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Superfine powdered activated carbon incorporated into electrospun polystyrene fibers preserve adsorption capacity

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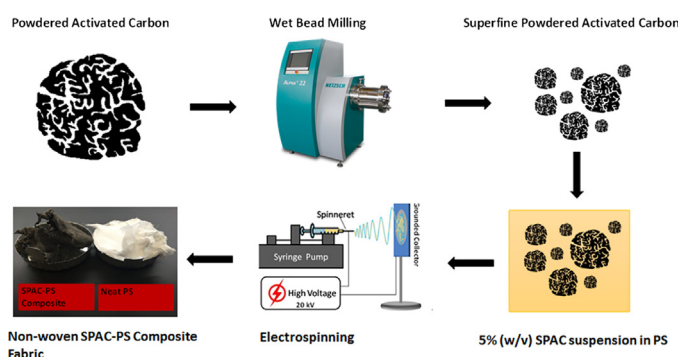
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HIGHLIGHTS

- Electrospun non-woven polystyrene fibers with superfine powdered activated carbon are fabricated.
- SPAC particles are fully or partially encapsulated by the polystyrene microfibers.
- Specific surface area of SPAC-PS composite is higher than neat PS fibers.
- SPAC particles conserve terminal adsorption sites after incorporation into PS fibers.
- Composition of SPAC-PS shows a synergistic increase in adsorption of phenanthrene from water.

GRAPHICAL ABSTRACT



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ABSTRACT

A composite material consisted of superfine powdered activated carbon (SPAC) and fibrous polystyrene (PS) was fabricated for the first time by electrospinning. SPAC is produced by pulverizing powdered activated carbon. The diameter of SPAC (100–400 nm) is more than one hundred times smaller than conventional powdered activated carbon, but it maintains the internal pore structure based on organic micropollutant adsorption isotherms and specific surface area measurements. Co-spinning SPAC into PS fibers increased specific surface area from 6 m²/g to 43 m²/g. Unlike metal oxide nanoparticles, which are non-accessible for sorption from solution, electrospinning with SPAC created porous fibers. Composite SPAC-PS electrospun fibers, containing only 10% SPAC, had 30% greater phenanthrene sorption compared against PS fibers alone. SPAC particles embedded within the polymer were either partially or fully incorporated, and the accessibility of terminal adsorption sites were conserved. Conserving the adsorptive functionality of SPAC particles in electrospun non-woven polymeric fiber scaffolding can enable their application in environmental applications such as drinking water treatment.

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1. Introduction

Electrospun non-woven polymeric fibers have a great potential for application in a multitude of areas including biomedicine, textile, electronics, optics and environmental remediation (Huang et al., 2003;

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Yoon et al., 2008). Fibrous structure of non-woven fabrics provides scaffolding advantages with tunable surface properties as well as high air and water permeability. In addition, electrospinning allows integration of functional nano- and submicro-sized particles to the fibrous macrostructure that can be applied in drinking water treatment. Providing access to clean water and maintaining the growing needs for quality and quantity is a National Academy of Engineering Grand Challenge that requires innovation and new strategies to treat water (Perry et al., 2008). Activated carbon adsorption is an existing best available technology capable of removing a broad spectrum of organic micropollutants (Crittenden et al., 2005). Recently, superfine powdered activated carbon (SPAC), which is activated carbon pulverized to sub-micron sizes, is gaining attention because of its favorable inherent properties as an adsorbent including very fine particle size, high porosity and large specific surface area (Matsui et al., 2013; Partlan et al., 2016; Ellerie et al., 2013). SPAC enables high adsorption capacity and fast adsorption kinetics because of its small size. Smaller SPAC particles contain a simpler inner pore structure, which subdues competition between natural organic matter and organic pollutants (Matsui et al., 2008; Partlan et al., 2016). However, it can be operationally challenging to settle SPAC from flowing water due to its small particle size and low density. As such, incorporating SPAC particles into a superior macroscale structure that preserves rapid adsorptive properties of SPAC would be desirable for treating water in large basins or as non-woven fabric reactor designs.

To create such a macroscale structure, composite sorbent was fabricated by co-spinning SPAC and polystyrene (PS), and a composite electrospun non-woven fabric matrix was produced. Electrospinning is a scalable, and cost-effective nano-fabrication method (Huang et al., 2003; Huang and You, 2013; Mohammadzadehmoghadam et al., 2015; de Faria et al., 2015). Electrospinning uses an electrically charged jet of polymer solution to produce polymer filaments by applying a high electrical potential difference (i.e., 10–40 kV). The surface tension on the fluid droplet at the tip of the syringe is overcome by the strength of the electric field, and a charged jet of fluid stretches and deposits onto the grounded collector, forming a layer of fibers with diameters in the micro- and nanometer scale. These fibers can be used in non-woven fabrics without post-treatment for a range of applications. To date, nano-additives including metal oxides (Hoogesteijn von Reitzenstein et al., 2016), single- and multi-walled carbon nanotubes (Jadhav et al., 2015; Sen et al., 2004; Mazinani et al., 2009; Bayley and Mallon, 2012), graphene (Das et al., 2013; Barzegar et al., 2015) and graphene oxide (de Faria et al., 2015) have been embedded as additives to enhance the mechanical, electrical and thermal stability of the electrospun fibers (Barzegar et al., 2015).

