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Removal of lignin, COD, and color from pulp and paper wastewater using electrocoagulation

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ABSTRACT

Electrocoagulation is an effective, fast, and economic method for treatment of industrial wastewaters. In this study, effects of different parameters including electrolysis time, voltage, and pH on the reduction of chemical oxygen demand (COD), lignin, and color in pulp and paper wastewaters were studied. Iron and aluminum were used as anode and cathode electrodes, respectively. Under the optimal conditions (pH 5, 60 min, 10 V), this treatment method led to 85% removal of COD and 78.5% removal of lignin. Furthermore, clear treated water with complete color removal was generated that suggests the application of electrocoagulation for industrial wastewater treatment, especially in pulp and paper industries.

Keywords: Pulp and paper; Wastewater treatment; Electrocoagulation; COD removal; Lignin removal; Color removal

1. Introduction

Discharging of wastewater into the rivers negatively affects the quality of the water resources and puts human and animal life in danger. This change in quality can be observed by physical, chemical, or biological characteristics of the water [1]. The wastewater from the pulp and paper industry is the sixth largest polluter (USEPA 1998) and possesses high organic matter, blackish color, chemical oxygen demand (COD), biological oxygen demand (BOD), and suspended solids. However, the exact characteristics of this wastewater depend on the type of raw material, type of treatment method, and internal recirculation of the effluent [2]. The strong color is a result of polymerization between tannin and lignin during the paper making operation [3]. In some cases, consumers may add dirt or impurities which cause a colored, odorous wastewater with higher BOD and COD concentrations. This impurity puts the aquatic life in danger and leads to mutation and release of carcinogenic materials in some cases [4–8]. Pulp and paper manufacturing companies utilize 76–227 m^3 water per ton of product [9].

In order to treat the industrial wastewater, different methods have been used including physical, chemical, and biological treatments [9–14]. Each method of treatment has its disadvantages. For instance, the cellulose and organic compounds with large molecular weight such as long cyclic groups of lignin cannot be decomposed by biological method [15]. This method is not efficient in color removal as well [16]. The biodegradability index of pulp and paper effluent is less than 0.4 which indicates that this specific wastewater cannot be treated well by biochemical method [3]. Anaerobic treatment is not a

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suitable method due to low efficiency and high cost [8,17]. Activated sludge plants have several problems such as poor settling characteristics, sensitivity to shock, toxicity, and inability of biodegrading poorly biodegradable substances such as lignin [15]. Ozonation, Fenton's reagent, and membrane technology are other methods which are useful but more expensive comparing to electrocoagulation [11,18]. Coagulation, flocculation, and surface adsorption, despite their effectiveness, are costly due to chemical materials needed through the treatment process. Furthermore, discharging these chemicals to the environment can cause new environmental problems [15]. The most important drawback for chemical oxidation methods is need of large amounts of reactive chemical reagents [19].

Electrocoagulation method has been examined for other effluents and demonstrated successful results [20–28]. Furthermore, electrocoagulation has several advantages compared to abovementioned methods of treatments. No chemicals are needed in this process, and the main reagent is the electron which is a clean reagent. Simple setup, producing less sludge, ability to remove the tiniest colloidal particles and shorter treatment time comparing to other methods are other advantages of electrocoagulation [29–31]. Mechanism of electrocoagulation is consisted of reactions in anode and cathode electrodes which are summarized in Table 1 [19].

There are a few published research studies about pulp and paper wastewater treatment using electrocoagulation. Khansorthong and Hunsom reported 97% of color and 77% of COD removal using iron and aluminum electrodes [15]. Kalyani et al. achieved color and COD removal efficiencies of 92 and 95%, respectively, against mild steel electrodes while using aluminum electrodes resulted in 84 and 89% removal efficiencies [3]. Uğurlu et al. studied the removal of lignin, phenol, BOD, and COD using aluminum electrodes observed 80%, 98%, 70%, and 75% removal efficiencies, respectively [19]. Removal of 98% COD, 92% polyphenols, and 99% color for black liquor utilizing aluminum as sacrificing electrode has been reported by Zaied and Bellakhal [8]. Al-Shannag et al. used iron electrodes and optimized initial pH, current density, circulating flow rate, and treatment time. They were able to achieve up to 80% removal efficiencies for both TSS and COD [32]. Sridhar et al. reported on reduction of 94% color, 90% COD, and 87% BOD under the optimum condition (15 mA/cm^2 current density, pH of 7, 1 g/L NaCl, 100 rpm, and 3 cm electrode distance) using aluminum as sacrificial electrode [2]. Shankar et al. achieved removal efficiencies of 77%, 78.8%, and 99.6% for COD, TOC, and color, respectively [33]. The COD removal efficiency of 85% and color removal of 94% were achieved by Sharma et al. under optimal conditions (15 mA/cm^2 current density, pH of 7, and treatment time of 2 h) [34].

