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Original Article

Removal Efficiency of Electrochemical Process by Iron Oxide Nanoparticles Impregnated on Rod Carbon for Arsenite Anions in a High Ionic Strength Solution

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Abstract

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The entry of heavy metals (e.g., arsenite anions) into water sources due to industrial and mining activities is considered a serious problem for the environment. Therefore, arsenite removal from polluted water has attracted the attention of researchers due to its toxic effects on human health. In the present study, the efficiency of the electrochemical (EC) purification of arsenite anions in a high ionic strength solution was investigated using a carbon electrode impregnated with iron oxide nanoparticles (NPs). These NPs were synthesized by the co-precipitation method and immediately impregnated on the carbon rod. Experiments were performed by two electrodes (carbon and carbon impregnated with NP electrode) in a 50 mL EC reactor. The effect of different parameters such as electrical current (0.1, 0.3, 0.5, 0.7 and 0.9 A), EC time (2, 5, 10, 15 and 20 minutes), pH (3, 5, 7, 9 and 11), and the initial concentrations of arsenite anion (2, 5, 10, 15 and 20 mg/L) was investigated on the removal efficiency to achieve the highest removal of arsenite anions. Arsenite anions (10 mg/L) were completely removed from the aqueous solution with an ionic strength of 0.141 M at an electrical current of 0.7 A, a pH of 8, and an EC time of 20 minutes. In addition, pH was the most effective parameter in removing arsenite anions from aqueous solution in the EC method. According to the results, EC treatment using an electrode impregnated with iron oxide NPs is highly efficient in removing arsenite anions from the contaminated water.

Keywords: Arsenite anions, Electrochemical process, Iron oxide nanoparticles, High ionic strength, Removal efficiency

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1. Introduction

At present, access to healthy water sources is difficult due to the industrialization of societies, rapid population growth, and reduced rainfall. The most important factor in the pollution of water sources is the entry of heavy and toxic elements into the environment caused by industrial and mining activities (1). Heavy metals in industrial effluents after discharge into the environment are serious threats to surface and groundwater sources. Arsenic usually exists in the form of arsenate (AsO_4^{3-}) or arsenite (AsO_3^{3-}) in water sources, where the arsenite anion is about 60 times more toxic and dangerous than the arsenate anion (2). Arsenic enters surface and groundwater sources naturally (due to weathering of minerals and rock and soil erosion) or due to human activities such as mining and the like (3, 4). The presence of arsenic in the water of some regions of the world can be considered a chronic problem, among which

we can mention East and Southeast Asian countries such as Bangladesh, Thailand, and Malaysia, as well as Western America, Chile, and Argentina (5). In Iran, in areas such as Sistan and Baluchistan, Kurdistan, and East Azerbaijan, the amount of arsenic in water sources is higher than the standard limit (6, 7). Contamination of drinking water with heavy metals, especially arsenite anions, is a problem that has endangered the health of many people (8). Depending on the arsenic concentration and contact time, arsenic can lead to acute (vomiting and tingling of hands and feet) and chronic (cardiovascular diseases, immune, nervous, and reproductive system disorders) poisoning (9). Due to the harmful effects of arsenic on humans, the Environmental Protection Agency has decreased the maximum concentration of arsenic in drinking water from 50 to 10 μ g/L (10, 11).

Different methods such as coagulation and flocculation,

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ion exchange, membrane processes, chemical precipitation, and surface adsorption have been used to remove arsenic anions from aqueous solutions (9, 12, 13). Contrary to their widespread use, the above-mentioned methods have many problems such as high maintenance costs, excessive consumption of chemicals, and sludge management (14, 15). In addition, most arsenic removal technologies are effective in removing arsenate and have a low capacity in removing arsenite because arsenite form is not normally hydrolyzed up to a pH rate of 9, and its solubility is low. In addition, arsenate has a greater affinity for inorganic compounds or has adsorption on solid surfaces such as iron oxide. Therefore, to completely remove arsenic compounds, first, the oxidation step must be performed to convert arsenite into arsenate.

