Modification of Polypropylene Fiber Applied in Concrete Via Silane Coupling Agent Treat

Xiaohui Li

China Nuclear Construction Concrete Co., LTD, Xi'an, Shaanxi, P. R. China

Abstract

Polypropylene fibers have potential for widespread application in industrialized concrete, and they have significant practical significance for the strength and waterproofing performance of the material. However, the dispersion performance of polypropylene fibers in concrete or aqueous solutions, as well as in mortar is poor, and they are prone to agglomeration and caking, which in turn adversely affects the strength of the concrete. This paper examines the influence of the treatment with coupling agents on the surface state of the dispersion of polypropylene fibers, and analyzes and studies their applications in aqueous solutions, mortars, and concrete.

Keywords

Polypropylene fiber, surface modification, silane coupling agent, dispersion, concrete.

1. Introduction

Polypropylene fiber has outstanding advantages such as light weight, high toughness, acid and alkali resistance, and low price, and has been widely applied in civil engineering, geotextiles, wastewater treatment and textile industry. However, the molecular chain of polypropylene consists only of C-C and C-H bonds, without high active functional groups. The water contact angle of pristine PP fiber can reach 135-140oC, showing strong hydrophobicity and chemical inertness. This poor interfacial compatibility has always been the core obstacle limiting the promotion of PP fiber reinforced composites. Chemical modification shows silane coupling agent treatment has become a research hotspot in recent years, because it can introduce polar functional groups on the fiber surface without seriously damaging the bulk tensile strength of PP fiber. Many recent studies have proved that appropriate SCA modification can significantly reduce water contact angle, increase surface roughness, improve fiber dispersion in cement paste, and greatly enhance fiber pull-out resistance and the mechanical performance of composite materials.

Silane coupling agents have the typical molecular structure of Y-Si(OR)3. The alkoxy groups (OR) at one end are hydrolyzed in aqueous or alcohol solution to produce silanol (-Si-OH). The silanol groups can form hydrogen bonds or covalent bonds with the hydroxyl groups generated on the oxidized PP fiber surface. The other end of the molecule is an organic functional group Y, such as amino group, epoxy group, vinyl group or methacryloxy group. These functional groups can undergo chemical crosslinking reaction with cement hydration products, polar polymers or inorganic fillers, so as to realize the "bridging" effect between PP fiber and matrix. KH550 (3-aminopropyltriethoxysilane) and KH560 (3-glycidoxypropyltrimethoxysilane) are the most widely investigated silane coupling agents for PP fiber modification. Sun et al. (2024)[1] systematically compared the modification effect of KH550 and KH560 at mass concentrations of 0.5%, 1.0%, 1.5% and 2.0% on PP fibers used in cement-based materials. Their SEM characterization showed that SCA treatment increased the micro-roughness of PP fiber surface. The FTIR spectrum verified that amino and epoxy functional groups were successfully grafted onto the fiber surface. The experimental results demonstrated that the

optimal concentration of both SCAs was about 1.5 wt%. Excessively high SCA concentration will lead to the formation of thick, brittle silane multilayer coating on the fiber surface. The thick coating is easy to peel off under stress, which reduces the interfacial performance instead. This paper is applied the KH-570 to modify the PP fiber for the further application in concrete, thus the surface of PP fiber was first activated by acid and then the KH-570, to promote the dispersion in water or in concrete.

2. Experimental Section

2.1. Surface Modification of Polypropylene Fibers

Polypropylene (PP) staple fibers were first cleaned with anhydrous ethanol and deionized water in an ultrasonic bath for 15 min to remove residual spinning oil and surface contaminants, followed by drying in an oven at 60oC to constant weight. For acid etching pretreatment, the cleaned PP fibers were immersed in dilute acid solution under continuous stirring at a preset temperature. The acid etching process introduced microscale pits and oxygen-containing polar groups on the chemically inert PP surface, increasing surface roughness and providing active sites for subsequent silane grafting. After acid corrosion, the fibers were repeatedly rinsed with plenty of deionized water until the pH of the eluate became neutral, then dried at 60oC for 12 h.

KH570 (3-(trimethoxysilyl)propyl methacrylate) silane coupling agent was adopted for surface modification. The KH-570 ethanol-water mixed solution was prepared and hydrolyzed for a certain period under weak acidic condition to generate reactive silanol groups.[2] The acid-etched PP fibers were submerged into the hydrolyzed KH-570 solution under gentle stirring to facilitate the adsorption and grafting of silane molecules onto the fiber surface. After the predetermined dipping duration, the fibers were taken out, rinsed with ethanol to wash off loosely bound and unreacted excess KH570. Subsequently, the samples were cured at a moderate temperature to complete the condensation reaction of silanol groups, and then cooled naturally to room temperature to obtain KH-570-modified PP fibers.

