Chemical Oxygen Demand and Turbidity Improvement of Deinked Tissue Wastewater using Electrocoagulation Techniques

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The goal of this work was to evaluate the efficiency of electrocoagulation technique in deinked tissue industry wastewater. The effect of two types of electrodes, three electrolysis times, four voltages, and three pHs were investigated. Experiments were conducted in batch process using a glass cell. The chemical oxygen demand (COD) removal and turbidity improvement of wastewater were measured and evaluated through the independent and interaction effects of variables. The results revealed that both electrodes reduced the COD and turbidity. However, the ability of their performance depended on the electrolysis time, voltage, and pH. For COD, greater improvement by electrocoagulation technique was obtained with an electrolysis time of 45 minutes, 24 volts, and alkaline conditions, while the turbidity reduction was achieved at lower voltage. Analysis of the treated water showed that the maximum COD and turbidity removal efficiencies were 81.12% and 89.43%, respectively. The treated effluent was very clear, and its quality met the industrial applications. Consequently, the electrocoagulation technique can be considered a reliable and safe method for deinked tissue effluent treatments to replace the other chemical methods.

Keywords: Electrocoagulation; Deinked tissue; Wastewater; Electrolysis time; Voltage; COD; Turbidity

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INTRODUCTION

Tissue manufacturing from waste paper is one of the largest paper industrial activities of wastewater. The process contains bath residues from pulp preparation, deinking separation and flotation, pulp cleaning and washing, dispersing, bleaching, and other important operations. These residues may cause severe pollution including dye, color, biological oxygen demand (BOD), and chemical oxygen demand (COD) if they are not properly treated before discharge into the receiving environments (Young and Akhtar 1998; Ali and Sreekrishnan 2001; Aksu and Gonen 2004; Ugurlu *et al*. 2008; Emamjomeh and Muttucumaru 2009; Terrazas *et al*. 2010).

The variation production process and kinds of waste papers used in recycled tissue causes the sophisticated composition of various pollutions in wastewater. It is difficult to treat and purify this wastewater and to estimate the hazards (Ali and Sreekrishnan 2001; Ragunathan and Swaminathan 2004). The treatment systems apply physical, chemical, biological, ozonation, or a combination of methods for effective purification of industrial paper wastewater due to the variation of wastewater characteristics (Basak and Gokay 1999; Fu and Viraraghavan 2001; Tezel *et al.* 2001;

Santos *et al*. 2002; Ragunathan and Swaminathan 2004; Terrazas *et al*. 2010; Tezcan UN *et al*. 2016; Hubbe *et al*. 2016).

Each of these methods has advantages and disadvantages. For example, the biological methods are inadequate due to the presence of lignin and other high molecular weight chemicals (Khansorthong and Hunsom 2009). Chemical methods are economically inefficient, due to their high costs (Cecen *et al.* 1992; Zaied and Bellakhal 2009). Ozonation is quite effective in decolorizing wastewater, but it is expensive and not efficient for COD and BOD_5 reduction (VanTran 2009).

Electrochemical coagulation oxidation has become an alternative for wastewater treatment and will be starting to replace traditional processes. Many researchers have investigated the electrochemical oxidation for various types of wastewater containing 1-4-benzoquinone, phenol, olive oil, vinasse, dichloromethane, ammonia, cadmium, unclear wastes, human and domestic wastes, textile wastewater, metal recovery, paint wastes, pulp, and paper waste waters (Korbahtia *et al.* 2007; Khansorthong and Hunsom 2009; Körbahtia and Abdurrahman 2009; Soloman *et al.* 2009; Demirci *et al*. 2015; Zayas *et al*. 2015).

The electrochemical coagulation of pulp and paper effluent has been studied and it is reported that the use of this method for wastewater treatment in the pulp and paper industry is technically possible. (Young and Akhtar 1998; Kobya *et al.* 2003; Mahesh *et al.* 2006; Parama *et al.* 2009; Soloman *et al.* 2009; Khansorthong and Hunsom 2009; Terrazas *et al*. 2010; Sridhara *et al*. 2011; Demirci *et al.* 2015; Zayas *et al.* 2015; Tezcan UN *et al*. 2016). However, the complete mineralization of heavily loaded effluent will not be economically viable. Thus, it may be possible to distribute treatment loads by integrating various techniques. The feasibility of electrochemical degradation using various electrode materials for wastewater treatment has been investigated with different components. But, in general, iron or aluminum is used more than other materials as electrodes. (Korbahtia *et al.* 2007; Körbahtia and Abdurrahman 2009; Soloman *et al.* 2009).

In this work, the electrocoagulation technique was investigated for treating the deinked tissue wastewater in order to improve the COD and turbidity and re-use of purified wastewater.

EXPERIMENTAL

Materials

All chemicals used were analytically pure and supplied by Latif Tissue Production Company from Alborz province, Karaj in Iran. Wastewater was sampled from Latif Tissue Production Company in overflow of physical treatment before chemical treatment. The wastewater was characterized for COD and turbidity using standard methods (APHA 2001). The characteristics of the wastewater are shown in Table 1.

Methods

Experiments were conducted in batch process using glass cell, 1000-mL capacity as a reactor. The iron and aluminum electrodes were positioned vertically and parallel to each other with an inner gap of 1 cm. Four pairs of electrodes of the same kind (Iron and Aluminum separately), each with active area of 50 cm^2 (10 cm × 5 cm) were used. The electrode plates were cleaned manually by abrasion with sandpaper and by treatment with

15% hydrochloric acid followed by washing with distilled water prior to every run. A total of 2 gL^{-1} sodium chloride was added to the wastewater as supporting electrolyte.

The wastewater was adjusted to the required pH by using either 0.1 molar sulphuric acids or 0.1 molar sodium hydroxide. One-thousand mL of the wastewater was poured into the glass reactor, where it was in contact with 50 cm^2 of the electrode surface. The solution was constantly stirred at 200 rpm with a magnetic stirrer to maintain a uniform concentration. Direct current (at 6, 12, 18 and 24 volt) was supplied to the electrodes in mono-polar mode according to the required current density, and the experiments were carried out under constant current conditions. Samples were collected for measuring COD and turbidity at three pH ranges (5.5, 7 and 8.5), three ranges of electrolysis time (15, 30 and 45 minute [min]), and four voltages (6, 12, 18 and 24 volt). Sampling was done in overflow of physical treatment before chemical injection in Latif Paper Company.

