

# Optimizing Coagulation-Flocculation Processes: Towards Enhanced Efficiency of Industrial Wastewater Treatment Stations

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**Abstract.** Wastewater treatment facilities at business parks may be optimized with the Box–Behnken design as an experimental tool to define the best combination of parameters with the least experimental trials possible. **Keywords:** textile wastewater, coagulation-flocculation, heavy metals, optimization using Box–Behnken design, computer modeling, industrial wastewater infrastructure. This paper outlines our use of the design in the optimization of the coagulation-flocculation processes used for heavy metal removal. The study achieved removal efficiencies for arsenic (91.2%), lead (82%), selenium (76.8%), nickel (70%), chromium (56.7%), and cadmium (42%) with optimization of parameters of coagulant dose, flocculant dose, and pH. Optimization improves not only efficiencies then also reduces year to year consumables while optimizing such that the effluent continues to be environmentally acceptable at lower operating costs.

**Keywords:** textile wastewater, coagulation flocculation, heavy metals, optimization using Box–Behnken design, Computer modeling

## 1 Introduction

Industrial wastewater treatment plants (IWTTPs) are industrial effluent treatment facilities that serve an important purpose in protecting our water resources and meeting socio-environmental regulations. Industrial waste treatment plants are usually designed to treat effluents based on certain pollutant loads. More importantly, treatment plants

should be evaluated and improved continuously in order to optimize treatment approaches and efficiencies, particularly for heavy metals that pose a major hazard to human health and the environment [1-2].

Heavy metals, including cadmium (Cd), lead (Pb), arsenic (As), nickel (Ni), and selenium (Se), occur in many industrial effluents, most notably from metallurgy, chemicals, and textiles. Heavy metals in industrial effluents, even at low concentrations, can be toxic and are bioaccumulating, which has detrimental impacts on humans and ecosystems [3-6]. Efficiently removing these metals from effluents before they create health hazards is an ongoing challenge for industrial waste treatment plants.

Electrolysis, reverse osmosis, ion exchange, and adsorption on activated carbon or montmorillonite, are some methods that can be used to treat industrial wastewater [7-8]. Despite success in treating wastewater, they can result in some unintended consequences such as costs, energy intensity, and residual removal issues. As a result, coagulation-flocculation has gained attention as a low-cost, simple and efficient method to remove heavy metals in industrial wastewater. Coagulation-flocculation consists of adding coagulants and/or flocculants to aid floc formation, which can adsorb the heavy metals, which in turn can be removed via sedimentation/filtration [9].

While coagulation-flocculation can be done anywhere, its effectiveness could be tailored with parameter optimization such as coagulant dosage, flocculant dosage, and solution pH. Parameter optimization is critical to improve treatment performance, reduce reagent usage and operating costs, and limits the environmental impacts [10]. Box-Behnken design, as is typically used for optimization of complex processes, can serve the purpose well since this design will minimize the number of trials while systematically testing multiple interactions of more than one independent variable [11].

Used in coagulation-flocculation will identify the parameters that provide an optimal combination of interaction parameters to desorb heavy metals from complex effluents, which is important for an industrial wastewater treatment facilities treating heterogeneous and dissimilar wastewater streams [12-13]. Although there has been industry work, especially with ferric chloride ( $\text{FeCl}_3$ ) as a coagulant to remove some metals, there has not been sufficient work studied of  $\text{FeCl}_3$  in combination with high performing flocculants, such as Himoloc DR3000. Nonetheless, this reaction is significant to constraints the process of coagulation-flocculation as well as optimize the removal efficiency of heavy metal from complex effluents (Cao et al., 2015; Moussavi et al., 2018).

Under this context, this study ultimately aims to examine the efficacy of a dual coagulation-flocculation process with the application of ferric chloride and Himoloc DR3000 in removal of Cadmium (Cd), lead (Pb), Arsenic (As), Nickel (Ni) and Selenium (Se) transition metal cations. The metals were chosen based on their relevance to industrial effluents and previously recognized environmental and health impacts. The specific research aims for this study include:

- Evaluate the effectiveness of ferric chloride and Himoloc DR3000 in removing transition metal cations from complex wastewater matrices.
- Study the synergistic effects of interactions between  $\text{FeCl}_3$  and Himoloc DR3000 on enhancing metal removal efficiency.

