Polymer Aerogels for
Efficient Removal of Airborne Nanoparticles

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ABSTRACT

This study evaluates the potential of polymer aerogel monoliths in removing airborne nanoparticles. Macroporous monolithic aerogels of δ -form syndiotactic polystyrene (sPS) are synthesized for this purpose via thermoreversible gelation of a solution of sPS in a good solvent followed by supercritical drying. The air permeability and airborne nanoparticle removal efficiency are determined as function of the bulk density of aerogels. The data reveal a power-law dependence of particle removal efficiency and air permeability on bulk density. The data also reveal that efficiency greater than 99.95% can be achieved if the bulk density is kept at 0.042 g/cm³ or higher. These materials show air permeability of the order of 10⁻¹⁰ m². The gradient density aerogels produced via sequential injections of sPS solutions show improvements in filtration efficiency attributed to additional skin layers. This idea is exploited in designing gradient density aerogel filters that offer higher efficiency and higher air permeability at a much lower bulk density than the single density monoliths of the same thickness.

Keywords: Filtration; airborne nanoparticles; aerogels; macroporous materials; air permeability

INTRODUCTION

It is widely known that exposures to airborne particles can cause severe health hazards to human being including nausea, breathing difficulties, bronchitis, birth defects, serious developmental delays, weakened immune systems, and even cancers ^{1, 2}. The significance of the hazards is a function of the size of the airborne particles. For example, smaller particles can easily penetrate into the human body and reach the pulmonary alveoli ³. Fine particles of size 0.1 μm -1 μm and ultrafine particles of size < 0.1 μm are more detrimental to human health than the coarse grain particles of size > 1 μm . The health hazards are compounded by the presence of pathogens of size 20-2,000 nm residing in these particles ⁴.

Removal of small particles and pathogens by filtration can significantly improve the quality of air and reduce the health hazards. The use of high efficiency particulate absorption (HEPA) filters fabricated from fiber mats are widely used for the purpose. The filtration efficiencies of HEPA filters are high at 99.95% for removing around 0.3 μm particles as per EN 1822-1:2009 classification at comparably low pressure drops. However, published literature on evaluation of HEPA filters for removal of airborne nanoparticles of sizes 100 nm and smaller are scarce.

A wide range of industrial applications use porous media for filtration due to their large surface area and pore volume ⁵. In this context, monolithic aerogels fabricated from inorganic or organic sources present several attributes as filter media. The aerogel monoliths have low solid contents, high porosity with various pore sizes, and large surface areas ⁶⁻²⁶. For example, silica aerogels derived from alkoxy silanes offer large surface areas, up to 1,000 m^2/g , significant mesopore fractions, and porosities over 90% ⁶⁻¹⁸. The δ -form syndiotactic polystyrene (sPS) aerogels also offer macropores and porosities up to 97%; the nanocavities of crystalline strands of sPS selectively absorb small organic molecules ¹⁹⁻²⁶. Despite offering several attractive characteristics, research on filtration of nanometric airborne particles by monolithic aerogels is scarce. A handful of studies used aerogel granules or microspheres in packed beds ²⁷⁻³⁰. Some literature reports are available on the use of aerogels impregnated into HEPA filters for airborne particle

filtration ³¹, colonization of bioluminescent organisms inside a monolithic silica aerogel to detect viral particles ³², and applications of heterogeneous catalyst for removal of pollutants, such as removal of cyanides or combustion gases by monolithic silica aerogel composites containing metal oxides ³³⁻³⁵. Literature on direct investigations of filtration of airborne nanoparticles by monolithic aerogels is scarce.

This study investigated the utility of monolithic δ -form sPS aerogels for nanometric airborne particle filtration. The values of permeability and filtration efficiency are two key measured properties needed to quantify the performance of the aerogels for use as filtration media. In this context, monolithic sPS aerogels of single density and gradient density were examined.

