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Research Article

Investigation of the effect of current density and PH on oil and grease removal from leather industry wastewaters by electrocoagulation method

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ABSTRACT

In this study, the effects of pH and current density on the removal of high concentrations of oil and grease in leather industry wastewater by the electrocoagulation method were investigated. Two different types of electrodes were used in the study. Studies with aluminum electrodes have shown that these electrodes are more effective than iron electrodes in removing oil and grease. In addition, aluminum electrodes are affected more by the system pH than iron electrodes. Studies have shown that higher oil and grease removal efficiencies are obtained below pH 4 for both electrodes. While the oil and grease removal efficiency with Al electrodes is 95% at pH 2, this value decreases to 83% at pH 6. In Fe electrodes, while the removal efficiency is 87.83% at pH 2, it decreases to 83% at pH 6. The effect of current density on oil and grease removal was examined and it was observed that the removal efficiency remained constant above a certain current density in aluminum electrodes. While the efficiency of Al electrodes is 83.31% at 0.6 mA cm^{-2} current density, this value has increased to about 98% at 1.2, 1.8, and 2.4 mA cm^{-2} current densities. In Fe electrodes, the oil and grease removal efficiency is 82.66% at 0.6 mA cm^{-2} current density, while it is 90% at 1.2 and 1.8 mA cm^{-2} current densities and 98% at 2.4 mA cm^{-2}. These results show that the electrocoagulation process removes the oil and grease in wastewater at a high rate. This shows that this process can also be used as pre-treatment before the biological treatment of industrial wastewater with high oil and grease content.

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INTRODUCTION

Water is one of the most important substances necessary for the survival of humans, animals, and plants. Water is constantly polluted due to overpopulation and uncontrolled industrialization [1]. Control of industrial pollution has become one of the most important environmental problems today.

Leather is one of the first materials used since the primitive ages. Humans who used the meat and milk of animals for nourishment realized that they could also use their skins [2]. The leather, which was used for veiling purposes in the early times, was later used for many purposes [3]. Leather processing processes can be classified as a liming process, tanning process, second tanning-dyeing process, and final processes [4, 5]. Abundant waste is generated in each of these units. The characteristics of the waste generated are affected by both the feed materials and the technology used [6, 7].

In the leather industry, a large amount of water is consumed in the leather processing process [8, 9]. Leather industry wastewater is a complex wastewater containing large amounts of pollutants in it due to the chemicals and processes used. The fact that leather industry wastewater is a complex wastewater is due to its high concentration of proteins, lipids, sulfur, sulfate, chlorine, dyes, surfactants, natural and synthetic tannins, as well as chemical substances and enzymes [10-13]. Leather industry wastewaters are problematic waters to be treated in conventional treatment systems, as it contains high amounts of oil and grease as well as organic and inorganic substances [14]. Therefore, new technologies are being researched for the treatment of these wastewaters. It attracts attention as one of these systems in electrocoagulation. This process seems very advantageous as it can remove many contaminants at the same time. However, in cases where electrical energy is expensive, optimization of the system is important.

Electrocoagulation; It is an electrochemical treatment system in which a soluble metal electrode with coagulant property is used as anode. The difference between electrocoagulation and chemical coagulation is the way of adding coagulants (aluminum or iron). Coagulation, flotation, adsorption, and chemical oxidation and reduction mechanisms occur together in electrocoagulation [15].

Electrode reactions when aluminum is used as electrode material [16];

At the cathode;

$$8H^{+} + H_2O + 8e^{-} \rightarrow 4H_{2(g)} \tag{1}$$

At the anode;

$$Al \rightarrow Al^{+3} + 3e^{-} \tag{2}$$

In the solution;

$$Al^{+3}_{(aq)} + 3H_2O \rightarrow Al(OH)_3 + 3H_{+(s)} \tag{3}$$

In the case of using iron as anode, two different mechanisms occur in the formation of $Fe(OH)_n$, depending on the pH of the environment, provided that n = 2 or 3 [17];

<u>Mechanism 1</u>

At the cathode;

$$8H^{+} + H_2O + 8e^{-} \rightarrow 4H_{2(g)} \tag{4}$$

At the anode;

$$4Fe(s) \rightarrow 4Fe^{2+}_{(aq)} + 8e^{-} \tag{5}$$

With dissolved oxygen in solution;

$$4Fe^{2+}_{(aq)} + 10H_2O_{(g)} + O_{2(g)} \rightarrow 4Fe(OH)_3 + 8H^{+}_{(aq)} \tag{6}$$

Finally, the total reaction can be summarized as;

$$4Fe_{(s)} + 2H_2O_{(g)} + O_{2(g)} \rightarrow 4Fe(OH)_{3(s)} + 4H_{2(g)} \tag{7}$$

<u>Mechanism 2</u>

At the cathode;

$$Fe_{(s)} \rightarrow Fe^{2+}_{(aq)} + 2e^{-} \tag{8}$$

At the anode;

$$2H_2O_{(g)} + 2e^{-} \rightarrow H_{2(g)} + 2OH^{-}_{(aq)} \tag{9}$$

Finally, the total reaction can be summarized as;

$$Fe_{(s)} + 2H_2O_{(g)} \rightarrow 4Fe(OH)_{2(s)} + H_{2(g)} \tag{10}$$

Although there are many articles about the use of the electrocoagulation process in industrial wastewater treatment in the literature, these articles mostly present studies on COD removal. The aim of this study is to determine how the oil and grease removal is affected by the pH and the current intensity of the wastewater, and its removal mechanisms while treating the industrial wastewater by electrocoagulation method.

