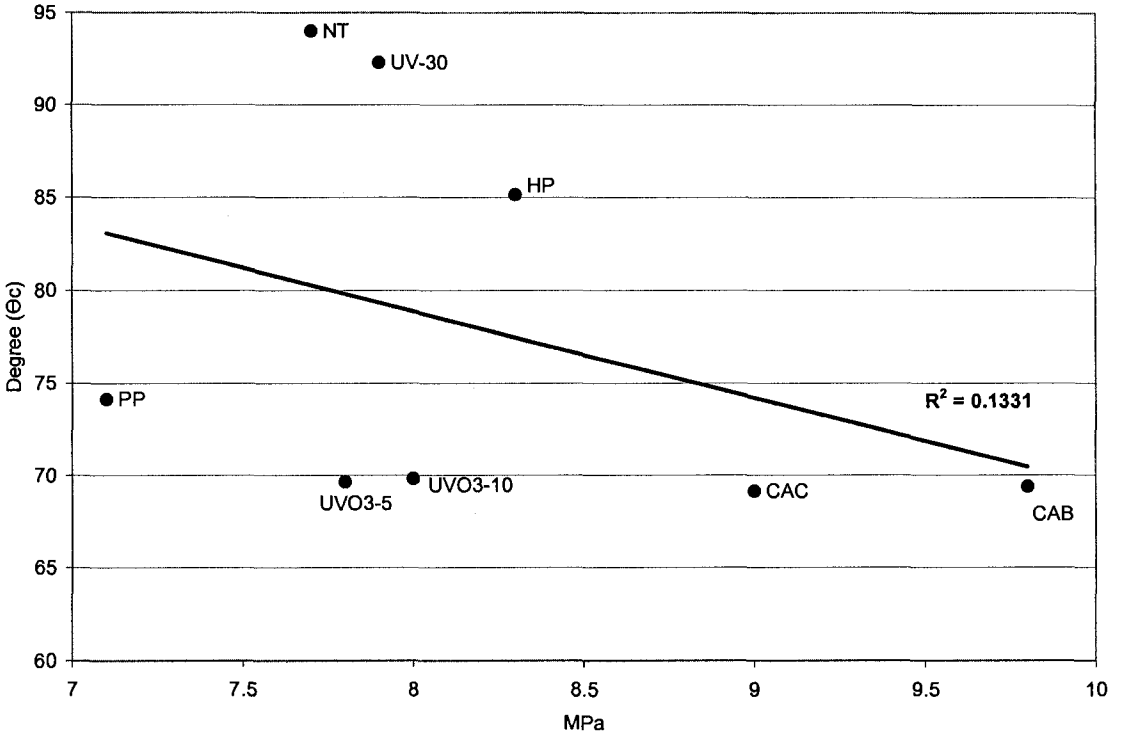


Figure 4.16— R-Squared of Comparison between Toughness versus Contact Angle (Θ_C).

It can be clearly seen that the toughness of fiber reinforced concrete prepared by different surface treated fibers was increased by decreasing the contact angle of fibers. The R-Squared line shows a value of 0.1331 which indicates insignificant relationship between the contact angle and toughness of the groups.

4.6.8 Comparison of Residual Strength at Span / 75 ($f_{75,3.0}$) versus Contact Angle

Figure 4.17 illustrates the R-Squared line of the results of residual strength at span / 75 ($f_{75,3.0}$) versus contact angle of fibers for all groups

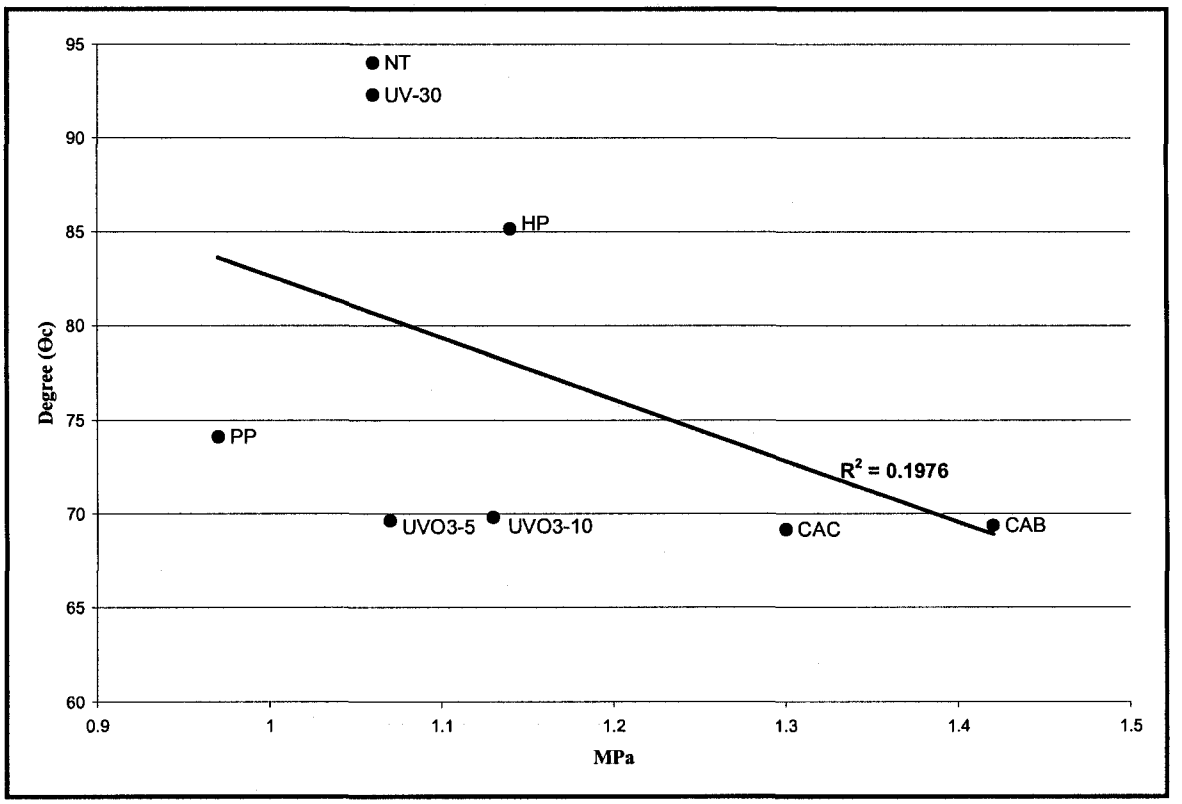


Figure 4.17— R-Squared Line of Comparison between Residual Strength at Span / 75 ($f_{75,3.0}$) versus Contact Angle (Θ_C).

The results of residual strength at span / 75 ($f_{75,3.0}$) was increased by decreasing of fiber's contact angle. Figures 4.15 and 4.16 prove the effectiveness of fiber surface treatment fibers by improvement in wettability of fibers. The R-Squared line shows a value of 0.1976 which indicates poor relationship between the contact angle and end-point residual strength ($f_{75,3.0}$), but higher than the relationship between contact angle and toughness.

The contact angle results show no relation between the residual strength of FRC and contact angle of the chemical surface treatment techniques since the contact angle for CAB and CAC groups are almost the same, but CAC group has lower residual strength than CAB group. Also, HP group has higher residual strength than PP group while has higher contact angle.

4.6.9 Comparison of Toughness ($T_{75,3.0}$) and Residual Strength at Span / 75 ($f_{75,3.0}$)

The Toughness ($T_{75,3.0}$) and Residual Strength at Span / 75 ($f_{75,3.0}$) of all groups as a proportion of the non-treated toughness and residual strength group in percent are presented in Figure 4.18.

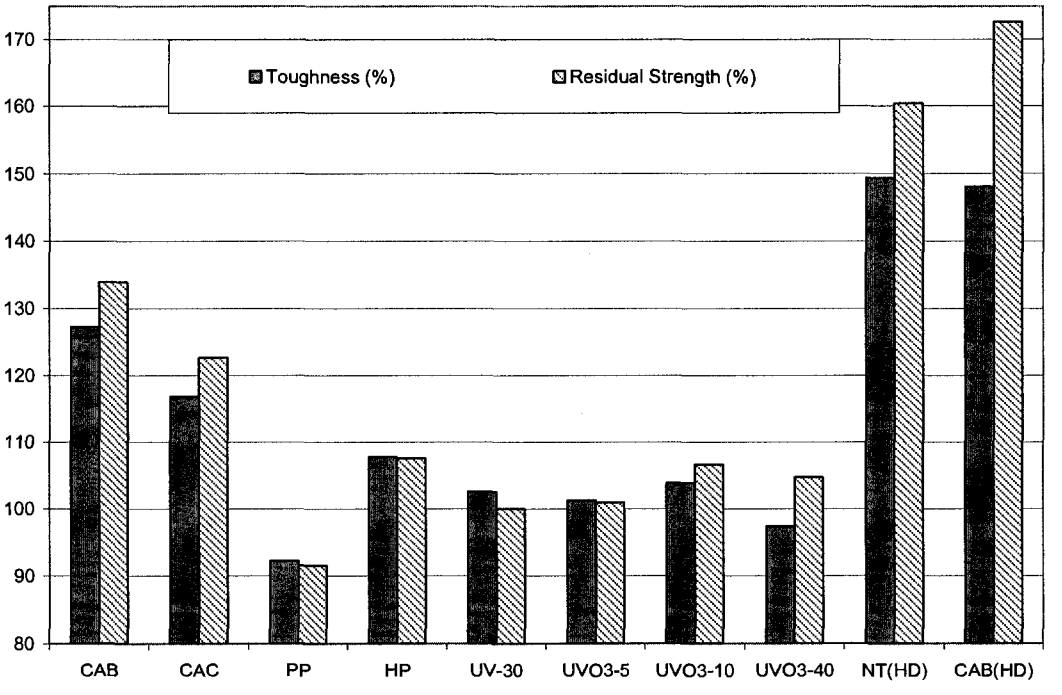


Figure 4.18— Comparison of Toughness ($T_{75,3.0}$) and Residual Strength at Span / 75 ($f_{75,3.0}$) as a Proportion of Non-Treated Toughness and Residual Strength Group in Percent .

It can be clearly seen that the ratio of end-point residual of treated groups/end-point residual of non-treated group shows higher values than those values of toughness except for UV-30 and UVO_3-5 which had almost the same results and PP which had lower flexural strength than non treated one.

The experimental results showed the measure changes in toughness values of fiber reinforced concrete were mostly depend on the Peak-Load strength. It was previously concluded in section 4.6.2 that the Peak-Load strength changes were not dependent on the technique of surface treatment. Since the main purpose of this work was the investigation on bonding strength, it could be concluded from sections 4.6.6 and 4.6.9 that considering the residual strength at the end-point is more reasonable rather than considering toughness strength of fiber reinforce concrete for evaluation the bonding strength.

Chapter 5

Summary, Conclusions, and Recommendations

5.1 Introduction

The major emphasis of the experimental program presented in this project was to investigate and examine the behavior of Fiber Reinforced Concrete (FRC) focusing on the bond characteristics between copolymer polypropylene/polyethylene (STRUX® 90/40) fibers and concrete by applying several fiber surface treatment techniques.

Summary and conclusions of the thesis are presented in this chapter. Recommendations are also given for further research in the area of the fiber surface treatment techniques and bonding between fiber and concrete of FRC.

5.2 Summary

The main objective of this research was to investigate the bonding behavior of FRC and partially the effectiveness of using various fiber surface treatment techniques on the bonding improvement. The focus of this experimental program was on the use of polyethylene/polypropylene fiber blend reinforcement which could be added to concrete as replacement for welded wire fabric, steel fibers, light temperature cracking rebars and other secondary reinforcement in slab-on-ground flooring and thin-walled non-structural precast applications.

The problem with using the organic polymeric fibers with inorganic concrete is their poor adhesion due to the ordered chemical structure and lack of polar groups on the fibers' surface. As a result, the separation of fiber and cement matrices occurs before reaching the potential tensile strength of the fibers. Many efforts were undertaken in this project to improve the bonding between fibers and concrete by using different surface treatment techniques of fibers. These fiber surface treatment techniques were mainly divided to two groups: Chemical and Physical.

Previous investigations introduced chemical etching techniques as an attempt to modify the surface of the fiber by abstraction of hydrogen atoms from the polymer backbone and replacement with polar groups (Landrock et al., 1985; Silverstein and Breuer, Polymer, 1993). In this thesis, several solutions for chemical surface treatment techniques such as: Chromic acid B (by mixing potassium dichromate), Chromic acid C (by mixing sodium dichromate dehydrate) Potassium Permanganate and Hydrogen Peroxide solution were performed and tested to investigate the effectiveness on adhesion improvement of fibers in concrete.

Surface treatment of polypropylene and polyethylene fibers by ozone has been considered as an efficient, economical, and potentially practical technique (Dasgupta, 1990). It has been found that using UV and Ozone (UVO_3) for surface treatment of polyethylene and polypropylene fibers directly affects on the contact angle between the fiber's surface and a water droplet resulting the improvement of their adhesion strength (Gongjian et al., 1996). In this work, both UV and UV and Ozone surface treatment techniques were performed and evaluated. To do this, the fibers were exposed to UV lamp for a number of durations and the contact angles were measured. The best duration

for treatment in both cases was determined by using contact angle measurement method and then several FRC samples were prepared and tested.

The effectiveness of the fiber surface treatment techniques on the bonding strength between fibers and concrete were investigated testing the compressive and flexural behavior of specimens. Also, the contact angles were measured for all groups of fiber surface treatment techniques and compared with the untreated ones. Finally all results obtained from compressive, flexural and contact angle measurements for different groups prepared by various fiber surface treatment techniques and those for plain concrete (unreinforced) were gathered and compared.

5.3 Conclusions

The results of compressive and flexural tests on FRC and the contact angle values for all surface treated fibers and untreated ones were presented and discussed in Chapter 4 and Appendix B. The following conclusions can be drawn from the present investigation:

1. The compressive strength of concrete was slightly decreased by introduction of fibers. This could be due to greater compressive strength of the concrete material and also due to the loss of the bonding between cement and aggregates in presence of the fibers.
2. The major advantage of the introduction fibers to concrete as reinforcement was the appearance of post crack residual strength of concrete members in bending. Plain concrete totally failed after reaching the peak load in flexural test, due to the brittle nature of unreinforced concrete materials.

3. Chemical surface treatment of STRUX® 90/40 fibers by Chromic Acid using Potassium Dichromate solution (Solution B) was found the most effective technique among all other fiber surface treatment techniques presented in this work in improving the mechanical properties of FRC.
4. STRUX® 90/40 fiber surface treatment techniques by Chromic Acid using sodium dichromate dehydrate solution (Solution C) and by Hydrogen Peroxide etching gave moderate flexural strength improvements among the chemical surface treatment techniques.
5. Use of Potassium Permanganate for chemical treatment of STRUX® 90/40 fibers had negative effect on bonding strength between the fibers and concrete.
6. UV surface treatment technique on STRUX® 90/40 was found to be an ineffective technique in improvement of bonding strength concluded from the results of flexural test and the contact angle measurements.
7. Using UV and Ozone to treat the surface of STRUX® 90/40 fibers greatly improved the contact angle which was directly related to the duration of treatment. Within first five minutes of treatment, the contact angle significantly decreased about 20 degrees and then continued to decrease slightly up to 90 minutes treatment
8. The results for 40 minutes UV and Ozone surface treatment of STRUX® 90/40 showed lower flexural strength than those that were treated for 10 minutes. The results of 90 minutes group indicated the same conclusion. This could due to shape deformation or degradation of fibers because of the heat generate of UV lamp.

9. Ten minutes UV and Ozone surface treatment of STRUX® was found the best duration of treatment among of all physical surface treatment techniques.
10. The measurement of Gradient of Decreasing Residual Strength was a method that introduced in this work to investigate the bonding strength between fibers an concrete. Comparison the results for the high dosage groups showed lower gradient value for the fibers treated by Chromic Acid using Potassium Dichromate than non-treated ones while both group had the same toughness. Also for UV and Ozone treated groups, increasing the duration of expose to UV lamp decreased the gradients values. As a conclusion, the lower the Gradient of Decreasing Residual Strength showed the higher bonding strength between the fibers and concrete.
11. The contact angle and Gradient of Decreasing Residual Strength with flexural strength of FRC showed no relationship for those groups which were treated by chemical surface treatment techniques. The Gradient of Decreasing Residual Strength values decreased with increasing the duration of fiber surface treatment by UV and Ozone technique, meaning the higher duration of UV and Ozone treatment the lower rate of losing the residual strength of FRC.
12. From an economical point of view, the UV and Ozone treatment is recommended in the fiber product industry to improve the bonding strength between STRUX® 90/40 and concrete since chemical surface treatment techniques are neither economical nor practical techniques.

13. Higher percentages of fiber gave improvements in mechanical properties exceeding all improvements achieved through surface treatments. This may in the end be the best and most economical option.

