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Abatement of pollutant load of pharmaceutical wastewater by electrochemically coagulated using aluminum electrodes

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Abstract. Pharmaceutical industry wastewater is characterized by high biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), turbidity and other pollution load. The purpose of this study was to investigate the effects of the operating parameters such as applied voltage, electrolysis time, pH, inter electrode distance and active electrode surface on ther removal of organic pollutants from the studied effluent by electrocoagulation process, using aluminum electrodes material. It has been shown that the treatment has allowed to reach a maximum reduction of 92 % for the COD, 87.5 % for BOD and 97 % for turbidity under the following conditions: a potential difference: 5V, electrodes surface: 140 cm^2, inter-electrode distance: 0.5cm, pH medium equal to 6 and a contact time of 60 min.

Keywords: *Pharmaceutical wastewater, treatment, pollution, electrocoagulation, aluminum electrodes*

1. Introduction

Pharmaceutical compounds are being used for several beneficial purposes in modern society but simultaneously pharma industries are releasing very toxic contaminants in the environment directly or after chemical modifications [1].

Many pharmaceutical industries are responsible to generate toxic effluent as a consequence of their operation. The waste water generated from these industries possess solids, biodegradable and non degradable organic compounds [2].

The main pollutant releasing from the industrial waste water is oxygen function is measured in chemical oxygen demand (COD) and biological oxygen demand (BOD). Lang and Enick estimated that approximately half of the pharmaceutical wastewaters produced worldwide are discarded without specific treatment [3,4].

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The effect of pharmaceutical wastes in the environment is a raising concern about the potential environmental consequences and it has an impact on the surface water, aquatic species, human health and surrounding lands, which may cause a serious problem to drinking water directly or indirectly. The effect of these contaminants depends upon the processing technology, nature of chemicals used, size, the complexity and characteristics of wastewater discharged [5].

Treatment of these wastes is therefore of paramount important. Various techniques employed in the treatment of pharmaceutical wastewater are essentially biological and physiochemical processes such as aerobic and anaerobic biological process, coagulation, flocculation, sedimentation, and activated carbon adsorption. These techniques have shown limited success for the treatment of pharmaceutical wastewater due to the nature and composition of pharmaceutical effluents. Therefore, other technologies have been explored with the aim to further reduce the concentration of pharmaceutical contaminants. These technologies include membrane separation [6], advanced oxidation technology [7], and electrochemical techniques [5,8]. These later have been investigated in environmental applications, especially for treating water and wastewater. One of these processes is electrocoagulation which has achieved much attention due to its attractive advantages: as simple, reliable and cost-effective operation for the treatment of wastewater [9]. The electrocoagulation process has been widely and successfully employed for the treatment of various kinds of water and industrial effluents such as olive mill wastewater [10,11], restaurant wastewater [12], domectic wastewater [13], tannery wastewater [14,15] textile industry wastewater [16], etc.

The electrocoagulation technique is based on the generation of the flocculating agent by electro-oxydation of a soluble anode, generally made of iron or aluminum, without the addition of any chemical coagulant or floculant. Thus, reducing the amount of sludge which must be disposed [17]. On the other hand, electrocoagulation is based on the insitu formation of the coagulant as the sacrificial anode corrodes due to an applied current and hydrogen gas is released from the cathode. The hydrogen gas would also help to float the flocculated particles out of the water.

Hence, in the present study an attempt was made on the evaluation of the efficiency of the EC process on reducing of pollution load of pharmaceutical wastewater, by removal of chemical oxygen demand, biological oxygen demand and turbidity, and using aluminum electrode. The experiments were conducted to examine the effect of the operating parameters such as applied potential difference value, electrolysis time, active electrode surface, inter-electrode distance and pH of the medium on pollutants removal.

2. Methodology

2.1. Material

The pharmaceutical wastewater studied was kept at 4°C before its use to avoid changes in the physico-chemical properties.

All parameters, may be altered, were analyzed in the laboratory in the shortest possible time in accordance with conservation rules and standardized analysis methods [18].