MATERIAL AND METHODS

Materials

The wastewater used in electrocoagulation experiments was supplied from a local tannery. The properties of tannery wastewater are given in Table 1.

1000 mL of plexiglass reactor with a cooling jacket shown in Figure 1 was used while conducting the experiments. Experiments were carried out with 850 mL wastewater. In the experiments, 10 plates connected as 5 cathodes and 5 anodes were used as electrode material. The electrode surface area is 1200 cm^2 and the distance between electrodes is 5 mm. Plates are arranged vertically. All experiments were carried out at room temperature (25 ± 3°C). Before starting the experiments, the pH of tannery wastewater was adjusted using 0.1 M NaOH and 0.1 M HCl. The potential difference in the cell was provided by a digitally controlled direct current power supply (Shenzhen Mastech HY3005 3). The current and potential difference passing through the system was measured with the help of two digital multimeters (Brymen 201). The reactor contents were agitated continuously with a magnetic stirrer (Heidolph MR 3004) at 150 rpm. During the experiments, the pH, temperature, and conductivity of the wastewater were determined by a multiple parameter meter (WTW pH/Cond 340i). Samples taken from the reactor at certain time intervals were passed through a membrane filter (Schleicher & Schüll) with a pore diameter of 0.25 μm and analyzed. Concentrations of oil and grease were measured by Wilksir HATRT 2.

Table 1. Characterization of wastewater used

Parameters	Units	Value Range
pH		7.2–7.9
Conductivity	μs cm^{-1}	5500–9000
Turbidity	NTU	220–440
TOC	mg L^{-1}	1100–1500
BOD_5	mg L^{-1}	1120–2300
COD	mg L^{-1}	6000–9000
PO_4–P	mg L^{-1}	13–17
Oil and grease	mg L^{-1}	130–350
Total nitrogen	mg L^{-1}	132–180
Total chromium	mg L^{-1}	2000–2300
Sulfate	mg L^{-1}	320–390
Chloride	mg L^{-1}	450–485

RESULTS AND DISCUSSION

The Effect of PH on Oil and Grease Removal in The Treatment of Leather Industry Wastewater

While investigating oil and grease removal from leather industry wastewater by electrocoagulation method, the pH was changed between 2 and 8. The experiments are carried out using both aluminum and iron electrode. In the studies, the pH of the wastewater was adjusted and it was monitored later but was not intervened. Experiments were carried out at 1.2 mA cm^{-2} current density (CD). The mixing speed (n) was kept constant at 150 rpm. The results obtained for the aluminum electrode are given in Figure 2.

When Figure 2 is examined, the highest removal efficiencies were obtained when 2, 3, and 8 of the initial pHs of the wastewater. The initial pH of the wastewater affects the oil and grease removal mechanism [18]. Since oil and grease compounds are dissolved in the water below pH 3, removal occurs as a result of the breakdown of oil and grease due to the electrooxidation mechanism. At pH 3, 4, and 5, since the pH of the wastewater is balanced between 5 and 7, at these points where the solubility of $Al(OH)_3$ compound is the lowest, oil and grease are adhered to $Al(OH)_3$ flocks and removed from the environment by sedimentation [19]. In

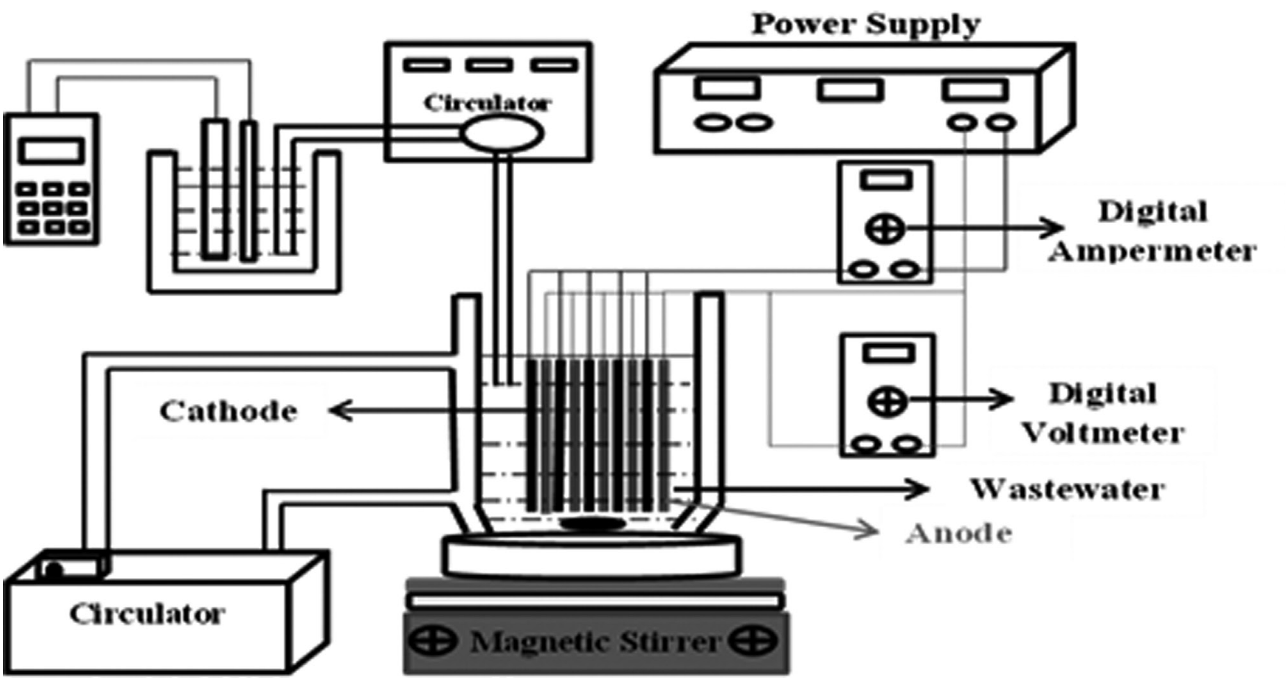

Figure 1. Electrocoagulation system used in experiments.

