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NANOCOMPOSITE MATERIALS FOR As(V) REMOVAL BY MAGNETICALLY INTENSIFIED ADSORPTION

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ABSTRACT

New nanocomposite adsorbent based on silica and iron(III) oxide, thermally treated at 200°C has been used for the arsenic(V) removal. Adsorbents were prepared by sol–gel method, the iron oxides being generated in situ during the sol–gel process. Nanocomposite materials thus obtained have a convenient porosity and nanosized iron oxide content. The sorption experiments have been carried out in a batch mode by using aqueous solutions containing 1000 ppm As. The influence of different experimental parameters

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on the adsorption capacity was investigated. The effect of magnetic field on the enhancement of adsorption process was also revealed.

INTRODUCTION

The problem of arsenic removal is very important because this metal exhibits a very high toxicity even at low concentrations and it is always present in wastewater.

There are many studies that present various adsorbent materials and their use to remove arsenic from wastewaters. It has been demonstrated^[1,2] that hydrous oxides of iron and aluminum have a high capacity to retain arsenic, thus playing a significant role in controlling arsenic presence and mobility in the environment.

The main purpose of this study is to find the optimum experimental conditions (pH, time, temperature, etc.) as well as the impact of magnetic field on enhancing adsorption of arsenic on different adsorbent materials synthesized by authors. Nanocomposite adsorbent materials doped with hydrous oxides of iron were used. A new chelating resin of iminodiacetate type, preloaded with iron(III), was also studied for arsenic adsorption. A comparison between arsenic adsorption capabilities of these new nanocomposite adsorbent materials and iminodiacetate resin will be presented.

EXPERIMENTAL

Adsorbents

As adsorbents, an iron-loaded iminodiacetate chelating resin and a composite material prepared by authors were used.

The preparation and the characterization of these new nanocomposite adsorbents has been presented elsewhere.^[1-3] The material used throughout the experiments was annealed for several hours at 200°C.

Analysis

Arsenic and iron in water were estimated by ICP technique using an ARL model 3410 with minitorch (Valencia, CA) and by AAS using an AAS 6 Vario model with flame (Karl Heinz, Jena, Germany). The emission lines used for this analysis were 197.262 and 259.94 nm for iron.

**As(V) REMOVAL****3695****Reagents**

Sodium arsenate ($\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$) and iron chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) were ACS grade from Aldrich.

Sorption Experiments

Weighed amounts of resin or composite nanomaterials were shaken together with measured volumes of arsenate solutions, 1000 ppm total As, in tightly stoppered tubes, for 15 hr (1 night) or 40 hr (nanocomposite materials), at room temperature. Suspensions were allowed to settle, centrifuged, and supernatant analyzed for As (total) and iron (total). pH was checked prior to sorption and after sorption. The equilibrium sorption was calculated from the residual concentration of As in the supernatant equilibrated solution.

For magnetically enhanced adsorption experiments a cylindrical magnet (purchased locally) with bore size of 2 cm and a length of 20 cm was used. Magnetic flux density inside the bore was measured with Model DG 4080 Gaussmeter (Laboratorio Elettrofizico, Milan, Italy) and shown to be in the range of 100 mT. Weighed amounts of composite materials were put in contact with measured volumes of arsenate solutions, 1000 ppm total As, in tightly stoppered tubes, for 40 hr, at room temperature. Tubes were vigorously shaken from time to time and then placed back in the magnet.

RESULTS AND DISCUSSIONS**Kinetic Considerations**

The sorption of arsenic on the nanocomposite material was measured as function of time at pH 2.6 and room temperature. Samples were continuously shaken from 1 to 96 hr on an orbital shaker, to check for optimum contact time and sorption kinetics. The sorption data are plotted in Fig. 1, and it can be observed that the adsorption capacity is almost constant after 40 hr. Therefore, all experiments involving nanocomposite materials have been considered as equilibrated after 40 hr. Similar sorption kinetic experiments were pursued for iron-loaded resin and it was found that the system reaches equilibrium after 10 hr. Therefore, all experiments involving iron-loaded resin have been considered as equilibrated after 10 hr (or overnight).