The pH, the turbidity and the conductivity of the solution were measured by using, respectively, a pH meter (HANNA HI 9812-5), a turbidimeter (HANNA HI 88713-ISO) and a conductivity-meter (ISO Method HACH 7027). The analysis of chlorides was carried out by Mohr method [18], the chemical oxygen demand (COD) and biological oxygen demand (BOD) was achieved via the experimental protocols of ISO 6060-1989 standard for the first parameter and the French standard (AFNOR T90: 103, 1994) for the second.

2.2. Method

The schematic representation of the experimental setup is shown in Figure 1. Experiments were performed in a batch reactor, of 1L of capacity, with two aluminum electrodes connected in parallel between which the effluent to be treated circulates. By the using a potentiostat (Constanter / Netzgerat Universel PHYWE), the electrodes are subjected to a constant voltage or a constant current density which leads to a uniform dissolution of the metal at the anode and hydrogen evolution at the cathode. The current control was done in the same time using this potentiostat and an ammeter (PHYWE) connected in series. To maintain homogenous mixing of reactor content, magnetic stirring unit was used. The samples were periodically taken from the reactor, filtered, to eliminate the flocs formed during the electrolysis, before being analysed to determine the residual concentration of the pollutant. At the end of each manipulation, the aluminum electrodes were cleaned with dilute hydrochloric acid (0.1 M), then rinsed with distilled water and dried at 105°C for about two hours. The reduction rate of the parameters pollution (turbidity, chemical oxygen demand and biological oxygen demand) expressed in percentage TX (%) was calculated using the Eq. 1:

$$\mathbf{T(X)} = \frac{(CiX - CfX)}{CiX}\mathbf{100} \qquad (1)$$

Where: Cix and Cfx are the concentrations of the element (X) before and after electrocoagulation treatment.

In order to optimize and understand well the treatment process, the different operating parameters such as the applied potential difference value (2, 5 and 10 V), electrolysis time (30, 60, 75, 90 and 120 min), active electrode surface (88 and 140cm^2), inter electrode distance (1 and 0.5cm) and pH of the solution (2, 4, 6, 9.2 and 12) have been investigate.

The choice of these values is based on studies already carried out but for other industrial effluent. The analysis of the parameters COD, BOD_5 and turbidity of the treated water were repeated two or three times depending on the difference between the analysis results.

Fig. 1: Schematic experimental set

3. Results and Discussion

3.1. Characterization of the pharmaceutical wastewater

The physicochemical analysis of the pharmaceutical wastewater studied is listed in Table 1. It shows a comparison between the obtained values of this wastewater with the Algerian national standards defining the values limits of industrial effluent liquids discharges [19]. From this table, it is clear that some values such as turbidity (5000 NTU), BOD (4100 mg/L) and COD (1380 mg/L) are exceeded the Algerian standards.

In view of these results, it is worth to note that this pharmaceutical wastewater has an organic nature. Thereby, the biodegradation of these matters causes oxygen consumption where a possible eutrophication of the receiving environment with a deterioration of the fauna and flora and the creation of harmful resistant species can take place. All these observations make sure that this wastewater has to be treated.

Table 1: Physicochemical characterization of the pharmaceutical wastewater

Analyses parameters	Values	Algerian standard discharge of industrial liquid effluent [19]
Temperature (°C)	80	30
pH	9.2	6.5-8.5
Conductivity (ms/cm)	3.43	/
Turbidity (NTU)	5000	/

COD (mg/L)	1380	130
BOD (mg/L)	4100	40
MES (mg/L)	7450	40
Chlorides (mg/L)	280.82	/

3.2.Results of treatment of pharmaceutical wastewater by electrocoagulation process

3.2.1. Effect of the applied potential difference

Applied potential is one of the important parameters that should be considered while determining the COD, BOD and turbidity removal efficiency [20]. Figure 2 presents the results of the reduction of COD, BOD and turbidity contents as a function of the applied potential difference for three values (2, 5 and 10V). At 2 V, the reduction rate of COD, BOD and turbidity is 45.8, 56.4 and 64.5% respectively, for a period of 30 min. The rise of the applied potential difference from 2 to 5V increases the reduction efficiency until 78.6, 69.4 and 89.2% of COD, BOD and turbidity respectively. After this difference potential value, a very slightly increase of the removal efficiency is observed.
By electrical potential increase, the amount of oxidized aluminum increases and consequently hydroxide flocs with high adsorption rate increase and this leads to an increase in the efficiency of hardness removal [21,22].

