CHARACTERIZATION AND MICRO AND NANOPARTICLES FILTRATION EVALUATION OF PAN NANOFIBERS PRODUCED BY CENTRIFUGAL SPINNING

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ABSTRACT

Centrifugal spinning has been used in the production of fibers for the most varied application areas. This paper aims to evaluate the filtration performance of substrate, and polyacrylonitrile (PAN) nanofibers covered at the same substrate, produced by Forcespinning (centrifugal spinning equipment). For this, the characteristics of the nanofiber layer and collection efficiency of micro and nanoparticles were investigated. Sodium chloride was used to generate nanoparticles, and hydrated lime was used as micro particles. Filtration experiments were performed with a superficial gas velocity of 4.8 cm/s. The fiber layer has a single layer with large fiber diameter and pore size dispersion. The results showed that the substrate was more permeable and presented the lower pressure drop than the covered substrate. For nanoparticle collection efficiency, the substrate had an increase of around 20%, due to the addition of PAN nanofibers. For micro particle collection efficiency, at the beginning of the test and after 30 minutes of filtration, the substrate increased collection efficiency from 50.5 to 69.6%, while the covered substrate increased from 71.0 to 97.2%.

KEYWORDS: centrifugal spinning, filtration, nanofiber.

CARACTERIZAÇÃO E AVALIAÇÃO DA FILTRAÇÃO DE MICRO E NANO PARTÍCULAS DE NANOFIBRAS DE POLIACRILONITRÍLO PRODUZIDAS POR FIAÇÃO CENTRIFUGA

RESUMO

A fiação centrífuga tem sido utilizada na produção de fibras para as mais variadas áreas de aplicação. Este trabalho tem como objetivo avaliar o desempenho de filtração do substrato e do mesmo substrato coberto por fibras de poliacrilonitrilo (PAN), produzidas por *Forcespinning* (equipamento de fiação centrífuga). Para isso as características da camada de nanofibras e a eficiência de coleta de micro e nanopartículas foram investigadas. Utilizou-se cloreto de sódio para gerar nanopartículas e cal hidratada como micropartículas. Os testes de filtração foram realizados com uma velocidade superficial do gás de 4,8 cm/s. A camada de fibra possui uma única camada com grande dispersão de diâmetro das fibras e de

tamanho dos poros. Os resultados mostraram que o substrato foi mais permeável e apresentou menor queda de pressão que o substrato coberto. Para eficiência de coleta de nanopartículas, o substrato teve um aumento de cerca de 20% devido à adição de nanofibras de PAN. Avaliando a eficiência de coleta de micropartículas, no início do teste e após 30 minutos de filtração, o substrato aumentou a eficiência de coleta de 50,5 para 69,6%, enquanto o substrato coberto aumentou de 71,0 para 97,2%.

PALAVRAS-CHAVE: fiação centrífuga, filtração, nanofibra.

INTRODUCTION

Air pollution poses a significant environmental risk to human health. Particles when inhaled increases the risk of respiratory and cardiovascular diseases, diabetes, lung cancer, which can lead to premature death (HAYES et al., 2019; KRAUSKOPF et al., 2019). Recent studies linked air pollution with Alzheimer's disease (WALTON, 2018), dementia (LEE et al., 2019), cerebral ischemic attack, epilepsy, and headaches (RADMANESH et al., 2019).

Filter media is commonly used to control and removal particles from the air. The spun fiber has been researched for filtration application with excellent performance (FENG et al., 2019; ZHU et al., 2016). Spun fiber has a high surface-volume ratio, small pore size, and small fiber diameter that promote the increase of the collection efficiency of micro and nanoparticles. Fundamental parameters for filtration are collection efficiency and pressure drop. Collection efficiency is the number of particles captured by the filter media from the gas stream and pressure drop measures the resistance of air passage. A good filter media should have the maximum collection efficiency with the lowest pressure drop (HINDS, 1999).

