

Contents lists available at ScienceDirect

Journal of Hazardous Materials



journal homepage: www.elsevier.com/locate/jhazmat

A parametric comparative study of electrocoagulation and coagulation using ultrafine quartz suspensions

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ARTICLE INFO

Article history: Received 12 February 2009 Received in revised form 27 May 2009 Accepted 28 May 2009 Available online 6 June 2009

Keywords: Electrocoagulation Coagulation Zeta potential Quartz Turbidity

ABSTRACT

This paper attempts to compare electrocoagulation using aluminum anodes and stainless steel cathodes with conventional coagulation by aluminum sulfate dosing on aqueous suspensions of ultrafine quartz. Several key parameters affecting the efficiency of electrocoagulation and coagulation were investigated with laboratory scale experiments in search of optimal parameter values. Optimal values of the parameters were determined on the basis of the efficiency of turbidity removal from ultrafine quartz suspensions. The parameters investigated in the study were suspension pH, electrical potential, current density, electrocoagulation time, and aluminum dosage. A comparison between electrocoagulation and coagulation was made on the basis of total dissolved aluminum, revealing that electrocoagulation and coagulation were equally effective at the same aluminum dosage for the removal of quartz particles from suspensions. Coagulation, however, was more effective in a wider pH range (pH 6–9) than electrocoagulation which yielded optimum effectiveness in a relatively narrower pH range around 9, where, in both methods, these pH values corresponded to near-zero zeta potentials of quartz particles. Furthermore, experimental results confirmed that electrocoagulation could display some pH buffering capacity. The kinetics of electrocoagulation was very fast (<10 min) in approaching a residual turbidity, which could be modeled with a second-order rate equation.

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1. Introduction

Mineral treatment processes generally produce wastewaters containing suspended and colloidal particles. Most colloidal particles are stable and remain in suspension, thus cause pollution in water into which they are discharged or degrade re-circulation water in processing plants [1]. The mutual repulsion amongst the colloidal particles due to the same sign of their surface charges is the main reason for the stability of the system. It is difficult to remove colloidal particles in gravitational sedimentation ponds or devices without any size enlargement treatment. Size enlargement treatment may involve destabilization of particles or collision of particles to form aggregates. Destabilization involves either an increase in ionic strength or a neutralization of the particles' surface charge by the addition of chemicals called coagulant or flocculant. One of the commonly used coagulants in practice is hydrolyzing metal coagulants. Those coagulants are mostly based on aluminum or ferric salts such as aluminum sulfate and ferric chloride [2]. These salts promote different processes involved in the charge destabilization, as they increase ionic strength, and adsorb on the colloidal surface compensating its former electrical charge, and they can promote the formation of precipitates. An alternative to the direct use of solution containing the coagulant salts, which causes secondary pollution when added at high concentrations, is the in situ generation of coagulants by electrolytic oxidation of an appropriate anode material (e.g. iron or aluminum). This process is known as electrocoagulation and has many advantages over conventional coagulation [3].

Metallic element used at the anode is oxidized to give its ion. These metal ions hydrolyze to some extent in water. When aluminum is used, AI^{3+} ions are produced in water, which form soluble monomeric and polymeric hydroxo-metal complexes. The main reactions occurring at the electrodes during electrolysis are as follows:

At the anode, aluminum oxidation occurs,

$$Al_{(s)} \rightarrow Al^{3+}{}_{(aq)} + 3e^{-} \tag{1}$$

(Standard electrode potential of aluminum is $E^{0} = -1.662 \text{ V}$) At the cathode, water reduction occurs,

$$3H_2O + 3e^- \rightarrow 3/2H_2 + 3OH^-$$
 (2)

(Standard electrode potential of water is $E^0 = -0.828 V [4]$)

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^{0304-3894/\$ -} see front matter @ 2009 Elsevier B.V. All rights reserved. doi:10.1016/j.jhazmat.2009.05.133

At alkaline conditions,

$$Al^{3+} + 3OH^{-} \rightarrow Al(OH)_3$$
 reaction (3)

At acidic conditions,

$$Al^{3+} + 3H_2O \rightarrow Al(OH)_3 + 3H^+ reaction$$
 (4)

occur.

