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Thermodynamic studies of the strontium adsorption on iron species generated by electrocoagulation

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ABSTRACT

In this research we present and discuss the results of the use of the Electrocoagulation (EC) process for strontium removal from wastewater. The phenomena associated with strontium removal is adsorption on iron magnetic species generated from the dissolution of iron electrodes of the electrolytic cell. With the EC process more than 99 percent of strontium was removed from wastewater without addition of chemical reagents. Sludge generated by EC was characterized by Chemical Analyses, X-Ray Diffraction, Scanning Electron Microscopy and Vibrating Sample Magnetometer (VSM) in order to document the adsorption of strontium on iron-species. The electrogenerated crystalline iron oxides magnetite (Fe_3O_4) and maghemite (γ-Fe_2O_3) powders have higher values for saturation magnetization, but lower values for remanent magnetization and coercive field than commercial strontium hexaferrite with micrometric particle size. The experimental data were correlated employing a Langmuir adsorption isotherm. Thermodynamic parameters such as ΔG^o, ΔH^o and ΔS^o were evaluated ($\Delta G^o = -23.49$ KJ/mol, $\Delta H^o = -24.92$ KJ/mol and $\Delta S^o = -0.00479$ KJ/mol K) and the adsorption process was found to be exothermic and spontaneous in nature. In addition, results indirectly indicate the feasibility of the EC process for strontium removal.

Keywords: Electrocoagulation; Strontium; Adsorption isotherm; Thermodynamics

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1. Introduction

In any place, where there are located plants that produce strontium carbonate, there is an excessive amount of strontium anions in the wastewater and the exposure to this, through drinking water poses a threat to human health. To prevent harmful consequences, especially problems that alter the growth of bones in children, the environment protection agency (EPA) fixed a maximum acceptable concentration of 4,000 micrograms per liter of strontium in drinking water (4,000 μg/l) [1].

The strontium ion is slightly toxic and the transport of these ions in the environment is strongly influenced by its chemical form. The toxic action of strontium compounds is closely associated with the anion of the compound concerned. Strontium behaves similarly to calcium both chemically and biologically. The human body absorbs strontium with the same easiness that absorbs calcium, but the stable forms of the strontium, seem not to represent a threat for human health [2].

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Strontium is deposited preferentially in bones and teeth. Strontium salts are not readily absorbed via the intestinal tract. Symptoms of acute toxicity are excessive salivation, vomiting, colic and diarrhea, and possibly respiratory failure. Strontium oxide and strontium hydroxide have a strongly irritant effect on the skin, mucous membranes and especially in the eyes.

Elimination of species of heavy metals present in polluted waters and aquifers has been constituted in an indispensable requirement in any outline of waters treatment, given the toxicity associated to those species and to their linking with specific diseases. The necessity of incorporation of simpler and decentralized processes has opened new challenges. It is therefore necessary to optimize the water purification methods for human consumption, as well as for the cleaning of residual waters of industrial effluents before discharging them toward other aquatic systems.

Various techniques have been developed to remove strontium and improve the quality of drinking water like chemical precipitation, coagulant operation and adsorption. Strontium ions removal from aqueous solutions has been traditionally carried out by chemical precipitation, ion exchange, membrane processes, which are expensive and inefficient, especially for low strength wastewaters. These techniques are compared in Table 1 [3,4]. Recovery efficiency ranges from 70% to 98%, some of them need the use of reagents, the support of other techniques to enhance performance and besides the required time in some cases is up to 24 h. Although these techniques have been widely applied, they present many limitations. For example, the regeneration of adsorbents by chemical and thermal procedures is expensive, while chemical coagulation may induce secondary pollution by using an excess of coagulants and it usually generates large volumes of sludge. It is also worthy to notice that adsorption is not able to remove strontium at high concentration.

These drawbacks have forced municipalities and various industries to search for effective alternative treatment technologies for strontium removal, ideally by electrochemical methods. Electrocoagulation (EC) process appears to be a good alternative for strontium removal, and thus may be beneficial for communities with better access to electricity than to chemicals. EC process appears therefore as an alternative due to its cost and efficiency.

