Experimental Design Methodology Applied to Phenol Removal from Refinery Wastewater by Electrocoagulation Process

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ABSTRACT

The present study investigates phenol removal from petroleum industry wastewater by electrocoagulation (EC) process. The effluent is treated in a batch reactor using successively iron and aluminum electrodes connected in a monopolar configuration. The effects of current intensity (0.5–3A), pH (5–10), treatment time (20–80 min), and electrode type (Fe-Al) were assessed through a full factorial design. The current intensity, the pH, and electrode type are the main influent parameters on phenol removal while treatment time has a negligible effect. Analysis of coefficients shows that current intensity and pH have positive effects on phenol removal. The coefficient of the electrode type indicates that Fe electrodes are more efficient than aluminum electrodes. The best performance of phenol removal is 90.36% and is obtained at a current intensity of 3A, electrolysis time of 80 min, and initial pH of 10. The mathematical model demonstrated a good correlation between predicted and experimental values with a high R^2 of 0.992. EC is a very effective process for removing phenol from water.

Key words: Phenol, Electrocoagulation, Full factorial Design.

1. INTRODUCTION

Faced with galloping demography and booming industrialization, there is an increase in the production of wastewater. These waters, discharged into nature, are often untreated or poorly treated, causing serious environmental problems such as surface and groundwater pollution [1,2], waters that constitute the major reserves for the production of drinking water. To date, nearly 1.1 billion people worldwide lack access to safe drinking water [3]. Wastewater from residential areas, commercial activities, and especially industrial activities are water laden with pathogenic microorganisms and/or organic matter, metals, sulfurous, or phenolic compounds [4-6]. Phenols and phenolic compounds come from oil refineries, oil mills, and pharmaceuticals… and are among the most common forms of refractory organic pollutants in wastewater. They are toxic to wildlife and their toxicity is even higher in water [7-9]. For this reason, the Environmental Protection Agency calls for a reduction of phenols in drinking and mineral waters to 0.5 ppb [10]. Adequate treatment of large quantities of wastewater is required. Thus, many methodologies have been exploited to eliminate phenol and its derivatives from wastewater before any discharge into the environment. The most used are separation by extraction, ultrasonic degradation, photocatalytic degradation, and electrochemical processes such as electrooxidation [8,10]. Many disadvantages such as high cost, low efficiency, and generation of toxic by-products have been reported for the mentioned methods [11,12]. Therefore, the search for an accurate, novel, and reliable alternative process has continued due to the introduction of stricter pollution regulations and sustainable development. Electrocoagulation (EC) has recently been presented as an emerging and alternative process [13]. The process is simple, less expensive, easily automated, and effective in the treatment of a wide range of pollutants including phenolic compounds [10,14]. EC process is performed in an electrolytic cell by immersing sacrificial electrodes in the solution to be treated. Aluminum and iron electrodes are the most commonly used due to their proven reliability and availability [15,16]. During electrolysis, the anode material undergoes electrooxidation generating *in situ* metal cations in solution [17] (Equations 1-2) with the simultaneous formation of hydroxide ions and hydrogen gas at the cathode (Equation 3).

At the anode (oxidation):

$$Al \rightarrow Al^{3+} + 3\,e^- \tag{1}$$

$$Fe \rightarrow Fe^{2+} + 2\,e^- \tag{2}$$

At the cathode (reduction):

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^- \tag{3}$$

The unstable ferrous ions can oxidize upon contact with dissolved oxygen to yield ferric ions [6] (Equation 4):

$$4Fe^{2+} + O_2 + 2H_2O \rightarrow 4Fe^{3+} + 4OH^- \tag{4}$$
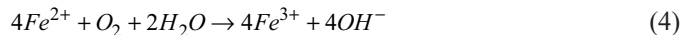
Within the solution, the metal cations released at the anode (Fe^{2+}/Fe^{3+} and Al^{3+}) react with the hydroxide ions (OH^-) to form different kinds of monomeric and polymeric coagulant species which finally transform into metal hydroxides precipitate $Al(OH)_3$, $Fe(OH)_2$, and $Fe(OH)_3$ [18]. The metal cations including the various coagulant species participate in water depollution [17]. For example, $Al(OH)_3$, $Fe(OH)_2$, and $Fe(OH)_3$ species have large specific surface areas that allow them to effectively adsorb phenol from water [19]. The flocs formed in the treated water are removed by settling, filtration, and by the action of the gaseous dihydrogen produced at the cathode. Indeed, the generated dihydrogen participates in the flotation of the flocs and favors both the removal of suspended matter and dissolved organic compounds adsorbed on the flocs.