Electrocoagulation can be used for many purposes such as treatment of clay suspensions [4], removal of boron from boroncontaining solutions [5,6], treatment of textile wastewater [7], removal of phosphate from aqueous solutions [8], separation of pollutants from restaurant wastewater [9], removal of arsenic from water [10], urban wastewater treatment [11], removal of chromium (Cr(VI)) [12,13] and removal of heavy metals [14].

In order to investigate the characteristics of electrocoagulation and coagulation of polluted water, a known pollutant material that causes pollution in water was selected. For this purpose, quartz was chosen. It is one of the common components present in soils and clays which may cause pollution in water. Quartz particles in water have negative surface charge in a wide pH range [15]. As an electrode material aluminum was chosen since it is cheap, readily available, and proven effective. Also, it is found that the effluent with aluminum electrodes is very clear and stable [9].

In this study, electrocoagulation and coagulation processes were investigated under different experimental conditions obtained by changing several key parameters including pH, electrical potential, current density, electrocoagulation time, and aluminum dosing. Optimal values of these parameters were determined on the basis of the efficiency of turbidity removal from ultrafine quartz suspensions.

2. Material and methods

Electrocoagulation and coagulation tests were conducted with suspensions of ultrafine quartz particles. The quartz was obtained from Eczacıbaşı Esan, Turkey (density of quartz is 2.65 g/cm^3). The median and 90%-finer sizes of the quartz samples, as determined by Mastersizer 2000, were 11.61 and 5.13 µm, respectively, with the most probable particle size corresponding to 7 µm. Mineral composition of the quartz sample was determined using X-ray diffraction analysis. XRD patterns were taken by using Rigaku Miniflex Diffractometer with Cu K α (30 kV, 10 mA, λ (1.54050 Å)) radiation, which revealed that the quartz sample was pure. Chemical composition of the quartz was determined with the X-ray florescence spectrometry using Benchtop X-ray Florescence Spectrometer. The results showed that quartz is composed of 99.5% SiO₂ and 0.0038% Al₂O₃.

Quartz suspensions were prepared by adding appropriate amounts of quartz powder into 250 mL distilled water to obtain a solid concentration of 0.32 g/L in suspension. The natural pH value of the prepared suspension was about 4. The suspension pH was then adjusted to the desired value for each experiment by using 0.1 M H₂SO₄ and 0.1 M NaOH solutions. The turbidity of suspensions before and after electrocoagulation and coagulation was measured in nephelometric turbidity units (NTU) with Lamotte Model 2008 Turbidimeter. The zeta potential of quartz particles was measured with Zetasizer Nano-Z meter in aqueous suspensions having pH values from 2 to 10. The values were varied in the range of -15 to -30 mV.

The batch electrocoagulation cell used in the experimental study was constructed of plexiglas with the dimensions of $65 \text{ mm} \times 65 \text{ mm} \times 110 \text{ mm}$ (Fig. 1). Two stainless steel cathodes were interspersed with two aluminum anodes, with brass rods used to connect the parallel plate electrodes. Aluminum anode and stainless steel cathode were made from plates with dimensions of $55 \text{ mm} \times 45 \text{ mm} \times 3 \text{ mm}$ and the spacing between electrodes



Fig. 1. Schematic diagram of electrocoagulation experimental setup. (1) Power supply; (2) magnetic stirrer; (3) conductivity meter; (4) voltmeter; (5) ampermeter; (6) pH meter; (7) turbidimeter; (8) electrocoagulation cell.

