

Characterization of Polymeric Fibers as Reinforcements of Cement-Based Composites

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ABSTRACT: In this study, three polymeric fibers (nylon 66, polypropylene, and acrylic) were used to improve the flexural and tension strength of cementitious materials. To characterize the performance of these fibers in a cement matrix, scanning electron microscopy, optical microscopy, dynamic mechanical analysis, tensile strength testing, and alkali resistance test were employed. The performance of cement-based composites containing the fibers was eval-

uated with a flexural strength test. The results indicated that the flexural strength increased with an increasing number of interfacial interactions between the fibers and cement. This finding was supported by dynamic mechanical analysis data. This has great application potential for fibers. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 115: 2779–2785, 2010

Key words: composites; fibers

INTRODUCTION

The use of fibers to strengthen materials that are much weaker in tension than in compression goes back to ancient times. Normally, fibers are used in two ways in cement-based materials:

- As primary reinforcements. Fibers can be used to strengthen cement-based products under flexural/tensile loads. In this case, fibers are used at high volume concentrations in a cement matrix (>2%).
- As secondary reinforcements. Crack creation and propagation in the matrix occur because of different factors. Fibers are used in low volume concentrations (<2%) to resist cracking.^{1,2}

In fact, it can be said that fibers change brittle cementitious products into tough composites that can bear high flexural stresses without rupture or even cracking. The load bearing and energy adsorption properties are most important, especially in some applications of concrete and cement-based materials (e.g., industrial engineering building, pavements, and fiber–cement boards).^{3–7} Fibers show rupturing, pull-out, bridging, and/or bonding to a cement matrix under a flexural load; Figure 1 shows the performance of fibers in a cement matrix at the crack tip.

Different fibers (natural and synthetic) are already used for the reinforcement of cement composites. Fiber–cement sheets, which are usually used for roofing, siding, cladding, and so forth, need ductility and energy adsorption capacity. Therefore, fibers as reinforcement materials are used in high volume percentages in such products. The mechanical performance of these sheets is usually determined with a three-point bearing flexural strength test.⁸

The performance of different low-modulus fibers in a cement matrix has been investigated by many authors.^{1,9–20} On the basis of this research, it is well known that nylon and polyester fibers are not alkali-resistant. Hence, they are not considered for the reinforcement of cementitious materials anymore.^{6,16–18} In contest, it has been found that acrylic fibers have better adhesion to cement than other low-modulus fibers.

In this work, three Iranian low-modulus fibers [nylon 66 (N66), polypropylene (PP), and polyacrylonitrile (PAN)] were used to improve the flexural strength of a cement paste. The specimens were tested for flexural strength with a three-point bearing test. Then, the physical/mechanical and chemical properties of the fibers were determined with tensile strength testing, optical microscopy, alkali resistance testing, and dynamic mechanical analysis (DMA). To investigate the fiber–cement interface, scanning electron microscopy (SEM) microphotographs were prepared, and interfacial interactions were studied. Finally, a correlation between the flexural behavior of the composites and the mechanical properties of the fibers was found.

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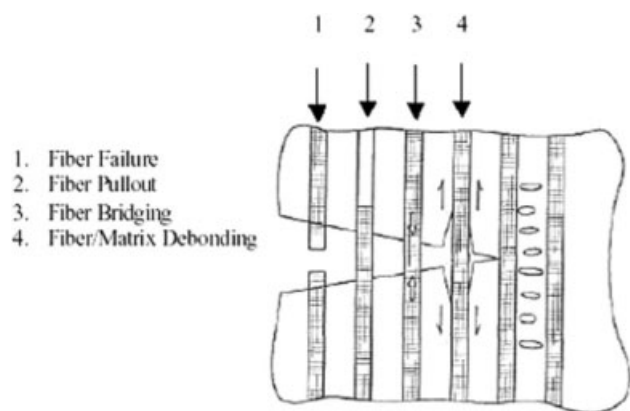


Figure 1 Fiber performance in the cement matrix at the crack tip.

EXPERIMENTAL

Materials

Cement

ASTM type II Portland cement was used in this investigation. The chemical composition and physical properties of the cement are shown in Table I.