EC process has been previously used to treat phenol wastewater [5,10,15,20,21]. According to the literature, the efficiency of the process depends on various parameters, including the initial pH of the effluent, the electrode type, the operating time, and the current intensity [22]. However, few studies have evaluated the interaction effects of these parameters on phenol removal. Moreover, the conventional method, which consists of varying one factor and holding the others constant, requires a large number of experiments, is time-consuming, and is not sufficient to explain correctly the phenomenon [23]. Studies on optimizing the parameters of phenol removal by EC process with the experimental design methodology (EDM) are limited [10]. The EDM, with a reduced number of trials, allows to keep the quality of information, to determine the main and interaction effects of factors on the response as well as the perfect optimization of the processes [24]. Of the many designs that exist, full factorial designs (FFDs) are the simplest. The main objective of this study is to optimize phenol removal from petroleum industry wastewater (PIW) by EC process using FFD. Specifically, it will be a question of quantifying and identifying the main and interaction effects of current intensity, electrolysis time, initial pH, and electrode type on the process efficiency.

2. MATERIALS AND METHODS

2.1. Wastewater Sampling and Analysis Techniques

The effluent used in this study was collected at the inlet of the Société Ivoirienne de raffinage (SIR) treatment plant. This oil refining industry is located in Abidjan (Southern Ivory Coast). The samples were collected and stored in polypropylene bottles and kept at 4°C before being physically and chemically characterized.

Turbidity was obtained with a HANNA HI 93703 turbidimeter, pH, and temperature with a pH meter (HANNA HI 98150 GLP pH/ORP meter), and conductivity with a conductivity meter (HANNA 9835 multiparameter). COD was determined by titration of excess potassium dichromate with iron ammonium sulfate in the presence of ferroin after mineralization of the sample under heat. The mineralization is done in sulfuric acid medium using the Hach DRB 200 apparatus (AFNOR standard). Phenol, ammonium, and suspended solids (SSs) were also determined according to AFNOR standards using a spectrophotometer (JASCO V-530 UV/Vis, Japan). The determination of the phenol content consists in condensing, in basic medium, the phenate ion on the 4-amino antipyrine in the presence of an oxidant, potassium hexacyanoferrate (III) (potassium ferricyanide).

2.2. Experimental Setup and Procedure

The experiments were carried out in batch mode in an electrolytic cell made of acrylic material with a capacity of 1.7 L at room temperature. Ten (10) iron and aluminum electrodes including five (5) anodes and five (5) cathodes of flat and rectangular shape (11.5 cm x 10 cm) were used. They are placed vertically in a monopolar configuration with an inter-electrode spacing of 10 mm. To ensure a constant homogeneity of the solution in the reactor, a magnetic stirrer (type Agimatic -N) mixes continuously the effluent at the same speed (700 rpm). The current intensity is produced by a direct current generator (ELC-AL781D: 0-5A; 0-30V, France) and its value is measured with an ammeter (ALDA DT- 830-D). Figure 1 shows the simplified diagram of the experimental setup.

After adjusting the pH of the effluent, a working volume of 1.7 L was poured into the reactor and then treated for a specific period of time with a pre-set current intensity. At the end of each test, the treated effluent was carefully collected in a 2 L graduated cylinder for settling for 24 h. Then, the supernatant was filtered through a glass microfiber filter Whatman™ (47-mm diameter circles) under vacuum and the residual phenol concentration is determined in the filtrate. The phenol removal rate Y (%) is calculated according to Equation 5:

$$Y(\%) = 100 \times (C_0 - C_r) / C_0 \tag{5}$$

Where C_0 and C_r are phenol initial and residual concentrations, respectively.