In addition, it was demonstrated that bubbles density increases and their size decreases with increasing current density [22,23,24] resulting in a greater upwards flux and a faster removal of pollutants and sludge flotation.

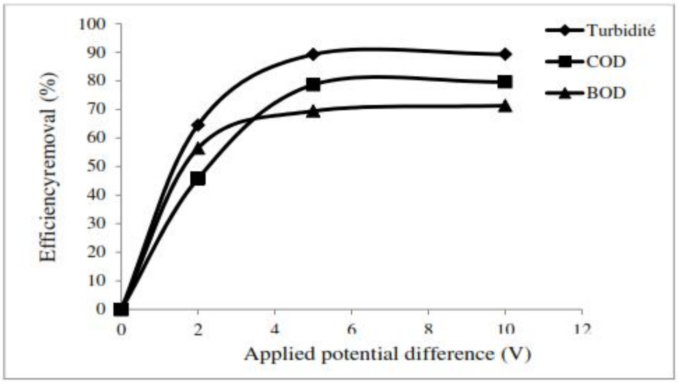

Fig. 2 : Effect of the applied potential difference on the removal of the turbidity, COD and BOD
Operating conditions: contact time: 30 min, temperature 20°C, initial pH of the wastewater: 9.2, conductivity: 3.4ms/cm, reactor volume: 1 liter, inter electrode distance: 0.5cm and electrode surfaces: 88cm^2.

3.2.2. Effect of the electrolysis time

Electrolysis time has influence on electrocoagulation process efficiency. This influence is translated by the production of ions concentration of the electrodes. The effect of electrolysis time was studied at constant voltage (5V), the result is shown in Figure 3. Increasing electrolysis time from 30 minutes to 60 minutes has increased removal efficiency from 89.2, 78.6 and 69.4 % to 94.2, 83.4 and 76.4 % for turbidity, COD and BOD respectively. This is because of the increase of consumption of coagulant over time that could lead to the increase of removal percentage and the decrease of the amount of pollutant [25]. After 60 minutes, the efficiency removal is almost stable.

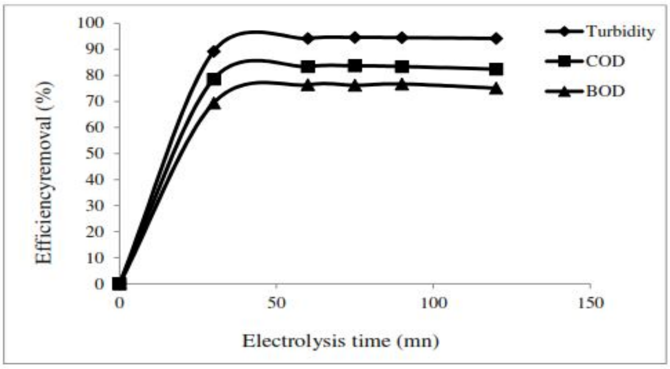

Fig. 3: Influence of electrolysis time on the removal of the turbidity, COD and BOD
Operating conditions: applied potential difference: 5V, temperature 20°C, initial pH of the wastewater: 9.2, conductivity: 3.4ms/cm, reactor volume: 1 liter, inter electrode distance: 0.5cm and electrode surfaces: 88cm².

3.2.3. Effect of pH

The pH is an important operating factor influencing the performance of electrocoaulation process [26,27,28].

To evaluate the pH effect on electrocoagulation process, a series of experiments were performed, using pharmaceutical wastewater with initial pH varying in the range (2-12).The solutions of these metals wereadjusted to the desired pH for each experiment using sodium hydroxide or hydrochloric acid.

Removal efficiency of COD, BOD and turbidity as a function initial pH are presented in Figure 4. The obtained results show that the pH has a noticeable effect on efficiency of COD, BOD and turbidity removal, the best removal results is obtained at pH of 6 and it equal 89.2 % for COD, 83.4 % for BOD and 96.7 % for turbidity. When the initial pH is increased above 6, a decrease of the removal efficiency is observed.

