International Journal of Science and Research (IJSR)
ISSN (Online): 2319-7064
Index Copernicus Value (2015): 78.96 | Impact Factor (2015): 6.391

Optimization of Influential Factors in Gold Electrowinning using Response Surface Methodology

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Abstract: ***Electrowinning is the extraction of metals from aqueous solution. The effects of three main factors namely temperature, caustic strength and current density on gold electrowinning were studied in order to obtain high electrowinning efficiency. Response surface methodology, in combination with central composite face-centered design (RSM-CCF), was used to fit the model and ridge analysis to optimize the selected factors. A series of 17 experiments arranged in a CCF design was carried out and the results fitted using ordinary least squares (OLS) method. Findings confirmed that the effect of caustic strength was found to be the most influential of the three factors followed by temperature and current density.For deposition of gold in the circuit current density was also vital. The average electrowinning efficiency at 683, 750 and 800 A/m^2 are 77, 82.1 and 86.2% respectively. Mathematical model was constructed to characterise the electrowinning efficiency. From the model, optimal ranges obtained were caustic strength of 2.28 - 2.43%, current density of 784.50– 797.00 Am^{-2} and temperature of 46.70 – 49.50*℃.

Keywords: Gold, Electrowinning efficiency, Optimization, Response surface methodology (RSM), Central composite face-centered (CCF) design, Ordinary least squares (OLS)

1. Introduction

Electrometallurgy is a technology developed after the discovery of electric current around the nineteenth century and has been employed extensively in separation and purification processes in the metal industries. Gold electrowinning is an electrolytic process in which a direct current carried by free electrons drives chemical reaction of reduction of aurocyanide to solid gold usingelectrodes immersed in an electrolyte [1].

Generally, the electrowinning process is affected by electrochemical and physical parameters. Some electrochemical parameters include concentration and composition of electrolyte, temperature, current density and strength of additives whiles physical parameters are current distribution, time and electrolyte flow rate [2].

Gold is contaminated by base metals such as copper, iron, nickel and cobalt that have been eluted during upstream operations [3]. The impurities depending on the operating conditions and concentration usually co-deposit with gold in the circuit [1]. Some of the impurities such as copper and iron can be easily removed to such an extent that they virtually do not affect gold deposition. However, some of them such as nickel and cobalt are not easily removed [4],[5].

Low base metals content will result in improve gold deposition and bullion fineness (quality of gold). A high electrowinning efficiency and bullion fineness can be obtained from highly purified electrolyte. However, severe electrolyte purification can be economically nonviable [4]. An alternative way to achieve increase electrowinning efficiency is optimization of gold electrowinning operating conditions.

The design of experiment (DOE) and statistical techniques are widely used to optimize process parameters [6]. Often the results which have been obtained from the traditional approach of studying “one factor at a time” were only valid for fixed experimental [7].

The response surface methodology technique provides an efficient and systematic method to optimize the response or performance design [8]. RSM is a collection of mathematical and statistical techniques for designing experiments, building models, evaluating the effects of factors and analysis of problems [9],[10]. It is employed to study the relationship between one or more response variables and a set of quantitative or qualitative experimental factors. RSM is often used after the important factors are identified and to find the factor settings that optimise the response [11], [12]. The application of RSM reduces the number of experiments required for the analysis of the main effects and interactions between factors [11],[13],[14]. An important point is that many different variables can be examined simultaneously.

In the present study, the objective was to optimize gold electrowinning operation by studying the influence of temperature,caustic strengthand current density on electrowinning efficiency and also optimum parameters forgold electrowinning. Plant scale data is used for the optimisation process.

2. Materials and Methods

2.1 Factors Selection

The independent factors selected for the study were temperature (x_1), caustic strength (x_2) and current density (x_3). For the three test factors selected, each one has three levels in the central composite design matrix. The design required 17 experiments. The effect of each independent

factor and their interaction over the considered response (electrowinning efficiency) was investigated.

2.2 Method of Analysis

Simple random sampling technique with replacement was used to sample the data.The function 'RANDBETWEEN' in Microsoft excel spreadsheet was employed to generate 17 individuals in each of the 17 groups. The averages of each group correspond to one experiment. The minimum and maximum level of each factor were determined as shown in (table 1)in order to use the CCF methodology [12],[15],[16],[17].The 17 experimental runs were designed in accordance with central compositeface-centered (CCF), which allowed a full quadratic model for the response under investigation. The minimal level, centre level and maximal level of the experimental parameters were coded as -1, 0 and 1 respectively. A detailed discussion of CCF design is documented elsewhere [18].The CCF design is often classified as anRSM design [18]. The actual values of the factors in natural units along with the response values are presented in table 2.

Response surface methodology which includes factorial designs and regression analysis was used for the experimental design, model fitting and validation, and condition optimization [14].The correlation matrix between the selected factors was determined using MODDE 10.1.1 Umetrics software. The RSM model was expressed as:

$$y = f(x_1, x_2, x_3) + e \qquad (1)$$

where dependent variable y was a function of x_1, x_2, x_3 and the experimental error term denoted as e.

A quadratic regression model was selected in the gold electrowinning operation.The quadratic regression model was used to predict response values for any factor combination in the region of interest, since it was a sound choice for the optimization objective and involves modelling the curved response functions. A second-order quadratic model was employed for curvature in the response surface of this study. For three independent variables, the second-order quadratic model was expressed as:

$$y = b_o + b_1x_1 + b_2x_2 + b_3x_3 + b_{11}x^2_1 + b_{22}x^2_2 + b_{33}x^2_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + e \qquad (2)$$

where b_o is the value of the fixed response at the central point of the experiment, b_1, b_2 and b_3 are the coefficients of the linear terms; b_{11}, b_{22} and b_{33} are the coefficients of the quadratic terms and b_{12}, b_{13} and b_{23} are the coefficients of the cross products.

