

TAILORING OF POLYPROPYLENE FIBERS FOR THE DESIGN OF SUSTAINABLE FIBER-REINFORCED CEMENT-BASED COMPOSITES

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ABSTRACT

Due to their low cost and widespread availability, polypropylene fibers are an attractive structural reinforcement for fiber reinforced concrete (FRC). But polypropylene is a hydrophobic polymer, creating a poor bond with cementitious matrices when mixed, thus limiting its potential as structural reinforcement. Copolymerizing polypropylene with non-ionic surfactants provides a solution by modifying the hydrophobicity of the fibers. This research investigated the micromechanical bonding properties between copolymerized polypropylene and a cementitious matrix. Single fiber pullout was employed in which both the chemical bond strength and frictional bond strength between fiber and matrix were measured. Results from this work show that the copolymerized polypropylene can be used to create hydrophilic fibers that are capable of developing substantially higher bonds with cementitious matrices. Ultimately, this increased bonding can result in higher strength polypropylene fiber reinforced cementitious composites with tighter crack widths, thus serving as a low-cost viable replacement for existing, more expensive FRC reinforcing fibers.

KEYWORDS:

Polypropylene fibers; chemical bonding, fiber reinforced concrete, single fiber pullout

INTRODUCTION

Fiber reinforced concrete (FRC) is now a commonly used building material that is used in the production of building façade panels, industrial floors, pipes, and roofing tiles (Banthia *et al.*, 2012; ACI, 2002). Depending on the application, strength requirements, and desired properties, there are a number of fibers that are used as primary or secondary reinforcement in concretes and cementitious composites (ACI, 2002).

Asbestos fibers were historically used, and continue to be used in developing countries, for their good mechanical performance and low cost (Aveston, 1969; Matthews & Rawlings, 1999; Shah, 1981). While affordable and suitable for easy manufacturing, asbestos fibers carry with them substantive human health impacts including lung cancer, mesothelioma, and asbestosis (Castleman, 2005; Selikoff & Lee, 1978; Terazono & Moriguchi, 2000).

More recently, synthetic fibers have gained acceptance throughout the industry. For example, investigations into synthetic fibers for FRC applications have focused on polyvinyl-alcohol (PVA) fibers, which are commonly used in high performance fiber reinforced cementitious composites (HPFRCC), strain hardening cementitious composite (SHCC) or Engineered Cementitious Composite (ECC) applications (Lepech, Li, Robertson, & Keoleian, 2008; Li, Wu, Wang, Ogawa, & Saito, 2002). PVA reinforced composites exhibit excellent mechanical properties, but at relatively high cost (Li, Wang, & Wu, 2001; Passuello, Moriconi, &

Shah, 2009). This excellent mechanical performance is, in part, due to the hydrophilic nature of PVA resin, causing PVA fibers to develop a strong bond to a cementitious mix. In some instances, this bond has proven to be too strong and can cause fiber rupture prior to pullout and associated desirable pullout energy dissipation. As such, PVA fiber surface treatments have been used (*i.e.*, oiling agents on the PVA fiber surface) to reduce the strength of the chemical bond between PVA fiber and cementitious matrix and improve overall composite performance (Wang, 2006).

In contrast to high performance and high cost PVA fibers, polypropylene (PP) fibers are used as reinforcement in concrete due to their low cost, in spite of their more modest mechanical properties (Biagiotti, Fiori, Torre, Lopez-Manchado, & Kenny, 2004; Guo-zhong & Shuai, 2010; Singh, Shukla, & Brown, 2004). Polypropylene is a common thermoplastic with a variety of applications in addition to fiber reinforcement for concrete. Applications include, but are not limited to medical, storage, and commercial uses (Xiangwan, 2013; Zhang & Chen, 2011). Currently, PP fibers in concrete are used in low volume fraction applications since they provide marginal mechanical improvement (Alavi Nia, Hedayatian, Nili, & Sabet, 2012; Guo-zhong & Shuai, 2010; Song, Hwang, & Sheu, 2005). This is due, in part, to the hydrophobic nature of polypropylene, which prevents strong bonding with hydrating cement paste that is rich in mix water. Thus, common applications of PP fibers in fiber reinforced concrete use between 0.25% and 0.5% fiber volume fraction to resist temperature and shrinkage crack formation.