- Discuss the involved mechanisms of metal cation fixation during coagulation-flocculation.

If the goals identified, are met, this study will expand coagulation-flocculation as a metal contaminated wastewater treatment process that is capable of being exploited, and expand it into industry standard use. The results may be used broadly to facilitate the development of even more environmentally-friendly and efficient water treatment technologies as part of the global initiative to protect water sources and public health.

## 2 Materiels and methods

### 2.1 Sampling and analytical equipment

Sampling took place during the time between week 1 and week 2 of June 2024, which is the dry season for northern Casablanca. Usually, low flow of natural water bodies means bulkier releases of industrial effluent and is an important period for estimating the impacts of pollutant in aquatic ecosystems. Sampling occurred in five campaigns from June 1–9, 2024 around the brewery outfall and surrounding stream area. Sampling was representative, with daily measurements of the key pollution indicators (pH, conductivity, turbidity) and samples for discharge was collected on max discharge. Samples were collected in sterilized vials, refrigerated to 4°C, and transported to the laboratory for analysis.

A complete series of analyses were performed which included chemical oxygen demand (COD), biochemical oxygen demand (BOD<sub>5</sub>), suspended solids (SS), and major ions (Ca<sup>2+</sup>, Mg<sup>2+</sup>, K<sup>+</sup>, Na<sup>+</sup>, Fe, Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>). pH, conductivity (CE), total dissolved solids (TDS), and temperature were measured in the fields using a portable multi-parameter kit (CONSORT) [16-17]. The metals were analysed using standard methods (3500-Co B, 3500-Cu B, and 3500-Ni B) along with an Atomic Absorption Spectrophotometer (PERKIN ELMER) [18-19]. The samples were pre-treated with concentrated nitric acid prior to analysis, and a deuterium background correction was board to reduce matrix interferences.

### 2.2 Coagulation-flocculation process

Coagulation-flocculation involves a first step of destabilizing the colloidal particles to allow these to aggregate to large flocs that are simple to remove through sedimentation. Coagulation is a neutralization of the surface charge of the particles, while flocculation causes agglomeration of the particles by means of a bridging mechanism by the aid of polymers [20].

The study utilized a Jar-test device (Jar Test Flocculate FC-6S, Velp Scientific) with six adjustable speed paddles to simulate the coagulation-flocculation process on a laboratory scale [21]. The device allowed control over important operating conditions: pH, agitation speed, detention time, and dose of reagents. The removal efficiency of heavy metals was calculated using the following formula:

Removal (%)

Where  $C_0$  and  $C_f$  are the initial and final concentration settings.

Coagulant Ferric chloride: Ferric chloride ( $FeCl_3$ ) was selected as the main coagulant because it readily destabilizes colloidal particles. The commercial solution used in this study was 41%  $FeCl_3$ , density 1.45 kg/L [22].

Flocculant Himoloc DR3000: Himoloc DR3000 is a biodegradable, cationic flocculant was used to facilitate metal complexation and floc formation. The flocculant has a wide applicability to pHs, and it is quite effective for treatment of effluents where separation is by spreading, capping, or flotation [23].

### 2.3 Optimization method

The Box-Behnken experimental design was used to optimize the coagulation-flocculation process. This design allows for investigations of both individual and interaction effects of multiple factors without necessarily running the highest number of experiments possible. We selected three important factors for optimization:

- One of the main parameters influencing the surface charge of particles and the floc formation.
- Coagulant dose ( $FeCl_3$ ) : Closely related to destabilization of colloidal particles.
- Flocculant dose (Himoloc DR3000) : Needed for the growth of big flocs and settling.

For each factor there were three levels (low, central, high), see Table 1. The variability between the levels was selected based on operational ranges routinely employed in coagulation-flocculation processes.