The method of ordinary least squares was used to estimate the parameters of the mathematical model. Finally, separate test was performed at the conditions predicted by the model.

2.3 Statistical Analysis of Results

A number of techniques were used to measure the adequacy of the regression model. The model fitting was evaluated by checking the coefficient of determination (R^2), reproducibility and prediction measure (Q^2). The optimal conditions for the factors were determined by the method of ridge analysis in RSM. The validity of the model was examined at 95% confidence interval.

Table 1: Maximum and minimum levels of the three factors

Factor	Description	Minimum value	Maximum value
Temperature	x_1	45.94	53.88
Caustic Strength	x_2	2.28	3.17
Current Density	x_3	781.3	797.1

Table 2: Central composite face-centered design arrangements of the factors in natural units along with the response

Experiment number	Run order	Independent factor			Response variable
		Temperaturex_1/(°C)	Caustic strengthx_2/(%)	Current densityx_3/(Am^{-2})	Electrowinning efficiency y/(%)
1	15	49.00	2.60	796.50	85.94
2	13	49.06	2.54	790.63	85.69
3	12	48.75	2.74	790.63	83.94
4	6	47.88	2.58	796.25	87.00
5	2	47.47	2.70	788.24	85.41
6	17	49.24	2.62	794.12	84.88
7	7	49.47	2.75	787.88	82.59
8	9	47.35	2.61	797.10	85.41
9	3	49.65	2.68	781.35	84.29
10	10	47.82	2.74	796.71	84.65
11	16	48.35	2.67	788.24	85.59
12	1	49.29	2.46	787.12	86.24
13	5	45.94	2.28	788.24	90.00
14	11	48.82	2.43	785.29	86.12
15	8	53.88	3.08	788.24	80.06
16	4	51.94	3.17	794.12	77.59
17	14	48.88	2.61	785.29	86.00

3. Results and Discussion

3.1 RSM Model Analysis of the Data

3.1.1Data Evaluation

The correlation between temperature, caustic strength, current density and electrowinning efficiency is shown in table 3.

Table 3: Correlation matrix of the selected factors

	Temperature	Caustic strength	Current density	Electrowinning efficiency
Temperature	1	0.77796	-0.14117	-0.84194
Caustic strength	0.77796	1	0.15286	-0.95412
Current density	-0.14117	0.15286	1	-0.0726654
Electrowinning efficiency	-0.84194	-0.95412	-0.0726654	1

From table 3 there exist strong negative relationshipsbetween caustic strength and electrowinning efficiency then temperature and electrowinning efficiency. A very weak negative relationship is recorded between current density and electrowinning efficiency.

3.1.2 Fitting the Model and Evaluation of Fit

The result of the quadratic response-surface model fitting for the response(y)is shown in table 4.The fitted model is given as

$$y = 85.619 - 1.382x_1 - 2.629x_2 + 0.645x_3 - 0.751x^2_1 - 0.746x^2_2 + 0.064x^2_3 - 0.676x_1x_2 + 0.249x_1x_3 - 0.574x_2x_3 \quad (3)$$

Table 4: The quadratic model for the response variable

Electrowinning efficiency	Coeff. SC	Std. Err.	P	Conf.int(±)
Constant	85.6193	0.624041	2.88246e-013	1.47564
Temperature	-1.38201	0.46118	0.0200362	1.09053
Causticstrength	-2.629	0.46118	0.000734898	1.09053
Current density	0.644999	0.46118	0.204648	1.09053
temp*temp	-0.751272	0.890973	0.426984	2.10684
cau*cau	-0.746271	0.890973	0.429926	2.10684
cur*cur	0.0637261	0.890973	0.944981	2.10684
temp*cau	-0.67625	0.515615	0.231053	1.21925
temp*cur	0.248752	0.515615	0.644215	1.21925
cau*cur	-0.573748	0.515615	0.302565	1.21925

Testing the goodness of fit of the model by checking whether the model is adequate is through examination of the plot shown in figure 1