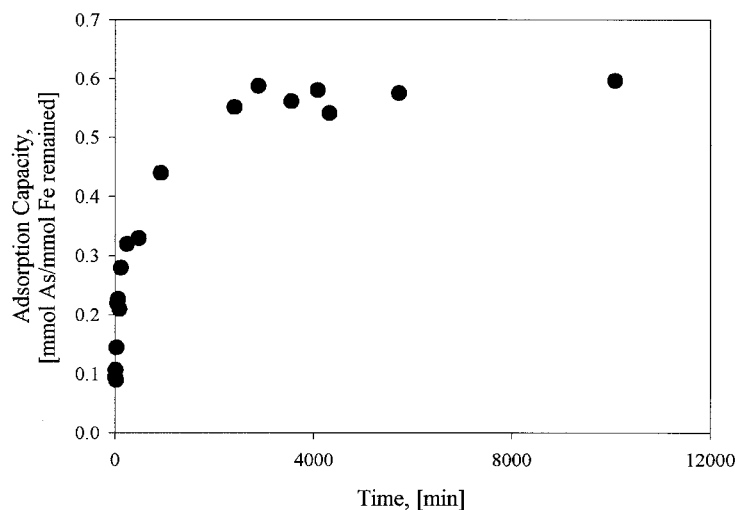


Figure 1. Kinetic study of As(V) adsorption.

Effect of pH on Sorption Capacity

The pH of solutions was increased or decreased using 1 M NaOH or HCl, respectively. All sorption experiments were carried out on an orbital shaker, samples being kept in contact overnight for the iron-loaded chelating resin and for 40 hr for the nanocomposite materials. Thus adsorption capacity was calculated for this equilibration time. The results are presented in Fig. 2.

During the experiments the pH of the supernatant solution decreased. Data considered as reference for adsorption equilibrium are those at the end of the adsorption equilibrium. Table 1 presents changes found.

The sorption of As(V) at equilibrium, with both types of adsorbents, is sharply influenced by pH, with the sorption drastically decreasing at low pH. The maximum sorption for nanomaterial adsorbents is observed at pH 2.6 when the adsorption capacity remained in resin is 0.58 mmol As/mmol Fe, while iron chelating resin maximum sorption shifts at even more acidic pH (pH 1.70), when the adsorption capacity remained in resin is 0.46 mmol As/mmol Fe.

The uptake of iron from adsorbents was also determined and the results are shown in Fig. 3a for iron-loaded chelating resin and in Fig. 3b for nanocomposite material, at each pH value. Pattern observed is similar amongst the two materials and it can be observed that at a very acidic pH (less than 1.50 for resin, and less

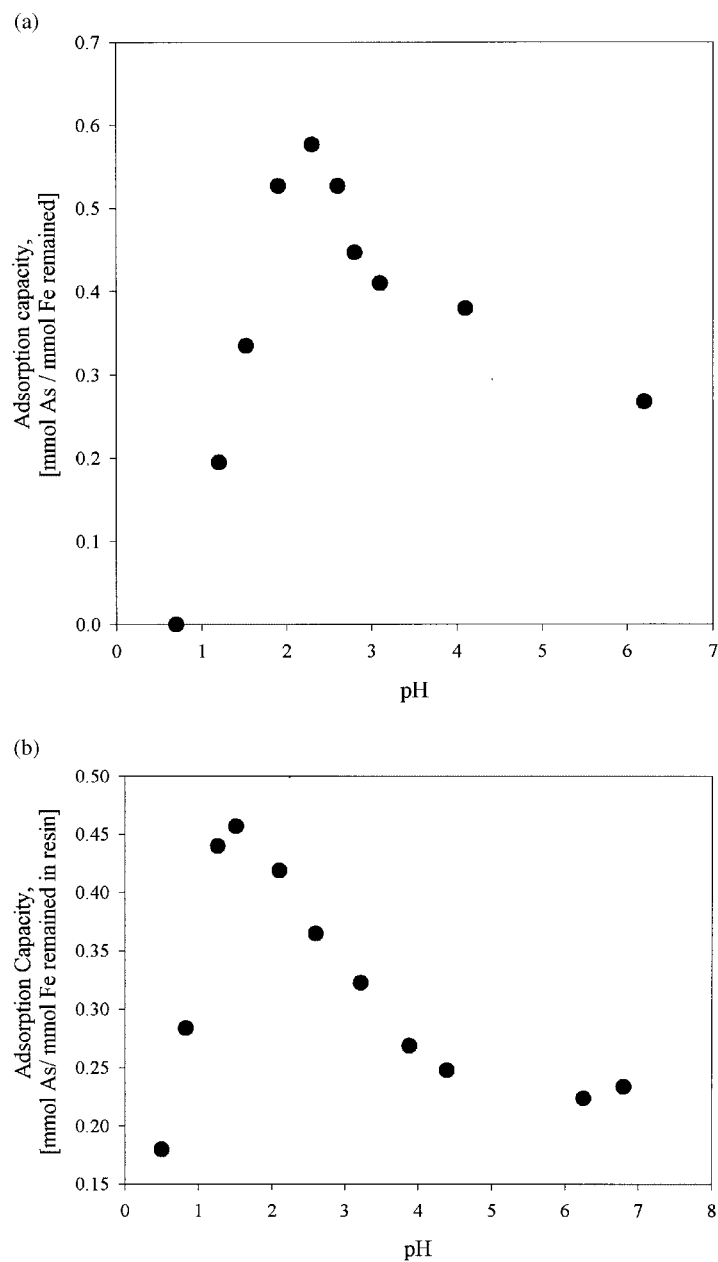
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Figure 2. (a) Sorption capacity of nanocomposite material as a function of pH. (b) Sorption capacity of iron-loaded resin as a function of pH.