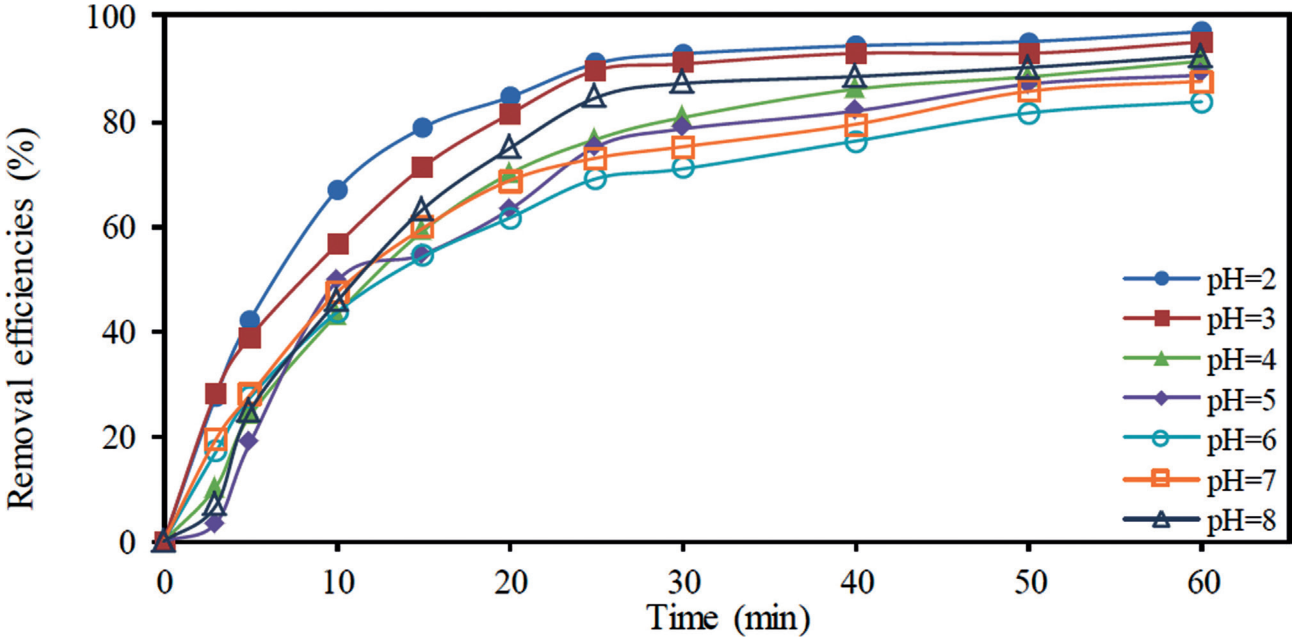

Figure 2. The change of oil grease removal efficiency versus time as a function of pH in the treatment of leather industry wastewater using aluminum electrodes.

