

Iron Electrocoagulation of Soybean Oil Refinery Wastewater with SuperPro Designer[®] Simulation

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Abstract—Iron electrocoagulation (Fe-EC) is a wastewater remediation method that eliminates various contaminants through the application of an electric current across iron electrodes. In this study, the efficacy of two Fe-EC procedures for eliminating chemical oxygen demand from soybean oil refinery wastewater was assessed. The first procedure entailed treating the wastewater by directly exposing it to the electrodes, while the second procedure comprised treating it with electrogenerated Fe coagulant flocs. The effects of operating factors: initial pH (4, 7 and 9) and applied current (0.1, 0.2 and 0.4 A) were examined. The study also sought to simulate the EC adsorption mechanism using software. The results showed that COD removal efficiency ranged from approximately 84% to 95% for both approaches with maximum removals of 93.61% and 94.56% by direct Fe-EC and preformed flocs, respectively. The most successful simulation of the adsorption process produced COD reductions ranging from 87.31 to 91.31%, using a two-stage reaction involving one iron (III) hydroxide molecule and five soybean oil molecules. **Keywords**— COD removal, iron electrocoagulation, process modelling, soybean oil

I. INTRODUCTION

Iron electrocoagulation (Fe-EC) is a form of electrocoagulation (EC) that employs an iron electrode as the sacrificial anode. Iron (Fe), like aluminum (Al), is frequently utilized as electrodes because of its abundant supply, cost-effectiveness, and proven efficiency in pollution removal. The principles behind the removal of pollutants from water or wastewater by EC are the same as for chemical coagulation. Both approaches work by destabilizing the repulsive forces that keep particles suspended in water [1, 2]. When the repulsive forces are neutralized, flocculation of the suspended particles causes them to form larger particles of reduced solubility that can settle out for easier separation from water [2]. The major difference between the two methods is the chemical dosing of the water/wastewater. In chemical coagulation, coagulating metallic salts such as ferric sulphate, ferric chloride and aluminum sulphate are added to the water or wastewater, whereas in EC, the coagulants are produced in-situ. Additional benefits of EC over traditional chemical coagulation include simple equipment design, user-friendly operation, little or no use of chemicals, and reduced sludge production [3, 4].

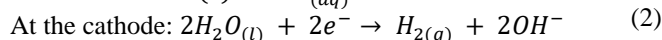
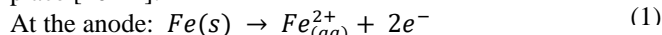
Electrocoagulation has been utilized in several wastewater treatment applications, primarily in experimental settings, and has demonstrated efficacy in decreasing a diverse array of contaminants. One potentially problematic characteristic of wastewater is its concentration of chemical oxygen demand

(COD). COD represents the oxygen-depletion ability of wastewater and its possible negative impact on the environment upon release. Fe-EC has been applied to various high COD wastewaters, yielding significant COD reduction results [5-9]. In this work, Fe-EC was used to treat high COD oily wastewater (OW) from a soybean oil refinery.

The study aimed to determine the effectiveness of Fe-EC in removing COD from OW and to use SuperPro Designer[®] for modeling the reaction(s) between Fe coagulant and oil molecules responsible for COD. The objectives of the study were to determine the maximum COD removal from OW by Fe-EC, to compare the COD removal efficiency from OW by direct Fe-EC and EC generated Fe coagulant flocs, and to use SuperPro Designer[®] to model Fe-EC based on data obtained from the Fe-EC experiments. The novelty of the study is the use of SuperPro Designer[®] to model Fe-EC and compare the results with experimental data to shed light on the possible adsorption mechanism for COD removal.

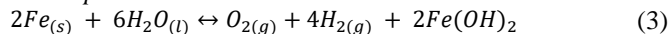
A. Theory

When a direct current (DC) passes through the electrolytical cell, the anode dissolves to release positively charged Fe ions. The following reactions are purported to take place [10-12]:

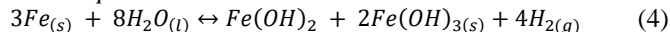


In the presence of dissolved oxygen, ferrous iron (Fe^{2+}) is oxidized to ferric iron (Fe^{3+}). Refs. [13] and [14] demonstrated through experiments that the amount of Fe^{3+} rises when the pH level increases from acidic to alkaline pH. Depending on the pH of the solution, different reactions are promoted. The following reactions have been proposed by [15] and [16]:

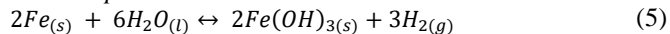
Acidic pH:



