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Arsenic removal via electrocoagulation from heavy metal contaminated groundwater in La Comarca Lagunera México

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Abstract

Arsenic contamination is an enormous worldwide problem. A large number of people dwelling in Comarca Lagunera, situated in the central part of northern México, use well water with arsenic in excess of the water standard regulated by the Secretary of Environment and Natural Resources of México (SEMARNAT), to be suitable for human health. Individuals with lifetime exposure to arsenic develop the classic symptoms of arsenic poisoning. Among several options available for removal of arsenic from well water, electrocoagulation (EC) is a very promising electrochemical treatment technique that does not require the addition of chemicals or regeneration. First, this study will provide an introduction to the fundamental concepts of the EC method. In this study, powder X-ray diffraction, scanning electron microscopy, transmission Mössbauer spectroscopy and Fourier transform infrared spectroscopy were used to characterize the solid products formed at iron electrodes during the EC process. The results suggest that magnetite particles and amorphous iron oxyhydroxides present in the EC products remove arsenic(III) and arsenic(V) with an efficiency of more than 99% from groundwater in a field pilot scale study. © 2005 Elsevier B.V. All rights reserved.

Keywords: Arsenic removal; Electrocoagulation; Drinking water; Iron electrodes

1. Introduction

The occurrence of arsenic in groundwater of La Comarca Lagunera is a major problem for México. The contamination is on a large scale and 30 years have passed since arsenic came into picture [1–3]. Arsenic concentration has been found in well water from several communities ranging from 0.24 to 1.0 ppm. Most of this region is reputed to have substrata rich in arsenic. Therefore, the arsenic in ground water is largely the result of minerals dissolving naturally from weathered rocks and soils, which is known to produce high-arsenic levels in well water. Other possible sources of contamination can be referred to, for example, the use of organoarsenical pesticides in the cotton fields and also millions of tons of slag or fallout from roaster off-gas emissions of one lead smelter in 1945 [1-5]. In La Comarca Lagunera México, thousands of people have already contracted the symptoms of arsenic poisoning and two million are at risk of arsenic contamination from drinking well water. Effects, such as changes on skin pigmentation, gastrointestinal disturbances, neurological changes, lung cancer and muscular weakness, characterize arsenic poisoning in humans. Arsenic toxicity has no known effective medicine for treatment, although drinking arsenic-free water can help the affected people to reduce or remove of the symptoms of arsenic toxicity and protect the health and well-being of rural people living in acute problem areas of México. Socio-economic conditions of México demand low-cost as well as efficient treatment

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Table 1	
A comparison of main arsenic removal technologies	[9-15]

Technologies	Advantages	Disadvantages	Removal (%)
Oxidation/precipitation			
Air oxidation	Relatively simple, low-cost but slow process In situ arsenic removal Also oxidizes other inorganic and	Mainly removes arsenic (V) and accelerate the oxidation process	80
Chemical oxidation	organic constituents in water Oxidizes other impurities and kills microbes Relatively simple and rapid process Minimum residual mass	Efficient control of the pH and oxidation step is needed	90
Coagulation/co-precipitation			
Alum coagulation	Durable powder chemicals are available Relatively low capital cost and simple in operation	Produces toxic sludges Low removal of arsenic	90
	Effective over a wider range of pH.	Pre-oxidation may be required	
Iron coagulation	Common chemicals are available More efficient than alum coagulation on weigh basis	Medium removal of As(III) Sedimentation and filtration needed	94.5
Lime softening	Most common chemicals are available commercially	Readjustment of pH is required	91
Sorption techniques			
Activated alumina	Relatively well known and commercially available	Needs replacement after four to five regeneration	88
Iron coated sand	Expected to be cheap No regeneration is required Remove both As(III) and As(V)	Yet to be standardized 9 Produces toxic solid waste	
Ion exchange resin	Well-defined medium and capacity The process is less dependent on pH of water	High cost medium Requires high-tech operation and maintenance	87
	Exclusive ion specific resin to remove arsenic	Regeneration creates a sludge disposal problem As(III) is difficult to remove Life of resins	
Membrane techniques			
Nanofiltration	Well-defined and high-removal efficiency	Very high-capital cost 95 Pre-conditioning High water rejection	
Reverse osmosis	No toxic solid waste is produced	High tech operation and maintenance	96
Electrodialysis	Capable of removal of other contaminants	Toxic wastewater produced 95	

systems that could be implemented in the rural areas or cities.

