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An investigation of the effect of NaCl concentration on the electrocoagulation of coal preparation plant tailings

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Abstract: Coal preparation is the process of separating the coal from mineral impurities to produce high-grade coal, and the wastewater of the coal separation process is mainly consisted of fine coal and clay particles. Electrocoagulation (EC) is one of the effective methods for wastewater treatment. In this context, this study was aimed to investigate the evaluation of EC process for coal preparation plant wastewaters. Several key parameters affecting the efficiency of EC were investigated with laboratory scale experiments in search of optimal parameter values. Most importantly, the effect of NaCl concentration on the precipitation of coal preparation plant tailings by EC was investigated. Current density, electrolysis time, mixing speed, mixing time, pH, and salt concentration were studied using an aluminum electrode. Based on the results obtained from this study, the optimum conditions were found to be as pH 7.88 (natural pH), current density 40 (A/m²), mixing speed 360 rpm, premixing time 120 sec, and electrolysis time 300 sec. Under the optimum operating conditions, the results indicated that EC can be successfully applied for the coal preparation wastewaters, and the turbidity was reduced from 1260 NTU to 63 NTU (95% efficiency) with an operating cost of \$5.67/Mg of tailings. By the increasing the salt content, a small increase in the turbidity was observed with a decrease in the voltage. With only a 1% decrease in the removal efficiency, the cost was reduced to \$3.19/Mg with a 44% operating cost reduction.

Keywords: coal, tailing, coagulation, electrocoagulation, electrode

1. Introduction

In coal washing plants, waste water containing very finely sized solids (coal dust and clay minerals) is produced. In order to prevent waste water from being hazardous to the environment, it is necessary to settle the solids. This process is usually performed in large tanks called thickeners that make use of conventional methods such as flocculation or coagulation. In both methods, the use of additional chemicals for the settling of the solids is necessary. Solids are filtered, and water is recycled to be used in the plant (Sabah et al., 2004; Sabah and Erkan 2006; Alam et al., 2011; Zhang and Wang 2012; Singh et al., 2012).

Electrochemical methods such as electro-oxidation and electrocoagulation (EC) have been developed due to some negative aspects encountered when classical settling methods are used. These aspects may be the formation of excessive amounts of slurry, excessive consumption of chemicals, and an increase in the conductivity of water. In addition, there is an increasing environmental sensitivity in today's world, and these electrochemical methods may offer an economical advantage (Mollah et al., 2004). Such methods can be used in many fields with the purpose of purification of clay suspensions, purification of textile waste waters, removal of phosphate from aqueous solutions, removal of pollutants from waste waters of restaurants, and removal of chrome and heavy metals from urban waste water (Mollah et al., 2004; Kilic et al., 2009).

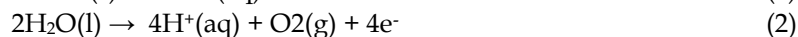
An EC system consists of an electrolytic cell, which has an anode and a cathode. The principle of the EC process is based on the dissolution of metal anodes, such as aluminum and iron, and the formation

of metal hydroxides such as $\text{Al}(\text{OH})_3$, $\text{Fe}(\text{OH})_2$, and $\text{Fe}(\text{OH})_3$ through hydrolysis in the environment (Mollah et al., 2001; Canizares et al., 2005a; Canizares et al., 2005b).

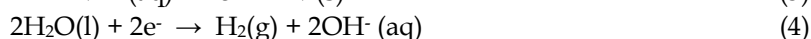
Metal ions released from these electrodes form metal complexes depending on ambient conditions and coagulate by absorbing the pollutants. In the EC process, it is generally accepted that there are three consecutive stages: 1. The formation of coagulant species by means of an electrode dissolved during electrolytic oxidation. 2. The breaking of particulate suspensions and emulsions to make the phases unstable. 3. Formation of flocks in the unstable phases (Holt et al., 2002; Canizares et al., 2005).

