



Preparation of Photocatalytic TiO₂-Polyacrylonitrile Nanofibers for Filtration of Airborne Microorganisms

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Abstract

Background: We aimed to investigate the efficiency of neat polyacrylonitrile (PAN) nanofibers and photocatalytic PAN/TiO₂ nanofibers for removal of airborne microorganisms.

Methods: Nanofibers were fabricated from 16 wt% of PAN dissolved in dimethyl formamide through the electrospinning technique. The efficiency of media for removal of *Staphylococcus epidermidis* and *Bacillus subtilis* was investigated at different conditions such as face velocity, relative humidity, air temperature and UVC radiation intensity. as face velocity (0.1 and 0.3 m/s), relative humidity (35±5% and 60±5%), air temperature (22±3 °C and 30±3 °C) and the UVC radiation intensity (dark, 1±0.09 mW/cm² and 1.8±0.07 mW/cm²) using air sampling from upstream and downstream of media by cascade impactor containing blood agar culture medium.

Results: The mean diameter of electrospun fibers and coefficient of variation were 194 nm and 15%, respectively. The amount of immobilized TiO₂ on the filter was 620±6.56 mg/m². Photocatalytic nanofiber filter media presented the best performance for removal of airborne *B. subtilis* at 60±5% relative humidity, 0.1 m/s face velocity, air temperature 22 °C, and 1.8 ± 0.07 mW/cm² UVC radiation.

Conclusion: The filtration efficiency of photocatalytic media was significantly higher than neat ones. Lower efficiency of media was found in the higher air velocity for all bioaerosols. High UVC radiation intensity increased filtration efficiency. Moreover, the increase in air temperature and relative humidity (except for TiO₂-coated media under UVC radiation) did not significantly affect the filtration efficiency of all media.

Keywords: Nanofiber; Electrospinning; Air filtration; Airborne microorganism; Photocatalytic

Introduction

The WHO has declared that air pollution is the sixth major cause of death globally (1). Indoor air pollution is grouped as one of the top five environmental risks (2) so that several studies showed that in indoors, the level of pollutants is higher

than outdoors (3). Additionally, people spend more than 80% of their time indoors, which inhalation risk of pollutants from these places is higher than outdoors (4).



Bioaerosols are particles of biological origin suspended in the air. They play significant role in indoor air pollution especially in healthcare settings (5). An anthrax attack on Sep 18, 2001, and the outbreak of airborne pathogens such as H1N1 Influenza and severe acute respiratory syndrome (SARS) in 2003 increased concerns about bioterrorism and have attracted public attention to bioaerosols and their purification methods (6,7). Therefore, it is important to reduce bioaerosol concentrations in contaminated environments to ensure the health of the workers and the public.

Filtration is a cost-benefit, efficient and the most common method to remove aerosols and improve indoor air quality (8, 9). In current years, polymeric nanofibers have gained vast attention in commercial air filtration applications due to their very large specific area, very small pore size, and high porosity, shown to improve the filtration efficiency of aerosol (10,11).

Different techniques exist for nanofibers production, but electrospinning is a simple and versatile technique producing nanofibers in different forms (12,13). Electrospun polymer nanofibers with a remarkably lower basis weight could usually gain a collection efficiency as same as the conventional HEPA (high-efficiency particulate air) filters (14-16). In this regard, polyacrylonitrile (PAN) nanofibers are easy to spin and have been widely used for filtration applications because of their excellent mechanical properties, well thermal stability, and chemical resistivity (17,18).

Studies on photocatalytic materials and their ability to remove organic pollutants have shown that titanium dioxide (TiO₂) is one of the most important compounds used for this purpose (19-21). TiO₂ is highly oxidizing and capable of destroying the outer membrane of bacteria including phospholipid, protein, and lipophosphosaccharide and ultimately causing bacterial destruction (22,23).

Few studies have been conducted on the application of photocatalytic electrospun nanofiber webs containing TiO₂ in the filtration efficiency of airborne microorganisms and the effects of face velocity and UV irradiation intensity on its filtra-

tion performance (24). So the present study aimed to assess neat PAN nanofibers and photocatalytic PAN/TiO₂ nanofibers and also to assess the effects of face velocity, relative humidity, air temperature and the UVC irradiation intensity on the filtration efficiency.

