

Arsenite removal from water by electro-coagulation on zinc–zinc and copper–copper electrodes

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Received: 3 November 2011 / Revised: 20 May 2012 / Accepted: 2 August 2012 / Published online: 13 November 2012
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Abstract Removal of arsenite from aqueous solution was carried out using electro-coagulation method. The experiments were conducted using copper–copper and zinc–zinc electrodes. The optimized experimental parameters were 2.0 mg/L initial concentration, 16.0-min processing time, 6.0 pH, 3.0-V applied voltage and 30 °C temperature for zinc–zinc electrodes while these values for copper–copper electrodes were 2.0 mg/L initial concentration, 20.0-min processing time, 7.0 pH, 5.0-V applied voltage and 30 °C temperature. The results demonstrated that zinc–zinc and copper–copper electrodes removed arsenite up to 99.89 and 99.56 %, respectively. The treated water was clear, colorless and odorless without any secondary contamination. There was no change in water quality after the removal of arsenite. The reported method is capable to remove arsenite from water at 6–7 pH range, which is a pH range of natural water. Therefore, this method may be the choice of arsenite removal from natural ground water.

Keywords Arsenic removal · Electro-chemical method · Eco-friendly · Ground water

Introduction

Nowadays, arsenic in the ground water has become a worldwide problem due to its toxic and pervasive nature. More than 70 countries are reported to have arsenic problem in drinking water and about 150 million people are at

risk. Moreover, about 70 % of 150 million people are living in ten Asian countries i.e. Bangladesh, Cambodia, China, India, Laos, Myanmar, Nepal, Pakistan, Taiwan and Vietnam. These countries have high-level alarm of arsenic havoc (Ali et al. 2011; Brammer and Ravenscroft 2009; Chen et al. 2011; Guo et al. 2003; Nickson et al. 2000). Prolonged exposure of arsenic contaminated water leads to various health hazards and side effects such as hyperkeratosis, skin lesions, and cancers of brain, liver, kidney, and stomach (Choong et al. 2007; Karim 2000; Smith et al. 1992). Generally, arsenic in natural water is found in two oxidation states viz. As(III) and As(V), which are called as arsenite and arsenate, respectively. Arsenite is more toxic due to its affinity for thiols group of proteins to form products that cause body malfunctions (Wang and Wai 2004). It has also been observed that arsenite and arsenate are found in anaerobic (highly reducing conditions) and oxygenated water, respectively (Gomes et al. 2010). Therefore, it is more difficult to remove arsenite in comparison to arsenate.

Some methods have been used for the removal of arsenite from water which include coagulation, adsorption, ion-exchange and reverse osmosis (Balasubramanian and Madhavan 2001; Ghimire et al. 2003; Hege et al. 2004; Jay et al. 2004; Katsoyiannis and Zouboulis 2004; Kim and Benjamin 2004; Seidel et al. 2001). But all these techniques have some draw backs such as production of large sludge, costly, time consuming and poor percentage of arsenite removal, making them economically unsuitable for large-scale arsenic removal. Contrarily, electro-coagulation is a simple, efficient and promising technique for water treatment (Ali et al. 2011). Moreover, the electrochemical methods of water treatment can be applied at large scale for supplying pure water to the communities, with minimum waste management. In view of these facts, attempts have

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been made to remove arsenite from drinking water by electro-coagulation method using Zn–Zn and Cu–Cu electrode assemblies. The results of these findings are discussed herein.

Materials and methods

Chemical and reagents

A. R. grade chemicals and Millipore water (Millipore Milli-Q, Bedford, USA,) were used to prepare all stock solutions. Sodium *m*-arsenite (NaAsO_2) was purchased from Merck, Germany. Ammonium molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$), stannous chloride ($\text{SnCl}_2\cdot 2\text{H}_2\text{O}$) and sodium chloride (NaCl) were obtained from Merck, India. The other chemicals such as glycerol and sulphuric acid were supplied by Qualigens, New Delhi, India. Zinc and copper electrodes were purchased from the local market, New Delhi and their purities were checked before using in electro-coagulation unit. All glasswares were soaked in 10 % HNO_3 (v/v) for 48 h followed by washing three times with Millipore water and air drying prior to use.