The main arsenic species present in natural waters are arsenate ions AsO_4^{-3} (oxidation state V) and arsenite ions H_3AsO_3 , $H_2AsO_3^{-}$ and $HAsO_3^{-2}$ (oxidation state III). However, As(V) ions are most prevalent in oxygenated water while As(III) is found in anaerobic conditions, like in well water or in groundwater. The literature on arsenic concludes that the most common valence states of arsenic in well water are As(V) or arsenate, and As(III) or arsenite. In the pH range of 4–10, the trivalent As(III) species are neutral in charge, while the As(V) are negatively charged. The removal efficiency for arsenic is often much lower for As(III) than for As(V) by using anyone of the conventional technologies for elimination of arsenic from water [6,7], so either elevation of pH [7] or oxidation of arsenite to arsenate [8] is considered a prerequisite for any treatment method to be efficient. Therefore, treatment of arsenic contaminated well water through appropriate technology is one option to mitigate the arsenic problem.

Various technologies have been used for removing arsenic from groundwater. The most commonly used technologies include co-precipitation with alum or iron, adsorption onto coagulated floc, ion exchange resin, reverse osmosis and membrane techniques. A review of these technologies along with their distinct advantages and disadvantages is shown in Table 1 [9–15]. The major disadvantage of most of the techniques presented in Table 1 is that they are unable to remove As(III) efficiently. In addition, none of these processes for the arsenic removal use electrocoagulation with air injection. It is intended to update the process development in arsenic removal and consider the economic factors involved in implementing lower drinking water standards for arsenic. These drawbacks have forced municipalities and various industries to search for effective alternative treatment technologies for arsenic removal, ideally by electrochemical methods. Electrocoagulation (EC) is one of the most efficient technologies for removal of both As(III) and As(V) from contaminated water. As removal is rapid with higher current densities [15,16]. Balasubramanian and Madhavan [15] reported about their EC experiments without air injection over a wide range of operational conditions and found that the efficient removal of arsenic takes about 7 h and the rate of arsenic removal for this technique depends on the initial arsenic concentration.

Therefore, the purpose of this research is to investigate the use of modified EC process with air injection to enhance the removal of highly water soluble As(III) and As(V) compounds from groundwater. Electrocoagulation with air injection integrates the high-arsenic removal efficiency of EC with the advantage of a shortened reactive retention time and minimum residual mass, and thereby it reduces the operating costs. This has received very little attention, although this process has the potential to eliminate the disadvantages of the classical treatment techniques. A review of the literature reveals that the potential of EC with air injection as an alternative to the conventional treatment processes has not yet been adequately explored due to technical and economical reasons [17]. Although also, the sludge from this process generates invaluable materials for other technologies such as Fe₃AsGa for semiconductor manufacturing and gallium arsenide (GaAs/Ge) for solar cell production [18]. A recent study [19] shows that EC can be combined with magnetic seeding to produce a magnetic aggregate that can be efficiently separated by high-gradient magnetic separation (HGMS) in-line with the EC reactor.