Statistical Analysis

Data were analyzed by SAS statistical software (Cary, NC, USA) based on a completely randomized design with a factorial test (Table 2). The comparison of the means was done employing Duncan's multiple range test (DMRT) to identify the groups that were significantly different from others at 95% confidence levels.

RESULTS AND DISCUSSION

The main characteristics of wastewater samples are shown in Table 1. The overflow of physical treatment of wastewater in deinked tissue paper making and the overflow of chemical treatments were considered as trial territory and target, respectively. In the tissue mill, the effluent from deinked and flotation systems were gathered in the equalization tank for homogenizing the characteristics. To reduce the cost of wastewater, before chemical treatments, the wastewater was sent to a physical purification stage and a considerable reduction of contaminants level was achieved in this stage. The fiber and other contaminants that settled in the bottom of system were drained and went to the sludge press section. The overflow of the physical stage had been considered as a sampling place. In general, after chemical treatment in the mill, the characteristics of the wastewater improved, as indicated by the values listed in Table 1. The goal was to replace the electrocoagulation process with chemical purification. So the overflow of physical stage was sampled and treatments were performed in order to achieve the best results in comparison with the physical and chemical purification stage. In traditional systems after chemical purification, the purified water can be used for internal applications. But, in order to send to receiving environments, it should be treated to biological stage. When the COD amount is higher than a certain level (for example 1500 mg/L), the efficiency in biological stage goes down.

As shown in Table 2, the study was mainly focused on the electrocoagulation of the deinked tissue industry wastewater for determining the effects of the operating parameters such as initial pH, kind of electrodes, voltage, and electrolysis time on COD and turbidity removal. The factorial test was used for all treatments.

Table 1. Characteristics of Wastewater Samples

Variables	COD	BOD	Turbidity	TDS	TSS	E.C	pH
Unit	mg/L	mg/L	NTU	mg/L	mg/L	(μs/cm)	-
Equalization tank	>5000	2050	>1000	2800	1075	3.4	7.02
Overflow of physical treatment as a blank	2808	1105	124	1339	135	2.6	7
Overflow of chemical treatment as a target in the mill	1276	510	65	1200	90	2.6	7

Table 2. Distinctions of Variables

Variables	1	2	3	4
Electrodes	Fe	Al	-	-
pH	7	5.5	8.5	-
Electrolyze Time (minute)	15	30	45	-
Voltage (V)	6	12	18	24

COD Removal

Initially, the experiments analyzed the independent effects of variables on COD improvement. The COD was reduced from 2808 mg/L to 1202 and 1315 mg/L, thereby giving 57.2% and 53.2% COD removal efficiencies for iron and aluminum electrodes, respectively (Fig. 1). The COD was decreased to 1169, 1330, and 1274 mg/L with the efficiency of 58.4%, 52.63%, and 54.6% for alkaline, natural and acidic conditions, respectively. The COD decreased to 1480, 1258, and 1039 mg/L with the efficiencies remaining at 47.3%, 55.2% and 63% for 15, 30, and 45 min of electrolysis time, respectively. The COD was reduced from 2808 mg/L to 1488, 1362, 1149, and 1034 mg/L, resulting in 47%, 51.5%, 59.1%, and 63.2% efficiency in removing COD from wastewater for 6, 12, 18, and 24 volts, respectively. Iron electrode reduced COD more than aluminum electrode. In alkaline conditions, the efficiency of electrocoagulation increased. COD further decreased with increasing electrolysis time or voltage (Fig. 1).

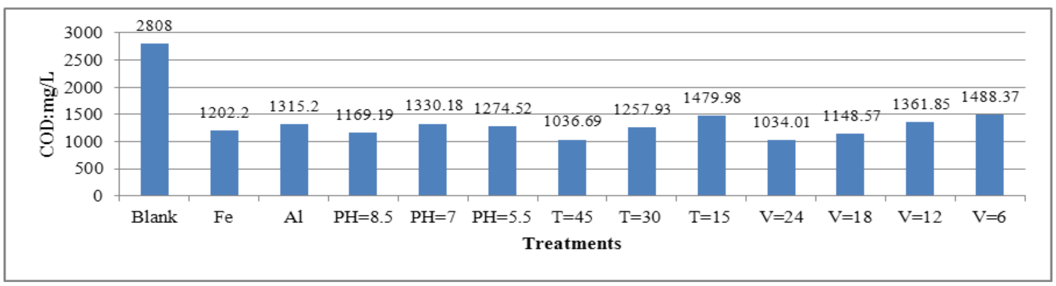

Fig. 1. Independent effects of variables on COD reduction in deinked wastewater

Next, the interactions of different levels of variables were studied for the reduction of COD. Under constant electrode type, when pH of wastewater was changed to alkaline conditions, electrolysis times were increased from 15 to 45 min. When the voltage was increased from 6 to 24 Volts, COD reduction took place faster than other treatments. In all treatments, the results for iron electrode were better than with the aluminum electrode. The best efficiencies of COD improvement obtained under the interaction of iron electrode with the alkaline pH, 45-min electrolysis time or 24 Volts were 60.27%, 65.47%, and 64.92%, respectively. Meanwhile, the COD removal efficiency was 56.44%, 60.68%, and 61.43%, respectively, for the aluminum electrode in the same conditions (Fig. 2). Regardless of electrode type, the interaction of electrolysis times, or voltage with different pH, under alkaline conditions with 45 min of electrolysis or 24 Volts, the COD removal efficiency was 66.81% and 67.62% respectively (Fig. 3).