N66 fibers

The N66 fibers had a fineness of 6 denier and a diameter of 26 μm . The fibers were tire-cord-grade and had a high molecular weight. They were cut to a length of 3–4 mm with a blade. Figure 2 shows a longitudinal image of the fibers. The typical structure of the N66 fibers is shown in Table II.

PP fibers

Discrete PP fibers (3 mm long) were used in this research. They had a fineness of 3 denier (per filament) and a diameter of 20 μm . Figure 3 shows a longitudinal image and Table II shows the typical structure of these fibers.

TABLE I
Chemical Analysis of the Cement

Chemical composition	Result (%)
SiO ₂	19.72
Al ₂ O ₃	3.65
Fe ₂ O ₃	4.2
MgO	3.4
CaO	60.48
SO ₃	2.14
Loss in ignition	4.76
Insoluble residue	0.46
C ₃ S	59.71
C ₂ S	11.49
C ₃ A	2.57
C ₄ AF + 2C ₃ A or C ₄ AF + C ₂ A	17.91
Na ₂ O + 0.0658K ₂ O	0.75

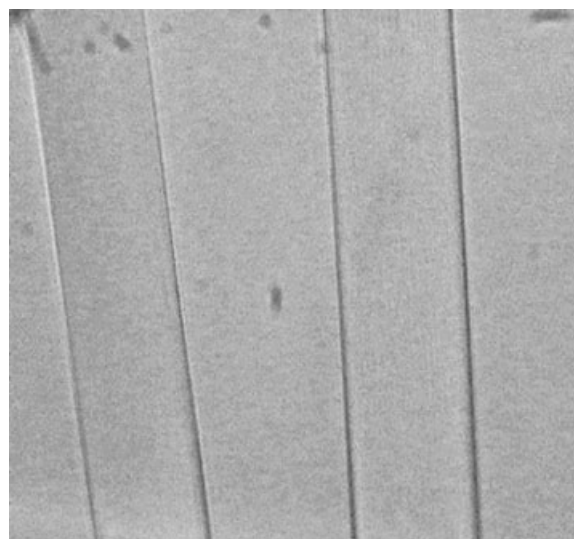


Figure 2 Longitudinal image of N66 fibers prepared by optical microscopy.

Acrylic (i.e., PAN) fibers

These fibers were PAN. They were textile-grade with a fineness of 4 denier (per filament). Apart from other fibers, the acrylic fibers were bean-shaped. The dimensions of these fibers were 14 \times 26 μm^2 . They were cut to a length of 3–4 mm with a blade in the laboratory. Figure 4 shows a longitudinal image and Table II shows the typical structure of these fibers.

Test apparatus

Flexural strength tester

The flexural stress of the fiber–cement sheets was evaluated with a Hounsfield H5KS apparatus with a three-point bearing clamp (see Fig. 5). The flexural force was determined continuously at different extensions (millimeters), and a force–extension curve was drawn with the instrument.

TABLE II
Typical Structures of PAN, PP, and N66 Fibers

Fiber type	Typical structure
N66	$\left[\text{N} \begin{array}{c} \text{H} \\ \\ \text{CH}_2 \end{array} \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{N} \begin{array}{c} \text{O} \\ \\ \text{C} \end{array} \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{CH}_2 \text{C} \begin{array}{c} \text{O} \\ \\ \text{C} \end{array} \right]_n$
PAN	$\left[\begin{array}{c} \text{N} \\ \\ \text{C} \\ \\ \text{H} \end{array} \text{C} \begin{array}{c} \text{H} \\ \\ \text{C} \\ \\ \text{H} \end{array} \right]_n$
PP	$\left[\begin{array}{c} \text{CH}_3 \\ \\ \text{C} \end{array} \text{C} \right]_n$

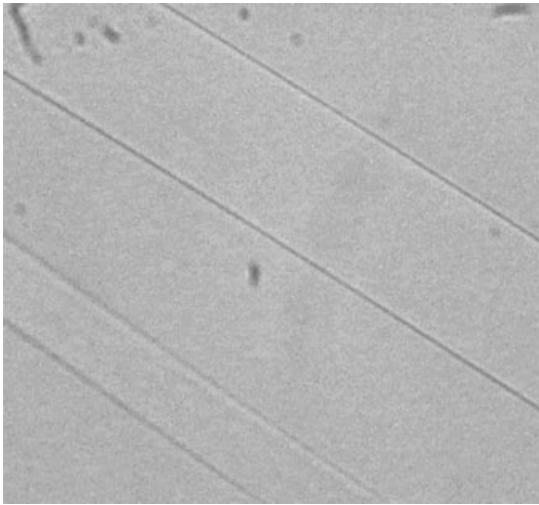


Figure 3 Longitudinal image of PP fibers prepared by optical microscopy.