Although these limitations to using PP fibers are due to their hydrophobic nature, the hydrophobicity of PP fibers can be tailored through the use of chemical additives. Such tailoring of PP fibers is analogous to the altering of PVA fiber bonding properties through surface oiling. This research hypothesizes that a small amount of chemical can be copolymerized into the polypropylene resin during manufacturing. These additives can then migrate to the surface of PP fibers, allowing the fibers to exhibit a stronger affinity towards water, thus increasing bond and making the fibers more suitable for use as structural reinforcement in cementitious composites. This paper describes the underlying chemistry of this process and the experimental verification of PP fiber bond modification through additive copolymerization.

BACKGROUND

Copolymerization has proven to be an effective modification tool for polymer materials. Datla *et al.* identified stearyl alcohol ethoxylated additives that can significantly increase the polarity of PP polymer resins (Datla, Shim, & Pourdeyhimi, 2011). Datla *et al.* found that through the copolymerization of polypropylene with polyoxyethylene 2 stearyl alcohol ($\text{CH}_3(\text{CH}_2)_{16}\text{CH}_2(\text{OCH}_2\text{CH}_2)_2\text{OH}$), polyoxyethylene 4 stearyl alcohol ($\text{CH}_3(\text{CH}_2)_{16}\text{CH}_2(\text{OCH}_2\text{CH}_2)_4\text{OH}$), and polyoxyethylene 6 stearyl alcohol ($\text{CH}_3(\text{CH}_2)_{16}\text{CH}_2(\text{OCH}_2\text{CH}_2)_6\text{OH}$), a significant increase in the hydrophilicity of polypropylene resin can be achieved. The effectiveness of each polyoxyethylene (POE) stearyl alcohol copolymer in altering hydrophilicity was controlled by its potential to disperse in the PP resin, a process also known as blooming, whereby additives migrate from the bulk of the fiber to the fiber surface.

With regard to copolymerization, blooming, and migration of stearyl alcohol ethoxylated additives in PP resin, Datla *et al.* made a number of important observations. First, the surface oxygen content ratio (and therefore the concentration of long ethylene chains on the film surface) changed over time after the copolymerized polypropylenes were extruded. As measured by differential scanning calorimeter (DSC) analysis, each additive/PP mixture indicated that the surface oxygen content ratio increased between 0 days and 20 days after extrusion before reaching steady state. This change in surface oxygen content indicates the possibility of

blooming and that migration of the additives occurs over a period of time after extrusion. Furthermore, each additive/PP mixture observed initially lower contact angle measurements at the time of extrusion when compared to neat PP. Four months after extrusion, the measured contact angle continued to decrease. These observed changes in the contact angle indicate the ongoing migration of copolymerized additives to the surface of the fiber over an extended timeframe.

Copolymerization is done through melt blending and spinning in which additives are melted together and then spun to uniformly distribute the additive within the polymer when still at high temperature (Brody, 1986). At this temperature, the polymer is spun again to create individual fibers. Unlike topical treatments, corona treatment, or plasma treatment, the process of melt blending and spinning disperses the additive throughout the bulk polymer. Thus, in order to change the surface polarity of the polymer, it is critical that blooming or surface migration of additives takes place. Based on preliminary investigations by Datla *et al.*, surface migration of stearyl alcohol ethoxylated additives is a promising method to modify the surface properties of PP fibers for use as structural reinforcement in fiber reinforced concretes and cementitious mortars (Datla, Shim, & Pourdeyhimi, 2011; Lee, Ivanova, Starov, Hilal, & Dutschk, 2008).

To investigate the effect of PP fiber surface modification through copolymerization, the primary composite material characterization technique used in this study is single fiber pullout (SFP). Redon *et al.* detailed the experimental determination of chemical and frictional bond between a fiber and its surrounding cementitious matrix for fiber reinforced cementitious composites using SFP (Redon *et al.*, 2001). The experimental setup was built by casting an individual fiber into a cementitious matrix with a section of the fiber extending from the matrix. A testing machine then slowly pulls the fiber out of this matrix while measuring the load and displacement response. The load-displacement (P - δ) response was used to quantify bond strength and pullout behavior. Figure 1 shows a schematic load-displacement response of a single fiber pullout (SFP) test.

