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Research on Equipment for the Preparation of PAN Photocatalytic Nanofiber Membranes

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Abstract: Efficient degradation of organic dye wastewater is a core urgent problem to be solved in the field of water pollution control. Conventional water treatment processes suffer from low degradation efficiency and severe secondary pollution, and cannot meet the discharge standards of industrial wastewater with complex compositions and fluctuating concentrations. This is the core reason for the rapid popularization and practical application of green photocatalysis technology. Visible light photocatalysis is driven by clean solar energy, featuring environmental friendliness, mild reaction conditions, and no demand for high-temperature and high-pressure auxiliary equipment, which makes it highly suitable for large-scale treatment of industrial dye wastewater. Polyacrylonitrile (PAN) nanofiber membranes possess unique merits including low cost, high flexibility, stable structure, and abundant pores. Through multi-step precise chemical modification to support heteropolyacid active components, recyclable visible-light-responsive photocatalytic membrane materials with high degradation efficiency and excellent stability can be fabricated.

Keywords: PAN photocatalysis; Nanofiber membrane; Preparation; Equipment research

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1. Equipment system for electrospinning of virgin PAN nanofiber membranes

The electrospinning equipment system for virgin PAN nanofiber membranes is developed to produce polyacrylonitrile substrate nanofiber membranes with uniform structure, stable performance, and low defect rate, which provides standardized, defect-free base materials for subsequent multi-step refined chemical modification and experimental research on visible light photocatalytic performance.

1.1. Equipment functional orientation

The complete electrospinning device consists of three independent units: pretreatment, main molding unit, and environmental control unit, which have clear divisions of labor and operate cooperatively throughout the whole process to form an integrated molding system. The pretreatment unit can effectively break up agglomerated powder particles, remove raw material impurities and bubbles, and prepare uniform, stable, non-delaminated spinning solutions. The main machine cooperates with the environmental control unit to jointly guarantee the overall molding quality and microstructural uniformity of nanofiber membranes. The equipment supports customized spinning parameters exclusive to polyacrylonitrile materials and can store mature process data for long-term use, which effectively eliminates errors caused by manual debugging and ensures consistency and repeatability of experiments across batches. The single molding duration can be precisely regulated, and the finished product size perfectly matches the feeding specifications of subsequent modification equipment, which minimizes mechanical loss during substrate cutting and transfer and improves raw material utilization [1].

1.2. Core equipment structure and operating parameters

The spinning solution pretreatment equipment is composed of an ultrasonic cleaner, constant-temperature stirrer, and sealed liquid storage device. All structural parts in contact with materials are made of corrosion-resistant materials to adapt to long-term operation with strong polar organic solvents. Stable dissolution and dispersion processes eliminate molding defects such as fiber beads and broken filaments at the source, and fully guarantee the comprehensive quality and long-term storage stability of spinning solutions. The TL-Pro fully enclosed electrospinning main machine serves as the core molding equipment. Its high-precision high-voltage electric field, micro quantitative liquid supply, and dynamic roller collecting system can precisely match the exclusive spinning process of high-viscosity PAN spinning solutions. The equipment is equipped with intelligent parameter monitoring and automatic overload protection functions to monitor voltage, flow rate, and rotational speed in real time, which ensures the molding precision and uniform diameter of nanofibers and greatly improves the overall safety of experimental operations. The supporting intelligent temperature and humidity control unit can precisely regulate the microenvironment inside the spinning chamber to maintain optimal conditions for steady, uniform volatilization of organic solvents and avoid structural defects caused by excessively fast or slow solvent evaporation. The integrated negative pressure exhaust and waste gas adsorption device stabilizes fiber-forming conditions and prevents uneven membrane thickness induced by airflow fluctuation, while eliminating safety and environmental hazards brought by organic solvent volatilization [2].

1.3. Adaptability analysis between equipment and PAN spinning process

The low-speed uniform output mode of micro-injection pumps is highly compatible with the refined molding demands of high-viscosity PAN spinning solutions, avoiding pulse output defects of conventional pumps. The continuous and stable fluid output fully stretches and refines fiber structures and sufficiently exposes cyano active groups on membrane surfaces, laying a solid structural foundation for subsequent cyclization and quaternization modification reactions. The stepless adjustable roller speed of the equipment generates moderate stretching orientation on deposited nanofibers and optimizes the internal molecular arrangement of fibers [3]. A reasonable rotational speed range balances fiber flexibility and overall structural integrity, and completely eliminates structural defects such as loose fiber stacking, fragile membranes, and overstretched broken filaments. Constant temperature and humidity conditions precisely control solvent volatilization rates to prevent fiber adhesion and pore closure, facilitating the preparation of high-quality fiber membranes with high porosity, large specific surface area, and loose structures. The abundant three-dimensional pore channels inside the membranes can boost mass transfer efficiency and reaction uniformity during subsequent functional group modification and ion exchange, reducing incomplete local reactions. The fully enclosed and corrosion-resistant structure of the equipment adapts to continuous experiments with organic solvents for a long time without pipeline corrosion, material leakage, or solvent volatilization. The standardized process parameters stored in the system minimize batch experimental errors and greatly enhance the stability and repeatability of substrate preparation.