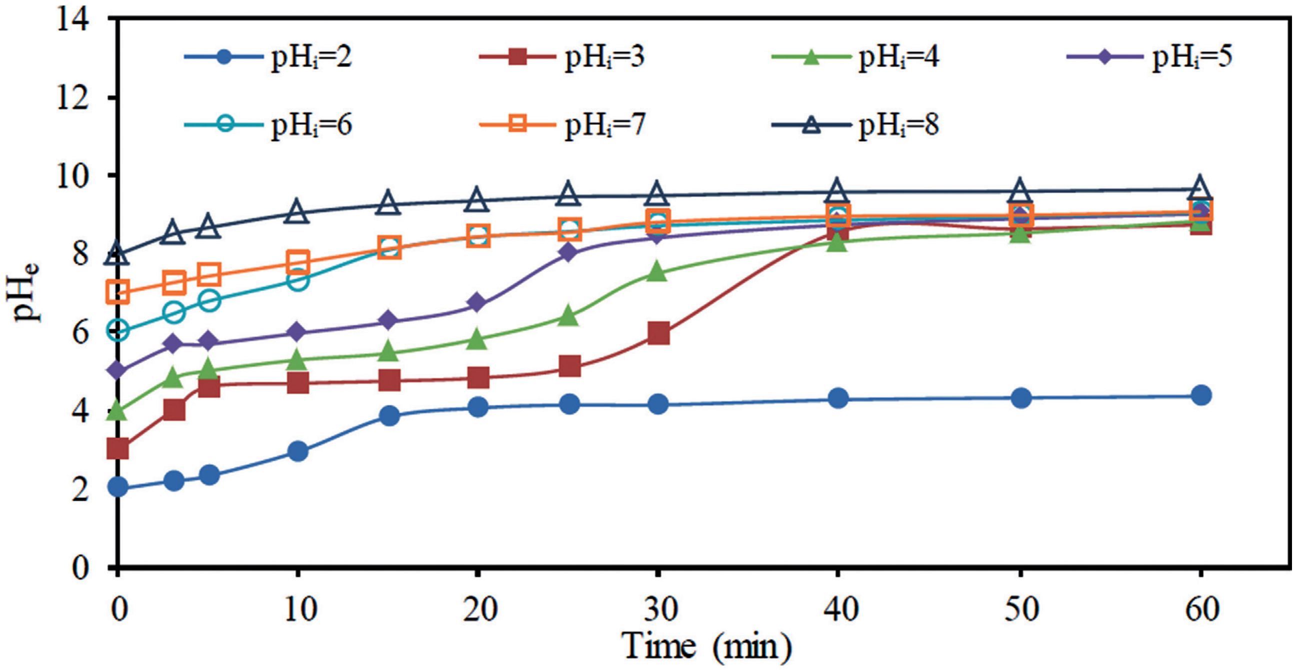

Figure 3. Change of pH of the wastewater in the treatment of leather industry wastewater using aluminum electrodes vs time.

addition, oil-grease removal is also carried out by the flotation effect caused by the hydrogen gas exiting the cathode at the all initial pH of the wastewater. If the pH is above 8, because soluble forms of aluminum are formed, flotation is the primary mechanism in oil and grease removal. The dimensions of the gas bubbles, which have a very important function in oil-grease removal, are also affected by the pH of the environment. Typical bubble sizes in electrocoagulation range from 20-70 µm, the smaller and more loaded bubbles, the better they function. These bubbles both

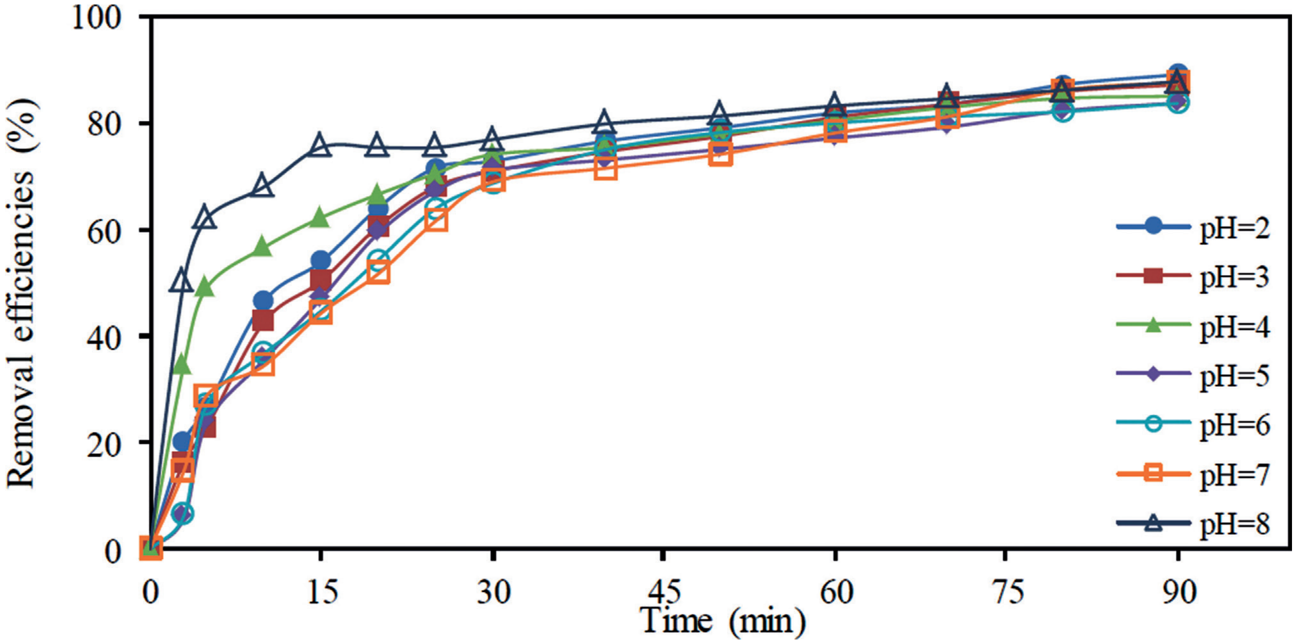

Figure 4. The change of oil and grease removal efficiency versus time as a function of pH in the treatment of leather industry wastewater using iron electrodes.

provide effective surface area for gas-liquid-solid interfaces and help to collect small unstable particles. Hydrogen bubbles that fit the normal size distribution are the smallest at approximately neutral pH. The change of pH of wastewater vs time is given in Figure 3.

When Figure 3 is examined, it is seen that leather industry wastewater has a buffering effect on pH. While the pH of the wastewater rises slowly up to 5.5, it suddenly rises when it exceeds the pH 5.5 threshold value. This makes the pH one of the most important parameters in the treatment of wastewater by the electrocoagulation method. The increase in pH if the initial pH is acidic and the decrease in pH, if it is alkaline, indicates that the electrocoagulation process neutralizes the pH of the wastewater. This makes it important to treat wastewater by electrocoagulation, as it eliminates the process of adjusting the pH of wastewater and enables it to operate effectively over a wider pH range as in this study [20, 21].

Experiments were made also using iron electrodes as electrode material under the same conditions as aluminum electrodes for leather industry wastewater and the data shown in Figure 4 were obtained for different initial pHs of wastewater.