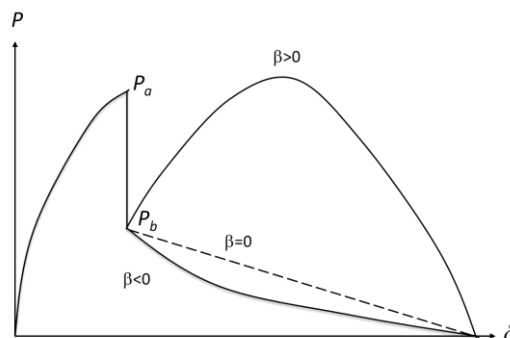


Figure 1: Schematic Representation of Single Fiber Pullout (SFP) Response (Redon *et al.*, 2001)

Figure 1 shows three distinct phases of fiber-matrix interaction during pullout. During the first portion of the response (elastic loading and inelastic fiber displacement), the fiber transfers load through interfacial shear to the surrounding cementitious matrix. This shear transfer is possible though chemical bonding between fiber and matrix. As load increases, small flaws along the interfacial transition zone (ITZ) between fiber and matrix coalesce to form a tunnel-shaped flaw near the cement surface, thus allowing some inelastic deformation before pullout. Once the load reaches a critical value (P_a), the tunnel-shaped flaw unstably propagates from the specimen surface to the embedded end of the fiber, instantly breaking the chemical bond along the fiber length, and resulting in a sudden drop to a lower load level, P_b . Following fracture of the chemical bond, the frictional bond between fiber and matrix provides varying pullout resistance. Beginning from load level P_b , the

fiber can undergo slip hardening ($\beta > 0$), slip softening ($\beta < 0$) or constant friction ($\beta = 0$). During this progression the fiber is in one of five states that includes active debonding, active pullout, rupture during debonding, rupture during pullout, or completed pullout (Wang, 2006).

Chemical bond, G_d , is calculated using the drop in load following the initial pullout response using Equation 1.

$$G_d = \frac{2(P_a - P_b)^2}{\pi^2 E_f d_f^3} \quad \text{(Equation 1)}$$

where G_d is the chemical bond in J/m^2 , P_a is the chemical debonding pullout load in N, P_b is the frictional pullout load in N, E_f is the fiber elastic modulus in Pascals, and d_f is the fiber diameter in meters. Equation 1 can be validated using the Griffith fracture criterion for a cylindrical-shaped crack in Mode II propagation (Redon *et al.*, 2001).

Following this unstable fracture and release of chemical bond, the frictional bond, τ_0 , is calculated using the load capacity remaining following the load drop through Equation 2.

$$\tau_0 = \frac{P_b}{\pi d_f l_e} \quad \text{(Equation 2)}$$

where, τ_0 is the frictional bond in Pascals, P_b is the frictional pullout load in N, d_f is the fiber diameter in meters, and l_e is the fiber embedment length into the test specimen in meters.

Redon *et al.* noted two typical scenarios for fiber pullout response. In the first scenario, the fiber begins to enter a slip-hardening phase. However, during slip hardening the strength of the fiber is exceeded resulting in fiber rupture before full pullout could be achieved. In the second scenario, the fiber similarly enters the slip hardening stage, but the strength of the fiber is never exceeded resulting in full pullout of the fiber. Since both scenarios capture the chemical debonding load drop, they are both useful in understanding the pullout behavior of fibers since G_d and τ_0 can be computed.

MATERIALS AND TESTING TECHNIQUES

FIBER PRODUCTION AND PROPERTIES

Fiber development and evaluation for this study was conducted in a two-stage process. During the first stage, a number of copolymer additives were tested to determine the best candidate to create a strong chemical bond within a cementitious matrix. Pullout testing was performed on fibers containing four different copolymerization additives, along with a set of control fibers (neat polypropylene). Ionic polymers, such as stearyl alcohol ethoxylated additives used by Datla *et al.* and also those used in this research, are termed surfactants since they are specifically used to lower the surface tension of water thus creating more hydrophilic behavior (Lee *et al.*, 2008). While partition ratio (used by Datla *et al.*) can be a good indicator of surfactant hydrophilicity, similar measures of polymer polarity are the hydrophilic-lipophilic balance (HLB) and the ratio of ethylene oxide (polar component) to propylene oxide (non-polar) within the polymer. Surfactants consist of a hydrophilic head and a hydrophobic/lipophilic tail. HLB values can be used to describe the relative ratio of the hydrophilic portion to the entire mass of a surfactant (Griffin, 1955). Equation 3 was used to calculate the HLB ratio for the additives used in the testing program.