5.4 Recommendations for Further Research

Further research is needed to investigate the effect of other parameters and to provide a comprehensive understanding of the bonding behavior of FRC. Some of the investigations needed in this area are recommended below:

1) The investigation could be continued by using other fiber aspect ratios to determine the effect of length/diameter of fibers on their bonding strength to concrete.
2) Investigation on standard specimen dimensions increases the compatibility of the parameters with those which specified by ASTM C 1609.
3) Equipment improvements such a vibrator equipped with frequency control and a compressive strength testing machine which the load rate could be controlled is recommended.
4) The effect of plasma fiber surface treatment technique, which is a relatively newer technique recommended by many studies, on bonding strength between fibers and concrete could be investigated.
5) Investigation on other dosage of fibers, more than that amount that specified by the producer company, gives the opportunity to find highest reachable flexural strength without sacrificing the required compressive strength of fiber reinforced concrete.

6) Standard Stress-Strain curve for compression test is recommended to determine the remaining compressive strength after the failure of fiber reinforced concrete occurs.

7) Fiber Pullout test is a method used by previous investigations to study the bonding strength between fiber and concrete. The examination of surface treatment techniques using this test method is recommended.

8) The chemical and physical fiber surface treatment techniques performed on Polypropylene/Polyethylene blend fibers could be extended to other polymeric fibers to investigate their effect on bonding strength improvement.

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Appendix A

STRUX® 90/40, Product Information

PRODUCT INFORMATION

STRUX® 90/40

Synthetic Macro Fiber Reinforcement

Description

STRUX® 90/40 synthetic macro fiber reinforcement is a unique form of high strength, high modulus synthetic macro reinforcement that is distributed throughout the concrete matrix. STRUX 90/40 gives toughness, impact and fatigue resistance to concrete. Unlike traditional microfiber reinforcement, STRUX 90/40 is specifically engineered to provide high, post-crack control performance. Reinforced concrete with STRUX 90/40 has been shown to reliably achieve average residual strength values in excess of 1.0 MPa (150 psi) at dosages that can easily be batched and finished in the field. It consists of synthetic macro fibers 40 mm (1.55 in.) in length with an aspect ratio of 90 that have specifically been designed to replace welded wire fabric, steel fibers, light rebar and other secondary reinforcement in slab-on-ground flooring and thin-walled precast applications. STRUX 90/40 is a user-friendly fiber reinforcement which is easier and safer to use, compared to these other types of reinforcement.

Uses

STRUX 90/40 is specially designed for ease of use, rapid dispersion, good finishability and improved pumpability in slab-on-ground flooring and many precast applications. STRUX 90/40 may be used in commercial floors, industrial floors, residential floors, other flat work applications and form work applications. The performance of STRUX 90/40 depends on the compressive strength of concrete. STRUX 90/40 is not intended as a substitute for steel reinforcing in any application other than slab-on-ground flooring and thin-walled precast applications. Always consult local building codes.

Advantages

STRUX 90/40 enhances safety during installation by eliminating the risk for potential injury caused by handling and placement difficulties commonly associated with steel fibers, welded wire fabrics or light rebar. Additionally, STRUX 90/40 does not corrode.

The geometry, strength and the elastic modulus of STRUX 90/40 were optimized to provide superior crack control. STRUX 90/40 fibers are uniformly built into the concrete, eliminating a concern over proper positioning of reinforcement. Also, STRUX 90/40 controls plastic shrinkage cracking and cracking due to drying shrinkage of the concrete.

Addition Rates

STRUX 90/40 addition rates are dependent on the specific application and desired properties and will vary between 1.8 to 7.0 kg/m^3 (3.0 to 11.8 lbs/yd^3). Please see STRUX 90/40 conversion tables for detailed information.

Mix Design and Mixing Requirements

The utilization of STRUX 90/40 may require the use of a superplasticizer such as ADVA® to restore the required workability. In addition, slight increases in fine aggregate contents may be needed.

STRUX 90/40 may be added to concrete at any point during the batching or mixing process. STRUX 90/40 can be added as fast as one bag every 5 seconds. After fiber addition, the concrete must be mixed in a drum at the recommended mixing speed for a minimum of 70 revolutions to ensure adequate dispersion.

Please contact your Grace representative with any questions.

Compatibility

STRUX 90/40 is compatible with all GRACE admixtures. Their action in concrete is mechanical and will not affect the hydration process of the cement or compressive strength. Each liquid admixture should be added separately to the concrete mix.

Packaging and Dispensing

STRUX 90/40 is available in 2.26 kg (5 lbs) Concrete-Ready Bags.

U.S. Patent Nos.: 6,569,525; 6,569,526; 6,758,897; 6,863,969

GRACE
Construction Products

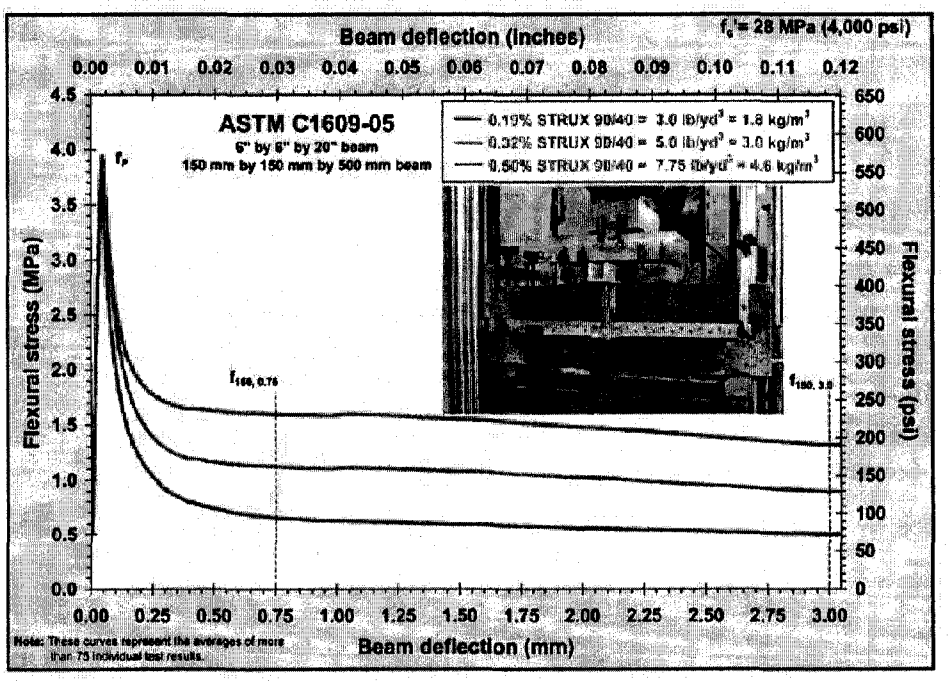
Flexural Strength and Toughness (Compressive Strength: 28 MPa) according to ASTM C1609-05

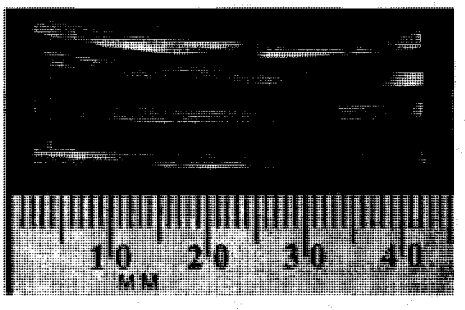
STRUX 90/40 Dosage Rate	Specimen cross-section		Peak Load P_p (N)	Peak Strength f_p (MPa)	Peak-load deflection δ_p (mm)	Residual loads		Residual strengths		Toughness $T_{150,3.0}$ (Joule)	JCI-SF4[1] $f_{e,3}$ (MPa)	TR34[2] $R_{e,3}$ (%)
	Width (mm)	Depth (mm)				$P_{150,0.75}$ (N)	$P_{150,3.0}$ (N)	$f_{150,0.75}$ (MPa)	$f_{150,3.0}$ (MPa)			
0.19% (1.8 kg/m³)	152	151	29,813	3.90	0.048	5,776	4,236	0.75	0.55	18	0.80	20.0%
0.32% (3.0 kg/m³)	152	152	31,422	4.10	0.050	8,472	6,932	1.10	0.90	27	1.15	28.5%
0.50% (4.6 kg/m³)	152	151	30,513	4.00	0.050	12,323	10,012	1.60	1.30	37	1.60	40.5%

Flexural Strength and Toughness (Compressive Strength: 4,000 psi) according to ASTM C1609-05

STRUX 90/40 Dosage Rate	Specimen cross-section		Peak Load P_p (lbf)	Peak Strength f_p (psi)	Peak-load deflection δ_p (in.)	Residual loads		Residual strengths		Toughness $T_{150,3.0}$ (lbf-in.)	JCI-SF4[1] $f_{e,3}$ (psi)	TR34[2] $R_{e,3}$ (%)
	Width (in.)	Depth (in.)				$P_{150,0.75}$ (lbf)	$P_{150,3.0}$ (lbf)	$f_{150,0.75}$ (psi)	$f_{150,3.0}$ (psi)			
0.19% (3.0 lbs/yd³)	6.00	5.95	6,702	565	0.0019	1,299	952	110	80	160	115	20.0%
0.32% (5.0 lbs/yd³)	6.00	6.00	7,064	595	0.0020	1,905	1,558	160	130	240	165	28.5%
0.50% (7.75 lbs/yd³)	6.00	5.95	6,860	580	0.0020	2,770	2,251	230	190	330	230	40.5%

1) Japan Concrete Institute (JCI): "Standard Test Method for Flexural Strength and Flexural Toughness of Fiber Reinforced Concrete, (Standard SF4)," JCI Standards for Test Methods of Fiber Reinforced Concrete, Japan Concrete Institute, 1983. 2) The Concrete Society. Technical Report 34 Concrete industrial ground floors – A guide to their design and construction, The Society, Crowthorne, 2003.





STRUX 90/40 Properties

Specific Gravity	0.92
Absorption	None
Modulus of Elasticity	1,378 ksi (9.5 GPa)
Tensile Strength	90 ksi (620 MPa)
Melting Point	160°C (320°F)
Ignition Point	590°C (1,094°F)
Alkali, Acid & Salt Resistance	High

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Appendix B

Details of Compressive, Flexural, Contact Angle Test Results

B.1 Determination of Mixing Proportion

A compressive strength of 30 MPa were chosen for this work so that a mixing proportion had to be determined to reach this strength. The initial mixing proportion recommended by St. Lawrence Company is appeared in Table B.1. For this single group only 3 cylindrical specimens for each age (7 and 28 days) were prepared. The results of compressive strength are presented in Table B.2.

Table B.1—Mixing Proportion of Concrete Recommended by St. Lawrence.

Concrete Materials	**kg/m^3**
Coarse aggregate	1140
Aggregate	760
Cement	345
Water	173

Table B.2—Compressive Strength Test Results for Mixing proportion Recommended by St. Lawrence.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa
PC1	81	8.9	101	11.2
PC1	74	8.2	93	10.3
PC1	83	9.3	105	11.6
Average	79	8.8	100	11.1

The compressive strength was much lower than the compressive strength that assigned for this work and also had very low slump (almost zero), so this mixing proportion was refused. The second mixing proportion was brought from Grace Company

recommendation to evaluate the compressive strength (Table B.3). For this mixing proportion seven specimens were prepared. Two specimens were tested for 7 days age and the rest were tested for 28 days. The results of compressive strength for this mixing proportion are presented in Table B.4.

Table B.3—Mixing Proportion of Concrete Recommended by Grace Company.

Concrete Materials	**kg/m^3**
Coarse aggregate	878
Aggregate	1074
Cement	270
Water	178

Table B.4—Compressive Strength Test Results for Mixing proportion Recommended by Grace Company.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa
PC2	152	16.9	161	17.8
PC2	120	13.3	165	18.3
PC2			165	18.3
PC2			157	17.4
PC2			148	16.4
Average	136	15.1	159	17.6

The compressive strength by using this mixing proportion apparently increased, but still did not reach the strength that had been proposed for this work. Also the slump of this mixing proportion was 4 cm which could be still low for this work, since adding the fibers to concrete result the lose of slump. At the next step, the mixing proportion

recommended by Grace Company was modified by adding more cement and water to increase the compressive strength and slump of concrete. For this mixing proportion ten specimens were prepared and five specimens were tested for both 7 and 28 days. The mixing proportion for this group and compressive test results are shown in Tables B.5 and B.6 respectively.

Table B.5—Mixing Proportion of Plain Concrete.

Concrete Materials	**kg/m^3**
Coarse aggregate	878
Aggregate	1074
Cement	330
Water	195

Table B.6—Compressive Strength Test Results for Mixing Proportion of Plain Concrete.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa
PC3	216	24.0	*	*
PC3	216	24.0	268	29.8
PC3	214	23.7	267	29.6
PC3	212	23.5	267	29.6
PC3	220	24.4	270	29.9
Average	216	24.0	268	29.7

* The maximum load exceeded the capacity of testing machine. From this point a bigger testing machine was used.

The results of this mixing proportion satisfied the compressive strength requirement of this work. Also, the slump was close to 60 mm which was desirable. From that point, above mixing proportion were chosen to make the concrete and to investigate on bonding characteristics of different surface treated fibers and concrete.

B.2 Flexural Strength Results of Plain Concrete

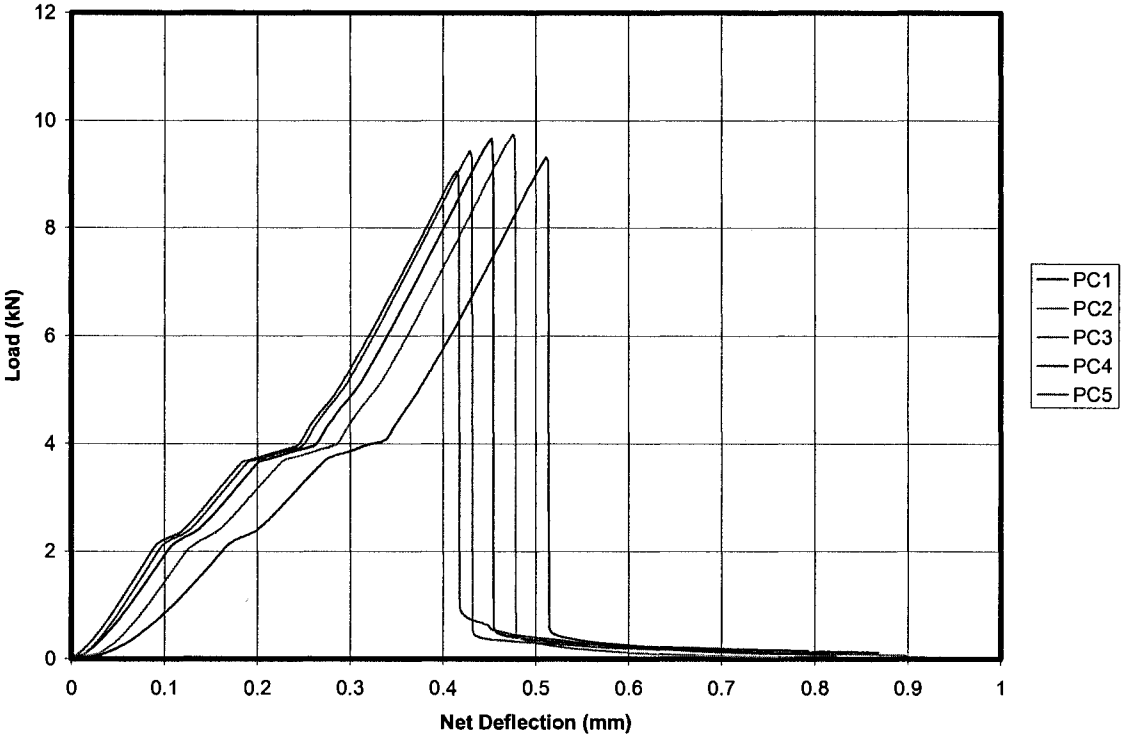

Figure B.1—Load-Deflection Curve for Plain Concrete (PC), 28 Days.

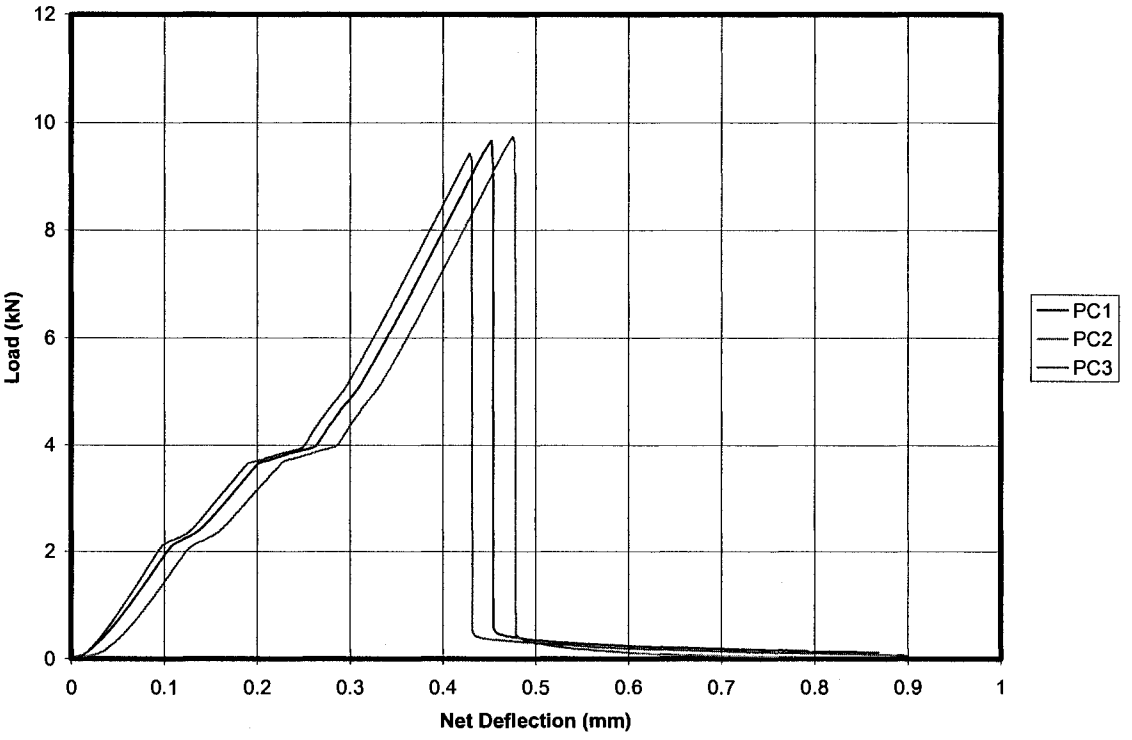

Figure B.2—Modified Load-Deflection Curve for Plain Concrete, 28 Days.