was 21 mm. The effective electrode area of each electrode was 23 cm². The electrodes were connected to a DC power supply. Suspensions were stirred using a magnetic stirrer (Velp Scientifica) adjusted to an optimal rate (300 rpm) so as to obtain the highest efficiency of turbidity removal. Electrical conductivity and temperature of suspensions were measured using a conductivity meter (Lovibond SensoDirect Con2000) and a digital thermometer (Hanna Checktemp 1), respectively. A water jacket was used to keep the suspension at room temperature (nominally 21 °C). The current that flows through the cell and the voltage across the electrodes were measured by using an ampermeter and a voltmeter. Before each electrocoagulation run, electrodes were washed with acetone to remove surface grease, and the impurities on the aluminum and stainless steel electrode surfaces were removed by dipping the electrodes for 1 min in a solution prepared by mixing 50 cm³ HCl solution (35%) and 100 cm³ of hexamethylenetetramine aqueous solution (2.80%) as described by Kobya et al. [16].

The amount of aluminum dissolved in an electrocoagulation experiment was theoretically calculated using Faraday's Law that expresses the relationship between the amount of metal dissolved or deposited and the current density:

$$w = \frac{ttM}{nF}$$
(5)

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where, w = aluminum dissolved (g Al/cm²); *i* = current density (A/cm²); *t* = time (s); *M* = molecular weight of Al (*M*=27 g/mol); n = number of electrons involved in the oxidation reaction (*n*=3); *F* = Faraday's constant, 96,500 C/mol.

Vik et al. [17] confirmed that there was a good correlation between the theoretically calculated and the experimentally determined amount of aluminum that went into solution in electrocoagulation.

Conventional coagulation tests were conducted using standard jar testing technique (Fig. 2) with the addition of aluminum sulfate solutions $(10^{-2}-10^{-6} \text{ mol/L})$ at room temperature (nominally 21 °C) and a constant stirring speed of 300 rpm. Stirring time was kept constant at its optimum value of 10 min as determined from a number of preliminary tests conducted over a wide range of stirring times (1–60 min). Experimental variables of electrocoagulation and conventional coagulation studies are presented in Table 1.

After each electrocoagulation or conventional coagulation run, the coagulated suspension was poured into a 300-mL beaker and a 20-mL sample was taken from a fixed depth at a predetermined settling time to measure the turbidity. Settling times of 0, 5, 10, 20, and 30 min were separately studied by running replicate electrocoagulation and coagulation tests. Ten minutes of settling time was found to be the most appropriate one in most of the experiments. The results reported in this paper refer to 10 min of settling time unless stated otherwise. The efficiency of turbidity removal, R (%),

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Fig. 2. Coagulation unit. (1) Magnetic stirrer; (2) pH meter; (3) digital thermometer; (4) turbidimeter.

was calculated from the formula given below;

$$R \ (\%) = \frac{T_{\rm o} - T_{\rm t}}{T_{\rm o}} \times 100 \tag{6}$$

where T_0 is the initial turbidity of the suspension just before electrocoagulation or coagulation, and T_t is the turbidity at the end of a predetermined settling time (*t*) after the electrocoagulation or coagulation run.

3. Results and discussions

3.1. Effect of initial pH on electrocoagulation and coagulation

pH is an important factor influencing the performance of both electrocoagulation and coagulation processes. To examine its effect in a comparative manner, electrocoagulation and coagulation experiments with different initial pH values were conducted in the pH range of 2–11. Figs. 3 and 4 display turbidity removal efficiencies and the zeta potential values of quartz particles for electrocoagulation and coagulation cases, respectively, where the zeta potentials refer to those determined after electrocoagulation or coagulation tests.

In the case of electrocoagulation (Fig. 3) turbidity removal efficiencies are much higher in the alkaline region than those in acidic or near-neutral pH values. The highest turbidity removal efficiency (about 90%) is near pH 9 where the zeta potential curve approaches the isoelectric point of the quartz particles having adsorbed aluminum species on their surfaces. The zeta potential curve indicates the highest positive value (53 mV) at about pH 5, and then decreases with increasing pH, leading to a sign reversal at pH 9, which are all closely related with the adsorption of hydrolyzed aluminum species in solution. In the case of coagulation (Fig. 4), turbidity removal efficiencies remain at their highest values in a wider pH range of 6–9 where the zeta potentials values are again close to zero. The zeta

Table 1

Experimental variables for electrocoagulation and coagulation experiments.