Metal electrodes used in the EC process must have a large surface area in order for them to be dissolved in sufficient quantities. Electrochemical reactions between "M" metal and the anode and cathode are as follows (Mollah et al., 2004):

Anode:



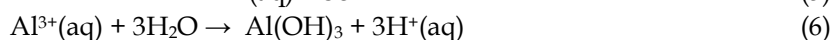
Cathode:



When an aluminum electrode is used, as a result of hydrolysis, Al^{3+} ions form $\text{Al}(\text{H}_2\text{O})_6^{3+}$, $\text{Al}(\text{H}_2\text{O})_5\text{OH}^{2+}$, and $\text{Al}(\text{H}_2\text{O})_4(\text{OH})_2^{2+}$. Within a wide pH range, hydrolysis products form several monomeric and polymeric types of hydroxide complexes such as $\text{Al}(\text{OH})^{2+}$, $\text{Al}(\text{OH})_2^{+}$, $\text{Al}_2(\text{OH})_2^{4+}$, $\text{Al}(\text{OH})_4^+$, $\text{Al}_6(\text{OH})_{15}^{3+}$, $\text{Al}_7(\text{OH})_{17}^{4+}$, $\text{Al}_8(\text{OH})_{20}^{4+}$, $\text{Al}_{13}\text{O}_4(\text{OH})_{24}^{7+}$, and $\text{Al}_{13}(\text{OH})_{34}^{5+}$ (Mollah et al., 2004).

The products of the aluminum anode in electrolyte solutions at low pH levels are, for instance, Al^{3+} and $\text{Al}(\text{OH})^{2+}$. At appropriate pH levels, these are polymerized first into $\text{Al}(\text{OH})_3$, and then finally into $\text{Al}_n(\text{OH})_{3n}$ (Vardar 2006). When an aluminum electrode is used, the reactions observed in the anode and cathode are the following (Mollah et al., 2004):

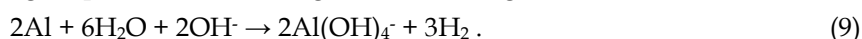
Anode:



Cathode:



In addition, depending on the pH value, other ionic types such as $\text{Al}(\text{OH})^{2+}$, $\text{Al}_2(\text{OH})_2^{4+}$, and $\text{Al}(\text{OH})_4^+$ could exist in the system. $\text{Al}(\text{OH})_4^-$ is formed as a result of the encircling of the cathode by OH^- ions formed chemically with H_2 at higher pH values according to the following reaction (Mollah et al., 2004):



Compared to conventional coagulation and flocculation practices, the EC has an advantage of removing colloid particles due to the presence of an electrical field (Pouet and Gramick 1995). Moreover, the formed slurry can be easily precipitated and dewatered since it essentially contains metal oxides or hydroxides (Vardar 2006).

Electrolytes such as NaCl , Na_2SO_4 , and Na_2CO_3 are widely used in the EC process to increase the conductivity of the solution. NaCl is the most preferred due to its low price and easy production (Chen 2004). In a study where the effect of salt used in the EC process for the treatment of paper mill waste was investigated, it was reported that chemical oxygen demand (COD) yield increased from 50% to 90% when the salt solution was added in a bipolar-connected reactor (Karabacakoglu and Tezakil, 2012).

In this study, the precipitation of coal preparation plant tailings using the EC method was investigated. In this purpose, the effects of pH, current density, electrolysis time, mixing speed, mixing time, and salt content were investigated in the EC experiments using an aluminum electrode.

2. Materials and methods

2.1 Materials

The coal waste water sample used in this study was obtained from a privately-owned mining company operating in Tekirdag in the Marmara Region of Turkey. The sample was taken from the input to the

thickener where the coal preparation plant tailings were settled. This pulp was transferred to the laboratory in plastic jerry cans.

In the EC experiments, six aluminum electrodes with a size of 50×1×60 mm each one and an active surface area of 373 cm² in total were used. For all experiments, the electrodes were placed into the test cell in monopolar-parallel connection. A 1 mol/dm³ 500 cm³ NaCl solution was prepared to adjust the salt ion concentration, and different amounts of this solution were used in the experiments.

Cation and anion values were determined by performing a chemical analysis of the plant process water, and the typical tap water used in the laboratory.

The particle size distribution analysis of the solids in the waste samples was measured with a Malvern Mastersize 2000, and the average particle size (d_{50}) was determined. The chemical analysis of the waste sample was conducted using an Axios SST-Max XRF analyzer.

A mineralogical analysis of the waste samples was performed with a Rigaku, D/Max-2200/PC XRD analyzer.

Zeta potential measurements of suspensions prepared with deionized water (DI, Milli Q, Millipore Corp., 18 MΩ × cm), process water, and tap water were made using a Brookhaven Zetaplus, USA branded device. This analysis investigated the effect of different ion concentrations on surface charge. The samples contained 1% solids for the measurements.