Neutral pH:



Alkaline pH:



Other Fe species such as oxides and oxyhydroxides are possible. The insoluble Fe species produced act as coagulants that destabilize pollutant molecules and aid their removal from the solution. Pollutants can be removed by surface complexation or electrostatic attraction [17]. The mass of the coagulant is related to the mass of Fe dissolved from the anode, m, given by Faraday's law:

$$m = \frac{ItMw}{nF} \quad (6)$$

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where I is the applied current (A), t is the electrolysis time (s), M_w is the molecular weight of the anode material, n is the number of electrons involved in the exchange, which is assumed to be 2 for ferrous ion, and F is Faraday's constant (96,485 C/mol). The coagulated particles are removed from the wastewater through sedimentation or electroflotation.

B. SuperPro Designer®

SuperPro Designer® Version 12 is a proprietary software developed by Intelligen, Incorporated (New Jersey, U.S.A). The software is used to model, evaluate, and optimize batch and continuous processes across many manufacturing industries. One of its features is the modelling of environmental operations such as air pollution control and wastewater treatment. This study focuses on the wastewater treatment capabilities, specifically the parameters to be reduced. The software database includes parameters like COD, TOC, BOD5, nitrates, and phosphate, which are associated with either a pure substance or a mixture as inherent characteristics. Although Fe-EC, and EC in general, is not at the stage to be modelled by proprietary simulation software due to lack of comprehensive knowledge on the intricacies of EC, SuperPro Designer® offers most of the procedures involved in a laboratory-based EC experiment. These are batch and/or continuous operation of a stirred tank reactor and filtration. The software has been used to model and evaluate several processes, including biodiesel production [18], polyhydroxyalkanoates production via bacterial fermentation of soybean oil [19], and fuel ethanol production from corn [20].

There are other modeling software options available, such as BioWIN and WEST, that are specifically designed for simulating municipal wastewater treatment plants. SuperPro Designer® provides the option to incorporate a pollution (air and water) treatment stage into a modelled chemical production facility, allowing for the simulation of reduced environmental effect. Therefore, because of this characteristic and a valid license to use the software, SuperPro Designer® was chosen for this study.

II. MATERIALS AND METHODS

A. Wastewater and its Characterization

The wastewater was obtained from a soybean oil processing facility located in Kingston, Jamaica, and analyzed for various parameters in accordance with standard methods [21]. The main characteristics of the wastewater were (mean values): COD = 3400 mg/L, pH = 8.42, total dissolved solids = 970 mg/L, total suspended solids = 1000 mg/L, phosphate = 60 mg/L, nitrate = 120 mg/L, and Fe content = 0.63 mg/L.

B. Supporting Chemicals

The supporting electrolyte was sodium chloride (NaCl), while hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used to adjust the pH of the effluent. Solutions were made using double-distilled water. All chemicals used in the study were of reagent grade.

C. Electrocoagulation Procedure

Fig. 1 shows the schematic diagram of the bench-scale EC reactor used in the experiments. The acrylic reactor was used in batch mode and had a 1-L working volume. Two mild steel rods were used as electrodes, each possessing an effective surface area of 13.36 cm². The electrodes had an interelectrode spacing of 3 cm and were positioned 4 cm vertically above the base of the reactor. Direct current (DC) was supplied via a Sencore PS402 Triple Output (0 - 30V, 3A) power supply, and the reactor's contents were stirred with a Corning PC-4100 magnetic stirrer.

The two factors selected for study were initial pH and applied current. The pH influences the speciation of the Fe coagulant and its solubility [2, 7, 22, 23], whereas the applied current determines the quantity of the Fe released from the anode [2, 4, 24, 25]. Table 1 shows the values of the factors.

1) *Direct Iron Electrocoagulation of OW*: The term direct Fe-EC refers to the typical EC process where the Fe electrodes are in direct contact with the wastewater for the duration of treatment. For this procedure, a 1-L mixture of 1:10 diluted OW, 2.925 g NaCl and distilled water was added to the reactor. The pH of the mixture was measured and adjusted to the desired initial value by adding either 0.1 M HCl or 0.1 M NaOH. The electrodes were immersed into the wastewater and subsequently connected to the power supply. The power was switched on for 8 minutes and 38 seconds to produce theoretical Fe(II) masses of 15 mg, 30 mg, and 60 mg, corresponding to currents of 0.1 A, 0.2 A, and 0.4 A, respectively. The mixture was agitated at 300 rpm for the same amount of time. After electrolysis, the electrodes were rapidly removed, the power source was turned off, and two 2-mL samples were taken for Fe content analysis. Following that, the content of the reactor was subjected to flocculation at 60 rpm for 12 minutes. At the end of treatment, samples were taken from the EC reactor, filtered with a Whatman No. 5 filter, and the filtrate analyzed for COD.