Centrifugal spinning has been a promising method of produces polymeric fibers. Centrifugal spinning increases the choice of the materials, productivity, and reduces production cost (PADRON et al., 2013; SARKAR et al., 2010). In recent papers, these fibers were used for battery (FLORES et al., 2018; KIM, 2019), tissue engineering (LUKÁŠOVÁ et al., 2019; MAMIDI et al., 2019a), adsorption (KUMMER et al., 2018; ROSTAMIAN; FIROUZZARE; IRANDOUST, 2019), colorimetric sensor for food (VALDEZ et al., 2019), fibrous color dosimeter (KINASHI et al., 2018), pharmaceutical application (AKIA et al., 2019; MAMIDI et al., 2019b).

Given the above background, this work aims to evaluate the filtration performance of the substrate (without cover), and PAN nanofibers covered at the same substrate produced by Forcespinning (centrifugal spinning equipment). For this, the characteristics of the fiber layer and collection efficiency of micro and nanoparticles were investigated.

MATERIAL AND METHODS

Fiber Preparation

Forcespinning (FS) employs the centrifugal spinning to produce the nanofibers. This equipment consists of a spinneret, needles, metal bar, and vacuum collection system, which can be seen in FIGURE 1. The substrate (filter medium with a weight of 75 g/m²) was fixed on the vacuum collection system at 5 cm from the lateral edges and 7.5 cm from the bottom edge. For fiber formation, the solution of 12 wt. % polyacrylonitrile (PAN, Mw: 150,000 g/mol, Sigma Aldrich) was dissolved in N,N-dimethylformamide (DMF, anhydrous, 99.8%, Sigma Aldrich), by heating (60 °C) and stirring at 3 hours. In the FS, the nanofiber was produced using 26 gauge half-inch needles, 10 cm needle-collector distance, 4000 rpm of rotation speed, 60 s of

collection time, and 100% VCS power.

FIGURE 1 - Schematic illustration of the arrangement of FS system.

Characterization of Fiber Layer

The nanofiber layer was characterized by scanning electron microscopy (SEM) using FEI Inspect S50. SEM images were used to determine the fiber diameter, the superficial porosity, the density of intersections, and pore size. The parameters were performed on DiameterJ plug-in of the ImageJ software package.

Filtration Test

The experimental unit used to perform the nanoparticle collection efficiency, permeability, and pressure drops of the substrate (without cover), and the substrate covered with PAN fiber can be seen in FIGURE 2. The apparatus consisted of aerosol generator (Model 3079, TSI), filtered air supply (Model 3074B, TSI), diffusion dryer (Model 3062, TSI), krypton-85 charge neutralizer (Model 3054, TSI), filter media apparatus, americium-241 charge neutralizer, flowmeter (Gilmont), and scanning mobility particle sizer (SMPS) composed of electrostatic classifier (Model 3080, TSI), differential mobility analyzer, and particle counter (Model 3776, TSI). The nanoparticles were generated by aerosol generator from 0.1 g/L sodium chloride (NaCl, 99.7%, J. T. Baker) solution. The particle concentrations before and after the filter media were determined using the SMPS, 30 minutes after the start of the test. The filtration area was 5.3 cm² and the superficial gas velocity of 4.8 cm/s.

FIGURE 2 - Flow chart of the system used to perform nanoparticle collection efficiency, permeability, and pressured drop of the filter media.

FIGURE 3 shows the experimental unit used to perform the micro particle collection efficiency. The experimental unit was composed of particle feeder (model 3433, TSI), diluter (model 3302A, TSI), aerodynamic particle sizer spectrometer (model 3320, TSI), flowmeter (Gilmont), and pump. The micro particles were hydrated lime powder, the particle density is 2.7742 g/cm^3 . The particle concentrations before and after the filter media were determined in APS, at the beginning and after 30 minutes of the start of the test. The filtration area was 17.6 cm^2 and the superficial gas velocity of 4.8 cm/s .

FIGURE 3 - Flow chart of the system used to perform micro particle collection efficiency of the filter media.

The collection efficiency was determined using Equation 1:

$$E = \frac{C_0 - C_1}{C_0} \quad (1)$$

where E is the collection efficiency, C_0 is the particle concentration before, and C_1 is the particle concentration after the filter media.

The pressure drop was measured using a digital manometer (Model 9555P, TSI) with a superficial gas velocity of 4.8 cm/s .