Electrocoagulation or enhanced coagulation accompanied by electro flotation is an emerging electrochemical water and waste water treatment technology. 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 EC [5]. This electrochemical method of waste removal requires small quantities of salt to increase the conductivity of the solution. Maintenance and operation of the EC cells are relatively simple. Since 1970, this technology has become increasingly popular around the world for treatment of industrial wastewater, electrocoagulation processes offer significant potential for removing soluble ionic species from solution, particularly heavy metals [6]. EC operating conditions are highly dependent on the chemistry of the aqueous medium, especially conductivity and pH.

Table 1
Techniques for strontium removal

Process	Advantages	Disadvantages
Solvent extraction	Well-defined medium and capacity. High removal of strontium.	Very high capital cost. Need solvents and additives to increase the removal.
Precipitation strontium hydroxide	Simple method for the separation. Centrifugation and filtration. Common chemicals are available.	Sedimentation and filtration needed. Readjustment of pH is required. Low removal of strontium.
Ion exchange	Well-defined medium and capacity. Ion specific resin to remove strontium. The process dependent on pH of the solution.	Short life of resins. High cost for resins. Requires high tech operation and maintenance.
Reverse osmosis	Highest water quality; treats wide range of dissolved salts, minerals, turbidity. No toxic solid waste is produced.	Expensive to install and operation pressure control to meet membrane tolerance. Inefficient for low strength wastewater.

Other important characteristics such as particle size, type of electrodes, retention time, plate spacing and chemical constituent concentrations dictate the operating parameters of the process [7]. The EC process operates on the principle that cations produced electrolytically from iron and/or aluminum anodes enhance the coagulation of contaminants from an aqueous medium. The sacrificial metal anodes are used to continuously produce polyvalent metal cations in the vicinity of the anode. These cations facilitate coagulation by neutralizing the negatively charged particles that are carried toward the anodes by electrophoretic motion. In the flowing EC techniques, the production of polyvalent cations from the oxidation of the sacrificial anodes (Fe and/or Al) and the production of electrolysis gases (H_2 and O_2) are directly proportional to the amount of current applied (Faraday's law). A schematic representation of the EC process is shown in Fig. 1.

The removal mechanisms in EC may involve oxidation, reduction, decomposition, deposition, coagulation, absorption, adsorption, precipitation and flotation [8]. However, it is the reactions of the metal ions that enhance the formation of the coagulant. The metal cations react to yield both soluble and insoluble hydroxides that will react with or adsorb pollutants 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 [9]. As mentioned above, the gas bubbles produced by electrolysis carry the pollutant to the top of the solution where it is concentrated, collected and removed.

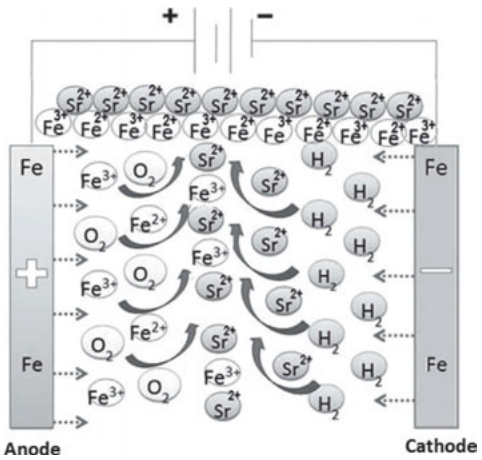

Fig. 1. An illustration of the EC mechanism (arrows indicate the migration of electrolysis gases O_2 and H_2).

