



A COMPARATIVE STUDY OF THE WETTABILITY OF STEEL, CARBON, AND POLYETHYLENE FIBERS BY WATER

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ABSTRACT

The wettability of fibers by water was found to increase in this order: polyethylene fiber, steel fiber, and carbon fiber. The order applies whether the fibers were surface-treated or not. Treatment with ozone was effective for improving the wettability of carbon and steel fibers. Treatment with acetone was effective for steel and polyethylene fibers. Treatments with HCl and NaOH were more effective for polyethylene fiber than acetone treatment. A contact angle of 0° was achieved by treating carbon fiber with ozone. © 1998 Elsevier Science Ltd

Introduction

The effectiveness of a reinforcement in a cement-matrix composite depends on the quality of the bond between the reinforcement and the cement matrix. This bond is in general poor compared to that between a reinforcement and a polymer matrix. As cement paste is water-based, a good bond between reinforcement and cement depends on the wettability of the reinforcement by water. Good wettability, as shown by a low contact angle, helps the development of a good bond.

The wettability depends on the type of reinforcement (e.g., steel, carbon and polymer) as well as the surface treatment of the reinforcement. Although the wettability of polypropylene fibers (1,2) and steel fibers (3) has been separately reported, with and without various surface treatments, a comparative study of the wettability of various types of fibers has not been previously reported. Because of the difference in conditions of dynamic contact angle measurement among different workers, a comparative study is needed in order to clearly show the differences in wettability among the different types of fibers. This paper is such a comparative study, involving polyethylene, carbon, and steel fibers.

The surface treatment of a fiber can be valuable for enhancing the wettability of the fiber. By surface treatment in the form of surface oxyfluorination, the receding contact angle of water on polypropylene fiber is decreased from 84° to 33° (1). By surface treatment in the form of SiCl₄-plasma activation, the receding contact angle of water on polypropylene fiber

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is decreased from 77° to 58° and the advancing contact angle is decreased from 100 to 66° (2). This paper reports on the effect of surface treatments on the wettability of polyethylene, carbon and steel fibers. These treatments include the use of ozone, acetone, hydrochloric acid, and sodium hydroxide.

Although previous studies on the wettability are limited (1–3), considerable work has been reported on the effect of surface treatment of the fiber on the bond between fiber and cement paste (4,5) and on the mechanical properties of fiber reinforced cement (6–9), in addition to the effect of surface treatment of steel rebar on the bond between rebar and concrete (10–13). In particular, ozone treatment has been reported to be effective for carbon fiber (4,6) and for steel rebar (10,12). Although ozone treatment has been reported to enhance the bond between steel rebar and concrete (10,12), it has not been previously reported for the bond between steel fiber and cement paste. In this work, steel fiber rather than steel rebar was used, partly because dynamic contact angle measurement for assessing the wettability involves measuring the wetting force, and a steel rebar is heavy compared to a steel fiber. It is also because the surface deformations (ribs) on a rebar interfere with the dynamic measurement. On the other hand, acetone treatment has been reported to be slightly effective for steel fiber (5,9) and rebar (13).

Experimental Methods

Steel, carbon, and polyethylene fibers were used in this comparative study. The steel fiber used was low-carbon, cold-drawn XorexTM steel fiber manufactured by Novocon International Inc. (Mt. Prospect, IL). The average diameter was 0.889 mm. The actual diameter of each fiber tested was separately measured. The length was 51 mm. The cross section was circular and uniform (without surface deformation). The fiber conforms to ASTM A820 (specification for steel fibers for reinforced concrete).

The steel fiber was surface-treated by the following methods: 1) washing with acetone (immersion in acetone for 1 h and then drying in air), 2) treating in ozone gas (exposure to an O_2 - O_3 mixture with 0.6 vol.% O_3 at 160°C for 5 min.), and 3) washing with acetone (as in 1)) followed by treating in O_3 (as in 2)).

The carbon fibers were isotropic-pitch-based and unsized, as obtained from Ashland Petroleum Co. (Ashland, KY). They were $15 \pm 3 \mu\text{m}$ in diameter, 690 MPa in tensile strength and 48 GPa in tensile modulus. As-received and ozone-treated fibers were used. The surface treatment involved exposure of the fibers to O_3 gas (0.6 vol.%, in O_2) for 5 min. at 160°C . Prior to O_3 exposure, the fibers had been dried at 110°C in air for 1 h.

