DOI: 10.17580/cisisr.2020.02.13

EXTRACTION BY ELECTROFLOTATION OF IRON, CHROMIUM AND ALUMINIUM HYDROXIDES FROM AQUEOUS SOLUTIONS OF SODIUM CHLORIDES AND SULPHATES IN THE PRESENCE OF Mg^{2+}, Ca^{2+} AND SURFACTANTS OF DIFFERENT TYPES

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Key words:

electroflotation, hydroxides, Fe, Cr, surfactants, electrolytes, sodium chloride, sodium sulphate, reagents, magnesium hydroxide, calcium hydroxide, electroflotation kinetics.

ABSTRACT

This paper examines an electroflotation process in which iron hydroxides are recovered from chloride-sulphate solutions in the presence of surfactants of different types, as well as calcium and magnesium cations used as reagents. It was found that calcium and magnesium cations lower the recovery of iron hydroxides by 30 to 40%. Introduction of surfactants intensifies the recovery of iron (III) hydroxides making it reach 89 to 98%. It was found that the anionic surfactant NaDDS produces a positive effect on the recovery of chromium (III) hydroxides by electroflotation by raising the recovery rate by 30 to 40% and expanding the effective pH range to 9. NaDDS improves the recovery of the mixture of iron, chromium and aluminium hydroxides in electrolytic solutions (10 g/l). Water treatment procedures are described aimed at removing iron (II, III) ions and the iron/chromium mixture.

Introduction

The wastewater generated as a result of steel surface treatment processes contains Fe^{2+}, Fe^{3+}, Cr^{3+}, Cr^{6+}, Ni^{2+}, Co^{2+}, as well as ions of some other metals [1, 2]. It is usually due to the use of HCl, H_2SO_4, H_3PO_4 that base electrolytes form in wastewater. Neutralization of the former results in the formation of NaCl, Na_2SO_4 (1–5 g/l), soluble salts of Mg^{2+}, Ca^{2+} (0.1–0.5 g/l), chlorides and carbonates [3, 4]. Wastewater generated by mining and metallurgical complexes has a more complex composition. Thus, it may contain oxides of Fe, Ca, Mg, Si, sulphides of Zn, Pb, Cu, Mn, hydroxides of Ti, Al, Fe [5, 6]. Even though wastewater contains ions of numerous different heavy and non-ferrous metals, it also contains 20 to 30% of iron (II, III) oxides and hydroxides. Some researchers [7] are working on developing basic techniques to help reduce the environmental impact of the ferrous metals industry. They are also working on developing the concept of waste-free metals production [8]. The majority of professionals in the field resort to modern technology to neutralize liquid, solid and gaseous wastes.

Reagents made with Fe(III), $FeCl_3$, $Fe_2(SO_4)_3$ are used as coagulants in wastewater treatment processes that help remove suspended particles, emulsions and surfactants [9, 10]. Fe(III) ions often enter the wastewater in great quantities due to the use of electrocoagulation and galvanocoagulation. In these processes, iron-containing wastes (shavings, scrap, oxides) are used as anodes [6, 9–12]. A study is carried out that looks at the steel industry wastes utilized as sorbents (iron oxides, hydroxides) in industrial water circuits [13].

All the above suggests that techniques are required that would help extract the disperse phase of $Fe(OH)_3$ from wastewater ponds. There are certain techniques for extracting the disperse phase of $Fe(OH)_3$ from aqueous solutions with complex compositions. They include electrochemical, membrane and flotation techniques.

The advantages and drawbacks of the above mentioned techniques are considered in detail in the following reviews and monographs: [14-17].

Analysis shows that the flotation process delivers certain advantages when it is used to remove suspended particles from wastewater in the presence of organic components, surfactants (SAA), lubricoolants and solvents.

This paper describes the findings of some recent studies that looked at the use of electroflotation to recover iron, chromium and aluminium hydroxides from aqueous electrolytic solutions, as well as at some wastewater treatment processes.

The paper examines the process of recovering iron hydroxides ($Fe(OH)_3$, $Al(OH)_3$, $Cr(OH)_3$) in the NaCl and Na_2SO_4 electrolytes in the presence of surfactants and the Ca^{2+}, Mg^{2+} cations by electroflotation.