Materials and Methods

Fabrication of PAN nanofibers

Polyacrylonitrile (PAN) polymer (Mw: 80,000) was provided from Polyacryle Co. (Isfahan, Iran) and 99% N-N, dimethyl formamide (DMF) solvent was purchased from Merck Co. (Germany) and Polypropylene spun bond as a substrate (basis weight 16.5 g/m² and thickness 100 μm) was obtained from Baftineh Co. A 16 wt % concentration of PAN solution was prepared in DMF solvent through magnetic stirring for 12-24 h at room temperature to reach enough homogenization. Nanofibers were fabricated by electrospinning instrument (Fanavaran Nano-Meghyas ES2000, Iran) according to the optimized values (8) as follow: applied voltages: 20 kV, nozzle-collector distance: 10 cm, temperature: 25 °C, flow rate: 0.9 ml/h, needle diameter: 0.84 mm (gauge 18) and speed of rotation of collector: 700 rpm. In the present study, nanofibers were spun on the nonwoven polypropylene substrate according to above optimum conditions for 4 h.

Preparation of TiO₂-coated nanofibers

A 20×20 cm nanofiber media dip-coated in a 0.8% TiO₂ (Nanopowder, Rutile: Anatase/ 85:15, 99.9%, 20 nm, Degussa-P 25, Germany) solution. They were later baked in an oven at 120 °C for 1 h and then put into a frame. The TiO₂ loading was determined from difference weights of the nanofiber media before and after coating.

Preparation of Airborne microorganism

A gram-positive cocci vegetative *Staphylococcus epidermidis* that inhabits the respiratory tract and the rod-shaped and a gram-positive *Bacillus subtilis* endospore were used in this study. *S. epidermidis* and *B. subtilis* were provided from the Depart-

ment of Pathobiology, Tehran University of Medical Sciences. Each of the microorganisms was inoculated at 37 °C on blood agar plates for 24 hours. Both microorganisms were suspended in distilled water to the required microorganism concentration of 10⁷ CFU/ml.

Investigation of removal efficiency

A schematic design of the filter test rig is shown in Fig. 1 (25). Nanofiber media was inserted into the filter holder of the test chamber. Microorganism suspensions with an initial concentration of 10⁷ CFU/ml were injected into the system upstream of media using a nebulizer at a pressure of 12 psi.

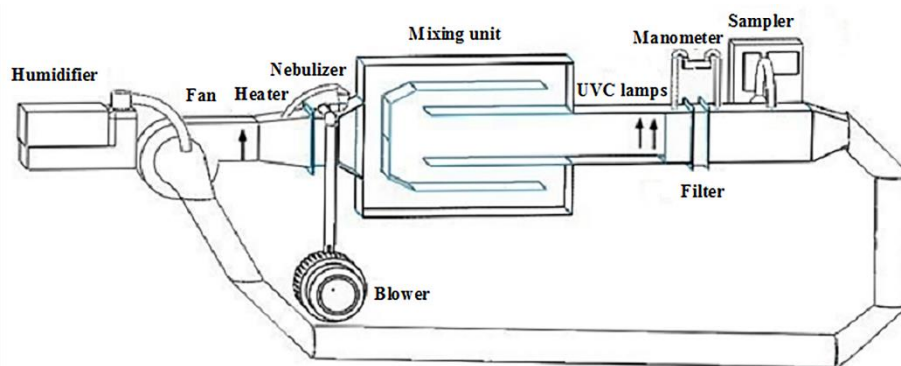


Fig. 1: Schematic of the closed-loop chamber

The air was circulated in the chamber at a velocity of 0.1 and 0.3 m/s by a blower, measured with a hot wire anemometer. Relative humidity (RH) was controlled at 35±5% and 60±5% by a humidity control device (JDR 800 device, Eskandari Industrial group, Iran). Air temperature also was controlled at 22±3 °C and 30±3 °C. At 5 cm before the filter, four fluorescent UVC lamps were inserted to irradiate at a UVC intensity of 1± 0.09 mW/cm² onto the media. For another radiation intensity, six 36-W UVC lamps (6WT, Japan) were inserted, and the radiation was 1.8 ± 0.07 mW/cm² measured on the filter surface.

