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Research on the Corrosion Modification Mechanism and Application of Polypropylene Fibers Based on a Combined System of Plasma Pretreatment and Chemical Etching

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Abstract: Polypropylene (PP) fibers, despite their advantageous properties, are limited by surface inertness and low chemical activity. This study develops a controllable modification technology using a combined plasma pretreatment and chemical etching system. The proposed "plasma activation-chemical etching synergy" mechanism first activates the fiber surface via plasma treatment, creating active sites. Subsequent chemical etching then constructs micro-nano rough structures and exposes more active groups. An experimental plan is designed to optimize this process and characterize the results using SEM, XPS, contact angle measurement, and tensile testing. Theoretically, this approach is expected to significantly enhance surface reactivity and wettability while maintaining mechanical properties, providing an efficient pathway to expand PP fiber applications in functional materials.

Keywords: Polypropylene fiber; Etching modification; Plasma pretreatment; Chemical etching; Surface activity

1. Introduction

1.1. Research Background and Significance

As one of the leading synthetic fibers in terms of production volume, polypropylene fiber boasts prominent advantages such as low density (only 0.91 g/cm^3), high specific strength, resistance to acid and alkali corrosion, and low processing cost, leading to its widespread use in various fields. In filtration, PP fiber filter media are used for air purification and wastewater treatment due to their high flux and low resistance. In biomedicine, they serve as base materials for tissue engineering scaffolds and medical sutures. [1] In composite materials, they are often used as reinforcing phases to improve the toughness and crack resistance of the matrix material.

However, the molecular chains of PP fibers consist of non-polar carbon-carbon backbones and methyl groups, resulting in very low surface energy (approximately 30 mN/m), strong chemical inertness, and a lack of active functional groups such as hydroxyl and carboxyl groups. These inherent characteristics lead to poor surface wettability and weak interfacial adhesion with other materials, becoming a key bottleneck restricting their functional expansion. In filtration applications, the inert surface has insufficient adsorption capacity for polar pollutants, limiting filtration efficiency. In biomedicine, the lack of cell adhesion sites on the surface makes it difficult to meet biocompatibility requirements. In composites, weak interfacial bonding with polar matrices (e.g., resins, cement) prevents the full utilization of their reinforcing effect. [2]

1.2. Necessity of Polypropylene Fiber Corrosion Modification

Surface corrosion modification of PP fibers, by constructing rough surfaces and introducing active functional groups to improve their surface chemical activity and interfacial interaction capability, has become key to breaking through application bottlenecks. Modification needs to achieve the following goals: (1) Construct controllable micro-nano scale rough structures on the fiber surface to enhance physical adsorption and mechanical interlocking; (2) Introduce polar functional groups such as oxygen-containing and nitrogen-containing groups to enhance chemical interactions with polar substances; (3) Ensure the modification effect while avoiding damage to the fiber's bulk mechanical properties, guaranteeing its stability in use. An ideal corrosion modification technology should feature controllable effects, simple process, moderate cost, and ease of industrial scaling.

1.3. Main Content, Approach, and Innovations of This Research

This study proposes a composite corrosion modification technology based on plasma pretreatment and chemical etching for the surface functionalization of PP fibers.

The main content includes: systematically reviewing the research status of PP fiber surface corrosion modification; elucidating the corrosion mechanism of the synergistic effect between plasma pretreatment and chemical etching; designing and optimizing the experimental plan, including plasma parameters, chemical etchant formulations, and process conditions; evaluating the modification effects through various characterization methods; analyzing the application potential and performance advantages of the modified fibers. [3]

Research Approach: The core is to construct a two-step "activation-etching" synergistic system. Step one: use plasma treatment (e.g., argon-oxygen plasma) to activate the PP fiber surface, where high-energy particle bombardment breaks surface molecular chains, generates free radicals, and introduces polar functional groups like carboxyl and hydroxyl, while increasing surface roughness. Step two: immerse the activated fibers into a specific chemical etchant, utilizing the selective corrosion of the fiber surface by the etchant to further amplify surface roughness while fully exposing the active functional groups, ultimately achieving a synergistic improvement in surface activity and interfacial bonding strength.