**Table 1.** Factors and levels of change

Factors	Unit	Variation (+/-)
pH	-	1.00
$FeCl_3$	mg/l	0.20
Flocculent	ml/l	1.00

To analyze the effects and interactions of factors on removal rates of heavy metals in a systematic manner, a second-order response surface methodology (RSM) mathematical model has been applied to describe the relationship that exists between the independent variables (pH,  $FeCl_3$  dosage, flocculant dosage) and the responses (removal rates of metals) in a second-order polynomial model. It is given by [24]:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j \quad (1)$$

Where  $Y$  is the response (metal removal rate),  $X_i$  and  $X_j$  are factors (pH,  $FeCl_3$  dosage, flocculant dosage),  $\beta_0$  is the constant of the model,  $\beta_i$  are the coefficients of the linear terms,  $\beta_{ii}$  are the coefficients of the quadratic terms, and  $\beta_{ij}$  are the coefficients of the interaction terms.

The Box-Behnken design consisted of 16 experiments with centre points for reproducibility. Such an amount is economical and fairly reasonable for examining the

parameter space. The construction of the experiments and analysis of the data were performed with NemrodW® software which uses a simulated annealing algorithm, a stochastic search method, that consumes a variety of random locations to efficiently probe the parameter space for the optimal conditions.

Model adequacy was verified using the coefficients of determination ( $R^2$ ) and adjusted  $R^2$  values, which also show what percentage of the data can be explained by the model. The interactions of the factors are described by the 3D response surface plots that can indicate combinations of parameters which give maximum process efficiency.

### **3 Results and discussion**

The existence of elevated levels of metals in industrial wastewater is a genuine environmental concern. Transition metals, including copper, cobalt and nickel are toxic. For this reason, regulations place limits on their concentrations in industrial wastewater discharges. This section will provide a summary of the main sources, properties and effects of each of these three transition metals. In addition, some of their chemical properties will be summarized. This will support a scientific discussion about the chemical behaviour of studied transition metals in the phenomenon of coagulation-flocculation.

#### **3.1 Characterization of wastewater**

The textile sewage is very alkaline with a pH of 8.1, and has a high concentration of suspended solids (SS) to treat reaching 863 mg/L. Evidence of the mineral contamination of the wastewater is found from the extremely high concentrations of iron (Fe = 320 µg/L), chromium (Cr = 2500 µg/L), lead (Pb = 610 µg/L), nickel (Ni = 700 µg/L), cadmium (Cd = 300 µg/L), arsenic (As = 210 µg/L), and selenium (Se = 260 µg/L). All of these exceed Moroccan standards for industrial discharges except for iron, arsenic and selenium.

Also, the effluent exhibited significant organic loading with a chemical oxygen demand (COD) of 2975 mg O<sub>2</sub>/L and biochemical oxygen demand (BOD<sub>5</sub>) of 780 mg O<sub>2</sub>/L, which exceeded the regulatory limits [25].

#### **3.2 System optimization and model equation**

This research aims to enhance the coagulation-flocculation process for the heavy metal extraction present in textile wastewater. The heavy metals of interest include cadmium (Cd), lead (Pb), nickel (Ni), chromium (Cr), arsenic (As), and selenium (Se). To assess the effectiveness of coagulation-flocculation methods on heavy metal extraction, a Box-Behnken experiment design was applied to establish the effects of three variables, pH, concentration of FeCl<sub>3</sub>, and dosage of flocculant on the removal efficiency of heavy metals present in textile wastewater.

A Box-Behnken experimental design was used to optimize the coagulation-flocculation process. To objectively evaluate the accuracy of the model, the  $R^2$  coefficient of determination and the adjusted  $R^2$  were used. A higher  $R^2$  value indicated

that there was a better relationship between predicted and experimental data, which means that the model is valid [26-27].

