Nanoparticle-Enhanced Bamboo-Like Tubular Nanofibers for Active Capture of Particulate Matter

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ABSTRACT: We proposed a thought of active capture of particles by improving the interaction force between fibers and particles. Nanoparticle-enhanced tubular nanofibers (Ag-SPNTs) were prepared by template-free cationic polymerization followed by surface modification. Ag-SPNTs have coarse surface and bamboo-like tubular structure with a diameter of approximately 80-150 nm. Ag nanoparticles were embedded on the nanofibers surface, and the content of Ag nanoparticles in the nanofibers could be tuned by changing the concentration of $[\text{Ag}(\text{NH}_3)_2]^+$ in the preparation process. *f-d* curve measured by AFM showed that increasing the content of Ag nanoparticles in the nanofibers resulted in the enhanced interaction force between the nanofiber surface and particles. Particle matter capture test showed that

the number of captured microscaled/naonoscaled particles on the fiber surface increased obviously for the nanoparticle-enhanced tubular nanofibers (Ag-SPNTs) compared to the nanofibers without nanoparticle (SPNTs), probably due to the increased interaction force and adhesion energy between fiber surface and particles. Filtration property test showed that the Ag-SPNTs fiber films had a better filtration performance with a higher filter efficiency and QF value than that of SPNTs. © 2019 Wiley Periodicals, Inc. *J. Polym. Sci., Part A: Polym. Chem.* **2019**

KEYWORDS: air filtration; interaction forces; particulate capture; tubular nanofibers

INTRODUCTION Nowadays, urban air pollution has attracted more and more attention along with the rapid economic development.¹⁻³ Especially the breakout of haze in many metropolitan cities, which was mainly caused by particulate matter (PM), has resulted in a serious impact on human health as well as the global climate.⁴⁻⁶ There are several common protective strategies for PM pollution, such as adsorption using water, plants, and filtration. Different particulate filters including solid foam filter,⁷ fiber filter,⁸ and wall-flow ceramics filter⁹ have been investigated. Among these particle-removal technologies, fiber filter was used extensively for air cleaning due to its simple structure and easily accessible raw materials.

At present, research on fiber filter for aerosol filtration was primarily focused on the control of fiber size and morphology.

Studies showed that the filtration efficiency increased with the decrease of fiber size because nanofibers with smaller diameter have a higher probability of inertial collision and interception for PM,^{10,11} while the pressure drop and energy cost also increased.¹² Nanofibers with diameter range from tens to hundreds of nanometers have been reported for air filters due to their high surface area and small pore size.^{13,14} Besides, fibers with various morphological structures such as straight, ribbon-like, spindle-like beaded have also been reported, and filters composed of beaded fiber showed the best quality in the air filtration.¹⁵ In addition, composite fiber filter consisting of two/three-tier, that is, a nano-fiber/net (NFN) top layer and a conventional nonwoven microfibrinous support, was demonstrated and showed highly efficient and low-pressure drop filtration for particulate filtration.¹⁶

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However, either the control of fiber size and morphology or constructing multitier composite fiber filter, particles were intercepted due to the restriction of pore size in the filter membrane, which was a simple and passive mechanical barrier for particles. In fiber filter for aerosol filtration, particles may deposit on the fiber through several mechanisms, including inertial impaction, interception, Brownian diffusion, gravitational force, and electrostatic forces.^{17,18} The first four mechanisms are known as mechanical capture mechanisms. Electrostatic forces arise when fibers or particles were electric charged or an external electric field is applied to the filter. Wang's group^{19,20} developed triboelectric nanogenerator-enhanced nanofiber air filter for PM removal using electrospinning, and the removal efficiency was enhanced when an electric field was used due to the electrostatic forces between fibers and particles.

Herein, we proposed a thought of active capture of particles by improving the interaction force between fibers and particles based on nanoparticle-enhanced fibers using metal or metal oxide nanoparticles with high surface energy for fiber modification. In this work, Ag nanoparticles were selected to construct the enhanced composite nanofibers because of its excellent performance. The synthesis and characterization of the nanoparticle-enhanced nanofibers were demonstrated, and the capture of particulate matter as well as filter performance using the fiber films was evaluated.