The pH of the effluent was adjusted with hydrochloric acid (0.1 N) and sodium hydroxide (0.1 N). The electrodes were cleaned in hydrochloric acid solution HCl (0.1 M) by scrubbing with a sponge and rinsed with tap water to remove grease deposits on the electrode surface.

2.3. EDM

In this work, a two-level (2^4) FFD was used to identify the main and interaction effects of four parameters (current intensity (X_1), electrolysis time (X_2), initial pH (X_3), and electrode type (X_4)) on phenol removal efficiency Y(%) as well as to determine the optimal conditions of the EC process. The real values of the high (+1) and low (-1) levels of the factors are summarized in Table 1.

The mathematical model associated to the FFD, taking into account only the first-order interactions, is given by Equation 6:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{12}X_1X_2 + b_{13}X_1X_3$$
$$+ b_{23}X_2X_3 + b_{14}X_1X_4 + b_{24}X_2X_4 + b_{34}X_3X_4 \tag{6}$$

Where Y, b_0, b_i, b_{ij}, and X_i represent the experimental responses (phenol removal efficiency), the average value of the observed
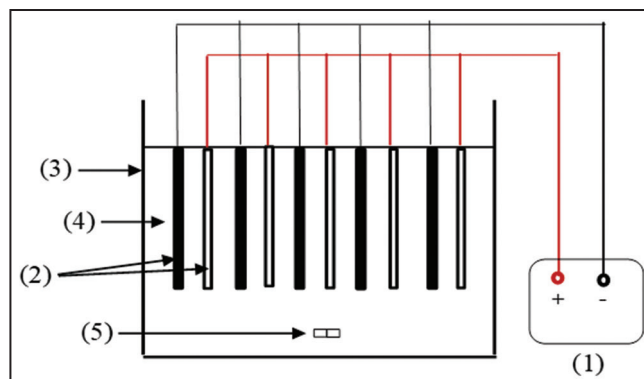

Figure 1: Schematic representation of electrocoagulation process setup: (1) Generator; (2) electrodes; (3) electrolytic cell; (4) effluent; (5) magnetic stirrer.

responses, the main effect of factor i on the response, the interaction effect between factors i and j, and the coded variable (−1 or 1) of factor i, respectively.

The calculation of the coefficients of the mathematical model and their significances, the ANOVA, the standard deviation, and the coefficients of determination were performed using the NEMROD - W software (NEMROD - W, version 9901 French LPRAI - Marseille Inc, France). ANOVA analysis was performed at the 95% confidence level.

2.4. Calculation of the Relative Contributions of the Factors on the Response

The relative contributions P_i of the factors and their interactions on phenol removal rate were calculated using Equation 7:

$$P_i = 100 \times b_i / \sum b_i \qquad (7)$$

Where b_i represents the main coefficients and interaction coefficients of the model.

3. RESULTS AND DISCUSSION

3.1. Physical and Chemical Characterization of the Effluent

The characteristics of the effluent are presented in Table 2.

Analysis of this table shows that several physic and chemical parameters such as conductivity, turbidity, TSS, COD, and phenol concentration are largely higher than the values recommended by Ivorian standards; hence the need for adequate treatment of this effluent before their discharge.

3.2. Establishment of the Mathematical Model

3.2.1. Calculation of the effect coefficients and their significance

The combination of the levels of the factors according to FFD 2^4 made it possible to construct the experimental plan. The execution of the

Table 1: Experimental domain.

Variables coded (X_i)	Factors (U_i)	Experimental Domain	
		(-1)	**(+1)**
X_1	Current intensity (A)	0.5	3
X_2	Treatment time (min)	20	80
X_3	Initial pH	5	10
X_4	Electrode type	Fe	Al

Table 2: Characteristics of raw effluent.

Parameters	Values	Ivorian standards
Temperature (°C)	26.5	≤40.00°C
pH	8.58	6.50–9.50
Conductivity (µS)	2710	200–1000
Turbidity (NTU)	192	<25
MES (mg/L)	100	30
COD (mg/L)	397.60	300
BOD$_5$ (mg/L)	110	50
[NH_4^+] (mg/L)	66.05	15
Phenol (mg/L)	1.63	0.3
Color	Dark	Colorless

tests gives the phenol removal rates, the different results of which are recorded in Table 3.