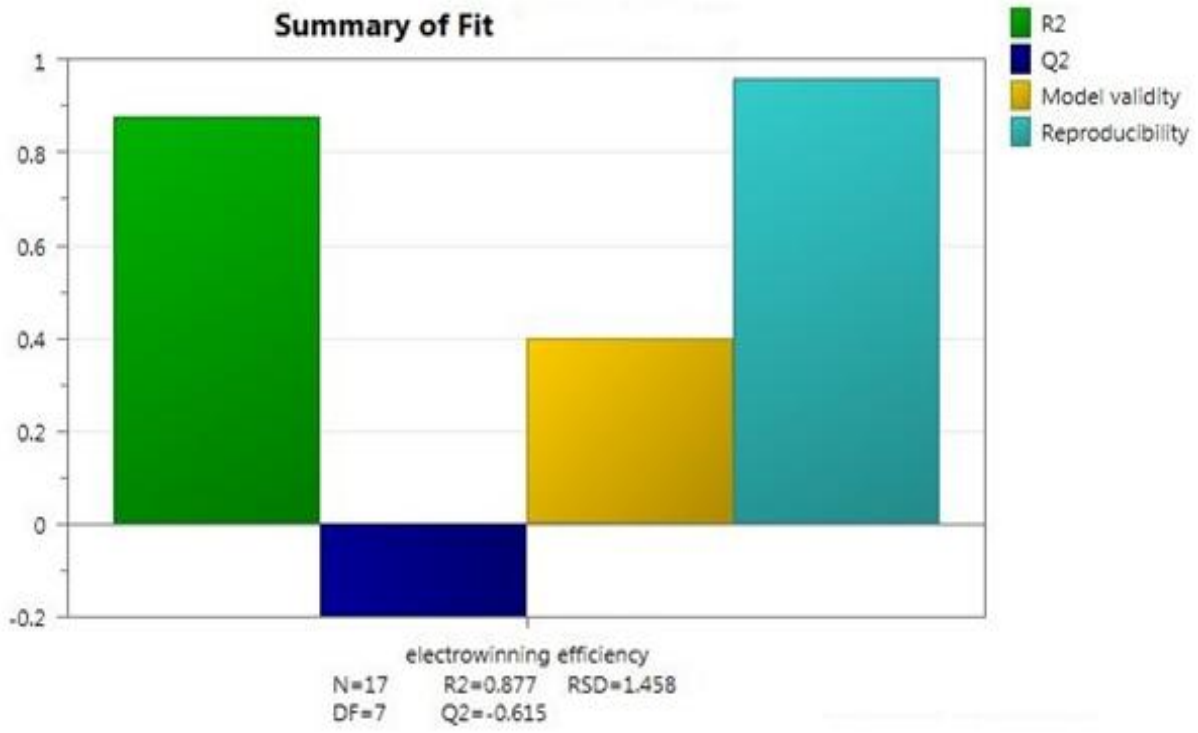

Figure 1: Summary of fit plot of the initial model

A model can be judged as good if $R^2–Q^2<0.2–0.3$, $Q^2>0.5$, model validity>0.25 and reproducibility is greater than 0.5 [12].From figure 1, the total measure of fit (R^2) is 0.877 which indicate that about 87.7% of the total variation in the response can be explained by the three independent factors. The model validity for the response is higher than 0.25, which means that there is no significant lack of fit. The reproducibility of 0.963 is above 0.5 which implies that there is a small pure error, good control of the experimental set up and the model validity evaluated. However, the predictive measure (Q^2) is -0.615 which is not acceptable for the model. A possible reason forthe low Q^2value could be that the regression model contains irrelevant term(s) [18]. This was checked through a bar chart of the regression coefficients.

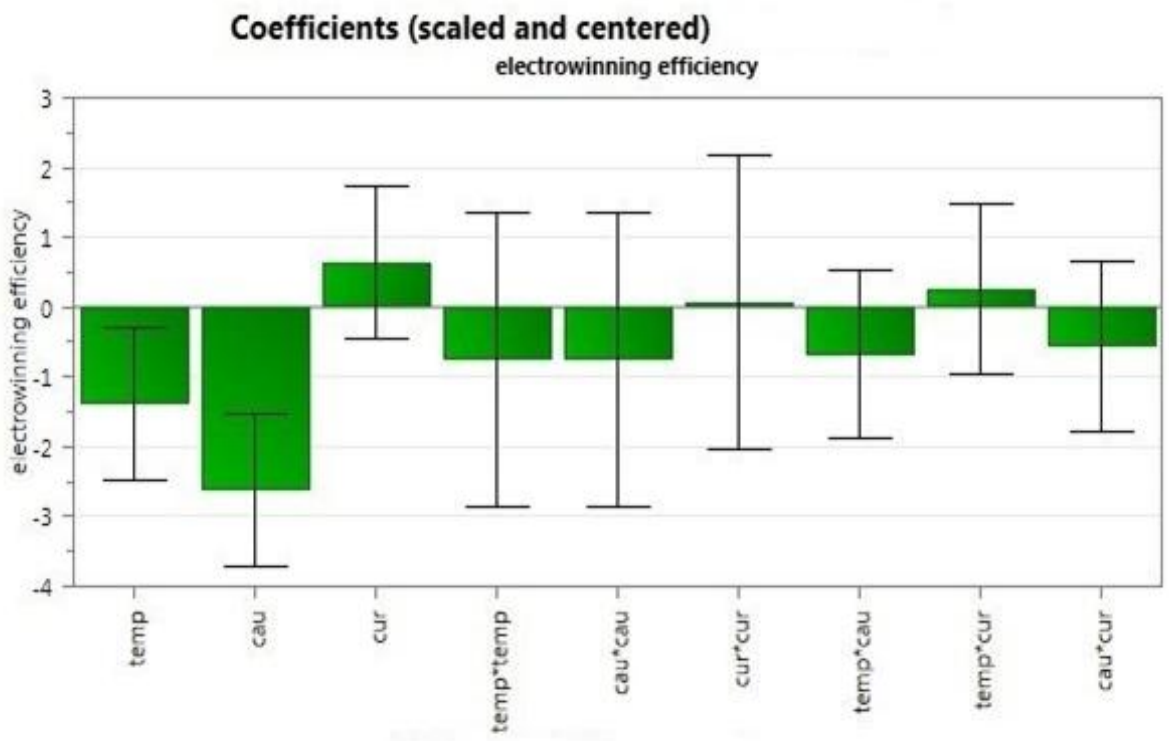

Figure 2: Bar chart of regression coefficients

Figure 2 provides the plot of the regression coefficients for the model. The plot shows the model term which is not significant for the response. From the plot the cur*cur quadratic term display a need to improve the model.

The N-plot of residuals lies between normal lines as shown in figure 3. It can be observed that the residuals are distributed normally. Some gaps between observations in the response probablestate that non-linear relationship exists between the factors and the response. The deviation of experiment 4 and 5 could as well be due to the irrelevant term seen in the regression coefficient.

