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Removal of Arsenic from Wastewaters by Airlift Electrocoagulation. Part 1: Batch Reactor Experiments

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Abstract: Arsenic removal from wastewater is a key problem for copper smelters. This work shows results of electrocoagulation in aqueous solutions containing arsenic in a newly designed and constructed 1 L batch airlift reactor. Iron electrodes were used in the cell. The airlift electrocoagulation reactor allowed simultaneously a) anodic Fe^{2+} production, b) Fe^{2+} to Fe^{3+} oxidation by air or oxygen, and c) precipitate/coagulate formation due to the turbulent conditions in the cell. A series of electrocoagulation experiments were carried out in the batch airlift reactor. The variables were: initial As(V) concentration, use of either a pure oxygen or an air flow, and electric current density. The results showed that the airlift electrocoagulation process could reduce an initial As concentration from $1000\,mg\,L^{-1}$ to $40\,mg\,L^{-1}$–corresponding to a reduction of 96%. At higher initial concentrations (e.g. $5000\,mg\,L^{-1}$ As) the oxidation of Fe^{2+} to Fe^{3+} seems to be rate determining. Oxidation with compressed oxygen was clearly more efficient than air at high initial As concentration. Arsenate removal from a solution with initially $100\,mg\,L^{-1}$ was efficient with both air and oxygen addition–more than 98% of As precipitated. When the electrocoagulation process was working efficiently, the arsenic removal rate in the cell was found to be around 0.08–0.1 mg As/C. The Fe-to-As (mol/mol) ratio, when electrocoagulation was working properly, was in the range of 4–6.

Keywords: Electric field, airlift reactor, iron electrodes, current density, oxidation

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INTRODUCTION

Pyrometallurgic copper processing generates large amounts of arsenic that vaporize as arsenic trioxide. This compound is absorbed from the gas flow leading into the sulphuric acid plant together with a variety of heavy metals, creating a highly acidic contaminated wastewater. Wastewater from copper smelters is acidic and contains typically considerable amounts of copper, lead, cadmium, zinc, arsenic and mercury (1). At the Codelco El Teniente copper smelter, heavy metals are precipitated as hydroxides but arsenic remains in the nearly pH-neutral wastewater (2). Combined $CaCO_3$ and $FeCl_3$ precipitation deals with the arsenic but since the arsenic concentration in the gas phase changes due to the batchwise operation of the smelter, it is difficult to predict and control the chemical dosage for the precipitation of the arsenic compounds.

Electrocoagulation is by now proven to be able to treat wastewaters from different sources such as textile industries (3), food and protein production (4), soluble oil wastes (5), restaurants (6), metal finishing (Mills, 2000) (7), and lately arsenic containing wastewater and groundwater (8–12). Typically, aluminium or iron plates are used as electrodes in the electrocoagulation process (13). When DC voltage is applied, the anodes sacrifice themselves to produce Al^{3+} or Fe^{2+} ions, which are good coagulants (14).

Hansen et al. (11) found analysing preliminarily the electrocoagulation process in a rectangular airlift reactor with cylindrical electrodes, that upto 99% of arsenic could be removed (from 5000 mg As/L solutions) using iron sacrificial electrodes. Here it was also shown that introduction of air was necessary to assure oxidation of dissolved iron. The effect of air bubbling not only oxidized Fe^{2+} to Fe^{3+} but also promoted the coagulation/flocculation process due to turbulence. Arsenic (III) had to be oxidized to As(V) before precipitation and subsequentially arsenic removal occurred. Inconveniences with the cell used were the presence of dead volumes in cell due to the rectangular configuration. Furthermore, it would be necessary to evaluate the potential of the electrocoagulation process at higher initial As(V) concentration than $100\ \mathrm{mg\ L^{-1}}$. If more concentrated solutions can be treated successfully, the electrocoagulation process could be considered as a primary or stand-alone unit.

The purpose of this work is to evaluate the As(V) removal from aqueous solutions by a newly developed airlift process. The airlift produces the necessary turbulence in the reactor. Parameters studied are:

a) initial arsenic concentration
b) the use of either oxygen or air as an oxidant
c) electric current density, and
d) remediation time.