Table 3 reveals a large variability in responses within the chosen experimental domain. This means that the chosen experimental domain is relevant [25]. The phenol removal rates vary between 42.92% and 90.36%. Understanding the effect of the factors is facilitated by calculating the coefficients of the mathematical model. Analysis of the data in Table 3, using the NEMROD-W software, gives the coefficient values and their significance. These data are summarized in Table 4.

The table shows positive and negative coefficient values. The coefficient $b_0 = 61.656$ represents the average value of the 16 trials. On average, a percentage of 61.656% of phenol is removed. Positive values of the coefficient mean that increasing the levels of the factor has a synergistic effect on the response, while a negative sign indicates an antagonistic effect by increasing the levels of the factor [26]. Furthermore, significant coefficients are those whose probability is

Table 3: Experimental plan and results.

Runs	Experimentation plan				Phenol removal Y (%)
	U_1	U_2	U_3	U_4	
1	0.5	20	5	Fe	42.92
2	3	20	5	Fe	48.03
3	0.5	80	5	Fe	43.28
4	3	80	5	Fe	45.84
5	0.5	20	10	Fe	74.12
6	3	20	10	Fe	85.62
7	0.5	80	10	Fe	83.43
8	3	80	10	Fe	90.36
9	0.5	20	5	Al	53.50
10	3	20	5	Al	54.96
11	0.5	80	5	Al	45.84
12	3	80	5	Al	49.83
13	0.5	20	10	Al	69.20
14	3	20	10	Al	70.29
15	0.5	80	10	Al	62.26
16	3	80	10	Al	67.01

Table 4: Values of the model coefficients.

Name	Coefficient	Significances (%)
b_0	61.656	***
b_1	2.337	*
b_2	−0.674	31.9%
b_3	13.631	***
b_4	−2.544	**
b_{12}	−0.058	92.5%
b_{13}	0.697	30.4%
b_{23}	1.153	11.5%
b_{14}	−0.926	18.7%
b_{24}	−2.202	*
b_{34}	−5.552	***

***$P<0.1$%; **$P<1$%; *$P<5$%

<5% ($P < 5\%$). Thus, in this study, the main effects (current intensity (X_1), pH (X_3), type of electrode (X_4), and interactions (X_2X_4) and (X_2X_4) are significant. The significance of the factors and their interactions are highlighted by the Pareto chart [Figure 2].

From the figure, it appears that the pH (X_3) has a strong contribution (78.55%), followed by the interaction X_3X_4 (13.03%), then the electrode type (2.74%), the current intensity X_1 (2.30%), and finally, the interaction X_2X_4 (2.05%). The influential factors have a cumulative contribution of 98.72%. Only 1.28% as the total contribution is attributed to non-influential factors.

3.2.2. Analysis of model quality
The values of the coefficients make it possible to write the first-order polynomial of the model expressed as follows (Equation 8):

$$Y = 61.656 + 2.337X_1 - 0.674X_2 + 13.631X_3 - 2.544X_4 \quad (8)$$
$$-0.058X_1X_2 + 0.697X_1X_3 + 1.153X_2X_3 - 0.926X_1X_4$$
$$-2.202X_2X_4 - 5.552$$

The comparison of the phenol removal rates given by the model with the experimental values makes it possible to construct the graph in Figure 3.