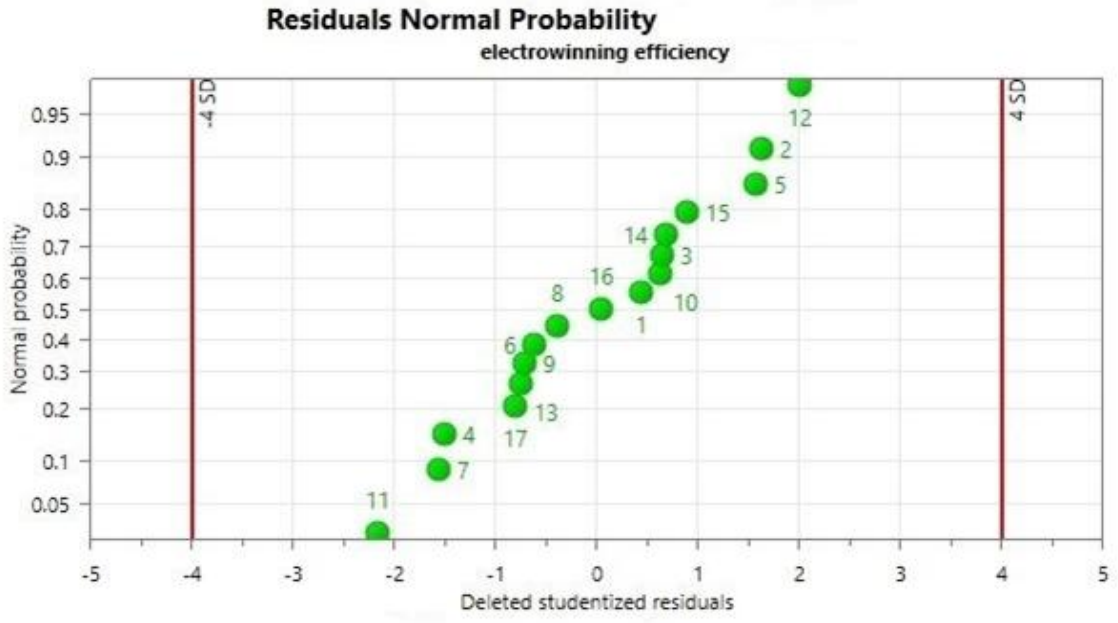

Figure 3: Normal probability graph for the response

Figure 4 shows the residuals plot against run order and it states that there is no systematic relationship between residual and the run order.

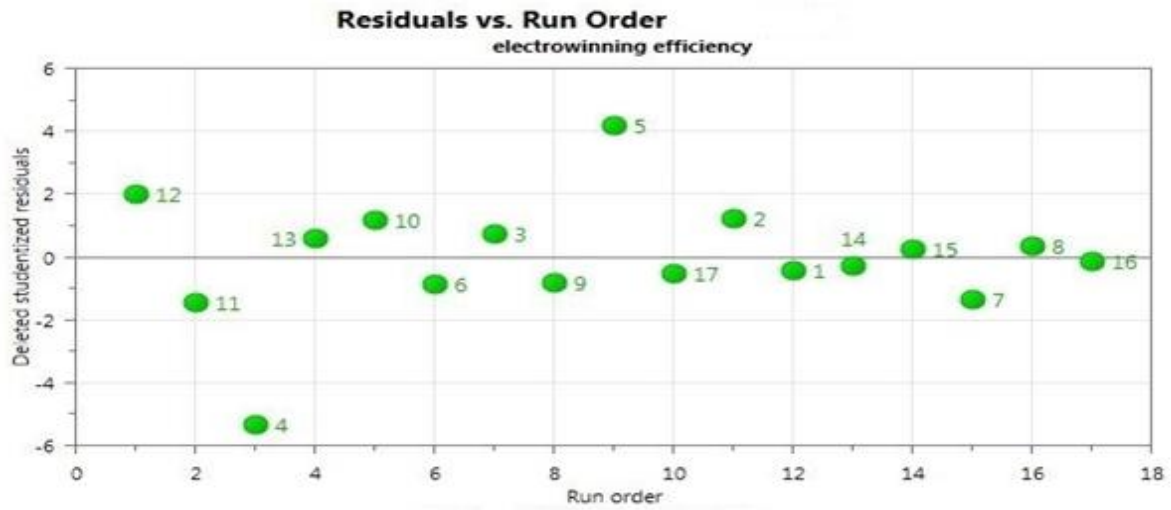

Figure 4: Residual versus run order for the response

The plot of the residuals against the predicted response is shown in figure 5. It can be observed that the plot is random with no patterns.

