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The use of carbon nanotube yarn as a filter medium to treat nitroaromatic-contaminated water

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Abstract: Carbon nanotube yarn (CNTY) is a promising material for the removal of organic contaminants from aqueous waste streams owing to its extraordinary mechanical strength, chemical stability, thermal stability and high surface area. CNTY was used to treat water contaminated with a model nitroaromatic compound, 2,4-dinitrotoluene (DNT). The isotherms and kinetics of DNT adsorption onto CNTY were investigated. The adsorption capacities of DNT were compared with the literature values of alternative sorbents. SEM-EDX, HRTEM, Raman spectroscopy and XPS were used to characterize the size, surface morphology and surface chemistry of the CNTY before and after DNT adsorption. Results indicate that adsorption isotherm of DNT onto CNTY could be fitted by the Freundlich isotherm with a Freundlich constant, K_F , of 55.0 mg/g (L/mg)1/n and a Freundlich exponent, 1/n, of 0.737. Adsorption kinetics can be formulated by the pseudo-second order kinetic model. This study demonstrates the ability of CNTY to remove organic contaminants from water.

Key Words: Carbon nanotube yarn; 2,4-dinitrotoluene; Adsorption; Freundlich isotherm; Emerging technology

1 Introduction

Activated Carbon (AC) is a common adsorbent that is used to remove contaminants from water [1]. Though AC is an excellent adsorbent, it is not ideally suited for removal of polar molecules. In addition, the application of AC in water treatment has several challenges, due to its slow adsorption kinetics and resource requirements for regeneration and reactivation [2]. Modified Carbon Nanotubes (CNT) have the ability to remove polar molecules $[3-6]$, adjustable structures and surface chemistries, and unique properties such as chemical stability, mechanical and thermal stability as well as high surface area. Given these characteristics, CNT have great potential application as a third generation carbonaceous adsorbent $[2]$. In addition to water treatment, CNT have found a number of other uses in hydrogen storage, protein purification, and biomedical applications $[7-11]$.

In addition to their use as sorbents $[2, 5, 12]$, CNT-based nanotechnologies have been applied as catalysts $[13, 14]$, membrane filters $[2, 7]$, and electrodes $[10, 15]$, in water treatment

applications. As sorbents, multi-wall CNT (MWCNT) and single-wall CNT (SWCNT) have been used to remove organic and inorganic pollutants from aqueous solutions as well as gaseous mixtures $[12, 16]$, . Although CNT have several advantages over conventional sorbents like ACs (e.g., faster kinetics and the ability to add functional groups to enhance adsorption and/or degradation of target contaminants $[12, 16]$, use in practical applications presents challenges $[2]$. Due to their small size, CNT cannot be filtered or settled, making recovery problematic $[2, 9]$. Therefore, recent research into water treatment applications of CNT as sorbents has focused on using CNT incorporated into sponge-like structures $[7, 12, 17]$. It has been demonstrated that such three-dimensional CNT structures can adsorb about 3.5 times more mass of a dissolved organic compound (e.g., o-dichlorobenzene) than an individual CNT $^{[18]}$. In addition, because of their structure, the CNT can be easily collected, regenerated, and reused $[9, 19]$. A new method of spinning CNT into a yarn has recently been developed ^[11]. CNTY consist of assemblies of individual CNT bonded together by van der Waals forces and mechanical

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interlocking $[20]$. The yarns were found to be mechanically robust with exceptionally high specific surface area. Although CNTY has been applied in a variety of ways (e.g., medical scaffolding, intelligent clothing, electrical sensors) $[12, 12]$ ^{14]}, to our knowledge, they have not been used in environmental applications. Based on the advantages that have been observed when using structured CNT as adsorbents [17, 19, 21], investigation of CNTY as a sorbent to remove organic contaminants from water is warranted.

 The main goal of this study is to investigate the application of CNTY to remove organic contaminants from water. A nitroaromatic compound such as 2,4-dinitrotoluene (DNT), frequently detected in surface and groundwater [22], will be used as a model organic contaminant. The main objectives of this study are to (i) characterize CNTY using microscopy and spectroscopic techniques, and (ii) determine the rate and extent of DNT adsorption onto CNTY, and compare these results to the rates and extent of adsorption of DNT onto alternative sorbents (e.g., AC and CNT powder) reported in literature.