EXPERIMENTAL

Materials

Divinyl benzene (80%) (DVB) was purchased from Aladdin Reagent Company (Shanghai, China). Boron trifluoride diethyl etherate complex ($\text{BF}_3 \cdot \text{O}(\text{Et})_2$) was purchased from TCI (Shanghai) Development Co, Ltd, Shanghai, China. Concentrated sulfuric acid (H_2SO_4 , 98%), ammonia solution ($\text{NH}_3 \cdot \text{H}_2\text{O}$, 28%), silver nitrate, glucose, ethanol, and *n*-heptane were purchased from Sinopharm Group Chemical Reagent Co, Ltd, Shanghai, China.

Preparation of the Sulfonated PDVB Nanotubes (SPNTs)

The PDVB nanotubes (PNTs) were synthesized as previously reported.²¹ Briefly, 64 μL of $\text{BF}_3 \cdot \text{O}(\text{Et})_2$ was immediately added into 75 mL of *n*-heptane containing 1 mL of DVB monomer under ultrasonic stirring at room temperature. The reaction was allowed for 10 min till a large amount of white precipitate was produced, and then 2 mL of ethanol was added to terminate the polymerization. The samples were filtered and washed with ethanol, and then dried in a vacuum oven at 50°C. Subsequently, 1 g of the as-prepared PDVB nanotubes was added into 30 mL of concentrated sulfuric acid under stirring at 50°C. After 12 h reaction, the mixture was dropwise added into excess ice water and filtered to collect the brown product of SPNTs.

Preparation of Ag Nanoparticles-Enhanced Sulfonated PDVB Nanotubes (Ag-SPNTs)

Ag-SPNTs were achieved by the following process. Briefly, 0.1 g of the as-prepared SPNTs were redispersed in 10 mL of freshly prepared aqueous solution of $[\text{Ag}(\text{NH}_3)_2]^+$ (0.5 or 1%), and the mixture was gently stirred at room temperature for 12 h.

Subsequently, the mixture was filtered and transferred to 10 mL of glucose aqueous solution (5 wt%) under stirring at room temperature for another 12 h. The products were collected by centrifugation and then dried in a vacuum oven at 50°C. Ag-SPNTs obtained from 0.5 and 1% $[\text{Ag}(\text{NH}_3)_2]^+$ were labeled as Ag-SPNTs-1 and Ag-SPNTs-2, respectively.

Characterization

Morphology and structure of the samples were characterized using SEM (Nova NanoSEM 450, FEI, 5 KV) and TEM (H-7650, Hitachi, 80 KV). Fiber diameters were measured with digital images using software Image J according to the previous report.²² Diameters of each fiber in SEM images were measured, and the average fiber diameters along with the standard deviation values were then calculated. Energy-dispersive X-ray spectroscopy (EDX) was conducted on the SEM to examine the surface composition of samples where needed. FTIR spectroscopy was tested using a Nicolet 6700 spectrometer with KBr-pressed pellets. The amount of Ag nanoparticles in the Ag-SPNTs was measured by TGA using a TG (SII TG/DTA7300, Exstar) with a heating rate of 10°C min⁻¹ from room temperature to 600°C under an air atmosphere. Powder XRD was measured using XRD (D8 Focus, Bruker).

Measurement of the Interaction Force

The interaction force between the probe and tubular nanofibers with or without Ag nanoparticles was estimated by *f-d* curve (force-versus-distance) measurement using AFM (Ntegra Spectra, NT-MDT). The *f-d* curve was obtained from the *DFL-Height* curve directly measured by AFM using a semi-contact mode. In detail, SPNTs and Ag-SPNTs were re-dispersed in ethanol at a very dilute concentration and air-dried to gain one single nanofiber on the silica substrate for the test. The current on the nanofiber surface (nA) was detected by the AFM probe when approaching or retracting the fiber surface. The interaction force (nN) between the probe and fiber surface was calculated using the following equation: $F = DFL \times K \times S$, where *DFL* is the current on the fiber surface detected by the AFM probe; *K* is the cantilever force constant, which is 1.068 nN·nm⁻¹; *S* (nm·nA⁻¹) is the reciprocal of the slope of the *DFL-Height* curve.

Particulate Matter Capture Test

The obtained SPNTs and Ag-SPNTs were redispersed in ethanol and spread out to form thin films of 5 × 5 cm. The prepared films were vertically fixed on a plastic scaffold and then placed at the roadside of the main street around Ningbo University with heavy traffic flow to allow free capture of particulate matters.