The above systems are often used for wastewater treatment by chemical, mining and metallurgical industries. Thus, for instance, when big pipes are treated in acid solutions (HCl, H_2SO_4) before protective coatings are installed on them, the Fe^{2+} and Fe^{3+} ions, sulphuric and hydrochloric acids, as well as iron oxides and other contaminants enter the wastewater as a result. The amount of generated wastewater can reach dozens of cubic metres an hour, as in the case of Novolipetsk Iron & Steel Works.

Research methods

Both laboratory and pilot units were used to study the electroflotation process.

The process efficiency was analyzed based on the recovery rate α (%), which is calculated with the help of the following formula (1):

$$\alpha = (C_{\text{initial}} - C_{\text{final}})/C_{\text{initial}} \cdot 100\%, \qquad (1)$$

The concentration of iron, chromium and aluminium was determined by means of atomic adsorption analysis on the KVANT-AFA unit (Russia) available at the Mendeleev University's Shared Knowledge Centre. Detailed descriptions of the techniques are given in the papers listed in the references.

Experimental results

A great number of papers and reviews [18–20] have been published that looked at the modelling of electroflotation processes, process kinetics and extraction of the disperse phase with the help of electrochemically generated H_2, O_2 bubbles. However, because of changing particle size, bubbles in time, trapping efficiency and a number of other parameters, it can be difficult to calculate certain parameters.

Analysis of the reviews shows that a great number of factors influence the disperse phase (MeX) trapping efficiency and the likelihood of a particle/bubble (H_2, O_2) complex being formed. Such factors include pH of the medium, the type of electrolyte, salt composition, cations, anions, flocculants and surfactants. [21–24]

The key steps towards an enhanced electroflotation process performance include the following: gas saturation control to ensure an efficient H_2-, O_2-to-particle contact; an increased particle size (flocculants, pH, coagulants); a changed ζ–potential (pH, surfactants, cations, anions); surface hydrophobization (anionic, cationic, nonionic surfactants).

Let's consider the results of experiments that looked at the extraction by electroflotation of iron and chromium hydroxides in the presence of alkali metal cations, which can often be found in wastewater generated by steel processing facilities or are used as precipitation reagents for metals and as neutralization reagents for sulphuric and hydrochloric acids [23, 24].

The study that looked at how the concentration of magnesium ions influences the extraction by electroflotation of iron hydroxides in sodium chloride solutions shows that the recovery rate tends to decrease as the concentration of Mg^{2+} rises from 0.1 to 1 g/l.

Analysis shows that Mg^{2+} ions inhibit the extraction of $Fe(OH)_3$ by electroflotation bringing the recovery rate down from 94 to 50%. This effect is associated with the adsorption of Mg^{2+} ions on $Fe(OH)_3$ which leads to a rise in the positive value of the ζ–potential to +20...+25 mV. $Fe(OH)_3$ has a negative potential, which is equal to –2 mV in NaCl solution at pH = 7 ± 0.5, and to –8 mV in sodium sulphate solutions. The iron hydroxide particle size at pH 7–8 is 45–50 μm.

A similar inhibiting effect was observed in the case of calcium and barium ions.

The electroflotation efficiency sees a dramatic decrease within the range of concentrations up to 500 mg/l, in which the adsorption of Mg^{2+}, Ca^{2+}, Ba^{2+} ions on $Fe(OH)_3$ is maximum. At higher concentrations (>500 mg/l), the adsorption of Mg^{2+}, Ca^{2+}, Ba^{2+} remains almost unchanged as it is already at its highest.

Introduction of surfactants (anionic surfactants in the first place) leads to a higher $Fe(OH)_3$ recovery rate due to surfactant adsorption through substitution of water molecules and surface hydrophobization. Thus, when the concentration of metals is 0.5 g/l, the recovery rate rises to 98% in the presence of Ba^{2+}, to 89% in the presence of Ca^{2+} and to 67% in the presence of Mg^{2+}.

The results of experiments that looked at the adsorption of surfactants on iron and aluminium hydroxides are described in the following papers [25, 26].