2. Electrocoagulation characteristics

Electrocoagulation has been known as an electrochemical phenomenon for the last century. It has been employed previously for treating many types of wastewater with varying degrees of success. However, most studies have focused on the efficiency of waste removal without exploring the fundamental mechanisms involved in the electrocoagulation process [20]. This electrochemical method of removal requires very small quantities of salt addition to increase the conductivity of the solution, and the maintenance and operation of the electrocoagulation cells is relatively simple. Since 1970, this technology has become increasingly popular around the world for treatment of industrial wastewater containing metals [20]. Electrocoagulation processes offer significant potential for removing soluble ionic species from solution, particularly heavy metals [21]. EC operating conditions are highly dependent on the chemistry of the aqueous medium, especially conductivity and pH. Other important characteristics such as particle size, type of electrodes, retention time between plates, plate spacing and chemical constituent concentrations dictate the operating parameters of the process [22]. The EC process operates on the principle that the cations produced electrolytically from iron and/or aluminum anodes enhance the coagulation of contaminants from an aqueous medium. Electrophoretic motion tends to concentrate negatively charged particles in the region of the anode and positively charged ions in the region of the cathode. The consumable, or sacrificial, metal anodes are used to continuously produce polyvalent metal cations in the vicinity of the anode. These cations neutralize the negative charge of the particles carried toward the anodes by electrophoretic motion, thereby facilitating coagulation. In the flowing EC techniques, the production of polyvalent cations from the oxidation of the sacrificial anodes (Fe and Al) and the electrolysis gases (H_2 and O_2) works in combination to flocculate the coagulant materials. Even inert electrodes, such as titanium and the passage of an alternating current have also been observed to remove metal ions from solutions and to initiate the coagulation of suspended solids. Fig. 1 illustrates the schematic diagram of the process. As mentioned above, gas bubbles produced by the electrolysis carry the pollutant to the top of the solution where it is concentrated, collected and removed. The removal mechanisms in EC may involve oxidation, reduction, decomposition, deposition, coagulation, absorption, adsorption, precipitation and flotation.

However, it is the reactions of the metal ions that enhance the formation of the coagulant. The metal cations of As(III) and As(V) react with the OH^- ions produced at the cathode during the evolution of hydrogen to yield both soluble and insoluble hydroxides that will react with or adsorb pollutants, respectively, from the solution and also contribute to coagulation by neutralizing the negatively charged colloidal particles that may be present at neutral or alkaline pH. This enables the particles to approach closely and agglomerate under the influence of van der Waals attractive forces. The chemical reactions that have been proposed to describe EC mecha-



Fig. 1. An illustration of the electrocoagulation mechanism.

nisms for the production of $H_{2(g)}$ and $OH^{-}_{(aq)}$ (cathode) and $H^{+}_{(aq)}$ (anode) [17] are:

When $M_{(s)} =$ Fe metal electrode:

$$Fe_{(s)} \rightarrow Fe^{2+} + 2e^-$$
 (1)

$$Fe^{2+} \rightarrow Fe^{3+} + e^{-}$$
 (2)

Anode:

$$a\mathbf{M}_{(s)} \to a\mathbf{M}^{n+} + an(\mathbf{e}^{-}) \tag{3}$$

$$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$$
 (4)

Cathode:

$$an(\mathrm{H}_{2}\mathrm{O}) + an(\mathrm{e}^{-}) \rightarrow \frac{an}{2}\mathrm{H}_{2} + an(\mathrm{OH}^{-})$$
 (5)

$$4\mathrm{H}^+ + 4\mathrm{e}^- \to 2\mathrm{H}_2 \tag{6}$$

Overall:

$$a\mathbf{M}_{(s)} + (2+an)\mathbf{H}_2\mathbf{O}$$

$$\rightarrow \left(2 + \frac{an}{2}\right)\mathbf{H}_2 + \mathbf{O}_2 + an(\mathbf{OH}^-) + a\mathbf{M}^{n+}$$
(7)

where constant *a* is a stoichiometric coefficient and *n* is number of electrons.

The pH of the medium rises as a result of this electrochemical process and the $Fe(OH)_{n(s)}$ formed remains in the aqueous stream as gelatinous suspension, which can remove the As(III) and As(V) from well waters, either by complexation or by electrostatic attraction followed by coagulation and flotation.

Generally, in the EC process, bipolar electrodes are used [23,24]. It has been reported that cells with bipolar electrodes, connected in series operating at relatively low current densities, produce iron or aluminum coagulant more effectively, more rapidly and more economically when compared to chemical coagulation.