BACKGROUND

Electrocoagulation consists of an in-situ generation of coagulants by an electrical dissolution of iron or aluminium electrodes. The generation of metallic cations takes place at the anode, whereas at the cathode, typically a H_2 production occurs together with OH^- release. When applying iron electrodes the process generates iron hydroxides, which would co-precipitate with arsenic anions. The main electrode reactions are at neutral pH:

Anodic Reactions

$$\mathrm{Fe} \rightarrow \mathrm{Fe}^{2+} + 2\mathrm{e}^- \tag{1}$$

$$2\mathrm{H_2O} \rightarrow \mathrm{O_2} + 4\mathrm{H}^+ + 2\mathrm{e}^- \tag{2}$$

Cathodic Reaction

$$2\mathrm{H_2O} + 2\mathrm{e}^- \rightarrow \mathrm{H_2} + 2\mathrm{OH}^- \tag{3}$$

When introducing air (or oxygen) to the process, Fe^{2+} is oxidized rapidly:

$$\mathrm{O_2(gas)} + 4\mathrm{Fe}^{2+} + 2\mathrm{H_2O} \rightarrow 4\mathrm{Fe}^{3+} + 4\mathrm{OH}^- \tag{4}$$

The rate of the oxidation depends on the availability of dissolved oxygen.

Typically at the cathode the solution becomes alkaline with time. The applied current forces OH^- ion migration towards the anode (and Fe^{3+} to the cathode), thus favoring ferric hydroxide formation:

$$\mathrm{Fe}^{+3} + 3\mathrm{OH}^- \rightarrow \mathrm{Fe(OH)_3} \tag{5}$$

Arsenate co-precipitates with or adsorbs to $Fe(OH)_3$:

$$\alpha\mathrm{Fe(OH)_3(s)} + \beta\mathrm{AsO_4^{3-}(aq)} \rightarrow [\alpha\mathrm{Fe(OH)_3^*}\beta\mathrm{AsO_4^{3-}}]\mathrm{(s)} \tag{6}$$

For effective arsenate removal due to precipitation, the ratio α/β should be higher than (15). During electrocoagulation, iron hydroxide particles are formed in the presence of As(V). This has shown to be more efficient for arsenic removal than adsorption to pre-formed Fe(III) particles (16–17).

It is expected that the As(V) would be removed more efficiently than As(III), since As(V) anions (AsO_4^{-3}, $HAsO_4^{2-}$, or $H_2AsO_4^-$) are adsorbed stronger by iron oxides than As(III) (18). If present, it would be necessary to oxidize As(III) to As(V). However, oxidised conditions in general favour arsenic removal in wastewaters (16).

During the electrochemical production of ferric hydroxide periodic current reversal is advisable for two reasons:

a) to use two iron based electrodes which alternate as anode and cathode, both of them contributing to ferric hydroxide production. This way, they are evenly consumed during the process;
b) to avoid anode passivation by periodic removal of excess ferrous and ferric ions from the vicinity of the anode and of loosely attached reaction products from the anode surface.

The major problem of the iron electrode is its passivation, which is caused by iron hydroxide produced during the discharge process and prevents further anodic utilization. Passivation of the anode surface is possible in these systems due to high current densities and high concentrations of Fe^{2+}, Fe^{3+}, and OH^- ions at the anode surface.

The electrocoagulation process has recently been applied with success for arsenic removal from wastewater containing $100\,mg\,L^{-1}$ As(V) (11). Different process designs were tested, such as a turbulent flow reactor, a combined electrocoagulation and sedimentation basin, and an airlift reactor. The airlift reactor gave most promising results but characteristics in the design showed the presence of dead volumes in the cell. In all cases turbulent conditions were obtained, which are essential for having an efficient electrocoagulation process (19).

EXPERIMENTAL

Wastewater Characteristics

The arsenic containing wastewater was prepared by dissolving adequate amounts of Sodium Arsenate (Na_2HAsO_4 analytical grade) in distilled water to reach the wanted concentrations of 5000, 1000, or $100\,mg\,L^{-1}$. One liter of solution was prepared for each experiment. The total arsenic and iron content in liquid samples was determined by an Atomic Absorbance Spectrophotometer (AAS). Detection limits were 2 mg/L and 1 mg/L for arsenic and iron, respectively. The pH of the solutions was in all cases initially around 7.