To study the removal efficiency, each of the microorganisms was nebulized into the system in a separate run for 30 min. The microorganism was sampled by two single stages Anderson impactors equipped with blood agar plates upstream and downstream of media every 10 min. The flow rate was set at 28.3 l/min and the time of sampling was 1 min. During each run (30 min), the injection of microorganisms into the test duct was continuous. After sampling, all sampled plates were incubated at 37 °C for 18-24 h. After

that, colonies were counted and then filtration efficiency was obtained by Equation 1.

$$\text{Filtration Efficiency} = \frac{\text{the average of colonies before the filter} - \text{the average of colonies after the filter}}{\text{the average of colonies before the filter}}$$

(Equation 1)

At the end of each run, the chamber was sterilized using UVC for several hours to remove the remaining microorganisms. Moreover, an inside surface swab was performed to verify sterilization. Five samples of the studied filter media were prepared and tested and then mean percentage efficiency was calculated (26).

Statistical Analysis

The data were analyzed using SPSS (ver. 21, Chicago, IL, USA). For quantitative variables, means and standard deviations were calculated. Quantitative variables were compared by the Independent Sample T-test and One-way ANOVA. The level was set at 0.05 for all statistical analysis.

Results

After weighing the coated filter, the amount of immobilized TiO₂ on the media was 620±6.56 mg/m². Figure 2 shows the morphology of nanofiber filter media with and without TiO₂ loading. The mean diameter of electrospun fibers and the

coefficient of variation was 194 nm and 15%, respectively. The initial mean pressure drop of the neat and treated filter media was 120±10.22 pa and 150 ± 13.43 pa, respectively at the 0.1 m/s face velocity.

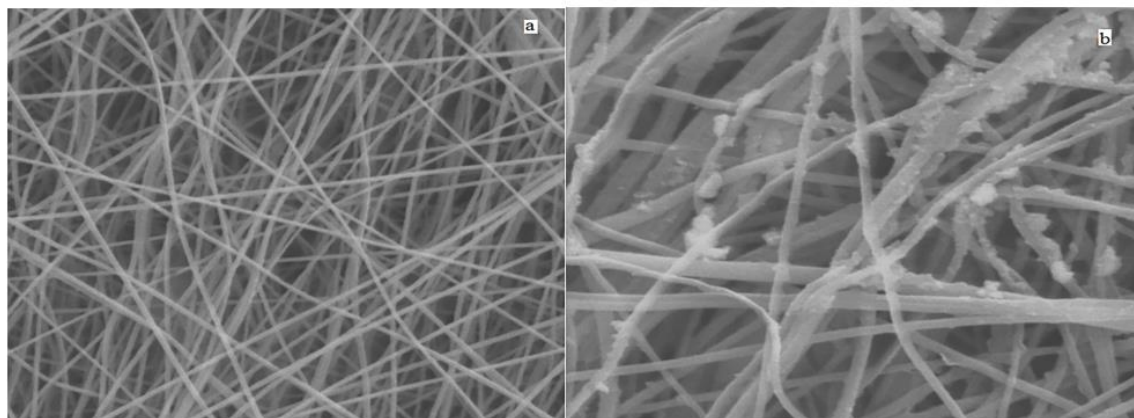


Fig. 2: SEM images of nanofiber filter: a) Neat filter media and b) TiO₂-treated filter media (5,000× magnifications)

Filtration efficiencies of neat and TiO₂-treated filter media for removal of the airborne microorganisms at different air temperatures (22 °C and 30 °C) and relative humidities (35% and 60%) are presented in Tables 1-4. Generally, in a fixed face velocity, a significant difference was found be-

tween the dark and UVC radiation in case of filtration of *S. epidermidis* and *B. subtilis* for both neat and treated filter media ($P < 0.01$), so that the mean efficiency in removal of the bioaerosols under radiation 1.8 mw/cm² was significantly higher than ones ($P < 0.01$).

Table 1: Mean filtration efficiencies of neat and TiO₂-treated filter media at air temperature of 22 ± 3°C and relative humidity of 35 ± 5%