When Figure 4 is examined, it is seen that the oil-grease removal in the treatment of leather industry wastewater by using iron electrodes has high efficiency at all pH values, that is, it is not affected much by pH. Because of the predominance of Fe2+ at the low and neutral pHs in the wastewater treated by electrocoagulation with iron electrodes, its color appears greenish, then the color turns yellow due to the predominance of Fe^{3+} at high pH [17]. Due to this reason, oil and grease in the wastewater have been removed with high efficiency at all initial pH values by adsorption, precipitation, and flotation effect. Similar results were obtained in the study conducted by Kongjao et al. [22]. The change in pH of the wastewater in the treatment of leather industry wastewater using iron electrodes is given in Figure 5.

Chen et al. (2000) conducted a study using iron and aluminum electrodes to remove oil and grease at pH 6.8 and 3-8 mA cm^{-2} CD in restaurant wastewater [23]. They said that both electrodes work at the same efficiency. Also, Chen et al. stated that the same efficiency is obtained at all pHs where pH is not effective in restaurant wastewater treatment. Kobya et al. (2006), in the study conducted on poultry slaughterhouse wastewater, found that if iron electrodes were used in oil and grease removal, the pH of the wastewater was not effective, but the Al electrodes were more affected from the pH of the wastewater. In addition, they found that although Al electrodes are more effective in COD removal, Fe electrodes are more efficient in oil and grease removal [24].

When compared with aluminum electrodes, it is seen that the pH of the wastewater increases faster at all initial pHs in iron electrodes. While pH increased up to pH 4.33 in the wastewater with an initial pH of 2 with aluminum

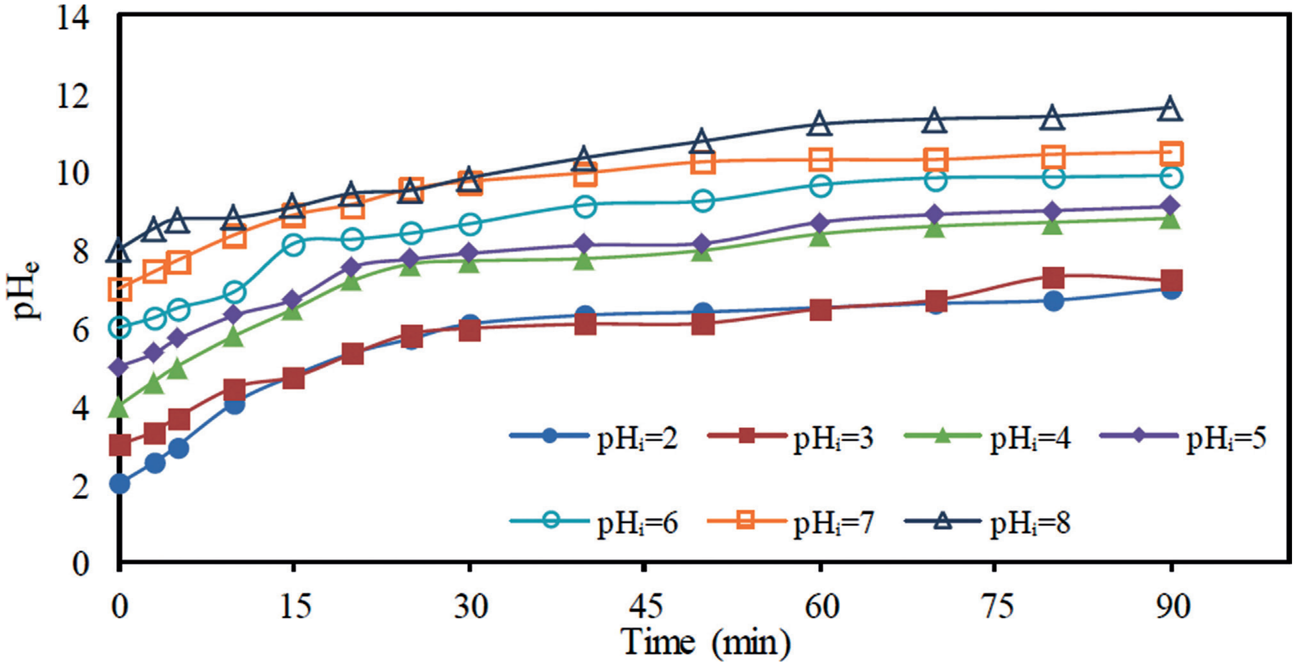

Figure 5. Change of pH of the wastewater in the treatment of leather industry wastewater using iron electrodes vs time.