The chemical reactions that have been proposed to describe EC mechanisms for the production of $H_{2(g)}$, $OH^-_{(aq)}$ and $H^+_{(aq)}$ at different pH values are [10]:

For pH <4

Anode: iron oxidation

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

$$Fe \rightarrow Fe^{+3} + 3e \tag{2}$$

Cathode: hydrogen evolution

$$2H^+ + 2e^- \rightarrow H_{2(g)} \uparrow \tag{3}$$

For 4<pH< 7:
Anode: reactions (1) and (2)
Comments:
In fact iron also undergoes hydrolysis

$$Fe + 6H_2O \rightarrow Fe\left(H_2O\right)_4\left(OH\right)_{2(aq)} + 2H^{+1} + 2e^{-1} \tag{4}$$

$$Fe + 6H_2O \rightarrow Fe\left(H_2O\right)_3\left(OH\right)_{3(aq)} + 3H^{+1} + 3e^{-1} \tag{5}$$

Fe(III) hydroxide begins to precipitate floc with yellowish color.

$$Fe\left(H_2O\right)_3\left(OH\right)_{3(aq)} \rightarrow Fe\left(H_2O\right)_3\left(OH\right)_{3(s)} \tag{6}$$

"Rust" may also be formed.

$$2Fe\left(H_2O\right)_3\left(OH\right)_3 \leftrightarrow Fe_2O_3\left(H_2O\right)_6 \tag{7}$$

Cathode:

$$2H^+ + 2e^- \rightarrow H_{2(g)} \uparrow \tag{8}$$

More hydrogen evolution takes place but $[H^+]$ now comes from weak acids and iron hydrolysis.
For 6<pH< 9:
Anode: reactions (1) and (2)
Comments:
Precipitation of Fe(III) hydroxide (7) continues, and Fe(II) hydroxide precipitation also occurs presenting a dark green floc.

$$Fe\left(H_2O\right)_4\left(OH\right)_{2(aq)} \rightarrow Fe\left(H_2O\right)_4\left(OH\right)_{2(s)} \tag{9}$$

The pH for minimum solubility of Fe(OH)n is in the range of 7–8. EC floc is formed due to the polymerization of iron oxyhydroxides.

Formation of rust (dehydrated hydroxides) occurs as shown in the following:

$$2\mathrm{Fe(OH)_3} \leftrightarrow \mathrm{Fe_2O_3} + 3\mathrm{H_2O} \quad (\text{hematite, maghemite}) \qquad (10)$$

$$\mathrm{Fe(OH)_2} \leftrightarrow \mathrm{FeO} + \mathrm{H_2O} \qquad (11)$$

$$2\mathrm{Fe(OH)_3} + \mathrm{Fe(OH)_2} \leftrightarrow \mathrm{Fe_3O_4} + 4\mathrm{H_2O} \quad (\text{magnetite}) \qquad (12)$$

$$\mathrm{Fe(OH)_3} \leftrightarrow \mathrm{FeO(OH)} + \mathrm{H_2O} \quad (\text{goethite, lepidocrocite}) \qquad (13)$$

Hematite, maghemite, rust, magnetite, lepidocrocite and goethite have been identified as EC by-products [11]

Cathode:

$$2\mathrm{H^+} + 2e^- \rightarrow \mathrm{H_{2(g)}} \uparrow \qquad (14)$$

More hydrogen evolution takes place but $[H^+]$ now comes from iron hydrolysis and weak acids.

Overall reactions are:

$$\mathrm{Fe} + 6\mathrm{H_2O} \rightarrow \mathrm{Fe(H_2O)_4(OH)_{2(s)}} + \mathrm{H_{2(g)}} \uparrow \qquad (15)$$

$$\mathrm{Fe} + 6\mathrm{H_2O} \rightarrow \mathrm{Fe(H_2O)_3(OH)_{3(s)}} + 1\tfrac{1}{2}\ \mathrm{H_{2(g)}} \uparrow \qquad (16)$$

