



The role of mesopores in achieving high efficiency airborne nanoparticle filtration using aerogel monoliths



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ABSTRACT

This study evaluates the role of small mesopore volume fraction in aerogel monoliths in achieving high efficiency airborne nanoparticle filtration. The diffusional flow in mesopores (diameter 2–50 nm) is more effective in nanoparticle capture than the viscous flow regime encountered in macropores (diameter > 50 nm). Substantial macropore fraction, however, is essential for achieving high air permeability. This idea is tested by evaluating the filtration efficiency of 25–150 nm diameter airborne sodium chloride nanoparticles and the permeability of air through hybrid aerogel monoliths with about 2–4% by volume mesopore content. The hybrid aerogels are prepared by growing discontinuous silica aerogel particles in the macropores of δ -form syndiotactic polystyrene (sPS). The results show that air permeability is not much affected by the silica content although the particle capture efficiency is increased to >99.95% due to the contribution of diffusional deposition aided by the mesopores. The aerogels develop cracks and reduce filtration efficiency at high silica content due to brittle nature of silica particle networks.

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1. Introduction

Small airborne particles, e.g., the fines with size 0.1 μm –1 μm and ultrafines with size <0.1 μm , are detrimental to human health as they can penetrate the human body and reach pulmonary alveoli [1]. These hazards become more alarming when the viral particles of size 20–2000 nm attach to the surfaces of airborne particles and enter the human body [2]. Nausea, breathing difficulties, bronchitis, birth defects, serious developmental delays, weakened immune systems, and even cancer are some of the aftermaths of exposure to and inhalation of these airborne particles [3,4]. In this context, air purification by filtration to remove the airborne nanoparticles is a logical measure for protection of human health.

The high efficiency particulate absorption (HEPA) filters improve air quality by filtering airborne particles at efficiency over 99.95% as per EN 1822-1:2009 classification. However, removal of airborne nanoparticles of sizes 100 nm and smaller using fiber mat-based HEPA filters still remains a challenge. In this context, aerogel monoliths offer several attractive features as filter media for capturing airborne nanoparticles. They are composed of solid networks with open pore structures, high porosity, large surface area, and cascading pore sizes [5–29]. Pores are defined as

micropores (pore diameter $d < 2$ nm), mesopore ($2 \leq d \leq 50$ nm), and macropores ($d > 50$ nm) as per IUPAC classification. Silica aerogels, the most studied aerogels to date, are mesoporous and offer surface area up to 1000 m^2/g and porosity over 90% [9–21]. The δ -form syndiotactic polystyrene (sPS) aerogels have porosity >97%, contain a small fraction of pores in the micropore range inside the nanocavities of crystalline strands, and a major fraction of pores as macropores [22–29].

Monolithic aerogels have the potential to serve as standalone filter media. However, only a few studies exploited the aerogel granules and microspheres in removal of airborne particles by arranging them in packed beds [30–33]. Several studies focused on viral particle detection by bioluminescent organisms colonized in silica aerogels [34], and removal of pollutants, such as cyanides or combustion gases by using monolithic silica aerogel composites as the medium for heterogeneous catalysts [7,35,36]. Prior work on direct usage of monolithic aerogels for filtration of airborne nanoparticles is scarce. Recently, we reported removal of airborne nanoparticles of size 25–150 nm (mean size 75 nm), with high efficiency (>99.95%) and air permeability of the order of 10^{-10} – 10^{-11} m^2 using macroporous monolithic sPS aerogels [37]. No detailed work exists on evaluation of airborne nanoparticle filtration potential of aerogel monoliths with the mesopores or with pore structures consisting of both the meso- and macropores.

The mesopores with pore dimensions 2–50 nm present interesting scenarios for fluid flow and thermal transport. These can

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be interpreted in terms of Knudsen number (Kn) as defined in Eq. (1) [38,39].

$$Kn = \lambda/d \quad (1)$$

In Eq. (1), λ is the mean free path of gas molecules and d is the mean diameter of the pores. A purely viscous fluid flow regime is obtained when $Kn \ll 1$ for monolith pore size much larger than the mean free path of the gas molecules. Note that for this regime, the viscous flow is driven by the pressure gradient and the viscosity of the fluid as per Darcy's law. The fluid flow becomes diffusional in nature when $Kn \gg 1$ for the mean free path much larger than the pore dimension. The diffusional flow is driven by the concentration gradient as per Fick's law. At an intermediate range, $Kn \approx 1$, the pore dimensions are similar to the mean free path of the gas molecules. In this case, the fluid flow is governed by both the viscous and the diffusional components. The mean free path of air is approximately 20 nm at a pressure of 370 kPa and temperature of 296 K, which corresponds to the size of mesopores [39]. It was reported that fluid flow through mesoporous silica aerogels at room temperature and pressure 6–200 kPa was in the intermediate regime [38]. The diffusional flow in the mesopores promote the capture of airborne nanoparticles of size less than 100 nm which experience Brownian motion and come in contact with the filter media. However, mesopores are not conducive to high air permeability due to their small sizes. An aerogel monolith with appropriate proportion of meso- and macropores, therefore, has the potential to offer both superior particle capture efficiency of mesopores and high air permeability of macropores.