First, about 5 g of the sample was dry ground for 15 min using a mortar and pestle. Then, the ground sample was screened through a 38 μm sieve, and the undersize was conditioned in the water samples (solid content of 1%) using a magnetic stirrer at 500 rpm for 15 min. Before each experiment, the suspensions were settled for 5 min to allow the coarse particles to settle. A small amount of suspension was taken from the top of the suspension and transferred to the measurement cell. Finally, ten measurements were performed, and the average value of the measurements was obtained. An average error of these measurements was about 3%. The experiments were carried out at room temperature (23 °C).

2.2 Methods

The EC experiments were conducted in a reactor made of plexi-glass having dimensions and volume of 150×90×85 mm and 1150 cm³, respectively. A DC, Echinni-C laboratory power supply was used as the power source. The experimental setup of EC is shown in Fig. 1.

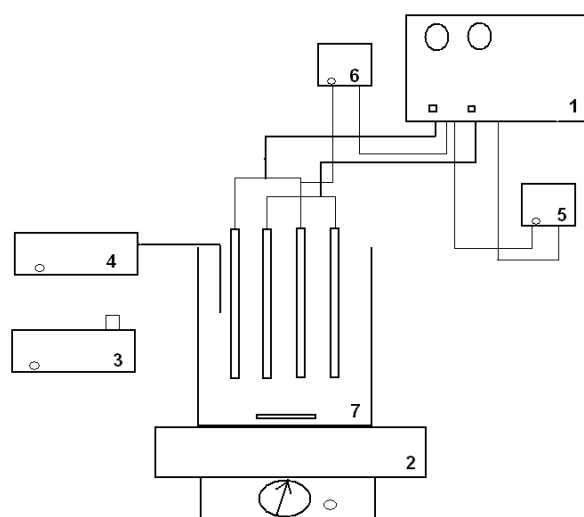


Fig. 1. EC experimental setup: (1) DC power supply (2) magnetic stirrer (3) turbidimeter (4) pH meter (5) voltmeter (6) ampere meter, and (7) electrocoagulation cell

The solid ratio of the slurry was determined as 3% (w/v), and the representative suspensions were prepared at 3% for the EC experiments. The solid ratio was kept constant in all experiments. Before each experiment, the suspension in the cell was homogenized using a magnetic stirrer. Electrical measurements in the cell were performed using an ampere meter and voltmeter.

Operating parameters such as pH, current density, electrolysis time, mixing speed, mixing time, and the salt content were investigated. In all experiments, the connection type of the electrodes (monopolar electrodes connected in parallel), the distance between the electrodes (2 cm), and temperature (room temperature) were kept constant.

Changes in pH and temperature were also monitored during the experiments. Electrodes were immersed in a 30% formic acid solution for 45 min prior to each experiment, and they were reused after being washed with water after cleaning in order to avoid corrosion that might occur on the surface of electrodes. The electrodes were weighed with precision scales before and after each experiment, and the differences were recorded and used in the cost analysis. The test results were evaluated by turbidity measurements. Turbidity removal efficiency (R) was calculated by Eq. 10:

$$R(\%) = \frac{T_0 - T_t}{T_0} \times 100 \quad (10)$$

where T_0 is the initial turbidity (NTU) of the suspension before exposure to EC and coagulation processes, and T_t is the turbidity (NTU) of the suspension after a specific precipitation time.

The amount of dissolved aluminum in the EC experiments were calculated based on Faraday's law. Faraday's law describes the relationship between current density and the quantity of dissolved metal. According to this law, the quantity (in grams) of dissolved material was calculated with Eq. 11:

$$w = \frac{itM}{nF} \quad (11)$$

where w is the aluminum dissolved (g/cm^2), i is the current density (A/cm^2), t is time (s), M is the molecular weight of Al (27 g/mol), n is the number of electrons involved in the oxidation reaction ($n=3$), and F is Faraday's constant (96500 C/mol).

In the EC experiment (presence of NaCl), the optimum experimental conditions obtained in the previous experiments (absence of NaCl) were applied. For these experiments, 500 cm^3 of a 1 mol/ dm^3 NaCl solution was prepared. The effect on EC was investigated by adding 10, 20, 30, and 40 cm^3 (0.58, 1.17, 1.76, and 2.34 g/ dm^3) of prepared NaCl solution into the suspensions.

