Chemical Treatments of Polypropylene Fiber Surfaces Used in Fiber Reinforced Concretes

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This paper reports the results of an experimental investigation to determine the effects of chemical treatment of polypropylene fibers (PPF) used in reinforced concretes. The control group concrete was designed at 4,000 psi. The chemical solution used to treat the fiber surfaces was a basic solution of linear alcohol alkoxylates. The investigation included comparison of some static strength as well as the interfacial bond characteristics of unreinforced, plain fiber reinforced, and chemically treated fiber reinforced concretes that were cured for two different periods of 28 and 45 days. Three separate methods of testing were conducted to ascertain the mechanical measures of the concrete samples: compression testing (ASTM 4.02, C-39), flexure strength tests (ASTM 4.02, C-78), and splitting tensile tests (ASTM 4.02, C-496). A random sampling of the failed PPF specimens was prepared and observed using the scanning electron microscopy (SEM) technique. The interfacial features of the fiber surfaces were evaluated to determine the bonding characteristics between the fiber and the concrete matrix. A correlation between the chemical surface treatment of the PPF and the mechanical measures were statistically analyzed.

Since the middle of the 1960s, extensive research has been completed relating to the mechanical bonding of fibers in fiber reinforced concrete composites. Such research brought forth the development and the standards for collated fibrillated polypropylene (CFP) (*1, 2*) which is in wide commercial use today.

Naaman et al. (*3*) cited the characteristics of polypropylene fiber (PPF) as being inert chemically and very stable because of the hydrophobic nature of its surfaces. They also enumerated some of the shortcomings of using such fibers, including its poor bonding with the cementitious matrix, particularly for untreated fibers. A careful review of the literature reveals a paucity of research directly related to the chemical bonding effects of PPF in concrete. Moreover, since the efficiency of fiber reinforcement depends on the interfacial bond strength as well as the mechanical properties of the fiber, studies of the interfacial characteristics of the fiber-matrix interface become very important.

To differentiate between fiber failure and fiber pullout mechanisms, a clearer understanding of the bonding and anchorage of fibers in the concrete matrix is needed. It has been established, however, that such bonding of CFP fibers is far less than that found with steel fibers, which in turn affects the energy absorbed in fiber pullout (*4*). Although there currently are few published reports about the chemical treatment of PPF, some recent studies (*5*) indicate that sulfur infiltrated and polymer impregnated fiber reinforced concretes did indeed exhibit better fibers bonding as well as improvements in the overall strength of the concrete composite.

Regarding techniques used in testing fiber reinforced concretes (FRC), many reports cite the static strength characteristics and properties of such composite systems. Some published studies of compression testing of fiber reinforced concretes, however, indicate conflicting data. Early reports, such as those by Hannat (*1*) found plain concrete to be stronger in compression than polypropylene fiber reinforced concrete (PFRC). It was also reported in the *Concrete Construction Journal* (*6*, p. 468) that PFRC enjoys higher compressive strength after 28 days of cure time than to plain concrete. Several researchers in the FRC field contend that standard compression testing should not be the only procedure used to assess the mechanical properties of such composites. Other mechanical measures should be employed to determine the static strength characteristics of FRC composites.

PPFs, in general, are known as low modulus materials and have the potential strength of between 60 and 100 ksi. Since this strength is generated in tension, it would appear that a more appropriate measure for FRC would be tensile or flexure tests. Empirical evidence has shown, however, that concrete by design is strongest in the compressive mode. It is also well known that, in industrial and commercial applications concrete is designed and subjected to flexure, tension and torque. From this assessment, a determination was made to test the FRC composite samples mechanically in flexure, splitting tensile, and compression modes.

An additional intent of this study was to provide a better understanding of the effects of chemically treating the surface of PPF before mixing it in the concrete matrix. A mild linear alcohol base solution was chosen as the agent for this chemical treatment. The chemical composition of the solution was a 28 percent water solution of the linear nonionic alkoxylate alcohol. This commercially available chemical, known as Basic-H, had revealed promise from a previous investigation (*7*), in which it was tested at only one concentration. In the present study, the chemical was administered at two different concentrations. The manufacturer, Shaklee Corporation, claimed that this chemical would improve the ultimate strength of the concrete composites due to its wettability characteristics, which was a persuasive factor in choosing it for this study.