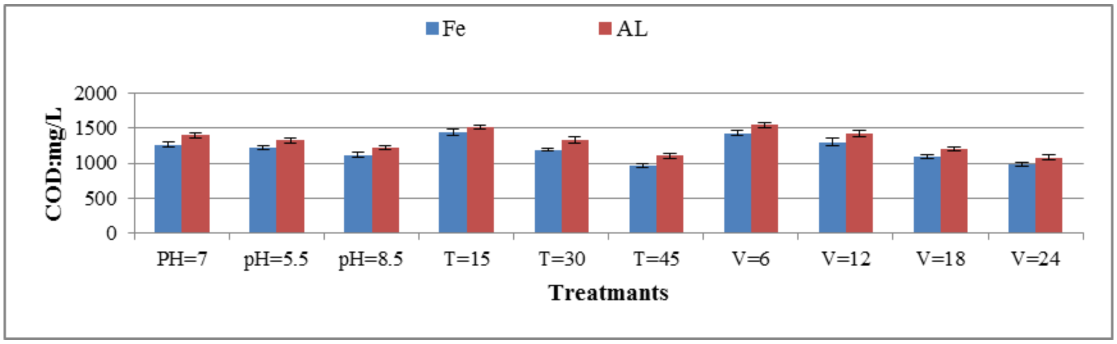

Fig. 2. The interaction of pH, electrolysis time, voltages, and electrodes for COD reduction in deinked wastewater

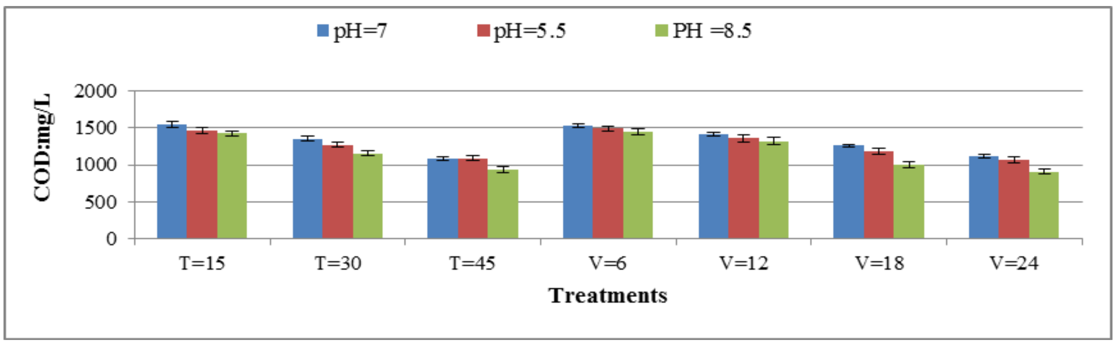

Fig. 3. The interaction of electrolysis time, voltages, and pH for COD reduction in deinked wastewater

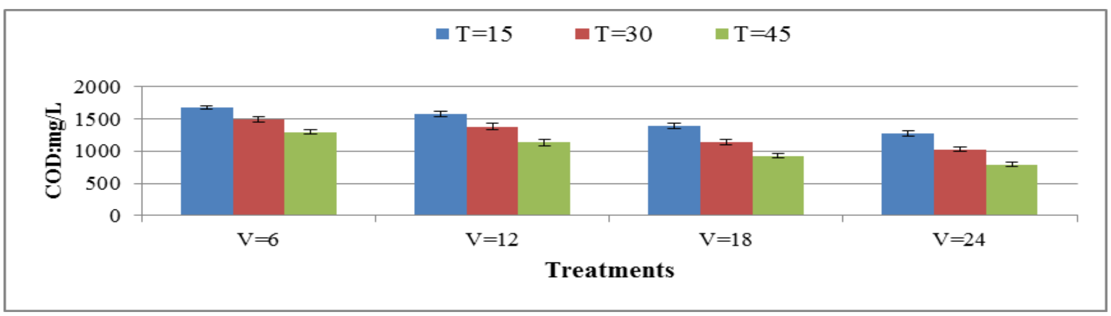

Fig. 4. The interaction of voltages and electrolysis time for COD reduction in deinked wastewater

When the electrolysis time or voltage increased at a certain pH, COD decreased. At the same electrolysis time or voltage, COD improved when the pH changed from neutral to acidic or alkaline conditions. At a constant voltage, COD decreased further with increasing time and *vice versa*. Therefore, the best result was obtained from an electrolysis time of 45 min and 24 Volts, resulting in a COD of 792.17 mg/L with efficiency of 71.79% (Figs. 4 and 5).

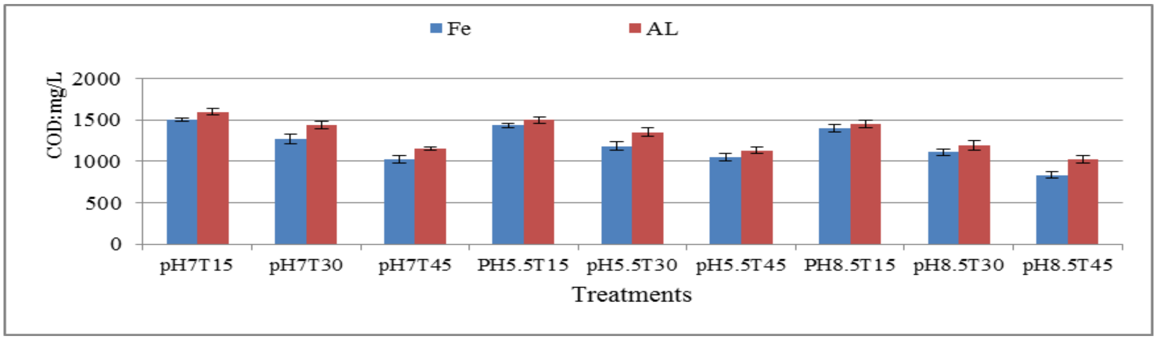

Fig. 5. The interaction of pH, electrolysis time, and electrodes for COD reduction in deinked wastewater

The interaction of pH, voltage, and electrode type showed that the best treatment for COD reduction was pH 8.5 and 24 V with an iron electrode, resulting in a reduction from 2808 mg/L to 834 mg/L, compared with reduction to 984 mg/L with aluminum electrodes (Fig. 6). When the time was replaced instead of pH, the reduction of COD continued to 721 mg/L and 863 mg/ L with the efficiencies of 74.32% and 69.26% for iron and aluminum electrodes, respectively (Fig. 7). The best results were obtained as 676 mg/L (75.93% removal efficiency) in alkaline pH, 45 min of electrolysis time, and 24 volts (Fig. 8). The interactions of triple variables showed the effect of “pH × electrolysis time × voltage” was more effective in COD reduction than “electrolysis time × voltages × electrode types” and “pH × voltages ×electrode types”.