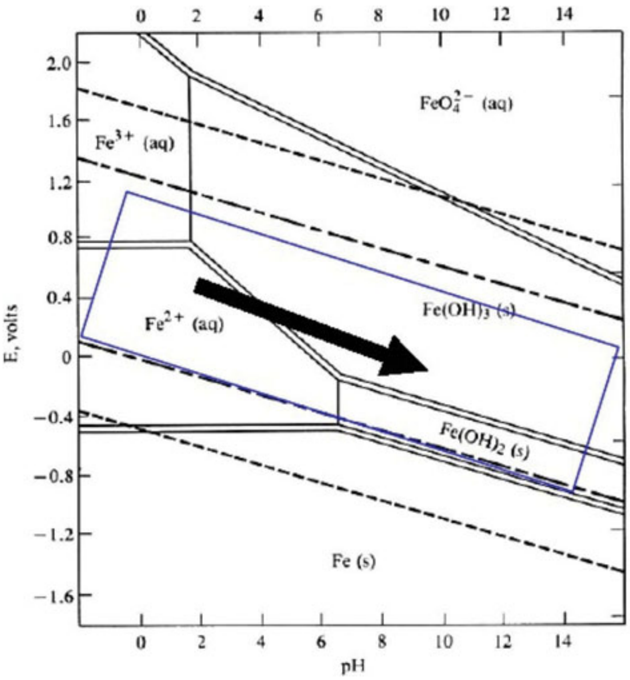

Fig. 2. Iron pourbaix diagram, showing the region and direction in which the EC process proceeds.

Conditions throughout the cell are not constant, concentrations, species and pH are changing. This may be illustrated with an iron pourbaix diagram. The process seems to occur in a region parallel to the hydrogen evolution line, and conditions change to the right as highlighted in Fig. 2.

Generally in the EC process bipolar electrodes are used. It has been reported that cells with bipolar electrodes connected in series operating at relatively low current densities produced iron or aluminum coagulant more effectively, more rapidly and more economically when compared to chemical coagulation [12].

2. Materials and methods

2.1. Experimental setup and methods of analysis

Electrocoagulation was carried out in a beaker size reactor (400 ml), equipped with two iron electrodes plates dipped in wastewater of 7 cm by 4 cm, the gap between the anode and cathode was 5 mm. The entire electrode assembly was fitted on non-conducting wedges and hanged from the top of the EC beaker. The assembly was connected to a DC power source. The experimental setup of the electrochemical cell is presented in Fig. 3. The current and voltage during the EC process were measured and recorded, using Cen-Tech multimeters. The pH values of the solution before and after EC were measured with a VWR scientific 8005 pH meter.

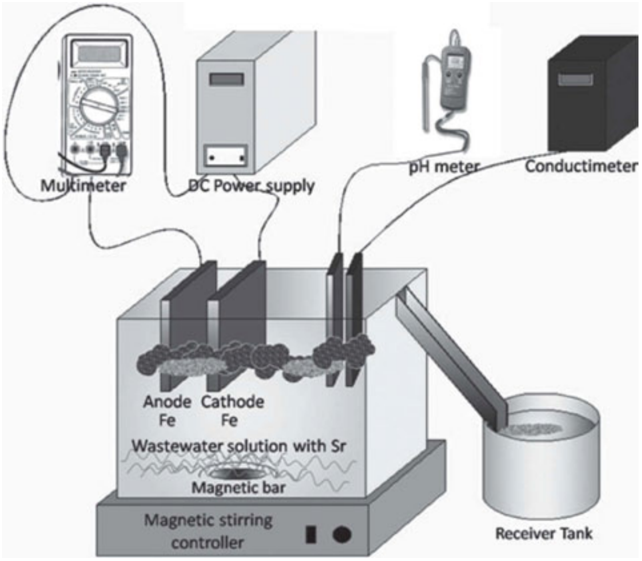

Fig. 3. Experimental set-up for strontium removal two-electrode electrocoagulation cell.

Table 2
Conditions for strontium adsorption test

Samples	Concentration (mg/l)	Temperature (ºC)	Intensity current (Amperes)	Voltage (Volts)	Time (min)
1	1.14	21	0.41	11.5	30
2	1.18	21	0.44	10.83	30
3	1.25	21	0.49	11.1	30
4	10	25	0.54	12.25	30
5	20	25	0.7	13.78	30
6	40	25	0.75	15.22	30
7	50	25	0.9	16.25	30
8	62	25	1	17.84	30

To determine the adsorption of strontium on iron species these steps were followed: 1) electrolytic solutions were prepared with strontium hydroxide, (reagent grade with a purity of 99% made by Monterrey chemical products) and deionized water with a conductivity of 0.95 µS (Aldrich Chemical Co. 99.5+%, A.C.S. reagent, Lot # DI02350AI).