Table 1. pH Changes Before and After Adsorption of As(V) on Nanocomposite Adsorbent

Sample	Initial pH	Final pH
1	0.6	0.6
2	1.1	1.1
3	1.5	1.5
4	2.0	1.9
5	2.5	2.3
6	3.0	2.6
7	3.5	2.6
8	5.0	2.8
9	6.0	3.1
10	7.0	6.0
11	8.3	6.2
12	1.5	1.5
13	3.0	2.6
14	7.0	6.0
15	11.0	6.9

than 2.00 for nanocomposites) iron uptake is quite important due to protons competition for the active sites of the adsorbent. Nevertheless, for pH values where arsenic adsorption is highest, this competition decreases and iron uptake from adsorbent becomes very small. Figure 4 exemplifies this correlation between As adsorption and iron uptake. It can be seen that there is a window where As adsorption is maximum but this does not rule out the interaction between As and adsorbent matrix itself in explaining this phenomena.

Effect of Magnetic Field on Sorption Capacity

Figure 5 shows the influence of magnetic field on the As(V) adsorption. It can be observed that for some pH values the adsorption of arsenic on nanomaterials is enhanced when samples are subjected to magnetic field exposure for 40 hr. Also, worth noting is the fact that magnetically influenced adsorption of arsenic is lower for acidic than for neutral pH. New sets of experiments have been designed by the authors for better understanding of the control parameters for magnetically enhanced adsorption of arsenic and mechanisms involved.

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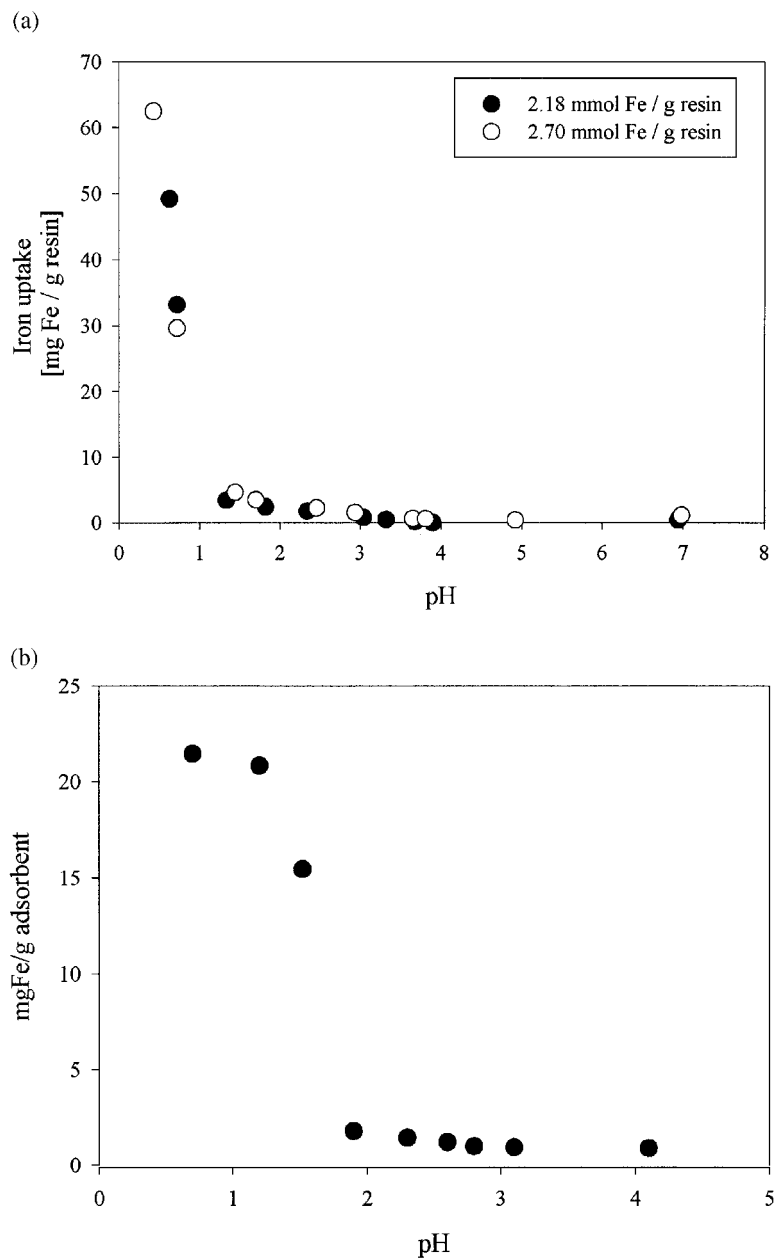