2 Methods Section

2.1 Materials and Chemicals

Chemicals (sodium hydroxide (NaOH), hydrochloric acid (HCL), nitric acid ($HNO₃$), and DNT) were purchased from Sigma-Aldrich (St. Louis, MO). All reagents were used as received. All experiments were conducted in ultra-pure water (18 MΩ-cm) produced by a Millipore water purification system (Millipore, Billerica, MA).

The CNTY (60-yarn) were procured from Nanocomp Technologies, Inc., who uses a direct spinning method to produce CNTY. The process is proprietary, but in general CNTY are made by injecting grain alcohols and iron-based catalysts into a horizontal reactor furnace with hydrogen as the carrier gas. Free CNT and CNT bundles are produced and exit the reactor as an aerogel-like material. In a post processing operation the CNTY is drawn through an acetone bath and twisted prior to collection on a spool $[23]$.

2.2 Methods

Batch kinetic and equilibrium adsorption experiments were conducted in triplicate with CNTY. The adsorption experiments were performed in 100 mL amber serum bottles having aluminum-coated septum caps. The pH values for all the samples before and after experiments were measured (Mettler Toledo Instruments). The ionic strength of the solution was maintained at 1 mM using NaCl, the pH was maintained at 6.5, and the experiments were conducted at room temperature.

For the kinetic experiments, 50 mL of 10 mg·L⁻¹ of DNT solution was added to 100 mL amber serum bottles containing $~1$ mg CNTY. Ionic strength was maintained at 1mM with the addition of NaCl. The pH of the solution was adjusted to pH 6.5 using NaOH. Bottles were prepared in triplicate and mixed using a magnetic stir bar. Aliquot samples of 0.2 mL were removed from the bottles at different time intervals (0-72 h) to determine the rate of adsorption. Samples were diluted with 0.8 mL of deionized water and filtered using a 0.2 μm filter and analyzed by a UV-Vis absorption spectrophotometer (UV-Cary 60, Agilent Technologies). Control samples (triplicate) were run in parallel to confirm significant DNT losses were not occurring without the adsorbent.

For the adsorption isotherm studies, a known mass of CNTY (-1 mg) was transferred to 100 mL bottles that contained 50 mL of solution at various DNT concentrations (0, 1, 2, 4, 8, 16, and 24 mg·L⁻¹). The isotherms were conducted using a similar method as reported by Randtke^[24]. The contents of the bottles were mixed with a magnetic stirrer. After 72 h the bottles were centrifuged and a 4 mL aliquot sample was withdrawn to determine the dissolved DNT concentration at equilibrium using gas chromatography/mass spectrometry (GC/MS) (GC 7890A; Agilent Technologies). The solid phase concentration of DNT was determined by mass balance. Preliminary work showed no further reduction in DNT concentration after 24 h and therefore 72 h was conservatively chosen as the experimental equilibrium time.

Details about the surface morphology of CNTY were obtained using a high resolution scanning electron microscope (Quanta SEM 450 (2 kV using EDAZ (20 kV), FEI). For SEM, CNTY was mounted as received onto an aluminum stub using carbon tape. Images were taken at multiple magnifications. Additional internal morphology of CNTY was determined using high resolution transmission electron microscopy (HRTEM) at 200 kV using a Philips TEM (CM200 LaB6, Philips Corporation).

Raman spectroscopy is an extensively used analytical tool for the characterization of structure and transformations of materials including CNT. Thus, to analyze the possible interactions between CNTY and DNT, Raman spectra were measured on the CNTY samples and controls using aLabRamHR 800, Horiba Scientific Inc. and a high resolution BX41 confocal microscope. The excitation source was a He-Ne laser (632.8 nm) set to an output of 17 mW. The laser beam (\sim) µm in diameter) was directed onto the solid samples placed on microscope glass slides through an Olympus 100x objective. Acquisition parameters were holographic grating of 600 grooves mm-1, confocal hole of 300 µm, and accumulation time of 5 s. Under these conditions, the spectral resolution was about 1 cm⁻¹. Each micro-Raman spectrum was averaged over 5 cycles, and was collected with the help of a thermo-electric cooled charge coupled device (CCD) detector (1024×526) pixels). Spectral data was processed and plotted in Origin 8 software.

Using molecular orbital theory and the binding energy of electrons, X-ray photoelectron spectroscopy (XPS) provides information about the chemical structure of materials. In this work, XPS was used to determine differences in the CNTY pre- and post-adsorption. An XPS (M-Probe, Surface

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