The innovations are mainly reflected in: combining the "mild modification" of plasma activation with the "deep regulation" of chemical etching to solve the problem of limited modification effects or damage to the fiber bulk inherent in single corrosion techniques; clarifying the synergistic corrosion mechanism to achieve precise regulation of fiber surface structure and functional groups; establishing a full-chain characterization system from surface microstructure to macroscopic performance to comprehensively evaluate the stability and practicality of the modification effect. [4]

2. Literature Review and Theoretical Basis

2.1. Structural Characteristics of Polypropylene Fibers and Bottlenecks in Corrosion Modification

Polypropylene is a semi-crystalline polymer with high molecular chain regularity. After melt spinning, the molecular chains are highly oriented along the fiber axis, forming a structure with high strength and low surface energy. The PP fiber surface lacks active functional groups, and the crystalline regions have a dense structure, making it difficult for conventional chemical reagents to penetrate and react. [5] This leads to two major challenges in corrosion modification: firstly, difficulty in forming uniform and controllable rough structures on the surface; secondly, the introduced active functional groups are few in number and poor in stability, unable to meet practical application requirements.

2.2. Research Progress in Surface Corrosion Modification Techniques for Polypropylene Fibers

Existing surface corrosion modification techniques for PP fibers are mainly divided into three categories: single chemical etching, plasma treatment, and composite modification.

2.2.1. Single Chemical Etching Method

This method uses strong acids, strong alkalis, or oxidants to corrode the PP fiber surface, destroying non-polar molecular chains and introducing polar functional groups. Commonly used etchants include chromic acid-sulfuric acid mixture, potassium permanganate solution, and sodium hydroxide solution. Studies show that the chromic acid-sulfuric acid system can effectively corrode the PP fiber surface, forming rough structures and introducing carboxyl groups. The advantages of this method are simple process and low cost, but the disadvantages are that the etching process is difficult to control, easily leads to uneven surface corrosion, the highly corrosive reagents cause significant environmental pollution, and they may damage the fiber's bulk mechanical properties.

2.2.2. Plasma Treatment Method

This method utilizes high-energy particles (e.g., electrons, ions, free radicals) in the plasma to bombard the fiber surface, achieving molecular chain scission and functional group introduction. Commonly used plasmas include argon, oxygen, and nitrogen plasma. Oxygen plasma treatment can introduce functional groups like carboxyl and hydroxyl on the PP fiber surface, improving surface wettability. [6] This method offers advantages such as fast modification speed, environmental friendliness, and surface-specific action. However, the modification effect of single plasma treatment is prone to decay, it is difficult to form deep rough structures, and long-term stability is insufficient.

2.2.3. Composite Modification Method

This method combines two or more modification techniques to exert a synergistic effect. For example, first activating the fiber surface via plasma pretreatment, followed by chemical etching or graft reaction. Research using plasma pretreatment followed by chemical etching found that both surface roughness and active functional group content were significantly higher than with single treatment. This method can balance modification effect and stability, but current research on the synergistic mechanism is not deep, and process parameter optimization lacks systematic guidance.

2.3. Mechanism and Application of Plasma Pretreatment

The core functions of plasma pretreatment are surface activation and roughness regulation. Its mechanisms include: high-energy particle bombardment causing PP molecular chain scission, generating a large number of free radicals; free radicals reacting with active species in the plasma (e.g., O_2^+, $OH\cdot$), introducing oxygen-containing polar functional groups; simultaneously, high-energy bombardment creates micro-etch pits on the fiber surface, increasing surface roughness. In material modification, plasma pretreatment is often used as an "activation step" for subsequent reactions, for example, to improve the interfacial bonding between fibers and resins, or to provide active sites for chemical etching, enhancing the reaction efficiency between the etchant and the fiber surface. [7]

2.4. Types of Chemical Etchants and Corrosion Mechanisms

Chemical etchants used for PP fiber corrosion are mainly divided into oxidative etchants and acid-base etchants. Oxidative etchants (e.g., chromic acid-sulfuric acid mixture, potassium permanganate solution) break carbon-carbon bonds in PP molecular chains through oxidation, generate polar functional groups, and dissolve part of the surface amorphous region, forming rough structures. Acid-base etchants (e.g., concentrated nitric acid, sodium hydroxide solution) destroy molecular chains through acid-base catalysis, achieving surface corrosion. Different etchants have varying corrosion mechanisms: the chromic acid-sulfuric acid system achieves selective corrosion through the strong oxidizing power of Cr^{6+} , preferentially attacking amorphous regions; the potassium permanganate system catalyzes oxidation reactions through the generated MnO_2 precipitate, resulting in a relatively mild corrosion process.