Procedure

Arsenite stock solution (100.0 mg/L) was prepared by taking the salt of sodium arsenite (NaAsO_2) in Millipore water. It was preserved by adding 2.0 mL of concentrated HCl and stored at 4 °C for longer duration use. The working arsenite solutions were prepared fresh prior to experiment by diluting arsenite standard stock solution with laboratory tap water (New Delhi, India), to achieve the natural ground water conditions. An electro-coagulation unit with anode and cathode of dimension 7.5×1.0 cm was used for the electro-coagulation of the arsenite samples. All the experiments were conducted in batch mode by diluting arsenite solution to the necessary concentrations with two sets of electrodes i.e. Zn–Zn or Cu–Cu electrodes. The electro-coagulation was carried out in a 100-mL beaker on magnetic stirrer, for constant agitation to reduce the mass transport over potential of the electro-coagulation cell. The space between the vertically positioned metal electrodes of EC cell was 1.0 cm and dipped in arsenite water. Total submerged surface area of each electrode was 13.36 cm^2 . pH of the working solution was adjusted by adding either dilute hydrochloric acid or sodium hydroxide. After each experiment, the electrodes were corroded with sand paper to remove scale and then washed with Millipore water. EC unit was connected to direct power supply for maintaining the current density. The treated water was allowed to settle down for 2–3 h and residue discarded. The filtrate solution

was used for determining the amount of residual arsenite spectrophotometrically.

Analysis

To determine arsenite [As(III)] in the filtrate spectrophotometrically, a stain was prepared by mixing two reagents i.e. stannous chloride and ammonium molybdate reagents. Stannous chloride reagent (110.8 mM) was prepared in glycerol while the ammonium molybdate was prepared in Millipore water followed by acidification by dilute hydrochloric acid with overall concentrations of ammonium molybdate and sulphuric acid as 20.23 mM and 5.2 M, respectively. To develop the stain, stannous chloride and ammonium molybdate reagent were mixed together (1:8, v/v), resulting into blue colour which was allowed to stand for 5 min. The stain was prepared fresh before each experiment and used within 20 min. 1.0 mL of stain was mixed with 50.0 mL of the filtrate arsenite solution and kept for 12 h, which gave blue colour with intensity directly proportional to arsenic concentration. The absorbance of the coloured solution was measured at 840 nm against a reagent blank. The concentration of arsenite was determined using a calibration graph against the blank.

Water quality of laboratory tap water (Jamia Nagar, New Delhi) was also determined before preparing arsenite solution in it. The parameters analyzed for ground water were conductivity (DB 1038, Decibel, India), pH (Control Dynamics), alkalinity, total dissolved solids (TDS), total hardness (temporary and permanent hardness), chloride, sodium, potassium and calcium ions.

Validation of the method

The validation of the electro-coagulation method was carried out for linearity, specificity, accuracy and precision. The values of standard deviations, correlation coefficients and confidence levels were calculated.

Linearity

The linearity of UV–Vis. Spectroscopic method was carried out by recording absorbance of different concentrations of arsenite solutions. The solutions of arsenite for linearity were prepared from 2.0 to 25.0 mg/L concentration ranges. The linear calibration graphs were plotted between different values of concentrations and absorbance. The analysis of linear regression by least squares method of the calibration curves was carried out to determine linearity range. The calibration graphs and the negligible scatter of experimental points were considered for calculation of

correlation coefficients and relative standard deviations (Miller and Miller 1984).

Specificity

The specificity was confirmed by observing any change in the results due to the presence of various dissolved species in the ground water. For this, the effect of ionic strength on the removal of arsenite was studied using 50.0–300.0 mg/L concentrations of sodium chloride.