$$HLB = 20 \frac{M_h}{M}$$

(Equation 3)

where M_h is the mass of the hydrophilic portion in daltons, and M is the mass of the entire molecule in daltons. Therefore, an HLB value of 0 indicates a highly hydrophobic substance whereas an HLB value of 20 indicates a highly hydrophilic substance. For the copolymerization additives used in this proposed work, ethylene oxide (EO) and propylene oxide (PO) serve as the respective hydrophilic and hydrophobic heads. Therefore, surfactants that contain more EO exhibit greater hydrophilicity. To this extent, the EO/PO ratio was used in this work as a relevant metric in understanding the hydrophilic/hydrophobic behavior of the surfactants.

Additives labelled “B”, “C”, and “D” are block copolymers formed by the reaction of alcohols with EO and PO. Additives B, C, and D follow the chemical formula of $R-O-(C_2H_4O)_x(C_3H_6O)_yH$ where R is an alkyl group, C_2H_4O is ethylene oxide (EO), and C_3H_6O is propylene oxide (PO). Additive B is a copolymer with 20% EO by mass. Additive C has an EO/PO ratio of 4:6 with an HLB value of 6.5. Additive D has an EO/PO ratio of 3:6 with an HLB ratio of 5.5. In the case of additives C and D, an increase in the HLB values can be seen with an increase in the EO/PO ratio due to the correlation of the EO with surfactant hydrophilicity. Additive “A” is a proprietary surfactant produced by PPG Corporation.

Once additives were identified for copolymerization, a batch of PP fibers with varying additive amounts was produced for study. The additives were incorporated using the melt-blending and spinning process and are intended to act as a polarity control agent, whereby the additives migrate from the bulk PP fiber to the surface to impart a change in the fiber hydrophobicity. Following manufacture, the produced fiber diameter was experimentally found to have a mean of 18.0 μm with a standard deviation of 2.0 μm . This fiber diameter is used in all subsequent fiber mechanical property, G_d , and τ_0 calculations.

Table 1 shows the five different fibers that were formulated using four different ionic polymer additives. Control fibers were spun from neat PP with no additive in order to observe changes in bonding strength that each individual additive produced when copolymerize with the PP. In Table 1, all treated PP was consistently copolymerized with 1.50% additive in order to best observe the effect of the additive in modifying fiber-matrix interfacial behavior.

Table 1: Experimental Program Fiber Designations and Polymer Additives

FIBER LABEL	PP RESIN ADDITIVE	COPOLYMERIZATION (%)
Control	None (control)	None (control)
A-1.5	A	1.50
B-1.5	B	1.50
C-1.5	C	1.50
D-1.5	D	1.50

Basic mechanical properties were experimentally determined for single fiber filaments containing each of the four copolymerization additives. These tests were done to independently determine the tensile strength (tenacity) and elastic modulus of the fiber in single filament form for chemical and frictional bond calculations. A minimum of 30 single filament tests were performed for each fiber type. The fiber filament performance is summarized in Table 2.

Table 2: Summary of Fiber Filament Mechanical Properties Testing

FIBER LABEL	AVERAGE STRENGTH (MPA)	STRENGTH STANDARD DEVIATION (MPA)	AVERAGE MODULUS (MPA)	MODULUS STANDARD DEVIATION
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(MPA)				
Control	805	160	3237	535
A-1.5	912	278	3144	711
B-1.5	688	146	2875	591
C-1.5	702	95.5	3002	440
D-1.5	765	131	2847	589

CEMENTITIOUS MATERIALS, MIXING, CURING, SPECIMEN PREPARATION, SPECIMEN GEOMETRY

Each of the SFP tests was performed within an identical cementitious mortar matrix. Mortar matrices were held constant to solely determine the effect of fiber modifications on chemical and frictional bonding. The mortar matrix mixing proportions are shown in Table 3. Single filament pullout testing was conducted at a specimen age of 28 days.