Experimental Setup

Figure 1 shows the experimental set-up used in this work. The cylindrical acrylic cell had a total volume of approximately 1.5 L. Two iron cylinders were placed inside the cell, and they were kept at approx. 3 cm from the bottom of the cell. In the gap between the two iron cylinders at the bottom,

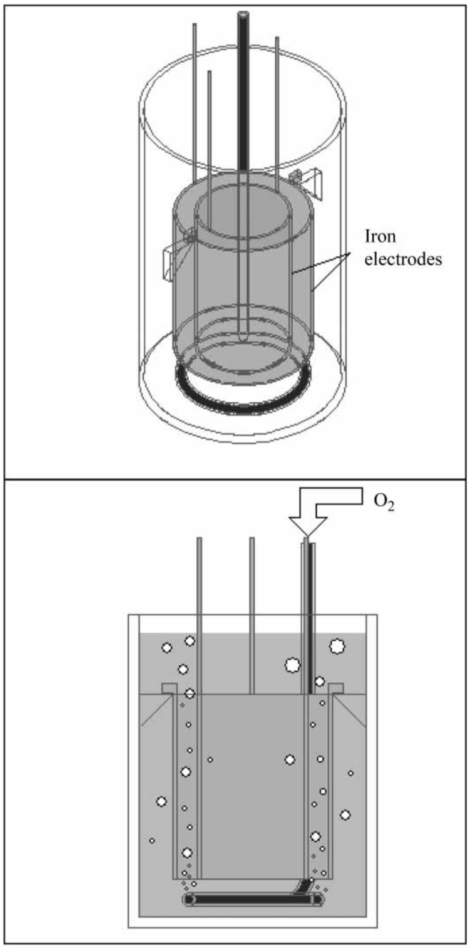

Figure 1. Experimental set-up. Cell height: 20 cm, Cell diameter: 10 cm, outer iron electrode diameter: 7 cm, inner iron electrode diameter: 5 cm, electrode height: 10 cm.

a perforated PVC tube was placed in order to produce an airflow in between the cylinders. This airflow sucked liquid with it and when reaching the top, the air "lifted" the surface level of the liquid, and the generated liquid flow created turbulence in the reactor. The electrode surface area/volume (S/V) ratio for the reactor was estimated to be 16.7 m^2/m^3. The cell was filled with 1 L solution in each experiment.

An Extech power supply, a homemade devise to produce electric current reversal, a multimeter, and an air compressor/oxygen gas container were used in the different experiments.

Electrocoagulation Experiments

Ten series of electrocoagulation experiments were carried out. See Table 1 for operational details. The experiments were done by placing one liter of arsenic containing solution. Operational variables were: Initial As(V) concentration, applied electric current, and either an air or pure oxygen flow to produce the combined oxidation/airlift effect. The same air or oxygen flow was used in all experiments: 5 L/min. The current was reversed each 2 minutes in order to minimize passivation of the iron anodes. Constant current was applied during experiments–either 1, 2, or 3 A, corresponding to a current density of 60, 120, and 180 A/m^2, respectively. The experiments were run for either 60, 120, 300, or 420 minutes depending on initial concentration and current strength. During and after experiments the As (all experiments) and Fe (only exp. 3–6 and 8) concentrations were measured in the solution.

RESULTS AND DISCUSSION

In Table 1 electrocoagulation results for all experiments in terms of final As and Fe concentration, removal efficiency, removed arsenic/charge and Fe-to-As ratio are given. Initial concentrations were measured before all experiments and the standard deviations were: $5000 \pm 25\ mg\,L^{-1}$, $1000 \pm 15\ mg\,L^{-1}$, $100 \pm 2\ mg\,L^{-1}$. In Table 1 the initial concentrations are given without the deviation in order have an easier overview.

In general it can be said that the airlift reactor is working successfully. A red-orange precipitate formed during most experiments (except exp. 1 and 5) indicating formation of ferric hydroxide. In the experiments with pure oxygen addition, the amount of precipitate was larger than with air bubbling. The cell potential was nearly constant during experiments. For example, in experiment 6 the voltage drop started at 10.0 V and ended on 9.6 V after running for four hours. The voltage drop in all other experiments applying 3 A was in the same magnitude. When applying 2 A, the voltage drop was in the range 5–6 V. The overall arsenic removal as a function of applied electrical charge is also given in the table as an indication if the electrocoagulation process is working at an acceptable level. It seems that for treating a $5000\ mg\,L^{-1}$ solution efficiently, the arsenic removal rate would be around 0.06–0.07 mg/C. Higher rates could be possible but the reaction kinetics and mass transfer determining steps should be evaluated. When treating a $1000\ mg\,L^{-1}$ solution, higher removal rates can be achieved (around 0.07–0.08 mg/C). The soluble iron content in the experiments was in all cases (except exp. 5) lower than the detection limit of the AAS–meaning all electrochemically dissolved iron is precipitated during the process. No residual Fe should therefore be removed from the cleaned wastewater.