Precision

To calculate precision data, all the experiments were carried out five times. Three different concentrations (2.0, 5.0 and 10.0 mg/L) of arsenite were selected and used.

Accuracy

The different concentrations of arsenite were used to determine the accuracy of the electro-coagulation method. Three different concentrations i.e. 5.0, 10.0 and 15.0 mg/L were used for this purpose. The experiments were carried out five times ($n = 5$) with these concentrations. The accuracy was determined by interpolation of the removal capacities of arsenite in all five experimental sets.

Results and discussion

The electro-coagulation cannot be carried out in distilled or Millipore water due to its almost zero conductivity. Therefore, serial dilutions (2.0–25.0 mg/L) of arsenite solution were prepared in natural ground water (Tap water) of New Delhi, India. General water quality of tap water was determined prior to arsenite removal by EC. Conductivity and pH of the ground water were found to be 1.45 mS/cm and 7.12, respectively. The values of total dissolved solids (TDS), total hardness, alkalinity, sodium, potassium, calcium and magnesium were 542.25, 448.0, 280.0, 2.15, 1.84, 204.0 and 244.0 mg/L, respectively, while iron was absent. As discussed above the electro-coagulation method was optimized by varying pH, arsenite concentration, voltage, processing time and temperature. The effect of these parameters is discussed in the following sub-sections.

Effect of arsenite concentration

The effect of arsenite concentrations on its removal efficiency was studied over a range of 2.0–25.0 mg/L and the percentage removal of arsenite is shown in Fig. 1. It can be observed that both zinc and copper electrode assemblies

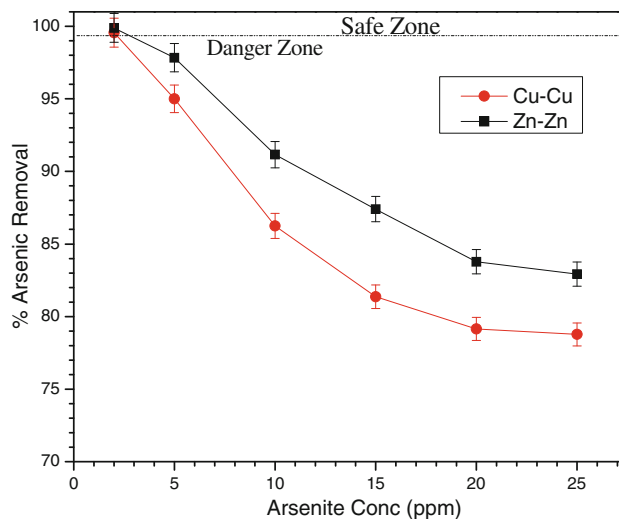


Fig. 1 Effect of initial arsenite concentration on percent removal efficiency (Conditions for zinc electrodes 16.0 min processing time, 6.0 pH, 3.0 V applied voltage and 30 °C temperature and for copper electrodes 16.0 min processing time, 7.0 pH, 5.0 V applied voltage and 30 °C temperature)

attained a significant removal of arsenite. For low arsenite concentration the percentage removal was much more as compare to the solution of higher concentration (Ali et al. 2012). The graph clearly indicates a sharp decrease in the percentage removal (99.89–82.92 and 99.56–78.77) for 2.0–25.0 mg/L for zinc and copper electrodes. This Figure is divided into two regions i.e. safe and dangerous zones. The safe zone started at 99.50 % removal at 2.0 mg/L concentration (10 ppb; permissible limit prescribed by WHO) for both electrode systems. Furthermore, in safe zone the maximum removal of arsenite was achieved up to 99.89 and 99.56 % for Zn–Zn and Cu–Cu electrode systems, respectively. It is interesting to note that 2.0 mg/L concentration of arsenite is quite high and rarely found in natural water resources (Mandal and Suzuki 2002). Therefore, this Figure indicates that the reported experimental conditions were quite satisfactory to reduce arsenite level up to prescribed limit of 10.0 µg/L or low.