Filter Media	Bacteria	Face velocity (m/s)	Dark	Radiation (mW/cm ²)		P-value**
				1	1.8	
Neat Nanofiber	<i>S. epidermidis</i>	0.1	83.9 ± 0.02	94.8 ± 0.06	97 ± 0.09	<0.001
		0.3	83.3 ± 0.04	94.6 ± 0.58	96.2 ± 0.08	0.001
	P-value*		0.001	0.010	0.001	-
	<i>B. subtilis</i>	0.1	93.8 ± 0.14	95.7 ± 0.16	97.1 ± 0.10	<0.001
0.3		90.6 ± 0.23	94.9 ± 0.08	96.6 ± 0.07	<0.001	
P-value*		<0.001	0.004	0.002	-	
TiO ₂ -treated Nanofiber	<i>S. epidermidis</i>	0.1	84 ± 0.05	96.9 ± 0.04	99 ± 0.03	<0.001
		0.3	83.3 ± 0.09	95.5 ± 0.12	98.5 ± 0.02	<0.001
	P-value*		0.001	0.001	0.002	-
	<i>B. subtilis</i>	0.1	94 ± 0.31	98.3 ± 0.12	99.6 ± 0.08	<0.001
0.3		91 ± 0.07	97.7 ± 0.19	99.3 ± 0.08	<0.001	
P-value*		<0.001	0.001	0.045	-	

*Comparison of efficiency between two face velocities

**Comparison of efficiency between three radiation conditions

Table 2: Mean filtration efficiencies of neat and TiO₂-treated filter media at air temperature of 22 ± 3°C and relative humidity of 60 ± 5%.

Filter Media	Bacteria	Face velocity (m/s)	Dark	Radiation (mW/cm ²)		P-value**
				1	1.8	
Neat Nano-fiber	<i>S. epidermidis</i>	0.1	83.7 ± 0.07	94.7 ± 0.05	97.2 ± 0.03	<0.001
		0.3	83.2 ± 0.04	94.2 ± 0.06	96.3 ± 0.09	<0.001
	P-value*		0.001	0.001	0.003	-
	<i>B. subtilis</i>	0.1	93.5 ± 0.24	95.5 ± 0.22	97.4 ± 0.18	<0.001
0.3		90.5 ± 0.05	94.7 ± 0.12	96.3 ± 0.13	<0.001	
P-value*		0.002	0.012	0.010	-	
TiO ₂ -treated Nanofiber	<i>S. epidermidis</i>	0.1	83.9 ± 0.07	97.1 ± 0.11	99.1 ± 0.08	<0.001
		0.3	83.2 ± 0.09	98.4 ± 0.07	98.3 ± 0.06	<0.001
	P-value*		0.001	<0.001	<0.001	-
	<i>B. subtilis</i>	0.1	93.9 ± 0.24	98.7 ± 0.07	99.7 ± 0.11	0.001
0.3		91.9 ± 0.17	98.4 ± 0.07	98.6 ± 0.13	0.001	
P-value*		<0.001	0.002	0.003	-	

Table 3: Mean filtration efficiencies of neat and TiO₂-treated filter media at air temperature of 30 ± 3°C and relative humidity of 35 ± 5%

Filter Media	Bacteria	Face velocity (m/s)	Dark	Radiation (mW/cm ²)		P-value**
				1	1.8	
Neat Nano-fiber	<i>S. epidermidis</i>	0.1	83.9 ± 0.11	94.8 ± 0.04	96.9 ± 0.13	<0.001
		0.3	83.3 ± 0.03	94.3 ± 0.08	96.2 ± 0.10	<0.001
	P-value*		0.007	0.005	0.004	-
	<i>B. subtilis</i>	0.1	93.9 ± 0.05	95.7 ± 0.10	97 ± 0.10	<0.001
0.3		90.6 ± 0.04	94.0 ± 0.08	96.1 ± 0.15	<0.001	
P-value*		0.001	0.001	0.013	-	
TiO ₂ -treated Nanofiber	<i>S. epidermidis</i>	0.1	82.2 ± 0.09	96.8 ± 0.05	98.9 ± 0.08	<0.001
		0.3	83.3 ± 0.12	95.6 ± 0.07	98.2 ± 0.06	<0.001
	P-value*		0.008	0.001	0.001	-
	<i>B. subtilis</i>	0.1	93.9 ± 0.37	98.4 ± 0.07	99 ± 0.11	0.001
0.3		91.9 ± 0.11	98.4 ± 0.07	98.6 ± 0.14	0.001	
P-value*		0.002	0.006	0.022	-	

Table 4: Mean filtration efficiencies of neat and TiO₂-treated filter media at air temperature of 30 ± 3°C and relative humidity of 65 ± 5%

