A Study of an Experimental Design Methodology for the Removal of Cadmium from Polluted Wastewater via the Electrocoagulation Process using Solar Energy

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ABSTRACT

The aim of this study was to optimize several parameters that affect the Electrocoagulation (EC) process of Cadmium (Cd) from polluted wastewater. To estimate the contribution of each parameter to the overall removal process, an experimental design methodology was employed. System performance was optimized using Response Surface Methodology (RSM), focusing on key factors, such as pH, amperage, and salt (NaCl) concentration. Minitab 19 software was utilized to develop a full model equation, including the

main effects and interactions between the variables. The analysis revealed that pH and amperage were the most influential factors, contributing 43.04% and 26.27%, respectively, to system efficiency. The optimal parameter range for achieving a yield (above 95%) was identified through response contour plots and interaction analyses: pH ranged between 6.9 and 11, amperage between 300 and 500 mA, and salt concentration between 0.1 and 0.6 g/L. Further refinement of these ranges was visualized using response surface diagrams and the Box-Behnken design. Practical experiments were conducted using the optimized values (pH = 6.2, amperage = 280 mA, salt = 0.46 g/L), and the results were compared to the theoretical model. A minimal error margin of less than 2% between the practical and statistical results validated the model's accuracy.

Keywords-Electrocoagulation (EC); heavy metal removal; Cadmium (Cd); amperage; pH; salt concentration

I. INTRODUCTION

Cadmium (Cd) is a heavy metal mainly found in the Earth's crust typically with zinc, and appears as a by-product in the extraction process of zinc, lead, and copper. This silvery-white material shares physical properties with zinc, with an atomic molar mass of 112.4 g/mol, and a specific gravity of 8.65 g/cm³. It is utilized in several industrial applications, including electroplating, soldering electrical and electronic circuits, producing low-melting-point alloys, and manufacturing cathodes for rechargeable nickel-cadmium batteries, as well as in television screens, nuclear reactor control rods, batteries, and dyes. Cadmium can pervade into nature through industrial processes, polluting sources of drinking water and food, with the accumulation of cadmium from high-cadmium foods, such as liver, mushrooms, mussels, crustaceans, cocoa powder, and dried seaweed, increasing bodily concentration significantly [1]. Additionally, tobacco smoke introduces it into the lungs, where it is absorbed into the bloodstream and distributed throughout the body, exacerbating dietary effects. It is initially transported to the liver via the bloodstream, it binds to proteins and forms complexes carried to the kidneys, where it accumulates and damages filtration mechanisms. This leads to the excretion of essential proteins and sugars, further exacerbating kidney damage. Health issues associated with cadmium exposure include diarrhea, abdominal pain, severe vomiting, bone fractures, infertility, central nervous system disorders, and an increased risk of cancer development [2-5].

Finding techniques to eliminate this pollutant is crucial. Several methods for removing heavy metals, including chemical precipitation, membrane separation, reverse osmosis, and electrodialysis, are economically unviable and energy consuming [6-7]. However, adsorption using natural materials, such as zeolites, clay, peat moss, and agricultural waste (rice husk, neem husk, black gram, tea waste, and fly ash), is a cheap, sustainable technique [8-27]. Inexpensive adsorbents, such as rice husk and fly ash, have indicated effective removal of heavy metals, like Fe, Pb, Ni, Cd and Cu, from wastewater. EC is an electrochemical technique that makes it possible to depollute wastewater using electricity instead of chemical reagents. This approach has been successfully adopted for various industrial water effluents, including pharmaceutical, textile, and wastewater effluents containing heavy metals, suspended solids, emulsified organics, and many other contaminants [28-38].

This study aimed to statistically optimize the parameters affecting the efficiency of the EC process for polluted wastewater by Cd. Aluminum plates were used as electrodes and photovoltaic cells as an energy source. The parameters to be optimized included pH, amperage, and the amount of salt added to improve the conductivity of the medium.