Effect of initial pH

pH of electro-coagulation system is one of the most essential factors for controlling the removal of contaminants (Can et al. 2003; Daneshvar et al. 2003; Elemer 1978; Kobya et al. 2003; Lin and Chen 1997). To study the effect of pH on the electro-coagulation process, experiments were carried out at various pH covering acidic, neutral and basic conditions (2–9 pH range) and results are shown in Fig. 2. The maximum arsenite removal occurred at pH 6–9 for both zinc and copper electrodes. The safe zone (99.5 %) started at 6.0 and 7.0 pH for zinc and copper electrodes,

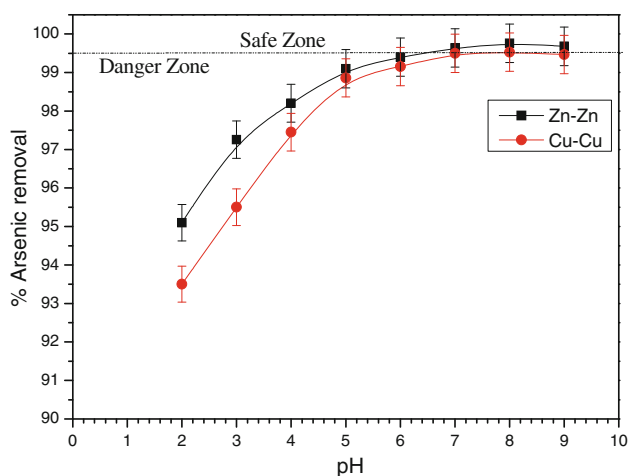


Fig. 2 Effect of pH on percent removal efficiency of arsenite (Conditions for zinc electrodes 2.0 mg/L initial arsenic concentration, 16.0 min processing time, 3.0 V applied voltage and 30 °C temperature and for copper electrodes 2.0 mg/L initial arsenic concentration, 16.0 min processing time, 5.0 V applied voltage and 30 °C temperature)

respectively. The maximum removals achieved were 99.89 and 99.56 % for zinc and copper electrodes, respectively, at pH 8.0, which became constant for further increase in pH. It was worthy to note that pH of solution increased during the electro-coagulation (Vik et al. 1984). Zinc and copper get dissolved to produce divalent ions Zn^{2+} and Cu^{2+} ions forming zinc and copper hydroxide. It is because of their low solubilities, ($K_S = 3 \times 10^{-17}$ for zinc; 4.8×10^{-20} for copper) (Pourbaix 1974) but thermodynamically these hydroxides are formed at pH >8.6 and 7.7 for zinc and copper, respectively, while obtained results showed good removal at pH 6–9 range. This was due to the interfacial pH increase during electro-coagulation process favouring the formation of these hydroxides.

Effect of voltage

Applied voltage influences directly the current density which determines the coagulant dosage rate and bubble production rate, size and growth of flocs, influencing the removal efficiency of any electrochemical process (Kobya et al. 2011). To investigate the effect of applied voltage on removal efficiency of arsenite, voltages applied were 1.0–7.0 with 2.0 mg/L as initial arsenite concentration for both the electrodes (Fig. 3). A perusal of this Figure indicates that the safe zone started at 3.0 and 5.0 voltages for zinc and copper electrodes, respectively. But the maximum removal of arsenite (99.89 and 99.56 % for zinc and copper electrodes) was achieved at 5.0 and 6.0 voltages for these electrodes, which became constant for further increase in voltage. As the voltage applied, divalent ions Zn^{2+} and Cu^{2+} were generated thereby forming $ZnHAsO_4$ and