3. Experimental

The electrocoagulation with air injection apparatus consisted of a flow-through reactor with parallel vertical electrodes, a peristaltic feed pump, an air pump and a power supply.

This EC system uses a modified EC process where the arsenic contaminated water is passed through a porous tube medium where air is injected before passing through the vertical electrodes in the EC cell. Seven vertical carbon steel electrodes were used as anode and cathode. The vertical geometry of the plates allowed the use of the gaseous O_2 and H_2 generated in the electrolysis of water to facilitate removal of the ferric and ferrous species associated with arsenic contaminants. The neighboring vertical electrodes (10.0 cm × 15.4 cm) were 6 mm apart and the internal volume of the reactor was 1200 ml. A variable transformer was



Fig. 2. A schematic diagram of the electrocoagulation reactor.

used to control the current and potential. The electrolyte solutions were prepared with deionized water with conductivity of 0.95 µS (Aldrich Chemical Co., +99.5% A.C.S. reagent, lot #DI02350AI) and the conductivity was controlled by adding 1 g of NaCl per liter of water (Fisher, 99.8% A.C.S. Certified, lot #995007). The arsenic contaminated well water was collected from groundwater wells in La Comarca Lagunera México. The pH was adjusted (as needed) using ~0.13 M NaOH solution prepared with NaOH pellets (EM, 97%, lot #36349739). Prior to each trial, the pH and conductivity of each solution were recorded. The aerated solution was pumped through the cell at a flow rate of about 600 ml/min and after 2.0-4.01 of well water solution were treated, the runs were stopped and the slurry pH and conductivity were recorded. The solutions and solids were then separated by filtration through cellulose filter paper. The sludge from the EC was dried either in an oven or under vacuum at room temperature. The samples were analyzed by atomic absorption spectroscopy (AAS, model Varian A 220). For the calibration curve of arsenic concentration, the arsenic standard solution (VW 4202-1) from Sigma-Aldrich was used. A schematic diagram of the cell is shown in Fig. 2.

4. Results and discussion

4.1. Arsenic removal by electrocoagulation with air injection

The $H_{2(g)}$ and $O_{2(g)}$ produced as a result of the redox reactions and the air injection may remove any suspended particles of iron coagulant impregnated with arsenic by Table 2 Summary of arsenic removal by EC analytical results by using with and without air injection methods

pH As (ppm) As removal $C_{\rm eff}$	(%)
(%)	
Feed solution 2.86 (in) 2.24 None None	e
Without air injection 6.36 (exit) 0.10 95.54 97.3	
With air injection 8.30 (exit) 0.005 99.77 102	

Note: The method of analysis was EPA 200.7 (which is an analytical method for identification of metals and trace elements by ICP/Atomic Emission Spectrometry).

electroflotation. Exact electrochemical reaction mechanisms involved are not yet completely understood. However, the EC of well water containing As(III) and As(V) produces an insoluble complex sludge, containing iron and arsenic oxides and hydroxides while leaving negligible or zero concentrations of arsenic from the well water. Table 2 shows a comparison of the results by using the same variables (40 V and 4 A) for removal of arsenic of the same feed solution without and with air injection for the laboratory EC cell. The increase in As removal efficiency with air injection in comparison to without air injection indeed justify the advantage of aeration in EC process for the removal of arsenic. An EPA report by Lowry and Lowry [25] on oxidation of As(III) by aeration and storage concluded that As(III) is not oxidized by oxygen on aeration or by dissolved oxygen and they suspected that removal of As(III) could be occurred due to its adsorption on precipitated iron oxide. Dissolved oxygen or aeration can enhance the oxidation process of iron. Kumar et al. [16] reported that As(III) removal mechanism in electrocoagulation with iron electrodes seems to be oxidation of As(III) to As(V) and surface complexation with iron hydroxides. Surface studies of the floc are essential for confirming these assumptions that will be carried out in our future research.