Table B.7—Flexural Strength Test Results for Modified Mixing proportion Plain Concrete, 28 Days.

PC	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$T_{75,3.0}$ Joule
1	9,659	4.99	0.45	0	0	0	0	1.97
2	9,734	5.03	0.48	0	0	0	0	1.94
3	9,420	4.87	0.44	0	0	0	0	1.85
Average	9,604	4.96	0.46	0	0	0	0	1.9

B.3 FRC without Fiber Surface Treatment Results

Table B.8—Compressive Strength Test Results for Non-Treated, Middle Dosage (NT), 28 Days.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa
NT	197	21.8	246	27.3
NT	200	22.2	242	26.8
NT	192	21.4	240	26.6
NT	192	21.3	228	25.8
NT	197	21.8	241	26.7
Average	196	21.7	239	26.6

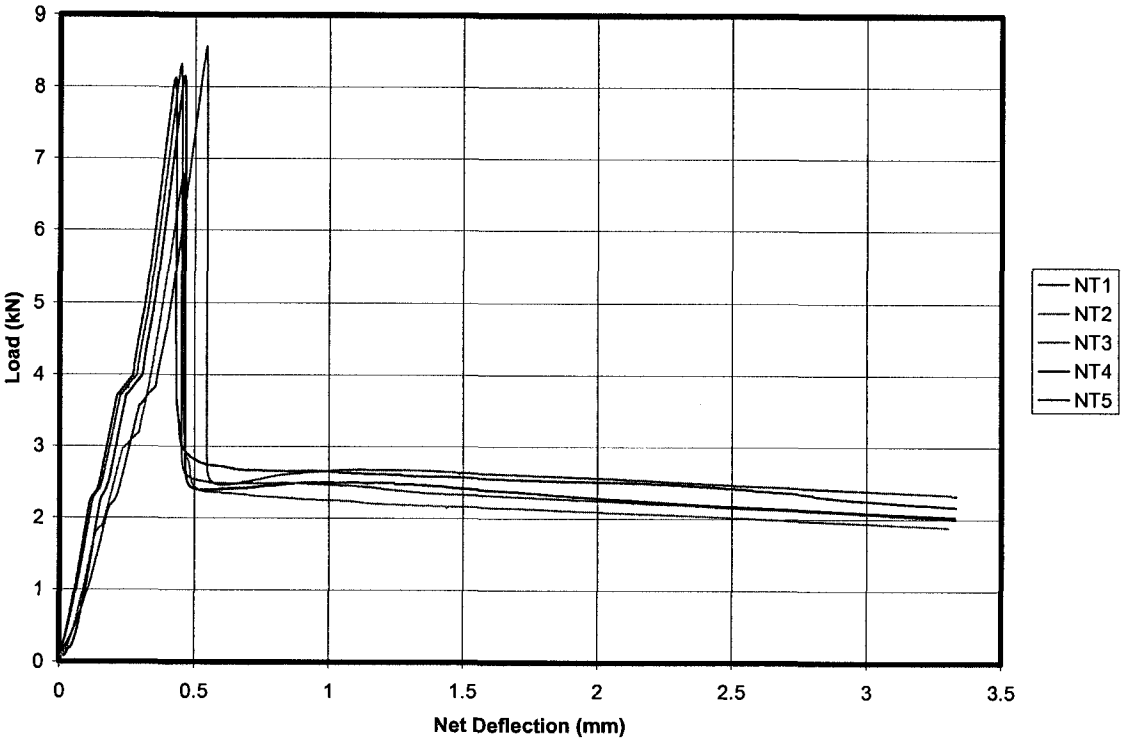

Figure B.3—Load-Deflection Curve for FRC without Fiber Surface Treatment (NT), 28 Days.

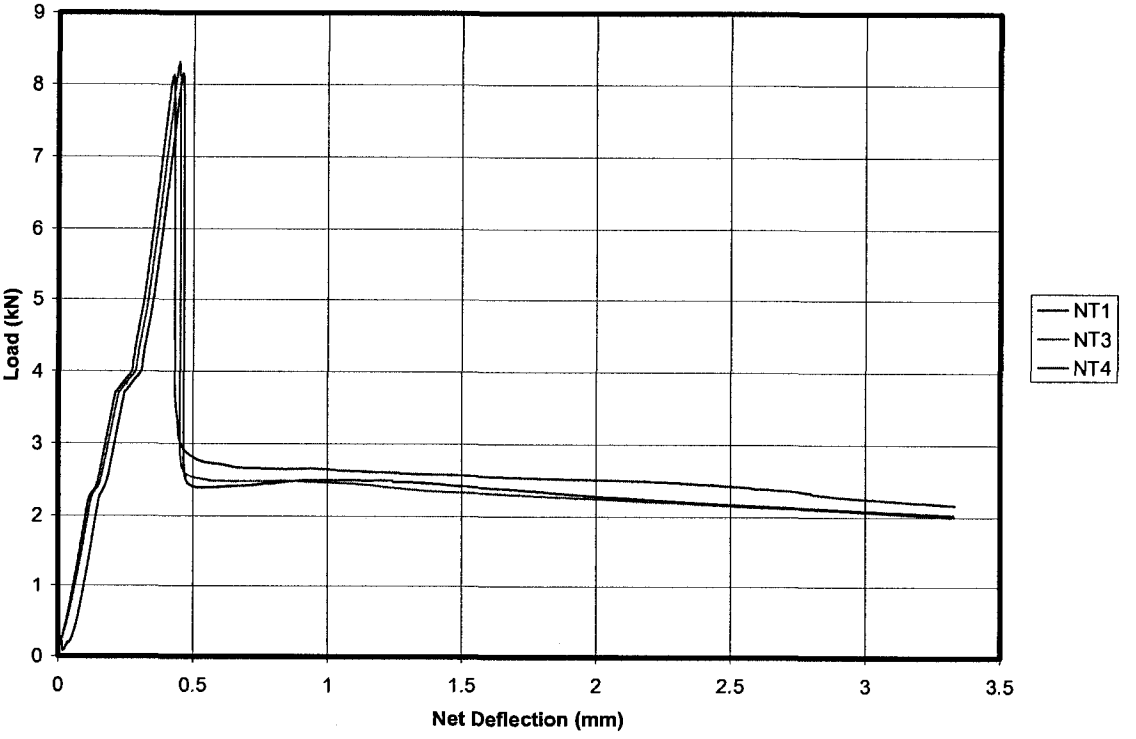

Figure B.4—Modified Load-Deflection Curve for FRC without Fiber Surface Treatment (NT), 28 Days.

Table B.9—Flexural Strength Test Results for FRC without Fiber Surface Treatment (NT), 28 Days.

NT	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	8,150	4.21	0.47	2,446	2,413	2,032	1.26	1.25	1.04	7.48
3	8,317	4.30	0.45	2,481	2,327	2,000	1.28	1.20	1.03	7.52
4	8,129	4.20	0.43	2,658	2,564	2,169	1.37	1.32	1.12	8.12
Average	8,199	4.24	0.45	2,528	2,435	2,067	1.30	1.26	1.06	7.7

Table B.10—Contact Angle Measurement for Non-Treated Fibers.

Group NT (Image Number)	Contact Angle, Degree
1	90.79
2	91.13
3	94.35
4	95.31
5	92.58
6	96.57
7	94.87
8	94.08
9	94.91
10	94.93
11	96.69
12	95.68
13	96.1
14	92.57
15	93.64
16	89.92
Average	94.01

B.4 Chemical Surface Treated Fiber Results

B.4.1 Chromic Acid Solution, Type B (Potassium Dichromate), (CAB)

Table B.11—Compressive Strength Test Results for Fiber Surface Treated by Chromic Acid Solution, Type B (CAB), 28 Days.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa
CAB	195	21.6	257	28.5
CAB	201	22.3	255	28.2
CAB	205	22.7	246	27.4
CAB	194	21.5	241	26.8
CAB	197	21.9	250	27.8
Average	198	22.0	250	27.7

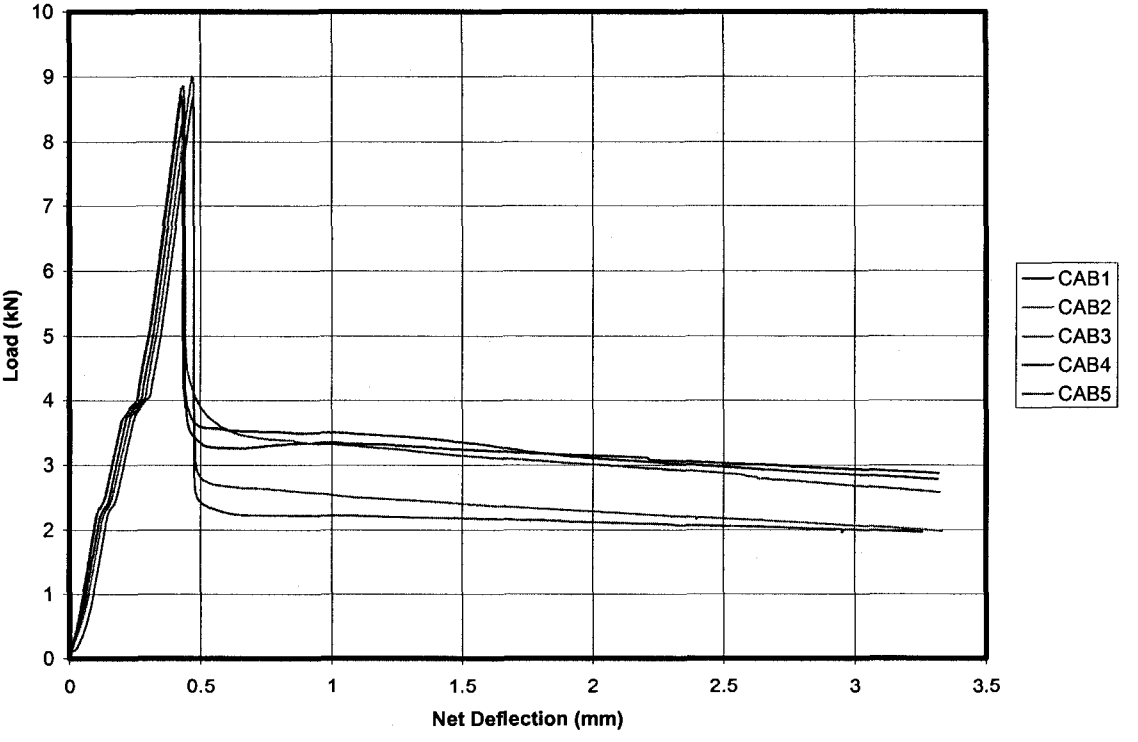

Figure B.5—Load-Deflection Curve for Fiber Surface Treated by Chromic Acid Solution, Type B (CAB), 28 Days.

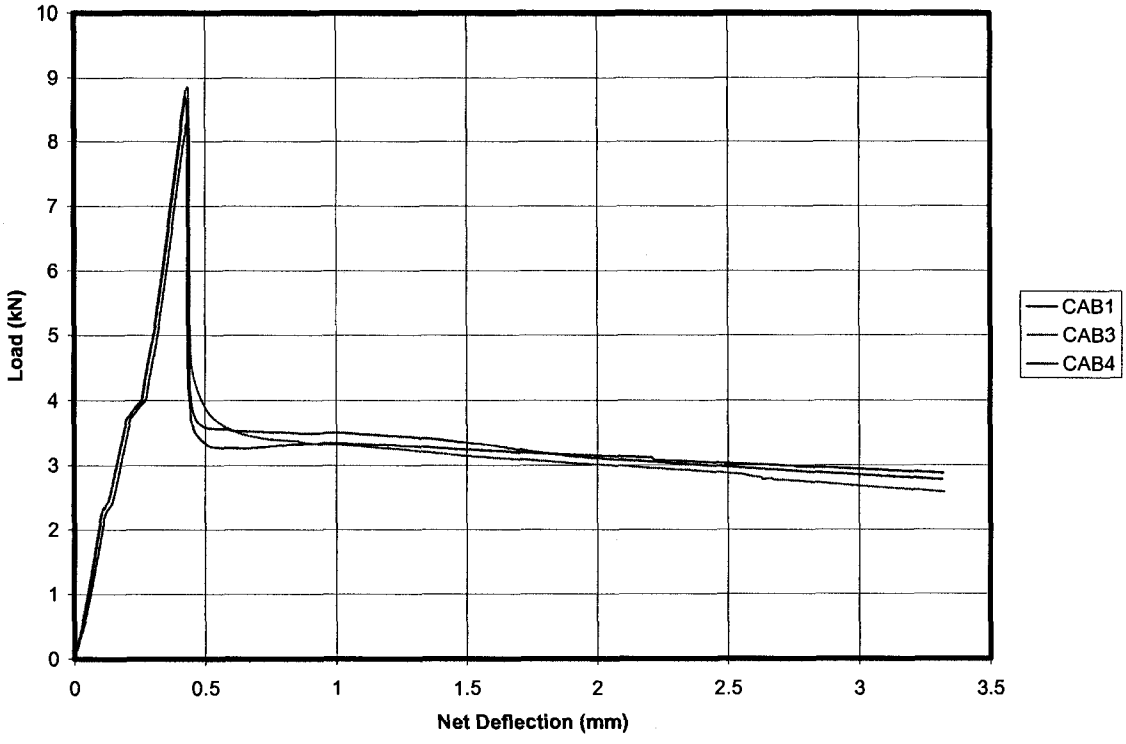

Figure B.6—Modified Load-Deflection Curve for Fiber Surface Treated by Chromic Acid Solution, Type B (CAB), 28 Days.

Table B.12—Flexural Strength Test Results for Fiber Surface Treated by Chromic Acid Solution, Type B (CAB), 28 Days.

CAB	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	8,695	4.49	0.44	3,291	3,238	2,878	1.70	1.67	1.48	9.9
3	8,856	4.58	0.44	3,401	3,138	2,591	1.76	1.62	1.34	9.8
4	8,276	4.28	0.45	3,511	3,348	2,787	1.81	1.73	1.44	9.9
Average	8,609	4.45	0.44	3,401	3,241	2,752	1.76	1.67	1.42	9.8

Table B.13—Contact Angle Measurement for Fiber Surface Treated by Chromic Acid Solution, Type B (CAB).