Table 1 shows how surfactants of different types influence the extraction by electroflotation of $Fe(OH)_3$ in the presence of Mg^{2+} , Ca^{2+}.

The main positive effect produced by surfactants includes surface hydrophobization. This is especially true for anionic and nonanionic surfactants as they help remove H_2O molecules from the $Fe(OH)_3$ surface.

Table 1. **Effect of surfactants of different types and calcium and magnesium ions on the recovery of $Fe(OH)_3$ by electroflotation**

Time τ, min	Recovery rate, α,%							
	Mg^{2+}				Ca^{2+}			
	w/o	cat.	an.	nonion.	w/o	cat.	an.	nonion.
5	50	60	72	40	50	53	73	80
10	68	72	76	62	70	94	80	95
20	75	85	89	64	75	76	90	98

Conditions: *c* (Fe^{3+}) — 100 mg/l, *c* (Me^{2+}) — 0.25 g/l; *c* (SAA) — 5 mg/l, *c* (NaCl) — 1 g/l, pH — 7.0, Jv — 0.4 A/l

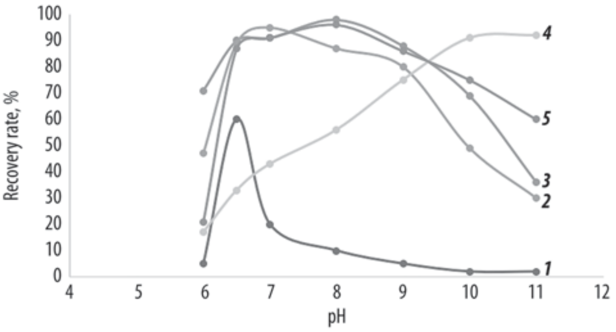

Fig. 1. Effect of pH and surfactants of different types on the $Cr(OH)_3$ recovery rate (Conditions: *c* (Cr^{3+}) — 50 mg/l; *c* (Na_2SO_4) —1 g/l; *c* (SAA) — 5 mg/l; J_v — 0.4 A/l; τ — 20 min):
1 — without SAA; *2* — NaDDS; *3* — NaDBS; *4* — katamin AB; *5* — NaDBS + katamin AB

A positive effect of surfactants can also be observed at low concentrations (5 mg/l) during electroflotation of the $Cr(OH)_3$ deposit in Na_2SO_4 solutions. It should be noted that chromium (III) hydroxide is not easily recoverable from sulphate solutions due to the disperse phase particles being too small: the average particle diameter is 13–14 µm at the pH of 7–8, the electrokinetic potential in a 1 g/l Na_2SO_4 solution is –5 to –7 mV.

Fig. 1 shows how the $Cr(OH)_3$ recovery rate changes in solutions with different pH values in the presence of surfactants.

Introduction of surfactant leads to a 30–40% increase in the $Cr(OH)_3$ recovery rate and a wider pH range associated with higher recovery rates (pH = 6–9). When the anionic surfactant NaDDS and the anionic flocculant M-10 are added, the size of the chromium hydroxide particles increases to 72 and 90 µm, correspondingly.

The presence of anionic and cationic surfactants at the same time helps expand the pH range to 10–11, in which high recovery rates are observed. Introduction of reagents containing Mg^{2+} and Ca^{2+} inhibits the electroflotation process in the case of both $Fe(OH)_3$ and $Cr(OH)_3$. The recovery rates can reach 18–20% (48–50% with no additives used) (see **Table 2**).

Table 2. **Effect of surfactants of different types on the extraction of $Cr(OH)_3$ by electroflotation in the presence of excess calcium and magnesium ions**

Time τ, min	Recovery rate, α,%							
	Mg^{2+}				Ca^{2+}			
	w/o SAA	cat.	an.	nonion.	w/o	cat.	an.	nonion.
5	11	14	88	10	11	12	96	11
10	14	18	93	12	14	14	99	13
20	20	22	95	15	18	20	99	18

Conditions: *c* (Cr^{3+}) — 50 mg/l, *c* (Me^{2+}) — 0.5 g/l; *c* (Na_2SO_4) — 1 g/l, pH — 7.0, J_v — 0.4 A/l, *c* (SAA) — 5 mg/l

As it is in the case of $Fe(OH)_3$, when anionic surfactant is added, it leads to chromium hydroxide surface hydrophobization and the recovery rate increased to 95–99%.