Specimen preparation apparatus

This is a well-known apparatus for sample preparation among asbestos cement sheet manufacturers. It was operated on the basis of dewatering from a dilute suspension of a fiber-cement mix and then pressing before the removal of the sheet from the die. The produced specimens had dimensions of about $200 \times 100 \times 6\text{--}10 \text{ mm}^3$.

The specimens were tested after 14 days of curing: 1 day in a humidity chamber and 13 days under the ambient conditions.

Tests and analysis

SEM analysis

This analysis was carried out with a Cambridge 1990 S360 instrument. The instrument was an old machine

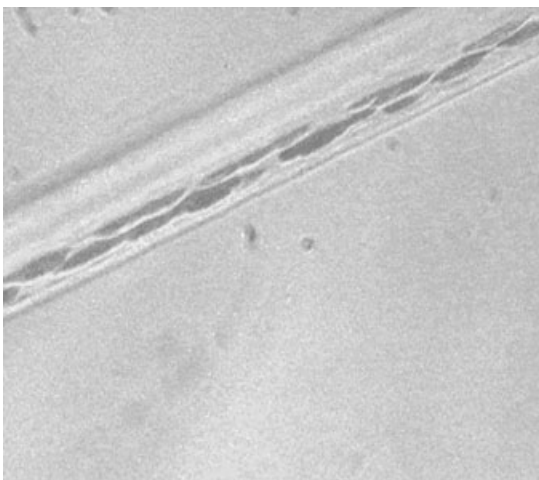


Figure 4 Longitudinal image of acrylic fibers prepared by optical microscopy.

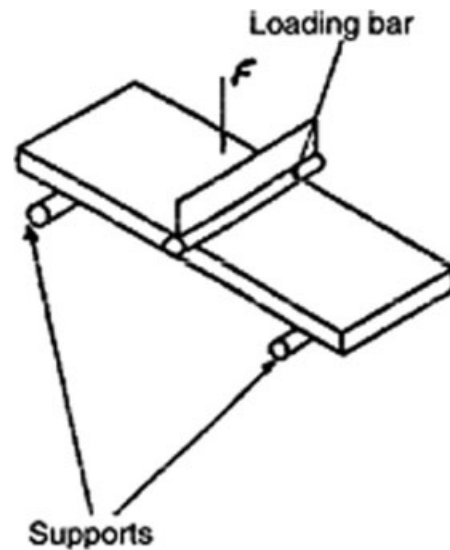


Figure 5 Schematic of the flexural strength testing apparatus (EN 12467).

that could not detect light elements similar to oxygen with respect to X-rays (energy-dispersive spectrometry analysis), but it had very good resolution for SEM images.

DMA

DMA was carried out with a TA Instruments 2980 apparatus. The temperature was constant, and the tensile strain was oscillated with an amplitude of 0.5% and a frequency of 1 Hz.

Tensile strength of the fibers

The tensile strength of different fibers was determined with a Fafegraph M (Textechna) equipped with a Vibromat M.

Then, the fibers were left in an alkali solution of sodium hydroxide (pH 12) for 28 days. They were removed and evaluated with respect to the tensile strength and surface degradation.