Considering the advantages of electrocoagulation as a promising treatment method in response to increasing pulp and paper wastewater, we carried out this research in order to assess the competence of this method against real industrial wastewater. Most previously conducted studies in this area have dealt with synthetic wastewater where few of them examined iron as a sacrificial electrode for lignin removal. In this research, treatment of real wastewater from a pulp and paper factory was investigated in electrocoagulation process using Al (cathode) and Fe (anode) electrodes. Electrolysis time, pH, and voltage were chosen as variable parameters to reach the maximum removal of lignin, COD, and color. The energy consumption in each experiment was calculated as well.

Table 1
Reactions occurring in electrocoagulation process [19]

Anode (general)	Cathode (general)
$4OH^- + 4e^- \rightarrow 2H_2O + O_{2(g)}$	$2H_3O^+ + 2e^- \rightarrow H_{2(g)} + 2H_2O$ (in acid solution)
$2H_2O + 4e^- \rightarrow O_{2(g)} + 4H^+$	$2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^-$ (in alkaline solution)
$2Cl^- + 2e^- \rightarrow Cl_{2(g)}$	$O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$
	$Cl_{2(g)} + H_2O \rightarrow HOCl + H^+ + Cl^-$
Anode (Fe-anode)	Cathode (Fe-anode)
$Fe_{(s)} + 2e^- \rightarrow Fe^{2+}_{(aq)}$	$Fe^{2+}_{(aq)} + 2H_2O \rightarrow Fe(OH)_2 + 2H_2O$
$Fe(OH)_3 + OH^- \rightarrow [Fe(OH)_4]^-$	$[Fe(OH)_4]^- + 2OH^- \rightarrow [Fe(OH)_6]^-_3$ (at very high pH)
$Fe(OH)_2 + HOCl \rightarrow Fe(OH)_3 + Cl^-$	
$Fe^{2+} + e^- \rightarrow Fe^{3+}$	
$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H^+$	

2. Materials and methods

The process of electrocoagulation occurred in a bench-scale batch plexiglass setup (Fig. 1) having dimension of $15 \times 10 \times 50\ cm^3$. As seen in Fig. 1, the bottom of the setup built with steep walls. This will expedite the process of producing sludge due to the thought that this change in the setup will increase the efficiency by inducing faster aggregation of the hydrolysis products [19]. Total volume of wastewater in every experiment was 2 L. Iron and aluminum were utilized in the process due to their low cost. The total active surface area of the electrodes was $6 \times 8 \times 10\ cm^2$, and iron was the sacrificial electrode. There were three iron electrodes in between of three aluminum electrodes arranged in monopolar configuration. The distance between the electrodes was constantly 2 cm. A peristaltic pump (Heidolph, Germany) utilized for circulation of the wastewater and a digital DC power supply (IMV Corporation, Japan) operated at galvanostatic mode was used. The voltage range of the power supply was 0–40 V, and current was 0–10 A. The properties of wastewater including lignin, COD, and color were analyzed before and after each treatment, and all the experiments were conducted at room temperature. After performing the treatment, treated solution was filtered. The electrodes used in the experiments were washed with distilled water and then with 15% HCl before every experiment.

The wastewater used in this study was obtained from Chouka Wood and Paper Industry, Talesh, Iran, and its characteristics are as followed in Table 2. The wastewater taken from aerated lagoons exactly before releasing to the Tajan River. It was taken to the laboratory in closed containers and kept at 4°C.