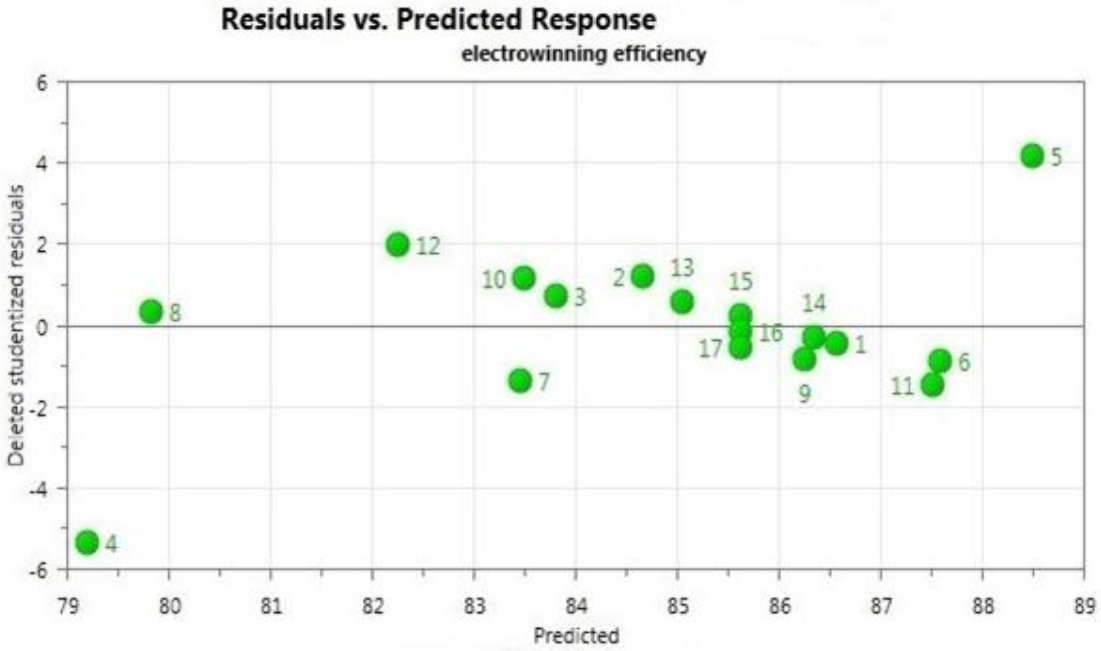

Figure 5: Plot of residuals against predicted response

In order to evaluate the fit, it is worthy to look at the observed against predicted values for the response. Figure 6 predicted the observed values very well and majority of the experiments are within the target efficiency.

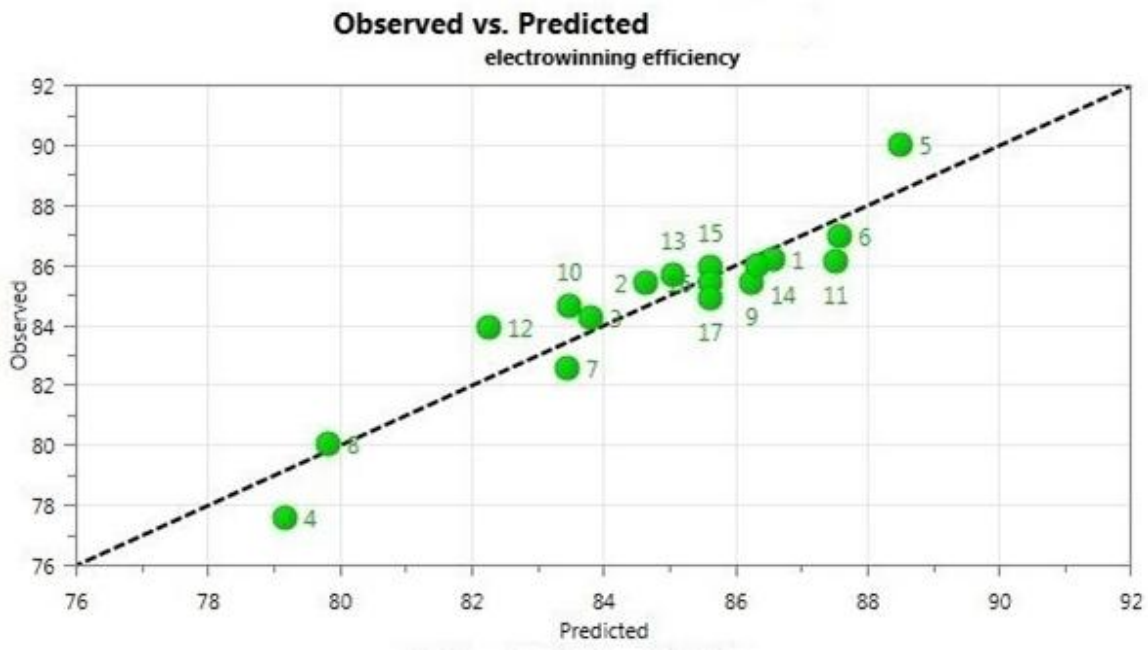

Figure 6: Graph of observed against predicted values for the response

The present statue of the model is the best possible fit and can be effectively used for explaining the relationship between the factors and response with a good reliability. However, the model is not a tractable tool in order to predict electrowinning efficiency for new series of data.The worst case scenario encountered for the predictive measure in figure 1 necessitate that the model needs to be improved.Logarithmic transformation is carried out for current density as detailed in table 5.

Table 5: Transformation factor table

Factor	Abbr.	Units	Type	Settings	Transformation	Precision
Temperature	temp	°C	Quantitative	45.94 to 53.88	None	0.199
Caustic Strength	cau	%	Quantitative	2.28 to 3.17	None	0.0223
Current density	cur	Am^{-2}	Quantitative	781.3 to 797.1	$10\log(C_1*Y + C_2)$	0.393

Where Y is the value of the current density and C_1 and C_2 are constants. The statistically non-significant term in the regression coefficient plot (Fig.2) is transformed. Thus the model was refined and simplified.The precision in table 5 tellshow close the measured values are measured reliably.

The summary of fit plot of the refined model is presented in figure 7.It can be seen from figure 7 that the Q^2 value has increased, and now amount to 0.556. The model show improved model validity of 0.7 as compared to the previous value of 0.4 in figure 1.