II. MATERIALS AND METHODS

A. Materials

1) Chemicals

All chemical reagents used in this study, including cadmium (Cd), sulfuric acid (H_2SO_4), phosphoric acid (H_3PO_4), hydrochloric acid (HCl), and sodium hydroxide (NaOH), were of analytical grade. These high-purity chemicals were sourced from Fluka and PanReac, both known for their stringent quality control standards. Each reagent was handled according to standard laboratory practices to prevent contamination and maintain the integrity of the experimental results.

2) Electrocoagulation Unit

The electrochemical cell employed in this study was based on a monopolar electrode configuration, as illustrated in Figure 1.

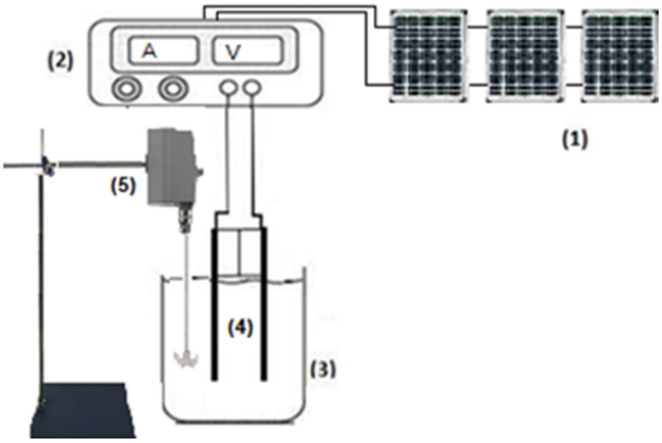

Fig. 1. EC reactor: (1) photovoltaic cells, (2) current regulator, (3) electrolytic cell, (4) electrodes, and (5) mechanical stirrer.

The chosen EC unit comprised a series of photovoltaic cells, whose characteristics are provided in Table I, namely a current regulator, a translucent Pyrex EC cell (allowing visual control of the EC process), aluminum electrodes, and a mechanical stirrer. The two electrodes consisted of parallel rectangular plates with dimensions of 120 mm × 25 mm × 1 mm, each having a nominal immersed surface area of 12.5 cm². The choice of electrode material was crucial as it significantly influenced the EC process [39]. Both the anode and cathode were exclusively made from recycled aluminum, making the electrodes a cost-effective choice for wastewater treatment applications. A characteristic spacing of 1 cm for the EC systems was maintained by a polycarbonate strip applied between them.

TABLE I. CHARACTERISTICS OF THE USED PHOTOVOLTAIC CELLS

Characteristics	Values
Dimensions (mm)	170 x 130 x 4
Maximum system voltage (V)	500
Open circuit voltage (V)	9.2
Short circuit current (A)	0.263
Maximum power current (A)	0.25
Maximum power voltage (V)	8.0
Maximum power (W)	2.0

B. *Methods*

All EC experiments followed a standardized procedure. Before each test, raw water was stirred in a container at 25 °C using a mechanical stirrer. For each experiment, 0.5 L of polluted water were added into the EC cell. The electrodes were linked to a photovoltaic panel (an electric current generator), operating with stabilized direct current, adjustable to the desired amperage. As the current passed through the electrodes, various physicochemical mechanisms were activated. Throughout this process, the contents of the EC cell were stirred using a magnetic bar, thereby enhancing the interaction between the pollutants and the sludge generated by EC. As for the equipment used to monitor the different parameters, a multi-parameter device branded CONSORT C3030 was utilized for pH and electrical conductivity measurements. Additionally, an atomic absorption spectrometer branded XPLOR AA was employed for heavy metal concentration measurements.

III. SIMULATION SOFTWARE

The software NemrodW was utilized to simulate and elucidate statistical approaches for optimizing the operational conditions. This statistical methodology enables the identification of suitable operating parameters through an appropriate model after optimization. To achieve effective optimization, it is essential to establish the minimum and maximum values of the parameters that significantly affect the efficiency of pollutant removal.