2. Complete equipment system for chemical modification of PAN fiber membranes

Virgin polyacrylonitrile fiber membranes only possess physical screening and supporting structures without any photocatalytic activity or pollutant degradation capacity. Multi-step refined chemical modification is mandatory to realize functional upgrading of materials. The complete set of modification equipment covers full procedures, including high-temperature reflux reaction, multi-solvent cleaning, constant-temperature ion exchange, and vacuum drying, and ensures stable, standardized, and efficient progress of modification reactions.

2.1. Equipment functional orientation

The four modification equipment units perform their respective duties and connect in sequence to form a complete functional modification production line, which sequentially completes core modification reactions such as cyclization and quaternization of fiber surface functional groups. Standardized equipment operating conditions largely reduce residual impurities and significantly improve the loading uniformity and interfacial bonding stability of active components. All material-contact parts of the modification equipment are made of anti-corrosion and solvent-resistant materials to fully adapt to complex organic systems and alternating acid-base working conditions. The exclusive flexible membrane clamping tool matched with the equipment effectively prevents stretching and breakage of fiber membranes during soaking and transfer, greatly improving the overall success rate and intact rate of experiments [4].

2.2. Core equipment structure and operating parameters

The double-layer glass reactor acts as the core modification reaction equipment. Its high-precision closed constant-temperature system perfectly meets the demands of long-term reflux reactions lasting several hours to more than twenty hours, with far superior temperature stability compared with conventional heating equipment. The matched condensation reflux and low-speed stirring structure maintains stable solvent concentration and temperature of the reaction system, fully guaranteeing sufficient modification reactions of fiber surface functional groups. The special membrane cleaning workstation abandons the extensive stirring and soaking mode of traditional equipment and adopts multi-angle spraying to treat soft, flexible fiber membranes in a targeted manner. The standardized multi-solvent alternating cleaning procedure set by the equipment removes residual organic reagents and free impurities layer by layer, ensuring the authenticity and accuracy of subsequent photocatalytic performance test data [5]. The constant-temperature magnetic stirring water bath provides mild, low-temperature liquid-phase environments for anion exchange reactions and avoids decomposition and deactivation of active components caused by high temperatures. The fully sealed oxygen-isolated design of the equipment prevents oxidation of heteropolyacid active components and remarkably improves their loading uniformity and bonding strength inside fiber membranes. The vacuum low-temperature drying oven rapidly removes residual organic solvents and water inside membrane pores under low-pressure vacuum conditions to shorten the drying cycle. The mild low-temperature vacuum working condition will not damage fiber skeletons or surface-active functional groups, and completely preserves the microstructures and visible-light photocatalytic performance of modified membranes [6].

2.3. Adaptability analysis between equipment and modification processes

The circulating heat conduction temperature control mode of double-layer glass reactors features tiny temperature drift and superior temperature stability to conventional water baths, which is highly compatible with long-term continuous organic modification reactions. The sustained stable high-temperature environment fully activates inert cyano groups on fiber surfaces and greatly raises the grafting efficiency and overall uniformity of cyclic active functional groups. Modified fiber membranes undergo an obvious decline in mechanical properties after multi-step chemical reactions, featuring reduced flexibility and loose structures, and are prone to breakage during cleaning. The special clamping tools matched with the equipment can fully protect membrane structures while thoroughly removing free impurities on membrane surfaces, effectively avoiding inflated deviations in photocatalytic experimental data [7]. The mild low-speed stirring condition of the equipment prevents shedding of functional groups on membrane surfaces and ensures smooth and ordered solid-liquid ion exchange reactions. Constant temperature accelerates anion diffusion and migration, enabling uniform loading of heteropolyacid active components inside the three-dimensional fiber skeletons and improving loading stability. Polyionic liquid functional groups have poor high-temperature resistance; traditional high-temperature drying easily causes decomposition and shedding of active components. The vacuum low-temperature drying process avoids this technical problem and stably retains intact microstructures and visible-light catalytic activity of modified membranes.

3. Complete equipment system for characterization and performance testing of photocatalytic membranes

The quality of modified photocatalytic fiber membranes cannot be intuitively judged only by preparation process parameters. Systematic and comprehensive testing is required to verify their microstructural characteristics and practical catalytic performance. The complete set of testing equipment covers full workflows of structural characterization, performance detection, and sample pretreatment, and provides reliable data support for material performance analysis and process optimization. The complete testing equipment system serves as a core link connecting material preparation and performance verification. It breaks the barrier between equipment processes, material structures, and catalytic performance, accurately identifies process defects and performance deficiencies generated during preparation, and provides an all-round, high-precision experimental basis for iteration of modification and optimization of equipment parameters. It is an important foundation for guaranteeing the scientificity and repeatability of overall experiments.