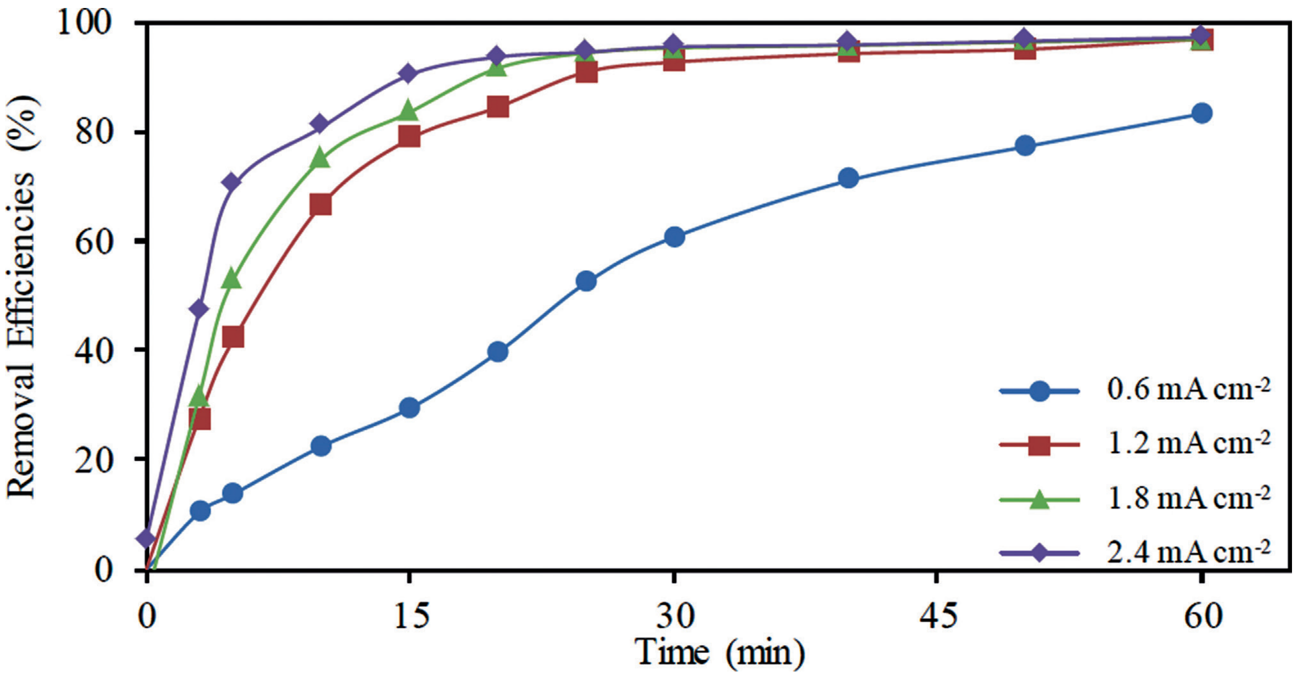

Figure 6. Change of oil and grease removal efficiency vs time as a function of CD in leather industry wastewater treatment using aluminum electrodes.

electrodes, this value was 6.48 for the same period with iron electrodes. Again, while pH increases to around 9 with Al electrodes, pH increases up to 12 with iron electrodes. In this situation, iron is thought to be caused by the bivalent and its more dissolution at high pH.

The Effect of CD on oil and Grease Removal in Leather Industry Wastewater Treatment

Experiments were carried out to determine how the CD applied to the system will affect the oil and grease removal while treating the leather industry wastewater at pH$_i$ 2 and

150 rpm stirring speed. The results obtained in the studies are shown schematically in Figure 6.

When Figure 6 is examined, it is seen that the oil-grease removal efficiency is almost the same for aluminum electrodes at other current densities except for 0.6 mA cm^{-2} and the steady state time decreases as the CD increases. Oil-grease particles are either adhered to the bubbles of hydrogen gas exiting the cathode, forming oil-grease and

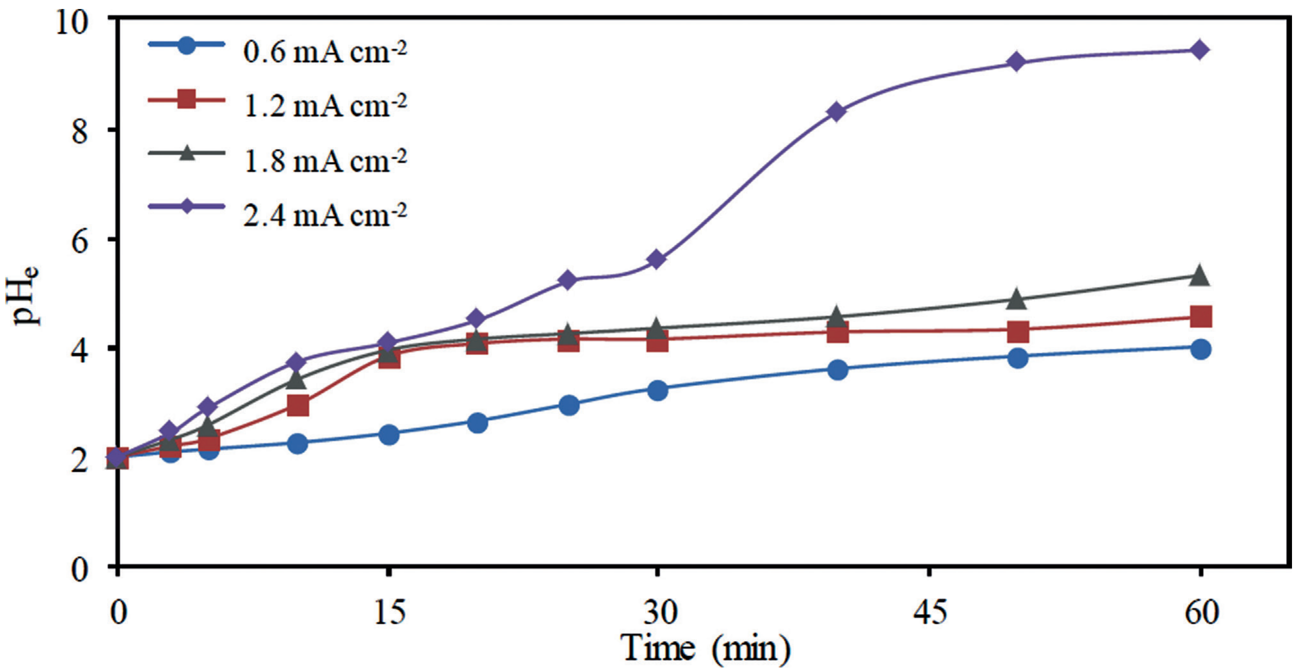

Figure 7. The change of wastewater pH vs time for different current densities in the treatment of leather industry wastewater using aluminum electrodes.