Based on the empirical experiments, it has been determined that the key parameters influencing pollutant removal efficiency are:

- X_1: pH of the solution.
- X_2: Amperage (current intensity) supplied by the generator (mA).
- X_3: Quantity of salt added to the reaction medium (g·L⁻¹).

The experimental matrix was determined using Yates' algorithm, which facilitated the systematic evaluation of these parameters to enhance the efficiency of the removal process Tables II and III present the coded and actual values, respectively, for each parameter.

TABLE II. CODES ASSIGNED TO THE MIN AND MAX VALUES OF THE PARAMETERS

Combinations	X_1	X_2	X_3	Y
1	-	-	-	Y_1
2	+	-	-	Y_2
3	-	+	-	Y_3
4	+	+	-	Y_4
5	-	-	+	Y_5
6	+	-	+	Y_6
7	-	+	+	Y_7
8	+	+	+	Y_8

TABLE III. ACTUAL VALUES ASSIGNED TO THE MINIMUM AND MAXIMUM VALUES OF THE PARAMETERS

Combinations	Factor A (pH)	Factor B (current intensity)	Factor C (NaCl)	*Y* (%)
1	2.8	100	0.2	46.23
2	11	100	0.2	49.71
3	2.8	500	0.2	58.39
4	11	500	0.2	62.47
5	2.8	100	1	48.53
6	11	100	1	56.42
7	2.8	500	1	66.78
8	11	500	1	72.51

IV. STATISTICAL STUDY

A. *Experimental Design*

The experimental design and statistical analysis were conducted using Minitab 19 software. This enabled the evaluation of the relative significance of the adjusted factors and their possible interactions. After data entry, the equation for the full model, including main effects and interactions, was obtained:

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{23}X_2X_3 + a_{13}X_1X_3 + a_{123}X_1X_2X_3 \quad (1)$$

Substituting the coefficients yields:

$$Y = 37.5 + 0.368X_1 + 0.161X_2 + 0.0549X_3 + 0.0059X_1X_2 - 0.0015X_2X_3 - 0.0013X_1X_3 - 0.00011X_1X_2X_3$$

A Pareto diagram of normalized effects was designed, as displayed in Figure 2. The analysis revealed that factor A emerged as the most important parameter influencing cadmium removal efficiency, accounting for 43.04% of the overall impact. This finding was in line with a previous study where the solubility and reactivity of coagulant species, such as aluminum hydroxides, were highly dependent on the pH level [32]. Amperage factor B exhibited a considerable importance of 26.27%. This finding was related to higher current densities, which facilitate electrode dissolution and particle destabilization. This improved dissolution generated more coagulant species, which in turn increased the adsorption capacity of heavy metal ions, leading to improved removal rates. Studies, such as [32], supported the idea that optimizing the current density was crucial to maximize treatment efficiency. Other factors with values below 15%, were considered statistically insignificant in this context, indicating that their effects were minimal compared to pH and amperage.

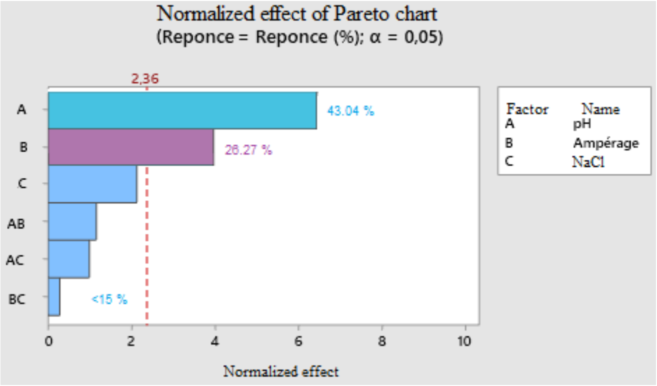

Fig. 2. Pareto diagram of normalized effect.