3. Results and discussion

3.1 Characterization of sample

Cation and anion values were determined by performing a chemical analysis of the plant process water, and the typical tap water used in the laboratory. The results of the analysis are presented in Table 1. Based on the test results, it was observed that cations in the process water were Na, Mg, and Ca, and the anions were SO_4 and Cl.

Table 1. Chemical analysis results of the process water and tap water

Ion type		Process water (mg/dm^3)	Tap water (mg/dm^3)
Anions	F	0.1	0.1
	Cl	93.5	29.1
	Br	0.1	0.1
	N	0.1	1.8
	SO_4	1537.8	41.7
Cations	Na	291.0	19.8
	K	17.6	2.5
	Mg	209.4	6.4
	Ca	111.9	45.2

The particle size distribution analysis of the waste samples was measured with a Malvern Mastersize 2000, and the average particle size (d_{50}) were determined to be 45 μm . The chemical analysis of the waste sample was conducted using an Axios SST-Max XRF analyzer, and the results of the analysis are

presented in Table 2. As a result of the analysis, the loss on ignition for the sample was found to be 30% which indicated that there was a significant amount of carbon, namely coal, in the sample.

A mineralogical analysis of the waste samples was performed with a Rigaku, D/Max-2200/PC XRD analyzer, and the presence of minerals such as quartz, muscovite, montmorillonite, chlorite, serpentine, calcite, and dolomite was identified. Lignite was also present.

The results for the zeta potential experiments are presented in Table 3. As shown in Table 3, the surface charges of the particles prepared with different types of water were negative and very similar. Therefore, it was concluded that water type would not have any effect on the EC. The conductivity of process water was found to be greater than the others, indicating that process water was saltier. As presented in Table 1, the SO_4 ion concentration of the process water was much higher than that of the tap water. It is known from literature that some ions such as SO_4 can considerably change the surface potential of the particles. Therefore, this can affect the flocculation behaviour of the particles. In order to eliminate this effect during the experiments, the tap water was used because it contained less sulphate ions.

Table 2. Chemical analysis of the sample

Analysis	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	K_2O	TiO_2	Na_2O	LOI
%	38.07	13.23	7.63	3.02	2.85	2.11	0.53	0.20	30

Table 3. Zeta potential of the sample in the deionized (DI), tap, and process water

Water type	pH (natural)	Zeta Potential (mV)	Conductivity (mS/cm)
DI	6.09	-17.53	0.5
Tap water	7.8	-22.12	1.3
Process water	7.66	-24.17	6.4

3.2 Electrocoagulation (EC) experiments in the absence of NaCl

3.2.1 Effect of current density on EC experiments

In order to determine the optimum current density (13.4, 26.8, 40, 53.6, 80.4, and 107.2 A/m^2) for the EC experiments in the absence of NaCl, the other parameters namely stirrer speed, premixing time, total EC time, and pH were kept constant as 360 rpm, 120 sec, 300 sec, 7.8, respectively. The effect of current density on EC (absence NaCl) is shown in Fig. 2.

A significant change was observed as a 1.5 A current intensity corresponding to 40 (A/m^2) current density. The initial turbidity of the suspension at a current density of 40 (A/m^2) was reduced from 1260 NTU to 104 NTU level with a 92% coagulation efficiency. The 40 (A/m^2) current density was determined to be the optimum value since an increase in the current increased electricity consumption as well as the costs.

3.2.2 Effect of electrolysis time on EC experiments

Following the determination of current density, the effect of electrolysis time on the EC was investigated. As the duration of the electrolysis was increased, the amount of metal ions transferred from the anode into the system also increased. This increased the efficiency of coagulation because the ions formed more hydroxide molecules. In order to determine the electrolysis duration (60, 180, 300, 420, 540, and 660 sec) for the EC experiments in the absence of NaCl, the other parameters namely current density, stirrer speed, premixing time, and pH were kept constant as 40 (A/m^2), 360 rpm, 120 sec, and 7.8, respectively. The effect of electrolysis duration on the EC is shown in Fig. 3.

As seen from Fig. 3 that there was a decrease in the turbidity values as the electrolysis duration increased. However, the decrease was almost stopped after approximately 300 sec of electrolysis, the turbidity and efficiency curves did not change further. Therefore, the optimum electrolysis duration was determined to be 300 sec. Therefore, the total duration of the experiment was 420 sec, including a

preliminary conditioning time of 120 sec. During the electrolysis for 300 sec, the initial turbidity of the suspension reduced from 1260 NTU to 63 NTU level with a 95% coagulation efficiency.