3.1. Equipment functional orientation

Multi-dimensional precision characterization equipment comprehensively analyzes the micro-morphology, chemical composition, crystal structure, and photoelectric properties of fiber membranes, and establishes an internal correlation between material microstructures and macroscopic catalytic performance. The special visible light testing system can quantitatively measure the dye degradation capacity and long-term cycling stability of materials, and objectively evaluate their practical industrial application value. The complete testing equipment is highly compatible with the material characteristics and testing requirements of PAN-based modified photocatalytic membranes, featuring strong equipment compatibility, high detection precision, and excellent repeatability. The platform supports simultaneous parallel testing of multiple groups of samples, which can intuitively compare performance differences of materials prepared via different modification processes and different active component loading routes [8].

3.2. Core equipment structure and operating parameters

Various precision characterization instruments have clear divisions of labor and complement each other. They conduct all-round quality detection from multiple dimensions, including micro-morphology and surface functional groups. The comprehensive detection combining multiple testing methods can accurately judge the completion degree of modification reactions and the actual loading quality and uniformity of active components. Mutual verification among different characterization devices avoids one-sided results caused by single detection means, fully analyzes the influence mechanism of equipment processes on material microstructures, and precisely locates tiny differences in fiber pore structures, functional group distribution, and active component loading, providing sufficient micro-data support for analyzing equipment adaptability and material performance rules. The visible light photocatalytic testing system accurately screens pure visible light bands via special filters, eliminating ultraviolet interference and fully conforming to laboratory research standards and practical water treatment scenarios [9]. The fully light-shielded, constant-temperature, and automatic continuous sampling design integrated into the equipment minimizes external environmental interference and guarantees precise and reliable degradation experimental data. The supporting sample pretreatment equipment standardizes basic operations such as sample preparation, unifying preparation standards for the whole experimental flow. Unified sample preparation standards eliminate random errors induced by manual operations and fully ensure the scientificity and comparability of multiple parallel experimental data.

3.3. Adaptability analysis between equipment and testing processes

The multi-dimensional integrated characterization system comprehensively verifies the practical effects of modification processes and accurately screens defects in material microstructures and unstable loading of active components. The equipment is highly adapted to the testing characteristics of flexible thin-film samples; it simplifies sample preparation without damaging the original microstructures and active systems of samples. The multi-layer optical shielding and band filtering design of the testing system completely eliminates experimental interference from ambient stray light and ultraviolet rays, ensuring single and stable light sources. The stable automatic sampling mode guarantees continuous progress of photocatalytic reactions and precisely fits the kinetic laws of dye degradation to truly restore the intrinsic photocatalytic performance of materials. The three types of testing, cleaning, and drying equipment form a closed-loop testing system to efficiently and continuously complete long-term cycling stability tests and shorten experimental cycles [10].

Fiber membranes can be directly clamped and recovered, which thoroughly solves the technical pain points of easy loss and difficult recovery of active components of traditional powder catalysts, making test results more consistent with practical industrial application scenarios. The complete closed-loop testing equipment system fits the operating logic of industrial water treatment equipment and can simulate continuous working states of actual water treatment processes. It effectively verifies the structural stability and performance retention rate of photocatalytic membranes during long-term service, accurately predicts the industrial service life and application feasibility of materials, and provides authentic experimental reference for subsequent large-scale equipment transformation and industrialization of technologies. High-precision spectrophotometers are specially adapted to micro-detection of low-concentration organic dye solutions with extremely low detection errors and excellent linear fitting, delivering highly credible test data. The instrument can capture tiny performance differences of materials under different process conditions and provide solid data support for subsequent process optimization, performance improvement, and breakthroughs in degradation efficiency.

4. Conclusion

The three major equipment systems of electrospinning molding, chemical modification, and performance characterization form a full-process closed-loop process for preparing PAN photocatalytic nanofiber membranes, realizing integration of preparation and detection. The equipment parameters of each system match each other with complementary functions and can stably produce recyclable photocatalytic functional membranes with intact structures and excellent performance. This customized special equipment system targets many industry pain points of traditional photocatalytic materials, such as difficult preparation, high recovery cost, and easy secondary pollution, and possesses remarkable advantages in production cost and service performance. The modular, upgradable, and automatically iterative characteristics of the equipment can adapt to the demands of pilot and industrialization in the future, providing solid equipment support and systematic technical reference for the industrial application of visible light photocatalytic water treatment technology.

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Disclosure statement

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