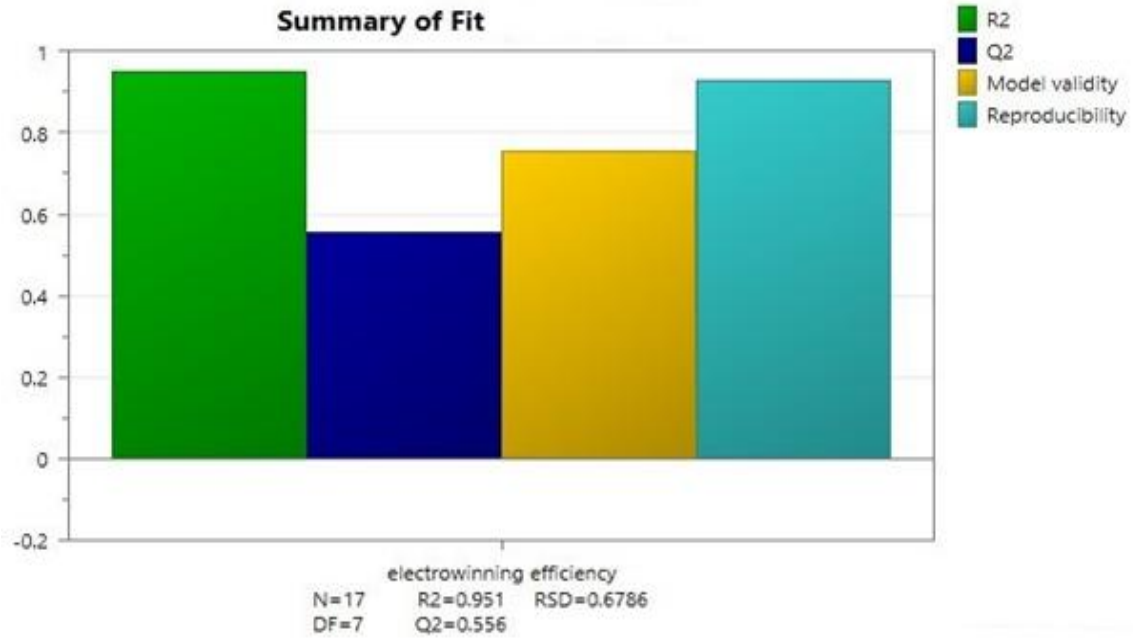

Figure 7: Summary of fit plot of refined model

The outcome of the normal probability plot of the residuals after refinement is shown in figure 8 and reveals that the model looks satisfactory as there were no outliers as seen earlier in figure 3.

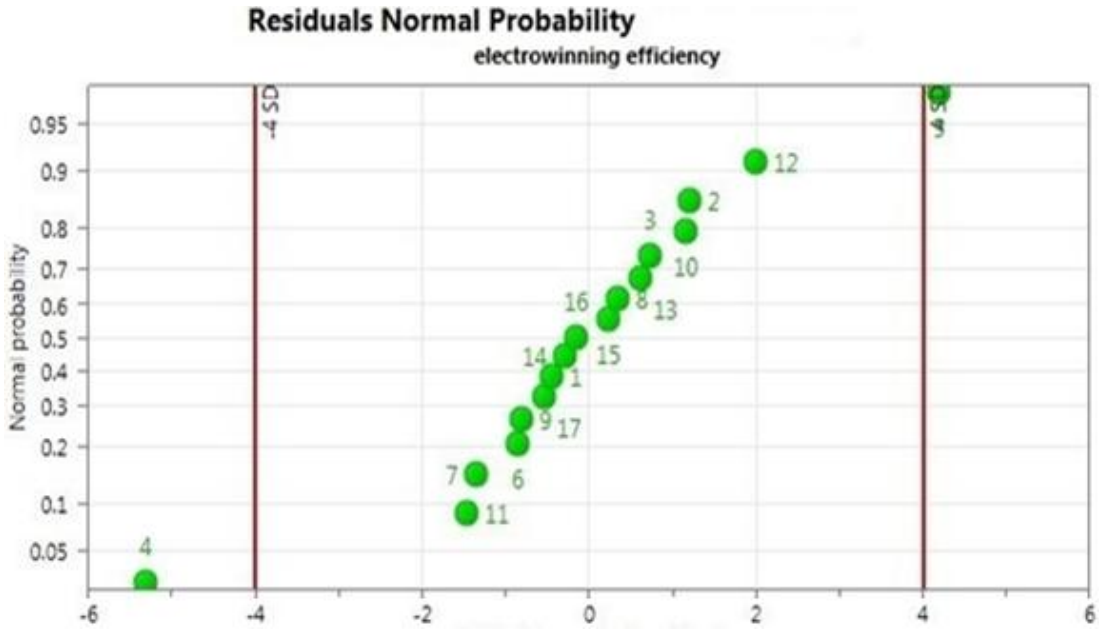

Figure 8: Normal probability plot of residuals after model refinement

The regression coefficient of the refined model was made in order to obtain information concerning how the input variables affect the response as shown in figure 9. The plot shows that temperature and caustic strength have a strong effect on the response.

