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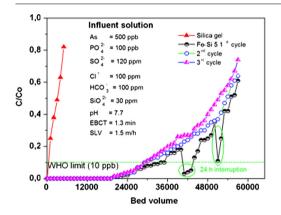
# Synthesis and characterization of hybrid iron oxide silicates for selective removal of arsenic oxyanions from contaminated water



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#### ABSTRACT

A series of hybrid silicates containing iron oxides was synthesized by adding various amounts of iron into silica on the basis of using post precipitation mechanism from aqueous solutions in order to form hydrated iron oxide species (HFO). The obtained materials were characterized by XRD, SEM, TEM, FTIR, Zeta potential and low temperature nitrogen sorption. XRD analysis confirmed that HFO-Si was amorphous and iron oxide crystals in the silica cannot be detected due to the lower size of HFO, which is the probability of its occupied interstitial positions in the silicate matrix. However, TEM image showed the presence of nano-sized HFO located inside the silicate matrix. The adsorption properties and selective efficiency of the synthesized materials were examined with respect to the removal of As(V) and As(III) species from contaminated water under different experimental conditions and in the presence of competing anions. Increasing iron loading enhanced the arsenic adsorption capacity of HFO-Si; the maximum level of arsenic removal was 300 mg As/g HFO-Si after 5 successive loads.

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#### 1. Introduction

Arsenic is among the rather hazardous metals which can be found as a natural element in the Earth's crust, and it occurs widely in the environment. In humans, long-term exposure to arsenic is associated with lung, liver, kidney, bladder and skin cancer. Inges-

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tion of large doses of arsenic may be lethal, while comparatively lower exposure levels can cause a variety of systemic effects including irritation of the digestive tract, vomiting and diarrhea, decreased production of erythrocytes and leukocytes, abnormal cardiac functions, blood vessel damage, liver and/or kidney damage and impaired nerve functions in hands and feet [1–4]. Arsenic pollution in drinking water supplies is reported in more than 70 countries, posing a serious health hazard to an estimated 150 million people worldwide [5,6]. International World Health Organization (WHO), United States Environmental Protection Agency (EPA) and many local organizations strive to reduce the occurrence of arsenic in drinking water from 50 to 10  $\mu g \, L^{-1}$  [7].

Leading techniques employed for the removal of arsenic include oxidation/precipitation, coagulation/electrocoagulation/co-precipi tation, sorption and ion-exchange based methods, and membrane-based methods. A number of bioremediation techniques are tried as well, but none was found effective enough for compliance with drinking water standards [8]. More recently, adsorption techniques involving the use of an adsorbent exhibiting high-capacity, durability, and high removal levels have been developed and finding wider applications. In this context, many materials are tried and analyzed as adsorbent for the removal of As (III) and As (V) from water. In addition to commercially available activated carbons [9], several newer types of activated carbons were synthesized and used for the removal of arsenic from water/ wastewater [10–12]. In addition, low cost adsorbents are also used for the removal of arsenic. The following is a non-exhaustive list of adsorbents used for this purpose: agricultural products and byproducts, industrial by-products/wastes such as charcoals [13,14], red sludge [15], blast furnace slag [16,17], Fe(III)/Cr(III) hydroxide waste [18], TiO<sub>2</sub> with different properties such as nanocrystalline TiO<sub>2</sub> particles [19,20], titanate nanotubes [21], hydrous TiO<sub>2</sub> [22,23], granular TiO<sub>2</sub> [24] and TiO<sub>2</sub>-impregnated chitosan beads [25], cupric oxide (CuO) nanoparticles [26-29], fly ash [30], soil [31], sand [32-34], clay minerals [35-37], zeolites [38,39], single or mixed oxides or hydroxides [19,24,40–44], hydrotalcites [45], phosphates [46], metal-based materials [47], and biosorbents [48–52].

A range of iron (III) oxides such as amorphous hydrous ferric oxide(FeOOH), poorly crystalline hydrous ferric oxide (ferrihydrite) and goethite (α-FeOOH) has been reported as promising adsorbents for removing As(III & V) from aqueous solutions [53–57]. Despite strong affinities of iron(III) oxides to As(III&V), their use so far has been limited due to reactor configurations incorporating large sedimentation and filtration units, which pose challenges in terms of solid/liquid separation [58]. In response, binary oxide was formed using amorphous hydrous ferric oxide (FeOOH), due to its selectivity and capability to remove high doses of As, with silica gel as a carrier in order to obtain granular adsorbent with high physical strength in water, chemical stability, scratch-proof characteristics, dynamic resistance, durability, in addition to high surface area and low cost. In addition, such binary oxides can be regenerated if required [57]. Furthermore, it is reported that the hybrid silicates modified with iron have high adsorption capacity and selectivity for arsenic removal [57,59].

