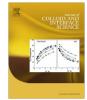
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## A novel arsenic removal process for water using cupric oxide nanoparticles

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#### ABSTRACT

Recent studies suggest that the cupric oxide (CuO) nanoparticles effectively adsorb aqueous arsenic species under a wide range of water chemistries. However, to develop CuO nanotechnology to a field application level, further studies are necessary. Batch adsorption kinetic experiments were conducted to determine the time course of uptake of arsenic by CuO nanoparticles. A reactor with CuO nanoparticles was developed to conduct continuous flow-through experiments to filter arsenic from groundwater samples. Groundwater samples spiked with 100  $\mu$ g/L of arsenic were passed through (1 L/h) the flow-through reactor. Samples from the flow-through reactor were collected at a regular interval and analyzed for arsenic and other chemical components (e.g., pH, major and trace elements). The CuO nanoparticles adsorbed with arsenic were regenerated with a sodium hydroxide (NaOH) solution and tested again in the flow-through reactor. Three natural groundwater samples with above  $10 \,\mu g/L$  of arsenic were also tested with the flow-through reactor. The arsenic adsorption process by CuO nanoparticles was kinetically rapid and followed the pseudo-second-order rate. The continuous flow-through reactor with CuO nanoparticles was effective in filtering arsenic from spiked or natural groundwater. The regenerated CuO nanoparticles were also effective in filtering arsenic from groundwater. Arsenic mass balance data from regeneration studies suggested that 99% of input arsenic concentration was recovered. The CuO nanoparticle treatment did not show any discernible effects on the chemical quality of groundwater samples. Results of this study suggest that CuO nanoparticles show potential for developing a simple process for field applications to remove arsenic from water.

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#### 1. Introduction

Arsenic in water is derived from natural and anthropogenic sources. The natural sources include weathering and dissolution of arsenic minerals. The anthropogenic sources include disposal of solid by-products from combustion processes (e.g., coal plants, solid waste incinerators, cement plants, and paper mills), discharge of produced water from in situ extraction processes (e.g., uranium, oil, and natural gas), and application of arsenic based pesticides [1,2]. The most common inorganic arsenic species found in natural water under anoxic and oxic environments include arsenite(III) and arsenate(V), respectively. Dissolved inorganic arsenic in water is toxic to humans. Studies suggest that arsenite is more toxic than arsenate to humans; long-term exposure to drinking water containing arsenic in excess of 50 µg/L causes increased occurrences of skin, lung, bladder, and kidney cancer resulting in premature death [3]. Widespread occurrence of natural arsenic (As) in groundwater has been reported from many parts of the world. The health of millions of people is at risk due to exposure to unsafe

levels of arsenic in groundwater. The World Health Organization (WHO) and US EPA recommend 10  $\mu$ g/L of arsenic as the limit for human drinking water.

Global awareness of arsenic contamination of drinking water supplies has skyrocketed during the 1990s when more than 35 million people in Bangladesh and West Bengal, India were inadvertently poisoned by drinking arsenic contaminated water [4,5]. Further studies illustrate that elevated levels of naturally occurring arsenic in groundwater are a prevalent problem affecting many regions of the world [6].

Widespread efforts are being made globally to develop effective and affordable technologies for the removal of arsenic from water. Conventional adsorbents such as aluminum, iron, manganese, titanium, and ferric phosphate were studied extensively to remove arsenic from water [7–11]. In addition to these, zeolites and coal combustion by-products (e.g., fly ash) were also used as sorbents to remove arsenic from water. However, it is difficult to remove both oxidation states simultaneously from water under a wide range of pHs and concentrations of competing anions including phosphate, silicate, and sulfate [12]. In addition, disposal of waste by-products (sludge or spent media) produced from conventional adsorbents is a problem. Due to these reasons, the conventional adsorbents are not sustainable [12–16].

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Reddy and Viswatej [17] were the first to observe that cupric oxide (CuO) can remove both arsenite and arsenate from water without adjusting sample pH or redox potential. Martinson and Reddy [18] expanded these studies and reported that CuO was effective arsenic adsorbent because it did not require pH or redox potential adjustments, and it worked well in the presence of competing anions. These studies attributed the effective removal of arsenic to CuO high zero point of charge (ZPC)  $(9.4 \pm 0.4)$  and the oxidation of arsenite to arsenate by the CuO surface. Martinson and Reddy [18] also modeled the arsenic adsorption equilibrium process using Langmuir and Freundlich isotherms and reported that adsorption of arsenic by CuO nanoparticles was best modeled with the Langmuir isotherm. These studies also reported that CuO nanoparticles were effective in removal of arsenic across a wide range of groundwater chemistries. Since Martinson and Reddy [18] studies were published, and other researchers also tested arsenic removal from water using CuO nanoparticles and reported similar results [19-23].