3.2.3 Effect of pH on EC experiments

pH is important in EC as well as in coagulation. Hydroxide and hydrogen ions released into the environment during EC changes the pH of the environment continuously. While hydroxide ions increase the pH level, metal ions form hydroxide compounds reacting with water molecules. In order to determine the optimum pH (6.02, 8.07 (natural), 9.0, and 9.71) for the EC experiments in the absence of NaCl, the other parameters namely current density, stirrer speed, premixing time, electrolysis time and total EC time were kept constant as 40 (A/m²), 360 rpm, 120 sec, 300 sec, 420 sec respectively. The effect of pH on the EC is shown in Fig. 4. Based on this, the optimum results were obtained at pH 7.8 (natural pH).

3.2.4 Effect of mixing speed on EC experiments

The mixing speed and duration were investigated to determine their effects on the interaction of ions and molecules in suspension with particles. The most efficient interaction rate of ions and molecules with solid particles formed a suspension, which also increased coagulation efficiency significantly. When the mixing speed and duration exceeded certain limits, flocs formed in the suspension might be disintegrated. This also reduced the efficiency of coagulation. In order to determine the optimum stirrer speed (120, 180, 360, and 540 rpm) for the EC experiments in the absence of NaCl, the other parameters namely current density, premixing time, electrolysis time, total EC time, and pH were kept constant as 40 (A/m²), 120 sec, 300 sec, 420 sec, and 7.8 (natural) respectively. The effect of mixing speed on the EC is shown in Fig. 5.

The initial turbidity of the suspension at a stirrer speed of 360 rpm reduced from 1260 NTU to 63 NTU level with a 95% coagulation efficiency. With speeds over 360 rpm, it was determined that turbidity values started to increase again in parallel with the disintegration of formed flocs. Therefore, 360 rpm was found to be the optimum mixing speed.

3.2.5 Effect of mixing time on EC experiments

The effect of mixing time was investigated in the EC experiments. In this context, the optimum parameters determined in the previous experiments were not changed, and different premixing times were tested. In order to determine the optimum premixing time (60, 120, 180, and 240 sec) for the EC experiments in the absence of NaCl, the other parameters namely current density, total EC time, stirrer speed, and pH were kept constant as 40 (A/m²), 420 sec, 360 rpm, and 7.8 (natural), respectively. The test results are given in Fig. 6. According to Fig. 6, the optimum premixing time was determined to be 120 sec. No significant change in the values was observed for the coagulation efficiency and turbidity values after 120 sec.

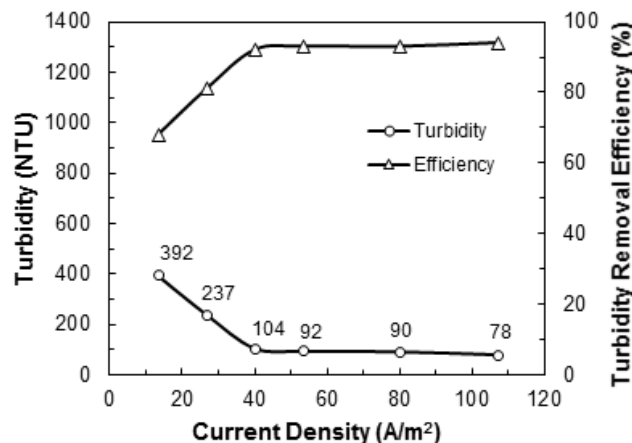


Fig. 2. Effect of current density on electrocoagulation

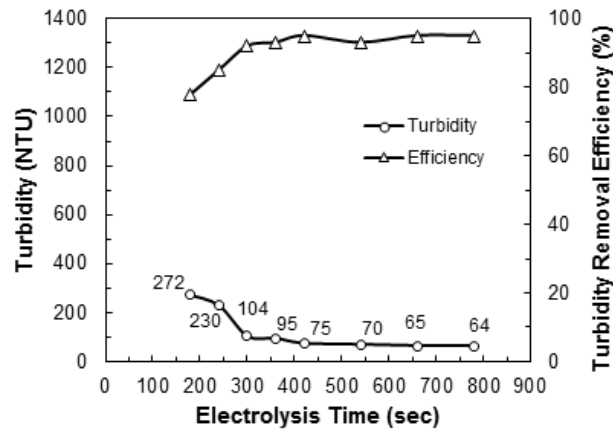


Fig. 3. Effect of electrolysis time on electrocoagulation

At the end of the experiments, the optimum test conditions were determined as follows; current density 40 (A/m²), electrolysis time 300 sec, mixing speed 360 rpm, mixing time for preconditioning 120 sec, and pH 7.8 (natural).

