

# Application of 3<sup>3</sup> Ffd Modelling for Removal of Zinc from the Industrial Wash Water

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**Abstract** — This present study focused, full factorial design modeling to achieve the optimum treatment conditions on removal of zinc from the industrial wash water. After experimentation, mathematical model for zinc removal was developed to correlate the influences of process parameters and output response. The main & interactive effects of three different experimentally controlled factors were investigated. The contribution percentage of each factor were obtained in the ascending order as Initial Concentration (15.49%) < Current Density (22.19%) < Time (25.29%). The experimental result analysis showed that the combination of higher level of initial concentration and lower levels of both current density and time is essential to achieve maximization of zinc removal rate. ANOVA was used to find out the most significant parameters which affect the response characteristics. Results also show that about 100% of zinc removal can be obtained at optimum conditions.

**Keywords**— Electro coagulation, Wash water, FFD, Removal of zinc, ANOVA.

## I. INTRODUCTION

Historical effluent discharges from a variety of anthropogenic activities have resulted in contamination of rivers, lakes and other water bodies. The explosive population growth and expansion of urban areas has exacerbated the adverse impacts on water resources [2]. Manufacturing of electronic devices, metal plating industries and mining are some human activities that produce wash water with considerable concentrations of heavy metals [17].

Zinc is one of the toxic metals that is available in industrial effluents involved in acid mine drainage, galvanizing plants, natural ores and municipal wash water treatment plants [11]. High release of zinc into environment is also from agricultural activities, sediment entrainment and ground water [5]. In recent years, various methods of zinc removal from industrial wash water have been developed such as chemical precipitation, solvent extraction, reverse osmosis, ion exchange, evaporation, filtration, adsorption, coagulation, oxidation and reduction. Among these methods, electro coagulation has been specially applied as an efficient method for Zinc removal.

Electrocoagulation is a simple and efficient method for the treatment of many water and waste waters. It has been tested successfully to treat Potable water [16 and 9], textile waste water [6, 7 & 8], to treat urban waste water [15], to treat

restaurant waste water [18] and dye stuff [13, 10 & 4] from waste water. It has also been used to remove heavy metals [14, 1 & 3]. This process is characterized by a fast rate of pollutant removal, compact size of the equipment, simplicity in operation and low capital and operating costs. The objective of the present study is to examine the feasibility of electrocoagulation in treating plating wash water and to determine the optimum operational conditions.

Design of experiment is a statistical procedure that can reduce significantly the number of experiment, keeping, however, the reliability of the conclusions at high standard. The traditional experiment method, one factor at a time approach can hardly be used to establish relationships among all the experimental input factors and the output responses. Factorial design technique was used to reduce the number of experiments, time, overall process cost and to obtain better response and many authors applied factorial designs for the development of chemical process in various applications. Compare with the traditional method, advantage of factorial design over one-factor-at-a-time experiment are that they are more efficient and they allow interactions to be detected [12].

## III. METHODOLOGY

The plating wash water was collected at source of the industry, M/s Sundaram fasteners, Aviyur, near Virudunagar using pre-cleaned and pre-sterilized 10L carbony cans from

the fresh stock maintained by the industrial officials, before treatment. The electrode used in this study consists of aluminium plates and stainless steel plates of 99.99% purity. All the chemicals were of analytical grade and the reagents were prepared using double distilled water, by Quartz double distiller. A raw (stock) solution containing zinc wash water of 433 mg/L was used in the experiment. The working samples consist of 866, 433 and 216.5 mg/L of solution prepared by dilution with distilled water to required levels. The samples were used freshly from the ice cold deep freezer by dilution from the stock as and when required. Before the experimentation, the pH of the solution was observed to be 6.3. Other pH conditions i.e., 6.0, 7.0 and 8.0 were obtained by using 0.1N H<sub>2</sub>SO<sub>4</sub> and 0.1N sodium hydroxide solution. All measurements were carried out at ambient temperature. The working sample was prepared by taking 400 ml aliquots of real industrial wash water added with calculated and constant amount of NaCl (1gpL) to avoid excessive ohmic drop and to restrict the formation of the passivation layer on anode (aluminium or mild steel) electrodes. The schematic diagram of the experimental setup employed in the present work is shown in study Figure 1.