Table 3. Mortar Matrix Mixing Proportions

COMPONENT	PROPORTION
Cement	1.0
Fly Ash	1.2
Sand	0.8
Water	0.55
Superplasticizer	0.01
Viscosity Modifier	0.0011

The superplasticizer used in these experiments was a polycarboxylate-based superplasticizer with tradename Melflux 2641[®] produced by BASF. The viscosity modifier used in these experiments was a hydroxypropylmethyl cellulose viscosity modifier with tradename Methocel[®] produced by Dow Chemical.

Ultimately, additive D (R-O-(C₂H₄O)_x(C₃H₆O)_yH with a EO/PO ratio of 3:6 and HLB of 5.5 was chosen for a more comprehensive second-stage test series. This testing series varied the percent of copolymerization in order to tailor the copolymer constituents for specific chemical and mechanical bonding properties. Table 4 details the specific testing series conducted for Additive D.

Table 4: Experimental Program Fiber Designations and Additive Amount for fibers containing Additive D

FIBER NUMBER	PERCENT COPOLYMERIZATION OF ADDITIVE D
Control	0.00
D-0.75	0.75
D-1.25	1.25
D-1.5	1.50

CHEMICAL AND FRICTIONAL BOND TESTING OF PP FIBERS

Single filament pullout testing was performed on a MTS Table Top 858 machine with a 10N load cell. Tested samples had a fiber embedment length between 0.5mm and 2mm. Testing speed was set at 0.003mm/min ± 0.002mm/min. Prior to loading, the fiber filament was affixed to a mounting plate with Loctite[®] glue in order to guarantee proper fixing of the fiber to the load cell. Given the small, 18 μm diameter of the fibers, this proved to be a more reliable method than using pneumatic, hydraulic, or mechanical testing grips that the fiber

could easily slip through during the course of the test. The low testing speed was necessary due to the fragile nature of the embedded fiber test set-up. A higher testing speed often resulted in scenarios where the fiber tenacity was exceeded well before the bond was engaged due to momentum effects. Only with a slower test speed was it possible to remain below fiber tenacity while engaging the fiber-matrix bond interface for useful test results. The SFP testing setup is shown schematically in Figure 2.

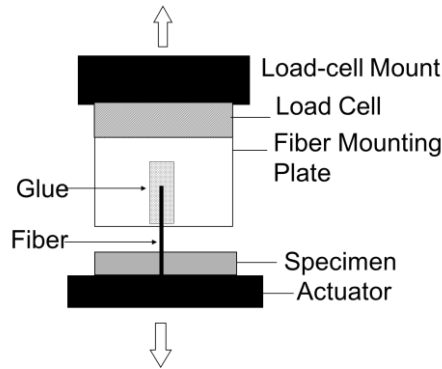


Figure 2: Schematic representation of single fiber pullout (SFP) test

RESULTS

Results from the two stages of testing show that significant changes in chemical and frictional bonding of polypropylene fibers with a cementitious matrix can be achieved through copolymerization with ionic additives. Figure 3 shows a pullout curve for neat PP (Control) that was unmodified. The curve shows a linear elastic response followed by a steady-state load capacity and slow load capacity deterioration. Since no drop is observed on the graph this pullout test displayed little chemical bonding (*i.e.* the load elastically increased and the pullout resistance is provided only by the frictional bond). Due to the hydrophobic nature of PP fibers and the aqueous nature of cement paste, this response is expected. PP fibers are known to exhibit little chemical bonding in cement-based matrices (Singh *et al.*, 2004), partially validating both the experimental setup and the control PP fibers being studied.