2.2. Characterization Methods

Fourier-transform infrared spectroscopy (FTIR) was used to identify the change of surface functional groups of PP fibers before and after modification. The fiber samples were ground and pressed into KBr pellets for FTIR measurement, and the spectra were recorded in the wavenumber range of 4000-400 cm-1 to verify the successful grafting of KH-570 onto the PP fiber surface.[3-5]

The static water contact angle test was performed to evaluate the variation of surface wettability. A deionized water droplet (5μL) was dropped onto the fiber surface, and the contact angle value was captured and calculated using a contact angle measuring instrument. At least five different positions on each fiber sample were tested, and the average value was reported.

Scanning electron microscopy (SEM) was applied to observe the micromorphology evolution of fiber surfaces. The fiber specimens were gold-sputtered before observation to enhance electrical conductivity. The surface microstructure, roughness change and coating morphology of pristine, acid-etched and KH-570-modified PP fibers were characterized under different magnifications.

The silane attachment rate of KH-570 on PP fiber surfaces was quantified to assess the loading amount of grafted silane coupling agent. The mass difference method was adopted: the mass of PP fibers before modification and the mass of fully dried modified fibers were accurately weighed using an analytical balance, and the KH570 attachment rate was calculated according to the mass increment after removing the physically adsorbed free silane.

The dispersion performance of PP fibers in aqueous solution was characterized for both unmodified and KH570-modified samples. Equal amounts of pristine PP fibers and modified PP fibers were separately added into deionized water under mild stirring. The dispersion state, floating behavior and agglomeration degree of fibers in water were visually recorded and compared after standing for a fixed time.

3. Results and Discussion

3.1. FTIR and Contact Angle

The surface of polypropylene fibers was treated with KH570 silane coupling agent with modification times of 0, 10, 20, 30, and 120 minutes. The infrared spectra (Fig. 1) of the treated polypropylene fibers are shown in Figure 1. The spectra indicate that as the treatment time with KH570 increases, the active functional groups on the surface of the polypropylene fibers gradually increase, particularly showing significant peak enhancements in the ranges of 3500-3400 cm-1 and 1700-1600 cm-1. These regions mainly correspond to two functional groups: hydroxyl and carbonyl groups from the silane coupling agent. The increased peak areas suggest that the functional groups of KH570 have effectively attached to the surface of the polypropylene fibers, indicating a successful chemical reaction. This significantly increases the number of active sites on the fiber surface, providing favorable reactive sites for subsequent applications in water and concrete, resulting in excellent modification performance.

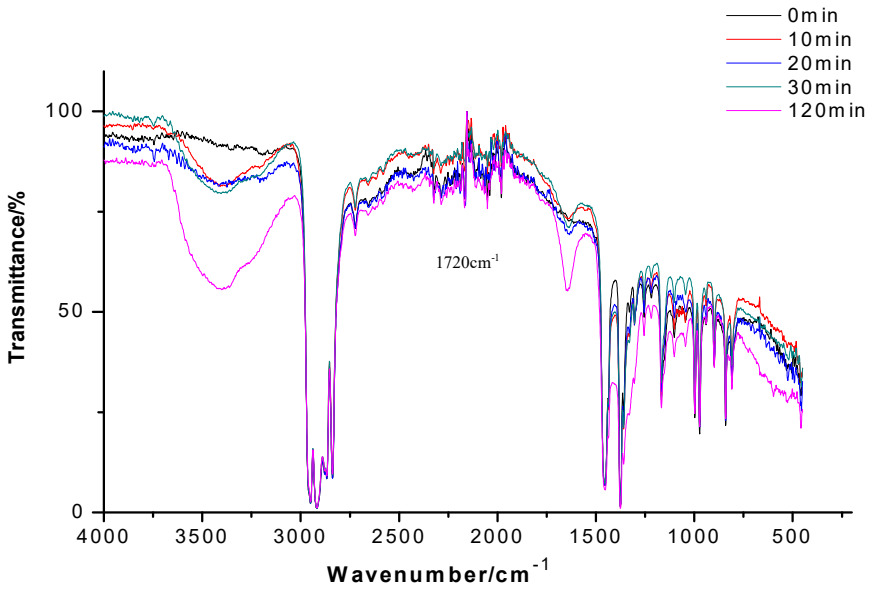

Fig. 1 The Infrared spectroscopy of KH-570 modify PP Fiber with different time

The contact angle tests (Fig. 2) on five types of modified polypropylene fibers. It was found that the contact angle of unmodified polypropylene fiber reached 125 degrees. After 10 minutes of modification, the contact angle decreased to 117 degrees; after 20 minutes, it was 97.2 degrees; after 30 minutes, it dropped to 84.2 degrees; and after 120 minutes of modification, the contact angle was 64.6 degrees. This study indicates that as the treatment time in KH570 increases, the contact angle of polypropylene fibers gradually decreases, resulting in improved hydrophilicity and enhanced compatibility with inorganic materials.