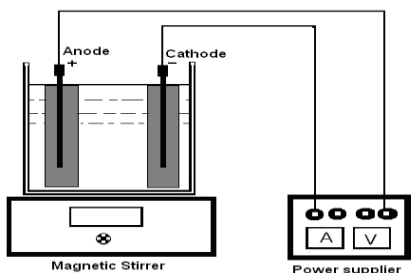


Figure 1 Schematic diagram of the experimental setup

A Statistical 3<sup>3</sup> full factorial design was used for the design of experiment and the process parameters optimization of zinc removal rate by electro-coagulation using plating wash water. Three operating factors viz., initial concentration, current density and time were taken into consideration. The parameters that varied during the present experimental investigation include Initial Concentration (mg/L), Current Density (A/dm<sup>2</sup>) & Time (minutes). Three levels were considered for each input parameter as mentioned in Table 2.

A statistical approach was used for improving the removal rate of zinc and for determining the interaction between the factors, the total of 27 experiments were conducted for three factors at three levels with single replicate as a single block is given in Table 1.

Table 1 Experimental Design, Plan and Results

Exper Number	Experimental			Experimental Plan			ZRR (%)
	X1	X2	X3	Ic (mg/L)	Cd (A/dm <sup>2</sup> )	T (Mins)	
1	-1	-1	-1	216.5	0.1	2	83.32
2	0	-1	-1	433	0.1	2	90.9
3	1	-1	-1	866	0.1	2	100
4	-1	0	-1	216.5	0.2	2	74.96
5	0	0	-1	433	0.2	2	100
6	1	0	-1	866	0.2	2	72.7
7	-1	1	-1	216.5	0.6	2	49.97
8	0	1	-1	433	0.6	2	88.87
9	1	1	-1	866	0.6	2	76.92
10	-1	-1	0	216.5	0.1	5	83.32
11	0	-1	0	433	0.1	5	90.2
12	1	-1	0	866	0.1	5	91.67
13	-1	0	0	216.5	0.2	5	49.98
14	0	0	0	433	0.2	5	80
15	1	0	0	866	0.2	5	72.7
16	-1	1	0	216.5	0.6	5	0
17	0	1	0	433	0.6	5	54.54
18	1	1	0	866	0.6	5	76.92
19	-1	-1	1	216.5	0.1	15	66.65
20	0	-1	1	433	0.1	15	81.8
21	1	-1	1	866	0.1	15	75
22	-1	0	1	216.5	0.2	15	0
23	0	0	1	433	0.2	15	60
24	1	0	1	866	0.2	15	54.54
25	-1	1	1	216.5	0.6	15	0
26	0	1	1	433	0.6	15	0
27	1	1	1	866	0.6	15	61.54

Table 2 Design Table of Process Parameters

Coded Variables	Process Parameters	Experimental Field		
		Lower	Middle	Upper
X1	Initial concentration	216.5	433	866
X2	Current Density	0.1	0.2	0.6
X3	Time	2	5	15

**IV. Results and Discussion**

**1. Selection of suitable mathematical model**

The experimental data was analyzed by model summary statistics in order to obtain regression models and decide about the adequacy of various models (linear, interactive, quadratic, cubic) to represent the electrocoagulation process significantly. The results are listed in Table 3.

Source	R-Squared	Adjusted R-Squared	Predicted R-Squared	Remarks
Linear	0.634	0.587	0.486	Not
2FI	0.737	0.659	0.496	Not
Quadratic	0.854	0.777	0.570	Suggested
Cubic	0.897	0.733	-0.388	Aliased

Table.3 Model Summary Statistics

From the Table3, it was found that, linear and interactive (2FI) models shows lower coefficient of determination ( $R^2$ ), adjusted  $R^2$ , predicted  $R^2$  and also having high p values, when compared with quadratic model. Cubic model was found to be aliased. Therefore the quadratic model is chosen to describe the effects of process parameters on the electro coagulation process to remove the zinc from the wash water.

**2a. Main Effect of Initial Concentration on ZRR**

By analysis the developed reduced quadratic response model, from the Figure 2 shows that with the increase of initial concentration the zinc removal rate increases. From Figure 2, suppose the value of initial concentration is 866mg/L then the zinc removal rate is 54.01% at cd and t are 0.45 A/dm<sup>2</sup> and 8.5mins respectively.