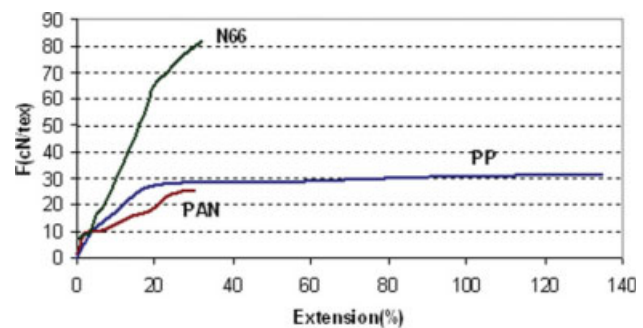


Figure 6 Tensile strength (F) of fibers. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

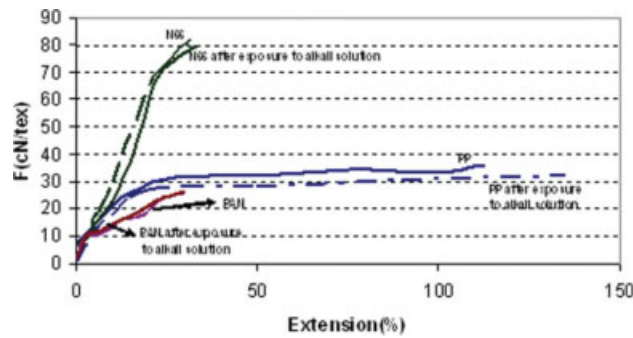


Figure 7 Tensile strength (F) of fibers after 28 days of exposure to an alkali solution. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

RESULTS AND DISCUSSION

Tensile strength of the fibers

Figure 6 shows the results for the tensile strength of the fibers: the tensile strength of the PP fiber is nearly equal to that of the acrylic fiber. In contrast, the elongation of the PP fiber is much more than that of the PAN fiber. In comparison, the N66 fiber has higher tensile strength and lower elongation. This means that with an equal tensile stress (e.g., under the flexure of fiber–cement sheets), N66 fibers show lower extension than the others.

Tensile strength of the fibers after exposure to an alkali solution

Some fibers degrade because of the alkali nature of the cement matrix. The polymeric fibers that were used were tested for alkali resistance, and after that, their tensile strength and surface properties were studied. Figure 7 shows the results for the tensile

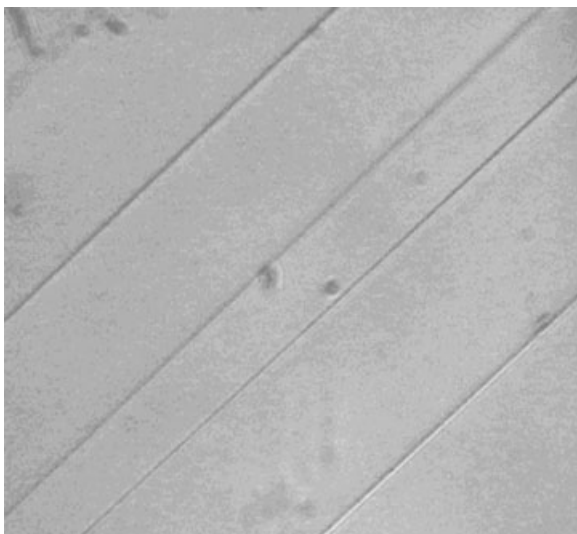


Figure 8 Longitudinal images of PP fibers after alkali aging.

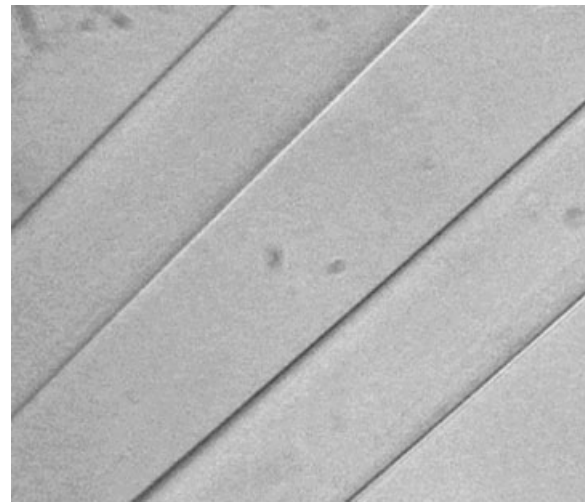


Figure 9 Longitudinal images of N66 fibers after alkali aging.

strength. The tensile strength of the fibers was not affected considerably by a sodium hydroxide solution (pH 12). Therefore, they resist cement alkali conditions, and their properties do not decrease in this matrix.