The concentrations of metal cations after coagulation flocculation of the cation-ferric chloride system allow for the calculation of the coefficients and corresponding heavy metal removal rate. They also allow for the equations for the removal rates for the elements to be given. These coefficients are provided in Table 2, as well as the residuals' results. The regression equations for each cation are provided below:

Equations :

$$\Psi1(\text{Cd}) = 39.0 + 7.4 Z1 + 1.8 Z2 - 1.0 Z3 - 1.7 Z1^2 - 6.0 Z2^2 - 7.3 Z3 \quad (2)$$

$$\Psi2(\text{Pb}) = 75.1 - 3.0 Z1 - 5.7 Z2 - 0.9 Z1^2 - 1.0 Z2^2 - 4.1 Z3^2 \quad (3)$$

$$\Psi3(\text{Ni}) = 58.7 - 1.3 Z1 + 3.2 Z2 - 1.5 Z3 + 1.5 Z1^2 - 0.7 Z2^2 - 2.7 Z3^2 \quad (4)$$

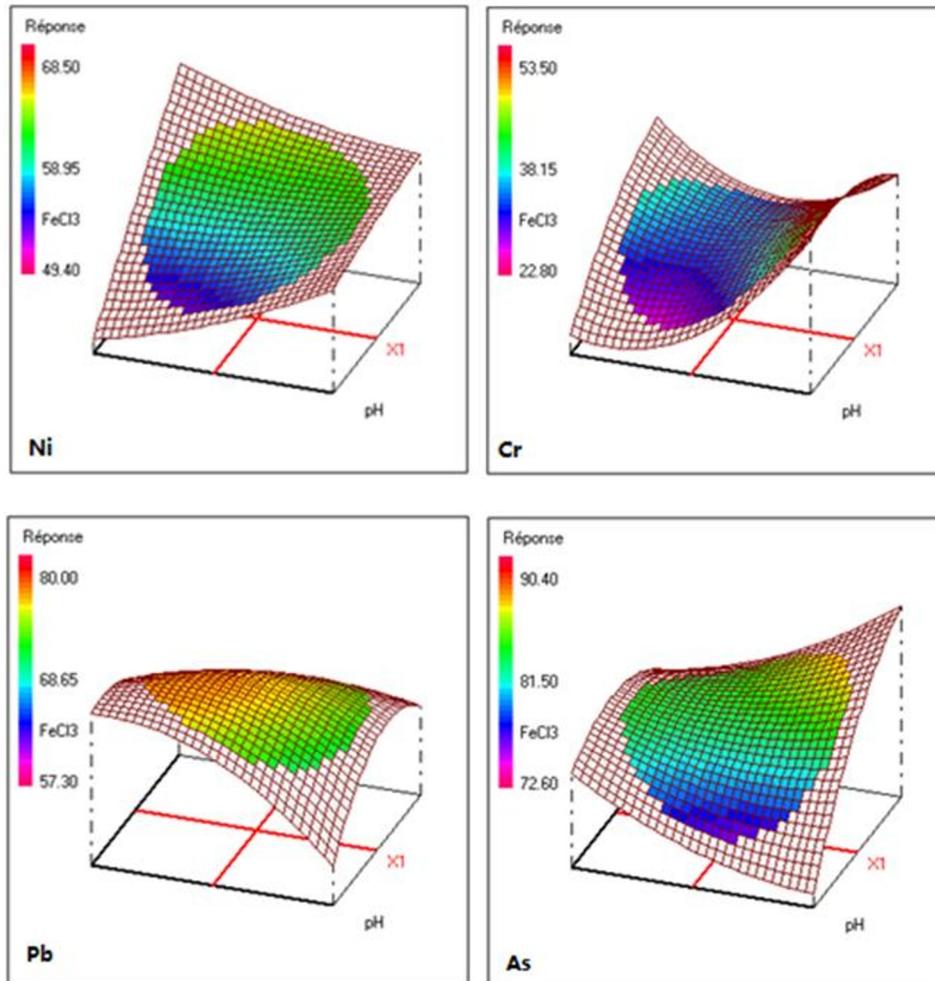
$$\Psi4(\text{Cr}) = 33.0 + 3.0 Z1 + 2.4 Z2 + 2.8 Z3 + 4.7 Z1^2 + 2.7 Z2^2 - 2.1 Z3^2 \quad (5)$$