Group CAB (Image Number)	Contact Angle, Degree
1	68.86
2	70.13
3	72.5
4	71.22
5	71.85
6	72.19
7	66.96
8	66.88
9	63.57
10	68.43
11	69.83
12	73.01
13	69.66
14	72.73
15	68.68
16	66.45
17	71.98
18	67.91
19	70.18
20	64.43
Average	69.37

B.4.2 Chromic Acid Solution, Type C (Sodium Dichromate), (CAC)

Table B.14—Compressive Strength Test Results for Fiber surface treated by Chromic Acid Solution, Type C (CAC), 28 Days.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa
CAC	195	21.6	259	28.7
CAC	206	22.9	253	28.1
CAC	208	23.1	255	28.3
CAC	205	22.7	252	27.9
CAC	199	22.1	263	29.1
Average	203	22.5	256	28.4

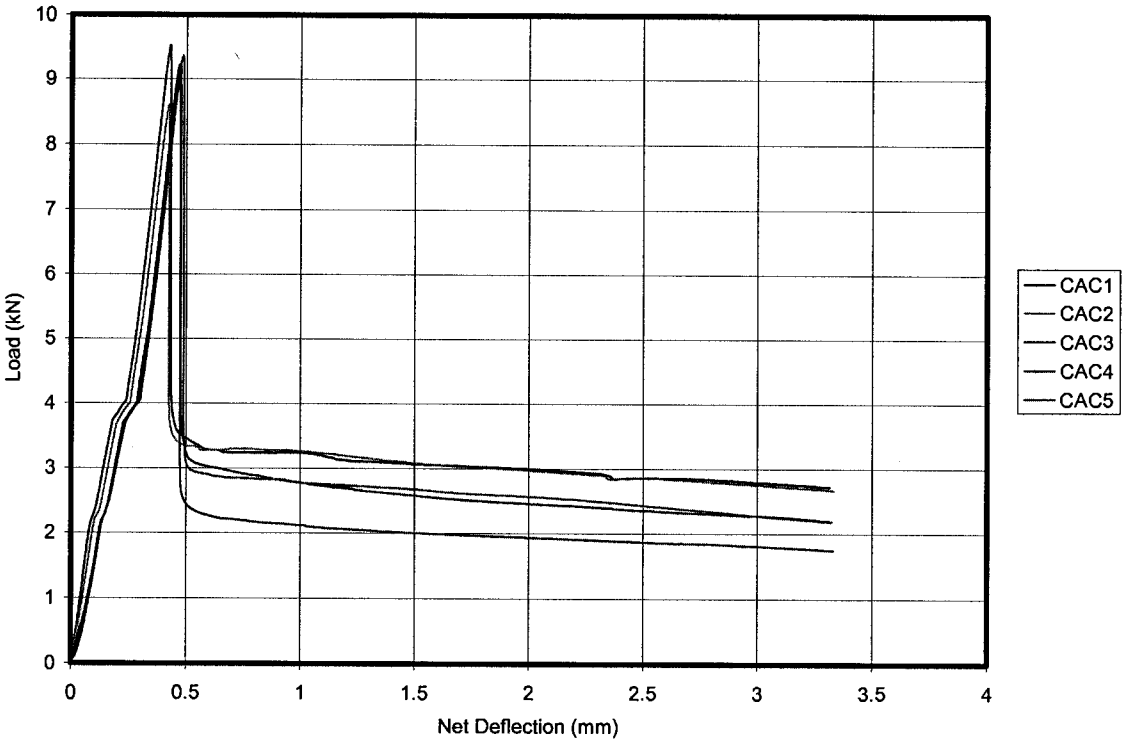

Figure B.7—Load-Deflection Curve for Fiber Surface Treated by Chromic Acid Solution, Type C (CAC), 28 Days.

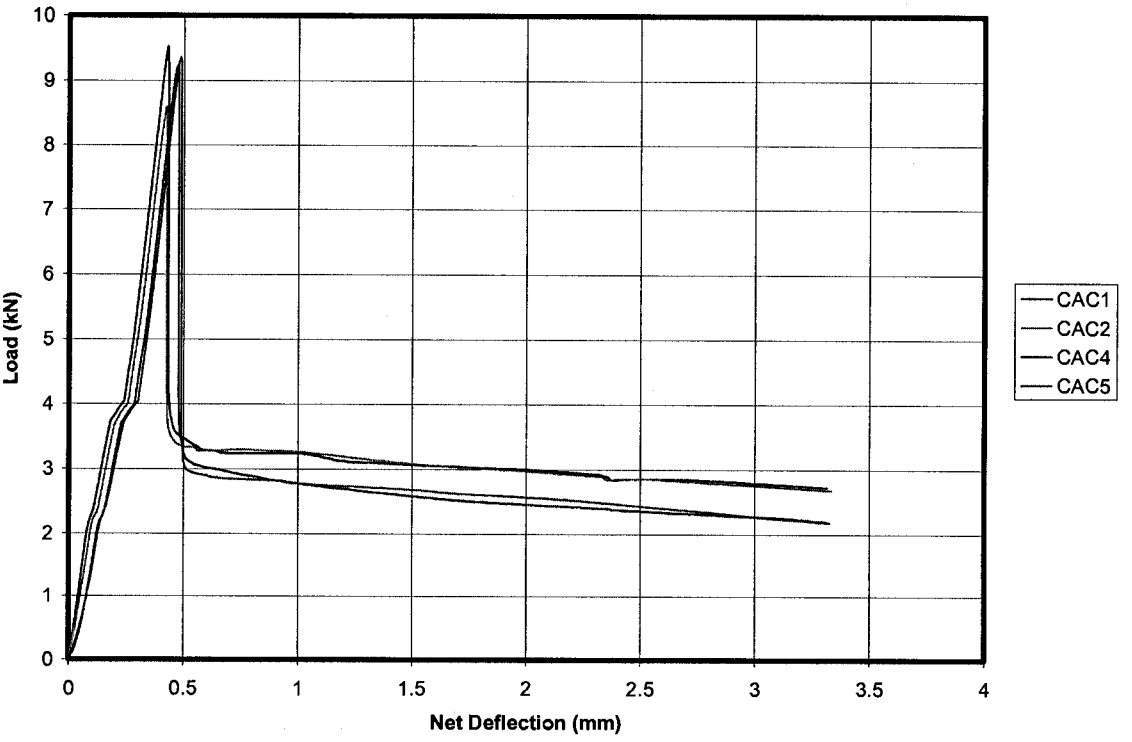

Figure B.8—Modified Load-Deflection Curve for Fiber Surface Treated by Chromic Acid Solution, Type C (CAC), 28 Days.

Table B.15—Flexural Strength Test Results for Fiber Surface Treated by Chromic Acid Solution, Type C (CAC), 28 Days.

CAC	P_p N	f_p MPa	δ_p mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	9,519	4.92	0.43	3,235	3,063	2,781	1.67	1.58	1.44	9.7
2	8,596	4.44	0.42	3,294	3,074	2,741	1.70	1.59	1.42	9.5
4	9,229	4.77	0.47	2,905	2,572	2,204	1.50	1.33	1.17	8.4
5	9,356	4.83	0.48	2,840	2,676	2,193	1.67	1.38	1.16	8.5
Average	9,175	4.74	0.45	3,068	2,846	2,513	1.59	1.47	1.30	9.0

Table B.16—Contact Angle Measurement for Fiber Surface Treated by Chromic Acid Solution, Type C (CAC).

Group CAC (Image Number)	Contact Angle, Degree
1	75.01
2	75.69
3	75.23
4	68.8
5	73.13
6	71.81
7	71.88
8	74.79
9	68.83
10	70.97
11	64.1
12	69.56
13	68.9
14	66.53
15	63.1
16	63.41
17	62.62
18	64.31
19	63.71
20	70.43
Average	69.14

B.4.3 Potassium Permanganate, (PP)

Table B.17—Compressive Strength Test Results for Fiber Surface Treated by Potassium Permanganate (PP), 28 Days.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa
PP	194	21.5	253	28.1
PP	194	21.5	251	27.9
PP	194	21.5	237	26.9
PP	206	22.8	252	28.0
PP	197	21.9	258	28.6
Average	197	21.8	250	27.9

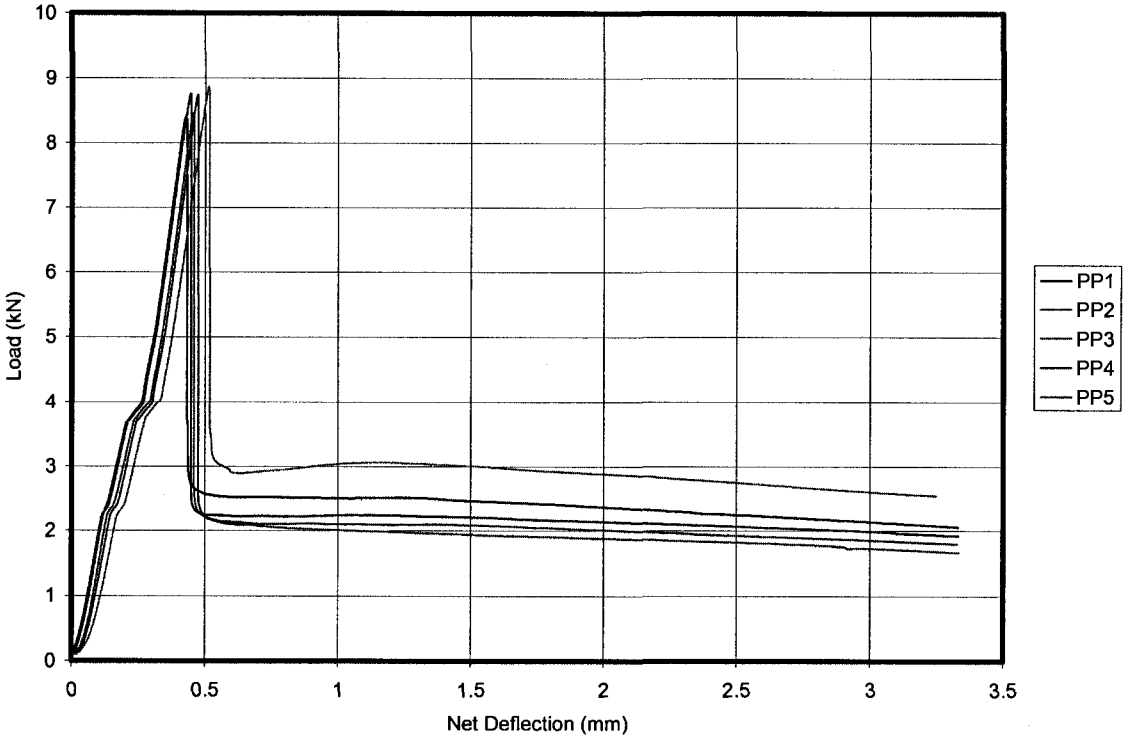

Figure B.9—Load-Deflection Curve for Fiber Surface Treated by Potassium Permanganate (PP), 28 Days.

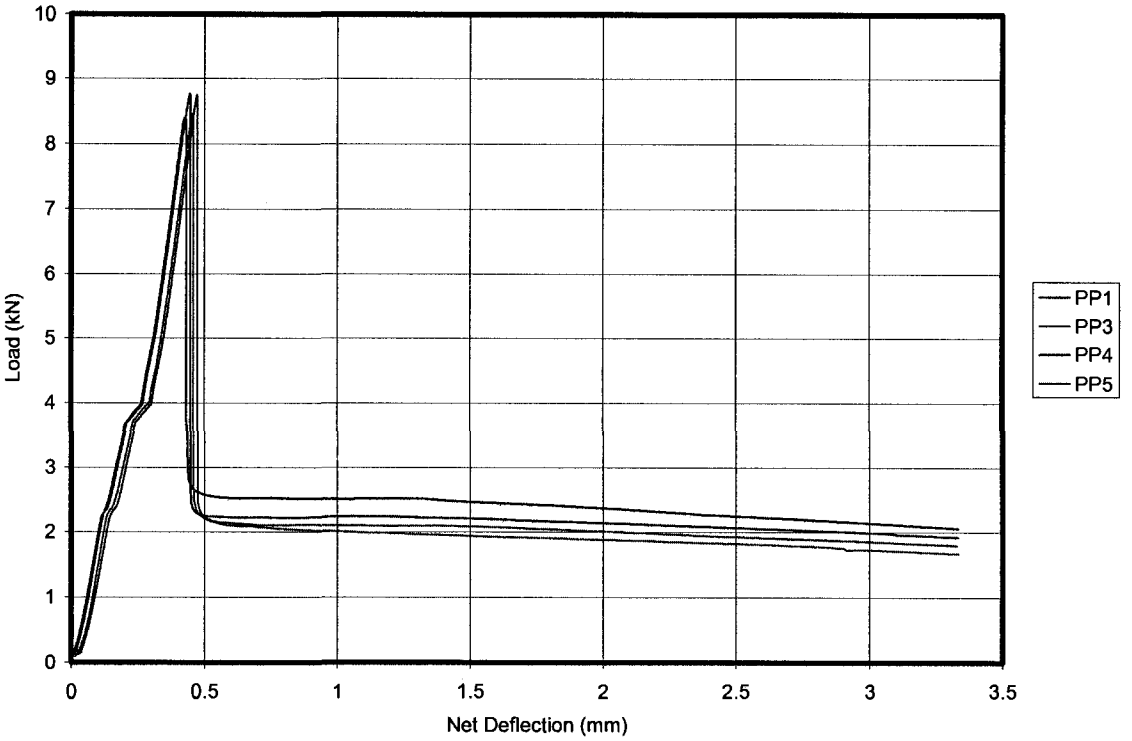

Figure B.10—Modified Load-Deflection Curve for Fiber Surface Treated by Potassium Permanganate (PP), 28 Days.

Table B.18—Flexural Strength Test Results for Fiber Surface Treated by Potassium Permanganate (PP), 28 Days.

PP	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	8,400	4.34	0.426	2,521	2,464	2,126	1.30	1.27	1.10	7.8
3	8,456	4.37	0.45	2,054	1,938	1,673	1.06	1.00	0.86	6.5
4	8,760	4.53	0.45	2,223	2,207	1,925	1.15	1.14	0.99	7.3
5	8,741	4.52	0.48	2,105	2,086	1,804	1.09	1.08	0.93	6.8
Average	8,589	4.44	0.45	2,226	2,174	1,882	1.15	1.12	0.97	7.1

Figure B.19—Contact Angle Measurement for Fiber Surface Treated by Potassium Permanganate (PP).

Group PP (Image Number)	Contact Angle, Degree
1	79.84
2	79.95
3	73.25
4	79.28
5	73.84
6	73.83
7	68.19
8	72.19
9	75.08
10	78.56
11	78.67
12	66.01
13	67.87
14	75.19
15	73.29
16	74.29
17	75.63
18	69.13
Average	74.12

B.4.4 Hydrogen Peroxide (HP)

Table B.20—Compressive Strength Test Results for Fiber Surface Treated by Hydrogen Peroxide (HP), 28 Days.

Group	Compression Test 7Days		Compression Test 28 Days	
	Maximum Load, kN	Compressive Strength, MPa	Maximum Load, kN	Compressive Strength, MPa
HP	204	22.6	251	27.9
HP	193	21.4	241	26.8
HP	199	22.1	250	27.7
HP	200	22.2	246	27.3
HP	208	23.1	255	28.2
Average	201	22.3	249	27.6

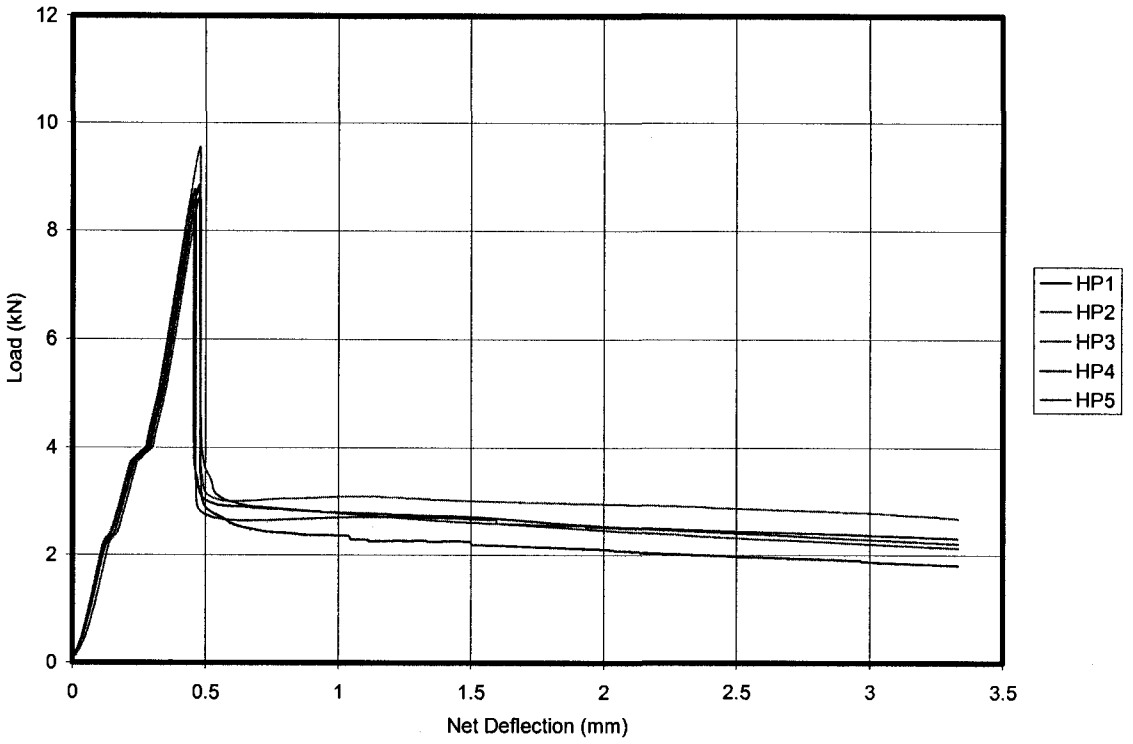

Figure B.11—Load-Deflection Curve for Fiber Surface Treated by Hydrogen Peroxide (HP), 28 Days.