For example, Pillewan et al. [20] examined arsenic removal from water using CuO incorporated mesoporous alumina. These studies reported that incorporation of CuO into alumina improved the removal of arsenic from water. Goswami et al. [21] reported that CuO found to be a potential nano-adsorbent to remove arsenic from water. In another study, Schilz et al. [23] treated in situ uranium produced water with CuO nanoparticles to remove arsenic. These studies reported CuO nanoparticles effectively removes arsenic and render in situ uranium produced water less toxic to cells. Overall, above studies demonstrate the effectiveness of CuO nanoparticles in removal of arsenic from water under a wide range of water chemistries and geo-hydrological conditions. However, in addition to batch equilibrium studies, a better understanding of adsorption kinetics and continuous flow-through experiments are required to develop CuO nanotechnology for field applications.

The objectives of this research were to (1) examine arsenic adsorption kinetics by CuO nanoparticles and (2) design and develop a reactor to conduct continuous flow-through experiments. Batch and continuous flow-through reactor experiments were conducted to determine the effectiveness of CuO nanoparticles in the removal of arsenic from groundwater samples. Cupric oxide nanoparticles adsorbed with arsenic were regenerated and tested again with the continuous flow-through system to test their effectiveness in the removal of arsenic from groundwater samples. Data from these experiments could help develop a practical one-step process for field applications to filter arsenic from natural groundwater.

#### 2. Materials and methods

#### 2.1. Materials

The groundwater used in this study was collected from the Casper aquifer at a residential site in eastern Laramie, WY, USA following the procedures of Wyoming Department of Environmental Quality [24]. The water was collected in high-density polyethylene (HDPE) bottle. Before collection of the sample, the well was purged until a water quality indicator (e.g., pH) was stable. The groundwater was spiked with equal volumes of 1000 µg/L As(III) and 1000 µg/L As(V) standard solutions to obtain 100 µg/L arsenicspiked water. The standard solutions were prepared by dissolving sodium arsenate heptahydrate (Na<sub>2</sub>HASO<sub>4</sub>·7H<sub>2</sub>O, Sigma) and sodium arsenite (NaAsO<sub>2</sub>, Sigma) in distilled water. The CuO nanoparticles used in this study were synthesized following the procedures as described in Martinson and Reddy [18]. The synthesized CuO nanoparticles were subjected to shape and surface area experiments.

#### 2.2. Batch experiments

All experiments were conducted in 50 mL polypropylene centrifuge tubes (Corning 430829). 200 mg and 10 mg of the nanoparticles were placed in the tubes, and then, the arsenic-spiked groundwater sample was added to set the concentration of nanoparticles to 4 g/L and 0.2 g/L, respectively. The tubes were sealed and placed on a bench top orbital shaker table (Labline Orbit Shaker) set at 250 rpm. The samples were allowed to react with nanoparticles for a period of time between 2 min and 12 h. After the set reaction time, the samples were centrifuged for 4 min to allow the nanoparticles to settle at the bottom of the tubes. The supernatant was then filtered using a syringe filter (Millipore 0.45  $\mu$ m) and then collected in a beaker for arsenic analysis. Samples were acidified with nitric acid and analyzed for arsenic concentrations.

#### 2.3. Flow-through experiments

#### 2.3.1. Design of flow-through reactor

An acrylic flow-through reactor was designed and developed to test arsenic removal with CuO nanoparticles (Fig. 1). The flow-

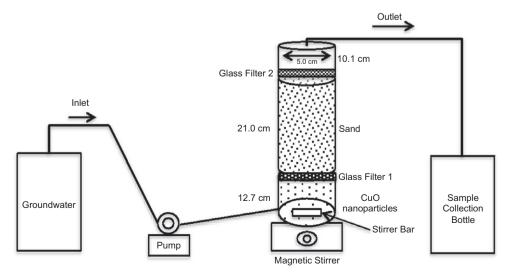


Fig. 1. Continuous flow-through reactor to remove arsenic from groundwater samples.

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