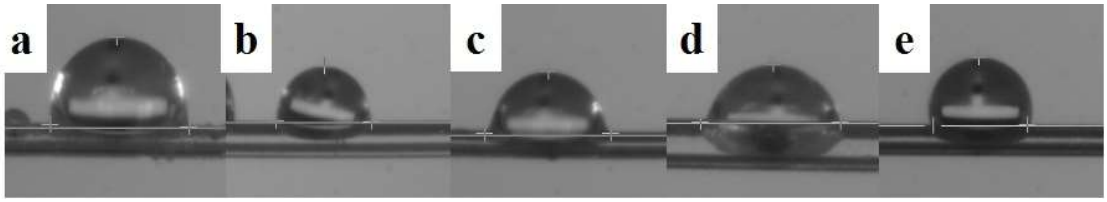

Fig. 2 The contact angle photos of the KH-570 modified PP Fiber with different time

3.2. SEM

The surface morphology of PP fiber is shown in Fig. 3, which SEM images of untreated PP Fiber and the KH-570 modify PP Fiber for 120 min.

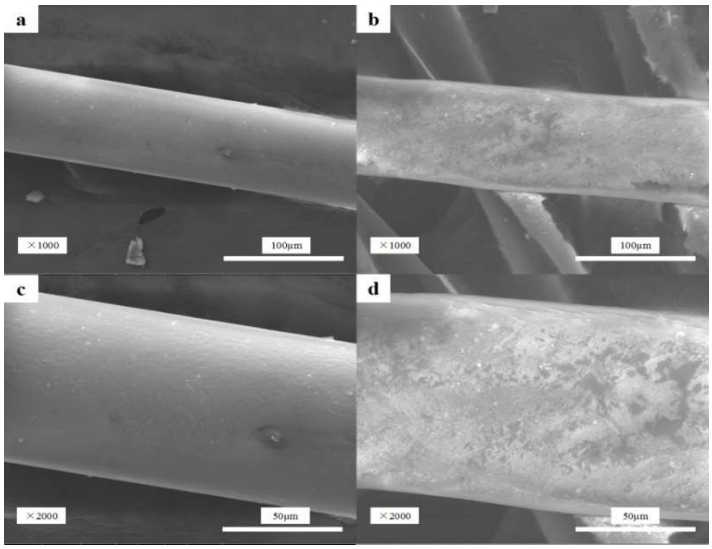

Fig. 3 The SEM images of untreated PP Fiber and the KH-570 modify PP Fiber for 120 min

The micro-morphology of the treated polypropylene fibers was characterized using scanning electron microscopy. It was found that as the treatment time increased, the fiber surface gradually became rougher and coated with silane coupling agent, resulting in decreased electrical conductivity and increased polarity. From the images, the fiber diameter was approximately 80-90 μm before modification,[6] increasing to 100-120 μm after modification, with an attached layer thickness of about 20-30 μm, achieving a satisfactory modification effect. Furthermore, the fiber surface was initially very smooth and contained only a few contaminant particles; after modification, the surface became rough and uneven, which is favorable for subsequent chemical reactions with polar substances or inter-facial bonding with cementation materials, indicating potential for industrial applications.

3.3. Inducing Rate of KH-570

The the results of Inducing rate of KH-570 on the surface of polypropylene fiber are shown in Table 1.

Table 1. Inducing rate of KH-570 on the surface of polypropylene fiber

Modify time/min	Silane coupling agent concentration/%	Weight after modify/g	Weight before modify/g	Inducing rate/%
10	5	0.2062	0.2038	4.59
20	10	0.2025	0.2006	4.83
30	20	0.5030	0.4998	5.15
120	50	0.2051	0.2037	6.55