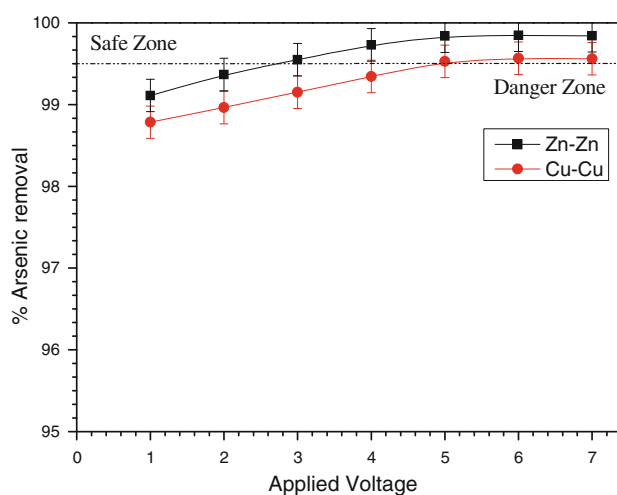


Fig. 3 Effect of voltage on percent removal efficiency of arsenite (Conditions for zinc electrodes 2.0 mg/L initial arsenic concentration, 16.0 min processing time, 6.0 pH and 30 °C temperature and for copper electrodes 2.0 mg/L initial arsenic concentration, 16.0 min processing time, 7.0 pH and 30 °C temperature)

$CuHAsO_4$ as discussed above. But below 3.0 and 5.0 volt for zinc and copper electrodes, metal hydroxides did not form sufficiently to remove arsenite.

Effect of processing time

The effect of time on the efficiency of arsenite removal by electro-coagulation process is shown in the Fig. 4. As usual the safe zone (99.89 and 99.56 % for zinc and copper electrodes) began after 16.0- and 20-min processing times

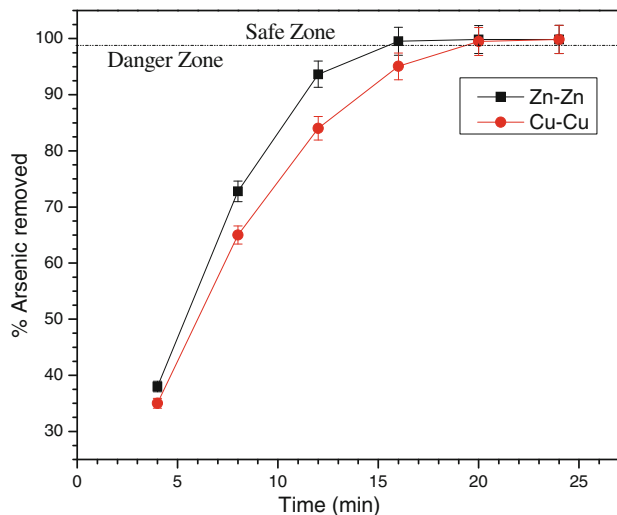


Fig. 4 Effect of processing time on removal percent efficiency of arsenite (Conditions for zinc electrodes 2.0 mg/L initial arsenic concentration, 6.0 pH, 3.0 V applied voltage and 30 °C temperature and for copper electrodes 2.0 mg/L initial arsenic concentration, 7.0 pH, 5.0 V applied voltage and 30 °C temperature)

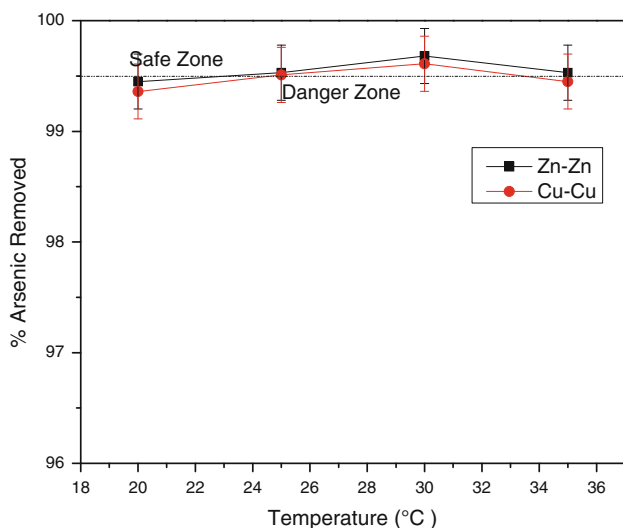


Fig. 5 Effect of temperature on removal percent efficiency of arsenite (Conditions for zinc electrodes 2.0 mg/L initial arsenic concentration, 6.0 pH, 3.0 V applied voltage and 16.0 min processing time and for copper electrodes 2.0 mg/L initial arsenic concentration, 7.0 pH, 5.0 V applied voltage and 20.0 min processing time)