The concentration of lignin and COD were determined by spectrophotometer at 700 and 600 nm, respectively. Both the dichromate method (standard methods 5220D), and lignin and tannin colorimetric removal method (standard methods 5550B) were used to determine the concentration of the COD and lignin, respectively [35]. Moreover, the color intensity was determined by measuring the treated sample absorbance at 450 nm by a spectrophotometer (Model named Unico—UV-2100). A digital calibrated pH-meter (HANNA pH 211) was used to measure the pH of the wastewater in experiments.

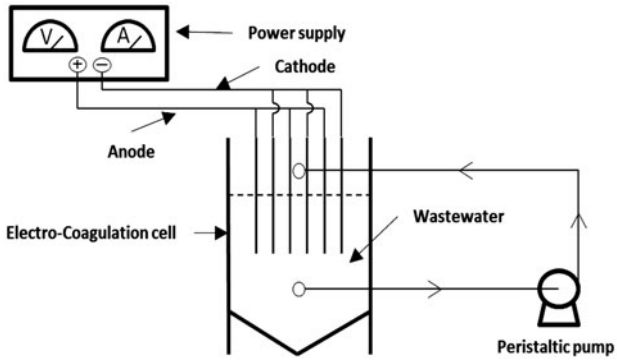


Fig. 1. Schematic diagram of the experimental electrocoagulation setup.

Table 2
Initial properties of the wastewater

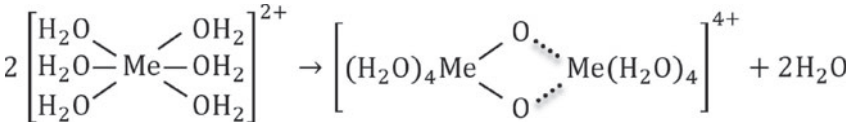
Property	Value
pH	7.5
Total COD	657 (mg/L)
Lignin	1,155 (mg Phenol/L)
Conductivity	1,175 (μS/cm)
Total dissolved solid	587 (mg/L)
Color	Brown

3. Results and discussion

Experiments were conducted on a wide range of operating conditions, and the results are presented in the form of figures.

3.1. Time effect

The removal of COD, lignin, and color at different electrolysis times (10, 20, 30, 40, 50, and 60 min) at the initial pH of 7.5, 4.0 V, and 600 mA were conducted. As shown in Fig. 2, 43% of lignin removal (82% of total removal) occurred in the first half of the operating time. The generation of Fe^{3+} in anode is the reason for particle aggregation. This aggregation and polymerization which takes place are depicted as Eq. (1) [19].



$$2\left[\begin{matrix}H_2O\\ H_2O-Me-OH_2\\ H_2O\end{matrix}\begin{matrix}\diagup\\ \\ \diagdown\end{matrix}\begin{matrix}OH_2\\ \\ OH_2\end{matrix}\right]^{2+} \rightarrow \left[(H_2O)_4Me\begin{matrix}\diagup O\cdots\\ \\ \diagdown O\cdots\end{matrix}Me(H_2O)_4\right]^{4+} + 2H_2O \quad (1)$$

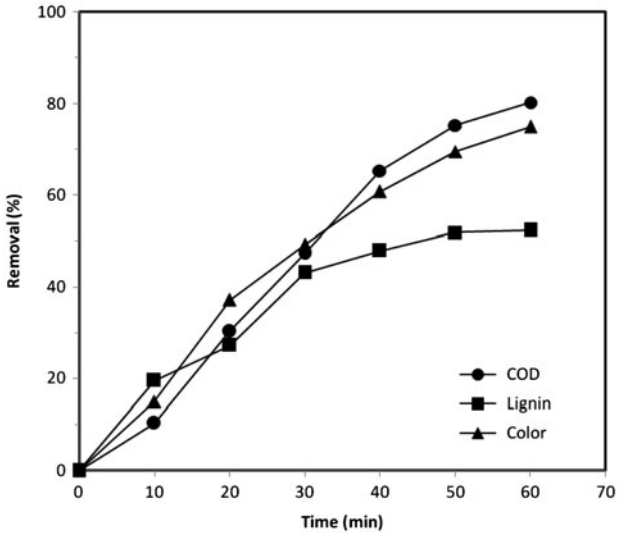


Fig. 2. Time course of COD, lignin, and color removal (initial pH of 7.5 and voltage of 4.0 V).