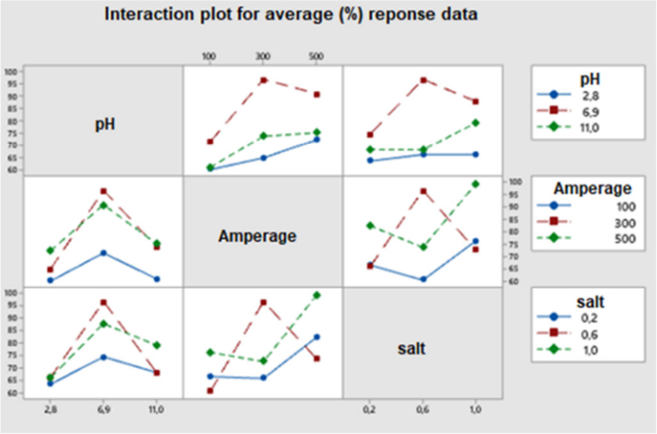

Fig. 3. Diagram of the interactions of adjusted means.

Interaction graphs were essential to evaluate how one factor influences the relationship between other factors and the continuous response, as depicted in Figure 3. In fact, the interactions between pH (X_1), amperage (X_2), and NaCl concentration (X_3) were closely examined. These results were visually confirmed by response contour plots $Y(\%)$, displaying the variations in system responses based on the parameter values, as portrayed in Figure 4. These plots suggested that, in order to maintain a response rate of greater than 95%, the factors should be fine-tuned within the following ranges.

The utilization of statistical tools, such as RSM, was crucial to explore the interplay between different factors comprehensively. The response surface diagram displayed the three-dimensional relationship in a two-dimensional format, with the variables being plotted on the X and Y axes and the response variable (Z axis) being shown as a smooth surface. To analyze responses in three dimensions, a method called the Box-Behnken design was used, involving the selection of a center value of the interval for each factor and is calculated by:

$$Y = b_0 + b_1.X_1 + b_2.X_2 + b_3.X_3 + b_{11}X_1.X_1 + b_{22}.X_2.X_2 + b_{33}.(X_3.X_3) + b_{12}.X_1.X_2 + b_{13}.(X_1.X_3) + b_{23}.(X_2.X_3) \quad (3)$$