2.5. Theoretical Basis and Feasibility Analysis of the Plasma/Chemical Etching Composite System

Theoretical Basis: Plasma pretreatment reduces the interfacial energy between the chemical etchant and the fiber surface by introducing active functional groups and increasing surface roughness, thereby improving the wettability and reactivity of the etchant. Simultaneously, the presence of surface free radicals can accelerate the etching reaction rate, realizing a synergistic "activation-etching" effect. [8] Chemical etching, based on the plasma pretreatment, can further expand the surface rough structure, fully expose the introduced active functional groups, and avoid the problem of effect decay associated with single plasma treatment.

Feasibility Analysis: At the material level, plasma equipment and chemical etchants are commercially available conventional products with stable sources and controllable costs. At the process level, both plasma pretreatment and chemical etching are mature processes, simple to operate, and easy to combine and scale up. At the performance level, based on the modification effects of single techniques, the composite system is expected to achieve precise regulation of surface structure and functional groups, significantly improving the stability and practicality of the modification effect. [9]

3. Experimental Plan Design

3.1. Experimental Materials and Equipment

3.1.1. Main Experimental Materials

Polypropylene fiber: Commercially available conventional PP short-cut fibers, diameter 20 μm, length 6 mm, recording original performance parameters before use.

Chemical etchants: Potassium dichromate (analytical grade), concentrated sulfuric acid (analytical grade), potassium permanganate (analytical grade), deionized water, for preparing composite etchants of different concentrations.

Auxiliary reagents: Anhydrous ethanol (analytical grade), acetone (analytical grade), for fiber pretreatment.

Other materials: P.O 42.5 cement, ISO standard sand, for composite material performance testing.

3.1.2. Main Experimental Equipment and Instruments

Preparation and processing equipment: Plasma treatment instrument (argon-oxygen atmosphere), electronic balance (accuracy 0.0001g), constant temperature water bath, numerically controlled ultrasonic cleaner, electric blast drying oven, vacuum drying oven.

Characterization and testing instruments: Scanning Electron Microscope (SEM, equipped with EDS attachment), X-ray Photoelectron Spectrometer (XPS), static contact angle measuring instrument, electronic single fiber tensile tester, universal material testing machine.

3.2. Parameter Design and Optimization of the Composite Corrosion System

3.2.1. Plasma Pretreatment Parameter Design

Set plasma treatment parameter gradients: treatment power (30W, 50W, 70W), treatment time (3min, 5min, 7min), argon-oxygen volume ratio (3:1, 2:1, 1:1). Optimize the best pretreatment parameters using surface functional group content and roughness as evaluation indicators.

3.2.2. Chemical Etchant Formulation and Parameter Design

Etchant formulations: Design three etchant systems, Group A (Chromic acid-sulfuric acid mixture: $K_2Cr_2O_7$ 5g/L + H_2SO_4 30% v/v), Group B (Potassium permanganate-sulfuric acid mixture: $KMnO_4$ 3g/L + H_2SO_4 20% v/v), Group C (Concentrated nitric acid-hydrochloric acid mixture: HNO_3 : HCl = 1:3 v/v).

Etching parameters: Etching temperature (30°C, 40°C, 50°C), etching time (5min, 10min, 15min). Optimize the etching system and parameters using surface roughness and active functional group content as evaluation indicators.

3.2.3. Composite System Parameter Optimization

Set up control groups (single plasma treatment, single chemical etching) and composite treatment groups (different plasma parameters + different etching parameters). Determine the optimal composite modification parameter combination by comparing surface morphology, functional group content, wettability, and other properties.

3.3. Corrosion Modification Process for Polypropylene Fibers

3.3.1. Fiber Pretreatment

Place PP fibers in a Soxhlet extractor and continuously extract with acetone for 6 hours to remove surface oils, waxes, and other impurities. Remove and dry to constant weight in a 60°C vacuum drying oven, then store in a desiccator for later use.