The polyethylene fibers were Spectra 900 of Allied-Signal, Inc. (Petersburg, VA). They were $38 \mu\text{m}$ in diameter, 2588 MPa in tensile strength, and 117.3 GPa in tensile modulus. As-received and four types of surface-treated fibers were used. The acetone treatment involved immersion in acetone for 1 h and then drying in air. The HCl and NaOH treatments involved immersion in 0.1 N HCl, 1.0 N HCl, or 1.0 N NaOH solution for 24 h, rinsing with water and then drying in air. Ozone treatment could not be applied because the polyethylene fibers could not withstand the temperature (160°C) required for ozone treatment.

The dynamic contact angle between fiber and doubly-deionized water was measured using the Sigma 70 tensiometer of KSV Instruments (Monroe, CT). The tensiometric method (micro-Wilhemmy technique) was used. The immersion depth was up to 2.5 mm and the stage

TABLE 1
Contact angle (degrees) between fiber and water.

Fiber	Advancing	Receding
Steel, as received	87.2 ± 3	81.4 ± 2
Steel, acetone treated	73.4 ± 2	40.6 ± 1
Steel, O ₃ treated	64.3 ± 2	50.2 ± 2
Steel, acetone then O ₃ treated	63.6 ± 1	32.4 ± 1
Carbon, as received	84.7 ± 2	29.0 ± 1
Carbon, O ₃ treated	0	0
Polyethylene, as received	115.2 ± 2	87.8 ± 1
Polyethylene, acetone treated	93.2 ± 4	60.8 ± 3
Polyethylene, 1.0 N NaOH treated	82.2 ± 3	49.5 ± 2
Polyethylene, 1.0 N HCl treated	78.1 ± 4	28.0 ± 2
Polyethylene, 0.1 N HCl treated	66.5 ± 3	60.0 ± 3

with a beaker of water was moved up (advancing) and down (receding) at a constant speed of 5 mm/min. At least six samples of each type were tested.

Results

The advancing and receding contact angles corresponding to the average over the first three cycles of advancing (increasing immersion depth) and receding (decreasing immersion depth) are shown in Table 1 for each of the as-received and treated fibers. Both advancing and receding angles were decreased by any of the treatments. In both as-received and surface-treated conditions, polyethylene fiber gave the highest contact angles and carbon fiber gave the lowest. Steel fiber was in between. Ozone was so effective for carbon fiber that the contact angles were reduced to zero. For steel fiber, ozone treatment was more effective than acetone treatment. Ozone treatment involved surface oxidation, whereas acetone treatment involved surface cleansing (probably degreasing). Acetone treatment improved the wettability of both steel and polyethylene fibers. However, the effect of acetone treatment on polyethylene fiber is less than those of HCl and NaOH treatments on the same fiber. Among the various treatments on polyethylene fiber, the 0.1 N HCl treatment gave the lowest advancing angle and the 1.0 N HCl treatment gave the lowest receding angle. The effects of HCl and NaOH treatments on polyethylene fiber are similar to those of surface oxyfluorination (1) and SiCl₄-plasma activation (2) on polypropylene fiber. On the other hand, HCl and NaOH treatments are inexpensive compared to oxyfluorination and plasma activation.

The wettability is expected to affect both fiber-matrix bond strength and the degree of fiber dispersion in cement. The superior wettability of carbon fiber compared to polyethylene contributes to explaining the high tensile strength of cement paste containing short carbon fibers compared to that of cement paste containing short polyethylene fibers of the same length and at the same volume fraction (14). The paste with carbon fibers has higher tensile strength, even though the carbon fibers themselves have much lower tensile strength (690 MPa) than the polyethylene fibers themselves (2588 MPa).

Conclusion

The wettability with water was found to increase in the order: polyethylene fiber, steel fiber, and carbon fiber. This order applies whether the fibers were surface treated or not. Ozone treatment was effective for improving the wettability of carbon fiber and steel fiber. Acetone treatment was effective for improving the wettability of steel fiber and polyethylene fiber. HCl and NaOH treatments were more effective for polyethylene fiber than acetone treatment. Ozone treatment was so effective for carbon fiber that the contact angle was diminished to zero.

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