for zinc and copper electrodes, respectively. However, the maximum removal was achieved after 20.0 and 25.0 min by zinc and copper electrodes assemblies, which became constant for further voltage increase. Initially, the removal of arsenic was rapid due the prevalence of arsenic ions, which formed complex with generated $Zn(OH)_2$ and $Cu(OH)_2$. However, as the experiment proceeds arsenic in the solution reduced, while the concentrations of hydrous zinc and copper oxides increased rapidly. As a result of these, the removal became constant after certain time period (Kumar et al. 2004).

Effect of temperature

The effect of temperature on arsenic removal in electro-coagulation is an important issue but nobody carried out it. Therefore, attempts have been made to explore the effects of temperature on arsenite removal. The effect of temperature on electro-coagulation was studied at 20–35 °C temperature range and the results are plotted in Fig. 5. It can be noticed from the figure that the maximum removal efficiency for both the electrodes was found at 30 °C but did not change much in this given temperature range. Of course, the safe zone was with 25, 30 and 35 °C temperatures. The effect of temperature may be explained by considering conductivity. Water conductivity increased at high temperature, leading to an increase in removal efficiency up to 30 °C. On the other hand, further increase in temperature destabilized the arsenic adsorption on hydroxide and, thus, reduced efficiency (Chen 2004).

Validation of the method

The validation of the electro-coagulation method was carried out by controlling various factors. The parameters studied for validation were linearity, specificity, accuracy and precision.

Linearity

Linearity of the data of electro-coagulation method was studied using 2.0–25.0 mg/L concentrations of arsenite. Linearity range was calculated from the calibration curve by least squares linear regression method. The calibration graphs and the negligible scatter of experimental points were considered for calculation of relative standard deviation (RSD), coefficient of determination (r^2) and confidence limits (%). It was observed that calibration graph was linear in the concentration range studied. The values of RSD, coefficient of determination (r^2) and confidence limits (%) were ± 1.50 , 0.9998 and 98.5 %, respectively.

Specificity

The ground water contains many soluble compounds and the method is likely to be effected due to these species. The specificity of the developed electro-coagulation method was ascertained by observing any change in the results due to the presence of various dissolved species in ground water. For this, the effect of various ions on arsenite removal was studied. The common ions used include sodium, potassium, magnesium, calcium, sulphate, phosphate, nitrate and chloride. It was observed that these ions did not affect the removal efficiency of arsenite, indicating the specificity of the reported method. The effect of chloride concentration (50.0–300.0 mg/L) on arsenite removal is shown in Fig. 6. It is clear from this graph that the maximum removal (<99.5 %) for both the electrodes occurred at 200.0 mg/L of sodium chloride. It is due to the fact that conductivity of water increased at increasing amount of sodium chloride. Therefore, 200.0 mg/L of sodium chloride was providing sufficient conductivity for the maximum arsenite removal. The conductivity of 200.0 mg/L of sodium chloride was measured and found to be 1.35 mS/cm. The conductivity of natural water was 1.43 mS/cm, which was comparable to 1.35 mS/cm conductivity of 200.0 mg/L of sodium chloride. The percentage removal of arsenite was <99.5 % for both the electrodes in both cases i.e. with 200.0 mg/L of sodium chloride and the natural ground water. Further increase in chloride concentration (>200.0 mg/L) did not affect arsenite removal, confirming the specificity of this electro-coagulation method.