The current efficiency (C_{eff}) was calculated using the following equation:

$$C_{\rm eff} = \frac{M_{\rm EXP}}{M_{\rm TH}} \times 100 \tag{8}$$

where the theoretical mass of iron (M_{TH}) was obtained by using the Faraday relation and the experimental mass (M_{EXP}) was obtained by the electrode mass difference before and after each experiment.

For various current and voltage values, arsenic concentration versus residence time plots showed the same profile for arsenic removal: the longer the residence time, the higher the removal of arsenic species from the solution (see Fig. 8). Since the current density applied $(3.7-4.6 \text{ mA/cm}^2)$ in these experiments did not differ appreciably and the resident time was also very low (90 s), obviously its effect on arsenic removal efficiency was not observed. Chen et al. [26] and Kumar et al. [16] also reported similar results. According to Chen et al. [26], current density has no significant effect on total pollutant removal and according to Kumar et al. [16], current density has no significant effect on the final total arsenic removal.

Since reaction Eqs. (4) and (5) contain protons or hydroxides, respectively, the pH value is an important factor for arsenic removal in both acid and basic solutions. In addition, it influences the stability of the adsorbed arsenic species on in situ generated iron oxide/hydroxide/oxyhydroxide species.

4.2. Product characterization

It is difficult to differentiate between the iron oxide and oxyhydroxide species using only a single analytical technique. In this study, powder X-ray diffraction (XRD), Scanning electron microscopy (SEM), transmission Mössbauer spectroscopy (TMS) and Fourier transform infrared spectroscopy (FT-IR) were used to characterize the solid products formed from carbon steel electrodes during EC.

4.2.1. X-ray diffraction

Diffractograms were obtained with a Bruker AXS D4 Endeavor diffractometer operating with a Cu K α radiation source filtered with a graphite monochromator ($\lambda = 1.5406$ Å). The samples were wet ground to a fine powder (isopropyl alcohol from Sigma–Aldrich) and pressed into a sample holder. The XRD scans were recorded from 20° to 80° 2 θ , with 0.02° step-width and with a 10 s counting time for every step-width (increment). Experiments were run at 40 kV and 40 mA power. Fig. 3 shows a diffractogram of the filtered solid products (the feed solution contained 2.86 ppm of arsenic and the pH of the solution after EC was ~7). It indicates the presence of magnetite, geothite, hydrogen arsenate hydrate, iron arsenate, iron hydroxide oxide, and lepidocrocite in the solid products.

4.2.2. Scanning electron microscopy

Fig. 4 shows an SEM image of As contaminated iron oxyhydroxide particles. This image and the image of the energy dispersive analysis of X-rays (EDAX) (not presented here) show that the surface of these particles were coated with a layer of As species.

4.2.3. Transmission Mössbauer spectroscopy

Fig. 5 shows the spectrum obtained for the filtered solid products (the pH of the solution after EC was \sim 7.0). Mössbauer Spectrum for each sample was obtained on a \pm 15 mm/s velocity scale, which allows for observation of wide magnetic hyperfine spectra expected from iron oxide compounds. The spectrum consists of a doublet magnetic spectrum, which is probably due to fine particles of iron oxides (non-stoichiometric magnetite, see Fig. 3) or iron hydroxides (Goethite etc., see Fig. 3).

4.2.4. Fourier transform infrared spectroscopy

FT-IR analysis were carried out by Thermonicolate FT-IR spectrometer and OMNIC software using potassium bromide pellets (sample:KBr = 1:50). The spectra were usually



Fig. 3. XRD diffractogram studies of the iron impregnated with arsenic.



Fig. 4. SEM image of the arsenic coated on the iron-based particles.