Together with Fe, Cr, Al, Zn, Ni, Cu ions, salts (such as chlorides and sulphates) are commonly present in wastewater, and their concentrations can reach 10–100 g/l. In the case of such process solutions, the extraction by electroflotation process can be less efficient [27, 28].

Brines are often the result of process solutions being dumped in wastewaters or of the ion exchange process, in which eluates form that contain ions of Fe, Cr, Al, Zn, Cu and inorganic salts.

Tables 3 and 4 below show the results of experiments that looked at the extraction by electroflotation of Fe, Cr and Al hydroxides from the following process solutions: 10 g/l NaCl and 10 g/l Na_2SO_4 (ion exchange eluates).

Analysis shows that only $Fe(OH)_3$ can be efficiently extracted without using a surfactant. The recovery rate (α) reaches 93%; 20% for $Al(OH)_3$; 14% for $Cr(OH)_3$. Introduction of 5 mg/l of anionic surfactant (NaDBS) results in a drastic rise in the recovery rate for all three metals.

Extraction by electroflotation of hydroxides from sodium sulphate solutions has a lower efficiency. Introduction of NaDDS helps intensify the electroflotation process bringing the recovery rate up to 93–97%. It should be noted that, when both $Cr(OH)_3$ and $Fe(OH)_3$ are present, the average disperse phase particle size is 45 µm, and it is 65 µm for the Cr–Fe–Al system.

Table 3. **Effect of anionic surfactant on the extraction by electroflotation of $Fe(OH)_3$, $Al(OH)_3$, $Cr(OH)_3$ hydroxides from the NaCl solution (10 g/l)**

Time τ, min	Recovery rate, α,%					
	without SAA			NaDDS		
	$Fe(OH)_3$	$Al(OH)_3$	$Cr(OH)_3$	$Fe(OH)_3$	$Al(OH)_3$	$Cr(OH)_3$
5	82	10	11	93	82	80
10	92	15	13	95	84	82
20	93	20	14	96	96	90

Conditions: *c* (∑Me^{3+}) — 300 mg/l; *c* (NaCl) — 10 g/l, pH — 7.0, J_v — 0.4 A/l, *c* (SAA) — 5 mg/l

Table 4. **Effect of anionic surfactant on the extraction by electroflotation of $Fe(OH)_3$, $Al(OH)_3$, $Cr(OH)_3$ hydroxides from the Na_2SO_4 solution (10 g/l)**

Time τ, min	Recovery rate, α, %					
	without SAA			NaDDS		
	$Fe(OH)_3$	$Al(OH)_3$	$Cr(OH)_3$	$Fe(OH)_3$	$Al(OH)_3$	$Cr(OH)_3$
5	45	11	16	90	62	44
10	60	15	18	93	76	52
20	65	18	20	97	83	60

Conditions: *c* (∑Me^{3+}) — 300 mg/l; *c* (Na_2SO_4) — 10 g/l, pH — 7.0, J_v — 0.4 A/l, *c* (SAA) — 5 mg/l (* — 10 mg/l SAA)