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EXPERIMENTAL DESIGN AND PROCEDURE

A 4,000-psi concrete mix was chosen for this study and also served as the control group (CG) for the modified factorial statistical design. The cementing material used was Type I portland bagged cement, which met ASTM C-150 specifications. The maximum allowable water/cement ratio was 0.47. The coarse aggregate used was a standard ¾-in. top size, AASHTO Size 67 material. The coarse and fine aggregates were obtained from a supplier approved by the Iowa Department of Transportation (DOT). The concrete sand (a natural sand) met fine aggregate requirements for ASTM C-33.

The PPF added to the basic concrete was furnished by a commercial supplier. The fibers were of the D-15 type and met the supplier's requirements for a ¾-in. top size aggregate. The fiber length of the CFP was 2.25 in. The fibers were added to the concrete design mix in the recommended ratio by the manufacturer of 1.6 lb/yd^3 of concrete. This weight ratio was held constant for all the three treatment groups used throughout this study.

As indicated earlier, the chemical chosen for this study was Basic-H, which is furnished by the supplier in full strength as a 28 percent water solution base. The chemical is a nonionic surfactant of linear alcohol alkoxylates. The chemical was diluted by volume fraction with distilled water into two denominations—1:2 and 1:5—for each of the treatment groups. The procedure followed in the chemical treatment of the fiber surfaces and the subsequent soaking and drying of the fibers prior to mixing is described below.

The chemical treatments were prepared first. The PPFs were then weighed and soaked in the chemical bath for 10 min. Next, the fibers were removed from the chemical bath and allowed to air dry. The concrete mix for each treatment group was weighed after drying and then packaged for future use.

The designation of the samples used in this study is as follows:

- CG refers to the control group, which was simply the concrete mix without any fibers added.
- Group A contained plain fibers that had not been treated chemically.
- Group B included fibers that were chemically treated with a diluted solution of 1:2 prior to mixing.
- Group C included fibers that were chemically treated with a diluted solution of 1:5 prior to mixing.

All mixing was done in a drum mixer with a capacity of 5 ft^3. The batching procedure followed ASTM C-92. First, the fine and coarse aggregates were placed in the mixer and rotated dry. Next, three-fourths of the required mixing water was added, and the contents were thoroughly mixed for 1 min. The cement was added next, followed by the remaining water. The fibers were then added, and the concrete was mixed for 3 min. Next followed a 3-min rest period and a final mixing period of 2 min.

There were 12 specimens cast for each test group to meet the required statistical replication factor. The size of the specimens used for both the compression and splitting tensile tests were 6 in. x 12 in. cylinders. The flexure samples were cast as beams with 6 in. × 6 in. × 20 in. dimensions. All the test samples were prepared to conform to ASTM 4.020.

The concrete samples were covered with plastic sheets and cured at room temperature (68°F to 72°F) for 24 hr. The samples were then demolded and placed in a lime-saturated water tank that was maintained at 72°F. Two sets of samples were prepared, with curing periods of 28 and 45 days. After the appropriate time, the samples were removed and tested. The test results were statistically analyzed to determine significant differences among all test groups. A random sampling of fibers was taken from failed specimens for SEM observations. The chunks of concrete containing the designated fibers were mounted and gold coated prior to SEM inspection.

RESULTS

Compressive Strength Tests

The statistical results obtained from the compressive tests were found to be consistent with previously obtained data (*7, 8*). The CG attained the highest compressive strength values among all test groups for both the 28- and 45-day cure periods. The average compressive strength values and their calculated standard deviations are presented in Figure 1.