From all of these results, it was determined that the turbidity of the suspension under the optimum experiment conditions specified reduced from its initial level of 1260 NTU to 63 NTU level with a 95% coagulation efficiency. Operating costs were calculated to be \$5.67 per Mg of solids for cost calculations made based on electrode consumption and energy costs.

In literature, although there is no specific study focused on this subject, particularly for the EC of coal plant tailings, there are several studies done for clay, marble, drinking water, textile waste water etc. For example, Ozyonar and Karagozoglu (2012) studied the removal of turbidity of drink water using the EC, and the results showed that the optimum conditions for the removal process were obtained as current density 10 A/m², natural pH (7.9), and electrolysis time 3 min with 99% of the turbidity removal efficiency. Moreover, Solak et al (2009) used the EC for dewatering of marble plant waste tailings, and found the similar results such as current density 15 A/m², pH 8, and electrolysis time 2 min with 99% of the turbidity removal efficiency. Additionally, the studies in literature indicated that the best turbidity removal efficiencies were obtained at pH values between 6 and 8, and the results obtained from this study also agree with the literature (Murugananthan et al. 2004; Kobya et al. 2006; Bentli 2010).

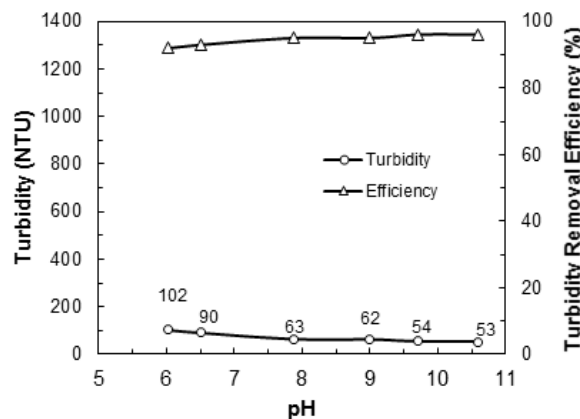


Fig. 4. Effect of pH on electrocoagulation

3.3 Electrocoagulation (EC) experiments in the presence of NaCl

Electrolytes such as NaCl, Na₂SO₄, and Na₂CO₃ are widely used in the EC process to increase the conductivity of the solution. NaCl is the most preferred due to its low price and easy production. In this context, the effect of different amounts of NaCl on EC was investigated.

The optimum experiment conditions obtained in the previous experiments were not changed in these experiments. The changes in pH and temperature were also monitored during the experiments.

For these experiments, 500 cm³ of a 1 mol/dm³ NaCl solution was prepared. The effect of NaCl on the EC was investigated by adding 10, 20, 30, and 40 cm³ (0.58, 1.17, 1.76, and 2.34 g/dm³) of prepared NaCl solution into the suspensions. The results of the experiments are seen in Fig. 7.