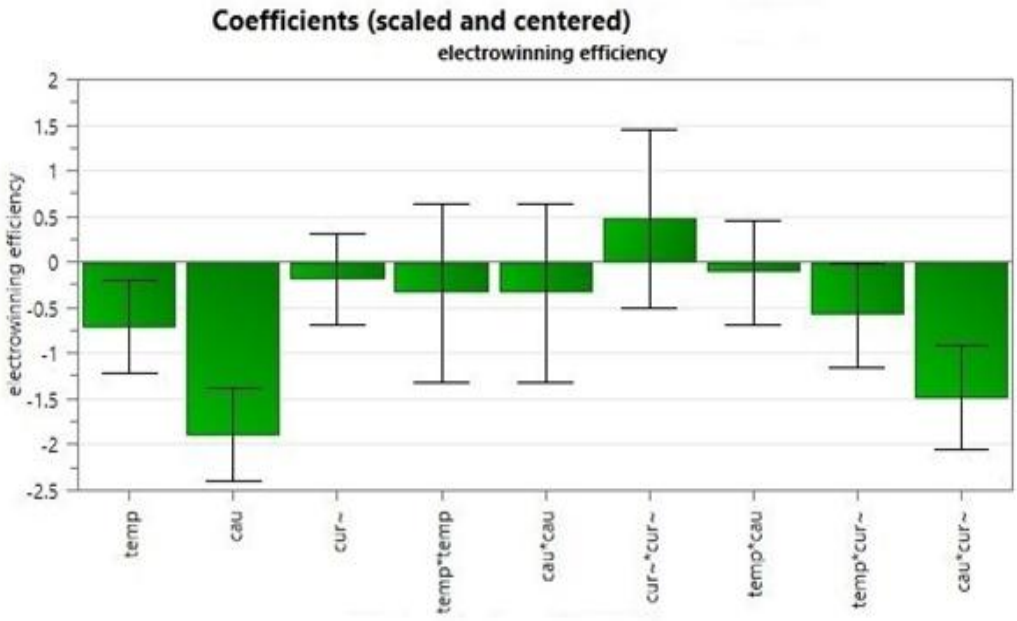

Figure 9: Regression coefficient plot for recovery of Au after model refinement

In addition the analysis of variance (ANOVA) of the refined model shown in table 6 reveals that the regression model is statistically significant with a 95% confidence level. The P-value for the regression is smaller than 0.05.

Table 6: Analysis of variance (ANOVA) for the quadratic m odel for the response variable (refined model)

Electro-winning efficiency	DF	SS	MS (variance)	F	P	SD
Total	17	123664	7274.37			
Constant	1	123598	123598			
Total corrected	16	66.2722	4.14201			2.03519
Regression	9	63.0487	7.00541	15.2125	0.001	2.64677
Residual	7	3.22353	0.460505			0.678605
Lack of fit	5	2.66173	0.532346	1.89513	0.380	0.3000272962
Pure error	2	0.561805	0.280903			0.5

From the ANOVA table 6, itcan be deduced that the lack of fit (model error) is of the samemagnitude as the replicate error for the response,because their P-values are greater than 0.05 at 95% confidence level. Therefore, the model has small error and good fitting power, meaning thatthe model shows no lack of fit.

Figure 10 shows that the observed response correlated well with the predicted values. Therefore, the model is considered as good and can be used for the predictions and optimization of the process.

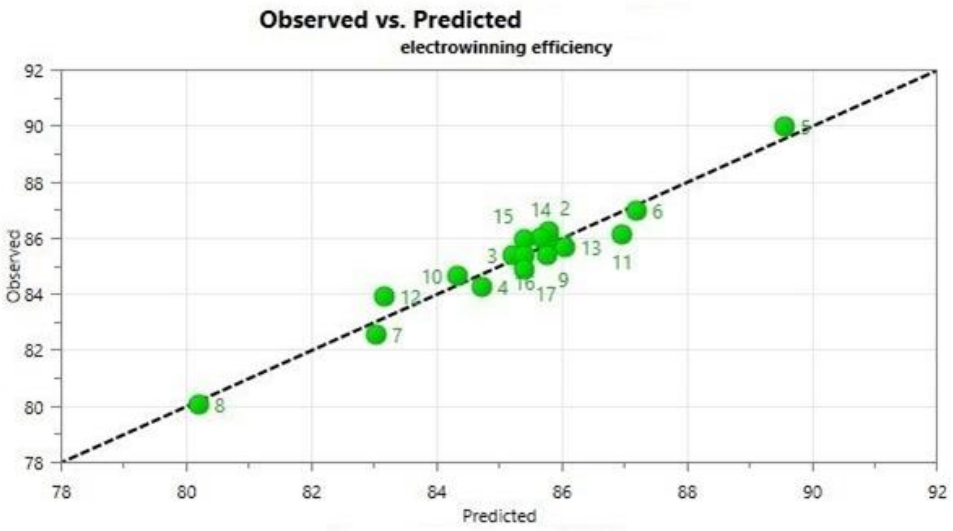

Figure 10: Relationship between observed and predicted values for response after model refinement

The regression model describing the relation of the response and the parameters investigatedafter transformation of the current density is given in table 7. The refined fitted model is given as

$$y = 85.384 - 0.713x_1 - 1.9x_2 - 0.190x_3 - 0.340\,x^2_1 - 0.335\,x^2_2 + 0.475\,x^2_3 - 0.117\,x_1x_2 - 0.587\,x_1x_3 - 1.485\,x_2x_3 \quad (4)$$

Table 7: Theestimated parameters of the refined model

Electrowinning efficiency	Coeff. SC	Std. Err.	P	Conf.int(±)
Constant	85.3841	0.290375	1.38913e-015	0.686637
Temperature	-0.712998	0.214594	0.0127205	0.50744
Caustic strength	-1.9	0.214594	4.74415e-005	0.50744
Current density	-0.190003	0.214594	0.405323	0.50744
temp*temp	-0.339661	0.414582	0.439623	0.980344
cau*cau	-0.334635	0.414582	0.446128	0.980344
cur*cur	0.475345	0.414582	0.289239	0.980344
temp*cau	-0.117496	0.239923	0.639302	0.567335
temp*cur	-0.587497	0.239923	0.0441922	0.567335
cau*cur	-1.485	0.239923	0.00044984	0.567335

The model equation (4) can be used with a good reliability to evaluate the main effects of the three independent factors on the response as well as inter-relation of both factors and response. A normalized coefficient of the refined model is shown in figure 11 is used to interpret the importance of the factors and their interactions on the response.