2) *OW Treatment with Electrogenerated Fe Flocs*: The procedure is the same as that used for direct Fe-EC but with two modifications. First, 0.9 L of a 0.05 M NaCl solution replaces the wastewater in the reactor, and second, a 100 ml of a 1:10 diluted OW was introduced prior to the initiation of flocculation.

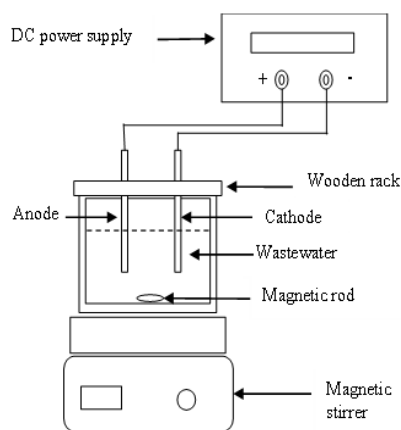


Fig. 1 Schematic diagram of the EC setup

TABLE I
VALUES OF THE FACTORS SELECTED FOR FE-EC
TREATMENT

Factor	Values		
	1	2	3
Initial pH	4	7	9
Current (A)	0.1	0.2	0.4

Three replicates of the experiments were conducted leading to a total of 27 runs for each treatment option. Before each run, the electrodes were subjected to a cleaning regime that included soaking in 1 M HCl, rinsing with distilled water, scrubbing with steel wool, rinsing again with distilled water, and wiping off any remaining residue with a paper towel. The runs were carried out at ambient temperatures of $30 \pm 2^\circ\text{C}$.

D. Analytical Methods

Total Fe concentration was measured using flame atomic absorption spectrophotometry (F-AAS) with an Agilent 240FS instrument. The measured Fe values were corrected by deducting the original Fe concentration in OW. The concentration of COD was estimated in accordance with the standard procedure for determining COD [21]. COD analysis was conducted using HACH reagents and a UV-VIS spectrophotometer (HACH DR 6000). The pH was measured using a HACH HQ440d multi-parameter meter. The removal efficiency of COD was calculated using the following equation:

$$\text{COD removal (\%)} = \frac{\text{COD}_i - \text{COD}_t}{\text{COD}_i} \times 100\% \quad (7)$$

where COD_i and COD_t are the initial COD concentration and the COD concentration at time t , respectively.

E. SuperPro Designer® Simulation

First, a representative process was designed in SuperPro Designer®. The EC process used in the study can be subdivided into three stages: electrolysis, reaction, and filtration. Electrolysis is not modelled in SuperPro Designer®; hence, only the reaction and filtration procedures are included. Fig. 2 shows the flow diagram of the process.

A batch reactor with the sequence of two charges, agitation, reaction and transfer out was set up. For the microfiltration procedure, a clarification process was added from which the filtrate and retentate streams are produced. The OW was simulated as a mixture of soybean oil and water only. By means

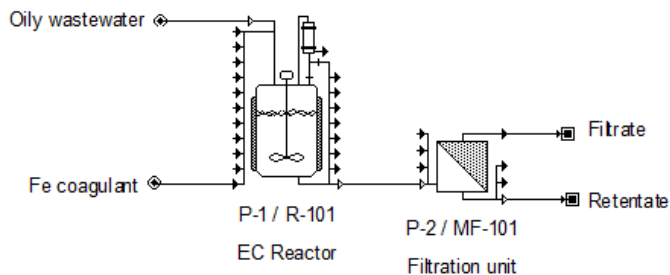


Fig. 2 EC process model in SuperPro Designer®

of trial and error, the percentage composition of the soybean was adjusted to achieve an effluent with a COD concentration comparable to the initial COD value for OW. The Fe coagulant was assumed to be iron (III) hydroxide ($\text{Fe}(\text{OH})_3$). The adsorption of soybean oil molecule onto the $\text{Fe}(\text{OH})_3$ coagulant was modelled as an either a single stoichiometric reaction or as two consecutive stoichiometric reactions. Eight product species representing potential adsorption products or intermediates were defined in the user database. The species were separated into two groups. Group 1 species were created on the assumption that 1 to 4 soybean oil molecules could bind to the solid surface of the coagulant, whereas group 2 species are formed when group 1 species combined with three molecules of soybean oil. Table 2 provides a listing of the user-defined adsorption products and their mode of formation. For both adsorption models, the simulations utilized the effective mass of Fe determined through direct EC experiments and reaction conversions ranging from 80 to 100%. Figs. 3 and 4 show the dialog boxes in SuperPro Designer® for user input of design information for the batch reactor and filtration units, respectively. The percentage COD removal results of the simulation runs were compared with those obtained from the EC runs.