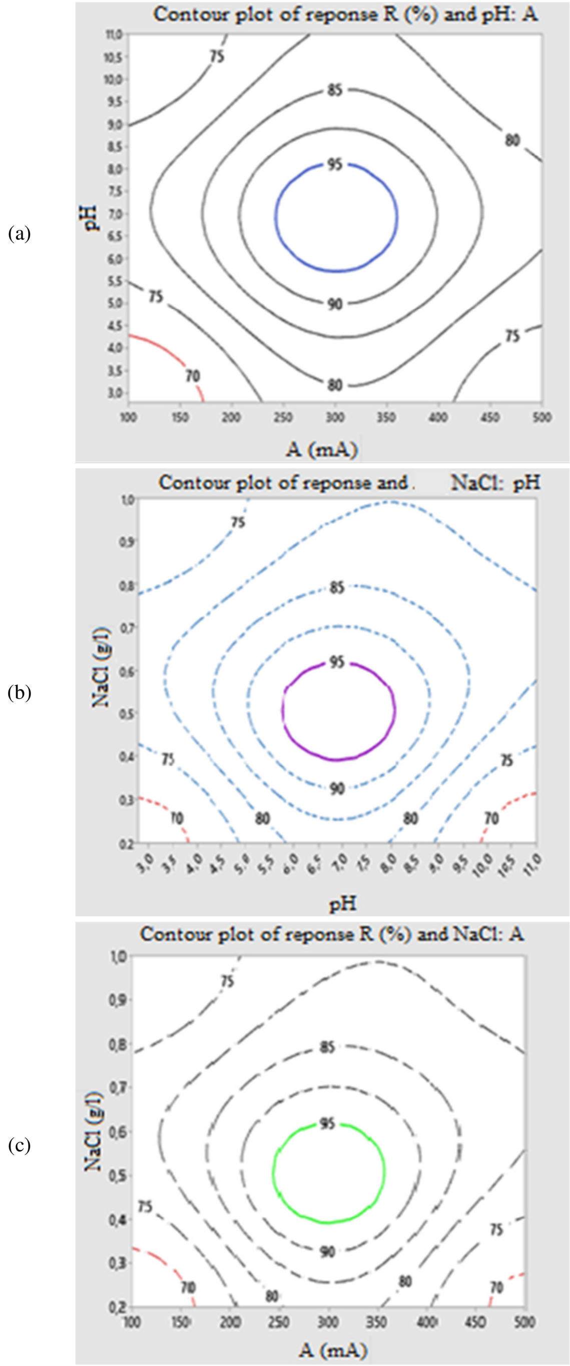

Fig. 4. Contour plot of reponse $Y(\%)$: (a) pH-A, (b) NaCl-pH, (c) NaCl-A.

The obtained results can be seen in Table IV.

TABLE IV. EXPERIMENTAL DOMAIN

	Factor	Units	Center	Step of variation
U_1	pH	-	6.9	4.1
U_2	Amperage	mA	300	200
U_3	Salt	g/L	0.6	0.4

Table V presents the new values after adding the center values of the intervals.

TABLE V. VALUES OF THE EXPERIMENTAL DESIGN PLAN

N° Exp	pH	Amperage (mA)	Salt (g/L)	Yield (%)
1	2.8	100	0.6	60.3
2	11.0	100	0.6	61.2
3	2.8	500	0.6	72.4
4	11.0	500	0.6	75.3
5	2.8	300	0.2	63.7
6	11.0	300	0.2	68.4
7	2.8	300	1.0	66.3
8	11.0	300	1.0	79.3
9	6.9	100	0.2	66.8
10	6.9	500	0.2	82.6
11	6.9	100	1.0	76.4
12	6.9	500	1.0	96.2
13	6.9	300	0.6	96.8
14	6.9	300	0.6	96.8
15	6.9	300	0.6	96.8

After running the provided data through the software, the results of Table VI were obtained, representing an estimation of the values of the model constants as well as the response (Y), standard deviation, and coefficient of error.

$$Y = 96.8 + 2.69X_1 + 7.72X_2 + 4.59X_3 - 20.29X_1X_1 - 9.21X_2 - 7.09X_3X_3 + 0.50X_1X_2 + 2.07X_1X_3 + 1.00X_2X_3 \quad (4)$$

TABLE VI. ESTIMATES AND STATISTICS OF COEFFICIENTS: RESPONSE Y (YIELD)

Coefficient	Value	F.Inflation	Standard deviation	t.exp.	Signif. %
b_0	96.80	1.00	1.77	54.71	***
b_1	2.69	1.00	1.08	2.48	5.5%
b_2	7.72	1.00	1.08	7.13	**
b_3	4.59	1.00	1.08	4.23	**
b_{11}	-20.29	1.01	1.59	-12.72	***
b_{22}	-9.21	1.01	1.59	-5.78	**
b_{33}	-7.09	1.01	1.59	-4.44	**
b_{12}	0.50	1.00	1.53	0.33	75.3%
b_{13}	2.07	1.00	1.53	1.35	23.3%
b_{23}	1.00	1.00	1.53	0.65	54.7%

Standard deviation of response: 3.06, R^2: 0.982, Adjusted R^2: 0.949, Predicted R^2: 0.711, PRESS: 751.320, Number of degrees: 5

Certain factors, like amperage (X_2) and salt concentration (X_3), significantly affected the response, with low p-values (< 5% significance level). Higher-order terms, like X_1^2, X_2^2, and X_3^2, were also significant, indicating nonlinear relationships between the factors and the yield. The adjusted R^2 of 0.949 and R^2 of 0.982 suggested that the model exhibited a good fit between the predicted and observed values. However, the predicted R^2 (0.711) was lower, indicating that the model's predictive ability could be weaker when used to predict outside the experimental range.