Considering that the only reaction at the anode is $Fe \rightarrow Fe^{2+} + 2e^-$ (which would be the case when no passivation of the anode is occurring),

Table 1. Electrocoagulation experiment details and results. C_0: Initial arsenic concentration, $C_{f,i}$: Final concentration of element *i*, EC time: Electrocoagulation time

Exp.	C_0 mg/L	Air/Oxygen addition	Electric current A	EC time min	$C_{f,\ As}$ mg/L	$C_{f,\ Fe}$ mg/L	As removal %	As removed/ charge mg/C	Produced iron mol	Fe-to-As ratio mol/mol
1	5000	Air	2	300	4700	—	6	0.0069	0.18656	42–62
2	5000	Air	3	300	4500	—	10	0.0139	0.27984	35–55
3	5000	Oxygen	3	300	1130	<1	77	0.0716	0.27984	5.4–6.0
4	5000	Oxygen	3	420	45	<1	99	0.0633	0.39178	5.9–7.7
5	1000	Oxygen	0	180	1010	60	0	—	0.000107	—
6	1000	Oxygen	1	180	170	<1	85	0.0768	0.05597	3.6–6.6
7	1000	Oxygen	2	180	60	—	94	0.0653	0.11193	3.8–8.9
8	1000	Oxygen	3	180	40	<1	96	0.0444	0.16790	3.8–13
9	100	Air	2	60	<2	—	>98	0.0136	0.037311	20–28
10	100	Oxygen	2	60	<2	—	>98	0.0136	0.037311	21–28

then the theoretical amount of produced Fe^{2+} can be calculated when knowing the total electrical charge passed through the system:

$$Fe = \frac{C}{2F}$$

where Fe is mol Fe^{2+} produced, C are the coulombs passed through the system and F is Faradays constant.

It must be stated that the theoretical amount of dissolved iron due to Faraday's, law does not take into account any chemically dissolved iron. In actuality, in experiment 5, where no current was applied, the dissolved iron was around $60\,mg\,L^{-1}$ after 180 minutes. This corresponds to 1% of what would be dissolved applying 2 A in the same time period. For treatment of more dilute arsenic contaminated waters the oxidation of zero-valent iron by air has proven to be more important (20). The amount of As removed is measured, and then the ratio Fe-to-As in mol/mol can be estimated (see Table 1). The Fe-to-As is given as a range observed during the whole period of electrocoagulation. From Table 1 it is seen that electrocoagulation with 1000 and $5000\,mg\,L^{-1}$ As with oxygen flow generally functions well showing Fe-to-As ratios around 4–6 mol/mol. This is in fact quite low in comparison with conventional iron hydroxide/arsenate precipitation. Hansen et al. (11) presented Fe-to-As ratio in an airlift reactor around 14 mol/mol, when carrying out electrocoagulation on a $100\,mg\,L^{-1}$ solution–compared to 20–28 mol/mol in this present work using the same current density. The configuration of the cells was somehow different (e.g. the S/V ratio). The higher Fe-to-As ratio with $100\,mg\,L^{-1}$ could be due to mass transfer being the limiting step.

Figure 2 shows the effect of the Fe^{2+}-to-Fe^{3+} oxidizing agent–either air or pure oxygen. In Fig. 2a, the As concentration is given as a function of electrocoagulation time for experiments with $5000\,mg\,L^{-1}$ solution applying different current strengths and oxidant. It is clearly seen, that the arsenic removal is better when adding oxygen than air at this arsenic concentration level. When using air only small amounts of arsenic are precipitated indicating that the Fe^{2+} oxidation step could be the limiting step in the process. Therefore, to have efficient electrocoagulation removal of arsenic at this concentration, stronger oxidants (such as pure oxygen) should be considered. For conventional arsenate/iron hydroxide precipitation purposes, several oxidants have been suggested, e.g. permanganate or ozone (16).