Electrocoagulation	
pH	2-11
Electrical potential	0-60 V
Current density	10-200 A/m ²
Electrocoagulation time	1-60 min
Coagulation	
pH	2-11
Aluminum dosage	0-100 mg Al/L



Fig. 3. Turbidity removal efficiency and zeta potential after electrocoagulation experiments at different initial pHs (40 V; electrocoagulation time: 10 min).

potential curve goes through two isoelectric points at around pH values of 3.2 and 6.8. The isoelectric point at pH 3.2, however, does not lead to good turbidity removals. Furthermore, the zeta potentials measured in the acidic pH values after coagulation are much lower than those measured in the electrocoagulation case for the same pH values.

The hydrolysis of aluminum either released from the electrodes in electrocoagulation or added into solution as aluminum sulfate in conventional coagulation, and the adsorption of the hydrolysis products on the particles are greatly affected by the varying pH of the suspension. Fig. 5 shows the solubility diagram for aluminum hydroxide Al(OH)_{3(s)}, assuming only monomeric species. Nonhydrolyzed Al³⁺ and cationic hydrolysis products, Al(OH)²⁺ and Al(OH)₂⁺ are the dominant species in solution at pH values less than 5. The cationic hydrolysis products, rather than the nonhydrolyzed Al³⁺ [18], are known to adsorb strongly on negatively charged particles leading to charge neutralization, and also to charge reversal with excess amount of aluminum ions [19]. This explains the high positive zeta potentials and low turbidity removal efficiencies due to electrical repulsion between the particles at pH values less than 5 in the case of electrocoagulation (Fig. 3). At pH values 5-8.5, especially with excessive aluminum concentration, amorphous aluminum hydroxide precipitation becomes important. This precipitation may occur either on the surface of guartz particles or the hydroxide precipitates formed in the bulk may attach to the particles. The aluminum hydroxide precipitate is known to have an isoelectric point in the region of pH 8-9 [20]; and, hence, it



Fig. 4. Turbidity removal efficiency and zeta potential after coagulation experiments at different initial pHs (15 mg Al/L; coagulation time: 10 min).



Fig. 5. Solubility diagram of aluminum hydroxide $Al(OH)_{3(s)}$ considering only monomeric aluminum species.

is positively charged at lower pH values. The adsorption of the positively charged precipitate on the negatively charged quartz particles leads to charge neutralization and lower zeta potential values, which results in better coagulation as depicted by increasing turbidity removal efficiencies in Fig. 3. At pH values higher than 8.5, the aluminate ion, $Al(OH)_4^-$, becomes the predominant species which cannot be adsorbed on the negatively charged quartz particles. Hence, the turbidity removal efficiencies deteriorate. In the case of coagulation, however, sulfate anion is introduced into solution by the added coagulant, aluminum sulfate. Sulfate can be adsorbed on positively charged aluminum hydroxide precipitate and reduces its positive charge at pHs below about 8 or 9 [2]. This explains the reason for the lower zeta potential values than those measured in electrocoagulation and a sign reversal in the acid region for the conventional coagulation as shown in Fig. 4.

Fig. 6 shows the pH change of the quartz suspensions after electrocoagulation. The suspension pH increases during electrocoagulation when the initially adjusted pH is less than 8, and a decrease occurs if the initial pH is above 8. As reported by previous studies [9,16], electrocoagulation exhibits some pH buffering capacity, especially in an alkaline medium. The increase in pH is attributed to formation of OH⁻ at the cathode. Dissolution of the amphoteric metal hydroxide Al(OH)₃ decreases the pH at high ini-



Fig. 6. pH change after electrocoagulation experiments (40 V; electrocoagulation time: 10 min).