In this work, a set of organic-inorganic hybrid macro- and mesoporous aerogel monoliths were produced and their effectiveness in nanoparticle filtration was evaluated. The hybrid monoliths were produced by growing discontinuous silica aerogel networks inside the macropores of δ -form sPS following a method published earlier [28]. This study answered the following questions. First, how sensitive is the air permeability data of the hybrid aerogel monoliths to mesoporous silica content? Second, how sensitive is the filtration efficiency data to mesoporous material content? In view of Kim, Chase, and Jana [37], macroporous sPS aerogel monoliths with porosity 96% or lower are effective as high efficiency filtration media for removal of airborne nanoparticles. In this context, can one achieve even higher efficiency without dramatically decreasing the solid porosity, for example, by incorporating appropriate quantities of mesoporous silica in a hybrid material with sPS? The answer to this question is not straight forward as introduction of mesopores may result in a reduction of air permeability and it is not known at the outset what fraction of mesoporous materials is appropriate. Third, considering the brittle nature of silica networks, do these monolithic filter media experience mechanical failure? Wang and Jana [28] observed synergy of compressive modulus in hybrid sPS-silica aerogel systems and reported an increase of compressive modulus by 250% over sPS aerogel monoliths.

2. Materials and methods

The monolithic δ -form sPS aerogels were considered a baseline material in this work. In conjunction, monolithic sPS-silica hybrid aerogel materials were developed to assess how mesopore content affects air permeability and filtration efficiency. sPS aerogel monoliths were fabricated using a two-step process consisting of thermo-reversible gelation and supercritical drying [22–29]. The hybrid aerogel monoliths were fabricated by growing mesoporous silica particle networks inside the sPS macropores following a procedure reported by Wang and Jana [28]. For this purpose, silica networks were grown inside the preformed sPS gels by double

catalyzed acid-base sol-gel reactions and the hybrid materials were dried under supercritical condition [28,29]. For materials, solid sPS pellets with 98% syndiotacticity and molecular weight 300,000 g/mol were purchased from Scientific Polymer Products Inc. (New York, NY) and converted into powder form for easy dissolution in reagent grade tetrahydrofuran (THF) purchased from Fisher Scientific. Tetraethyl orthosilicate (TEOS, reagent grade 98%) purchased from Sigma-Aldrich was used as the silane precursor for silica gel. Ethanol (EtOH, 200-proof) purchased from Decon (King of Prussia, PA) was used as the co-solvent of TEOS along with deionized water to conduct sol-gel reactions. Nitric acid (64–66%, Sigma-Aldrich) and ammonium hydroxide (28–30%, Fisher Scientific) served respectively as the acid and base catalysts in sol-gel reactions.

sPS solutions with solids concentration 0.020, and 0.025 g/mL were prepared by dissolving sPS powder in THF in sealed glass containers kept in oil bath at 120 °C. The porosity of sPS aerogel monoliths varied with the solid content. The solutions were poured in a disc-shaped mold of 30 mm internal diameter and allowed to cool down to room temperature to form δ -form sPS gels [26–29]. The thickness of the disc shaped gels was maintained at around 4.0 mm. The gels were aged in the mold for a day and washed and solvent exchanged with ethanol for at least 3 days. Ethanol in sPS gel was exchanged with liquid carbon dioxide and the materials were subjected to supercritical drying.

In case of hybrid gels, disc-shaped sPS gels filled with ethanol were produced as above from sPS solutions of concentration 0.020 and 0.025 g/mL. TEOS solutions were prepared in a mixture of ethanol and deionized water (DIW) in the molar ratio TEOS: ethanol:DIW = 1:x:5 with $x = 37, 46, \text{ and } 64$. TEOS was hydrolyzed to form hydroxysilanes under stirring for one hour at room temperature after the pH of these solutions was adjusted to 2.0 by adding nitric acid. The sPS gel specimens were soaked in hydroxysilane solution for one day and transferred to another container where a solution of ammonium hydroxide and DIW in 1:1 volume ratio was poured over the gel specimen drop by drop using a pipette so as to increase the pH to around 9.0 and to promote condensation of hydroxysilanes into silica particle networks. The hybrid gels were aged for a day, washed and solvent exchanged with ethanol for 3 days, and finally solvent exchanged with liquid carbon dioxide before drying under supercritical condition. Table 1 lists the concentration of solutions used in synthesis of hybrid aerogels. sPS aerogel monoliths are represented by sPS20 and sPS25 while the hybrid aerogels are represented by Hx-y where x represents sPS concentration in g/mL and y represents moles of ethanol per mole of TEOS in the solution.

The permeability of air was obtained by Frazier air permeability tester (Frazier Precision Instrument, Hagerstown, MD). The disc shaped aerogel specimen of thickness 3.5–4.0 mm was placed in a sample holder that covers the lateral face of the aerogel and prevents lateral flow of air [37]. Vacuum grease was applied to seal the interface between the sample holder and the specimen. Filtration

Table 1
Specific concentration of sPS and TEOS used in synthesis of hybrid aerogels.

Specimen	sPS conc. (g/THF mL)	TEOS conc. (mol/L) [TEOS:ethanol molar ratio]
sPS20	0.020	–
sPS25	0.025	–
H20-64	0.020	0.25 [1:64]
H20-46	0.020	0.33 [1:46]
H20-37	0.020	0.40 [1:37]
H25-64	0.025	0.25 [1:64]
H25-46	0.025	0.33 [1:46]
H25-37	0.025	0.40 [1:37]

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