Figure 2b shows the arsenic concentration over time during electrocoagulation in $100\,mg\,L^{-1}$ solutions using either air or oxygen flow. Here it can be seen that the oxidation process is not the limiting step, since the arsenic removal is similar for both cases. This correspond well with the findings of Hansen et al., (11) where efficient arsenic removal was obtained using air as an oxidant. The Fe^{2+} oxidation by air is enhanced at alkaline pH (21), therefore with time the oxidation could be faster, since pH rises during the

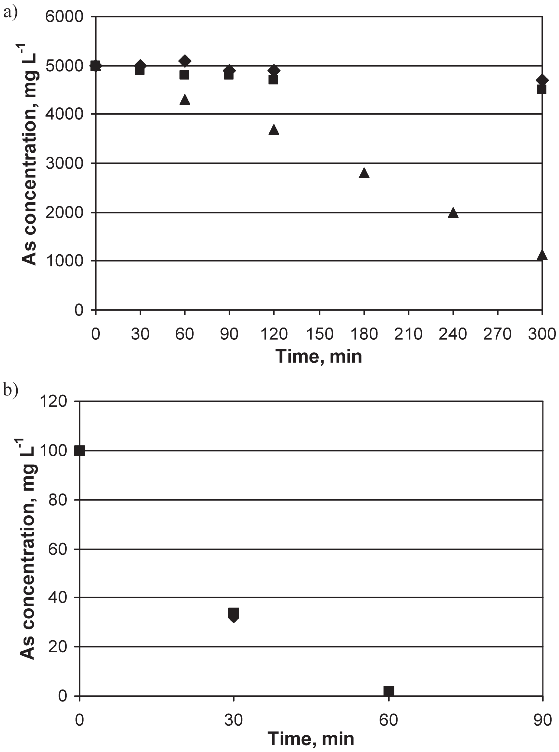

Figure 2. Arsenic removal by electrocoagulation with time. a) Initial concentration 5000 mg L^{-1}. b) Initial concentration 100 mg L^{-1}. Legend 2a) ◆: 2 A, air; ■: 3 A, air; ▲: 3 A, oxygen. Legend 2b) ◆: 2 A, air; ■: 2 A, oxygen.

electrocoagulation process. The low arsenic removal per charge (see Table 1) can be contributed to other rate determining steps such as mass transfer and diffusion.

Figure 3 shows the arsenic concentration in the solution as a function of time for exp. 5–8 when applying 0, 1, 2, or 3 A. Initial As concentration was 1000 mg L^{-1}. In the figure the effect of the current can be evaluated. It looks like the arsenic removal is somewhat proportional with the applied current–at least until around 90% of the arsenic is removed.

Figure 4 shows the total removed arsenic as a function of charge for the first 120 minutes of exp. 6 and the first 60 minutes of exp. 7 and 8. It seems that the removal is proportional with the charge–as also expected from Fig. 3. This indicates that in all three cases the electrocoagulation process is working efficiently–producing the necessary Fe^{2+} cations, which are oxidized rapidly by oxygen. Figures 3 and 4 show that the current could be raised even further, if faster arsenic removal is desired. The limiting current density has not yet been reached at 180 A/m^2 (3 A).

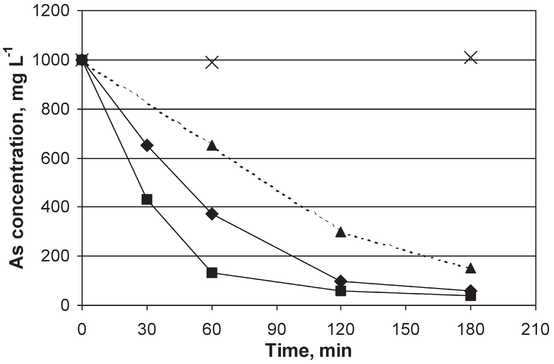

Figure 3. Arsenic removal by electrocoagulation with time. Initial concentration 1000 mg L^{-1}. Legend: ×: 0 A, oxygen; ▲: 1 A, oxygen; ◆: 2 A, oxygen; ■: 3 A, oxygen.

If the electrocoagulation results applying oxygen and 3 A are combined, one could expect a nearly 100% As removal from 5000 mg L^{-1} initial concentration. From Figure 2a it can be estimated by extrapolation that a concentration of 1000 mg L^{-1} in the solution from initially 5000 mg L^{-1} is reached after around 310 min. From Fig. 4 it can be approximated that it takes 70 min to lower the concentration from 1000 to 100 mg L^{-1}. This means that it would take 380 min (310 + 70) to reach 100 mg L^{-1} from 5000 mg L^{-1}. From this one can make an estimation of what could happen when "adding" the time difference—or displacing the start time—in the experiments.