According to Bani Salameh [29], the aluminum can form different species depending on the pH of the solution. Al^{+3} ions hydrolysis may generate the aqueous complex $Al(H_2O)_6^{+3}$, which is prevalent at pH > 4.5. Between 5.5 and 6.5 the prevalent hydrolysis products is $Al(OH)_3$ also found that solubility of $Al(OH)_3$ was minimum at pH around 6, insoluble form $Al(OH)_3$ predominate at the pH near 6.5.This species has an amphoteric character and its solubility increases as the solution becomes more acidic or basic.

On the other hand, for pH < 6, the protons in the solution get reduced to H_2 and thus, the proportion of hydroxide ion produced is less and consequently there is less removal efficiency [30].

These findings are in the line with the results of Bani Salameh [29] and Yazdanbakhsh [31] about the electrocoagulation process for pollutants removal from olive mill wastewater.

As observed by others investigator, the pH of the medium changed during the electrocoagulation process depending on the type of electrode material and initial pH [32].

If the initial pH of the solution is low, an increase will occur in the final pH of the solution (Figure 5).

Vik et al. [33] ascribed this increase to hydrogen evolution at cathodes. In the alkaline medium, the final pH is not changed.

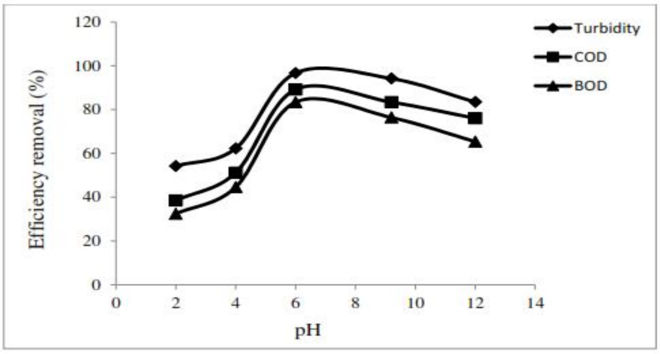

Fig. 4: Effect of pH on the removal rate of the turbidity, COD and BOD

Operating conditions: applied potential difference: 5V, temperature 20°C, contact time: 60 min, conductivity: 3.4ms/cm, reactor volume: 1 liter, inter-electrode distance: 0.5cm and electrode surfaces: 88cm^2.

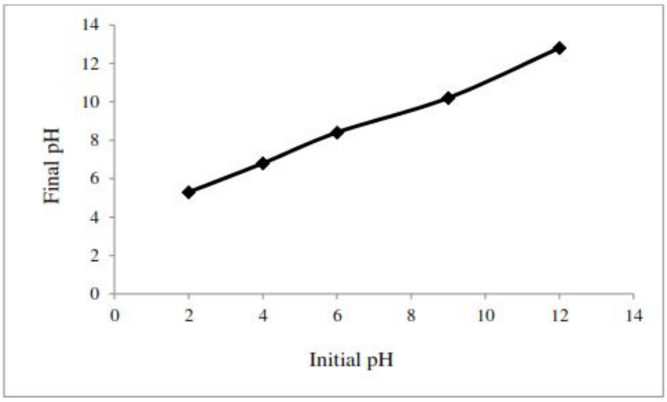

Fig. 5: pH variation after electrocoagulation

3.2.4. Influence of electrodes surface

Figure 6 shows the experimental results of the reduction of turbidity, COD and BOD as a function of the surface of the aluminum electrodes used. It can be seen that the increase of surface of electrodes from 88 to 140 cm^2 leads to increase the treatment efficiency. The removal rate of COD, turbidity and BOD increases from 89.2, 96.7 and 83.4 % to 92.2, 97.6 and 87.5 % respectively.

According to Battula [34], this can be attributed to a greater electrode area that produced larger amounts of anions and cations from the anode and cathode.

The greater the electrode is increased the rate of flock's formation, which in turn influenced the removal efficiency [35,36].