The different models of combination of these products may be responsible for the trend shown in Fig. 2. Gurses et al. [36] reported that Fe^{3+} may form ferric hydroxo complexes and polymeric species such as $Fe(H_2O)_6^{3+}$, $Fe(H_2O)_5OH^{2+}$, $Fe(H_2O)_4(OH)_2^{+}$, $Fe_2(H_2O)_8(OH)_2^{4+}$ and $Fe_2(H_2O)_6(OH)_4^{2+}$ depending on the initial pH of the wastewater. The variety and amount of these species which are key factor in removing of contaminants depend on electrolysis time.

Fig. 2 also demonstrates the COD removal percentage which is increased with increasing of the operating time without any fluctuation. This is due to the fact that by applying current in the electrodes, the Fe $(OH)_3$ flocks are generated and these flocks adsorb the organic molecules of the effluent resulting in COD reduction thorough the electrolysis time. A similar trend was observed in the color reduction of the effluent. As shown in Fig. 2, the trends of these factors show an approximate steadiness for the lignin removal after 60 min but there is a considerable COD removal between 50 and 60 min. Therefore, 60 min of operation time was selected for further studies.

3.2. Voltage effect

Several researchers reported that current density and voltage are important factors in the electrocoagulation process for contamination removal [20,21,37,38]. Higher voltages increase the amount of ferric ions that are produced from sacrificial electrodes and leads to more removal. In our experiments, the electrolysis time was set to 60 min and initial pH of the wastewater was 7.5. The effect of different voltages (4, 6.5, 10, 14 V) was investigated on COD, lignin, and color removal. It is crucial to find the optimum value for voltage since applying more current for the laboratory-scale setup will lead to waste of energy by heating the water without any positive effect on the removal [39].

As shown in Fig. 3, in the first experiment (at 4 V) more than 70% of color and COD were removed. The diminished rate of COD removal after 4 V can be attributed to the generation of high amount of Fe^{2+} which is able to react with dissolved oxygen of the wastewater according to Eq. (2) [15].

$$4Fe^{2+} + O_2 + 10H_2O \rightarrow 4Fe(OH)_3 + 8H^+ \quad (2)$$

There was also a declination of lignin removal between 10 and 14 V which is a result of attending lignin in the electrode surface. Therefore, the optimal voltage for the process was 10 V which results in 81.6% removal of COD, 83.2% removal of color, and 78.1% removal of lignin.

3.3. pH effect

Another factor in contaminant removal by electrocoagulation is pH which is an important factor for possible reactions that can happen in the electrocoagulation process. Particularly for iron, it can shift the

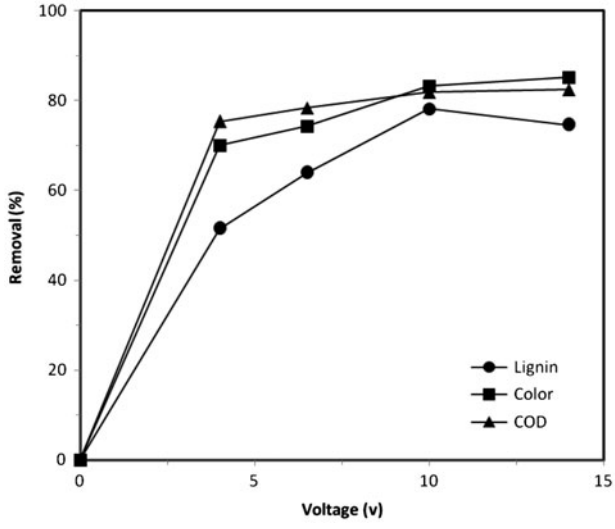


Fig. 3. Effect of voltage on COD, lignin, and color removal (initial pH of 7.5 and operation time of 60 min).