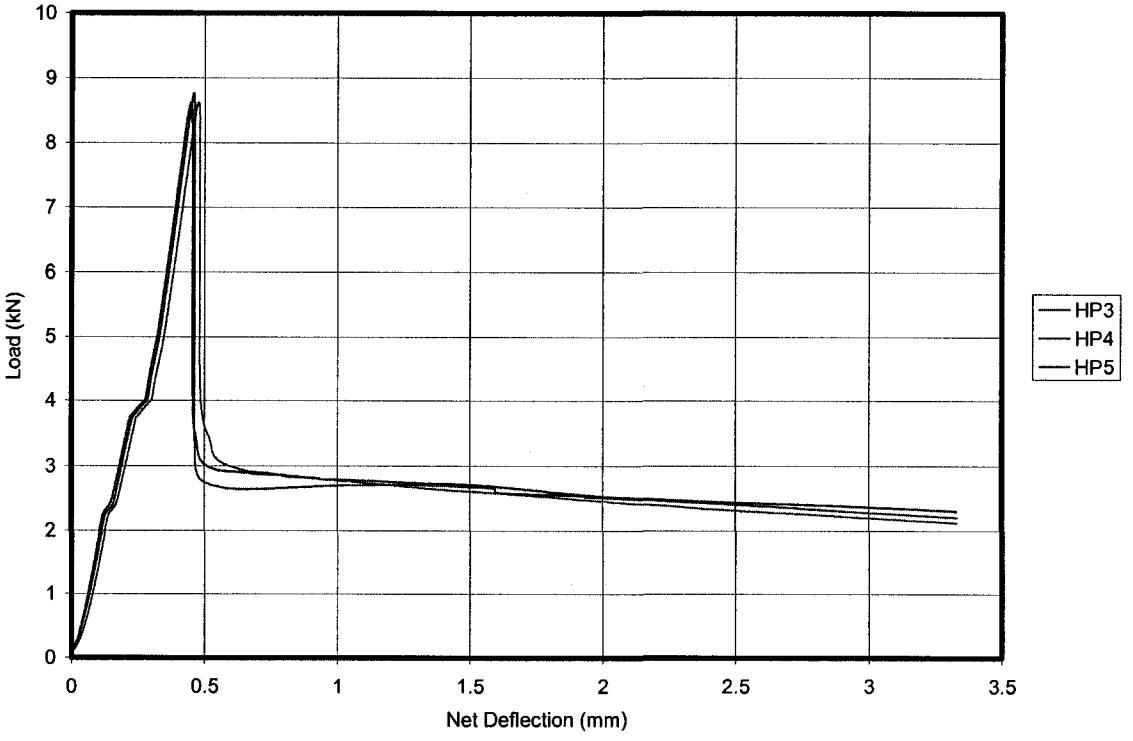

Figure B.12—Modified Load-Deflection Curve for Fiber Surface Treated by Hydrogen Peroxide (HP), 28 Days.

Table B.21—Flexural Strength Test Results for Fiber Surface Treated by Hydrogen Peroxide (HP), 28 Days.

HP	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
3	8,620	4.45	0.48	2,878	2,604	2,126	1.49	1.35	1.10	8.3
4	8,620	4.45	0.45	2,864	2,706	2,311	1.48	1.40	1.19	8.5
5	8,768	4.53	0.46	2,652	2,668	2,212	1.37	1.38	1.14	8.2
Average	8,669	4.48	0.46	2,798	2,659	2,216	1.45	1.38	1.14	8.3

Table B.22—Contact Angle Measurement for Fiber Surface Treated by Hydrogen Peroxide (HP).

Group HP (Image Number)	Contact Angle, Degree
1	90.95
2	84.54
3	84.96
4	87.51
5	85.6
6	86.82
7	80.39
8	81.53
9	81.55
10	82.4
11	79.27
12	81.47
13	86.01
14	89.08
15	90.06
16	88.47
17	84.35
18	87.62
19	81.73
20	88.98
Average	85.16

B.5 Physical Surface Treated Fiber Results

B.5.1 UV

To find the best time of treatment by UV the fibers were exposed to the UV lamp for 10, 30, 60, 90 and 120 minutes. The results are presented below:

Table B.23—Contact Angle Measurement for Fiber Surface Treated by UV.

Image No.	UV-10	UV-30	UV-60	UV-90	UV-120
1	87.87	92.51	94.37	95.84	91.74
2	88.01	91.8	94.71	96.94	89.15
3	88.2	92.89	92.44	94.69	91.33
4	87.7	96.55	94.8	96.4	92.37
5	91.42	95.84	92.53	93.87	92.52
6	95.48	95.12	93.11	93.04	95.26
7	94.5	95.05	94.27	92.34	92.4
8	88.21	94.16	95.3	92.72	94.47
9	92.65	92.59	88.63	94.17	95.06
10	91.39	90.7	93.69	93.54	96
11	92.98	92.66	91.16	92.23	94.59
12	97.03	88.51	91.56	92.24	90.89
13	97.45	89.88	92.02	95.55	92.7
14	92.89	92.6	92.13	92.68	88.93
15	93.54	91.87	93.73	92.75	89.37
16	96.24	88.48	94.34	85.67	90.2
17	97.74	90.89	94.73	85.91	92.41
18	99.83	92.84	91.9	93.73	90.04
19	95.93	94.34	92.3	92.06	
20		90.29	93.72	93.06	
21		90.73	93.27	88.45	
22		89.58	91.39	94.8	
23		92.86		93.79	
24		92.31			
Average	93.11	92.29	93.00	92.89	92.19

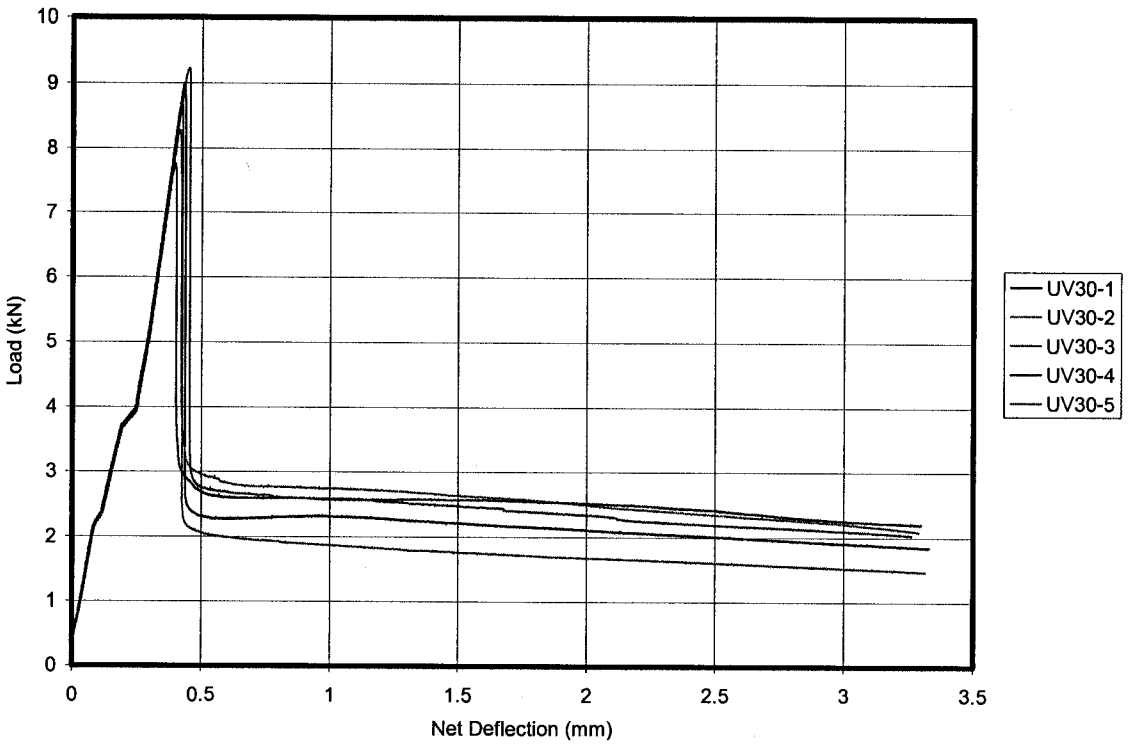

Figure B.13—Load-Deflection Curve for Fiber Surface Treated by UV for 30 Minutes (UV-30), 28 Days.

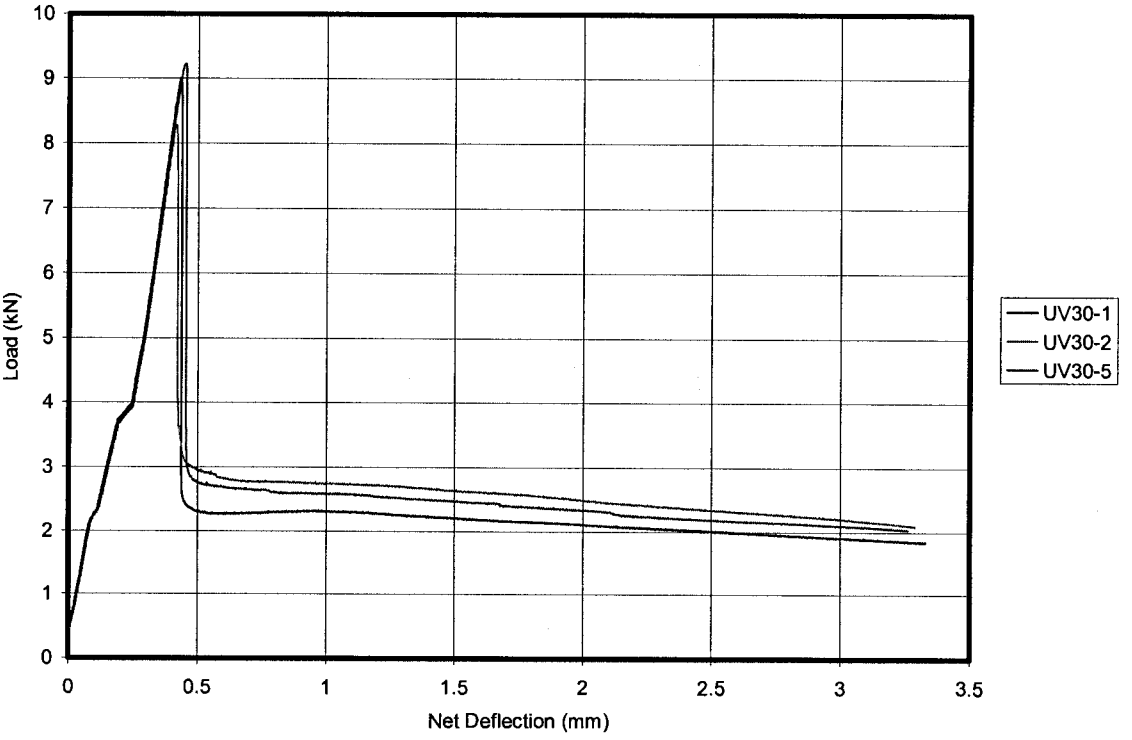

Figure B.14—Modified Load-Deflection Curve for Fiber Surface Treated by UV for 30 Minutes (UV-30), 28 Days.

Table B.24—Flexural Strength Test Results for Fiber Surface Treated by UV for 30 Minutes (UV-30), 28 Days.

UV-30	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	9,001	4.65	0.43	2,293	2,209	1,898	1.18	1.14	0.98	7.4
2	8,271	4.27	0.42	2,784	2,628	2,185	1.44	1.36	1.13	8.3
5	9,229	4.77	0.45	2,644	2,459	2,089	1.37	1.27	1.08	8.1
Average	8,834	4.56	0.43	2,574	2,432	2,057	1.33	1.26	1.06	7.9

B.5.2 UV and Ozone (UVO_3)

To find the best time of treatment by UV and Ozone the fibers were treated for 5, 10, 15, 20, 30, 40, 60 and 90 minutes. The results are presented in Table B.25:

Table B.25—Contact Angle Measurement for Fiber Surface Treated by UV and Ozone.

Image	UVO_3-5	UVO_3-10	UVO_3-15	UVO_3-20	UVO_3-30	UVO_3-40	UVO_3-60	UVO_3-90
1	69.6	75.11	71.08	69.89	65.61	63.66	62.68	63.2
2	70.54	72.01	69.46	71.32	64.79	65.23	66.85	62.68
3	70.87	69.39	68.72	62.71	61.85	65.64	64.66	62.15
4	70.85	70.17	70.81	61.99	67.58	62.39	63.67	63.87
5	67.64	72.95	68.55	68.27	64.76	66.98	66.59	57.58
6	66.13	66.36	67.35	65.01	68.00	62.00	65.68	58.96
7	72.84	69.13	66.1	66.66	60.08	60.26	60.29	60.67
8	66.08	69.81	70.4	66.41	65.8	61.26	63.87	64.93
9	69.63	64.77	67.96	67.13	66.95	61.76	65.28	57.32
10	66.97	73.82	69.01	71.87	63.87	61.36	62.12	60.02
11	72.32	75.64	67.79	62.69	62.79	61.69	65.28	59.53
12	70.78	68.22	66.89	60.83	65.78	64.97	66.36	64.27
13	72.23	69.55	64.25	59.88	61.88	62.71	60.65	58.44
14	70.05	70.99	70.63	64.82	61.32	67.61	61.53	58.74
15	68.75	64.19	67.52	66.29	66.91	66.22	62.34	60.42
16	67.5	64.80	68.51	65.58	64.19	61.05	63.22	55.81
17	67.6		70.71		57.58			56.31
18	69.93		71.33		64.15			55.79
19	72.44		68.38		64.95			60.19
20	69.99		71.86		61.68			58.08
21			69.82		59.26			64.09
22			67.51		59.75			58.49
23			69.18		62.26			63.43
24			65.78		62.07			58.65
25					61.93			56.47
26								64.51
27								62.75
28								63.95
Average	69.64	69.81	68.73	65.72	63.43	63.42	63.82	60.40

B.5.2.1 5 Minutes UV and Ozone (UVO_3-5) Results

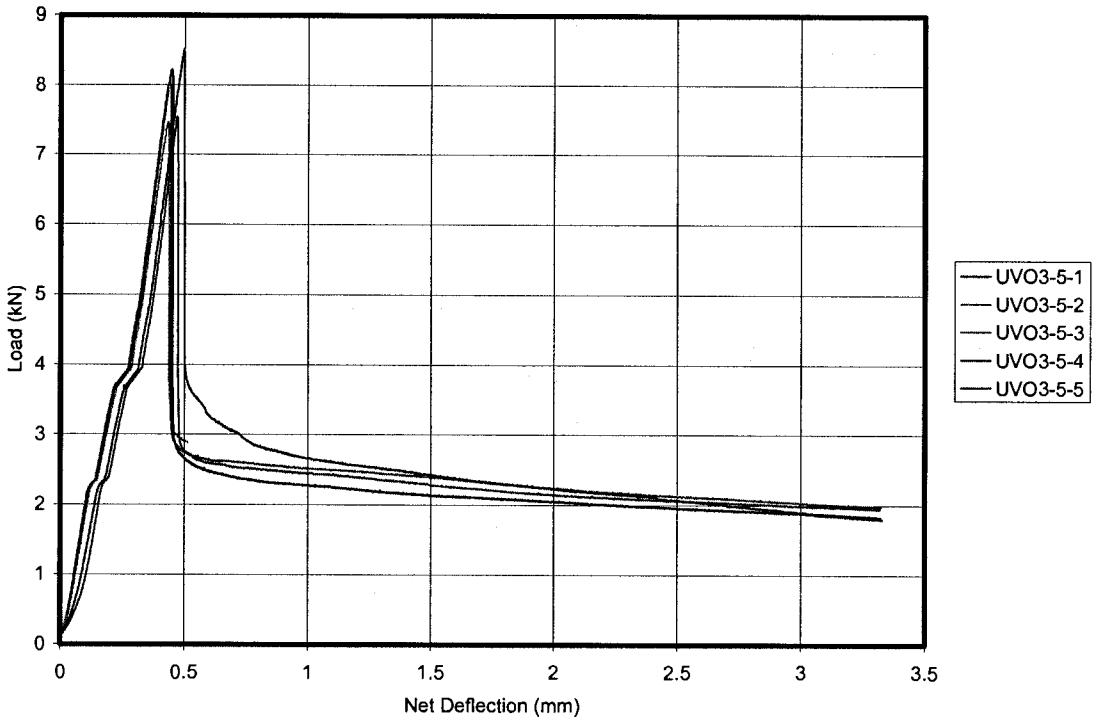

Figure B.15—Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 5 Minutes (UVO_3-5), 28 Days.