Splitting Tensile Tests

The data obtained from the splitting tensile tests of the cylindrical samples are shown in Figure 2. As illustrated, the results indicate almost no differences between the CG and the highest values attained by the treatment groups. Curing these specimens for two different cure durations did not result in any observable difference in the split tensile values attained by each group.

Flexure Strength Tests

As shown in Figure 3, the flexure strength tests clearly indicate that the FRC groups assumed part of the applied loads. In two of the three treatment groups, the FRC outperformed the CG with plain concrete.

SEM Observations

A previous study (*7*) reported the morphology of untreated plain fiber surfaces taken from a failed specimen. Small CH crystals with sharp edges were seen to be the major precipitates on these fiber surfaces. The density of these precipitates, however, was not high but appeared to be rather scarce. The same study reported that, when the fibers were presoaked in a full-strength Basic-H solution, more CH crystals with the same morphological features precipitated on the fiber surfaces. The density of these precipitates was still not of high magnitudes. In the study described in this paper, the precipitated crystals retained the same morphological features as reported before when the fibers were soaked in diluted solutions of Basic-H. Figure 4 illustrates this finding for treatment group C, which is representative of both groups B and C. The amount of the precipitated crystals, however, seem to be more dense as a function of the dilution of the solution than that

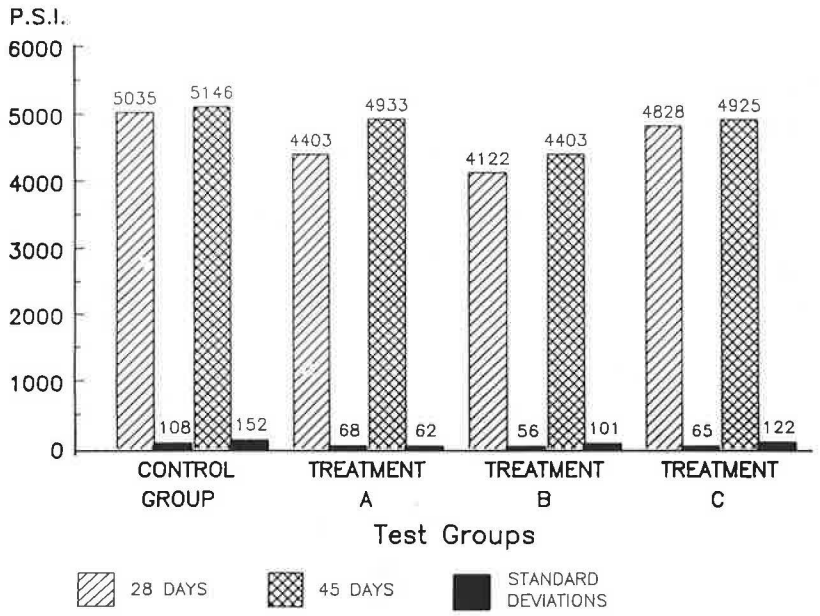

FIGURE 1 Compressive strength test results: comparison between the 28- and 45-day cure time.