MATERIALS AND METHODS

sPS aerogel monoliths were fabricated using a two-step process involving thermoreversible gelation and supercritical drying ¹⁹⁻²⁶. The thermoreversible gelation of sPS occurred via spinodal decomposition of the polymer-solvent binary system. sPS pellets with 98% syndiotacticity and molecular weight of 300,000 g/mol was provided by Scientific Polymer Products Inc. (New York, NY). The sPS pellets were converted into powders for easy dissolution in a solvent. Reagent grade tetrahydrofuran (THF) purchased from Fisher Scientific was used as the solvent. The solutions of sPS in THF was turned into δ -form sPS gel via natural cooling ²³⁻²⁶. The concentration of sPS in the solution was varied to obtain different values of porosity. sPS solutions with concentration 0.010, 0.015, 0.020, 0.025, 0.030, 0.040, 0.050, 0.060, 0.070, and 0.080 g/mL were prepared by dissolving sPS powder in THF in sealed glass containers kept in oil bath at 120 °C. The solution was poured in a disc shaped mold of 30 mm diameter and allowed to gel as the solution gradually cooled to room temperature. The thickness of the disc shaped gels was maintained at around 3.5-4.0 mm. The gels were aged in the mold for a day and washed and solvent exchanged with 200-proof ethanol for at least 3 days. sPS aerogels were obtained by supercritical drying in carbon dioxide.

For this purpose, ethanol in sPS gel was exchanged with liquid carbon dioxide before the supercritical drying step.

Monolithic gradient density aerogels were produced by a sequential injection method, whereby sPS solutions of different solid content were sequentially injected into the mold before or after gelation of the previously injected solution. For example, one gradient density aerogel was obtained by first injecting a solution of 0.05 g/mL sPS into the mold followed by injection of 0.03 and 0.01 g/mL solutions. The time interval of each injection was varied based on the gelation time of each solution. The gelation time of the solution containing 0.05 g/mL sPS was about 4.5 min. Thus, the 0.03 g/mL solution was injected before the 4.5 min mark or shortly afterwards. In this study, two types of gradient density aerogels were produced respectively by injecting solutions before and after gelation of the previously injected solutions.

The value of bulk density, ρ_b was obtained from the weight-to-volume ratio of the specimens. The permeability of air was obtained from Darcy's law, as in equation (1):

$$Q = \frac{kA}{\mu} \frac{\Delta P}{L} \quad (1)$$

In equation (1), Q is the measured volumetric flow rate of air, k is permeability, A is cross-sectional area of the face normal to the fluid flow, μ is the viscosity of air, ΔP is the measured pressure drop, and L is the thickness of the aerogel specimen. An air permeability tester (Frazier Precision Instrument, Hagerstown, MD) was used for this purpose. The disc shaped aerogel specimens were placed at the center of a perforated bottom plate with a central hole which allowed air flow (Figure 1). The hole sizes were 25, 22, and 5 mm in diameter. Darcy's law can be modified with the shape factor, G for holes much smaller than the diameter of the aerogel specimen³⁶. A sample holder covered the specimen and prevented lateral flow of air. The gaps between the specimen, the bottom plate, and the sample holder were eliminated using vacuum grease. Finally, the specimen with the assembly was placed inside the Frazier tester to measure the permeability (Figure 2). As seen schematically in Figure 2, a vacuum pump drew air through the aerogel specimen.

(a)

(b)

Figure 1. (a) Schematic and (b) experimental image of the assembly of sPS aerogel specimen, sample holder, and bottom retaining plate for permeability measurement. Vacuum grease is used to seal any gaps.

(a)

(b)

Figure 2. (a) Schematic and (b) experimental image of the arrangement in Frazier permeability tester.

The power of the vacuum pump was controlled to obtain several values of pressure drop, ΔP and volumetric flow rate, Q . Figure 3 presents a set of representative data on ΔP vs. Q used in the evaluation of air permeability, k .

Figure 3. Representative plot showing pressure drop vs. volumetric flow rate relationship.