Strontium concentrations in the prepared solutions were 1, 10, 20, 40 and 62 mg/l. 1) Conductivity was adjusted by adding one gram of NaCl per liter of water (Fisher, 99.8% A.C.S. Certified, lot #995007). 2) All EC runs were performed at a constant temperature of 25ºC and a stirring speed of 200 rpm. 3) Solution and solids were separated by filtration through cellulose filter paper. 4) The sludge from the EC was dried in an oven. 5) The solid precipitate was characterized by XRD, SEM/EDAX, VS Mand 6) the filtrate was used for determining the amount of residual strontium. Table 2 presents the initial conditions for EC, voltage, current and residence time for each run.

3. Results and discussion

3.1. Calculus of strontium adsorbed on iron species (N)

The amount of strontium adsorbed at equilibrium N (mg Sr/g Fe) on iron species was calculated from the following equation:

$$N = V * \left(\frac{C_0 - C_e}{W}\right) \tag{17}$$

where C_0 (mg/l^1) is the initial concentration of strontium in the aqueous phase, V (l) is the volume of the liquid phase, C_e (mg/l^1) is the Sr concentration at equilibrium in the aqueous phase and W (g) is the mass of adsorbent used (in this case the Fe dissolved from the electrodes during the EC).

Table 3
Strontium moles adsorbed on iron species (N)

Sample	C_{0Sr} (mg/l)	Ce_{Sr} (mg/l)	Sr Removal (%)	W (g)	N (mg/g)
1	1.14	0.006	99.5	0.143	31.77
2	1.18	0.043	96.35	0.153	27.97
3	1.25	0.058	95.35	0.170	29.66
4	10	0.588	94.12	0.188	199.99
5	20	1.545	92.28	0.243	303.37
6	40	3.826	90.44	0.261	553.63
7	50	5.21	89.58	0.313	564.38
8	62	12.549	87.45	0.35	571.61

The metal amount of iron that dissolves (W in Table 3), depends on the quantity of electricity that passes across the electrolytic solution and the residence time. A simple relation between the current density and the quantity of substance dissolved (g) may be established from the Faraday's law [13]:

$$W = \frac{(D * A * t * M)}{n * F} \tag{18}$$

where W is the mass of the sacrificial electrode dissolved (g), D the current density (A/cm^2), A represents the surface area of the anode (cm), t the EC treatment time (seconds), M the molar mass of the electrode material, n the valence of the dissolving metal and F the Faraday's constant (96,485 coulombs**).**

The number of strontium moles adsorbed on iron species are calculate by Eq. (17) and presented in Table 3. Results show that N increases with increasing concentration.

3.2. Langmuir's isotherm

The Langmuir equation is given in the following equation:

$$N = \left(\frac{N_{\max} K_L C_e}{1 + K_L C_e} \right) \tag{19}$$

where N is the solid phase adsorbate concentration in equilibrium (mg/g), $N_{\max}$ the maximum adsorption capacity corresponding to complete monolayer coverage on the surface (mg/g), C_e the concentration of adsorbate at equilibrium (mg/l) and K_L is the Langmuir constant (l/mg). Eq. (19) can be rearranged to a linear form:

$$\frac{C_e}{N} = \frac{1}{N_{\max} K_L} + \frac{C_e}{N_{\max}} \tag{20}$$

Following the model of the Langmuir's isotherm Figs. (4 and 5) shows the plot and results of the strontium adsorption capacity on iron species.

The constants can be evaluated from the intercepts and the slopes of the linear plots of C_e/N versus C_e. It was observed that the equilibrium adsorption data followed Langmuir's Isotherm. Table 4 shows the results of the linear regression. The value of $N_{\max}$ indicates the maximum adsorption capacity corresponding to complete monolayer coverage on the iron surface. The value of K_L is the constant of adsorption of Langmuir.