Figure 5 shows the As concentration over time, where the electrocoagulation results starting from 1000 mg L^{-1} As (exp. 8) and 100 mg L^{-1} (exp. 10) have been "added" 310 and 380 (310 + 70) min, respectively. The figure

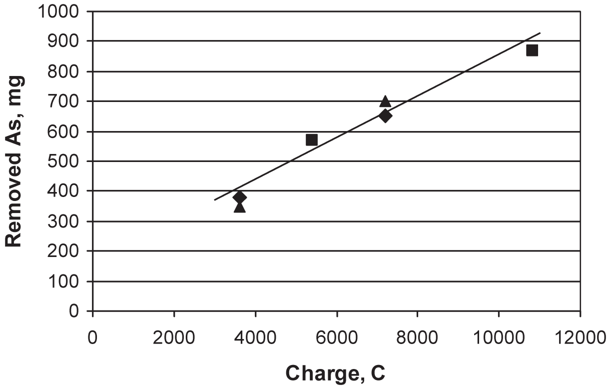

Figure 4. Removed arsenic from 1000 mg L^{-1} solution by electrocoagulation as a function of electrical charge. Legend: ▲: 1 A, oxygen; ◆: 2 A, oxygen; ■: 3 A, oxygen.

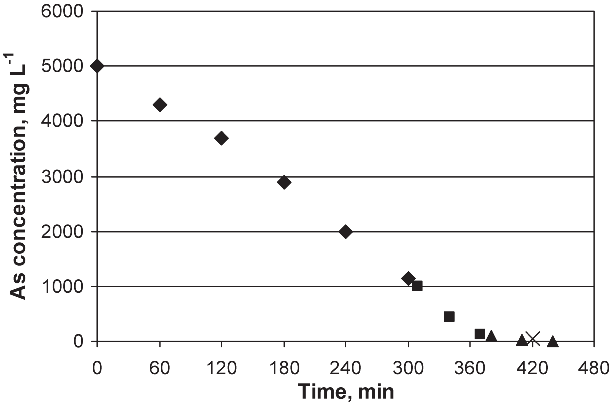

Figure 5. Arsenic concentration with time by combining the electrocoagulation results treating 5000 mg L^{-1}, 1000 mg L^{-1} and 100 mg L^{-1} As solutions. Legend: ◆: Initial 5000 mg L^{-1}; ■: Initial 1000 mg L^{-1}; ▲: Initial 100 mg L^{-1}; ×: Exp. 4.

indicates that a total As removal by electrocoagulation would be possible after approximately 400 min, applying 3 A and compressed oxygen. Exp. 4 was carried out to validate this combination of experiments. This experiment is added to Fig. 5, and it can be seen that the level of arsenic reached nearly the expected value. The effect of accumulated iron has to be analysed, together with the effect of the produced precipitate, and therefore further experiments are necessary to prove this.

CONCLUSIONS

Electrocoagulation of As(V) in wastewaters is a promising remediation tool to remove arsenic as adsorbed to or co-precipitate with iron(III)hydroxide. In a newly developed batch airlift reactor As removal efficiencies higher than 98% were obtained in 100 mg L^{-1} As(V) solution. Since detection limit of the used AAS was 2 mg/L arsenic, it is not sure which level electrocoagulation can reach with respect to arsenic removal. Typical lower levels (e.g. US-EPA recommends 0.1 mg/L) are needed. Further research should reveal this doubt.

In more concentrated arsenic solutions electrocoagulation functioned well, too. Here the process did not show high total arsenic removal since the experiments were stopped for time reasons, and further As removal could be expected.

Oxidation of Fe^{2+} to Fe^{3+} seems to be a limiting step when treating highly concentrated arsenic solutions. Only small amounts of arsenic was removed from a 5000 mg L^{-1} solution by electrocoagulation in the batch airlift reactor using air as an oxidant. On the other hand, when using pure oxygen the removal rate increased remarkably.

The arsenic removal seems to be proportional with the charge, when working with current densities in the range 120–180 A/m^2. Around 0.1 mg As is removed per Coulomb.

When the electrocoagulation process is working properly, the Fe-to-As ratio (taken as the total anodically produced Fe divided by the amount of removed arsenic at a given time) lies around 4 to 6. This low value makes the electrocoagulation process very competitive to conventional precipitation techniques.

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