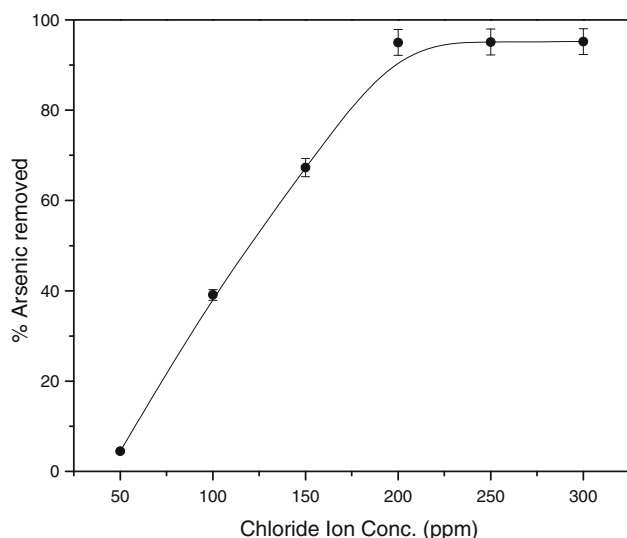


Fig. 6 Effect of chloride ion concentration on arsenite removal (Conditions for zinc electrodes with 2.0 mg/L initial arsenic concentration, 6.0 pH, 3.0 V applied voltage and 16.0 min processing time and 30 °C temperature)

Precision

To calculate precision data, all the experiments were carried out five times. The data was calculated using five sets of experiments for pH, arsenite concentration, voltage and temperature. The values of relative standard deviation (RSD), coefficient of determination (r^2) and confidence limits (%) were calculated. The values of RSD, coefficient of determination (r^2) and confidence limits (%) were 1.50–1.59, 0.9996–0.9998 and 96.0–99.0, respectively. These values indicated electro-coagulation method as the precise one.

Accuracy

Three different concentrations of arsenite (5.0, 10.0 and 15.0 mg/L) were used for determining accuracy of the reported method. The experiments were carried out five times ($n = 5$) with these concentrations. The accuracy was determined by interpolation of the removal capacities of arsenite in all five experimental sets. The percent error was calculated for each concentration and found in the range of 0.50–2.0 %. This range indicates a good accuracy of the developed and reported method.

Secondary contamination of treated water

The electro-coagulated arsenite treated water was investigated for secondary contaminants. Various parameters like conductivity, pH, hardness (temporary and permanent hardness), alkalinity, total dissolved solids (TDS), chloride,

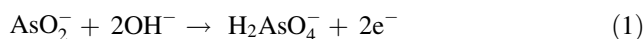
sodium, potassium, calcium and magnesium ions were analyzed before and after the electro-coagulation to notice any change in water quality. It was observed that all these values were almost similar before and after the electro-coagulation with a slight increase in pH, indicating no change in water quality. Moreover, treated water was analyzed for zinc and copper metal ions too. It was observed that both these elements were absent in the treated water. These results dictated that there was no secondary contamination in treated water after electro-coagulation process.

Mechanisms of removal

The removal of arsenite from contaminated water by electro-coagulation involved a little bit complex mechanism. The overall removal of arsenite can be described by dividing mechanism into two parts. The first part was the conversion of arsenite to arsenate while the second part discussed the removal of arsenate.

Conversion of arsenite into arsenate

First of all, arsenite converted into arsenate during electro-coagulation, which was monitored by measuring the concentrations of arsenite and arsenate in electro-coagulation medium. It was observed that arsenite converted into arsenate with respect to electro-coagulation process. Besides, coagulation of arsenate started side by side, which will be discussed in second part of the mechanism. The chemical reaction involving the conversion of arsenite to arsenate is given below.



This is the most abundant arsenate species at 2–7 pH (Sullivan et al. 2010).