The present study is based on hybrid silicates containing iron oxides, synthesized by loading hydrated iron oxide species (HFO) using post precipitation mechanism to add various amounts of iron from aqueous solutions into silica. The effect of iron content, adsorption kinetics, isotherms, and pH on arsenic adsorption using hybrid silicates containing iron oxides were studied in a number of experiments. Finally, the interference of phosphate, sulfate chlorate and carbonate on the adsorption of arsenic were reviewed.

#### 2. Materials and Methods

#### 2.1. Synthesis of HFO-Si

High-purity silica gel (pore size 60 Å; mesh size 70–230) and  $FeCl_3$  (anhydrous, powder,  $\geqslant$  99.99% trace metals basis) purchased from Sigma Aldrich were used for the synthesis of iron loaded materials. Silica gel (25 g) was first treated with 5% (wt.) NaOH solution and kept under vigorous shaking for 5 min, followed by washing with deionized (DI) water for several times till neutralization. The washed material was divided into 5 parts and then treated with different doses of  $FeCl_3$  (1–5 g). For example, 5 g of silica was treated with solution containing 1 g of  $FeCl_3$ , dried at 100 °C over night before further thermal treatment at 200 °C for 24 h and thereafter denoted as HFO-Si1. In this vein, each sample was denoted in accordance with the naming scheme HFO-Si (1–5), with reference to the number of Fe loadings.

#### 2.2. Adsorbent characterization

HFO-Si was characterized by scanning electron micrograph (SEM), (JEOL/JSM-6610), combined with energy dispersive X-ray (EDX), (OXFORD INSTRUMENTS INCA X-Act/51-ADD0013), to provide a complete map of the formed HFO-Si nanoparticles. In addition, TEM analysis for the synthesized samples was performed using JEOL-2100.

The crystal structure of the synthesized HFO-Si was investigated using X-ray Diffraction (XRD) on a Rigaku Smart Lab X-ray diffractometer using non-monochromotographic Cu K $\alpha$ 1-radiation (40 kV, 40 mA,  $\lambda$  = 1.5). Scans were performed in the range 5–65° of 20.

The specific surface area and micropore volume of the samples were measured using  $N_2$  adsorption—desorption (AUTOSORB 1C) at - 196 °C. Prior to adsorption, the samples were evacuated, heated up to 200 °C and evacuated until a pressure of 1.3 Pa was reached, only to be maintained through the night. The surface area, total pore volume and micropore volume were determined by multipoint BET (Brunauer, Emmett and Teller), t-plot and DR (Dubinin–Radushkevic), respectively.

Infrared adsorption measurements were carried out using a Fourier Transform Infrared (FTIR) spectrophotometer (Bruker Optics-Alpha) before and after arsenic adsorption of HFO-Si. The FTIR spectra were obtained in the wavenumber range 400–4000 cm<sup>-1</sup> using single bounce ATR with diamond crystal.

The content of loaded iron as well as initial and non-adsorbed concentrations of arsenic in supernatants was determined by atomic absorption spectroscopy (AAS) (GBC A 4382).

Iron content of silica was estimated via the treatment of 0.1 g of all samples with 10 ml (2 M) HCl to leach out the precipitated iron hydroxide before analysis with AAS.

Zeta potential measurements were conducted using a zetameter (Zetameter Inc., USA). 0.005 g of samples was suspended in 100 ml of water and the particles were homogenized at 2 h using an ultrasonic bath. After ultrasonication, the aqueous suspension was equilibrated at different pH values for 30 min. The equilibrated slurry was injected into the micro electrophoresis cell using disposable syringes. Zeta potentials of HFO-Si (1–5) were also measured at 7 pH, as well as at different pH values for HFO-Si3 and HFO-Si5. Prior to each measurement, the electrophoresis cell was thoroughly washed and rinsed with deionized water, followed by the introduction of the sample solution to be analyzed.

0.01 g of silica gel and HFO-Si samples was added to each flask containing 25 ml water at varying initial pH levels (1.5–12), by adding a few drops of HCl and/or NaOH, and all the flasks were shaken in a shaker bath at 200 rpm for 24 h. The final pH levels of the

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