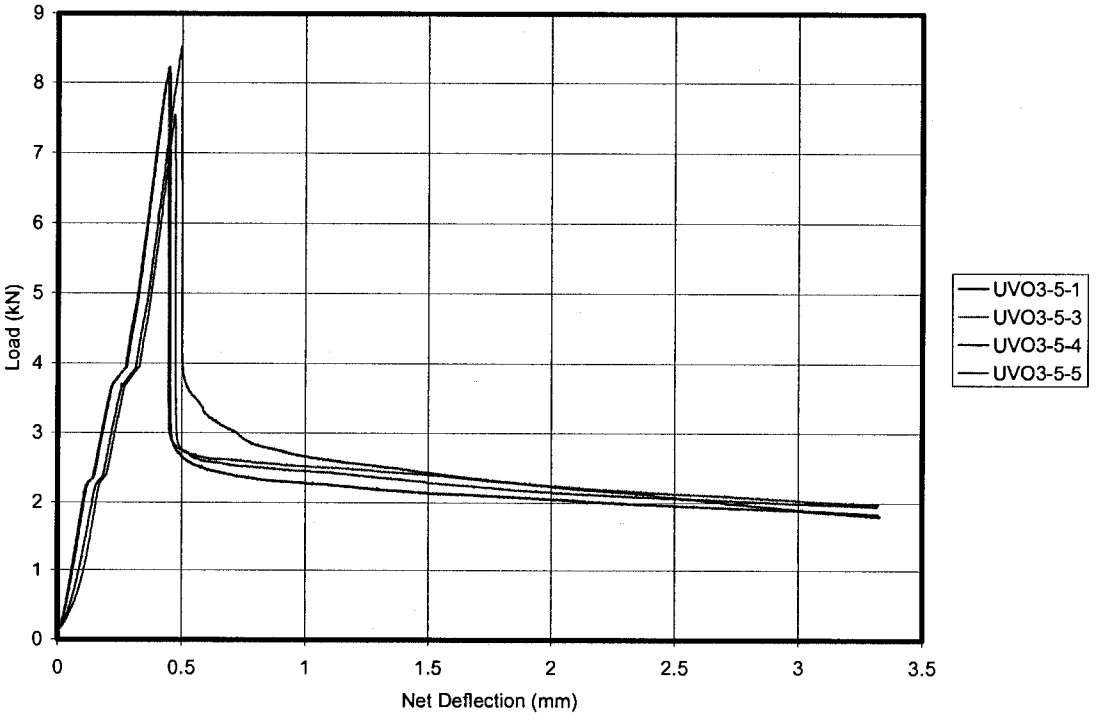

Figure B.16—Modified Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 5 Minutes (UVO_3-5), 28 Days.

Table B.26—Flexural Strength Test Results for Fiber Surface Treated by UV and Ozone for 5 Minutes (UVO_3-5), 28 Days.

UVO_3-5	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	8,223	4.25	0.45	2,357	2,129	1,876	1.22	1.10	0.97	7.1
3	8,105	4.19	0.45	2,588	2,389	2,021	1.34	1.23	1.04	7.6
4	8,515	4.40	0.50	2,897	2,411	1,876	1.50	1.25	0.97	7.8
5	7,544	3.90	0.47	2,521	2,276	1,981	1.30	1.18	1.02	7.2
Average	8,097	4.18	0.47	2,591	2,301	1,939	1.34	1.19	1.00	7.4

B.5.2.2 40 Minutes UV and Ozone (UVO_3-40) Results

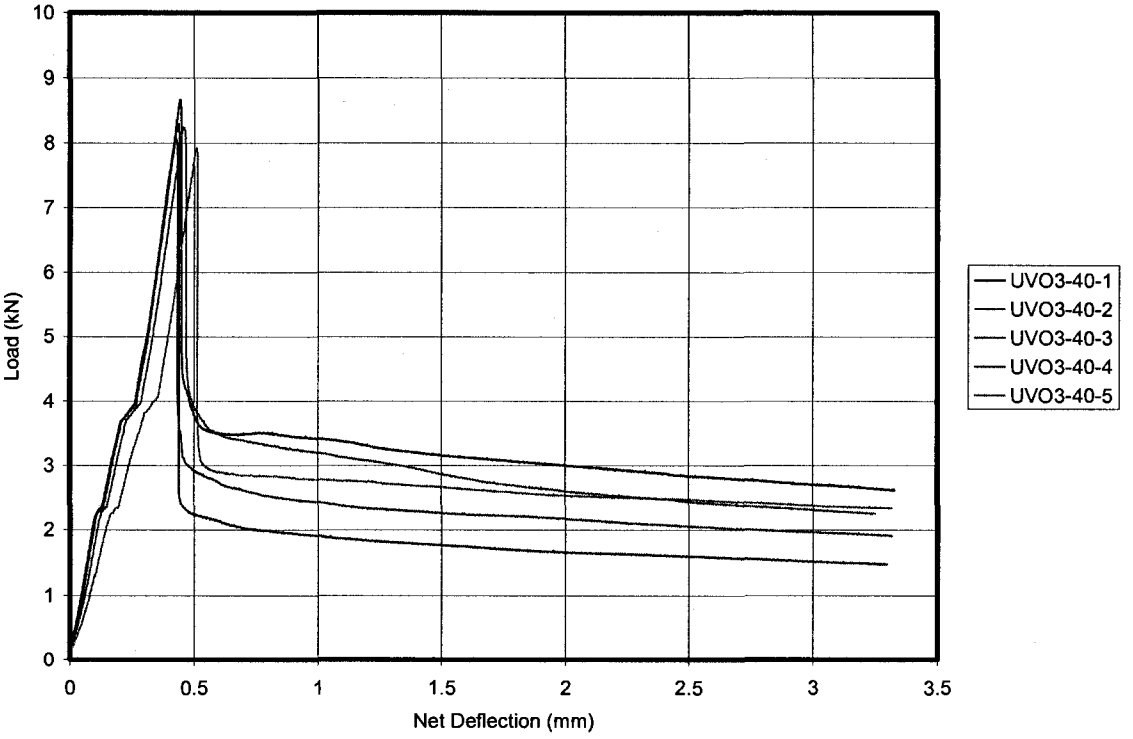

Figure B.17—Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 40 Minutes (UVO_3-40), 28 Days.

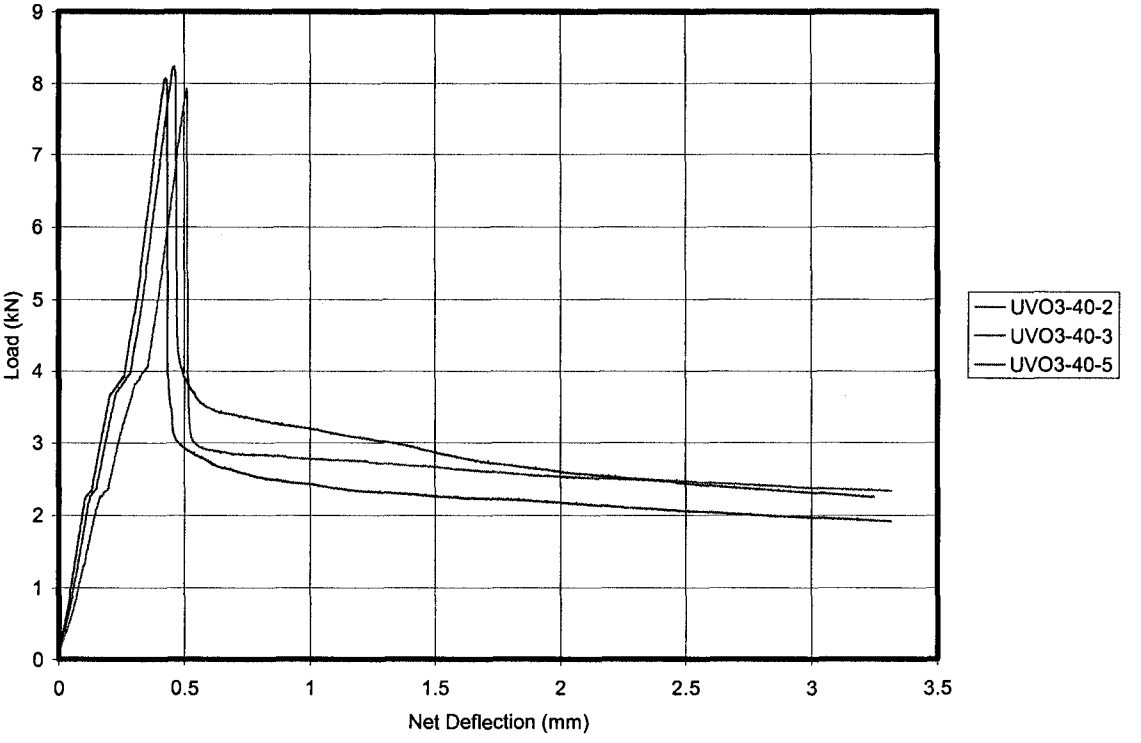

Figure B.18—Modified Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 40 Minutes (UVO_3-40), 28 Days.

Table B.27—Flexural Strength Test Results for Fiber Surface Treated by UV and Ozone for 40 Minutes (UVO_3-40), 28 Days.

UVO_3-40	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
2	7,923	4.09	0.51	2,832	2,650	2,362	1.46	1.37	1.22	8.3
3	8,236	4.26	0.46	3,342	2,856	2,290	1.73	1.48	1.18	8.9
5	8,067	4.17	0.43	2,556	2,252	1,957	1.32	1.16	1.01	7.5
Average	8,076	4.17	0.47	2,910	2,586	2,203	1.50	1.34	1.14	8.2

B.5.2.3 90 Minutes UV and Ozone (UVO_3-90) Results

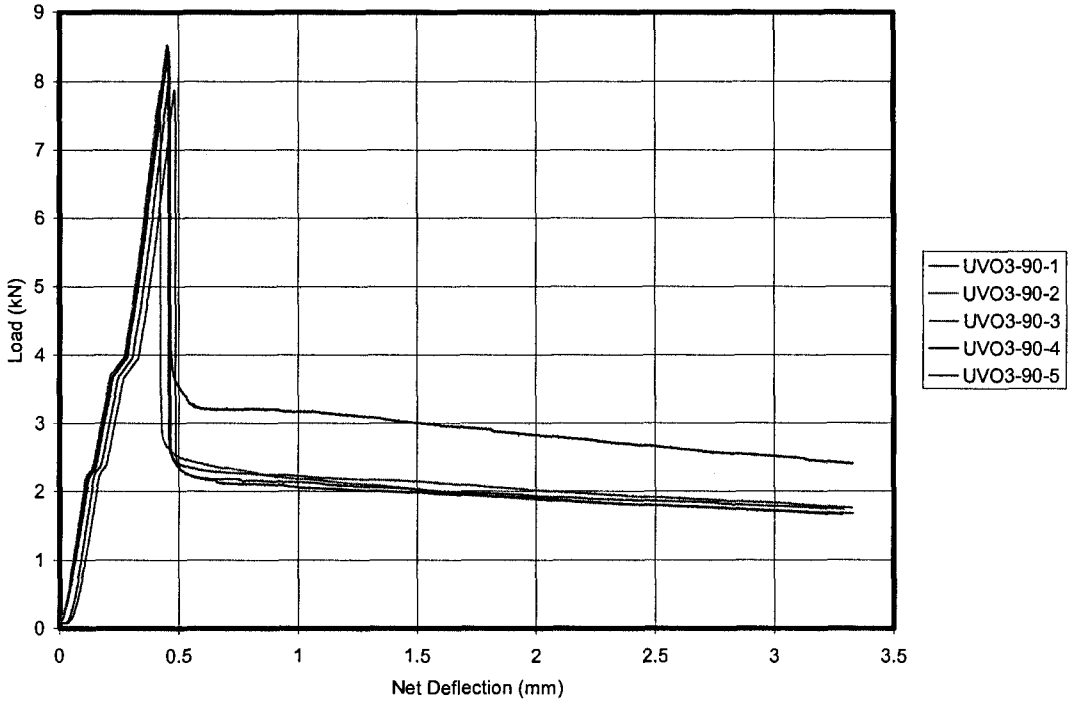

Figure B.19—Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 90 Minutes (UVO_3-90), 28 Days.

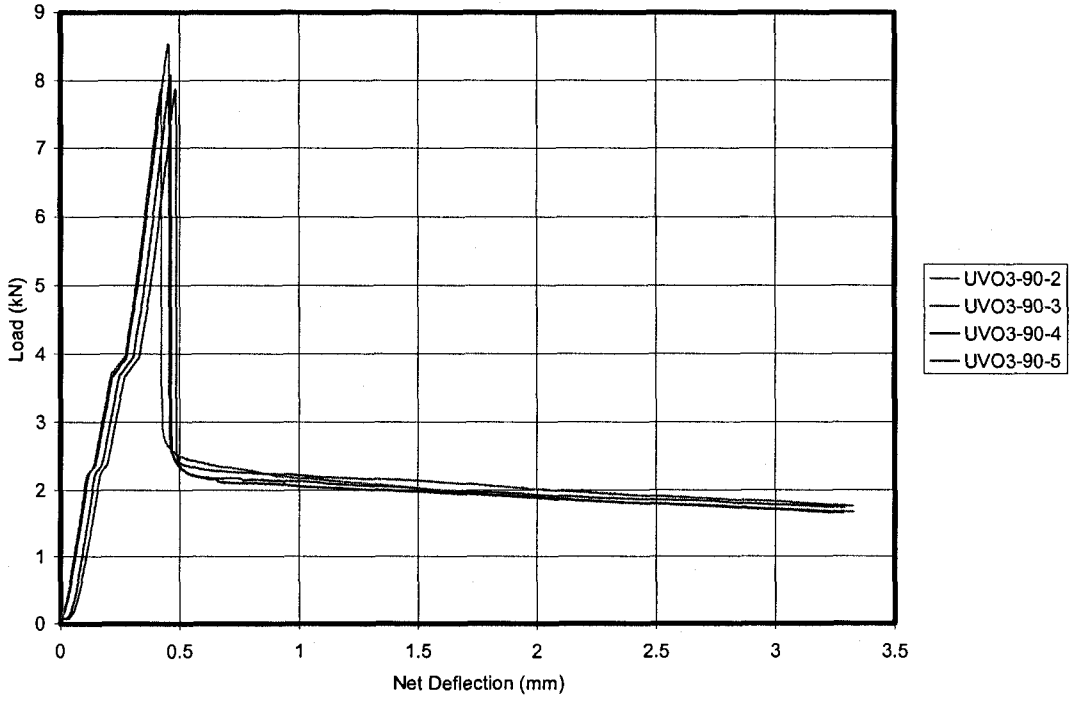

Figure B.20—Modified Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 90 Minutes (UVO_3-90), 28 Days.

Table B.28—Flexural Strength Test Results for Fiber Surface Treated by UV and Ozone for 90 Minutes (UVO_3-90), 28 Days.

UVO_3-90	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
2	7,868	4.07	0.42	2,311	2,019	1,723	1.19	1.04	0.89	6.710
3	8,531	4.41	0.45	2,260	2,140	1,828	1.17	1.11	0.94	7.029
4	8,072	4.17	0.46	2,102	1,973	1,718	1.09	1.02	0.89	6.506
5	7,871	4.07	0.48	2,153	2,019	1,783	1.11	1.04	0.92	6.568
Average	8,086	4.18	0.45	2,207	2,038	1,763	1.14	1.05	0.91	6.7

B.5.2.4 Repeated FRC without Fiber Surface Treatment Results

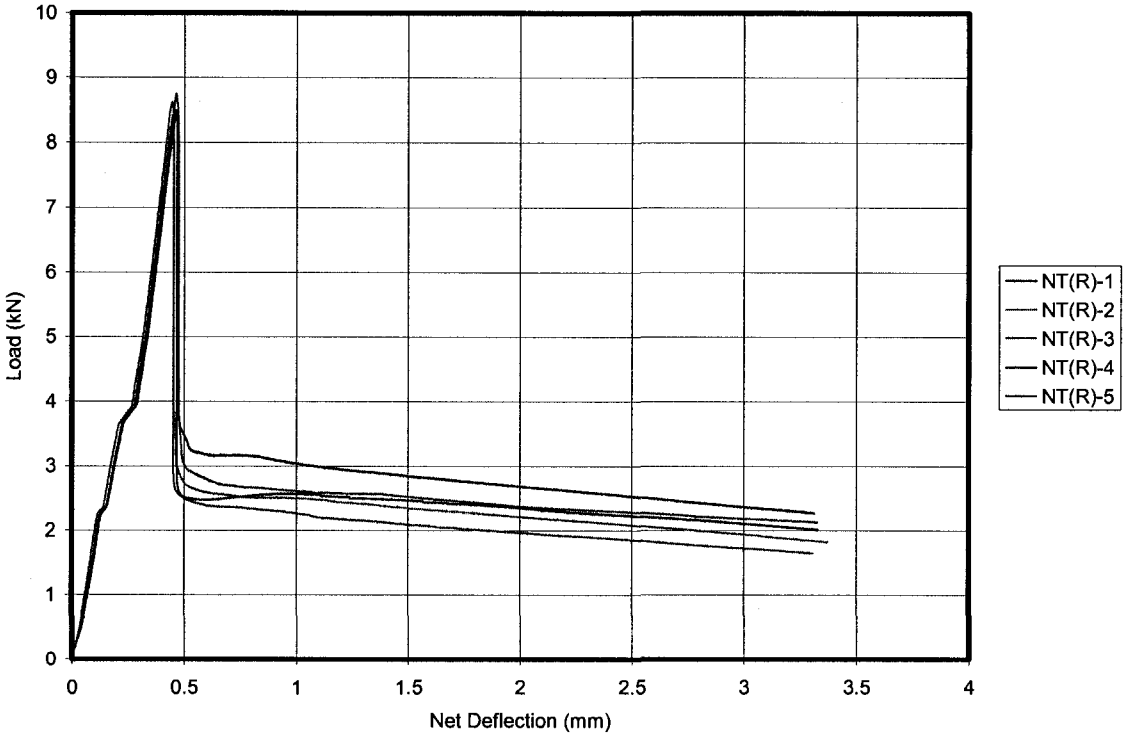

Figure B.21—Load-Deflection Curve for Non-Treated, Middle Dosage (NT, Repeated), 28 Days.