3.3.2. Plasma Pretreatment Process

Spread the pretreated fibers flat on the sample stage of the plasma treatment instrument. Introduce the set ratio of argon-oxygen mixed gas, adjust the vacuum to 10 Pa, and perform plasma activation treatment according to the optimized parameters. After treatment, proceed immediately to subsequent chemical etching to avoid deactivation of surface free radicals. [10]

3.3.3. Chemical Etching Process

Completely immerse the plasma-activated fibers into the prepared chemical etchant and etch at the set constant temperature. After etching, quickly rinse the fibers with copious deionized water until neutral, then clean once with anhydrous ethanol to remove residual etchant.

3.3.4. Post-treatment Process

Place the etched fibers in a 60°C blast drying oven for 2 hours to remove surface moisture, obtaining the modified PP fibers.

3.4. Testing and Characterization Methods

3.4.1. Surface Morphology and Elemental Analysis (SEM/EDS)

Fix fiber samples on the sample stage with conductive adhesive, sputter-coat with gold, and observe surface micro-morphology via SEM, recording the size, distribution of etch pits, and roughness changes. Use EDS to analyze surface elemental composition, focusing on changes in O element content.

3.4.2. Surface Chemical State Analysis (XPS)

Use XPS to test the elemental composition and chemical bonding states of the fiber surface, scanning range 0-1000 eV, resolution 0.1 eV. Focus on analyzing the C1s spectrum, fitting and decomposing characteristic peaks for functional groups such as C-C, C-O, C=O, and calculate the relative content of each functional group.

3.4.3. Surface Wettability Test (Contact Angle)

Use the sessile drop method. Arrange fiber bundles parallel and tightly fixed on a glass slide. Add a 2 μL droplet of deionized water. Record the water droplet shape and calculate the static contact angle using the contact angle instrument. Measure 5 different locations per sample and take the average.

3.4.4. Mechanical Performance Test (Single Filament Tensile Strength)

Refer to ASTM D3822 standard. Use the electronic single fiber tensile tester to test the breaking force and elongation at break of fibers before and after modification. Gauge length 10 mm, tensile speed 2 mm/min. [11] Test 30 single filaments per group, calculate the average and standard deviation.

3.4.5. Composite Material Performance Evaluation

Refer to GB/T 17671 standard. Prepare reference mortar, mortar mixed with unmodified PP fibers, and mortar mixed with modified PP fibers into test specimens (40mm × 40mm × 160mm), with a fiber volume fraction of 0.1%. After standard curing for 28 days, test the flexural strength using the universal material testing machine to evaluate the reinforcing effect of the modified fibers on the composite material. [12]

4. Expected Results and Discussion

4.1. Analysis of Synergistic Effects of the Composite Corrosion System

The composite modification system with the optimal parameter combination is expected to achieve a synergistic effect between plasma activation and chemical etching. Compared to single treatment groups, the composite treatment group's fiber surface is expected to form more uniform and controllable micro-nano scale rough structures, with O element content and polar functional group content significantly increased. This indicates that plasma pretreatment effectively improves the reaction efficiency and uniformity of chemical etching, and the modification effect of the composite system is superior to that of single techniques. [13]

4.2. Evolution and Analysis of Modified Fiber Surface Chemical State (XPS)

The XPS spectrum of unmodified PP fibers is expected to show very low O element content (<1%), with the C1s spectrum dominated by the C-C bond (284.8 eV). After single plasma treatment, the O element content is expected to increase to 3%-5%, with characteristic peaks for C-O (286.3 eV) and C=O (288.5 eV) bonds appearing. After single chemical etching, the O element content is expected to be 5%-7%, with polar functional group content slightly higher than the plasma treatment group.

After composite treatment, the O element content is expected to reach 8%-12%, with the characteristic peak intensities for C-O and C=O bonds significantly enhanced. [14] This demonstrates that the composite system successfully introduces a large number of polar functional groups onto the fiber surface through synergistic action, laying the chemical foundation for enhanced surface activity.

4.3. Analysis of Modified Fiber Surface Morphology (SEM) and Elemental Distribution (EDS)

Unmodified PP fiber surfaces are expected to be smooth, with only a few spinning grooves. After single plasma treatment, slight etch pits are expected to appear on the surface, with a slight increase in roughness. After single chemical etching, irregular etch pits are expected to form, but with uneven distribution. [15]

After composite treatment, SEM images are expected to show uniformly distributed micro-nano scale rough structures on the fiber surface, with consistent etch pit size and moderate depth, and no obvious over-etching phenomenon. EDS analysis is expected to show uniform distribution of O elements on the composite-modified fiber surface, with content significantly higher than control groups, proving uniform distribution of polar functional groups on the surface.

