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A nanofilter composed of carbon nanotube-silver composites for virus removal and antibacterial activity improvement

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ABSTRACT

We have developed a new nanofilter using a carbon nanotube-silver composite material that is capable of efficiently removing waterborne viruses and bacteria. The nanofilter was subjected to plasma surface treatment to enhance its flow rate, which was improved by approximately 62%. Nanoscale pores were obtained by fabricating a carbon nanotube network and using nanoparticle fixation technology for the removal of viruses. The pore size of the nanofilter was approximately 38 nm and the measured flow rate ranged from 21.0 to 97.2 L/(min·m²) under a pressure of 1–6 kgf/cm² when the amount of loaded carbon nanotube-silver composite was 1.0 mg/cm². The nanofilter was tested against Polio-, Noro-, and Coxsackie viruses using a sensitive real-time polymerase chain reaction assay to detect the presence of viral particles within the outflow. No trace of viruses was found to flow through the nanofilter with carbon nanotube-silver composite loaded above 0.8 mg/cm². Moreover, the surface of the filter has antibacterial properties to prevent bacterial clogging due to the presence of 20-nm silver nanoparticles, which were synthesized on the carbon nanotube surface.

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Introduction

The continued advancements in the field have shown that filter separation of particles from fluids to achieve high purity is a critical area in research and development of industrial technologies. Nanoparticle separation is becoming increasingly important as needs arise in diverse fields including the semiconductor, chemical, food, pharmaceutical, medical, and biochemical industries, as well as the environmental field. Especially in the environmental field, where the need for clean water and awareness of water shortages has increased, nanofiltration technology is a potentially viable solution.

Since their discovery in 1991 (Iijima, 1991), carbon nanotubes (CNTs) have attracted much attention in various scientific communities, with a myriad of applications in

electronics, composite materials, fuel cells, sensors, optical devices, and biomedicine. Moreover, CNTs, because of their high surface area, electronic properties, and ease of functionalization, have excellent nanosorbent properties for filtering contaminants from water (Diallo and Savage, 2005; Upadhyayula et al., 2009). Previous studies in this field have focused on the use of bare CNTs or CNTs functionalized with inorganic nanoparticles for adsorption of inorganic contaminants and toxic metals from water (Di et al., 2006; Li et al., 2003; Peng et al., 2005). Other studies have explored the use of CNTs for adsorption of low molecular weight organic contaminants (Lu et al., 2005) and toxins (Yan et al., 2006) from water.

In particular, CNTs have been studied as filters for the removal of viruses or bacteria. Single-walled carbon nanotube

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(SWCNT) filters have shown high bacterial retention (Brady-Estévez et al., 2008), and multi-walled carbon nanotube (MWCNT) filters have high viral removal efficiency at low pressure (Brady-Estévez et al., 2010a), both through the effects of size exclusion. Moreover, a SWCNT-MWCNT hybrid filter achieved efficient bacterial inactivation and viral retention at low pressure (Brady-Estévez et al., 2010b). The application of an external electric field markedly enhanced the viral removal by the CNT filter, because of the increased viral particle transport (Rahaman et al., 2012). Furthermore, in order to enhance the antibacterial ability of the CNT filter, vertically aligned MWCNT arrays were combined with silver nanoparticles (Akhavan et al., 2011), and CNT/cotton membrane was combined with silver nanowires (Schoen et al., 2010). A recent study demonstrated scalable applications of this technology that use low-cost and widely available CNTs for inactivation of microbes (Kang et al., 2008a,b, 2009). For viral removal by CNT-hybrid filters, filtration performance was also tested under various solution chemistries (Brady-Estévez et al., 2010c). These CNT-based filters remove the micrometer-sized bacterial cells through a sieving mechanism, whereas depth (physicochemical) filtration governs the adsorption of nano-scale viruses throughout the thickness of the matrix.

In this study, we describe a novel, highly permeable, MWCNT-silver (Ag) nanofilter and demonstrate its use for the effective removal of bacterial and viral pathogens from water at low pressure. Although previous studies have provided a proof-of-concept for viral removal by filters of various forms using CNTs, a CNT-Ag nanofilter in which Ag nanoparticles are synthesized on the CNT surface has not been reported. This filter was fabricated by utilizing the following key properties of CNTs and Ag nanoparticles: (1) the small diameter and high surface area of CNTs; (2) the tendency of CNTs to aggregate and form highly porous structures; (3) the low melting point of Ag nanoparticles; and (4) the antibacterial activity of Ag nanoparticles. We demonstrated that bacteria were completely retained on the CNT-Ag nanofilter and were effectively inactivated upon contact with Ag on the MWCNT. We also showed that viruses could be completely removed by a depth-filtration mechanism, that is, capture by nanotube bundles inside the CNT-Ag layer.