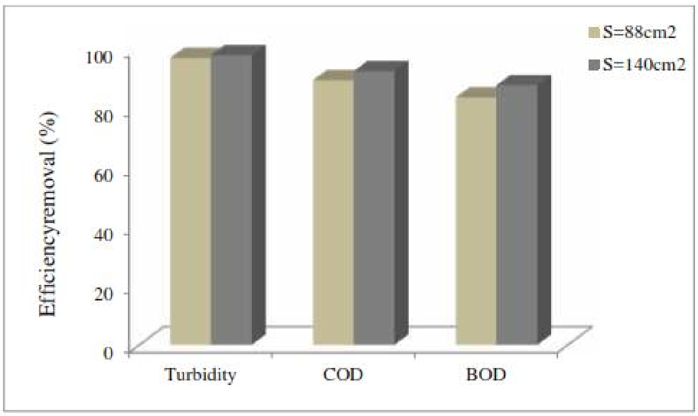

Fig. 6: Influence of electrodes surface on the removal of the turbidity, COD and BOD

Operating conditions: applied potential difference: 5V, temperature 20°C, contact time: 60min, conductivity: 3.4ms/cm^{-1}, reactor volume: 1 liter, inter-electrode distance: 0.5cm.

3.2.5. Effect of inter electrode distance

Inter electrode spacing is a vital parameter in electrocoagulation process for the removal of pollutant from effluent [37]. Two distances inter electrode are tested: 0.5cm et 1cm. the electrical potential and electrolysis time were kept respectively 5V and 60minutes. The COD, BOD and turbidity removal as a function of inter electrode distance are presented in Figure 7. At 0.5cm of distance between electrodes, the removal efficiency of COD, BOD and turbidity is 89.2, 83.4 and 96.7%; when the distance increases up to 1cm, the removal efficiency will decrease to 74.8% for COD, 71.6 % for BOD and 83.1 % for turbidity. Similar results are found by researchers [38,39]. According to Mansour and Hasieb [40], with increasing electrodes distance, the internal resistance (or the ohmic) between electrodes increases and that leads to the decrease of aluminum ions production and therefore a decrease in removal efficiency. The decreased inter electrodes distance reduces resistance, electricity consumption and the cost of the wastewater treatment [41].

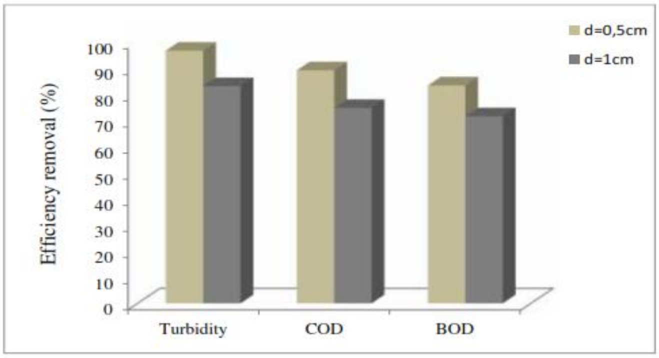

Fig. 7: Influence of the inter electrode distance on the removal of turbidity, COD and BOD

Operating conditions: applied potential difference: 5V, temperature 20°C, contact time: 60min, conductivity: 3.4 ms/cm, reactor volume: 1 liter, electrode surfaces: 88cm^2.

4. Conclusion

This work was focused on the clarification of the pharmaceutical wastewater by electrocoagulation in a static system. The content analysis of this wastewater showed a high pollutants load expressed especially by COD, turbidity and BOD. The results of the treatment showed the high efficiency of the electrocoagulation process for reducing COD, turbidity and BOD contents. The study of the effects of different operating parameters has shown that under the following conditions : applied potential difference: 5V, room temperature 20°C, contact time: 60 min, pH of medium equal to 6, electrode surfaces: 140 cm^2 and inter electrode distance: 0.5cm, the removal rate is 92% for the COD, 87.5 % for BOD and 97 % for turbidity. Finally, the results demonstrated the technical feasibility of electrocoagulation as a reliable technique for removal of COD, BOD_5 and turbidity using aluminum electrodes to effectively treat pharmaceutical wastewater, by means of additional tests on new reactors, to predict the fate of the sludges formed which limit, in time, the performance of the reactor.

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