Percent Absorption vs Velocity

Fig. 5. Mössbauer spectrum with arsenic at pH 7, indicating the presence of magnetite.

recorded in the range of 4000–400 cm⁻¹ with 2 cm⁻¹ resolution. Sixty-four scans were collected for each specimen. Fig. 6 shows the FT-IR spectrum of the by-product. Infrared analysis of iron electrode by-product showed OH stretching at 3738 and 3447 cm⁻¹, hydroxyl bending and γ' (OH) water bending vibration or overtones of hydroxyl bending around 1637 cm⁻¹ [27,28]. Bands for lepidocrocite phase showed up at 1120, 1023 and 745 cm⁻¹ [29]. Magnetite (Fe₃O₄ or Fe_{3-x}O₄) band at 575 cm⁻¹ and Fe–O vibration band is seen at 469 cm⁻¹ [29,30]. For details of FT-IR analysis, see Table 3. XRD analyses also confirmed the presence of these species detected by FT-IR.

4.3. EC pilot plant

The well water from La Comarca Lagunera has arsenic levels between 0.025 and 0.05 ppm with conductivity between 600 and 4000 μ S and pH between 5.5 and 7.1. The samples were treated by using a residence time of 1 min in the reactor, a voltage between 20 and 30 V and a current of



Fig. 6. FT-IR spectrum of iron electrode by-product.

Table 3 FT-IR vibrations and their corresponding wavenumbers and region for the bands observed for the EC by-product

Electrode material	Type of vibrations	Vibration wavenumbers (cm ⁻¹)	Vibration range (cm ⁻¹)
Iron	OH stretching	3738	3689-3787
	-	3447	3550-3000
	Hydroxyl bending	1637	1572-1813
	$\gamma'(OH)$ water bending	1637	1572-1813
	Overtones of hydroxyl bending	1637	1572–1813
	Magnetite (Fe ₃ O ₄ or Fe _{3-r} O ₄)	575	526-840
	Fe–O	469	416–510
	Lepidocrocite	1120	1090-1245
		1023	923-1057
		745	730-790

5 A. After the EC reaction, the residual arsenic in the well water was 0.002 ppm with final pH 8.5 and final conductivity between 500 and 2000 μ S. The arsenic removal efficiency of the EC process was higher than 99%.

The EC process is ideal for mobile units and is proving its worth in field trials in México using the 30 l/min Lamar Mobil Pilot Plant as shown in Fig. 7. Well #50, in the city of Torreón, was the test site for removal of arsenic. Well water containing 0.040 ppm of arsenic and having pH 7 was drawn directly from the well to the pilot plant system that has EC reactors, separation tanks, pumps, a rectifier operating from 480 V AC three-phase electrical power with automatic periodic reverse. The rectifier reverse timers were initially set at 5 min (both forward and reverse), but this value was increased to 20 min near the end of the first pass since the voltage was stable. A filter press was used for to collect residuals after the EC reaction. Analysis results of the effluent from SIMAS laboratories (water supply company for Torreón) demonstrated 0.002 ppm residual arsenic, which also indicates above 99% arsenic removal efficiency of EC. Arsenic concentration versus res-

Fig. 7. A picture of the Lamar Mobil Pilot EC Reactor that is being used in Torreón Coahuila México for field trials.



Fig. 8. Arsenic removal at various current and voltage values.

idence time curve profile of the pilot plant is similar to the results shown in Fig. 8. For this well producing $2500 \text{ m}^3/\text{day}$, the power requirements are about 150 W/min. For comparative purposes, the energy cost is US\$ 0.002 for cubic meter, but the cost to treat water in this system is directly proportional to the amount of contaminants.

5. Conclusions

The full potential of EC with air injection as an alternative water treatment technique to remove arsenic from well water is yet to be fully realized. EC comprises complex chemical and physical processes involving many surface and interfacial phenomena. Also, the results of this study suggest that EC produces magnetic particles of magnetite and amorphous iron oxyhydroxides, and that this process can be used to remove As(III) and As(V). The 99% arsenic removal in the experimental EC reactor is usually completed within 90 s or less for most experiments with approximately 100% current efficiency. A pilot plant EC study yielded 99% removal of total arsenic from well water. These results for the removal of arsenic show that a pilot plant system can be used to remove arsenic most efficiently from contaminated well waters. Hence, EC with air injection technique built in a pilot robust system can be a very promising technology for removal of toxic and heavy metals especially arsenic, where adjustment of pH is not required during the EC process.

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