In this study, we aim to demonstrate incorporation of porous SPAC particles into electrospun polymeric matrices in a single step without post-treatment while preserving accessibility to SPAC's inner pore network. This novel material is demonstrated to remove organic pollutants from water when embedded in a polymeric fiber. SPAC particles were produced from parent powdered activated carbon via wet milling and then incorporated into PS via electrospinning. To the best of our knowledge, there is no previous literature related to incorporating SPAC particles into electrospun polymeric matrices. Freely suspended SPAC particles were tested side-by-side with SPAC-PS composite sorbents for nitrogen gas and phenanthrene (PNT) adsorption.

2. Experimental methods

2.1. Preparation of superfine powdered activated carbon (SPAC) by wet milling

Previous work with SPAC characterization (Partlan et al., 2016) was used to guide selection of bulk powdered activated carbon. Coal-based powdered activated carbon (PAC, WaterCarb-800) obtained from Standard Purification (Palm Beach Gardens, FL) was pulverized to sub-micron particle size via wet bead milling. The pulverization was

conducted by a Netzsch Premier Technologies LLC (Exton, PA) MiniCer Horizontal Bead Mill using 0.5 mm steel beads as grinding media. The milling chamber was 85% full of beads. An aliquot of 200 g parent PAC was suspended in about 800 g of deionized water, with additional water (a few hundred grams) added incrementally to decrease viscosity during milling. The slurry was recirculated through the machine for a total milling time of 7 h, with an agitator speed of 3935 rpm. SPAC-slurry was dried overnight in a vacuum oven at 105 °C.

2.2. Characterization of superfine powdered activated carbon (SPAC)

Specific surface area measurements were conducted with 30 mg of material degassed for ~18 h at 60 or 300 °C prior to nitrogen gas adsorption experiments. Lower temperature (i.e., 60 °C) was selected for polymer-containing samples to prevent structural losses via thermal decomposition. Nitrogen adsorption at 77 K was performed with a physisorption analyzer (Micromeritics ASAP 2020). The Brunauer-Emmett-Teller (BET) equation was used to calculate surface area from nitrogen gas adsorption isotherms. The density functional theory (DFT) model was used to calculate the pore size distribution. Total pore volume (V_T) was calculated from single point adsorption capacity at $P/P_0 = 0.99$.

Elemental analysis was performed using a Flash Elemental Analyzer 1112 series (Thermo Electron Corporation). Sizes for particles <6 μm in diameter were measured using dynamic light scattering (DLS) with a Zetasizer NanoZS (Malvern, Worcestershire, UK). Readings were taken in distilled water after bath sonication, and z-avg hydrodynamic diameters are reported. Particles larger than 6 μm were measured by optical microscopy imaging using a Zeiss Axioskop 2 Plus optical microscope with a Zeiss AxioCam MRC5 camera attachment running AxioVision AC version 4.2 software. Particles were sonicated before imaging, and Zeiss Immersionsol 518C immersion oil was used to view the particles at 40× magnification. The images were processed using ImageJ, an image processing software (Schneider et al., 2012), to determine the average diameter of the particles.

pH_{PZC} in the bulk material was measured by a pH drift method where the point of zero charge is defined as the pH where no drift occurs after 48 h. For each pH point and carbon, 100 mg of dry SPAC was added to 20 mL of pH adjusted 0.1 M NaCl in a CO₂-free background. After a minimum of 48 h on a shaker table, pH was measured in each vial and compared to a no-carbon blank (Partlan et al., 2016).

2.3. Fabrication of SPAC-PS composite material

Pristine PS pellets (MW 350,000 g/mol) and organic solvent (*N,N*-dimethylformamide, DMF) were purchased from Sigma-Aldrich (St. Louis, MO). SPAC-PS composite sorbents were prepared by dispersing 5% (w/v) of SPAC in DMF by bath sonication (Branson 2510, Branson Ultrasonic, Danbury, CT, USA). SPAC was weighed on a laboratory scale and poured from a plastic weigh boat into a 40 mL borosilicate glass vial with a Teflon-lined septa screw cap. An aliquot of 10 mL of DMF was then added to the SPAC inside the glass vial. The SPAC and the DMF were sonicated for 1 h in a bath sonicator. Finally, PS was weighed on a laboratory scale and mixed with the SPAC/DMF suspension. A small magnetic stirrer was added to the vial, and the vial was set on a heated stir plate at 300 rpm for 24 h at 55 °C. Polystyrene was added last to avoid its gelation when contacted SPAC in the sonicator. Bath sonication was used to minimize SPAC aggregation.

An electrospinning apparatus (see Fig. S1 in SI for a schematic diagram) similar to previously published electrospinning systems was utilized for fabricating neat (i.e., pristine with no additives) and composite fibers (Fong et al., 1999; Leach et al., 2011; Thavasi et al., 2008; Casper et al., 2004; Yang et al., 2004). Following our previously published procedure, electrospinning was performed using a high voltage power supply that provided 40 kV (Gamma High Voltage, Ormond Beach, FL), a syringe pump (New Era NE-300, Farmingdale, NY), a 10 mL plastic

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