Longitudinal images of fibers after exposure to an alkali solution were prepared (see Figs. 8–10). These images comply with the results for the tensile strength because any imperfection or degradation due to the alkali reaction was not observed on the surface of the fibers.

DMA

The fibers were characterized with DMA to study their mechanical behavior under cyclic tensile stress. The extension–time results for all the fibers are shown in Figure 11. N66 fibers had the lowest irreversible

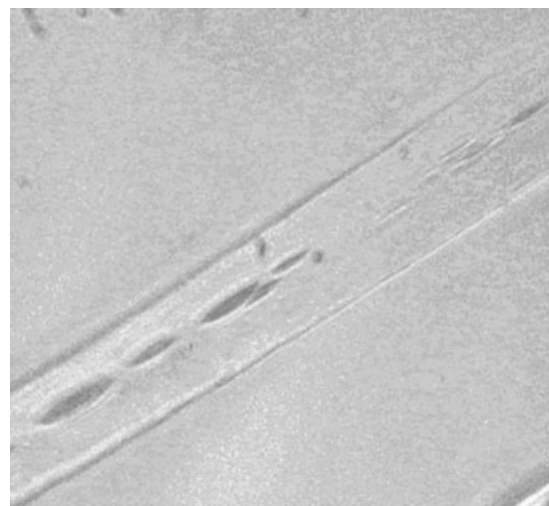


Figure 10 Longitudinal image of acrylic fibers after alkali aging.

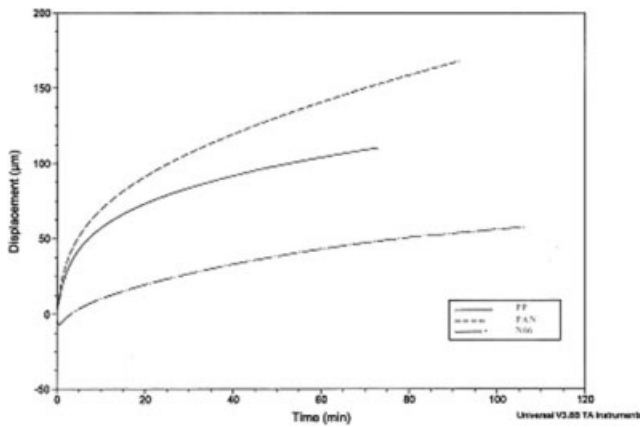


Figure 11 Extension versus time for different fibers.

elongation due to cyclic tension with time. It is well known that irreversible elongation and failure in fibers due to a low modulus and stiffness lead to weak performance of a composite. When a cyclic flexural load is applied to such composites, the fibers do not withstand the load after a short time of loading because of irreversible elongation, so microcracks appear in the matrix, and this is followed by macrocracking and rupturing of the sheet. The results show that N66 fibers have lower irreversible elongation, so they are expected to do better in a cement matrix under flexural testing as well.

Figure 12 shows a curve of $\tan \delta$ versus time for the same fibers. $\tan \delta$ is a representative parameter for showing energy dissipation in materials. The PP fibers have lower irreversible elongation than acrylics, but their energy dissipation is higher than that of PAN fibers. This can be attributed to the crystalline structure of PP fibers, which break under tension.

From these findings, it was postulated that N66 fibers must perform better under flexural strength testing because of the lower loss of energy and irreversible elongation under tension.

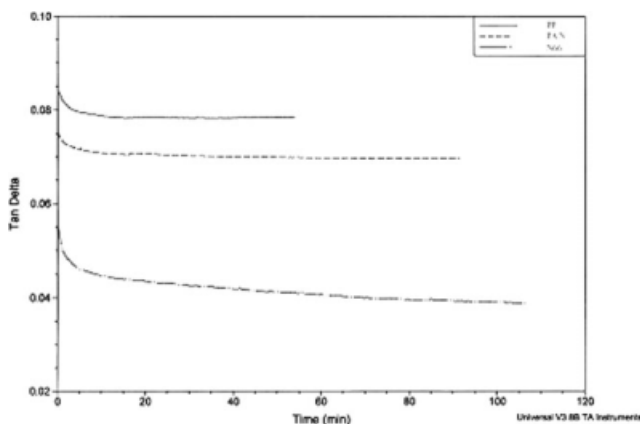


Figure 12 $\tan \delta$ versus time for different fibers.