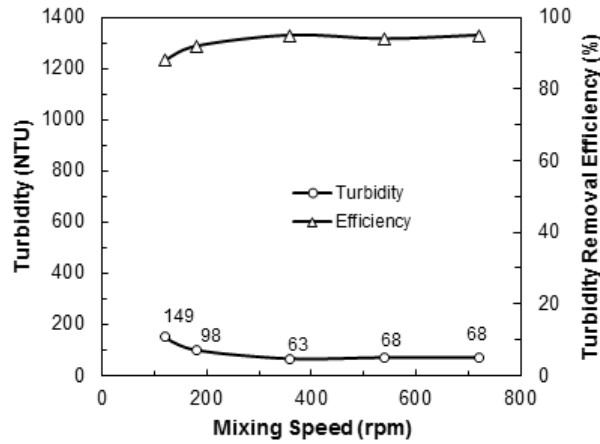


Fig. 5. Effect of mixing speed on electrocoagulation

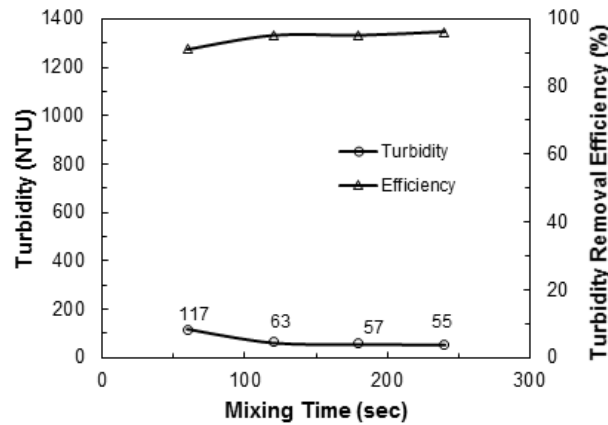


Fig. 6. Effect of mixing duration on electrocoagulation

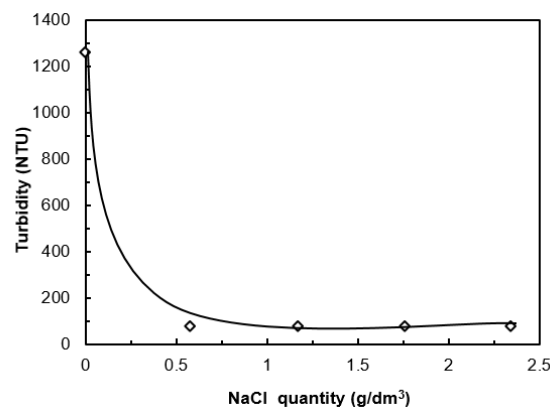


Fig. 7. Effect of NaCl on electrocoagulation

In the case of EC experiments in the absence of NaCl, the initial turbidity of the suspension (1260 NTU) decreased to 63 NTU level with 95% coagulation efficiency. The EC experiments in the presence of NaCl at different amounts showed that the best removal efficiency was obtained as 94% at 0.58 g/dm³ NaCl concentration. It seemed that there was no considerable change on the efficiency with the use of

NaCl, however, the calculations indicated that the electricity consumption for the EC experiments in the presence of Na⁺ ions reduced the energy consumption and saved up to 44% in operating costs. Likewise, Kilic et al. (2010) also found the similar results for the kaolinite suspensions using the EC.

The turbidity values increased to some extent (from 63 NTU to 74-78 NTU), and the coagulation efficiency reduced approximately 1% (from 95% to 94%) when the NaCl solution was added to the coal preparation plant tailings. If the Na⁺ ions caused the surface charge to be even more negative due to ion exchange of the clays contained in the suspension, then there would be a decrease in the coagulation of the particles.

Despite the 1% decrease in coagulation efficiency, a one-third decrease in the voltage values was observed during the experiment. Therefore, the costs of electricity consumption were significantly reduced. The operating cost, which was calculated to be \$5.67 per Mg of solids in the initial experiments, was reduced to \$3.19 per Mg of solids with the use of NaCl as a result of the decrease in the energy consumption. This allowed a 44% savings in the operating cost.

4. Conclusions

Electrocoagulation (EC) process has an advantage of removing colloidal particles because applied electric field sets them faster motion, thereby facilitating the coagulation. Additionally, the EC process avoids uses of chemicals, and therefore there is no problem of neutralizing excess chemicals, and no possibility of secondary pollution. In this study, the EC method in the absence and presence of Na⁺ ions (in order to increase the conductivity of the suspension) was used for the coal processing tailings. An Al electrode was used for the EC experiments, and several parameters were investigated to obtain the optimum parameters. The results for the EC experiments in the absence of NaCl showed that the optimum conditions were: pH value 7.88 (natural pH), current density 40 (A/m²), mixing speed 360 rpm, premixing time 120 sec, and electrolysis time 300 sec, and the turbidity of the suspension was also reduced from 1260 NTU (initial turbidity) level to 63 NTU level with a 95% coagulation efficiency. The operating cost for the EC process was calculated as \$5.67/Mg of solids. Additionally, the EC experiments in the presence of NaCl at different amounts showed no significant change on the turbidity. However, the addition of the NaCl reduced the energy consumption and saved up to 44% in operating costs. These results clearly indicated that this method can be successfully applied for the coal processing tailings.

Acknowledgements

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