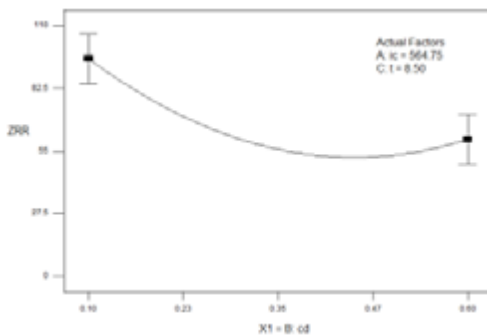


Figure 2 Main Effect of Initial Concentration Plot on ZRR

**2b. Main Effect of Current Density on ZRR**

By analysis the developed reduced quadratic response model, from the Figure 3 shows that with the decrease of current density the zinc removal rate decreases. From Figure 3, suppose the value of current density is 0.1 A/dm<sup>2</sup> then the zinc removal rate is 95.64% at ic and t are 564.75mg/L and 8.5mins respectively.

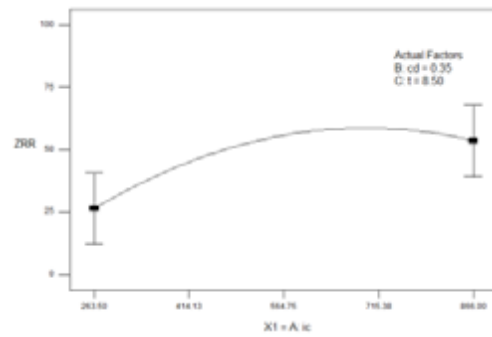


Figure 3 Main Effect of Current Density Plot on ZRR

**2c. Main Effect of Time on ZRR**

By analysis the developed reduced quadratic response model, from the Figure 4 shows that with the slight decrease of time the zinc removal rate decreases. From Figure 4, suppose the value of time is 15mins then the zinc removal rate is 34.6% at ic and cd are 564.75mg/L and 0.45 A/dm<sup>2</sup> respectively.

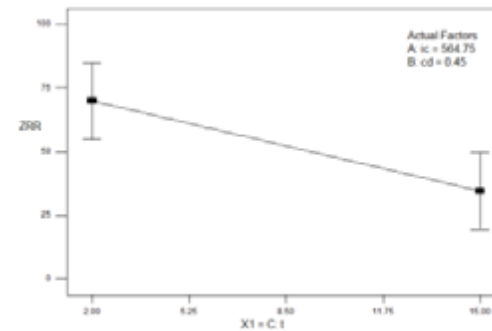


Figure 4 Main Effect of Time Plot on ZRR

Hence larger value of initial concentration and smaller values of current density and time must be selected in order to achieve higher efficiency of zinc removal rate during the electro coagulation process.

**3a. Interaction Effect between ic and cd on ZRR**

By analysis the interaction effects between two factors while kept third factor as a constant in the response model, from the Figure 5 shows that with the higher value of initial concentration and lower value of current density, higher value of zinc removal rate was obtained while keeping the time factor as a constant one. From Figure 5 shows that the value of initial concentration and current density are 866mg/L and 0.1 A/dm<sup>2</sup> respectively, the zinc removal rate is 83.6% at t is 8.5mins and at the same initial concentration (866mg/L) and time (8.5mins), the zinc removal rate is 67.87% when the current density is 0.6 A/dm<sup>2</sup>. Similarly, at the same current density (0.1 A/dm<sup>2</sup>) and time (8.5mins), the zinc removal rate is 76.1% when the initial concentration is 263.5mg/L. From the above relationship the zinc removal rate is higher (83.6%) when the current density is Lower (0.1

A/dm<sup>2</sup>) with the higher value of initial concentration (866mg/L) at time t is 8.5mins.