Filter Media	Bacteria	Face velocity (m/s)	Dark	Radiation (mW/cm ²)		P-value**
				1	1.8	
Neat Nano-fiber	<i>S. epidermidis</i>	0.1	83.8 ± 0.04	94.7 ± 0.09	95.5 ± 0.06	<0.001
		0.3	93.2 ± 0.14	94.3 ± 0.03	94.9 ± 0.11	<0.001
	P-value*		0.013	0.006	0.004	-
	<i>B. subtilis</i>	0.1	93.6 ± 0.08	95.5 ± 0.03	96.2 ± 0.08	0.003
0.3		91.5 ± 0.17	94.8 ± 0.16	95.7 ± 0.12	<0.001	
P-value*		<0.001	0.016	0.009	-	
TiO ₂ -treated Nanofiber	<i>S. epidermidis</i>	0.1	83.9 ± 0.11	96.9 ± 0.03	98.9 ± 0.02	<0.001
		0.3	83.3 ± 0.05	95.7 ± 0.10	98.4 ± 0.07	<0.001
	P-value*		0.004	<0.001	0.005	-
	<i>B. subtilis</i>	0.1	93.9 ± 0.30	98.5 ± 0.08	99.3 ± 0.10	<0.001
0.3		90.9 ± 0.03	97.9 ± 0.17	99 ± 0.09	0.001	
P-value*		0.003	0.011	0.010	-	

The filtration efficiency of the neat and treated filter media for both microorganisms has been significantly decreased with an increase in the face velocity at different UVC radiation conditions ($P < 0.01$). There was no significant difference in filtration efficiency of both microorganisms between the neat and treated filter media under dark conditions ($P = 0.20$), however, this difference was statistically significant ($P < 0.05$) under UVC radiation, so that the mean efficiency of treated media in the removal of the bioaerosols was higher than ones ($P < 0.01$). Totally, under different test conditions, the nanofiber filter media has higher performance for removal of *B. subtilis* compared to *S. epidermidis* ($P < 0.01$). For the relative humidity effect, there was no significant difference between low and high relative humidity in coated and uncoated filters efficiency under dark conditions but under radiation conditions, the filtration efficiency of TiO₂-coated media was significantly increased with an increase in relative humidity ($P < 0.05$). Moreover, relative humidity did not affect the filtration efficiency of uncoated media at different UVC radiation conditions. Statistical analysis has been shown that the air temperature effect on the filtration efficiency of *S. epidermidis* and *B. subtilis* was not significant for both neat and treated filter media under different conditions of radiation.

Discussion

In this study, filtration efficiency of neat nanofiber filter media was achieved 83.9% and 93.8% for *S. epidermidis* and *B. subtilis* at dark condition, respectively and was obtained 94.8% and 95.7% at 1 ± 0.09 mW/cm² UVC radiation intensity for *S. epidermidis* and *B. subtilis*, respectively. There was a significant difference between dark and 1 ± 0.09 mW/cm² UVC irradiation intensity in filtration efficiency of uncoated filter that showed UVC irradiation increase filtration efficiency that was similar to the results of another study (25, 27) that reported use of UVC on TiO₂-coated HEPA filters can reduce the percent penetration

of bioaerosols within them. In the current study, the filtration efficiency of the TiO₂-treated filter was obtained 84% and 94% at the dark condition for *S. epidermidis* and *B. subtilis*, respectively. Moreover, these values were 96.9% and 98.3% at 1 ± 0.09 mW/cm² UVC irradiation intensity for *S. epidermidis* and *B. subtilis*, respectively. Using TiO₂ and UVC radiation (photocatalysis) have a positive effect on the filtration efficiency of *S. epidermidis* ($P < 0.001$) and *B. subtilis* ($P < 0.001$). This is in accordance with other findings reported (28) that used UVA radiation on pleated HEPA filter coated with TiO₂. In dark condition, a significant difference did not exist between the filtration efficiency of the neat and treated nanofiber filter media for *S. epidermidis* and *B. subtilis*. These findings are in accordance with Pal et al (2) that it could be due to the insensitivity of microorganisms to TiO₂ in the absence of UV light. A significant difference in filtration efficiency occurred between neat nanofibers and photocatalytic nanofibers in UVC radiation condition. The photocatalytic oxidation (PCO) was more successful for disinfecting *S. epidermidis* and *B. subtilis* than UVC photolysis alone. This result is in agreement with Donlup et al. (29), it can be because of the production of more oxidizing radical species in the photocatalytic oxidation reaction than photolysis reaction.