Filtration efficiency (E) measures the fraction of the incident particles that a filter can capture after passing through the filter once, given in equation (2):

$$E [\%] = \frac{N_B - N_A}{N_B} \times 100 \quad (2)$$

In equation (2), N_B and N_A are respectively the number of particles before and after passing through the filter. Penetration, P is obtained from the value of E as follows:

$$P = (1 - E) \quad (3)$$

In this work, a TSI-8130 filter tester (TSI Inc., Shoreview, MN) that generated in situ polydispersed sodium chloride nanoparticles was used. The diameter of generated particles ranged from 25-150 nm with

an average diameter of 75 nm³⁷. A wide-mesh metal net attached at the bottom of a specimen holder (Figure 4) supported the aerogel specimen. The periphery of the aerogel specimen was sealed by a rubber O-ring. The face velocity (V_f) of air flow was varied between 25, 40, and 50 cm/sec. For each specimen, the particles were allowed to go through the specimen 10 times and the value of filtration efficiency corresponding to each incident was measured and an average value was determined. **The maximum efficiency that can be measured using TSI-8130 filter tester is 99.999%.**

Figure 4. (a) Schematic showing incident particles and the location of O-ring in reference to the aerogel specimen. (b) Experimental image of the assembly used in filtration efficiency measurement.

The internal structures of the sPS aerogel specimen were examined by scanning electron microscope (JEOL JSM5310) to visually identify how the trapped nanometric NaCl particles were retained. For this purpose, the aerogel specimens were recovered after filtration and frozen in liquid nitrogen. The frozen specimens were cut into pieces and a representative piece was mounted on an aluminum stub using carbon tape. The samples were sputter coated by silver (ISI-5400 Sputter Coater, Polaron).

The absorption capacity of the particles by the filter media is an important attribute as it relates to the life-time of a filter. In this work, the absorption capacity was determined by the value of porosity (Π) as in equation (4).

$$\Pi = 1 - \frac{\rho_b}{\rho_s} \quad (4)$$

In equation (4), ρ_b and ρ_s are respectively the bulk and skeletal density of aerogels. The skeletal density of sPS aerogels was found to be almost constant, 1.056 g/cm^3 , regardless of the sPS concentration in the precursor solutions and matched with data reported elsewhere²³⁻²⁶.

RESULTS AND DISCUSSION

The values of bulk density, porosity, permeability, and filtration efficiency of the monolithic δ -form sPS aerogels are presented in Table 1. The averaged values and standard deviation are reported where at least four multiple measurements were made. The bulk density of sPS aerogels varied from 0.02 to 0.1 g/cm^3 producing aerogel monoliths of porosity values in the range 90-98%.

Table 1. Bulk density, porosity, permeability, and filtration efficiency of monolithic δ -form sPS aerogels. The number in specimen designation, e.g., 0.010 in “sPS-0.010”, indicates polymer concentration in g/mL in original solution.

Specimen	Bulk density, ρ_b (g/cm^3)	Porosity, Π (%)	Permeability, k ($\times 10^{-10} \text{ m}^2$)	Filtration efficiency, E (%) @ $V_f \approx 50 \text{ cm/s}$	Pressure drop* $\Delta P/L$ (mmH ₂ O/cm) @ $V_f \approx 50 \text{ cm/s}$
sPS-0.010	0.020 $\pm$ 0.0012	98.11	9.74 $\pm$ 1.06	93.468 $\pm$ 0.4449	9.7
sPS-0.015	0.025 $\pm$ 0.0029	97.63	9.42 $\pm$ 0.30	95.860 $\pm$ 0.6092	10.1
sPS-0.020	0.030 $\pm$ 0.0007	97.16	8.17 $\pm$ 0.23	98.889 $\pm$ 0.9980	11.6
sPS-0.025	0.034 $\pm$ 0.0028	96.78	7.71 $\pm$ 0.27	99.734 $\pm$ 0.1170	12.3
sPS-0.030	0.042 $\pm$ 0.0018	96.02	7.40 $\pm$ 0.42	99.998 $\pm$ 0.0014	12.8
sPS-0.040	0.055 $\pm$ 0.0062	94.79	6.74 $\pm$ 0.26	99.999 $\pm$ 0.00	14.1
sPS-0.050	0.061 $\pm$ 0.0018	94.22	6.62 $\pm$ 0.62	99.999 $\pm$ 0.00	14.3
sPS-0.060	0.074 $\pm$ 0.0009	92.99	5.99 $\pm$ 0.06	99.999 $\pm$ 0.00	15.8
sPS-0.070	0.093 $\pm$ 0.0031	91.19	5.39 $\pm$ 0.58	99.999 $\pm$ 0.00	17.6
sPS-0.080	0.100 $\pm$ 0.0039	90.53	5.29 $\pm$ 0.18	99.999 $\pm$ 0.00	17.9