This figure shows a good correlation between the experimental and calculated responses because the different points are mostly aligned on a straight line. The high coefficient of determination ($R^2 = 0.992$)
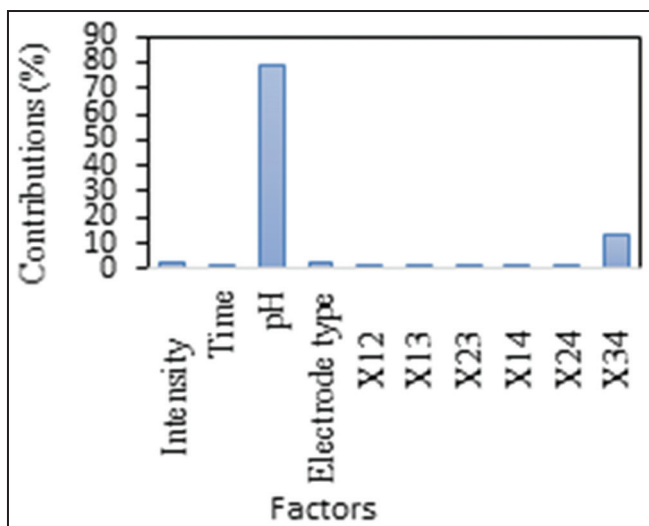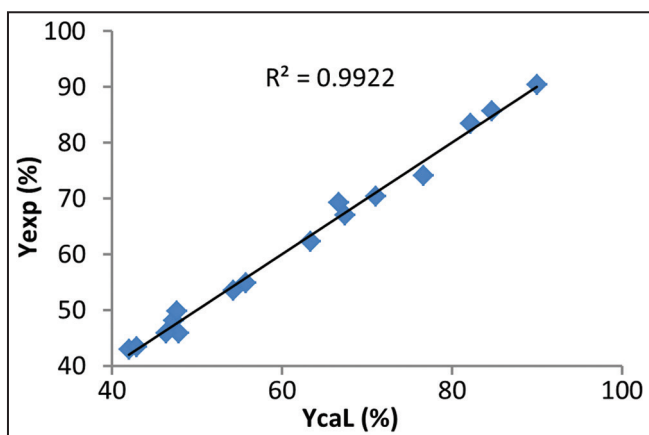

Figure 2: Pareto chart.

Figure 3: Comparison between experimental and calculated values.

shows that the model explains a large percentage of the phenol removal rate. For the ANOVA analysis of the model, the calculated F value ($F = 64.0674$) is higher than the critical value in the Fisher–Snedecor table ($Fc = 4.735$) with a $P = 0.0121 < 5\%$. This demonstrates that the model is valid and robust [26].

3.3. Main Effects and Interactions
3.3.1. Main effects
Table 4 shows that phenol removal is strongly influenced by initial effluent pH with a positive effect ($b_3 = 13.63$). The phenol removal rate increases on average by 27.26 (2×13.63) for an increase in the initial effluent pH from 5 to 10. It has been previously established that pH is a crucial parameter in the EC pollutant removal process, as it affects the predominance of the different coagulant species in equilibrium in water [27]. Consequently, the coagulation and the adsorption of phenol on the surface of metal hydroxides depend on this parameter. Phenol removal is low at acidic pH because metal cations (Fe^{2+} or Al^{3+}) are predominant at this pH range. These cations are unable to remove phenol. However, as pH increases, $Fe(OH)_3$ and $Al(OH)_3$ species become increasingly predominant thus increasing phenol adsorption sites [19]. The different results obtained with the FFD coincide with different works [10,19]. However, these authors found that a very alkaline solution (pH > 10) can have a negative effect on the effectiveness of the treatment.

The second most important factor is the type of electrode. Its coefficient of effects is negative ($b_4 = -2.544$), which indicates that the change of iron electrode instead of aluminum-induced a decrease in the removal rate of 5.09% (2×2.544). In this study, iron electrodes perform better than aluminum ones. This result could be explained by the differences in metal hydroxides formed by the two types of electrodes. Iron hydroxides would have a good affinity in the phenol adsorption process. The results obtained are similar to those obtained by Mahvi *et al.* [7]. Indeed, these authors eliminated 98% of phenol with iron electrodes and 95% with Al electrodes in refinery effluent.

The last significant coefficient is related to the current intensity. The sign is positive ($b_1 = 2.337$), which means that increasing the levels of this factor helps to increase the phenol removal rate on average by 4.67% (2×2.337) when the current intensity varies from 0.5 to 3 A. The current intensity is generally an important factor in electrochemistry. In the case of EC, this factor determines the amounts of Al^{3+} or Fe^{2+}, hydroxide ions according to Faraday's law as well as the amount, the size, and the distribution of dihydrogen bubbles [12,15]. This affects the growth of precipitates ($Al(OH)_3$, $Fe(OH)_2$, $Fe(OH)_3$) responsible for phenol adsorption.

The factor related to the electrolysis time is not significant on the phenol removal rate in the range defined for this study.