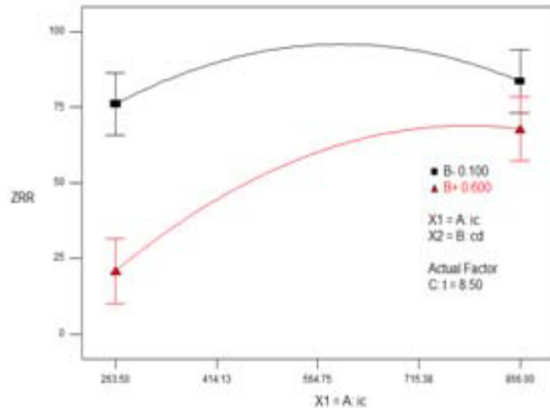


Figure 5 Interaction Effect between ic and cd plot on ZRR

3b. Interaction Effect between ic and t on ZRR

By analysis the interaction effects between two factors while kept third factor as a constant in the response model, from the Figure 6 shows that with the higher value of initial concentration and lower value of time, higher value of zinc removal rate was obtained while keeping the current density factor as a constant one. From Figure 6 shows that the value of initial concentration and time are 866mg/L and 2mins respectively, the zinc removal rate is 71.79% at cd is 0.45 A/dm<sup>2</sup> and at the same initial concentration (866mg/L) and current density (0.45 A/dm<sup>2</sup>), the zinc removal rate is 36.42% when the time is 15mins. Similarly, at the same current density (0.45 A/dm<sup>2</sup>) and time (2mins), the zinc removal rate is 36.56% when the initial concentration is 263.5mg/L. From the above relationship the zinc removal rate is higher (71.79%) when the time is Lower (2mins) with the higher value of initial concentration (866mg/L) at current density cd is 0.45 A/dm<sup>2</sup>.

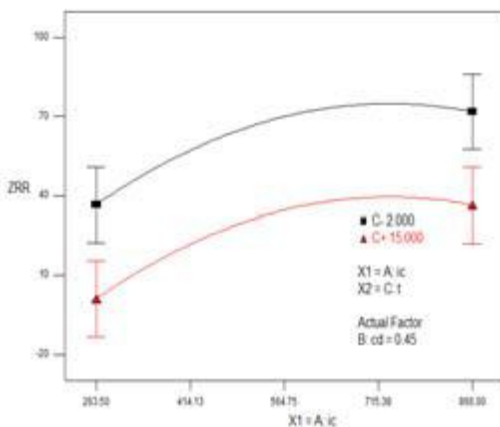


Figure 6 Interaction Effect between ic and t plot on ZRR

3c. Interaction Effect between cd and t on ZRR

By analysis the interaction effects between two factors while kept third factor as a constant in the response model, from the Figure 3 shows that with the lower values of current density and time, higher value of zinc removal rate was obtained while keeping the initial concentration factor as a

constant one. From Figure 3 shows that the value of current density and time are 0.1 A/dm<sup>2</sup> and 2 mins respectively, the zinc removal rate was 99.99% at ic is 321.5mg/L and at the same current density (0.1 A/dm<sup>2</sup>) and initial concentration (321.5mg/L), the zinc removal rate is 64.61% when the time is 15mins. Similarly, at the same time (2mins) and current density (0.6 A/dm<sup>2</sup>), the zinc removal rate was 48.63% when the initial concentration as 321.5mg/L. The above relationship concluded that the zinc removal rate is higher (99.99%) when the time is Lower (2mins) with the lower value of current density (866mg/L) at initial concentration ic is 321.5mg/L.

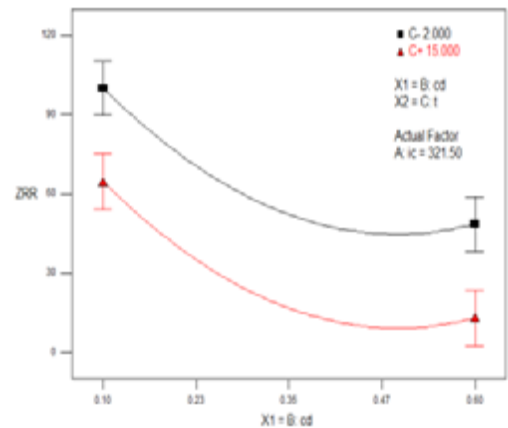


Figure 7 Interaction Effect between cd and t plot on ZRR

4. Analysis of ZRR Model

The Full Factorial Polynomial Design cube diagram with the factors level is shown in the Figure 8. Figure 8 indicates the experimental mean results for the respective levels of initial concentration, current density and time. Figure 8 shows that the effects of all independent variables like initial concentration, current density and time on Zinc Removal Rate about the particular design point. This perturbation chart (Figure 9) was used to assessed the effect of each factor by holding other two factors are constant. A curvature in a factor shows that the response is more sensitive to that factor (B – Current Density). The negative slope of curve B, points that indicates higher current density, lower the zinc removal rate. The other parameter initial concentration A shows, positive slope of curve that conclude lower initial concentration, higher zinc removal rate. The parameter time C has relatively little influence on zinc removal rate at the level 8.5 mins.