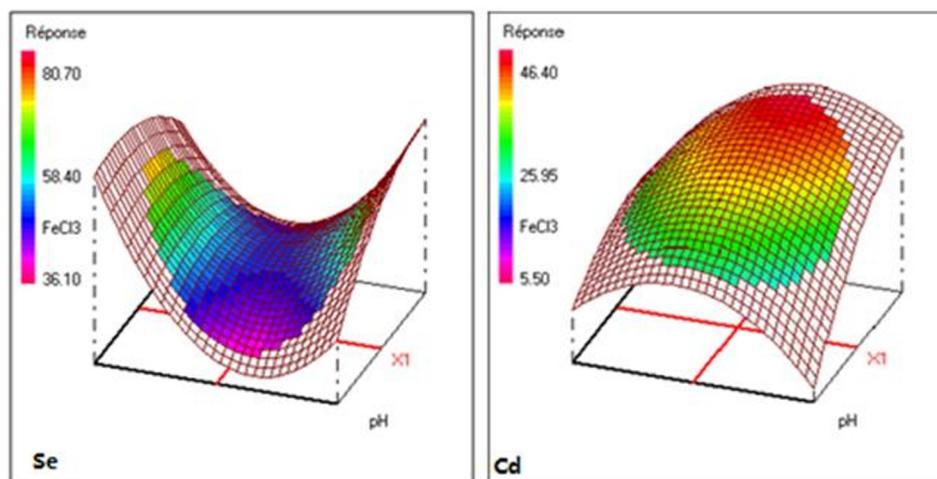
$$\Psi5(\text{As}) = 82.0 + 1.5 Z1 - 0.5 Z2 + 2.7 Z3 + 1.3 Z1^2 + 0.8 Z2^2 - 2.0 Z3^2 \quad (6)$$

$$\Psi6(\text{Se}) = 48.3 - 3.6 Z1 - 1.4 Z2 + 2.5 Z3 + 13.5 Z1'' + 8.9 Z2^2 - 4.0 Z3^2 \quad (7)$$

**Table 2.** Test conception and experimental results for wastewater of textiles

N°Exp	Factors			Reposes					
	<i>pH</i>	<i>FeCl3</i>	<i>Floc culant</i>	<i>Cd</i>	<i>Pb</i>	<i>Ni</i>	<i>Cr</i>	<i>As</i>	<i>Se</i>
Unit	-	mg/l	ml/l	(%)	(%)	(%)	(%)	(%)	(%)
1	7.1	0.44	2.6	24.0	76.1	51.0	32.6	76.4	73.4
2	9.1	0.44	2.6	12.0	72.0	52.0	32.4	74.0	42.0
3	7.1	0.84	2.6	41.5	62.0	70.0	45.6	84.4	63.1
4	9.1	0.84	2.6	42.0	70.0	63.4	32.0	91.2	52.7
5	7.1	0.64	1.6	21.0	82.0	54.0	23.4	86.0	76.4
6	9.1	0.64	1.6	32.0	76.5	63.1	45.0	86.2	76.8
7	7.1	0.64	3.6	20.5	67.4	54.0	35.4	82.1	56.7
8	9.1	0.64	3.6	31.0	62.1	56.4	56.7	80.7	70.0
9	8.1	0.44	1.6	24.0	76.0	58.1	34.0	82.3	42.6
10	8.1	0.84	1.6	32.0	76.2	54.0	36.0	80.0	60.0
11	8.1	0.44	3.6	31.0	65.7	54.8	32.5	81.0	56.0
12	8.1	0.84	3.6	36.0	66.0	58.0	33.7	82.4	57.3
13	8.1	0.64	2.6	36.0	75.2	55.6	52.0	84.0	54.7
14	8.1	0.64	2.6	36.0	75.2	55.6	34.0	82.4	52.0
15	8.1	0.64	2.6	42.0	75.1	62.4	23.0	81.4	43.5
16	8.1	0.64	2.6	42.0	75.0	61.2	23.0	80.5	43.1





**Fig. 1.** Variation in response - Ni; Pb; Cr; As; Se; Cd in plane: pH, FeCl3 FIXED FACTORS: - Flocculent = 2.60 ml/l

These findings show that the proposed method demonstrates high effectiveness for As and Pb removal, it achieved 91.2% maximum removal levels for arsenic and 82% maximum removal level for lead. It also demonstrates considerable performance related to nickel and selenium removal with maximum removals of 70% for nickel and 76.8% for selenium. It has lower removal efficiencies of chromium and cadmium since it achieved maximum removals of 56.7% for chromium and 42% for cadmium respectively. The residual concentrations of metals achieved after treatment and shown in Table 3 are compared against the Moroccan standards for industrial discharges. The results showed that the treated discharges met the Moroccan limits for all metals studied including cadmium and selenium.