Figures 5-7 present the responses obtained during the variations of the parameters. It was noticed that optimal values were obtained at 6.2 for pH, 280 mA for current intensity, and 0.46 gL^{-1} for the added salt.

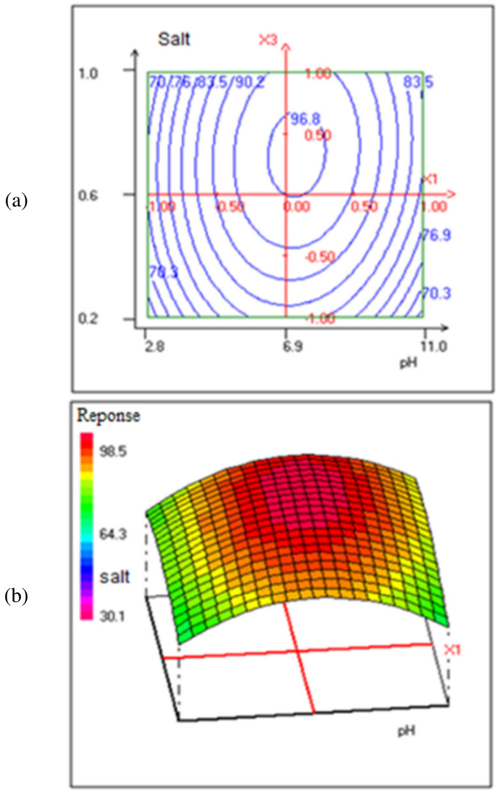

Fig. 5. Variation of (a) 2D and (b) 3D responses of yield in the plane: pH, current intensity, salt = 0.6 $g.L^{-1}$.

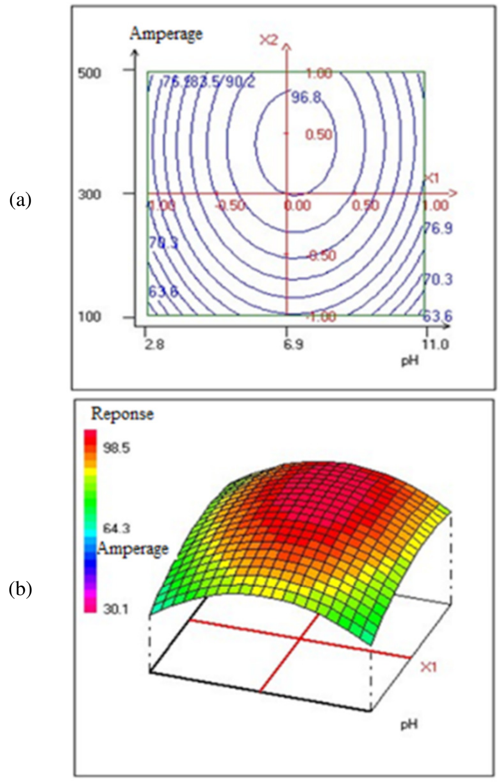

Fig. 6. Variation of (a) 2D and (b) 3D responses of yield in the plane: pH, salt, current intensity = 300 mA.