In the case of face velocity effect, the results of the current study showed that an increase in face velocity from 0.1 m/s to 0.3 m/s caused reduced filtration efficiency of neat and TiO₂-coated nanofibers for *S. epidermidis* and *B. subtilis* at different UVC radiation condition. Moreover, some studies, had similar results to our study. This can be due to the decreased retention time of the microorganisms in the filter media and its exposure to UVC radiation in high air velocity (30,31).

In terms of UV-C radiation, the filtration efficiency of the TiO₂-coated filter was increased for both microorganisms in higher radiation intensity. This also agrees with another study (25) that observed a significant decrease in the penetration rate of *S. epidermidis* and *B. subtilis* in photocatalytic HEPA filter when the UVC intensity was in-

creased. Although the findings of some studies were in contrast with our results (2, 28). An increase in UV radiation intensity leads to a competition between UV and photocatalysis for dissolved O_2 , which is an essential reactant for generating strong oxidant species and also superoxide radicals generated from UV at high intensity which can combine with hydroxyl radicals or holes at the TiO_2 surface, decreasing the microorganism inactivation effectiveness. This difference can be due to differences in the type of radiation source and radiation intensity used in present work with the above-mentioned studies.

In the case of relative humidity effect, the filtration efficiency of coated and uncoated nanofiber filters did not change significantly at dark conditions with increase in relative humidity. Moreover, under UVC radiation conditions, the filtration efficiency of uncoated nanofiber filter was not significantly different between RH 35% and 60%. Relative humidity did not affect photolysis reaction. This result agrees with another study that showed photolysis resulted in similar inactivation rates of *E. coli* K-12 over a range of RH (32). Although the filtration efficiency of coated nanofiber filter was significantly increased for both microorganisms in UVC irradiation conditions at higher relative humidity. This finding is in contrast with other studies that indicated in high relative humidity, the efficiency of photocatalytic filter media was significantly decreased (28, 30). The finding of their studies can be explained by Li et al (33), although the existence of water vapor improves a promotion of hydroxyl radical formations, the radical formations may not increase with the increase in water vapor and even the occupation of the adsorption site on the TiO_2 surface can decrease them and as a consequence, high humidity decreases the efficiency. Moreover, complete inactivation of *Serratia marcescens* was achieved at 50% RH in about 13 h, but 10% of the microorganisms remained alive at higher humidity (85%) (34). High humidity may lead to reactivation of organisms or most of the TiO_2 sites may occupy by water; hence, less available sites remained for microorganisms. In the current study with increasing humidity, the

filtration efficiency of TiO_2 -coated media was increased which agrees with Pham et al. (10) that reported among three relative humidity of $40\pm 5\%$, $60\pm 5\%$ and $80\pm 5\%$, which the relative humidity of $60\pm 5\%$ had maximum disinfection efficiency. The difference between the finding of this study and other studies (28,30) may be due to difference in type of UV radiation source and higher relative humidity levels used in their studies.

In terms of air temperature, the filtration efficiency of *S. epidermidis* and *B. subtilis* did not change significantly for both neat and treated nanofibers under dark and UVC radiation conditions when the air temperature was increased. This finding is in agreement with that stated the increasing temperature in the duct did not play a significant role in the bacteria inactivation (6).

Conclusion

The TiO_2 -coated nanofiber filter media was shown higher filtration efficiency than uncoated nanofiber filter for removal of *S. epidermidis* and *B. subtilis* bioaerosol under UVC radiation. All produced media had higher filtration efficiency at 0.1 m/s air velocity compared to 0.3 m/s in the different test conditions. The Efficiency of photocatalytic nanofibers was prompted by an increase in UVC radiation intensity from 1 mW/cm^2 to 1.8 mW/cm^2 . Moreover, an increase in the air temperature from 22 °C to 30 °C and relative humidity from 35% to 60% (except for TiO_2 -coated media under UVC radiation) did not significantly affect the filtration efficiency of nanofibers. Finally, under test conditions, it can be concluded produced photocatalytic nanofiber filter media present the best performance for removal of airborne *B. subtilis* at $60\pm 5\%$ relative humidity, 0.1 m/s face velocity, air temperature 22 °C, and $1.8 \pm 0.07 mW/cm^2$ UVC radiation.

Journalism Ethics considerations

Ethical issues (Including plagiarism, informed consent, misconduct, data fabrication and/or fal-

sification, double publication and/or submission, redundancy, etc.) have been completely observed by the authors.

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Conflict of interest

The authors declare that there is no conflict of interest.

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