**Table 3.** Study of residual responses

Response	Initial Value (µg/L)	Average Removal Rate (%)	Residues (µg/L)	Conformity to standards
Cd	300	42	174	Yes
Pb	610	82	109.8	Yes
Ni	700	70	210	Yes
Cr	250	56.7	108.25	Yes
As	210	91.2	18.5	Yes

Response	Initial Value ( $\mu\text{g/L}$ )	Average Re- moval Rate (%)	Residues ( $\mu\text{g/L}$ )	Conformity to standards
Se	260	76.8	60	Yes

### 3.3 Analysis of factor interactions

The results obtained for metals demonstrate the obvious significant considerations of coagulant concentration and pH on the response, as well as the comparatively small role of coagulant concentration, taken as a parameter on its own. The quadratic and interaction components are relatively large t except for the interaction term between pH and Flocculants concentration. The coefficient corresponding to the 2nd order interaction is almost negligible compared to the other coefficients as in the case of residual nickel t [28-29].

With regard to the figures generated for chrome removal, the following points can be addressed. The three figures, showing the relationship of Cr removal rate as a function of pH on coagulant mass ratio, represent that the Cr removal rate is low at low pH which becomes adjusted as a function of Flocculant mass ratio t which is as follows:

- For a low initial concentration of ferric chloride, the removal rate increases with the increase in the amount of flocculant;
- For an initial average initial concentration of ferric chloride, the elimination rate passes through a maximum before decreasing;
- For a high initial concentration of ferric chloride the rate elimination decreases as the amount of flocculants increases.

The three figures representing the variation in metal removal rate as a function of initial ferric chloride concentration and flocculent mass ratio show that

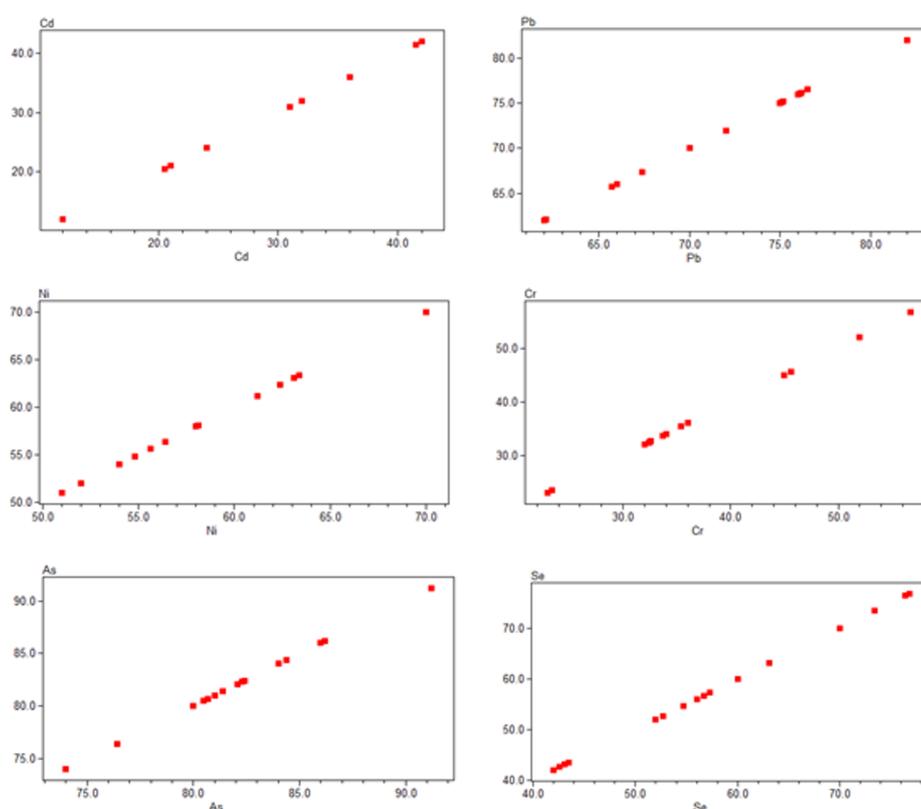
- At low pH, the removal rate passes through a maximum for a flocculants weight ratio of about 40%;
- Medium and high pH, the removal rate increases with increasing concentration of ferric chloride and goes through a slight maximum for a flocculant concentration.