* Pressure drop per unit thickness of sPS aerogels calculated from permeability and air flow rate.

The permeability data reported in Table 1 followed a power-law relationship with the bulk density, as presented in equation (5).

$$k = (c\rho_b)^{-d} \quad (5)$$

In equation (5), c and d are constants with values 0.123 and 0.380 respectively determined from the relationship presented graphically in Figure 5.

Figure 5. Plot of permeability as a function of the bulk density of monolithic δ -form sPS aerogels.

A similar trend was reported for mesoporous carbon aerogels, although the permeability of carbon aerogels was much lower, 10^{-16} - 10^{-14} m² ³⁸. It is apparent from the data presented in Figure 5 that macroporous sPS aerogels offered much higher permeability, in the order of $\sim 10^{-10}$ m², due to high values of porosity, 90-98% (Table 1). The permeability of sPS aerogels reported in Figure 5 are within the range appropriate for practical filtration applications and is about one order of magnitude higher than the reported value for packed beds ²⁹.

The filtration efficiency (E) also follows a power-law relationship with the bulk density for $E < 1$, as seen in Figure 6. Such power-law behavior can be expressed as in equation (6):

$$E = [p(\rho_b - q)]^{1/r} \text{ for } E < 1 \quad (6)$$

In equation (6), p , q , and r are constants with values 50, 0.015, and 19 respectively obtained from fitting the permeability data presented in Table 1. The data in Figure 6 reveal that high efficiency of nanoparticle

filtration can be achieved using monolithic δ -form sPS aerogels of bulk densities of 0.042 g/cm^3 or higher. The permeability values corresponding to such high efficiency were found to be of the order of 10^{-10} m^2 . The values of pressure drop per unit thickness of aerogel specimen ($\Delta P/L$) computed from air flow rate and permeability data as listed in Table 1 fell in the range of 10-18 mm water per cm of aerogel specimen.

Figure 6. Filtration efficiency as a function of the bulk density measured at $V_f \approx 50 \text{ cm/s}$.

It is important to determine the organization of airborne nanoparticles that were retained inside the pores of aerogel monoliths during filtration test. Figure 7 presents SEM images of sPS aerogel before and after the filtration test. The open pore structures of sPS aerogel monoliths are apparent in Figure 7(a). It is seen in Figure 7(b) that the open pores seen in Figure 7(a) are now filled with sodium chloride particles indicating that the nanoparticles penetrated into the aerogel materials during filtration. However, the pore structures in Figure 7(b) appear condensed in comparison with the materials before filtration test, as in Figure 7(a). We attribute this to the artifact of specimen preparation for SEM. Recall that the aerogel specimens recovered after filtration were placed in liquid nitrogen and then fractured to reveal the cross-sectional plane. In the process, the aerogel specimen experienced substantial visible shrinkage, which helped retain the NaCl nanoparticles within the pores. The images of salt particles shown in Figure 7b

reveal that the largest particles were of a few hundred nanometer in size. In view of this and the fact that incident particles were in the size range of 25-150 nm³⁷, we infer that the aerogel filters used in this work were efficient in removing the nanometric particles.