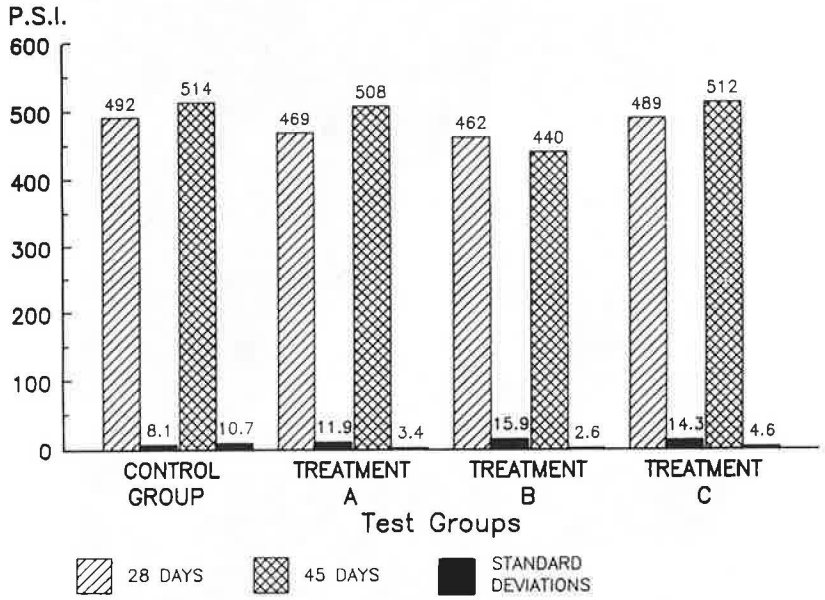

FIGURE 2 Splitting tensile test results: comparison between the 28- and 45-day cure time.

reported earlier. It should be noted that the fibers taken from the split tensile specimens also showed some areas with clear tears. It appears as if the fibers started to fail in shear rather than pullout from the matrix under load.

DISCUSSION

The date obtained from the compressive strength tests indicate that there is almost no change in this property due to fiber reinforcement and chemical treatment. This finding supports what Zollo (*8*) reported a few years ago. In his study, Zollo also concluded that the compressive test does not indicate the fiber contribution in the reinforced concrete samples. One observation that is worth recording is that, during the compression testing of cylinders in the course of this study, one of the cylinders failed and could no longer support further loads even though its surface did not show any major visible cracking. In this case, the specimen obviously failed because of a network of microcracks while the PPFs resisted the propagation of any major cracks to the cylinder surface. Such an observation substantiates the findings of ACI Committee 544

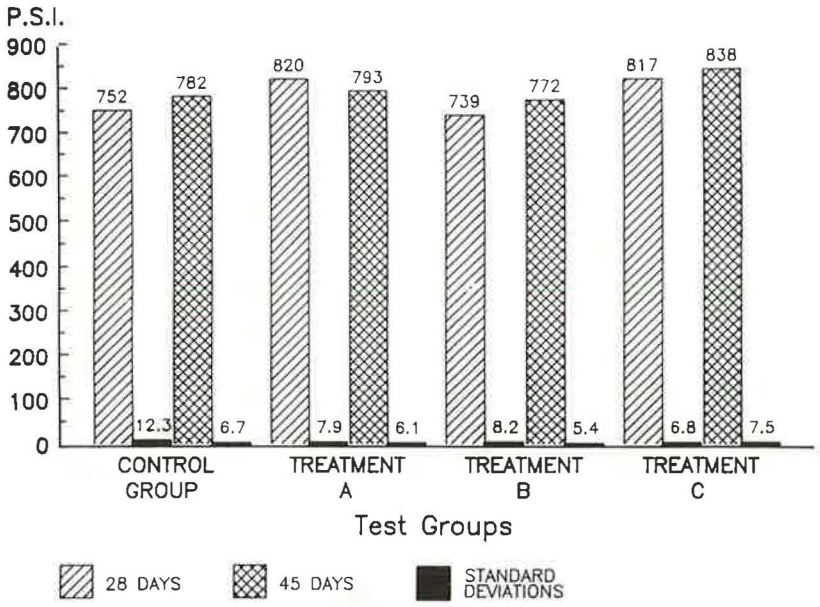

FIGURE 3 Flexural strength test results: comparison between the 28- and 45-day cure time.