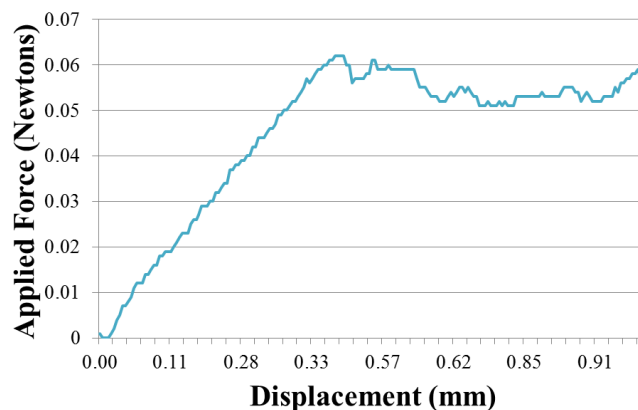


Figure 3: Representative Load versus Displacement Relationship for Control Fiber Single Filament Pullout

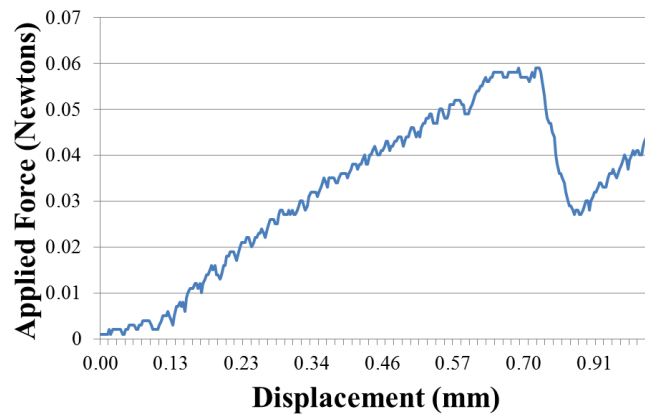


Figure 4: Representative Load versus Displacement Relationship for D-1.5 Single Filament Pullout

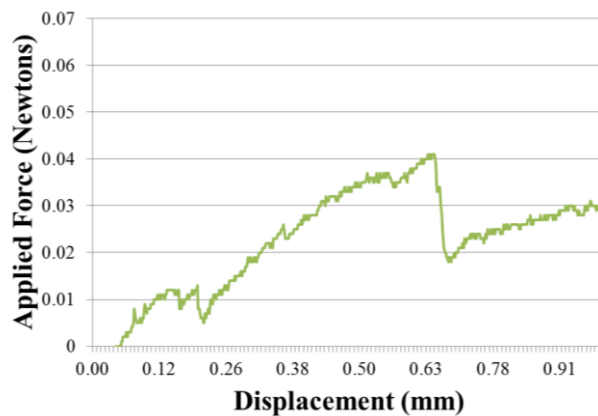


Figure 5: Representative Load versus Displacement Relationship for D-1.5 Single Filament Pullout

Figures 4 and 5 show representative pullout curves for Fiber D-1.5. In both graphs, a linear response is followed by a sudden drop in load. This drop in load is indicative of the unstable propagation of the tunnel crack and the loss of the chemical bond between the fiber and the surrounding cementitious matrix. In contrast to the control PP fiber, D-1.5 exhibits a significant chemical bond, supporting the hypothesis that stearyl alcohol ethoxylated additive copolymerization with polypropylene can improve chemical bonding properties of PP fiber through increased hydrophilicity. Table 5 summarizes the measured chemical bond for each of the copolymers studied, where a significant increase in chemical bond was achieved as compared to the control fibers. Approximately 5 tests were conducted for each fiber type. The largest increase in the chemical bond was seen for additive D.

Table 5: Bonding and Pullout Properties of PP Fibers D-1.5 and Control embedded in cementitious matrix

<i>Fiber Label</i>	<i>Average G_d (J/m^2)</i>	<i>σ_{Gd} (J/m^2)</i>	<i>Average τ_0 (MPa)</i>	<i>σ_{τ_0} (MPa)</i>
Control	0	0.2	1.01	0.53
A-1.5	1.9	0.7	0.55	0.25
B-1.5	0.4	0.08	0.30	0.13
C-1.5	1.5	1.0	0.40	0.18
D-1.5	4.7	2.5	0.81	0.47

For reference, it is useful to compare these results with previously reported single fiber pullout testing data. Presently, most of this data exists for poly(vinyl)alcohol (PVA) fibers, a strongly hydrophilic polymer as discussed previously. PVA chemical and frictional bonding properties are taken from Redon *et al.* (2001) and are shown in Table 6. These fibers are produced by Kuraray under the trade name Kuralon-KII and have diameters ranging from 44 microns up to 700 microns. PVA fibers serve as a viable comparison fiber due to their similar chemical bonding behaviour and their need to also be modified for effective use as a reinforcement material in concrete. Due to copolymerization with additive D, the PP fibers produced in the present study exhibit chemical bonding properties comparable to those displayed by hydrophilic PVA fibers. The control PP fiber exhibits little chemical bonding properties.