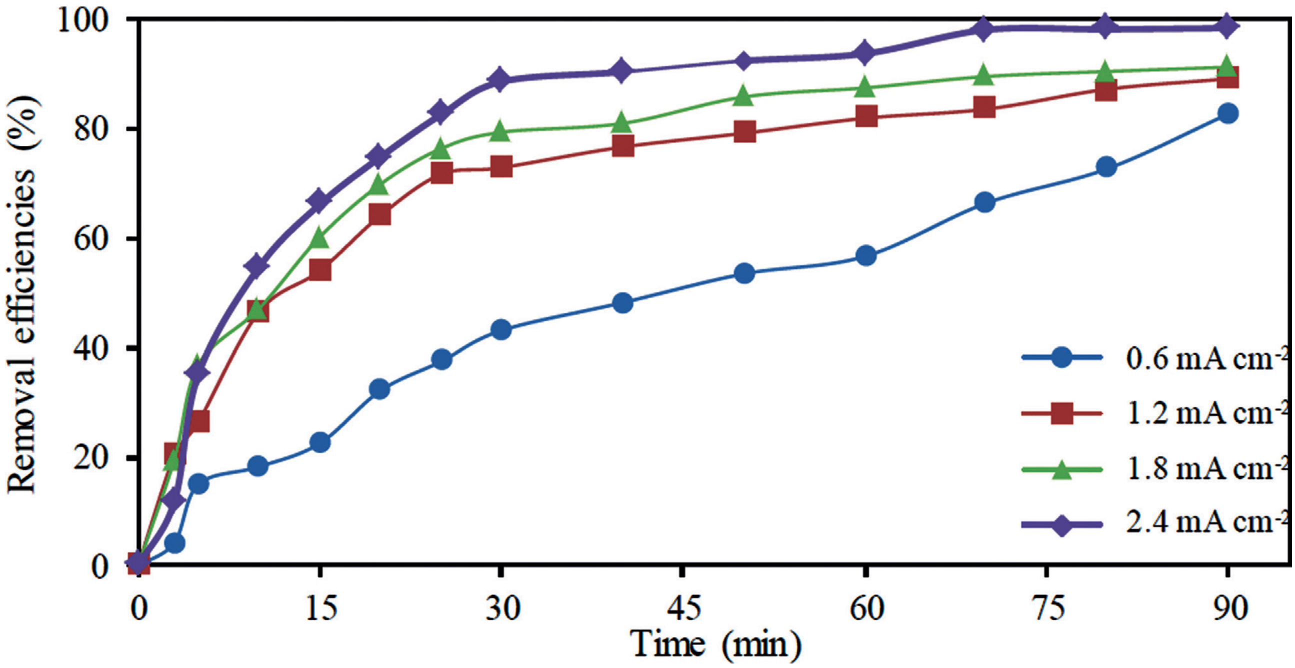

Figure 8. Change of oil and grease removal efficiency vs time as a function of CD in leather industry wastewater treatment using iron electrodes.

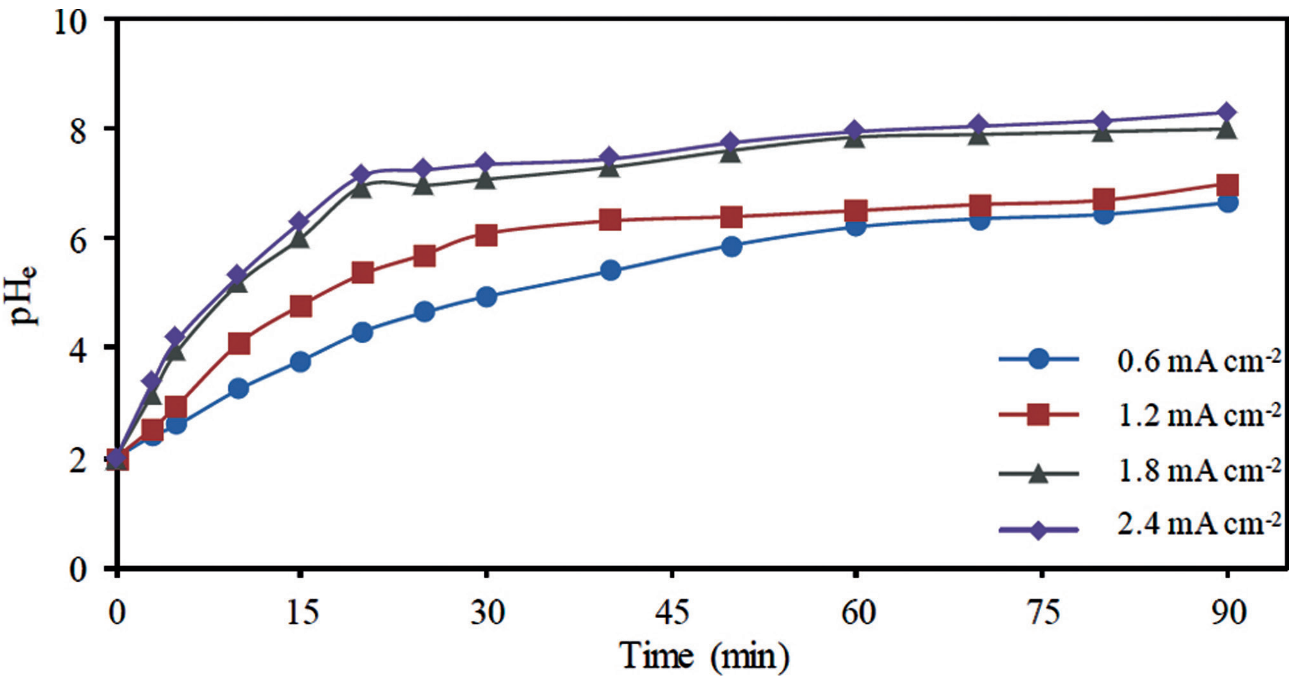

Figure 9. The change of wastewater pH vs time for different current densities in the treatment of leather industry wastewater using iron electrodes.