Filtration Property Measurement

The obtained SPNTs and Ag-SPNTs were redispersed in water and spread out on PP nonwovens (thickness of 2.6 μm) scaffolds to form thin films with a diameter of 15 cm. The filtration efficiencies and pressure drops of the prepared fiber films were tested using an automated filter tester (model 3601, TSI) to evaluate the filtration performance of the films according to GB2626-2006. The dioctyl-phthalate (DOP)

FIGURE 1 Illustrative synthesis of SPNTs and Ag-SPNTs. [Color figure can be viewed at wileyonlinelibrary.com]

particles with a size of 0.33 μm were generated from an oil generator. Then, the DOP aerosols were processed by dilution and mixed with air through the filters. The face velocity of the supplied air was 5.5 cm/s, and the number of the DOP particles was analyzed by a condensation particle counter before and after the filters. The quality factor (QF) of the filter, which is often used to evaluate the filtration performance of filters, is defined as follows: $QF = -\ln(1-E_f)/P_d$, where E_f is the filtration efficiency, and P_d is the pressure drop across the fiber filter.²³

RESULTS AND DISCUSSION

Preparation and Characterization of SPNTs

As shown in Figure 1, the PDVB nanotubes (PNTs) synthesized by cationic polymerization were further sulfonated by concentrated

sulfuric acid to introduce the surface of polymer tubes with sulfonic acid groups ($-\text{SO}_3\text{H}$) and harvest the SPNTs. SEM and TEM displayed that SPNTs were bamboo-like tubular nanofibers with a diameter of approximately 80–150 nm, and the surface of SPNTs was as smooth as that of PNTs (Fig. 2). In comparison with the original PNTs, new peaks at 1175 and 1025 cm^{-1} appear in the FTIR spectrum of SPNTs (Fig. 3), confirming that the sulfonic acid group was successfully grafted to the polymer tubes.²⁴ The introduced $-\text{SO}_3\text{H}$ could facilitate the following adsorption of $[\text{Ag}(\text{NH}_3)_2]^+$ and be conducive to a feasible loading of noble metal.²⁵

Preparation and Characterization of Ag-SPNTs

Ag-SPNTs composite nanotubes demonstrated much coarse surface with many nanoscaled particles embedded on the tube surface in comparison with PNTs and SPNTs [Fig. 2(c,f)]. Element analysis upon the coarse nanoparticles in Ag-SPNTs showed that the nanoparticles were composed of silver, confirming the successful loading of Ag nanoparticles (Supporting Information Fig. S1). The fiber diameter of Ag-SPNTs (239 ± 62 nm) was a bit increased compared to that of SPNTs (152 ± 32 nm) and PNTs (163 ± 40 nm), possibly due to the swelling of sulfonated

FIGURE 2 SEM, TEM images and the diameter distribution of PNTs (a, d, g), SPNTs (b, e, h) and Ag-SPNTs (c, f, i).

FIGURE 3 FTIR spectra of PNTs, SPNTs, and Ag-SPNTs. [Color figure can be viewed at wileyonlinelibrary.com]

layers in the $[\text{Ag}(\text{NH}_3)_2]^+$ adsorption process. The content of Ag nanoparticles in Ag-SPNTs could be tuned by changing the concentration of $[\text{Ag}(\text{NH}_3)_2]^+$ in the adsorption process. As confirmed by the TGA, the content of Ag in Ag-SPNTs increase from 8.4 wt% (Ag-SPNTs-1) to 19.7 wt% (Ag-SPNTs-2) when the concentration of $[\text{Ag}(\text{NH}_3)_2]^+$ increased from 0.5 to 1 mg mL⁻¹, as shown in Figure 4. XRD analysis confirmed the crystal structures of Ag nanoparticles in the Ag-SPNTs composite nanotubes (Fig. 5). The sharp and strong diffraction peaks at 2θ angles of 37.9, 44.1, 64.3, 77.2, and 81.4° were precisely indexed to Ag with an fcc (face-centered cubic) structure, reflecting (111), (200), (220), (311), and (222) crystalline planes (JCPDS No.04-0783), respectively. It was interesting to note that the residues of Ag-SPNTs after TGA under an air atmosphere were Ag not Ag₂O, thus the content of Ag nanoparticles in Ag-SPNTs could be measured directly by TGA. This was because that Ag oxidation and Ag₂O reduction were reversible reactions at the critical temperature (148°C), and the rate of reduction of silver oxide is rapid enough, while the oxidation of silver is sluggish, so silver will not form an oxide in a reasonable period of time upon cooling below the critical temperature.²⁶

FIGURE 4 TGA analysis of SPNTs and Ag-SPNTs obtained from different concentration of $[\text{Ag}(\text{NH}_3)_2]^+$. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 5 XRD patterns of SPNTs, Ag-SPNTs before and after TGA test. [Color figure can be viewed at wileyonlinelibrary.com]

Interaction Forces between the Fiber Surfaces and Particles

Ag nanoparticles were loaded in the polymer tubes in order to enhance the interaction force between the fiber surfaces and particles due to their high surface energy. The interaction forces between particles and polymer tubes with different content of Ag nanoparticles were evaluated through the force-distance (f - d) curve. AFM was utilized to directly measure the interaction force between the tubular nanofiber surface and AFM probe in an air atmosphere at room temperature. Figure 6 showed the AFM images of the single tubular nanofiber of SPNT and Ag-SPNTs with different content of Ag nanoparticles, displaying a fiber shape with a diameter similar to the SEM results.