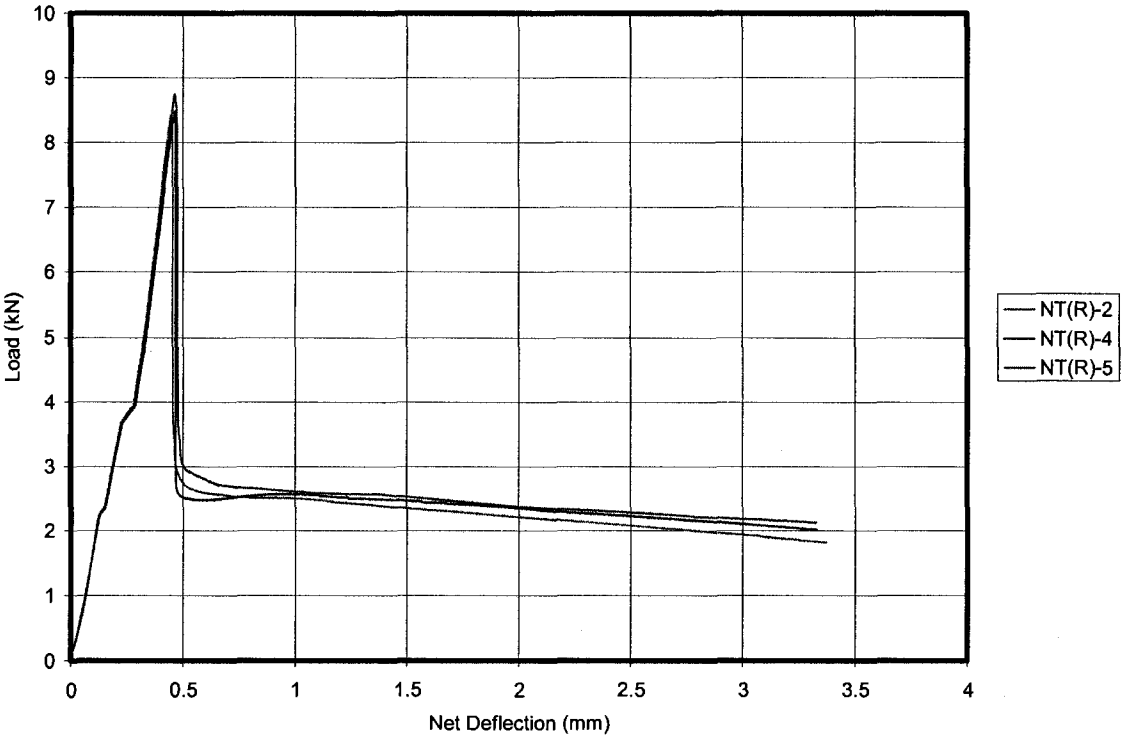

Figure B.22—Modified Load-Deflection Curve for Non-Treated, Middle Dosage (NT, Repeated), 28 Days.

Table B.29—Flexural Strength Test Results for Non-Treated, Middle Dosage (NT, Repeated), 28 Days.

NT(R)	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
2	8,427	4.35	0.45	2,513	2,338	1,927	1.30	1.21	1.00	7.6
4	8,488	4.39	0.46	2,534	2,464	2,091	1.31	1.27	1.08	7.8
5	8,738	4.51	0.46	2,679	2,526	2,177	1.38	1.31	1.12	8.1
Average	8,551	4.42	0.46	2,575	2,443	2,065	1.33	1.26	1.07	7.8

B.5.2.5 Repeated 5 Minutes UV and Ozone (UVO_3-5) Results

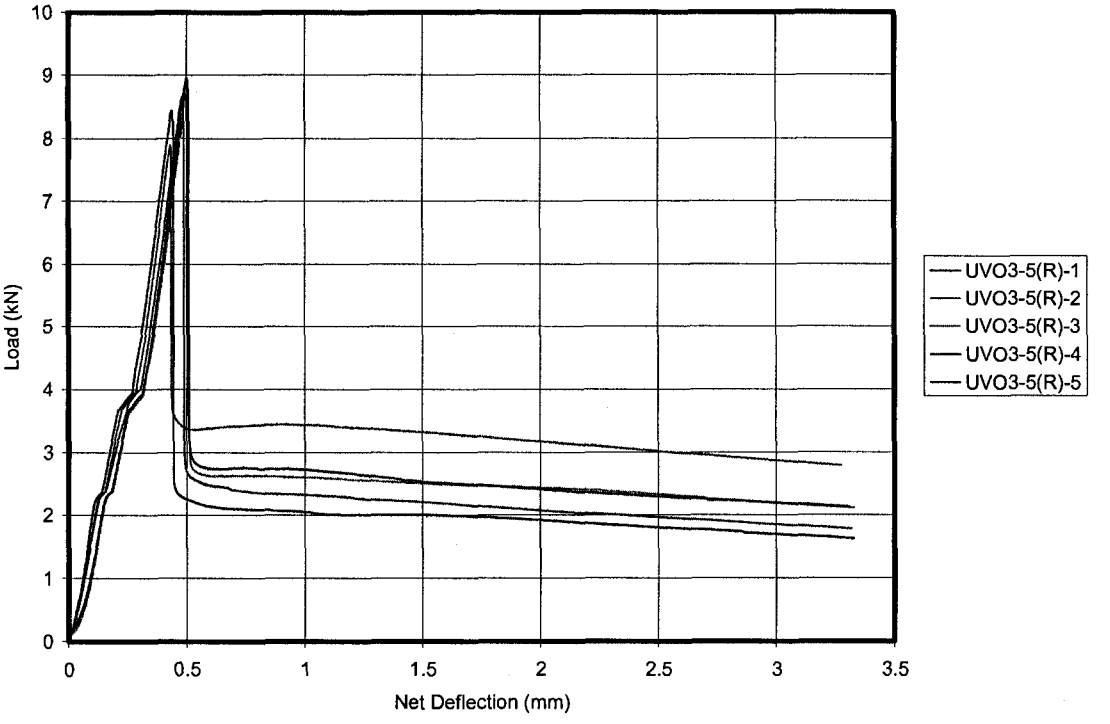

Figure B.23—Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 5 Minutes (UVO_3-5, Repeated), 28 Days.

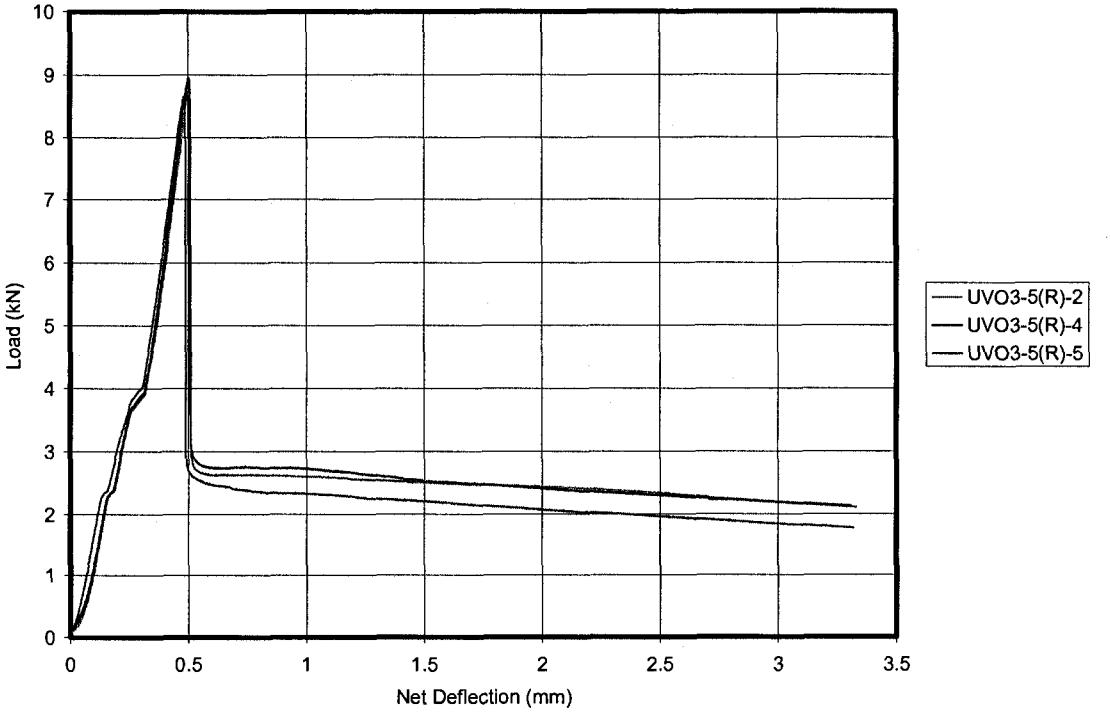

Figure B.24—Modified Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 5 Minutes (UVO_3-5, Repeated), 28 Days.

Table B.30—Flexural Strength Test Results for Fiber Surface Treated by UV and Ozone for 5 Minutes (UVO_3-5, Repeated), 28 Days.

UVO_3-5 (R)	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
2	8,819	4.56	0.50	2,639	2,502	2,174	1.36	1.29	1.12	7.979
4	8,950	4.62	0.50	2,746	2,518	2,188	1.42	1.30	1.13	8.112
5	8,679	4.48	0.49	2,376	2,207	1,836	1.23	1.14	0.99	7.256
Average	8,816	4.55	0.50	2,587	2,409	2,066	1.34	1.24	1.07	7.8

B.5.2.6 10 Minutes UV and Ozone (UVO_3-10) Results

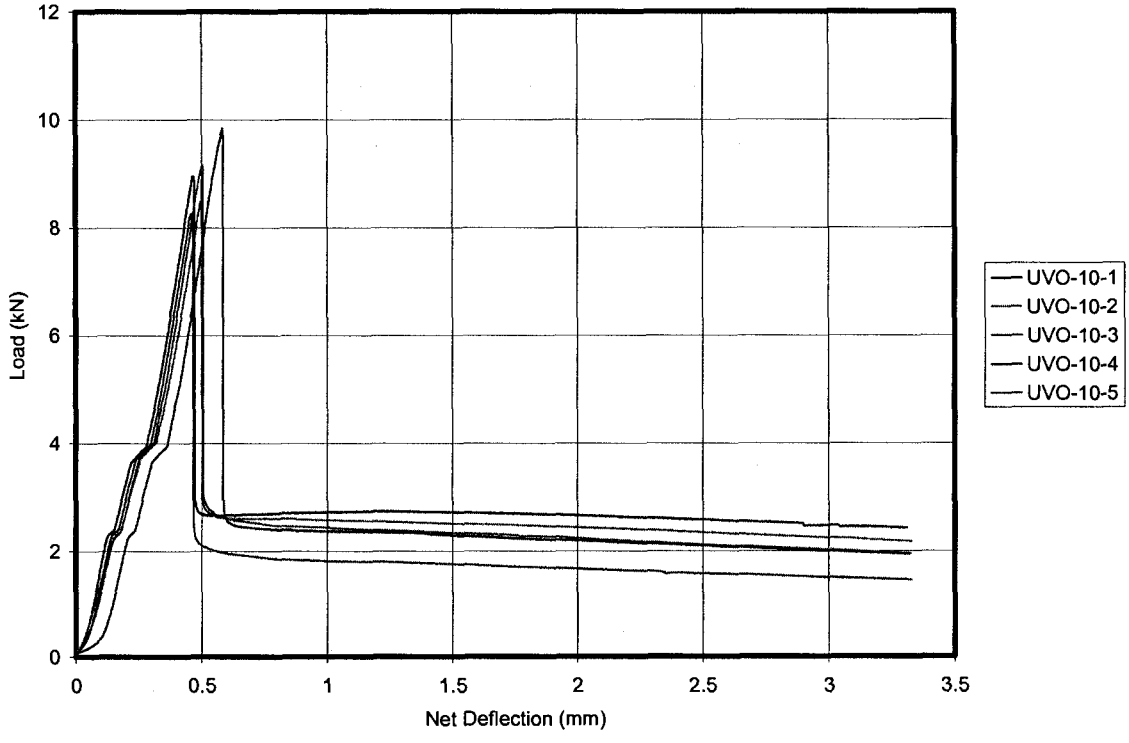

Figure B.25—Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 10 Minutes (UVO_3-10), 28 Days.

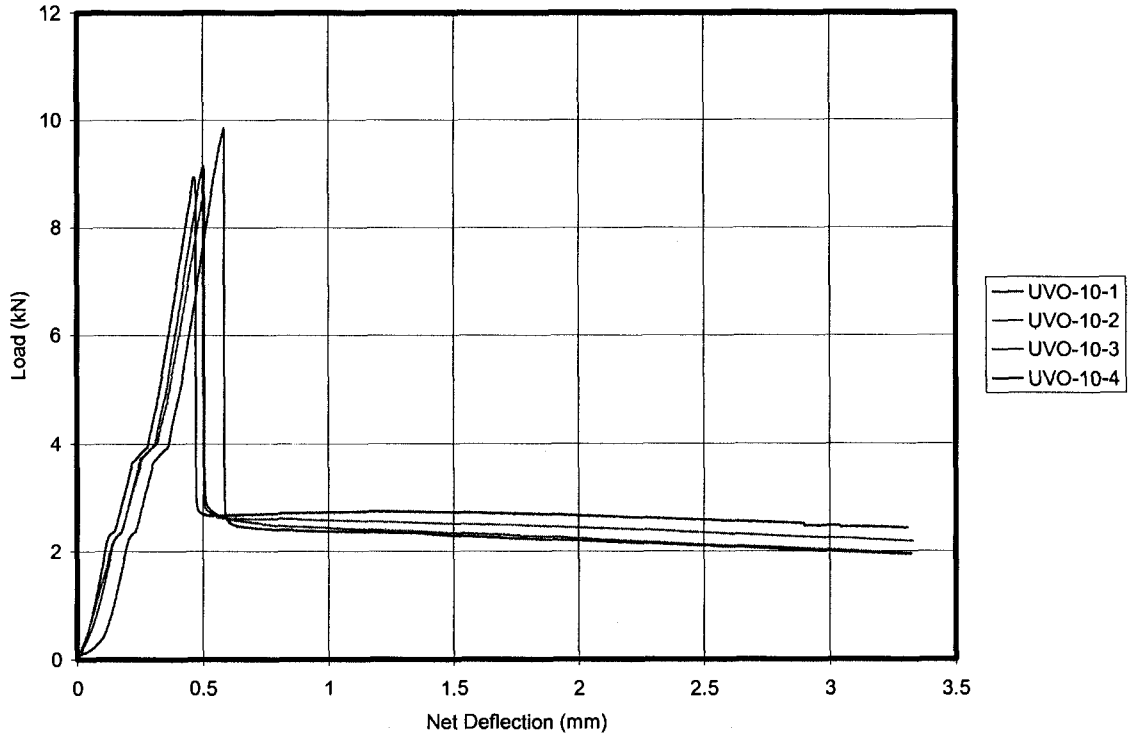

Figure B.26—Modified Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 10 Minutes (UVO_3-10), 28 Days.

Table B.31—Flexural Strength Test Results for Fiber Surface Treated by UV and Ozone for 10 Minutes (UVO_3-10), 28 Days.

UVO_3-10	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	8,937	4.62	0.47	2,703	2,727	2,481	1.40	1.41	1.28	8.6
2	8,494	4.39	0.50	2,601	2,510	2,247	1.34	1.30	1.16	8.00
3	9,157	4.73	0.50	2,491	2,336	2,013	1.29	1.21	1.04	7.7
4	9,847	5.09	0.58	2,413	2,285	1,989	1.25	1.18	1.03	7.6
Average	9,109	4.71	0.50	2,552	2,464	2,183	1.32	1.27	1.13	8.0

B.5.2.7 Repeated 40 Minutes UV and Ozone (UVO_3-40) Results

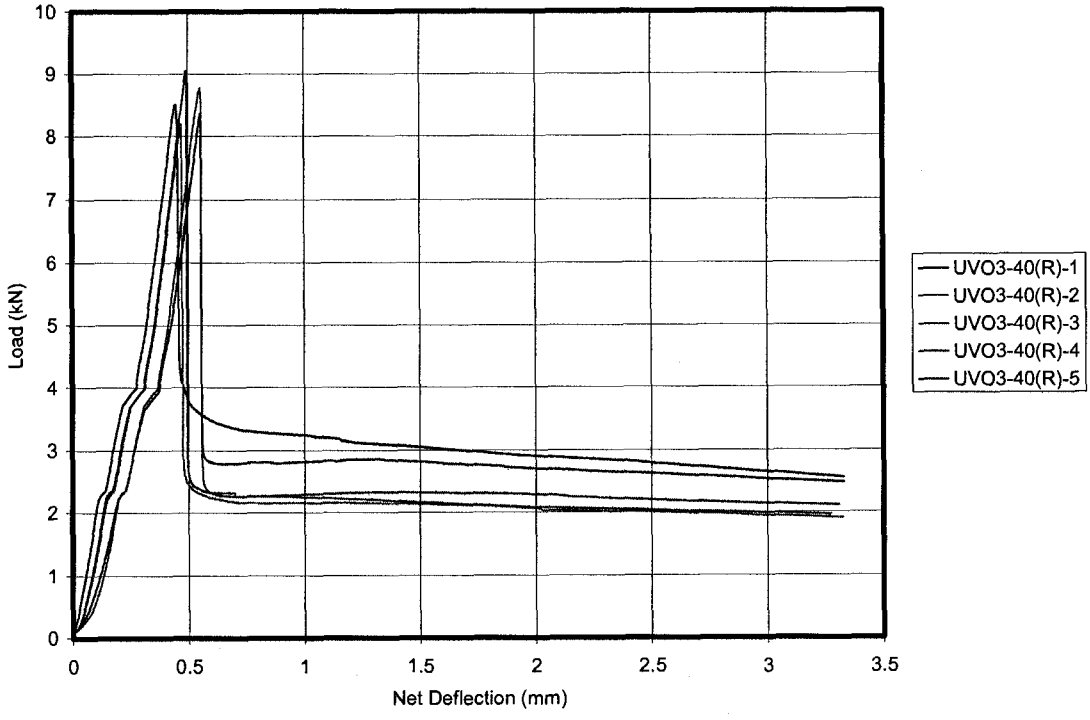

Figure B.27—Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 40 Minutes (UVO_3-40, Repeated), 28 Days.