III. RESULTS AND DISCUSSION

A. Iron Content

Fig. 5 shows the total Fe content in the wastewater and the sodium chloride solution after electrolysis. The results indicate that the measured quantities of Fe were less than the theoretical masses expected for 0.1, 0.2, and 0.4 A. The mass of Fe in NaCl was generally higher than that in the wastewater with 53.19 mg/L obtained at 0.4A compared to 46.37 mg/L for OW. Ref. [26] similarly found that the anode released more Fe in the NaCl solution than in pretreated wastewater. The decline in Fe dissolution from the anode suggests that both solutions have a passivating effect on the anode. The influence of pH and current on the passivating effect of the solutions are further discussed in [27]. The higher mass of Fe in the NaCl solution can be attributed to the chloride ions whose presence facilitates pitting corrosion of the anode. On the contrary, oxyanions, like nitrates and phosphates in wastewater, have a negative effect on anode dissolution [22, 28-30].

TABLE II
DEFINED ADSORPTION PRODUCTS IN SUPERPRO DESIGNER®

Adsorption product	Proposed formation
FeSoyA	$\text{Fe}(\text{OH})_3 + 1 \text{ soybean oil} \rightarrow \text{FeSoyA}$
FeSoyB	$\text{Fe}(\text{OH})_3 + 2 \text{ soybean oil} \rightarrow \text{FeSoyB}$
FeSoyC	$\text{Fe}(\text{OH})_3 + 3 \text{ soybean oil} \rightarrow \text{FeSoyC}$
FeSoyD	$\text{Fe}(\text{OH})_3 + 4 \text{ soybean oil} \rightarrow \text{FeSoyD}$
FeSoyA-3	$\text{FeSoyA} + 3 \text{ soybean oil} \rightarrow \text{FeSoyA-3}$
FeSoyB-3	$\text{FeSoyB} + 3 \text{ soybean oil} \rightarrow \text{FeSoyB-3}$
FeSoyC-3	$\text{FeSoyC} + 3 \text{ soybean oil} \rightarrow \text{FeSoyC-3}$
FeSoyD-3	$\text{FeSoyD} + 3 \text{ soybean oil} \rightarrow \text{FeSoyD-3}$