It can be seen from Table 1 that the improvement of KH570 on the surface of polypropylene is relatively good. When the modification time is 10 minutes and the concentration of the silane coupling agent is 50%, the weight of the fiber after attaching the silane coupling agent is approximately 0.2062g. [7]The weight of the fiber before attachment is 0.2038g. The introduction rate is 4.59%. When the modification time is 20 minutes and the concentration of the silane coupling agent is 10%, the weight after modification is 0.2025g, and the weight before modification is 0.2006g, with an introduction rate of 4.83%. When the modification time is 30 minutes and the concentration of the coupling agent is 20%. The weight of the fiber after modification is 0.5030g, and the weight before modification is 0.4998g, with an introduction rate of 5.15%. When the modification time is 120 minutes and the concentration of the silane coupling agent is 50%, the weight after modification is 0.2051g, and the weight before modification is 0.2037g, with an introduction rate of 6.55%. The above results indicate that the modification time and the concentration of the silane coupling agent will have an impact on the introduction of surface active substances on the fiber. Among them, the concentration of the silane coupling agent has a more significant impact, and the modification time has a secondary impact. Therefore, when modifying the surface of polypropylene fibers, it is necessary to first use a high concentration of silane coupling agent, and then consider extending the modification time.

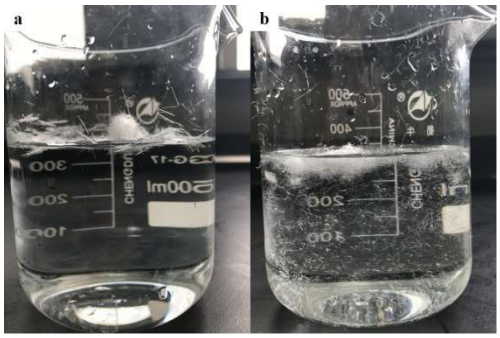

Fig. 4 The dispersion situation of KH-570 modified polypropylene fiber(left: unmodified PP; right: modified PP)

The polypropylene fibers modified with silane coupling agent were placed in a beaker and deionized water was added. The dispersion properties of the solution were characterized. According to the results of the characterization, the unmodified polypropylene fibers completely floated on the water surface and did not show any tendency to disperse when exposed to water. However, the modified polypropylene fibers significantly enhanced their affinity for water, and a large number of fibers were suspended in the solution. But some fibers still floated on the surface of the solution. Compared with the unmodified polypropylene fibers, its hydrophilicity significantly increased, which formed a correlation evidence with the contact angle test results. It reveals that it has a better interface explanation effect and dispersion performance in water-based or polar environments and can be used in mortar, concrete, etc.

4. Conclusion

This paper conducted chemical modification of polypropylene fibers using KH-570 silane coupling agent. The effects of modification time and the concentration of KH-570 silane coupling agent on the modification results were investigated. The infrared spectra, contact angle, surface microstructure, modification introduction amount, and suspension state of the products were studied. It was found that longer modification time and higher silane coupling agent concentration resulted in better modification effects. The experiment revealed that when

the modification time was 120 minutes and the silane coupling agent concentration was 50%, the introduction rate was the highest and the dispersion performance was the best. The research results of this paper indicate that simple modification of polypropylene fibers with silane coupling agent can make the polypropylene fibers have better dispersion performance, and have better dispersion performance in water-based solutions, mortars or concrete, possessing potential industrial application.

References

[1] Sun, J., Liu, X., Wang, C., et al. (2024). Study on properties of cement-based composites with silane coupling agent modified polypropylene fiber. Bulletin of the Chinese Ceramic Society, 43(7), 2355–2362.

[2] Tran, T., & Zhang, J. (2022). Pullout and flexural performance of silane groups and hydrophilic groups grafted polypropylene fibre reinforced UHPC. Construction and Building Materials, 324, 126674. https://doi.org/10.1016/j.conbuildmat.2022.126674

[3] Goldfein, S. (1965). Fibrous reinforcement for Portland cement. Modern Plastics, 42(8), 156–160.

[4] Liu, H., Fei, Y., Sun, S., et al. (2024). Research on hydrophilic modification performance of polypropylene fiber treated by KH570 silane coupling agent. Contemporary Chemical Research, (13), 52–54.

[5] Guo, L., & Yang, Q. (2024). Surface activation of polypropylene fiber via vinyl monomer grafting for cementitious composite applications. Polymers, 16(11), 1645. https://doi.org/10.3390/polym16111645

[6] Zollo, R. F. (1984). Collated fibrillated polypropylene fibers in FRC. In Fiber reinforced Concrete International Symposium (pp. 397–409). American Concrete Institute.

[7] Mindess, S., & Vondran, G. (1988). Properties of concrete reinforced with fibrillated polypropylene fibers under impact loading. Cement and Concrete Research, 18(1), 109–115. https://doi.org/10.1016/0008-8846(88)90127-5