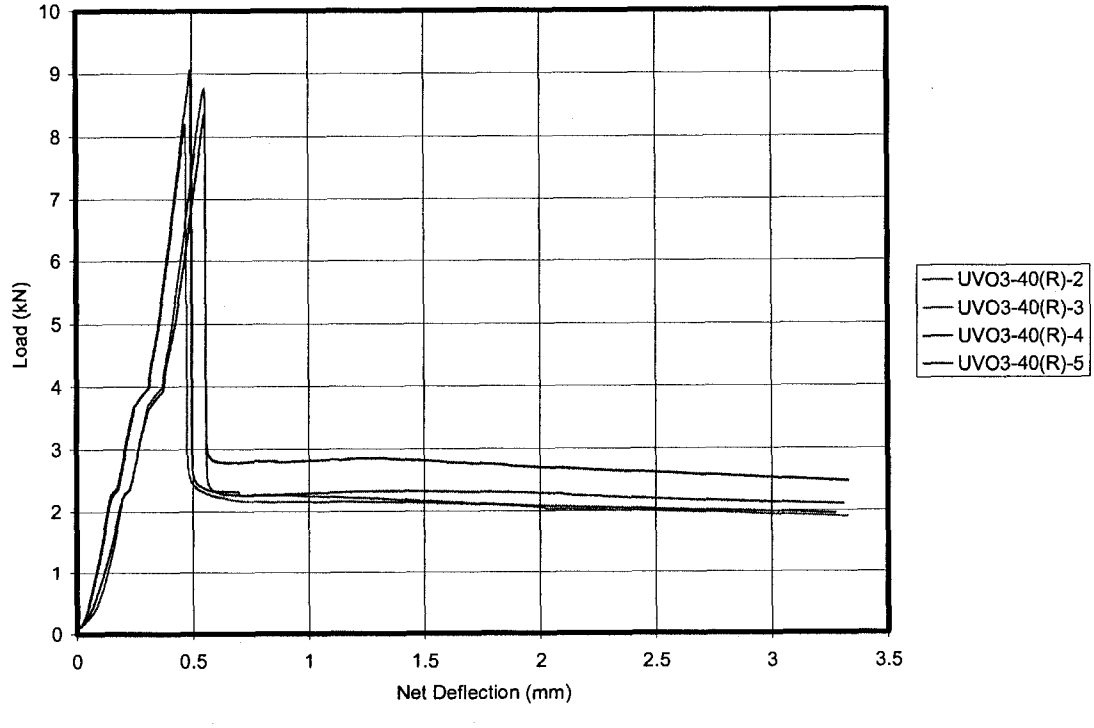

Figure B.28—Modified Load-Deflection Curve for Fiber Surface Treated by UV and Ozone for 40 Minutes (UVO_3-40, Repeated), 28 Days.

Table B.32—Flexural Strength Test Results for Fiber Surface Treated by UV and Ozone for 40 Minutes (UVO_3-40, Repeated), 28 Days.

UVO_3-40 (R)	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
2	8,196	4.23	0.47	2,158	2,137	1,936	1.12	1.10	1.00	6.9
3	8,773	4.53	0.55	2,260	2,156	1,984	1.17	1.11	1.03	7.1
4	8,365	4.32	0.56	2,800	2,800	2,515	1.45	1.45	1.30	8.5
5	9,058	4.68	0.49	2,263	2,314	2,134	1.17	1.20	1.10	7.5
Average	8,589	4.44	0.52	2,370	2,352	2,142	1.23	1.22	1.11	7.5

B.5.3 High Dosage of Fibers Results

B.5.3.1 Non-Treated High Dosage of Fibers Results

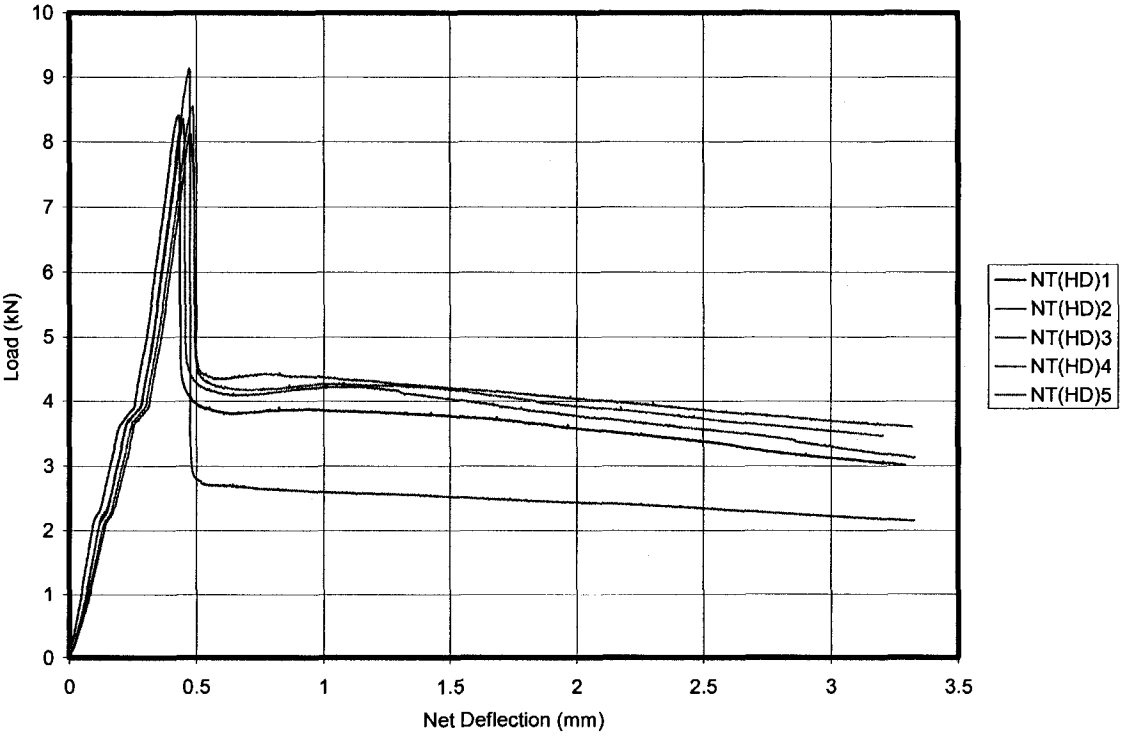

Figure B.29—Load-Deflection Curve for FRC without Fiber Surface Treatment, High Dosage (NT, HD), 28 Days.

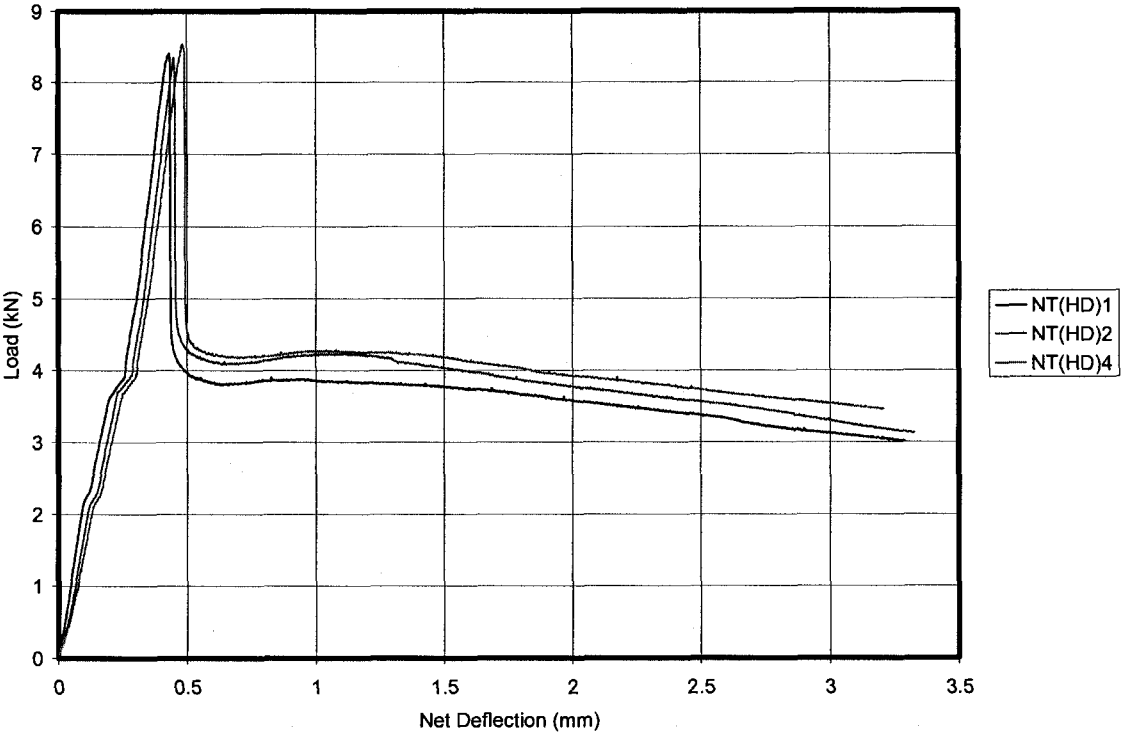

Figure B.30—Modified Load-Deflection Curve for FRC without Fiber Surface Treatment, High Dosage (NT, HD), 28 Days.

Table B.33—Flexural Strength Test Results for FRC without Fiber Surface Treatment, High Dosage (NT), 28 Days.

NT (HD)	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	8,405	4.34	0.43	3,850	3,769	3,114	1.99	1.95	1.61	11.03
2	8,539	4.41	0.49	4,207	4,172	3,517	2.17	2.16	1.82	11.87
4	8,341	4.31	0.45	4,121	4,019	3,264	2.13	2.08	1.69	11.54
Average	8,428	4.35	0.46	4,059	3,987	3,298	2.10	2.06	1.70	11.5

B.5.3.2 Chromic Acid Solution, Type B (Potassium Dichromate), High Dosage Results

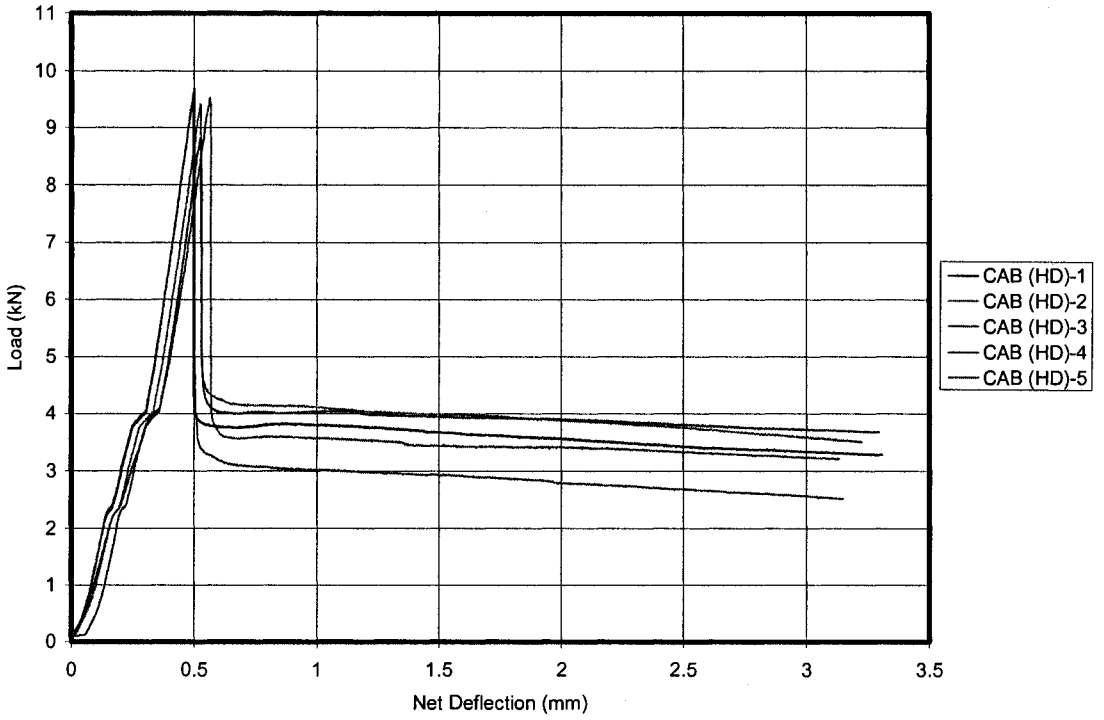

Figure B.31—Load-Deflection Curve for Fiber Surface Treated by Chromic Acid Solution, Type B (CAB, High Dosage), 28 Days.

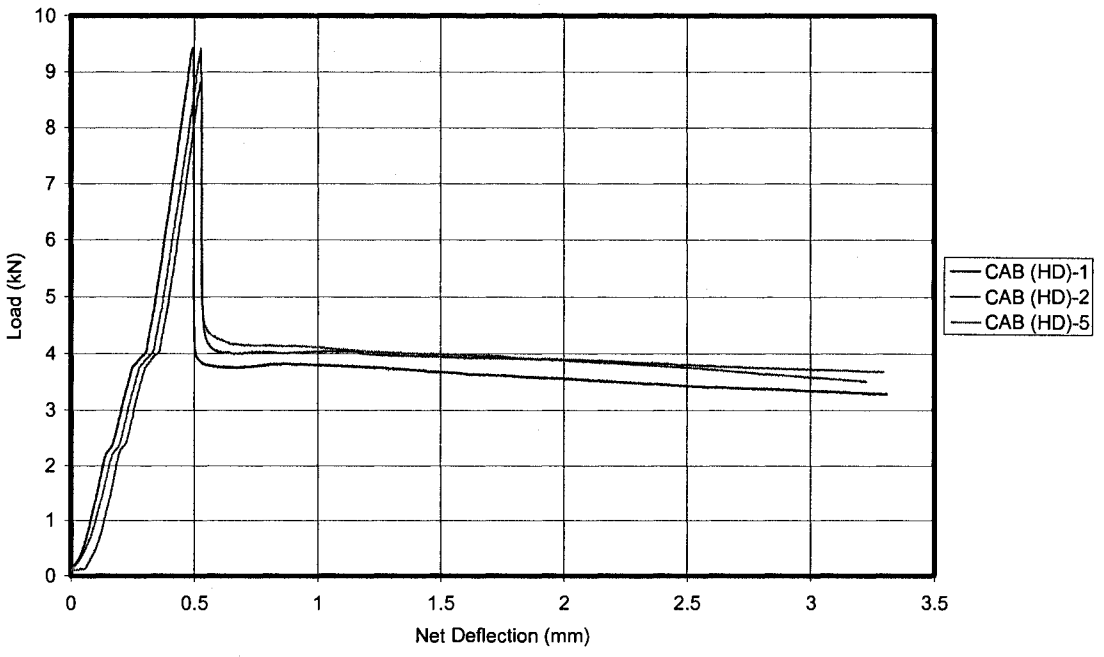

Figure B.32—Modified Load-Deflection Curve for Fiber Surface Treated by Chromic Acid Solution, Type B (CAB, High Dosage), 28 Days.

Table B.34—Flexural Strength Test Results for Fiber Surface Treated by Chromic Acid Solution, Type B (CAB), 28 Days.

CAB (HD)	P_p N	f_P MPa	δ_P mm	$P_{75,0.75}$ N	$P_{75,1.5}$ N	$P_{75,3.0}$ N	$f_{75,0.75}$ MPa	$f_{75,1.5}$ MPa	$f_{75,3.0}$ MPa	$T_{75,3.0}$ Joule
1	9,423	4.87	0.49	3,792	3,671	3,352	1.96	1.90	1.73	11.0
2	9,420	4.87	0.53	4,143	3,984	3,564	2.14	2.06	1.84	11.9
5	8,816	4.55	0.53	4,037	3,933	3,715	2.09	2.03	1.92	11.4
Average	9,220	4.76	0.52	3,990	3,863	3,544	2.06	2.00	1.83	11.4