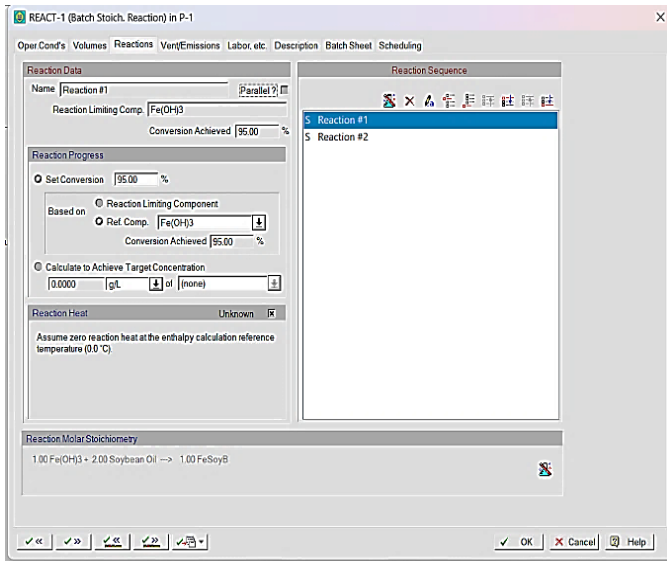


Fig. 3 Batch reactor dialog box for reaction setup

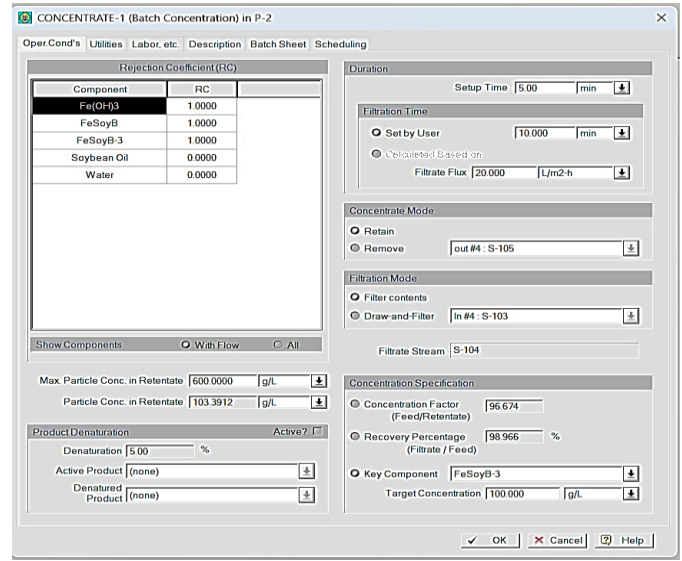


Fig. 4 Microfiltration unit dialog box for filtration setup

B. EC Treatment of OW

Table 3 presents the results of the direct Fe-EC treatment as well as the treatment utilizing preformed electrogenerated flocs. The aspect of the research aimed to determine the maximum COD removal and if flocs generated in close contact with pollutant molecules are more efficient at capturing and eliminating pollutants from wastewater. The results indicate a minimal difference between the two sets of data, which was deemed statistically insignificant at $\alpha=0.05$ ($p > 0.05$). The data shows that the average COD removal rates were notably high, ranging from 84.66% to 93.61% for direct Fe-EC and from 86.51% to 94.56% for treatment using preformed flocs. Maximum COD removal by direct Fe-EC and preformed flocs were 93.61% and 94.56%, respectively. Both values were obtained at an initial pH of 9 but at different current values. The effects of the factors on COD removal from OW are further discussed in [31].

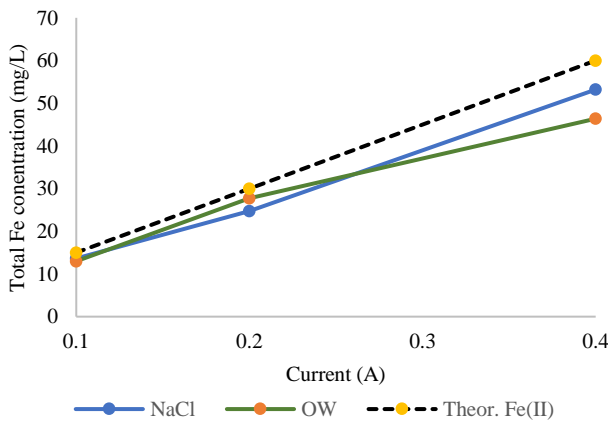


Fig. 5 Total Fe content present in the NaCl and OW at the applied currents

Due to the closeness of the removal efficiencies at all current values, there appears to be a minimum current value or mass of Fe coagulant that is effective in removing COD. Research done by [24, 32-34] has suggested that increased current levels do not always lead to a notable increase in pollution removal, as shown in Fig. 6.

These and other studies have shown that there is an optimal current value beyond which either no further substantial increase occurs or pollution removal decreases. Since there was no statistically significant difference between the two methods in COD removal at the initial pH and current values, the simulation of the EC process in SuperPro Designer® was approached from the perspective of preformed $\text{Fe}(\text{OH})_3$ entering the batch reactor.

TABLE III
COMPARISON OF COD REMOVAL BY DIRECT EC AND ELECTROGENERATED FLOCS

Method of EC treatment	Current	Percentage COD removal/mgL ⁻¹ (mean)		
		Initial pH		
		4	7	9
Direct Fe-EC	0.1 A	84.66	87.39	89.89
Preformed flocs		86.85	86.51	90.64
Direct Fe-EC	0.2 A	90.21	89.39	91.65
Preformed flocs		89.95	93.73	94.56
Direct Fe-EC	0.4A	90.53	89.94	93.61
Preformed flocs		91.57	92.35	86.62