The interaction force increased when the AFM probe was approaching the nanofiber surface and decreased when retracting. Unsurprisingly, the interaction force increased with the increasing content of Ag nanoparticles owing to the higher surface energy of metal nanoparticles than organic matter.²⁷ As shown in Figure 6(d), the force for SPNTs was 1348 nN when the AFM probe touched the fiber surface, and the value increased to 2917 and 6301 nN for Ag-SPNTs containing 8.4 and 19.7 wt% Ag nanoparticles, respectively. It has been reported that particles are deposited and captured upon collision with the filter surface due to the adhesion energies between particles and a surface when using filter for the particle-removal,¹¹ and a variety of theories have been developed for calculating the adhesion energy between a particle and a surface, including Bradley-Hamaker (BH), Johnson-Kendall-Roberts (JKR), Derjaguin-Muller-Toporov (DMT), and Maugis-Pollock (MP) models. Both of these theories took van der Waals interactions into account, considering that the surface adhesive energy per unit area results from van der Waals interactions.^{11,28,29} Thus, the increased and enhanced interaction force between Ag-SPNTs surface and particles would increase the surface adhesive energy, which would be facilitated for particles capture. Studies also showed that nanoparticles would escape from capture by fiber as the gas molecules do if it is sufficiently small (< 10 nm) due to the thermal rebound, and thus reduce the filter efficiency.^{11,17,30} The adhesion of a particle to a surface is

FIGURE 6 AFM images of a single fiber of SPNTs (a), Ag-SPNTs-1 (b), and Ag-SPNTs-2 (c); the approaching and retracting trace of the f - d curve between the single fiber and AFM probe in an air atmosphere at room temperature (d). [Color figure can be viewed at wileyonlinelibrary.com]

related to its impact velocity. If the kinetic energy of the particle exceeds the adhesion energy, it will rebound from the surface. Critical velocity is the threshold impact velocity above

which rebound takes place. Wang and Kasper found that the critical velocity is proportional to the 5/6 power of the specific adhesion energy and inversely proportional to the same power

FIGURE 7 SEM images of the fiber film before and after dust capture at the roadside, SPNTs (a, d), Ag-SPNTs-1 (b, e), and Ag-SPNTs-2 (c, f). Inset is the magnified images. The white arrow shows the captured microscaled dust particles, and the red arrow shows the captured nanoscaled particles. [Color figure can be viewed at wileyonlinelibrary.com]

FIGURE 8 Illustrative graph of particulate matter captured on the SPNTs film (a), and active capture of particulate matter on the nanoparticle-enhanced Ag-SPNTs film (b). [Color figure can be viewed at wileyonlinelibrary.com]

of particle diameter in their analysis of thermal rebound using the JKR model.^{17,30} Thus, for small particles with a definite size, the increase of interaction force between fiber surfaces and particles could increase the specific adhesion energy and improve the capture of small particles.

Particulate Matter Capture Test

To evaluate the particle capture performances of the tubular nanofibers, the obtained SPNTs and Ag-SPNTs were redispersed in ethanol and then spread out to form thin films of 5×5 cm. The prepared films were vertically fixed on a plastic scaffold and then placed at the roadside of the main street around Ningbo University with heavy traffic flow to allow free capture of particulate matters for 1 h. As shown in Figure 7, SPNTs and Ag-SPNTs demonstrated relatively neat surface with no particles deposited before dust capture test [Fig. 7(a-c)]. There were a few microscaled particles deposited on the SPNTs surface after dust capture test, and the number of deposited microscaled particles on the film surface increased obviously for the Ag-SPNTs fiber films [Fig. 7(d-f)], probably because of the increased adhesion energies between particles and Ag-SPNTs surfaces due to the enhanced interaction force, as illustrated in Figure 8. Besides the capture of microparticles, nanoparticles were also deposited on the surface of the Ag-SPNTs film, as shown in the inset magnified images (red arrow), while seldom nanoparticles were observed on the SPNTs film surface. Thermal rebound theory considered that airborne nanoparticles striking a filter surface will rebound if their initial kinetic energy surpasses the adhesion energy between particles and surfaces for particles between 1 and 10 nm,^{30–32} consequently decrease the capture efficiency of such small particles. Hence, the capture of nanoparticles for Ag-SPNTs film may be attributed to the increased adhesion energy between the Ag-SPNTs