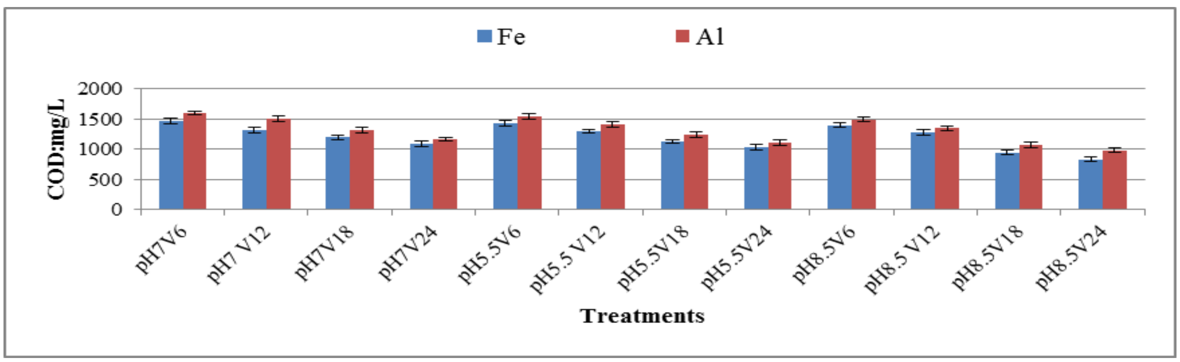

Fig. 6. The interaction of pH, voltages, and different electrodes for COD reduction in deinked wastewater

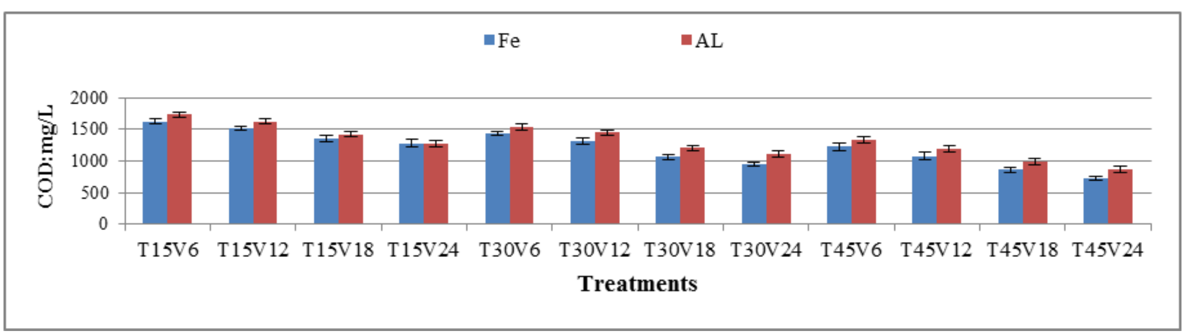

Fig. 7. The interaction of electrolysis times, voltages and different electrodes for COD reduction in deinked wastewater

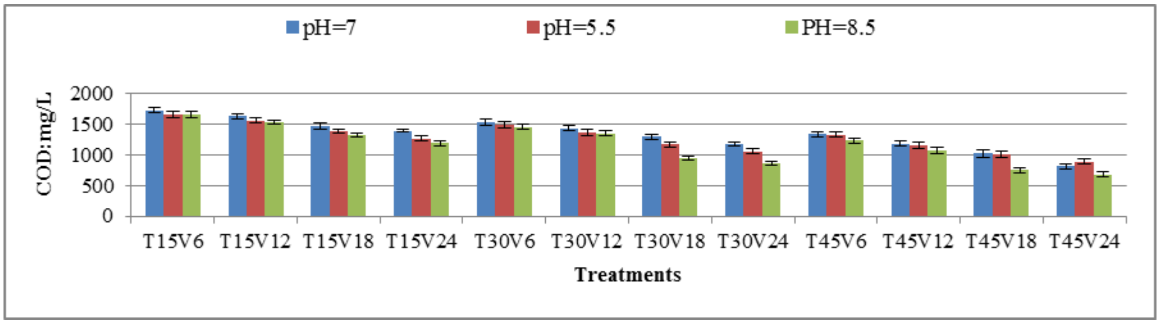

Fig. 8. The interaction of electrolysis times, voltages and pH for COD reduction in deinked wastewater

At a certain pH and electrolysis time, COD was decreased as voltage was increased. In this situation, the slope of the decrease was higher with iron electrodes than aluminum electrodes. At a certain pH, COD was decreased with the increase in electrolysis time. Considering all conditions, the treatments with pH 8.5, electrolysis time 45 min, 24 volts with iron or aluminum electrodes improved the COD from 2808 mg/L to 530 mg/L and 822 mg/L, respectively (Fig. 9). The efficiency of COD removal was calculated 81.12% and 70.72% for iron and aluminum electrodes, respectively.

The impact of voltage on COD reduction can be attributed to anode dissolution, which was greater at higher voltages, Consequently, cationic hydroxy compounds in the electrolyte were increased, which neutralize the pollution level, increase the force of attraction between them, and improve coagulation to result in a reduced COD. In addition, the increase in voltage with volume and speed of bubbles and the reduction of their size intensifies the upward flow generated within the reactor. This process increased the release of metal hydroxy, reinforced the formation of floated sludge on the surface of the liquid, and reduced the load of COD in the wastewater (Kobya *et al.* 2003; Chen 2004).