The image in Figure 7(b) raises an interesting question about the filtration mechanism, e.g., if filtration occurred by cake filtration, depth filtration, or a combination of the two. The filtration efficiency data obtained for all aerogel specimens considered in this study revealed that filtration efficiency remained unchanged with the number of passes up to 10 passes that was exercised in this work. This indicates the absence of surface cake formation up to 10 passes. This also indicates that filtration occurred primarily by depth filtration mechanism. It is quite possible that some degree of cake filtration occurred due to trapping of larger NaCl particles inside the macropores, but its effect on overall filtration efficiency was not apparent as filtration experiments were conducted for only 10 passes.

Figure 7. SEM images of the sPS aerogel before filtration (a) and sPS aerogel with captured nanometric NaCl particles after filtration (b).

We now examine the filtration efficiency and permeability data of gradient density aerogel materials. Previously, gradient density aerogels were investigated in the context of interstellar grains collectors and acoustic impedance matching materials^{39, 40}. One anticipates that gradient density aerogels can also be used in filtration due to its unique feature in the form of gradients in porosity. For this purpose, sPS

aerogels fabricated with variation of bulk densities across the specimen thickness were prepared by sequential injection of solutions differing in sPS concentration e.g., 0.05, 0.03, and 0.01 g/mL in the order. The performances of these materials were compared with that of a single density material (sPS-0.040) with bulk density 0.055 g/cm^3 and the same thickness. The specimen sPS-0.040 provided 99.999% filtration efficiency as presented in Table 1 and Figure 5.

It is not intuitive if the gradient density sPS aerogel synthesized from one discrete layer of bulk density 0.055 g/cm^3 or higher and the other layer from lower bulk density materials, such as 0.02 g/cm^3 can also provide a filtration efficiency of 99.999%. To answer this question, one can invoke composite layered structures of successively larger pores or higher porosity. Such composite layered structures are more attractive as they provide higher porosity to potentially have greater volume for collection of the particles and thus to offer higher retention capacity. Two kinds of gradient density sPS aerogels were produced in this work. Specimen sPS-g1 was produced by sequential injection of sPS solutions of 0.010, 0.030, and 0.050 g/mL concentration. Each solution was injected after the previously injected solution formed a gel and the monolith had a clear boundary - a skin of each injected gel - between the layers. The skin or the skin layer was denser than the inner body of the structure of the gels⁴¹. The gel specimen sPS-g2 was also a gradient density material obtained from the same solutions as sPS-g1 except that the sequential injections were performed before the previously injected solutions had time to gel. In this case, no clear boundaries were observed in the specimens. Table 2 summarizes the values of bulk density, porosity, permeability, and filtration efficiency obtained for aerogel specimens sPS-g1 and sPS-g2. It is evident from the comparison of the data presented in Tables 1 and 2 that gradient density aerogels sPS-g1 and sPS-g2 come very close to single density aerogel sPS-0.030 in terms of bulk density and porosity. The air permeability of the gradient density aerogels is, however, about 20-30% lower. The data in Figure 8 show that the permeability of sPS-g1 and sPS-g2 gradient density aerogels fell below the power-law line obtained previously (Figure 5) for single density aerogels.

Table 2. Bulk density, porosity, permeability, and filtration efficiency of gradient density aerogels.

Specimen	Bulk density, ρ_b (g/cm ³)	Porosity, Π (%)	Permeability, k ($\times 10^{-10}$ m ²)	Filtration efficiency, E (%) @ $V_f \approx 50$ cm/s	Filtration efficiency, E (%) @ $V_f \approx 40$ cm/s	Filtration efficiency, E (%) @ $V_f \approx 25$ cm/s	Pressure drop* $\Delta P/L$ (mmH ₂ O/cm) @ $V_f \approx 50$ cm/s
sPS-g1	0.041 $\pm$ 0.0034	96.12	5.03 $\pm$ 0.27	99.999 $\pm$ 0.0 delamination	-	-	18.9
sPS-g2	0.043 $\pm$ 0.0021	95.93	5.94 $\pm$ 0.13	99.999 $\pm$ 0.0	-	-	16.0
sPS-g3	0.022 $\pm$ 0.0017	97.92	7.55 $\pm$ 0.53	99.999 $\pm$ 0.00	99.999 $\pm$ 0.00	99.981 $\pm$ 0.0090	12.6

* Pressure drop per unit thickness of sPS aerogels calculated from permeability and air flow rate.