The permeability of each filter media was obtained from the equation for the linear plot of superficial gas velocity (ranged from 0.3 to 6.4 cm/s) against P/L , calculated using Darcy's law on Equation 2:

$$\frac{P}{L} = \frac{\mu v_L}{k} \quad (2)$$

where P is the pressure drop, L is the thickness of the filter media, μ is the fluid viscosity, v_L is the superficial gas velocity, and k is the permeability constant of the filter media.

RESULTS AND DISCUSSION

Characterization of fiber layer

SEM images were used on ImageJ software to measure the fiber diameter, superficial porosity, density of intersections, and pore size. PAN nanofiber layer was $1040 \pm 400 \text{ nm}$ of the mean fiber diameter, $86 \pm 5\%$ of superficial porosity, $0.01 \pm 0.01 \text{ int}/\mu\text{m}^2$ of the density of intersection, $7.0 \pm 2.5 \mu\text{m}$ of pore size. FIGURE 4 shows the SEM image of PAN nanofibers with a) $5000 \times$ and b) $200 \times$ of magnification, and c) fiber diameter distribution. The most frequently diameter were between 700 and 1000 nm . These data report a single fiber layer with large fiber diameter dispersion and pore size.

FIGURE 4 – a) SEM image of PAN nanofiber layer with 5000 x and b) 200 x of magnification and c) fiber diameter distribution.

Evaluation of filtration test

The permeability shows the ease or difficulty of air passing through the filter medium. FIGURE 5 shows P/L versus superficial gas velocity of the substrate and covered substrate. The determination coefficient (R^2) of the substrate and covered substrate were 0.989 and 0.999, respectively. It demonstrated that the curves are well adjusted. The substrate with PAN nanofibers presented a higher pressure drop than the substrate. Thus the linear regression of covered substrate had a high regression coefficient and the steepest curve. The substrate and covered substrate with PAN nanofiber permeability were $3.3 \pm 0.0 \cdot 10^{-5} \text{ m}^2$ and $1.5 \pm 0.6 \cdot 10^{-5} \text{ m}^2$, respectively. These values indicate that the substrate is more permeable than the covered substrate.

FIGURE 5 – P/L versus superficial gas velocity of substrate and substrate with PAN nanofibers.

Collection efficiency performed on substrate and PAN nanofibers covered substrate for nanoparticles and micro particles, can be seen in FIGURES 6 a) and 7 a), respectively. For the collection efficiency of nanoparticles, the particles were generated from 0.1 g/l of NaCl solution by an aerosol generator in the experimental unit of FIGURE 1. The nanoparticle size ranged from 5.9 to 224.7 nm, and the largest concentration of particle was from 25 to 50 nm, as can be seen in Figure 6 b). The concentration of particles before and after filter media was measured 30 minutes after the beginning of the experiment. The substrate had a collection efficiency of 11.5%, while the substrate with a nanofiber layer had 31.3%, resulting in a difference of 19.8% (TABLE 1). This effect occurs because the addition of the fiber layer promotes increased particle capture. PAN nanofibers have a fiber mean diameter of 1040 nm, which fibers of this size are on the slip flow regime. On the slip flow regime, the gas fluid slip over the fiber surface, promoting greater interaction between the particles and the fiber, which increases the collection efficiency (LI et al., 2014). Particles smaller than 100 nm have an increase in collection efficiency due to the predominance of diffusion collection mechanisms (CHUANFANG, 2012). This behavior can be noticed in both curves, FIGURE 6 a). Similar results were obtained in the study by BORTOLASSI et al. (2019).

FIGURE 6 – a) Collection efficiency performed on substrate and PAN nanofibers covered substrate for nanoparticles, and b) distribution of nanoparticles size.