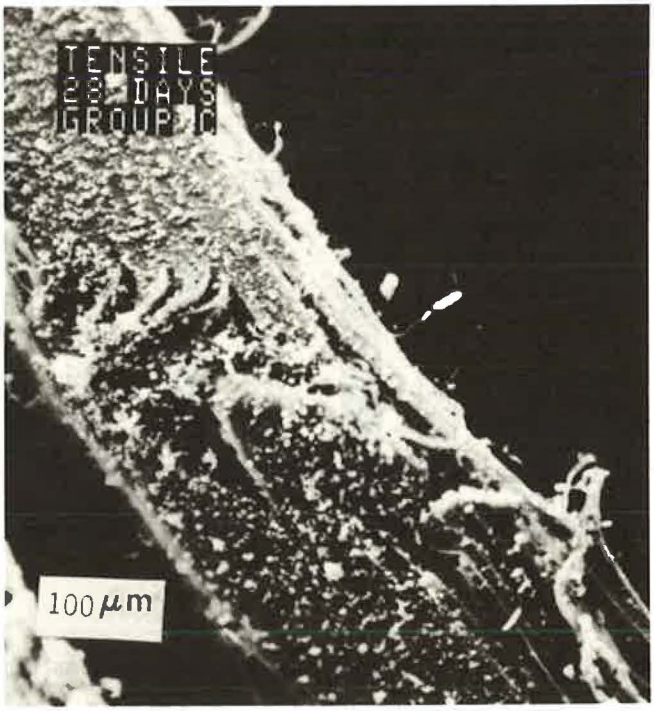

FIGURE 4 Scanning electron micrograph of fiber surface morphology of a failed fiber from the splitting tensile test of treatment group C (28-day cure).

(*5*). This committee concluded that major cracking in concrete beams is usually preceded by slow microcrack growth, which leads to major crack development and the subsequent failure of the beams. This phenomenon also indicates that the PPFs are very efficient in load transfer, especially at peak loading and post loading, where the fibers are believed to have assumed nearly the total applied load.

The post peak load transfer phenomenon was discussed by Ramakrishnan (*9*). During the course of this study, it was noticed that the applied load at failure was sustained for up to 15 sec on the average. This further indicates the ability of fibers to continue being load-bearing members of the composite and to contribute to the post peak load transfer phenomenon. The fibers, in fact, are so efficient in the load transfer that they started to fail themselves (see Figure 4).

The data obtained from the splitting tensile tests revealed

no clear improvement in composite strength, perhaps because this type of testing, as viewed by many researchers, is not in itself a measure of the tensile properties of the composites. Rather, it is another measure indicative of the compression properties of these composites.

The statistical flexure results have the most promising possibilities. Statistical improvement of the FRC becomes apparent when compared to the CG of plain concrete. The almost 10 percent improvement in flexural strength of both group A and the chemically treated group C over the CG is in agreement with Zollo's findings (*8*).

As indicated in the tests and in the figures presented earlier, the 45-day curing resulted in better mechanical static strength properties in almost all cases.

Regarding the bonding between the PPFs and the matrix, the soaking of fibers into the chosen solution prior to mixing improved the wettability of the fiber surfaces. The nonionic linear alcohol alkoxylates present in the solution did indeed promote the formation of CH crystals on the fiber surface. As reported earlier (*7*), the pre-treatment of the fiber surfaces prompted a localized effect on the cement matrix in the fiber vicinity, which resulted in the precipitation of the CH crystals on the otherwise inert fiber surface. The mechanical interaction between the fiber surface asperities and the matrix provided the friction forces necessary for bonding in spite of the absence of direct fiber-matrix bonding. The dilution of the alkali solution up to a 1:5 ratio resulted in more CH crystal density on the fiber surface, hence increasing the mechanical bonding between the fiber and the matrix.

CONCLUSIONS

- The chemical treatment of PPFs with diluted concentrations up to 1:5 by volume of the Basic-H solution improved the flexural strength of the concrete composite up to about a 10 percent increase.
- The surface treatment of fibers with different concentrations of the Basic-H solution improved the precipitation of CH crystals on the fiber surfaces, hence increasing the mechanical friction bonding of the fiber to the cementitious matrix.
- The mechanical bonding of fibers to the matrix increased due to the precipitation of CH crystals to the extent that the fibers started to fail rather than pull out of the matrix under loading.
- Although the addition of PPFs and the chemical treatment used did not significantly improve such static strength characteristics of the composite as the compressive and split tensile strengths, it did prevent massive major crack propagations through the composite.
- The use of chemically treated PPFs is important as secondary reinforcement and crack control.
- The PPFs sustain the load bearing role in post peak loading (for up to 15 sec in compression loading).

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