1. Materials and methods

1.1. Materials

MWCNTs were provided by Hanwah Nanotech (CM-95 grade) in South Korea. Polyvinylpyrrolidone (PVP) was purchased from Fluka (Mw: 40,000). Ethylene glycol, sodium dodecyl sulfate (SDS), oleylamine, silver nitrate (AgNO_3), ethyl acetate, and hexane were purchased from Sigma-Aldrich. Poliovirus 1 (ATCC No. VR-1562), Coxsackie type A9 virus (coxA9 virus, ATCC No. VR-186), and Norovirus GI RNA (ATCC No. VR-3199) were purchased from the American Tissue Culture Collection (ATCC). *Staphylococcus aureus* (KCTC 1928) and *Escherichia coli* (KCTC 1039) were purchased from the Korean Collection for Type Culture (KCTC).

1.2. Preparation of the CNT-Ag composite

Thin MWCNTs (0.3 g) were loaded into a 500-mL round flask reactor, to which 280 mL of ethylene glycol was added, followed by stirring for 30 min. Ethylene glycol acts as a weak reductant that slows down the growth of the Ag nuclei. (Ducamp-Sanguesa et al., 1993). Subsequently, the reactor was placed in an ultrasonic cleaner, followed by dispersion of the carbon nanotubes in ethylene glycol for 3 hr using ultrasonic waves at a temperature of under 50°C. Post-ultrasonication, a stirrer was attached to the reactor and a thermometer and condenser, for cooling, were connected. While stirring the reactor, 1.68 g of PVP and 5.6 mL of oleylamine, to which 1.102 g of AgNO_3 was added in a stepwise manner, were added. A vacuum pump was connected to replace air in the reactor with nitrogen. While the nitrogen was continuously supplied, nitrogen was forced to circulate within the reactor to prevent oxygen inflow. A mantle was attached to the bottom of the flask and the temperature of the reactor was raised to 200°C for 40 min, followed by reduction for 1 hr. The temperature of the reactor was lowered slowly to room temperature for 3 hr upon completion of reduction. The generated CNT-Ag composites were filtered with a filter paper, followed by washing with ethyl acetate and hexane several times (Cha et al., 2005; Kim et al., 2007, 2008).

1.3. CNT-Ag nanofilter preparation

The generated CNT-Ag was uniformly coated on a glass fiber (GF) membrane with a pore size of 0.7 μm (Whatman, USA) by using a sonication/filtration procedure. Specified quantities of CNT-Ag (0.03–0.15 g) were suspended in 500 mL of deionized water containing 0.5 g of SDS. The suspension was sonicated for 1 hr, and then vacuum-filtered through a GF membrane to achieve the various loadings of CNT-Ag on the base filters. Ethanol (100 mL) followed by 500 mL of deionized water were passed through the CNT-Ag nanofilter to remove residual SDS and ethanol. Then, the fabricated CNT-Ag nanofilter was dried in an oven for 12 hr at 60°C. Finally, the CNT-Ag nanofilter was heat-treated for 10 min at 250°C.

1.4. Characterization of CNT-Ag composite and nanofilter

A field-emission transmission electron microscope (FE-TEM, JEOL, JEM-2010, Japan) at an acceleration voltage of 200 kV was used to investigate the size and distribution of the silver on the CNT surface. TEM specimens were prepared by placing a few drops of sample solutions on a carbon grid. The crystalline structures of the synthesized CNT-Ag nanocomposite were investigated using an X-ray diffractometer (XRD). Lyophilized and powdered samples were used, and the diffraction patterns were recorded in the scanning mode on an X'pert Pro diffractometer (PANalytical, Almelo, the Netherlands) operated at 40 kV and with a current of 30 mA, with $\text{Cu}/k\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) in the range of 20°–80° 2 θ .

The surface morphology of the CNT-Ag nanofilter was studied using field emission scanning electron microscopy (FE-SEM, S-4300SE, Hitachi, Japan). Wettability measurements were performed using a Video-Based Optical Contact Angle Meter (Dataphysics, OCA15EC). Distilled water was used as the

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