Table 4
Linear regression results for strontium adsorption

R^2	0.991
$N_{\max}$ (mg Sr /g Fe)	625
K_L (l/mg)	14.54

Table 5
Covered fraction θ for strontium

$N(\mathrm{mg_{Sr}/gr_{Fe}})$	$N_{\max}$ $(\mathrm{mg_{Sr}/gr_{Fe}})$	Θ
27.97	625	0.045
29.66	625	0.047
31.77	625	0.05
199.99	625	0.32
303.37	625	0.485
553.63	625	0.886
564.38	625	0.903
571.61	625	0.915

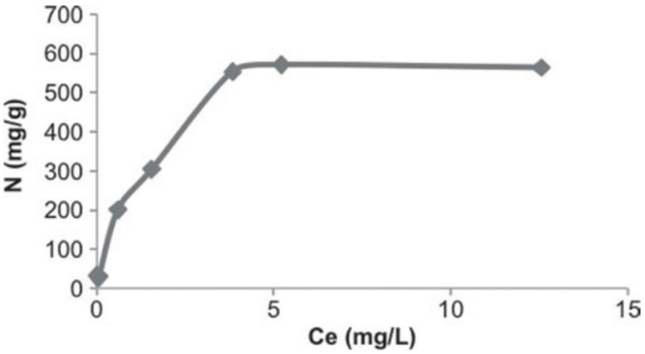

Fig. 4. Plot of number of strontium moles adsorbed on iron species versus concentration of adsorbate at equilibrium.

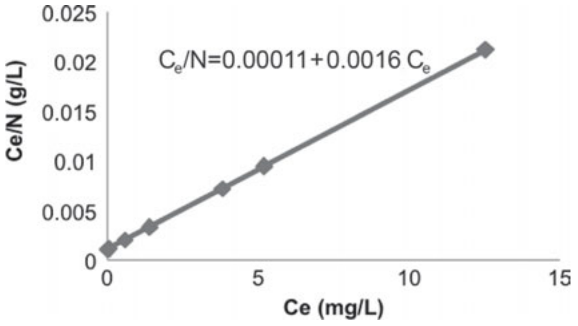

Fig. 5. Langmuir isotherm for strontium adsorption on iron species.

3.3. Covered fraction of strontium on iron species

The equation $\theta = N/N_{\max}$ calculates the covered fraction θ (Table 5). According to the obtained results of N and $N_{\max}$, it may be observed that the covered fraction θ approaches 1 as concentration increases.

3.4. Thermodynamics results

The thermodynamic parameters such as change in standard free energy ($\Delta G°$), enthalpy ($\Delta H°$) and entropy ($\Delta S°$) were determined by using the following equations [14]:

$$\ln\left(\frac{1}{K_L}\right) = \frac{\Delta G^{\circ}}{RT} \tag{21}$$

$$\ln\left(K_L\right) = K_{L0} - \left(\frac{\Delta H^{\circ}}{RT}\right) \tag{22}$$

$$\Delta G^{\circ}{}_{\mathrm{ads}} = \Delta H^{\circ}{}_{\mathrm{ads}} - T\Delta S^{\circ}{}_{\mathrm{ads}} \tag{23}$$

where R is the gas constant, T(°K) the absolute temperature, K_L and K_W are Langmuir's constant to final and initial concentration. Table 6 show the negative value of obtained, indicating that the strontium adsorption on iron species by EC is a spontaneous process. The negative value of represents an exothermic process and

Table 6
Thermodynamic parameters for strontium adsorption

ΔH_{ads} (kJ/mol)	ΔG_{ads} (KJ/mol)	ΔS_{ads} (KJ/mol°K)
–24.92	–23.49	–0.00479

suggests a physisorption behavior according with the liberated heat between –20 and –40 KJ/mol [15]. The negative value of $\Delta S°$ suggests that a significant change does not happen in the internal structure of the adsorbent during the adsorption of Sr.

3.5. Product characterization

In this study, Powder X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Vibrating Sample Magnetometer (VSM) were used to characterize the solid products formed from carbon steel electrodes during EC.