H_2 complexes and lifted to the top of the reactor or removed by adsorbed to metal hydroxides [25].

While finding the optimum parameters of the system in the electrocoagulation process, not only high efficiency but also energy consumption should be taken into account. The increase in the CD means the increase in the potential difference applied to the system. This situation increases energy consumption. However, since the potential difference applied to the system in the treatment of water with high conductivity, such as in the leather industry, will be low, the energy consumption is also reduced.

Priya and Jeyanthi (2019) carried out studies on oil grease removal by using Cu and Al electrode pairs in the automobile wash water and achieved approximately 80% efficiency at 2.5 mA cm^{-2} CD and pH 6.5 [18]. However, due to the insufficient conductivity of the wastewater, they had to reach a high potential difference.

When aluminum electrodes are used in the experiments, the change of the pH of the wastewater vs time for different current densities is given in Figure 7.

When Figure 7 is examined, it is seen that as the CD through the system increases, the pH of the wastewater increases faster. Especially when the CD is 2.4 mA cm^{-2}, it increases suddenly because it exceeds the pH 5.5 threshold. Since the increase in CD increases the amount of Al^{3+} dissolved in the anode and the electrolysis of the water, more hydroxyl compounds are formed and the pH rises faster.

Using iron electrodes, oil and grease removal at different current densities in the treatment of leather industry wastewater has been examined and the results obtained are given in Figure 8.

When Figure 8 is examined, it is seen that when iron electrodes are used, the oil and grease removal increases relatively as the CD increases. It can be said that the reason for this is that as the CD increases, both Fe^{2+} and Fe^{3+} ions dissolved in the anode create more flocs and also the number of H_2 gas bubbles formed on the cathode increases. The change of wastewater pH according to time for different current densities is given in Figure 9.

When Figure 9 is examined, it is seen that as the CD increases, the pH of the wastewater increases faster. In this case, as the Fe(OH)n compounds formed in the reactor dissolve better at high pH, the flotation rate of the H_2 gas bubbles formed at the cathode increases more.

CONCLUSIONS

Leather factory wastewater contains high amounts of COD as well as high amounts of oil and grease. For this reason, it is difficult to treat these wastewaters with conventional treatment methods. In this study, it was determined that the electrocoagulation process in the treatment of leather industry wastewater provides removal of a high amount of oil and grease. This will reduce the organic load of a biological treatment system that will follow electrocoagulation and will ensure high efficiency.

When both aluminum and iron electrodes are used in the electrocoagulation process, the oil and grease removal efficiency is maximum when the pH of the wastewater is below 4. While the oil and grease removal efficiency with Al electrodes is 95% at pH 2, this value decreases to 83% at pH 6. In Fe electrodes, while the removal efficiency is 87.83% at pH 2, it decreases to 83% at pH 6. When aluminum electrodes are used, the oil and grease removal efficiency is relatively higher. However, when aluminum electrodes are used, oil and grease removal efficiencies are more affected by pH.

It has been determined that as the CD increases in the electrocoagulation system, the oil and grease removal efficiency increases. As the CD increases, both the amount of Al and Fe dissolved in the anode and the H_2 gas formed at the cathode increase. As a result, the oil and grease removal efficiency is also increased. While the efficiency of Al electrodes is 83.31% at 0.6 mA cm^{-2} CD, this value has increased to about 98% at 1.2,1.8 and 2.4 mA cm^{-2} current densities. In Fe electrodes, the oil and grease removal efficiency is 82.66% at 0.6 mA cm^{-2} CD, while it is 90% at 1.2 and 1.8 mA cm^{-2} current densities and 98% at 2.4 mA cm^{-2}. Since leather factory wastewater contains high amounts of dissolved material, its conductivity is high. In this case, high current densities can be achieved under low potential differences. This increases the cost and applicability of the process. In this system, oil and grease are removed by four different mechanisms. These are electrooxidation, precipitation, adsorption, and flotation mechanisms. In this study, the effective mechanism is searched depending on the pH of the wastewater and the applied intensity of the device. As a result, when the electrocoagulation process is used to remove oil and grease from leather factory wastewater, high yields are obtained at every pH and every CD. This situation provides a flexible working opportunity in the operation of the system. Wastewater with high oil and grease concentration, which causes problems in conventional treatment systems, can be easily treated with the electrocoagulation system.

As a result of this research, the COD amount of treated water decreases to 900-1000 mg L^{-1} and the amount of oil and grease to 0-20 mg L^{-1}. As a result of electrocoagulation, discharge standards are met in terms of oil and grease amount. Even if they do not meet the discharge standards in terms of COD, electrocoagulation as a pre-treatment can provide a high-efficiency biological treatment.

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AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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