4.4. Discussion on Surface Wettability and Activity Enhancement Effects

Unmodified PP fibers are expected to have a water contact angle greater than 90°, exhibiting hydrophobic characteristics. After single plasma treatment, the contact angle is expected to drop to 70°-80°, but gradually recover after 7 days of storage. After single chemical etching, the contact angle is expected to drop to 60°-70°, with stability slightly better than the plasma treatment group.

After composite treatment, the fiber's water contact angle is expected to drop to 40°-50°, with no significant recovery after 30 days of storage. This indicates that composite modification not only significantly improves surface wettability but also enhances the long-term stability of the modification effect. The mechanism lies in: the synergistic effect of the rough structure constructed by the composite system and the large number of polar functional groups enhances the interaction between the surface and water molecules; simultaneously, the stable rough structure formed by chemical etching prevents the loss of active functional groups, ensuring long-term stability. [16]

4.5. Impact of Modification on Fiber Bulk Mechanical Properties

Single filament tensile test results are expected to show that the breaking strength and elongation at break of the composite-modified fibers change by no more than 5% compared to unmodified fibers (no statistically significant difference). This indicates that this composite modification process only acts on the fiber surface without damaging the internal crystalline regions and molecular chain structure, preserving the original excellent mechanical properties of PP fibers and meeting application requirements in fields like composite materials. [17]

4.6. Analysis of Reinforcement Effect and Mechanism in Composites

The 28-day flexural strength of the reference mortar and mortar with unmodified fibers is expected to be similar, with an improvement of less than 5%. The mortar with composite-modified fibers is expected to show a 15%-25% increase in flexural strength, demonstrating a significant reinforcement effect.

Reinforcement Mechanism: The rough structure on the modified fiber surface forms a mechanical interlocking effect with cement hydration products, enhancing physical anchoring force. Simultaneously, the surface polar functional groups chemically interact with components of cement hydration products (e.g., Ca^{2+} , Si-OH in C-S-H gel), forming chemical bonds and enhancing interfacial bonding strength. [18] When the mortar is stressed, the strong interface can effectively transfer stress, fully utilizing the fiber's bridging effect, thereby improving the flexural strength and toughness of the composite material.

5. Conclusions and Outlook

5.1. Research Conclusions

Through systematic literature review, theoretical analysis, experimental plan design, and in-depth discussion of expected results, the following conclusions are drawn:

The proposed composite corrosion system of plasma pretreatment and chemical etching is theoretically and technically feasible for achieving efficient and stable surface modification of PP fibers.

The "plasma activation-chemical etching synergy" mechanism is reasonable. Plasma pretreatment provides active sites for chemical etching, while chemical etching expands the surface rough structure and stabilizes active functional groups, achieving a synergistic improvement in the modification effect.

Expected characterization results will confirm from multiple aspects — chemical state, microstructure, macroscopic performance—that composite modification can significantly enhance the surface activity, wettability, and interfacial bonding strength of PP fibers without damaging their bulk mechanical properties. [19]

This composite corrosion modification technology provides a precise and efficient new pathway for the surface functionalization of PP fibers, promising to promote their application expansion in fields such as filter materials, biomedicine, and composite materials.

5.2. Future Work Outlook

Experimental Verification and Parameter Optimization: Conduct experiments according to the design plan to obtain first-hand data, verify and revise theoretical expectations, and optimize plasma parameters, etchant formulations, and process conditions. [20]

In-depth Mechanism Research: Use methods such as molecular simulation and fine XPS analysis to reveal the synergistic corrosion mechanism at the molecular level and clarify the intrinsic relationship between functional group introduction and rough structure formation.

Development of Eco-friendly Processes: Explore green etchants (e.g., bio-enzymes, ionic liquids) to replace traditional highly corrosive reagents, reducing the environmental impact of the process.

Durability and Application Expansion: Systematically evaluate the performance stability of modified fibers under environments such as humidity, heat, and UV aging; apply modified fibers to specific products (e.g., high-efficiency filter media, biological scaffolds) to verify their practical application value. [21]

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