3.3.2. Interaction effects
Among the interaction effects, only X_3X_4 (electrode type- pH) and X_2X_4 (electrode type-electrolysis time) are the most significant. Their effects are worth -5.55 for X_3X_4 and -2.202 for X_2X_4. These interactions have a negative effect on the abatement of phenol. Interpretation of interaction effects is easier with Figure 4.

The summits of the squares represent the combination of the lower and upper levels of initial pH (X_3) and electrode type (X_4) [Figure 4a]. For Figure 4b, it is the combination of the levels of electrolysis time (X_2) and electrode type (X_4) that represents the summits of the squares.

In Figure 4a, when Fe electrodes are used and the pH changes from 5 to 10, there is an increase in removal rates from 45.02 to 83.38% (a gain of 43.36%). For the same pH variation with aluminum electrodes, an increase in removal rates from 51.03 to 67.19% (a gain of 16.16%) is
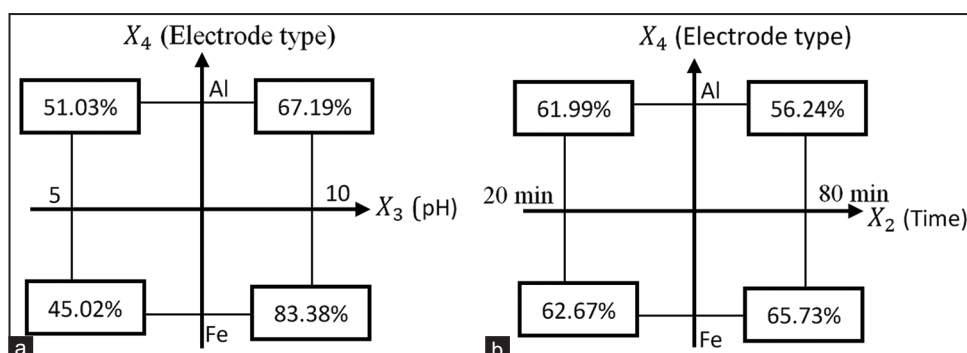
Figure 4: Interaction graphs (a): pH - Electrode type interaction and (b): Electrolysis time - Electrode type interaction.

obtained. The effect of the initial pH on the phenol removal depends on the nature of the electrodes.

Regarding the X_3X_4 interaction [Figure 4b], a slight increase in removal rates from 62.67 to 65.73% is obtained with Fe electrodes for treatment time varying from 20 to 80 min. For the same variation of treatment time (20–80 min) with Al electrodes, the opposite effect is obtained. A decrease in the rate of 5.75% is obtained. The effect of the treatment time on the phenol abatement depends on the nature of the electrodes used.

3.4. Optimal Conditions

The highest phenol removal rate was obtained in test N° 8 with a reduction rate of 90.36% (residual concentration of 0.151 mg/L) using iron electrodes. These conditions represent the optimal conditions. To achieve this rate, a current intensity of 3A was applied to the effluent of pH 10 for a treatment time of 80 min. When focusing on the other pollutants, the residual contents of the treated water in COD, ammonium, MES, and turbidity are 43.64 mg/L; 20.54 mg/L; 9.6 mg/L; and 9 NTU, respectively. Apart from the residual ammonium content, the residual content of other pollutants is below Ivorian standards.

4. CONCLUSION

The objective of this work was to optimize the treatment of phenol from PIW by the EC process. An FFD was used to model the process, quantify, and know the effects of current intensity, reaction time, initial pH, and electrode type on the phenol removal rate. The model obtained was of good quality. Analysis of the principal coefficients related to the factors revealed that an increase in current intensity and initial pH increased the efficiency of phenol treatment. The effect of electrolysis time was not significant. The results also showed that iron electrodes perform better than aluminum electrodes. The optimization of the process with the iron electrodes gave the following conditions: current intensity = 3A, reaction time = 80 min, and initial pH = 10. These conditions allowed us to reach up to 90.36% phenol removal rate for a residual concentration of 0.157 mg/L. This residual rate is largely lower than 0.3 mg/L as recommended in the Ivorian standards. In conclusion, we can say that the electrochemical EC process has been very effective for the remediation of phenol-containing oil effluents as revealed in the present study.

5. ACKNOWLEDGMENTS

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