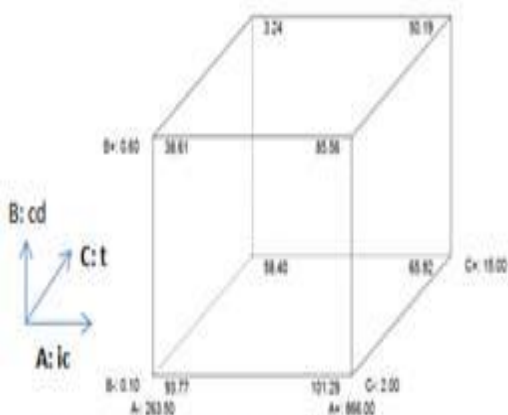


Figure 8 Full Factorial Polynomial Design Cubic Diagram for ZRR

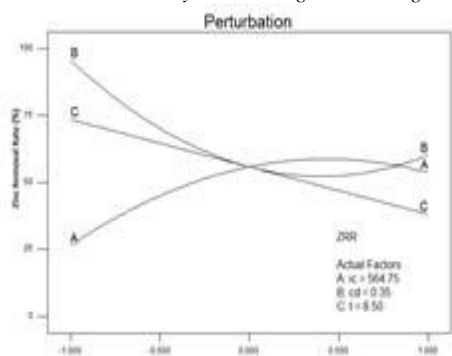


Figure 9 Deviation from the reference point

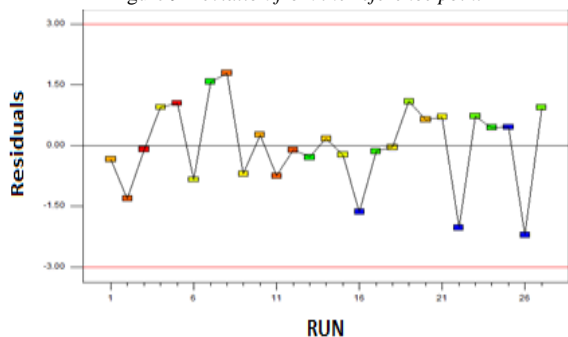


Figure 11 Residuals Vs Runs

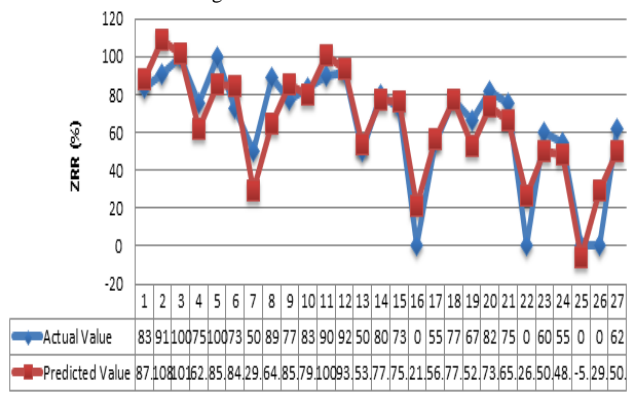


Figure 10 Experimental Vs Predicted Values

Figure 10 presents a plot of an experimental versus the predicted values of zinc removal rate. Since all the predicted values are close to the experimental values confirming that the model could predict the responses accurately. Similarly, internally studentized residuals obtained were plotted against run for the model of zinc removal rate is shown in Figure 11. Residuals were calculated as a difference between the measured and predicted values, whereas internally studentized residuals are the ratio of residual to the estimated standard deviation of that residual. It measures the number of standard deviation separating the actual and predicted values. It was found that internally studentized residuals for regression model of zinc removal rate are between + 1.790 to - 2.207. Since all the standardized residuals lie within the limits ( $\pm 3\sigma$ ) without any outliers, further confirmed that the model can be used to predict the response.

### V. Conclusion

The present work was concerned with exploring to determine the optimum setting of process parameters for single response optimization and analysis of results were carried out. Using three level full factorial design to avoid the traditional one - factor at - a - time experiments. The model has been developed FFD resulted, improved responses, reduced process variability and closer confirmation of responses to targeted requirements. According to experimental results, the optimum parametric values within investigated range have been suggested for the zinc removal rate as the 866 mg/l of initial concentration, 0.1 A/dm<sup>2</sup> of current density and 2 mins of time. Under these conditions, the zinc removal rate of 101.29% was achieved.

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