In the three figures showing the variation of the rate of removal of Cd as a function of coagulant mass ratio and pH, it can be seen that the rate of removal is increasing with the increase of both of these two parameters.

The variation in rate of removal of Ni (Fig. 1) and coefficients of the Pb elimination pattern have a similar trend to that observed for As. For the measured response, as well as the figures, it is apparent that there is a similarity there. Similarly, the coefficients in the models explaining the variation in rate of change of the rates of removal (Se) were similar to those for Cd and Ni.

Figure 1 shows the experimental and predicted value graphs for Cd, Pb, Ni, Cr, As and Se. A statistical association between the elimination of Cd, Pb, Ni, Cr, As and Se, was suggested by the model p-value  $<0.05$ , when accompanied by the coefficient estimation (figure 1) at 95% confidence level. In figure 1, a 3-dimensional response surface was constructed to show the effects of coagulant dosage and pH on Cd, Pb, Ni, Cr, As and Se abatement for a textile wastewater by coagulation-flocculation processes with the dosage of coagulant of 2.6 mL/L. The figure clearly shows that as the factors of FeCl<sub>3</sub> and pH interact, the results incur the most variation in Cd, Pb, Ni, Cr, As and Se; for the sample if the dosage of coagulant fixed at 0.64 mg/L and the pH fixed at 8.1; the potential amounts of lost Cd, Pb, Ni, Cr, As and Se, can be up to 42%, 76%, 59%, 26%, 78% and 41% respectively.

### 3.4 Validation of the Model



**Fig. 2.** Experience values in relation to the values predicted for the removal of Cd, Pb, Ni, Cr, As and Se (%)

The previous results demonstrate that coagulation-flocculation differs related to pH values and coagulant and flocculant being tested. Many studies have considered investigation of the coagulation-flocculation process for wastewater, particularly with

regard to performance optimization for coagulant/flocculant, experimentation parameters, pH consideration and analysis of added flocculant [30-31].

The R2 (0.86 and 0.59) value represented the adjustment quality of the heavy metal removal. The 86% observed variation in the sample regarding Se removal was explained by the variables chosen (pH, doses of coagulant and flocculant), while 17% of the change was not explained by the model. Conversely, the fit quality of the model can be assessed based on the predicted values versus the observed values [32]. The model output approximately represented actual experimental data over the complete range of the study. The best model fit was also indicated in the figure as it can be demonstrated based on the regression index.

#### **4 Conclusion**

In conclusion, the following were the primary goals of the study. The first was to use a Box-Behnken response-surface methodology to comprehend how the corresponding treatment process worked, employing ferric chloride as a coagulant and a polymer as a flocculant. The study also sought to determine how the three crucial input factors—pH, flocculant dose, and coagulant dose—affect the effectiveness of metal removal. Since all three have been shown to be important, they are all necessary to improve the process's performance.

Results obtained lead to next conclusions:

- **Polymer Efficiency:** The polymer was shown to be effective in removing the metals of interest. Higher pH and higher ferric chloride concentration in solution increase its efficiency.
- **Optimal Conditions:** To obtain maximum efficiency, they were pH of 8, flocculant volume of 2.6 mL/L, and coagulant dosage of 0.64 mg/L.
- **Industrial Applications:** The process studied shows great potential for semi-industrial applications, especially in treating wastewater from electrochemical industries and for the demetallization of water contaminated by mining activities.

This study demonstrated the efficacy of coagulation-flocculation in the removal of heavy metals from textile wastewater by employing ferric chloride and a polymer. The research findings offer promising opportunities for growing and optimizing this effort to safeguard the environment and manage water supplies in a sustainable manner.

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