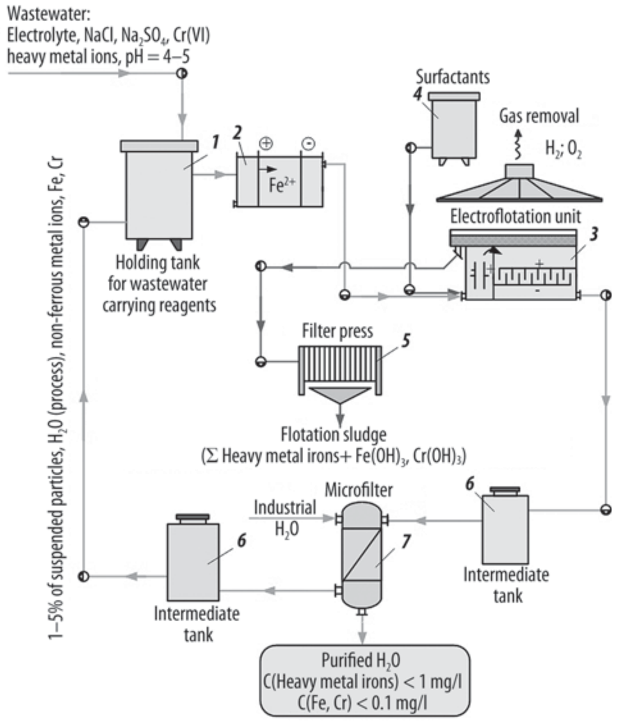

Fig. 2. Principal diagram: Treatment of wastewater carrying heavy metal ions, Cr(IV) through electrocoagulation and electroflotation:
1 — holding tank; *2* — electrocoagulator; *3* — electroflotation unit; *4* — container with anionic surfactant; *5* — filter press; *6* — intermediate tank; *7* — Microfilter

Along with laboratory studies, there is some process work that is carried out to examine the application of electroflotation and coagulation for wastewater treatment using $FeCl_3$ and $AlCl_3$, $Ti(OH)_4$, flocculants and surfactants [29–31]– primarily for poorly extractable systems, such as oil-containing wastewater generated by steel rolling mills [29].

On the basis of the conducted studies and practical experience, the authors propose certain treatment processes applicable to wastewater containing iron and chromium hydroxides, as well as organic contaminants.

Fig. 2 shows a principal diagram of the electrocoagulation and electroflotation processes aimed at removing Cr(III), Cr(VI) and Fe(III) from wastewater.

Two basic modules are used for treatment of water carrying iron (II, III) ions: an electroflotation unit and a microfilter. The electroflotation unit recovers 95–98% of the deposit in 10–15 minutes, and 1–5% of suspended particles are recovered by the filter. The main deposit is formed in the electroflotation unit. The moisture content of the deposit is 96–97%. The amount of the deposit is dictated by the initial concentration of Fe^{3+} (g/m^3) and the capacity of the unit (m^3/hour). This process works if the concentration of the NaCl and Na_2SO_4 salts is 1–50 g/l, that of the suspended particles is < 200 mg/l, and that of Fe^{3+} is < 500 mg/l.

The treatment process for wastewater carrying suspended particles and hexavalent chromium (see Fig. 2) involves the processes of electrocoagulation, electroflotation and microfiltering. What differentiates this process from the approach described above is that this process is based on the use of an electrocoagulator with a steel anode (metal, shavings) that serves to reduce Cr(VI) to Cr(III). It is possible due to Fe(II) accumulated in wastewater. Then, an electroflotation unit is used to separate a mixture of $Fe(OH)_3$ and $Cr(OH)_3$. The recovery rate is 95–97%; the concentration of NaCl and Na_2SO_4 salts is 1–50 g/l.

To realize the above described techniques, the authors propose to use electroflotation units with the capacities of 1, 5 and 10 m^3/h. More information on such units can be found in the following papers: [15, 21]. These units are characterized with a low power consumption (0.3–0.5 kW·h/m^3 treated water), a short separation (extraction) time (not exceeding 10 minutes) and a recovery rate reaching 95–98%; the throughput is 5 m^3 of water per 1 m^3 of the unit; the cost of the unit per 10 m^3/h does not exceed 1.5 million rubles.

Conclusion

The conducted study shows that it is in the presence of anionic surfactants that the process of extracting hydrophilic Fe, Al, Cr hydroxides by electroflotation reaches its highest efficiency.

Though Ca^{2+} and Mg^{2+} ions inhibit the electroflotation process, positive effects are observed when the concentration of surfactant is low: Me:SAA — 100:(5–10). This can be attributed to hydroxide surface adsorption and surface hydrophobization.

The described effects lead to a shorter electroflotation time, a higher recovery rate and lower power costs. $Fe(OH)_3$–SAA and $Al(OH)_3$–SAA used as flocculants prove to be efficient in extracting the powders of TiO_2, 'OU-B', TiC, SiC, SiO_2 by electroflotation with the recovery rate reaching 95%.

This research study was funded by D.Mendeleev University of Chemical Technology of Russia, Project no. 3-2020-004.

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