In recent years, electrochemical (EC) processes as an advanced and efficient process for the treatment of industrial wastewater have attracted the attention of many chemical and environmental health researchers (16, 17). The base of the EC process is to apply direct current to the electrodes of an EC cell and adsorb ions along with oxidation and reduction reactions on the surface of the electrodes. The most important advantages of this method are operation at ambient temperature and pressure, low use of chemicals and thus less sludge production, the ability to adapt to changes in the composition and initial flow rate, and the ability to remove a wide range of pollutants (18).

Iron oxide has been extended to remove pollutants from water and wastewater and has an important role in a variety of industrial applications. Several kinds of iron oxide have been studied for arsenic anion adsorption from contaminated solutions. Some types of iron oxide are used as the adsorbents of arsenic anions in the solution, including goethite, magnetite, hematite, siderite, limonite, and ferrihydrite. The most widespread types of iron oxide are magnetite (Fe₃O₄) and ferrihydrite (FeOOH). The results of the study by Thirunavukkarasu et al indicated that arsenite is more strongly adsorbed on ferrihydrite than arsenate in the pH range of 3-11 at a high initial arsenic concentration (19).

Thus, this study was conducted to determine EC efficiency for the treatment of arsenite anions in a high ionic strength solution using a rod carbon electrode impregnated with iron oxide NPs. The influences of electrical current, EC time, solution pH, and initial arsenite concentration were studied in batch experiments.

2. Materials and Methods

This experimental study was conducted on a semi-scale in the Chemistry Laboratory of Yazd Health Faculty. First, a rod carbon electrode was modified with iron oxide by the co-precipitation method, and an EC reactor was designed to investigate the arsenite removal. After variable determination and according to study objectives, EC examinations were performed to obtain high removal efficiency by the EC reactor. The steps of the study are specified as follows:

2.1. Synthesis of iron oxide NPs

Iron oxide NPs were synthesized by the co-precipitation method for impregnation on the rod carbon electrode. For this purpose, 0.1 M solution of iron (III) by dissolving 5.35 g of FeCl₃, and 0.1 M solution of iron (II) by dissolving 8.10 g of FeCl₂.4H₂O were separately prepared in 500 mL of water. Then, its temperature was raised to 80°C, and two solutions were mixed together. Finally, 0.1 M of ammonium hydroxide solution was slowly added dropwise for 20 minutes until the pH of the solution reached about 9. The obtained solution was placed in an ultrasonic bath for 10 minutes (20, 21).

2.2. Impregnation of NPs on Rod Carbon

The used electrode in this study was a rod carbon of the applied batteries with a length of 4.5 cm and a diameter of 0.5 cm. The rod carbon was washed with soapy water and cleaned with a plastic brush. Then, its surface was smoothed and polished with the 3% HNO₃ solution. The polished rod carbon was immersed in the freshly synthesized iron oxide NP solution for 2 hours on the shaker. The formation of iron oxide NPs on the rod carbon was controlled by the scanning electron microscope (SEM). The rod carbon impregnated with iron oxide NPs was washed 3 times with distilled water and ethanol (70:30) and dried at laboratory temperature, and this electrode was kept in a desiccator for further EC experiments. The schematic of the iron oxide NP synthesis process and impregnation on rod carbon is illustrated in Fig. 1.

2.3. EC Process

To perform the experiments, a cubic reactor $2 \times 2 \times 5$ cm with a volume of 50 mL was employed with two carbon rods (one without NPs and the other impregnated with iron oxide NPs) at a distance of 3 cm, connected to the power source as cathode and anode. Fig. 2 depicts the schematic of the reactor:

To prepare the desired solutions of arsenite in the



Fig. 1. The Schematic of Synthesis and Impregnation of Nanoparticles on Rod Carbon

laboratory, first, the mother solution of arsenite was prepared from sodium arsenite (Na, HASO,). The desired concentrations were daily prepared by diluting the mother solution with distilled water (6, 22). All experiments were performed at a temperature of $20 \pm 2^{\circ}$ C. To increase the ionic strength and electrical conductivity of the solution, 2 mL of Na₂SO₄ electrolyte with a concentration of 10 g/L was added to the reactor. Next, 0.1 M of the H₂SO₄ or M NaOH solution was also used to adjust the pH. The effect of different parameters, including electrical current (0.3, 0.5, 0.7, & 0.9 A), EC time (2, 5, 10, 15, 20 minutes), pH (3, 5, 7, 9, & 11), and the initial concentration of arsenic (2, 5, 10, 15, 20 mg/L), on the removal efficiency of arsenite anions in high ionic strength solutions (0.141 M) was determined as a factor at any time in a batch reactor. At the end of each test, the electrodes were washed with distilled water, placed in a 1% hydrochloric acid solution for 20 minutes, then rinsed well, and dried. The experiments were repeated twice, and the average results were reported at the end. To measure the residual arsenic concentration in the water after the EC process, the solution was centrifuged and measured using an atomic absorption spectrometer at a wavelength of 193.7 nm by an acetylenenitrous oxide flame (23).

2.4. Data Analysis

The removal efficiency of arsenic in the solution in each step was calculated by Eq. (1) as follows (24):

$$\% R = \frac{C_0 - C}{C} \times 100$$
 (1)

The adsorbed mass in the EC process was estimated by Faraday Eq. (2) as (25):

$$m_{cal} = \frac{1.t.E}{96485} \tag{2}$$

The efficiency of the EC cell was obtained by Eq. (3):





% eff =
$$\frac{(C_0 - C) \cdot V_R \cdot E}{m_{cal}}$$
 (3)

The energy consumption of the EC process (kWh/kg analyte) was computed by Eq. (4) as follows (26):

Energy consumption =
$$\frac{VIt}{3600} \times \frac{10^3}{(C_0 - C) \times V_R}$$
 (4)

where C₀ and C are the initial and residual concentrations (arsenite anions) in the solution (mg/L), and I is the applied current (A). In addition, t, E, and V_p represent EC time (s), the equivalent of pollutant (arsenite anions), and the reactor volume (L), respectively. Further, m_{cal} and V denote the calculated removal mass active specie (arsenite anions) and the applied voltage (V), respectively.

3. Results and Discussion

The morphology of the electrode surface was considered by SEM (Fig. 3). The SEM image illustrated that the whole surface of the rod carbon electrode had been coated with iron oxide NPs.

3.1. The Effect of Arsenite Anion Concentration

To determine the effect of arsenite anion concentration, experiments were performed at an electrical current of 0.5 A, initial pH solution, time interval of 10 minutes, and different concentrations of 2, 5, 10, 15, and 20 mg/L. Based



Fig. 3. The SEM of the Surface of a Rod Carbon Electrode Impregnated With Iron Oxide. Note. SEM: Scanning Electron Microscope





on the obtained results (Fig. 4), first, the removal efficiency increased up to a concentration of 10 mg/L (76.6%). By increasing the initial concentration of arsenite anions from 10 to 20 mg/L, the removal efficiency decreased from 76% to 49%.

As shown in Fig. 4, the maximum removal efficiency is at the concentration of 10 mg/L of arsenite anions. It is clear that in the EC process, the removal efficiency decreases under the constant conditions of electrical current and EC time when increasing the initial concentration of the substance. In a constant electrical current and voltage, the speed of movement of charged particles (anions and cations) toward the electrodes is constant and the EC process can adsorb and remove a certain amount of ions in a constant time. Therefore, if the initial concentration of the ionic substance in the solution increases, the amount of anions and cations in the EC reactor increases and the time is not enough to remove excess ions; because of the increase of the initial concentration of the substance, not all ions participate in the EC process, and as a result, the efficiency of substance removal represents a reduction (27). Hence, in the present study, it was also observed that removal efficiency decreased with an increase in the concentration of arsenite anions. In the study conducted by Argiani et al, removal efficiency decreased with increasing the aniline concentration (28). for the removal of phosphate during the EC process using iron and aluminum electrodes, İrdemez et al found that removal efficiency decreased with an increase in the initial concentration of phosphate (29). Thus, the effective concentration to remove arsenite anions in these conditions was 10 mg/L.