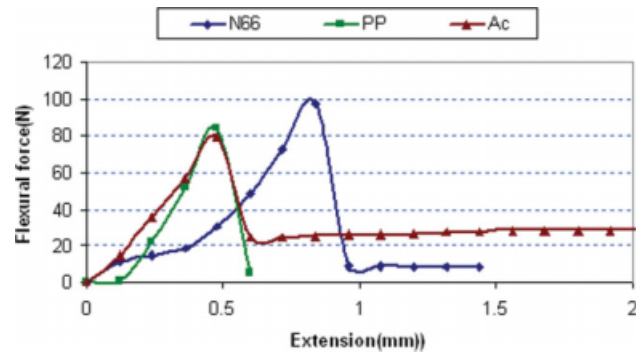


Figure 13 Flexural force versus extension for fiber-cement sheets (Ac = acrylic). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Flexural strength test

The flexural strength test results for 5-mm-thick sheets containing a 1 vol % fiber concentration are shown in Figure 13.

It is evident that the N66 fiber-cement sheet was ruptured at a higher extension and flexural force than the other fibers. The higher slope of the curve (i.e., it is closer to the vertical axes) before maximum flexural force means better performance due to the greater modulus of elasticity of the composite. All the composites were ruptured thoroughly after the maximum flexural force, whereas they were expected to show ductile behavior.

The PP and PAN fibers performed similarly, but the acrylic-containing composite had a little energy adsorption capacity after initial cracking and did not rupture immediately after cracking.

For further information, composites were made with different volume fractions of fibers.^{21,22} The PP, N66, and PAN fibers performed better at 1.2, 3.7, and 2.6 wt %, respectively (Fig. 14). The fibers clearly caused an improvement in the load-bearing capacity of the cementitious composites. Although

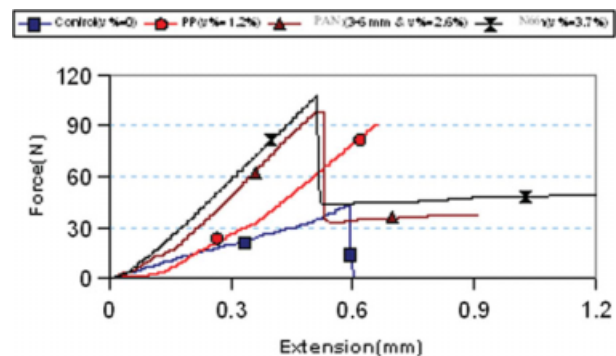


Figure 14 Flexural force versus extension for fiber-cement sheets at the best fiber volume fractions. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

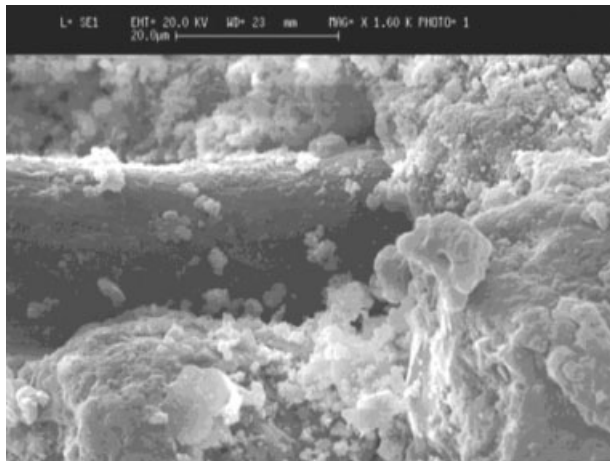


Figure 15 SEM image of the N66–cement paste interface.

the maximum flexural force made by the different sheets was nearly the same, as mentioned previously, it is not suitable for composites to extend more before the maximum flexural force (e.g., in the PP specimen). Therefore, N66- and PAN-fiber-containing cement sheets performed better than PP-fiber-containing composites.