Removal of arsenate from contaminated water

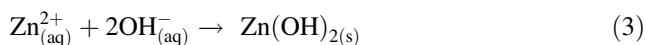
The produced arsenate was removed from water via the generation of coagulant due to oxidation of sacrificial anode. It was a three step process with oxidation of sacrificial anode (formation of coagulant), adsorption/replacement of the contaminants and settling of loosely aggregated mass of coagulant material along with arsenate. During first process, anode material converted into its hydroxides (insoluble in water), which was nothing but coagulant. In second step, arsenate got adsorbed on the surface of coagulant. Besides, arsenate also replaced hydroxyl groups of produced $\text{Zn}(\text{OH})_2$ and $\text{Cu}(\text{OH})_2$ (Eqs. 9 and 10), giving rise to an insoluble ZnHAsO_4^- and CuHAsO_4 complexes (Maldonado-Reyes et al. 2007). Third step comprised the settling of flocs followed by its removal from water by sedimentation

and filtration. The experiments were carried out at 2.0–9.0 pH i.e. under both acidic and alkaline conditions. The removal of arsenite was observed in both acidic and basic mediums, confirming the formation of hydroxides in both the situations. The electrochemical reactions of hydroxide formation under these conditions are shown below.

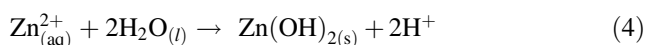
For zinc anode



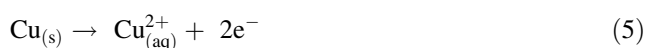
Alkaline conditions:



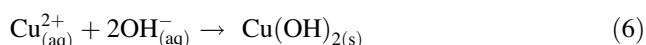
Acidic conditions:



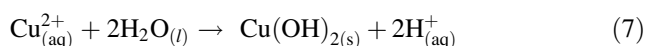
For copper anode



Alkaline conditions:



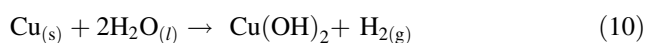
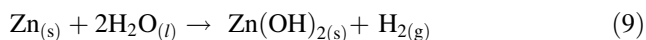
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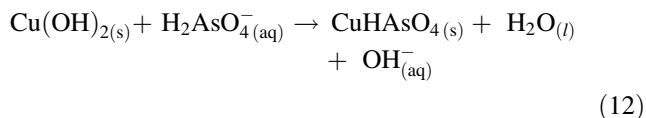
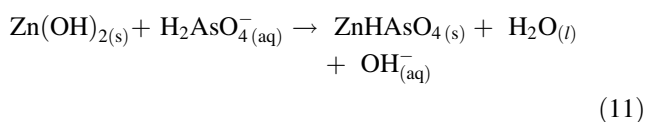
The reactions at the cathode for both Zn and Cu electrodes:



Overall reactions for both Zn and Cu electrodes:



The formation of arsenic complex occurs:



Energy consumption was calculated as per the standard method (Martínez-Villafane et al. 2009). The values of energy consumption for Cu–Cu and Zn–Zn electrodes were found to be 0.704 and 2.66 kWh/L, respectively.

Conclusion

The developed electro-coagulation method for the removal of arsenite was fast, selective, effective and economic.

The optimization of various parameters such as initial concentration of arsenite, voltage, electro-coagulation time, initial pH, and temperature indicated 99.50 % as the safe zone at 2.0 mg/L arsenite concentration. The maximum removals were 99.89 and 99.56 % for zinc and copper electrodes, respectively. But overall zinc electrodes proved much better than copper ones. The best conditions for zinc–zinc electrode assembly were 2.0 mg/L initial arsenite concentration, 16.0-min processing time, 6.0 pH, 3.0-V applied voltage and 30 °C temperature, while these values for copper–copper electrodes were 2.0 mg/L initial arsenite concentration, 20.0-min processing time, 7.0 pH, 5.0-V applied voltage and 30 °C temperature. Briefly, the treated water was safe without any secondary contamination. Therefore, the developed electro-coagulation method can be applied at large scale for removing arsenite.

Acknowledgments The authors are thankful to University Grant Commission (UGC), New Delhi, India for providing BSR Fellowship to Mohd. Asim.

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