Figure 3. (a) Iron uptake as a function of pH for iron-loaded resin for two different values of iron loadings. (b) Iron uptake as a function of pH for nanocomposite material.

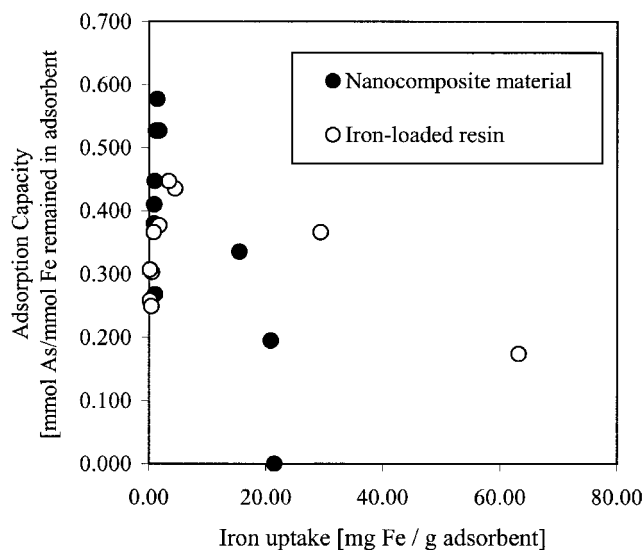


Figure 4. As(V) sorption capacity as a function of iron uptake.

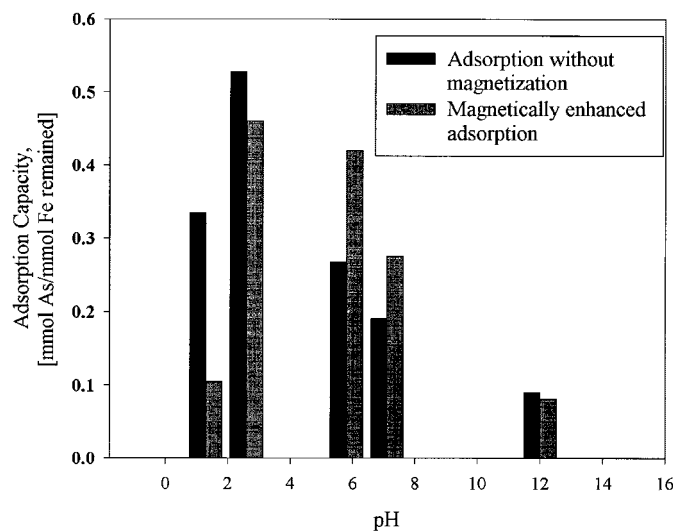


Figure 5. Effect of magnetic field on sorption of As(V) on nanocomposite material.



CONCLUSIONS

The main purpose of this study was to find the optimum experimental conditions for adsorption of arsenic on different adsorbent materials. A nanocomposite adsorbent prepared by the authors and doped with hydrous oxides of iron has been tested against an iron-loaded iminodiacetate chelating resin. Higher As(V) adsorption capacity is shown by the nanocomposites when compared with an iminodiacetate resin. The adsorption of As(V) on nanocomposites adsorbents presents a maximum at pH 2.6, while Fe(III)-loaded iminodiacetate resin presents a maximum at pH 1.5.

When subjected to magnetic field exposure the adsorption of As(V) on nanocomposite materials prepared by the authors changes in a complex way: in acidic conditions the sorption capacity decreases when compared with samples not subjected to magnetic exposure, while at neutral pH the effect of magnetically intensified adsorption is revealed. New nanocomposite adsorbents including ferrites are under preparation to evaluate magnetically enhanced adsorption of arsenic.

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