3.2. The Effect of pH

According to the initial arsenite concentration determined in the previous step, to explore the effect of pH on the removal efficiency of arsenite anions, experiments were performed with a concentration of 10 mg/L, an electrical current of 0.5 A, and a time interval of 10 minutes at different pH rates of 3, 5, 7, 9, and 11 (Fig. 5). Based on the results, the removal efficiency was low at highly acidic and basic pH rates, and the highest removal efficiency was obtained at a pH rate of 8.

According to data in Fig. 5, the highest removal



 $C_0 = 10 \text{ mg/L}, I = 0.5 \text{ A}, t = 10 \text{ min}$







efficiency was 85% (pH = 8). Moreover, a relative increase was observed in removal efficiency at the pH range of 5-8. The pH of the solution significantly affects the speciation of arsenic in the solution, thus the hydrolyzed arsenite species is a function of pH. Arsenite is in the form of neutral H₃AsO₃ at pH < 9; when pH increases to 9-12, 12-13, and>13, these particles change from neutral to mono-anionic form H₂AsO₃, di-anionic form HAsO₃²⁻ , and AsO₃³⁻, respectively. Therefore, the capacity of the chemical adsorption of arsenic on iron oxide surfaces varies with a change in solution pH (2). In this study, as the pH increased, the effective anions in the solution increased, leading to an increase in the removal percentage of arsenite anions. In addition, at pH>6, the electrode impregnated with iron oxide NPs has a surface charge and can adsorb with the formation of a complex by arsenite anions. Previous studies also demonstrated that As(III) adsorption on these materials had a maximum pH value of around 8.5, and pH had a direct effect on removal efficiency due to its effect on the electrode surface and arsenic form (30). Accordingly, the arsenite removal from polluted water increased with increasing pH; other researchers attributed it to the difference between HAsO₃²⁻ dissociation constants (31, 32).

3.3. The Effect of Electrochemical Time

To investigate the effect of the EC time on the removal efficiency of arsenite anions, experiments were conducted at a concentration of 10 mg/L, electrical current of 0.5 A, a pH of 8, and time intervals of 2, 5, 10, 15, and 20 minutes. At constant electrical current, the removal efficiency increased with increasing time so that it reached 89% at 20 minutes (Fig. 6).

Based on Fig. 6, the removal efficiency increased with the increase of the EC time. In the EC process, with increasing time, further electric charge passes through the solution, causing the transfer of more charged particles from the solution to the surface of the electrode while increasing removal efficiency. In the current study, the appropriate reaction time for removing arsenite anions was 20 minutes. The results obtained from the removal of azo dye by the EC process also indicated that by increasing the reaction time from 2 to 14 minutes, the removal efficiency increased to 98% (33). Likewise, Arqiani et al reported that the removal efficiency of aniline increased with an increase in the reaction time (28).

3.4. The Effect of Electrical Current

To determine the effect of the electrical current on the removal efficiency of arsenite anions, the experiments were performed at currents of 0.1, 0.2, 0.3, 0.5, and 0.9 A at the initial concentration of arsenite anions, pH, and time constant (Fig. 7). According to the findings, the maximum removal efficiency (99%) was obtained at an electrical current of 0.7 A.

Fig. 7 shows that the removal efficiency of arsenite anions increases with increasing electrical current. Thus, the adsorption of arsenite anions in the anode was significant in all studied currents. Considering the increase in the removal efficiency with increasing in electrical current in the EC process, the transfer rate and the number of charged particles from the solution to the surface of electrodes, which are responsible for pollutant removal, increased with an increase in the electrical current and could increase in the adsorption of arsenite anions on the electrode impregnated with NPs.