FIGURE 9 EDX spectra of the selected area (red box) of the film before and after dust capture, SPNTs (a, c) and Ag-SPNTs-2 (b,d). [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 1 Parameters, Filter Efficiency, Press Drop and QF of the SPNTs and Ag-SPNTs-2 Fiber Films

Samples	Packing density (g/cm ²) ^a	Thickness of the film (μm) ^b	Filter efficiency (%)	Pressure drop (Pa)	QF(Pa ⁻¹) ^c
SPNTs	0.0113	5.4	61.6	44	0.022
Ag-SPNTs-2	0.0113	6.0	84.4	60	0.031

^a Calculated by the following formula: the mass of fibers/the film area.

^b Measured by a vernier caliper.

^c Calculated using the equation, $QF = -\ln(1-E_f)/P_d$.

surface and particles, which was resulted from the enhanced interaction force between nanoparticles and Ag-SPNTs containing Ag nanoparticles with high surface energy.

EDX spectra showed C, O, and S element in the SPNTs film, which is originated from the sulfonated PDVB polymer tubes (Fig. 9). In comparison, a new peak assigned to Ag appeared in the spectra of Ag-SPNTs film, confirming the successful loading of Ag nanoparticles. For both films with dust, there is a high level of Si, Al, and O because the main components of fly ash in the atmosphere consisted of SiO₂ and Al₂O₃.^{33,34} Other nonvolatile elements in fly ash including Fe, Na, Mg, K, and Ca^{33,35} have also been found in the captured particles for both SPNTs and Ag-SPNTs films. The elementary component of the captured dust has no significant difference between SPNTs and Ag-SPNTs films because of the same source of the captured dust. It is reported that Si, Al, Ca, and Fe are the major bounded elements of diesel engine emissions.^{36,37} Hence, the element analysis result of the captured dust was consistent with the captured environment, that is the roadside of the main street with heavy traffic flow.

Filtration Performance of the Fiber Films

The filter efficiency, pressure drop, and QF are the main factors to evaluate the filter performance.²³ Fiber films with an effective filtration area of 176.7 cm² (a diameter of 15 cm) were loaded on a TSI-automated filter tester for measurement at room temperature. DOP aerosol with a particle size of 0.33 μm and a face velocity of 5.5 cm/s were used in the test. The results were demonstrated in Table 1. It was found that SPNTs fiber film had a filter efficiency of 61.6% and a pressure drop of 44 Pa. As expected, the Ag-SPNTs-2 fiber film showed a higher filter efficiency (84.4%) than that of SPNTs fiber film. The pressure drop of Ag-SPNTs-2 fiber film (60 Pa) increased slightly in comparison with that of SPNTs fiber film, possibly due to the accumulation of captured particles on the film surface, as demonstrated in the particulate matter capture test (Fig. 7). The QF values of the SPNTs and Ag-SPNTs-2 fiber film were 0.022 and 0.031 Pa⁻¹, respectively. Since a filter with a better filtration performance should have higher efficiency and QF value,²³ the results suggested that the Ag-SPNTs-2 fiber film was better for filtration than SPNTs, which was in accordance with our proposal as well as the result of interaction forces and particulate matter capture test.

CONCLUSIONS

Nanoparticle-enhanced tubular nanofibers with a diameter of approximately 80-150 nm were prepared by template-free cationic polymerization followed by surface modification.

Ag nanoparticles with high surface energy were used to modify the nanofiber surface to improve the interaction force between fibers and particles for active capture of particulate matters. *f-d* curve measured by AFM showed that the interaction force increased when the AFM probe was approaching the nanofiber surface and decreased when retracting. Increasing the content of Ag nanoparticles in the nanofibers resulted in the enhanced interaction force between the nanofiber surface and particles and thus increased the adhesion energy between the particulates and filter fibers. Particulate capture and filter performance tests demonstrated that the Ag-SPNTs fiber films facilitated the capture of both microparticles and nanoparticles and improved the filter efficiency of the fiber films, showing better filtration performance than that of non-particle fibers.

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