3.5.1. X-Ray diffraction results

For the strontium samples, a diffractogram was obtained with a Bruker-AXS D4 Endeavor diffractometer operating with CuK_α radiation source filtered with a graphite monochromator (λ = 1.5406 Å). The samples were ground to a fine powder in isopropyl alcohol (Sigma-Aldrich) and loaded into a sample holder. The XRD scans were recorded from 10 to 70 degrees 2θ with 0.020° step-width, and with 10 sec counting time for every step-width. Experiments were run at 40 kV and 40 mA. Fig. 6 shows the powder diffractogram of sample 5. The species identified were magnetite and maghemite in crystalline form. The positions and relative intensities of all diffraction peaks match well with those from the reference database JDPCS card (19–0629) for magnetite and (39–1346) for maghemite.

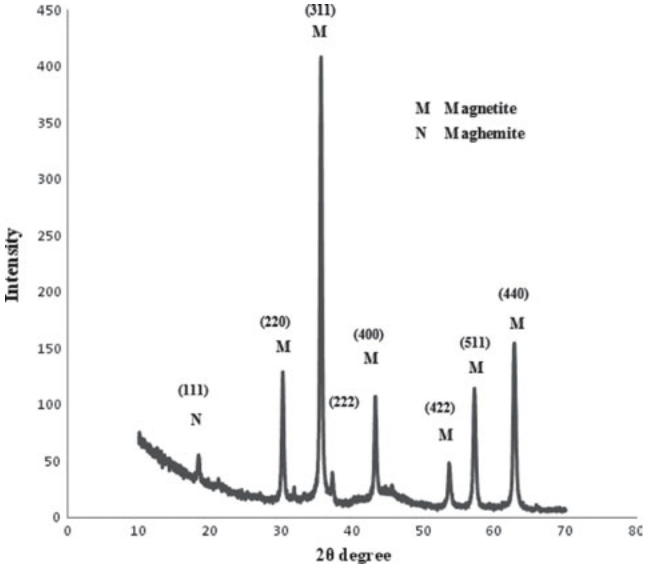

Fig. 6. XRD diffractogram of iron species of sample 5.

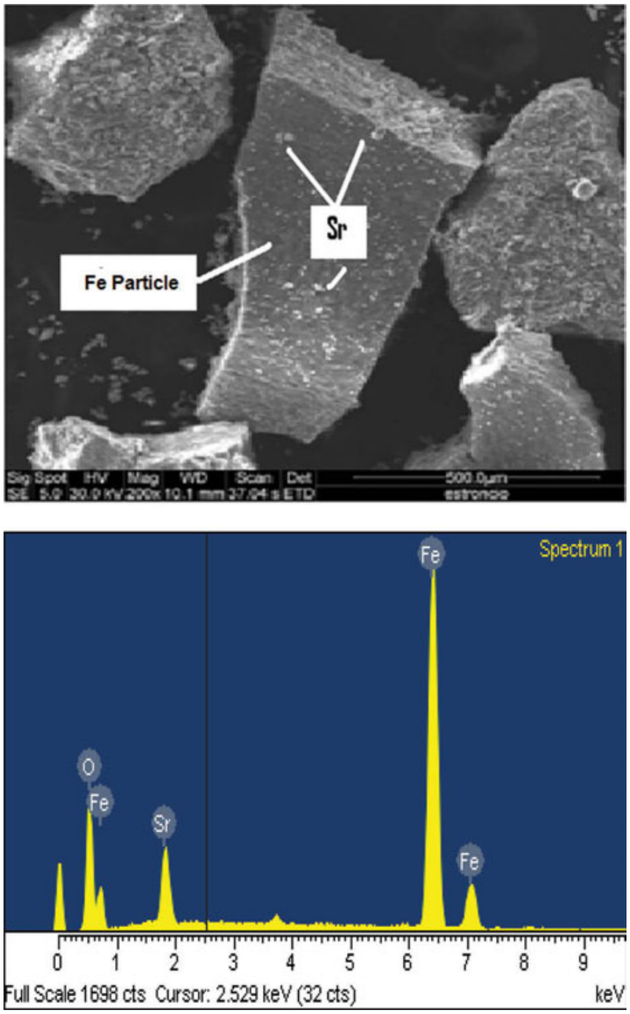

Fig. 7. SEM image and EDAX spectra for by-products generated from sample 5.