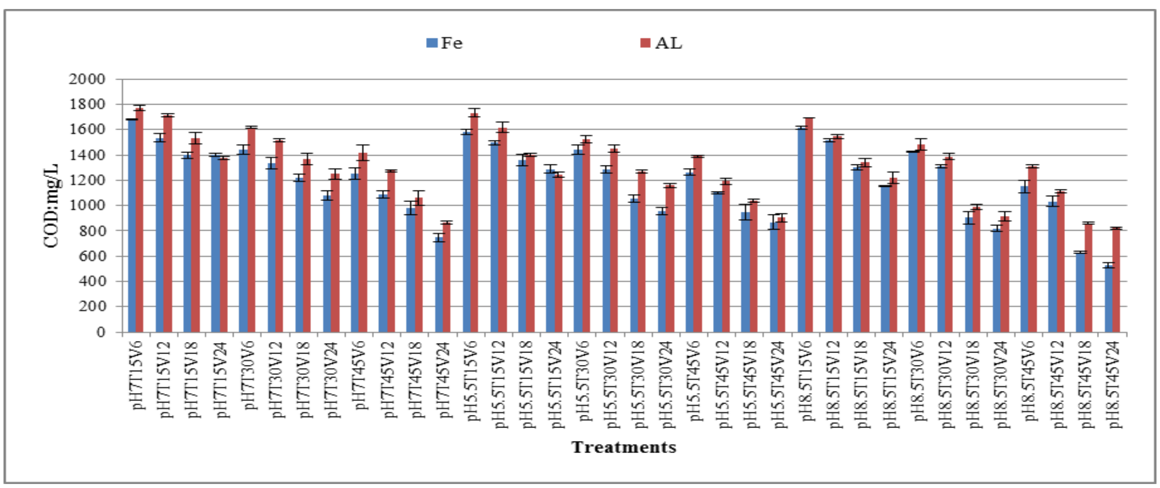

Fig. 9. The interaction of pH, electrolysis time, and voltages with different electrodes for COD reduction in deinked wastewater

The importance and the effect of pH on electrochemical process have been studied (Chen 2004; Zayas *et al.* 2015). As shown, the maximum reduction in COD took place in alkaline conditions with higher electrolysis time and voltage (Fig. 9). As reported (Khansorthong and Hunsom 2009), the COD of wastewater was affected by different factors such as electrolysis time, voltage, and pH. The COD was significantly affected by the initial pH of paper mill wastewater. The results showed that the maximum improvement in COD reduction was obtained in the range 4.5 < pH < 7, which is not consistent with the results of the present study. In a study using electrochemical technique with iron electrodes, Zaied and Bellakhal (2009) showed reduced COD in paper mill wastewater with increased electrolysis time and voltage. Tezcan *et al.* (2016) reported that the COD improved with an increasing reaction time from 15 to 60 min. In addition, a maximum COD removal was achieved after 15 min.

In this research, the effect of electrode material was examined using iron and aluminum electrodes with the same shape and dimensions. As can be seen from Figs. 1, 2, 5, 6, 7, and 9, the iron electrode was found to be more effective than aluminum electrode. It seems that a greater amount of iron was released from the anode compared with amount the aluminum released, which can result in higher removal efficiency. Tezcan *et al.* (2016) announced that the results are an agreement with Faradays low. Kobya *et al.* (2003) indicated that the efficiency of iron electrodes was higher than other electrodes and that the best results were obtained at pH 6 to 9, which is consistent with the results of this study.

Turbidity Improvement

Turbidity of deinked wastewater can be improved in alkaline conditions with higher electrolysis times and voltages using aluminum electrodes as compared with control samples and iron electrodes. The effect of independent variables on turbidity reduction in deinked wastewater showed that the turbidity was improved from 124 FTU in a blank sample to 38.66 and 36.24 FTU with the efficiencies at 68.82% and 70.77% for iron and aluminum electrodes, respectively. Turbidity was decreased to 31.98, 46.94, and 33.42 FTU with the efficiencies of 74.2%, 62.14%, and 73.04% for alkaline, neutral, and acidic conditions, respectively. The turbidity was decreased to 52.52, 39.10, and 20.71 FTU with the efficiencies at 57.64%, 68.46%, and 83.29% for 15, 30, and 45 min electrolysis time, respectively. The turbidity was reduced from 124 FTU to 48.88, 33.28, 35.55, and 32.06 FTU, equaling 60.58%, 73.16%, 71.33%, and 74.14% efficiency in removing turbidity in 6, 12, 18, and 24 Volts, respectively. Consequently, the aluminum electrode reduced the turbidity more than iron electrode. The efficiency of electrocoagulation techniques was increased in alkaline conditions. The turbidity was decreased more with increasing electrolysis time or voltage (Fig. 10).

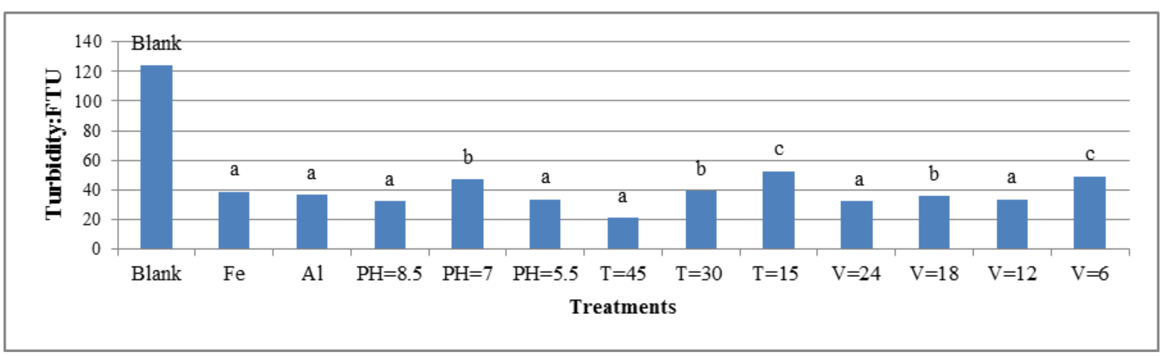

Fig. 10. The effect of independent variables on turbidity reduction in deinked wastewater