3.5. EC Mechanism

The adsorption mechanism of arsenic on iron oxide has been considered by several researchers on the samples of arsenic anion removal by iron oxide using ultraviolet photoelectron spectroscopy and infrared spectroscopy techniques and represented that arsenic anions form monodentate and binuclear bidentate complexes with hydroxyl groups on the iron oxide surface (12-15). Arsenic anions replace one or two OH groups on iron oxide surfaces to form Fe-O-As(OH)-O and Fe-O-AsO(OH)-O-Fe mono or dinuclear bridge complex (34).

The complex form is the most abundant species on iron oxide surfaces and is chemically stable. Therefore, the proposed mechanism involves two steps, including the formation of a monodentate surface complex with a high adsorption rate as covalent bonds between the adsorbed ion and the functional group (-OH) of the surface (35). The anodic oxidation reaction of the arsenite ion and its





conversion to an arsenate ion leads to the formation of an internal bidentate complex and increases the affinity of this ion on the surface of iron oxide.

Based on the above discussion, the mechanism of the removal of As(III) by iron oxide surface is adsorption along with the oxidation approach. The whole process can be summarized by reaction equations (5-7).

Anode

 $C-O-Fe-OH+H_2AsO_3^{-} \Rightarrow C-O-Fe-O-As(OH)_2 + HO^{-} adsorption (5)$ $C-O-Fe-O-As(OH)_2 + H_2O \Rightarrow C-O-Fe-O-AsO_3H_2 + 2H^+ + 2e \text{ oxidation (6)}$

Cathode

$$SO_4^{2-} + 2H^+ + 2e \rightarrow SO_2^{2-} + H_2O$$
 reduction (7)

where (C-O-Fe-O-) represents the adsorption site on the iron oxide adsorbent surface. In addition, C—O-Fe-O-As(O₂H) and C—O-Fe-O-AsO₃H₂ denote arsenic anions (As(III) and As(V)) adsorption on the surface, respectively. This mechanism of adsorption and oxidation can well explain the higher and faster adsorption of As(III) by the EC process.

3.6. Consumed Energy and Adsorbed Mass

Electrical energy consumption is a beneficial factor in the EC process. In such processes, energy consumption is related by several important parameters, including average applied current and voltage, EC time, and mass value reacted in the reactor. Using Eq. (4), in optimum conditions (electrical current of 0.7 A, time interval of 20 minutes, and constant voltage of 11 V), the energy consumption of the unit removal of arsenite ions was 52.3 kWh/kg. Based on electrical energy consumption at optimum conditions, the use of an EC process is a suitable process in the removal of arsenite contaminant from an aqueous solution.

Faraday's law predicts the mass of a species that can be precipitated, reduced, or oxidized by the amount of electricity that passes through the solution (36). The adsorbed mass on the anode was 35.7 mg, which is calculated by Faraday's equation (2). The mass of arsenite anions in these deposits is lower than the calculated theoretical values. Using Eq. (3), it was possible to obtain the electric efficiency of the EC process for arsenite removal. The electrical efficiency of the EC process was 75.6%, which is a good practical approach for arsenite anion removal. The obtained result in the present study determined that EC treatment with an electrode impregnated with iron oxide NPs had an acceptable performance for removing arsenite anions.

4. Conclusion

It was found that arsenic anions can be efficiently and rapidly removed from the aqueous solution in the modified EC process. The arsenite anions migrated to the anode by the electrical positive potential, followed by the adsorption and oxidation by the iron oxide on the rod carbon electrode of the anode. The capability of the EC process was closely related to the current, pH, and initial arsenite concentration of the solution. The results indicated that EC efficiency increased with increasing the electrical current, EC time, and proper pH. Under optimal conditions, with a current of 0.7 A, a time of 20 minutes, pH 8, and an initial concentration of arsenite anions of 10 mg/L, 99% of arsenite anions could be removed from the solution. The EC process was used for the removal of arsenite anions to decrease the toxicity of arsenic. Meanwhile, with low energy consumption and eco-friendly conditions, the modified carbon electrode develops the EC process as a promising technology to remove arsenite anions.

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Competing Interests

The authors declare that they have no conflict of interests.

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