We attribute the above reduction in permeability in the case of sPS-g1 and sPS-g2 specimens to additional skin layers that formed in the composite structures due to sequential injection process. The reduction (30%) is more apparent for aerogel specimen sPS-g1. Recall that sequential injection took place in this case after a previously injected solution gelled. Even in the case where sequential injections were made before the gelation of a previously injected solution, e.g., in the case of sPS-g2 aerogel specimen, the presence of additional skin layers could not be completely ruled out due to the time gap between the injections. This accounts for lower permeability value of sPS-g2 compared to the power-law trend for single density aerogels.

Figure 8. Permeability of gradient density aerogels.

The filtration efficiency of aerogel specimens sPS-g1 and sPS-g2 was 99.999% (Table 2). The 99.999% efficiency mark was reached for aerogels of single density when the bulk density was 0.055 g/cm³ or higher (Table 1). However, a lower overall bulk density of 0.041 g/cm³ was enough in the case of gradient density aerogels to reach 99.999% efficiency value. We attribute this to the skin layers that formed due to sequential injections.

We elaborate the effect of skin layer further by expanding the discussion to another gradient density aerogel (sPS-g3) prepared by sequential injection of only two solutions. The solution with 0.02 g/mL sPS was injected first into the mold followed by injection of 0.01 g/mL solution without giving much time for gelation of the former solution. The data in Table 2 show that sPS-g3 offered filtration efficiency 99.98% at the face velocity of 25 cm/s and 99.999% efficiency at face velocities of 40 and 50 cm/s. It is known in literature²⁹ that lower face velocity results in an increase of filtration efficiency of nanoparticles due to an increase of contribution of the diffusional deposition mechanism. However, the data obtained in this study is counterintuitive, as is evident from the data reported in Table 2. This interesting observation will be further explored in a future communication.

The trend observed in Figure 9 reveals another interesting information. The sPS-g3 gradient density aerogel with bulk density of 0.022 g/cm³ provided the same filtration efficiency (99.999%) as sPS-g1 and sPS-g2 at face velocity of 50 cm/s. However, the data in Table 2 show that this aerogel had much lower bulk density than the other two gradient density aerogels mentioned above. This indicates that the skin layers generated by sequential injections even in a two-layer material helped increase the filtration efficiency with air permeability of 7.55×10^{-10} m². A complete investigation of the expanse of the skin layer and its influence on air permeability will be communicated shortly.

Figure 9. Filtration efficiency of the gradient density aerogels measured at $V_f \approx 50$ cm/s.

CONCLUSIONS

The study established the applicability of monolithic sPS aerogels in nanometric particle filtration and presented several strategies for achieving high filtration efficiency while keeping high values of air permeability. The data presented in this paper established that both permeability and filtration efficiency of single density sPS aerogels have power-law relationships with bulk density. It was also revealed that monolithic sPS aerogels with bulk density 0.042 g/cm^3 or higher are needed to obtain high efficiency ($> 99.95 \%$) while permeability values are at an acceptable level $\sim 10^{-10} \text{ m}^2$. The internal porous structures of aerogels were able to retain the nanometric NaCl particles. The study also showed that design of gradient density in aerogels by sequential injection of polymer solutions can be an effective method to obtain higher porosity for retention of particles, lower bulk density, and 99.999% efficiency in comparison to single density aerogels. Skin layers formed during sequential solution injection were found responsible for higher efficiency.

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Macroporous aerogel monoliths can remove airborne nanoparticles by retaining the particles in the pores.