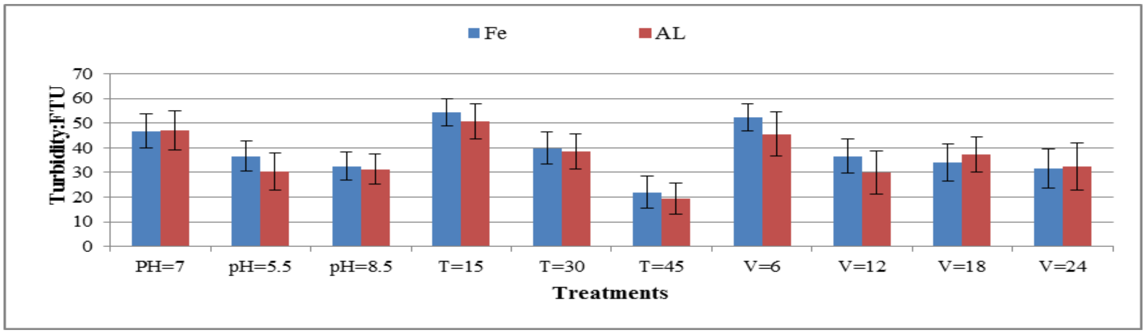

Fig. 11. The interaction of pH, electrolysis time, voltages, and electrode for turbidity reduction in deinked wastewater

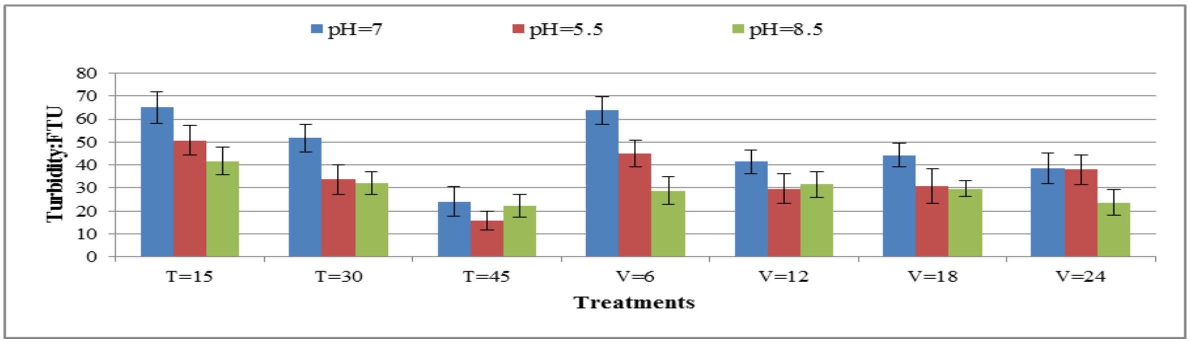

Fig. 12. The interaction of electrolysis time, voltages, and pH for turbidity reduction in deinked wastewater

The interaction of pH and electrode type, and voltage and electrode type did not cause significant differences ($p > 0.05$). The interaction of electrolysis time and electrode type, and voltage and electrode type influenced turbidity reduction in deinked wastewater. At higher electrolysis time, a significant reduction in turbidity was observed ($p < 0.05$). The lowest turbidity was obtained with aluminum or iron electrodes with 45 min of electrolysis from 124 FTU to 19 and 22 FTU, with efficiency of 84.46% and 82.25%, respectively. These results were obtained in acidic and alkaline conditions with higher voltages (Figs. 11 and 12).

The interactions of voltage and electrolysis time showed that turbidity was reduced at higher voltages and electrolysis times. The lowest turbidity was obtained as 14 FTU under the voltage of 18 Volt and electrolysis time of 45 min resulting in 88.70% removal of color efficiency (Fig. 13). In most treatments, different electrodes resulted in the same outputs but under a certain pH, turbidity was reduced with increasing electrolysis time. In a constant electrolysis time with different pH, the alkaline condition resulted in lower turbidity (Fig. 14). The best result for turbidity was achieved at pH 8.5 with 24 volts using iron electrode (Fig. 15). At a certain electrolysis time, turbidity was decreased with increasing in voltage or at a certain voltage with increasing the electrolysis time. Turbidity was reduced to 13 and 15 FTU in 45 min combined to 24 volts using iron or aluminum electrodes, respectively (Fig. 16).

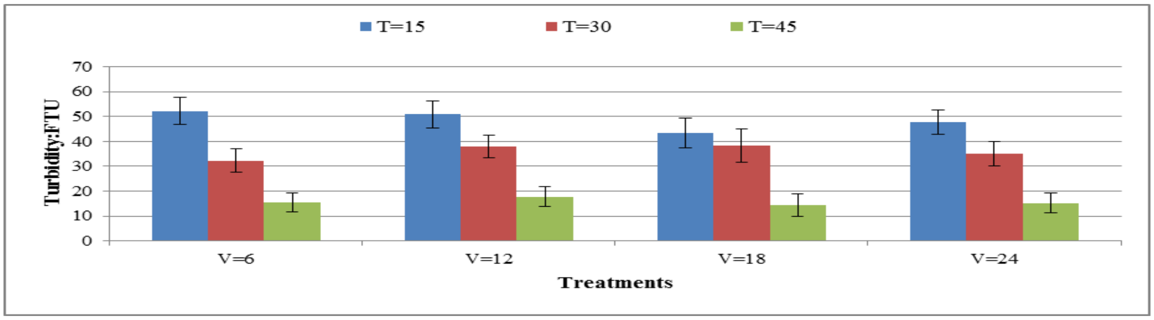

Fig. 13. The interaction of voltages and electrolysis time for turbidity reduction in deinked wastewater