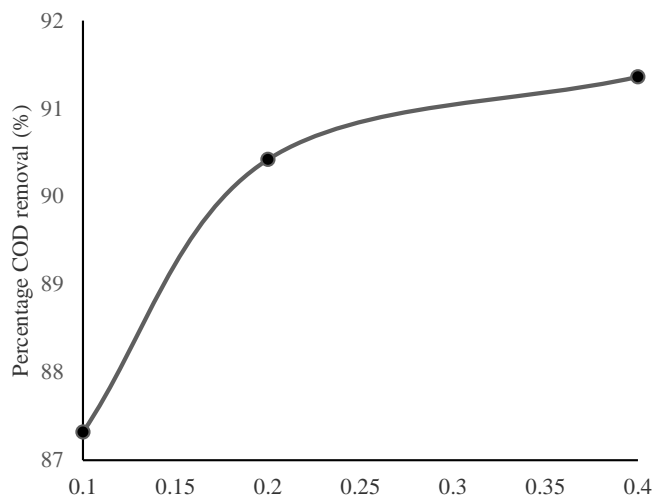


Fig. 6 Main effect of applied current on COD removal from OW by direct Fe-EC

C. SuperPro Designer® Simulation Results

The 1-L soybean oil-water mixture used had 0.12% soybean oil (1193.49 mg) composition. This yielded a COD value of 3431.29 mg/L, which was the closest estimate to the COD measured in real OW.

The mass of the coagulant $Fe(OH)_3$ used in the simulations was assumed to be equal to the quantity of iron dissolved from the anode. Based on Fig. 5, the effective mass of $Fe(OH)_3$ used was 27.70 mg, the average amount produced at 0.2 A. Any current beyond this point resulted in less than 1 mg/L elimination in COD.

Modelling the adsorption of COD constituents as single stoichiometric reactions did not yield meaningful results. As expected, the larger the molecular weight of the adsorption product and the greater the conversion of $Fe(OH)_3$, the greater the COD removal from the filtrate (S-104). At 95% conversion of $Fe(OH)_3$, 54.80 and 73.07% were the maximum COD removal obtained for FeSoyC and FeSoyD, respectively. Full conversion of the assumed mass of coagulant was not modelled as guided by Ref. [35] who reported 97.4% conversion of the dissolved Fe to solid phase under fully oxic conditions.

The second model of adsorption yielded better results. The first reaction was the same as that for the first model, whereas the second reaction used a conversion range of 80 to 100% of the group 1 product. Despite using this conversion range, the software occasionally outputs a maximum conversion lower than the input value due to the limiting reactant principle. Table 4 provides a summary of the results for the 80% and 100% conversions. The product FeSoyD-3 is not included as invalid results were obtained.

From Table 4, the method of producing FeSoyB-3 is the best representation of the adsorption mechanism taking place as the COD removals obtained are relatively close to those achieved from the direct Fe-EC treatment of OW (see Table 3). The pH of the OW could be responsible for the variations in the

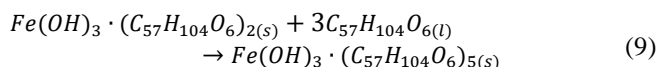
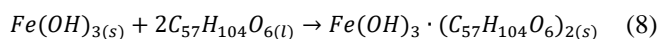
TABLE IV
SUPERPRO DESIGNER® SIMULATION RESULTS FOR COD REMOVAL BY
2-REACTION ADSORPTION MODEL

Adsorption product	COD removal (%) at 80% conversion	COD removal (%) at 100% conversion
FeSoyA-3	73.07 ^a	-
FeSoyB-3	87.31	91.31
FeSoyC-3	98.5	100

^a Maximum conversion was 67.05%

comparisons; however, the factor was not included in the simulations.

No other studies were found that have used a proprietary software to model the EC process at the time of this investigation, hence there is no data available for comparison. Although the simulations were based on mass balances and several assumptions, the study suggest the following adsorption of soybean oil ($C_{57}H_{104}O_6$) scheme:



IV. CONCLUSION

In this study, Fe-EC was applied to treat OW from soybean oil processing. The treatment of the wastewater was approached from two perspectives: direct contact of the wastewater with the electrodes or by electrogenerated Fe coagulant flocs in NaCl solution followed by the addition of the OW. It was found that no significant difference existed between the two methods and the respective maximum COD removals were 93.61% and 94.56%. Application of SuperPro Designer® was successful in modelling a representation of the adsorption process in EC, which consisted of a 2-stage reaction with one $Fe(OH)_3$ molecule and five soybean oil molecules. Although the process is expected to be more intricate than depicted, the similarity between the simulation findings and experimental data suggests that adsorption involves numerous phases and potentially many products. As research progresses to unravel the mechanisms of EC, the use of software to model the process will prove to be invaluable. Recommendations for future research include investigating the actual nature of the insoluble adsorption products and identifying the types of the attachments between the coagulant and the pollutant as a function of solution pH.

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