SEM analysis

SEM images were prepared from fiber–cement interfaces, as shown in Figures 15–17. The hydration products of cement could be seen as a continuous film on the surface of the N66 fiber (Fig. 15). There were no pores around these fibers, so good adhesion of the N66 fiber to the cement matrix could be considered. Thus, it can be concluded that there are interactions between N66 fibers and cement hydrates that lead to the formation of primary centers for crystal propagation on the fiber surface. This image confirms the flexural strength and DMA results.

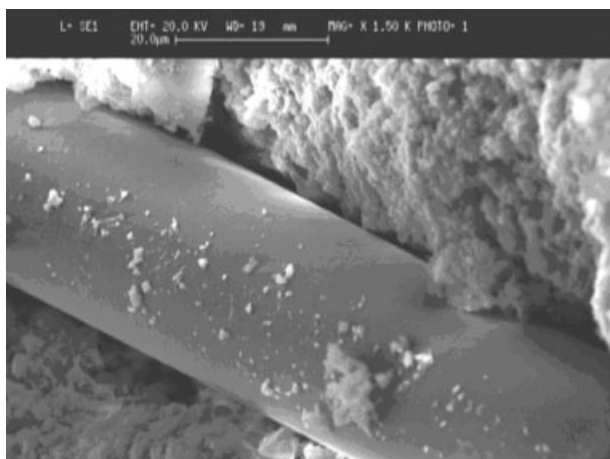


Figure 16 SEM image of the PP–cement paste interface.

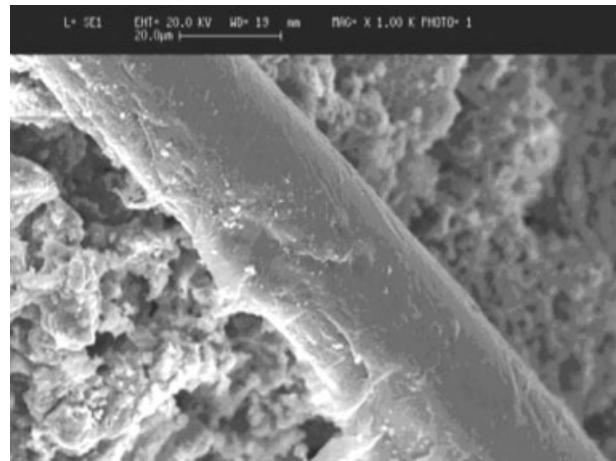


Figure 17 SEM image of the acrylic–cement paste interface.

The PP fibers, as olefin fibers (nonpolar), are hydrophobic. They do not have chemical interactions with cement paste, so they do not show adhesion to it. Figure 16 shows pores around the PP fiber. Therefore, PP fibers will readily pull out from the matrix under a flexural load. This low pullout energy causes the low energy adsorption capacity and lack of ductility in the composite.

Cement hydrate crystals formed on the PAN fibers as a continuous film, showing good adhesion and chemical interactions between the fiber and matrix (Fig. 17). After hydration of the cement, a large amount of energy had to be used to pull out the fibers in comparison with the PP fiber.

CONCLUSIONS

The physical and mechanical properties of polymeric fibers (N66, PAN, and PP) as reinforcements of a cement matrix were studied with different methods. It was found that the nylon fibers had better mechanical properties, especially under a load. They had lower irreversible displacement and energy dissipation than the other fibers, and this could make them candidates for good reinforcements for cement-based composites.

The selected N66 and PAN fibers that were not made for the reinforcement of cement-based materials performed better than PP fibers prepared especially for cement-based composites. Therefore, it can be concluded that they have potential for utilization in cement-based composites.

The flexural strength of the fiber-reinforced cement sheets was evaluated also. These results confirmed the findings from DMA. A good correlation was found between the flexural strength test results and DMA. SEM images were prepared, and they were also evidence for these findings.

It can be concluded that apart from fiber–cement interactions, the mechanical properties of fibers are

the other controlling mechanism for performance. Because of these findings, we suggest the use of DMA to study the feasibility of using fibers in cement-based materials. It is a precise, fast, and simple method for pre-estimating the performance of fibers in cementitious materials.

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