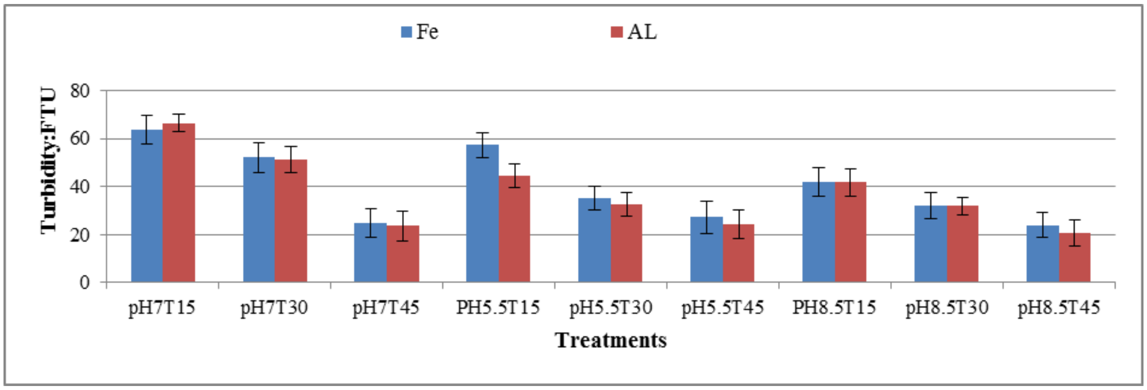

Fig. 14. The interaction of pH, electrolysis time and electrode for turbidity reduction in deinked wastewater

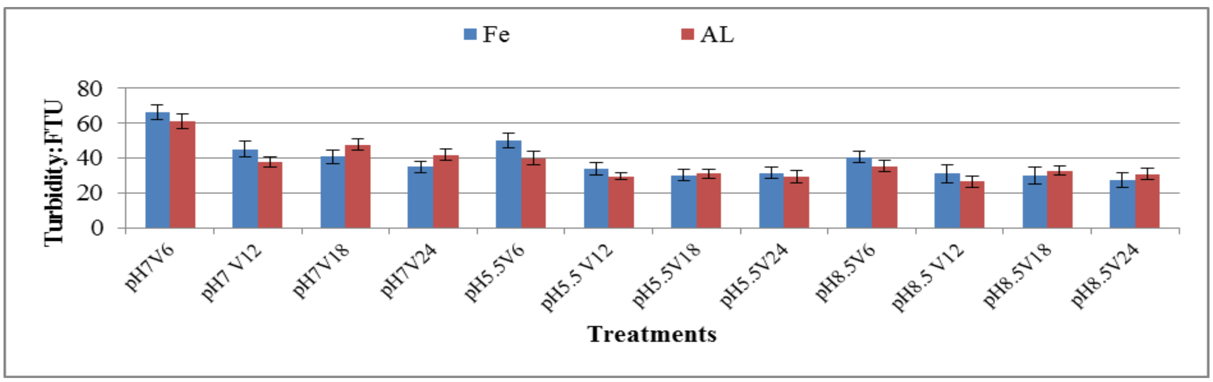

Fig. 15. The interaction of pH, voltages, and electrode for turbidity reduction in deinked wastewater

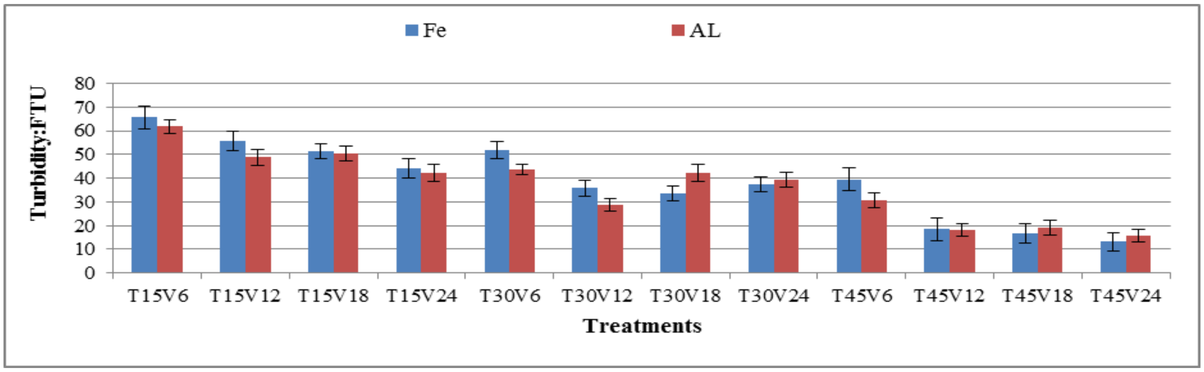

Fig. 16. The interaction of time, voltages and electrode for turbidity reduction in deinked wastewater

At a certain pH and electrolysis time, the increase in the voltage resulted in lower turbidity. Turbidity was reduced with increasing electrolysis time at a certain pH and voltage. The greatest reduction took place with electrolysis time of 45 min and 24 volts in neutral, acidic and alkaline conditions (Fig. 17). There were no significant differences ($p > 0.05$) in turbidity between electrode types. Turbidity was decreased with increasing voltage at a certain pH and time. Turbidity was reduced by changing pH of wastewater from neutral to alkaline or acidic conditions. The lowest turbidity was obtained from the interaction of acidic condition, 45-min electrolysis time, and 24 volts with iron or aluminum electrodes. Consequently, turbidity was decreased from 124 FTU to 13 and 14.33 FTU with the efficiencies of 89.51% and 88.44% for iron and aluminum electrodes, respectively (Fig. 18).

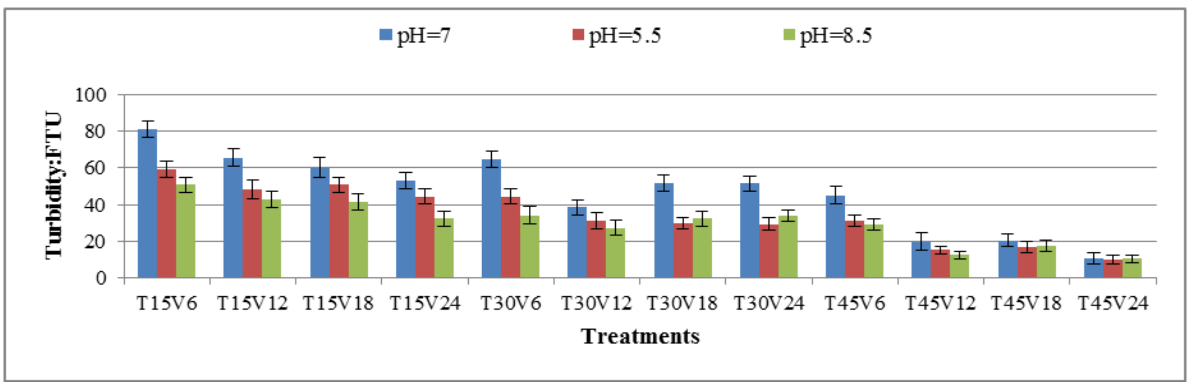

Fig. 17. The interaction of electrolysis time, voltages and pH for turbidity reduction in deinked wastewater