Fig. 7. Effect of applied voltage on current density and turbidity removal efficiency (pH 9; electrocoagulation time: 10 min).

tial pHs according to the following reaction [9,16,17,21]:

$$Al(OH)_3 + OH^- \rightarrow Al(OH)_4^-$$
(7)

In addition, there is also oxygen evolution reaction that leads to decrease in pH. When the anode potential is sufficiently high, a secondary reaction, especially oxygen evolution, may occur [21–23]:

$$2H_2O \rightarrow O_2 + 4H + + 4e^-$$
 (8)

3.2. Effect of voltage on electrocoagulation

In order to examine the effect of applied electrolysis voltage on turbidity removal efficiency and current density in the electrocoagulation process, experiments were carried out in a wide range of applied voltage from 0 to 60 V at fixed values of pH, suspension concentration, and electrocoagulation time (Fig. 7). The turbidity removal efficiency reaches an optimum value at about 40 V, while the current density expectedly increases with increasing voltage. The current density at the optimum turbidity removal efficiency is 23.9 A/m² corresponding to a power consumption of 1.87 kWh/m³ of suspension after 10 min of electrocoagulation conducted with suspensions having about 30 µS/cm conductivity. The conductivity had no significant change during each experimental run for the range of voltages tested. It was also observed during the experiments that, at long electrocoagulation times (>30 min) and high voltages, flotation of particles attached to hydrogen gas bubbles released from the cathode became an important turbidity removal mechanism, as also previously reported by Holt et al. [24].

3.3. Effect of current density and time on electrocoagulation

Waste mineral suspensions may have a range of dissolved ionic species changing the ionic strength and hence the conductivity of the suspensions, which, in turn, change the current density at constant applied voltage. Therefore, the effect of increasing current density on turbidity removal efficiency was studied at constant voltage by adding appropriate amounts of NaCl to quartz suspensions. Table 2 shows the amounts of NaCl added and the resulting current densities and conductivities at 40 V electrocoagulation experiments.

The effects of current density on the turbidity removal efficiency, conductivity and the theoretically calculated amount of aluminum released from the electrodes are shown graphically in Fig. 8. It appears that the turbidity removal efficiency increases up to 92% at 87 A/m² current density and decreases at higher current densities. The increase in the turbidity removal efficiency can be explained by higher degree of destabilization of particles due to charge neu-



Fig. 8. The effect of current density on the turbidity removal efficiency, total dissolved aluminum and conductivity (pH 9; 40V; electrocoagulation time: 10 min).

tralization as more aluminum is released from the electrodes with increasing current density. However, with excessive amount of aluminum released from the electrodes at higher current densities, the most likely explanation for the decrease in turbidity removal is the formation of slow-settling, low-density flocs in the extensive presence of aluminum hydroxide precipitates [4]. Adding NaCl to increase suspension conductivity in constant voltage electro-coagulation experiments increased the power consumption. The optimum turbidity removal (92%) in Fig. 8 corresponds to a power consumption of 2.67 kWh/m³ which is considerably higher than the power consumption (1.87 kWh/m³) required for obtaining the same degree of turbidity removal in the absence of NaCl at the same voltage (Fig. 7).

Fig. 9 shows the removal efficiencies and quartz concentrations as a function of electrocoagulation time at constant electrolysis voltage. In this case, turbidity removal efficiencies were measured after 5 min of settling time following the electrocoagulation experiment. The turbidity removal efficiency increased sharply within 10 min of electrocoagulation time, beyond which no significant improvement was observed. As a result, quartz concentration in suspension decreased rather sharply within first 10 min of electrocoagulation, approaching a residual level of concentration which did not change appreciably at prolonged electrocoagulation. The form of the concentration vs. time profile shown in Fig. 9 suggested fitting a second-order rate equation for the reduction of quartz concentration in suspension in the following form:

$$\frac{\mathrm{d}C}{\mathrm{d}t} = -k(C - C_{\mathrm{r}})^2 \tag{9}$$

where *C* is the concentration of quartz particles in suspension at any time *t*, C_r is the residual value of the quartz concentration at prolonged times, and *k* is the second-order rate constant of electro-

Concentration of NaCl added (g/L) and the resultant changes in current density and conductivity.