The collection efficiency was also measured for hydrated lime micro particles, at the beginning ($t=0$) and after 30 minutes of the start of the test ($t=30$), as shown in FIGURE 7 a). The micro particles ranged from 1 to 19.8 μm , with the highest concentrations between 1 and 5 μm , as can be seen in FIGURE 7 b). The particle concentrations before and after the filter media were determined by APS in the experimental unit of FIGURE 2. At $t=0$, the substrate had a collection efficiency of 50.5%, and the covered substrate had 71.0%. The collection efficiency increased because of the addition of the fiber layer, just like in the previous case. But in this case, how the particles are large diameter, the predominant collection mechanisms are interception and inertial impaction, which promotes the increase of collection efficiency with the increase of particle size (CHUANFANG, 2012). This behavior can be noticed in all curves, FIGURE 7 a).

After 30 minutes of filtration, the substrate remains in deep filtration, i.e., the powder has not accumulated sufficiently to form the cake. For the covered filter, the PAN fiber layer promoted surface filtration enabled the dust cake formation, which significantly increased particle collection. Thus, after 30 minutes of filtration, the

substrate increased collection efficiency from 50.5 to 69.6%, while the covered substrate increased from 71.0 to 97.2% (TABLE 1). For example, for particle size 1.0 μm the collection efficiency of substrate + PAN fiber at $t=0$ was 36.7% and the substrate with PAN fiber at $t=30$ min was 92.9%, showing how the dust cake filtration influenced the collection efficiency.

FIGURE 7 – a) Collection efficiency performed on substrate and substrate covered with PAN nanofibers for micro particles at beginning and 30 minutes after the start of the test, and b) distribution of micro particles size.

In terms of pressure drop, the substrate had an initial pressured drop of 0.6 Pa and the covered filter of 1.6 Pa. For nanoparticle filtration, there was no significant increase in pressure drop for both the substrate and the covered substrate. However, for micro particle filtration, the substrate increased from 0.6 to 1.2 Pa, and the covered substrate increased from 1.6 to 21.3 Pa, demonstrating that during the 30 minutes of filtration there was the formation of dust cake, that is, a larger amount of particles was retained on the surface of the covered substrate, increasing the pressure drop value (LEUNG; CHOY, 2018).

TABLE 1 – Pressure drop and collection efficiency of micro and nanoparticles on the beginning and 30 minute after the start of the test.

Filter media	Filtration time (min)	Collection efficiency (%)		Pressured drop (Pa)	
		nanoparticles	micro particles	nanoparticles	micro particles
Substrate	0	-	50.5 ± 12.7	0.6 ± 0.1	0.6 ± 0.1
Substrate + PAN fiber	0	-	71.0 ± 7.1	1.6 ± 0.6	1.6 ± 0.6
Substrate	30	11.5 ± 5.8	69.6 ± 6.7	0.6 ± 0.2	1.2 ± 0.1
Substrate + PAN fiber	30	31.3 ± 5.1	97.2 ± 2.2	2.6 ± 0.3	21.3 ± 1.0

The addition of the fiber layer promoted a significant increase in collection efficiency without a significant increase in pressure drop. Wang et al. (2008) studied filters composed of a layer of fibers on a substrate and the results show that collection efficiency and pressure drop increase with the addition of the fiber layer. For 30 minutes of micro particle filtration, there was a collection efficiency of 97.2%.

Collection efficiency increase of around 20% for nanoparticle filtration and for $t = 0$ micro particle filtration. Thus, the addition of a single PAN nanofiber layer positively affected filtration performance, mainly for micro particle evaluation. Thus, Forcespinning equipment could be a promising device for the production of nanofibers for air filtration applications.

CONCLUSIONS

This paper study the filtration performance of substrate and PAN nanofiber covered substrate produced by Forcespinning (centrifugal spinning equipment), evaluating the collection efficiency of micro and nanoparticles, and the characteristics of the fiber layer. Fiber layer had a single layer with large fiber diameter dispersion and pore size.

Evaluating filtration performance, the substrate presented more permeable than the covered substrate. The covered substrate was higher pressure drop and collection efficiency than the substrate. For nanoparticle, collection efficiency had an increase of around 20% with the addition of PAN nanofibers. For micro particle collection efficiency, at the begging of the experiment substrate had a collection efficiency of 50.5%, and the covered substrate had 71.0%. After 30 minutes of filtration, the substrate remains in deep filtration, while the PAN fiber layer promoted surface filtration enabled the cake formation, which significantly increased collection efficiency, 97.2%.

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