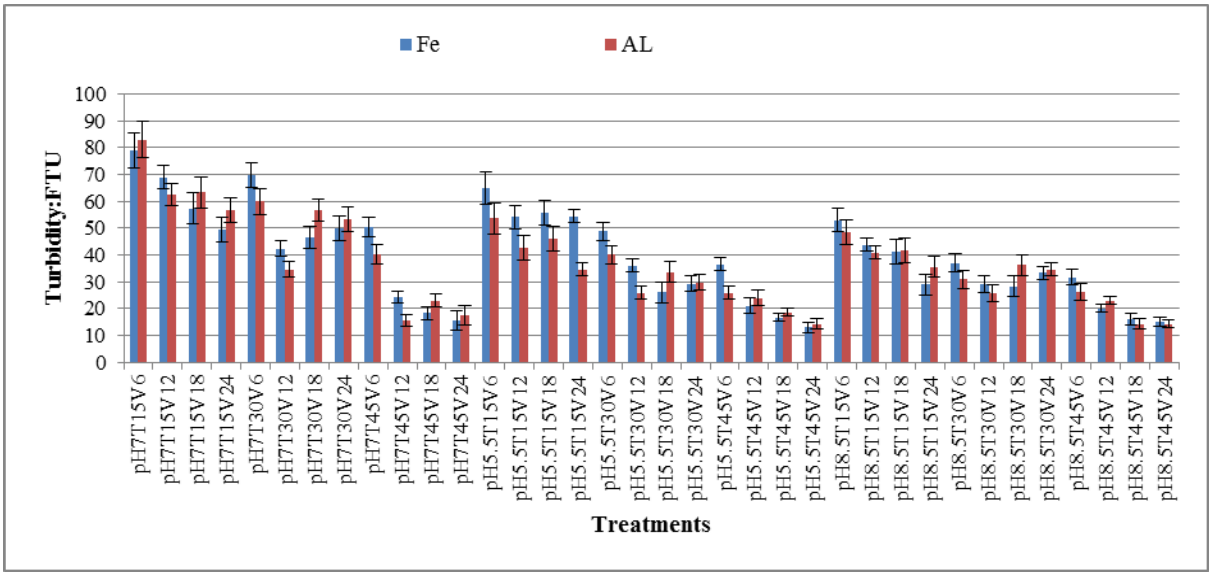

Fig. 18. The interaction of pH, electrolysis time and voltages with electrode for turbidity reduction in deinked wastewater

Turbidity was decreased with increasing voltage. The increasing of voltage increased the coagulation rate by production of air bubbles and reduced their size on the liquid surface at reactor. The efficiency of turbidity reduction was increased at higher electrolysis times under certain conditions, which has been attributed to the greater solubility of anode (Kobya *et al*. 2003; Terrazas *et al*. 2010).

Turbidity was significantly reduced by the electrochemical process (Parama *et al*. 2009). Some researchers reported a positive effect of voltage and electrolysis time on the reduction of turbidity in an urban restaurant (Chen *et al*. 2002; Terrazas *et al*. 2010; Demirci *et al*. 2015). Ben Mansour *et al.* (2007) showed that turbidity was decreased with increasing of electrolysis time and voltage in the electrochemical process. They also stated that the initial pH was more effective on turbidity reduction and proposed that alkaline condition (pH 8.5) had a suitable efficiency in reducing turbidity of wastewater.

Table 3. The Importance of Variables on Output Improvement in the Research Using SPSS Clementine Software

Turbidity	COD	Variable
0.359	0.403	Voltage
0.390	0.315	Time
0.247	0.234	pH
0.003	0.048	Electrode Kinds

The results showed (Table 3) the importance of voltage for COD reduction was greater than electrolysis time and pH, but the importance of electrolysis time was greater in turbidity improvement than voltage and pH. The effect of electrodes was less important than other variables for both outputs. Given the whole restrictions, the effect of voltage was more important than electrolysis time. After the electrocoagulation process, the wastewater should be treated in biological sections, and it is fed into the biological stage with lower COD that is safer and has higher performance.

CONCLUSIONS

1. The efficiency of electrocoagulation technique for deinked pulp wastewater treatment was evaluated. The electrocoagulation process was effective in decreasing Chemical oxygen demand and turbidity of deinked wastewater effluents.
2. Electrode materials, time of reaction and electrolysis, pH, and voltages are significant parameters affecting COD removal and turbidity improvement.
3. The turbidity and COD reduction are very important for deinked pulp wastewaters. With electrolysis time of 15 to 45 minutes, COD and turbidity were reduced in all cases. The results showed that the turbidity and COD were reduced by 89.52% and 80.06%, respectively, in comparison to the overflow of the physical stage and 80% and 56.11%, respectively, to the overflow of the chemical stage (based on data in Table 1).
4. The deinked wastewater purified with electrocoagulation technique can be consumed directly in internal processes. The treated wastewater has the advantage of containing less organic loads (lower COD and turbidity) in case of feeding into biological treatment stage.
5. The electrocoagulation process is a reliable, safe, and cost-effective technique for deinked wastewater purification. It can replace a chemical purification stage in sequence of physical, chemical, and biological stages.

ACKNOWLEDGMENTS

This manuscript is a part of a research plan entitled, ‘‘Efficiency of electrochemical treatments on purification of waste water at paper making mills,” performed at Islamic Azad University, Karaj branch, Iran. The author is thankful for this institutional support.

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Article submitted: November 16, 2016; Peer review completed: March 30, 2017; Revised version received: April 13, 2017; Accepted: April 15, 2017; Published: April 28, 2017.
DOI: 10.15376/biores.12.2.4327-4341