3.5.2. Scanning electron microscopy (SEM) and energy dispersive analysis of X-rays (EDAX).

Fig. 7 show the SEM image of the precipitates obtained during the EC treatment of sample 5 and the corresponding EDAX spectrum. As can be seen the EDAX analysis exhibits a content of Fe, Sr and Oxigen.

3.5.3. Vibrating sample magnetometer (VSM)

The magnetic properties of precursor powder sample obtained, was studied with a vibrating sample magnetometer (Lake Shore 7300) with a maximum field of 12 KOe. Fig. 8 illustrates the saturation magnetization (M_s) and coercivity.

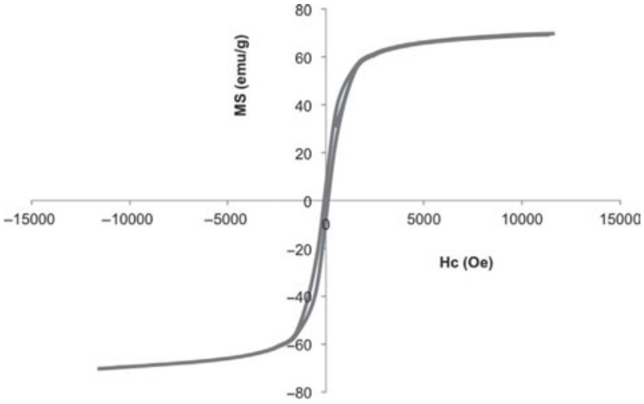

Fig. 8. Hysteresis loop shows the saturation magnetization for by-products generated from sample 5.

Results showed that the best value for coercivity is 65.49 Oe, for saturation magnetization 69.9 emu/g and for retentivity magnetization 2.34 emu/g which indicates that the product obtained has an acceptable magnetization but shows a low coercivity. The M_s of 69.9 is close to the theoretical M_s value of 74.3 for strontium ferrite single crystal [16].

4. Conclusions

EC was tested as an alternative method to remove strontium from wastewater. This study indicates that strontium can be succesfully adsorbed on iron species by electrocoagulation process. Based on the Langmuir's isotherm analysis, the maxim adsorption capacity was determinated to be 625 mg/g for strontium. The negative value of ΔG^o obtained indicated that strontium adsorption on iron species by EC is a spontaneous process. The negative values of ΔH^o represents an exothermic process and suggests a physisorption behavior. The negative values of ΔS^o suggests that a significant change does not happen in the internal structure of the adsorbent during the adsorption of Sr. X-Ray Diffraction, Scanning Electronic Microscopy, Transmission Electronic Microscopy and Vibrating Sample Magnetometer techniques demonstrate that the formed species are of magnetic type with good saturation magnetization, but lower remanent magnetization and coercive field, which adsorbed the strontium particles on his surface due to the electrostatic attraction between both metals. The 95% average strontium removal in the experimental EC reactor is usually completed within 30 min or less for most experiments with a current efficiency of 100%.

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Symbols

N — Number of strontium moles adsorbed on iron species (mg/g^1)
C_0 — Initial concentrations of strontium (mg/l^1)
C_e — Equilibrium concentrations of strontium (mg/l^1)
V — Volume of the solution (l)
W — Mass of the sacrificial electrode dissolved (g)
D — Current density (A/cm^2)
A — Surface area of the anode (cm^2)
t — EC treatment time (seconds)
M — Molar mass of the electrode material ($55.84\ g/mol^1$)
n — Valence of the dissolving metal ion (n=3)
F — Faraday's constant (96,485 coulombs)
N_{max} — Maximum adsorption capacity (mg/g^1)
K_L — Langmuir adsorption constant at final concentration
K_{L0} — Langmuir adsorption constant at initial concentration
θ — Covered fraction
ΔG^o — Standard free energy (KJ/mol^1)
ΔH^o — Standard enthalpy (KJ/mol^1)
ΔS^o — Standard entropy ($KJ/mol^1/K^1$)
R — Universal gas constant ($J/mol^1/°K^1$)
M_s — Saturation Magnetization (emu/g)
H_c — Coercitive Force (Oe)

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