Table 2

NaCl addition (g/L)	Current density (A/m ²)	Conductivity (µS/cm)
0.005	17.39	24
0.009	21.74	32
0.014	30.44	41
0.021	43.48	57
0.056	86.96	125
0.093	130.44	200
0.130	173.91	274

coagulation. The integrated rate equation takes the form

$$C = \frac{C_0 + C_r(C_0 - C_r)kt}{1 + (C_0 - C_r)kt}$$
(10)

The experimental data of Fig. 9 showed very good fit to the above kinetic model. The predicted values of the model parameters C_r and k from a nonlinear regression analysis were found to be 0.034 g/L and 5.48 min⁻¹, respectively, with a corrected sum of squares value of 0.99 in regression.

3.4. Effect of aluminum dosage on electrocoagulation and coagulation

In order to compare electrocoagulation and coagulation processes on the basis of the amount of aluminum either released from the electrodes or added as aluminum sulfate into the suspension, tests were conducted under the optimum conditions of each process. Fig. 10 presents the experimental conditions and the effect of aluminum dosage on the turbidity removal efficiencies obtained with electrocoagulation and coagulation. It appears from the figure that, as long as the operating conditions are optimized for each process, the source of aluminum does not matter from the turbidity removal point of view. Equal dosages of aluminum provided into suspensions lead to quite similar effectiveness in removing the suspension turbidity. A detailed comparison of the two processes,



Fig. 9. Effect of electrocoagulation time on the turbidity removal efficiency of quartz from suspension and quartz concentration (g/L) in the suspension (pH 9; 40 V; 22 A/m^2).



Fig. 10. Effect of aluminum dosage (mg/L) on quartz removal by electrocoagulation (pH 9; 40 V; 10 min) and coagulation (pH 6; 10 min).

however, requires the consideration of many other operational and economic factors, which is beyond the scope of this study.

4. Conclusions

- The initial pH of the suspension and the current density were two major parameters that affected the removal of quartz from suspensions with electrocoagulation process. The optimum pH for removal of quartz from suspension was found to be 9. The turbidity removal efficiency increases up to 92% at a current density of 87 A/m². In a particular experiment, when aluminum was used as a sacrificial anode and the initial concentration of quartz was about 0.32 g/L, the optimum electrical potential between the electroces and electrocoagulation time were found to be 40 V and 10 min, respectively.
- The coagulant used in the coagulation process was aluminum sulfate whose amount in coagulation process was studied together with the pH effect. The value of the optimum coagulant dosage was 15 mg Al/L with a turbidity removal efficiency of 93% at an initial concentration of 0.32 g/L quartz. The optimum value for pH in coagulation was found to be in a wider pH range of 6–9.
- In both electrocoagulation and coagulation, suspension pHs for optimum turbidity removal were around the isoelectric point of quartz particles having adsorbed species, suggesting similar mechanisms of particle aggregation through charge neutralization and/or enmeshment in aluminum hydroxide precipitates.
- Electrocoagulation shifted the initial suspension pHs toward neutral pH values during the process whereas aluminum sulfate addition acidifies the water in coagulation.
- Electrocoagulation and coagulation removed turbidity equally well if the same amount of aluminum was introduced into the suspension provided that all other operating parameters were optimized specifically for each process.
- The kinetics of electrocoagulation was very fast, leading to an optimum removal of turbidity within 10 min. The kinetics of electrocoagulation could be modeled by a second-order rate equation.

Acknowledgements

The authors gratefully acknowledge Dr. Ali İhsan Arol for his helpful discussions. The financial support provided by the Research Fund of the Middle East Technical University in the scope of BAP-2006-07-02-00-01 project is greatly appreciated. The authors are also thankful to the METU-Central Laboratory for the measurement of particle size distributions.

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