Table 6: Bonding and Pullout Properties of PVA Fibers Embedded in Cementitious Matrix (from Redon *et al.*, 2001) Compared to those For Control and D-1.5

<i>Fiber Type</i>	<i>Chemical Bond (J/m²)</i>	<i>Frictional Bond (MPa)</i>
PVA	4.2	5.9
Control	0	1.01
D-1.5	4.7	0.81

As seen in Table 5, in the experimental investigation additive D proved to be the most promising additive in affecting the hydrophilicity of the fibers so as to bring about an improved chemical bond. To further investigate the impact that the additive amount has on the chemical bond strength, and to investigate the potential for fiber-matrix interface tailoring, further analysis was conducted on copolymerized fibers where the amount of additive D present was varied (Table 4). The objective of this round of experiments was to assess whether or not an optimum amount of additive might exist that would serve to improve the bond strength to a desired level. Figure 6 displays the relationship between the amount of Additive D and the chemical bond.

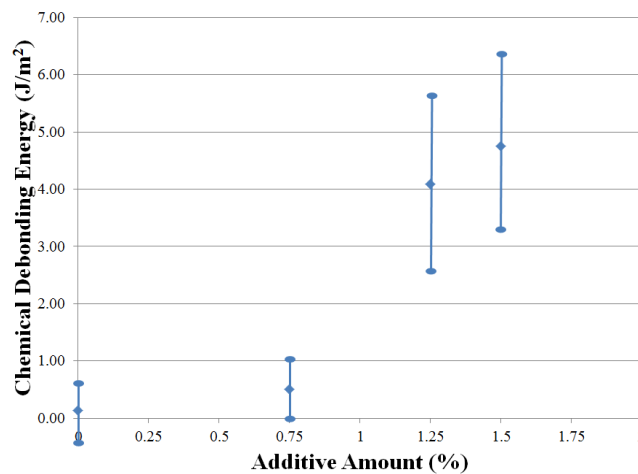


Figure 6: Percent of PP Copolymerization versus Chemical Debonding Energy Relationship for Additive D Single Filament Pullout Tests

As can be seen in the graph above, it is clear that as the amount of additive increases so does the chemical bond of the fiber to the matrix. This is most likely due to the greater amount of bloomed additive present on the surface of fibers copolymerized with high amounts of additive. Currently, this hypothesis is being investigated through the use of Raman Spectroscopy. Moreover, the extent to which copolymerization is

increasingly effective at modifying chemical bonding of PP fibers is unclear in Figure 6. Therefore, additional SFP testing is currently being done with fibers copolymerized with larger percentages of Additive D. These tests look to identify the maximum effective copolymerization level using Additive D for PP fibers used in this cementitious matrix.

Finally, although the chemical bond of the PP fiber can be modified when the chemical additives are used, the frictional bond does not see a similar modification. This is mainly driven by the fact that the frictional bond is primarily a function of the roughness of the fiber. Chemical additives that are copolymerized with the fiber resin in small amounts do not change the roughness and are only modifying the surface affinity to water.

CONCLUSION

This study investigated the effect of copolymerizing polypropylene (PP) resin with stearyl alcohol ethoxylated additives to control the hydrophobicity of the copolymer and improve PP fiber bonding with cement matrices. Experimental results show that an increase in chemical bonding brought about by the use of additives can be clearly observed. Furthermore, higher concentrations of the additive within the matrix result in stronger bond between the copolymerized fiber and the cementitious matrix. At a copolymerization level of 1.5% of stearyl alcohol ethoxylated additive with an EO/PO ratio of 3:6 and an hydrophilic-lipophilic balance ratio of 5.5, the chemical bond strength of PP fibers was equivalent to that of poly(vinyl)-alcohol (PVA) fibers, which are currently used for structural reinforcement of cementitious composites.

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