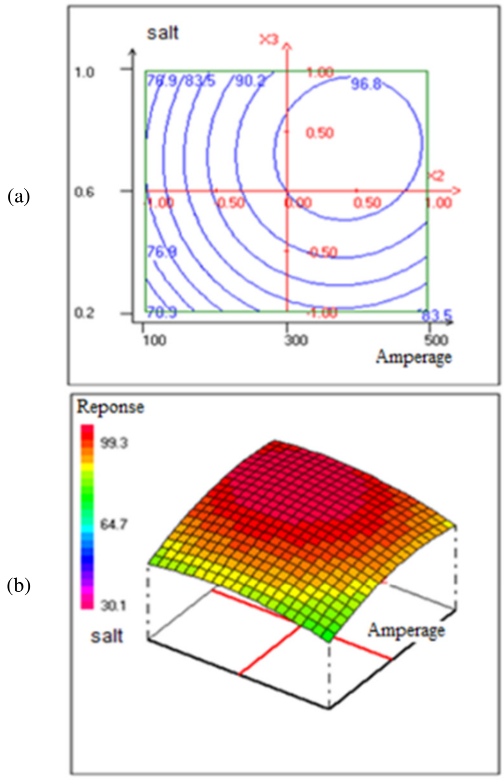

Fig. 7. Variation of (a) 2D and (b) 3D responses of yield in the plane: salt, current intensity, pH = 6.9.

Table VII demonstrates a compilation of optimal values for achieving the best elimination yield.

TABLE VII. EXPERIMENTAL OPTIMAL VALUES

	Factor	Units	Center
U_1	pH	-	6.2
U_2	Amperage	mA	280
U_3	Salt	gL^{-1}	0.46

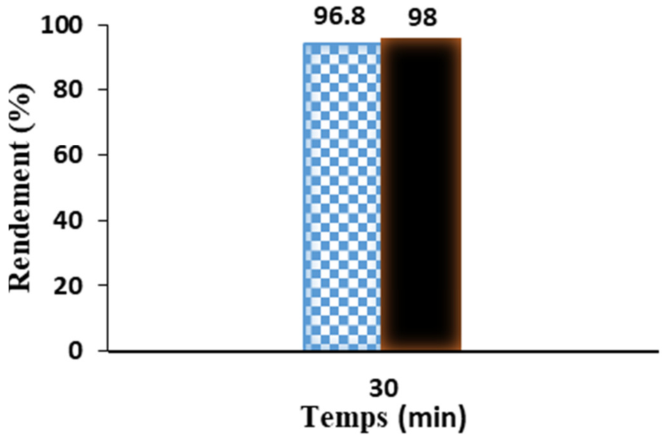

Fig. 8. Comparison of the practical (96.8%) and statistical (98%) yields.

To validate the results obtained through the statistical method, the practical experiment was repeated using the optimal values: pH = 6.2, A = 280 mA, and salt = 0.46 g/L, which were compared with the theoretical ones, as illustrated in Figure 8. It was obvious that the practical results are very close to those obtained through the statistical method, with an error margin of less than 2%. This error margin was a clear indicator of the robustness of the model. In statistical optimization, such a small margin suggested that the experimental setup, data collection, and modeling were highly accurate, confirming the effectiveness of the quadratic response model in capturing the relationships between the variables. According to literature, margins below 5% are typically considered acceptable for the practical validation of statistical models [39].

V. CONCLUSION

Cadmium (Cd) contamination in wastewater presents a critical environmental challenge, requiring innovative solutions. This study addressed a significant knowledge gap in understanding the optimal conditions for cadmium removal using Electrocoagulation (EC) powered by solar energy. Several parameters, including pH, amperage, and salt concentration, were investigated to enhance removal efficiency. The experiment was conducted using Minitab 19 software to analyze the impact of the adjusted factors and their correlations on system performance. The key findings indicated that pH and amperage were the most significant factors, with pH contributing 43.04% and amperage 26.27%. Interaction plots and response contour plots helped determine the optimal ranges for pH (6.9-11), amperage (300-500 mA), and salt concentration (0.1-0.6 g/L), ensuring a yield above 95%. Response surface diagrams and the Box-Behnken design were used to visualize factor interactions. Practical experiments were conducted with specific parameter values: pH = 6.2, amperage = 280 mA, and salt concentration = 0.46 g/L. The results revealed an error margin of less than 2% compared to the theoretical predictions, confirming the statistical model's accuracy.

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