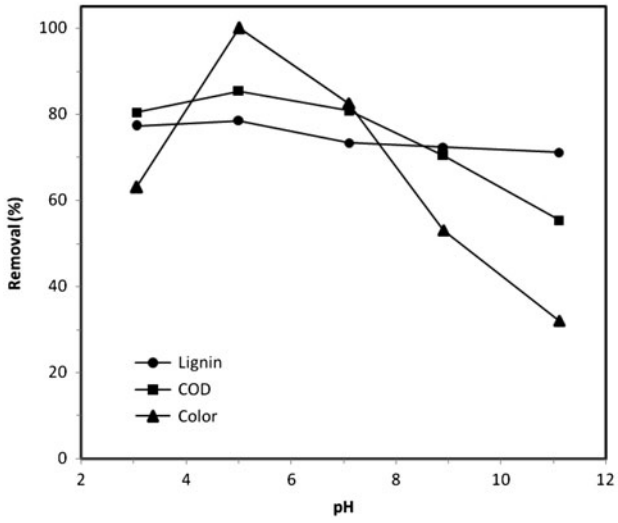


Fig. 4. Effect of pH on COD, lignin, and color removal (operation time of 60 min and voltage of 10 V).

Table 3
pH variation of the wastewater before and after treatment

Before treatment	After treatment
3.5	7.8
5	8.2
7.1	9.6
9.8	9.5
11.1	10.1

redox reactions between $Fe^{2+}$and Fe^{3+} which directly affects the contaminant removal. Therefore, the removal efficiency for lignin, COD, and color was investigated in different pH values as shown in Fig. 4. These experiments were carried out in 60 min and 10 V, which were the optimum amounts acquired in the previous sections. For adjusting pH, the H_2SO_4 and NaOH solutions were used.

As demonstrated in Fig. 4, at pH 5, the highest removal efficiencies for color (100%), COD (85.4%), and lignin (78.5%) have been achieved. The amount of sludge produced under acidic condition was more than that of the alkaline condition which is consistent with higher removals at pH 4. A sharp reduction in removal efficiencies was observed after pH 7 which is due to higher $Fe(OH)_3$ solubility that leads to the formation of $Fe(OH)_4$ which is completely soluble. Higher removal efficiency in weak acid condition is a result of large amount of released Fe^{2+} which reduces and removes contaminants.

According to Table 3, in pH values less than 7.1, wastewater demonstrates an increase in pH after treatment by electrocoagulation. However, the pH decreased when the initial pH of the wastewater was over 9.8. This increase under acidic condition is due to CO_2 release because of H_2 bubbles disturbance [21]. It can be understood from present results that electrocoagulation acts as a buffer for the pH of solution. This is consistent with findings of Zaied and Bellakhal [8].

As exhibited in Fig. 4, the highest removal efficiency was achievable under acidic condition. This can be attributed to formation of iron hydroxides which have higher adsorption capacity in this pH.

3.4. Energy consumption

The electrical energy consumption was calculated under optimum conditions (pH 5, 10 V, 60 min) using Eq. (3) [40]:

$$\text{Energy Consumption } (\text{kWh/m}^3) = \frac{V \cdot I \cdot t}{\text{treated volume (l)}} \quad (3)$$

where V is the cell voltage in volt, I is the current in ampere (A), and t is the treatment time (h). The energy consumption at the optimal condition was 8.334 kWh/m^3 which is less than a previous study [2].

4. Conclusion

The present research demonstrated a successful application of electrocoagulation in the treatment of pulp and paper wastewater. The impact of electrolysis time, voltage, and initial pH was investigated for the removal of COD, lignin, and color. Under optimum condition (10 V, current density of 4.167 mA/cm^2, initial pH of 5, operating time of 60 min), the removal efficiencies of lignin, total COD, and color were 78.5%, 85% and 100%, respectively. The treated wastewater under the optimum condition was visually clear with complete color removal. This reduction of COD, color, and lignin were directly proportional to the electrolysis time. pH of the wastewater after treatment and electrical energy consumption under optimum condition were 8.2 and 8.334 kWh/m^3, respectively. According to our results, this method can be successfully replaced or added to the existing treatment units of pulp and paper wastewater treatment such as biological methods.

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