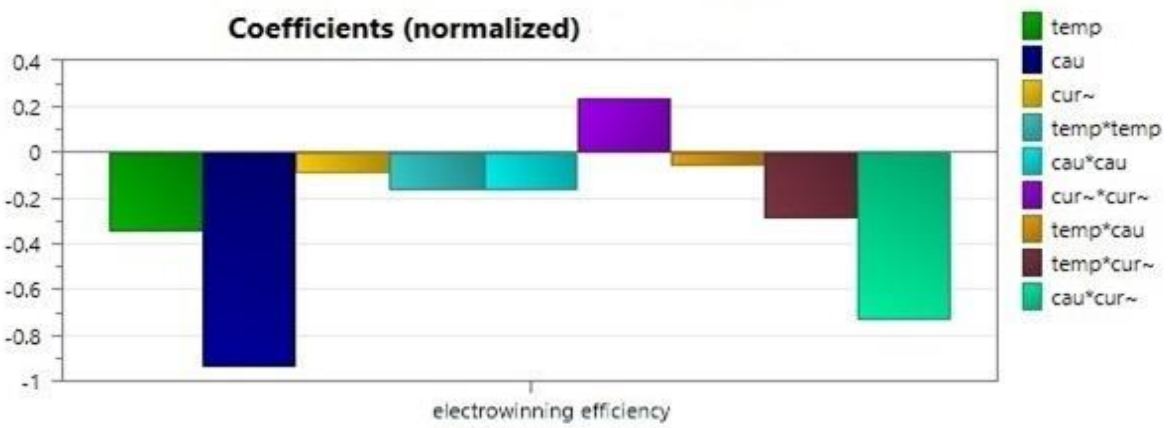

Figure 11: Normalized coefficients for the response

The effects of the factors areagain confirmed in figure 11. The cur*cur quadratic term shows a positive effect whiles the remaining quadratic terms shows negative effects. The interaction termsalso shows negative effect on electrowinning. The presence of squares and interaction terms in the regression equation confirms a quadratic behavior and non-linear combining effects of the factors.

3.2Elucidation of Data by Response Surface Methodology

Response surface plots were developed, which provide a better understanding of the effect of the experimental factors on the response variable. Figure 12, 13 and 14 show the contour plots at various current densities where electrowinning efficiency is represented by varying simultaneously the temperature from 45.94 to 53.88°C and caustic strength from 2.28 to 3.17%. From figure 12, 13 and 14, it can be observed that gold electrowinning efficiency increases with the decrease of both caustic strength and temperature. This could be that the effect of temperature and caustic strength on gold electrowinning efficiency can be explained by the fact that the gold ore is free milling in nature**.**Perhaps the anode plate resists corrosion by sodium hydroxide at mild temperatures [19].

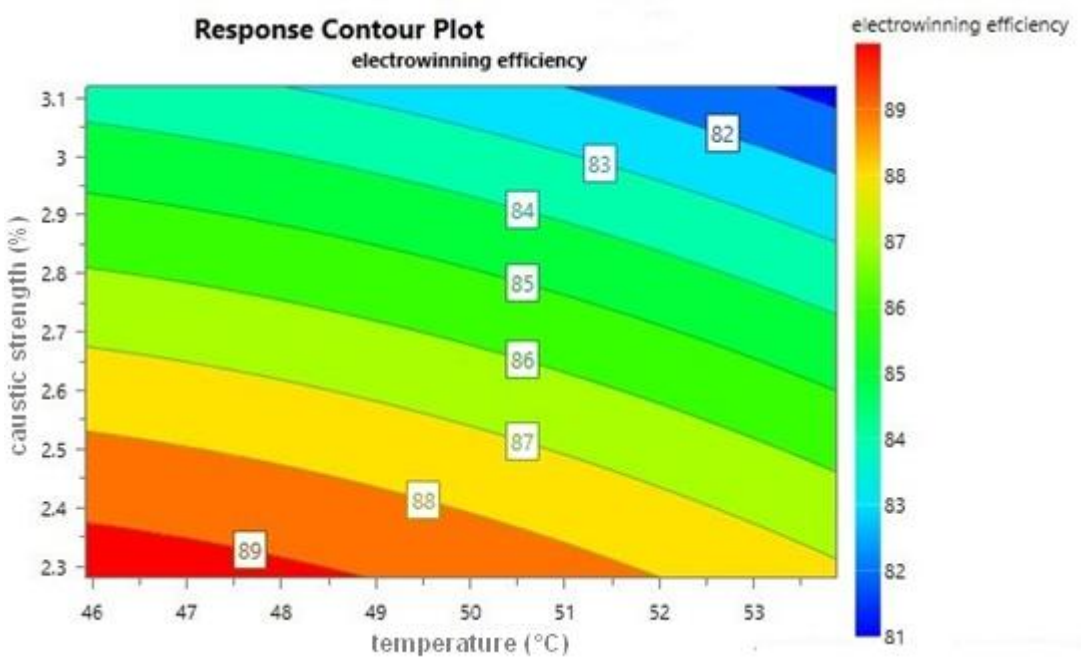

Figure 12: Contour plot of electrowinning efficiency showing interaction between caustic strength and temperature at current density of 781.35 Am^{-2}

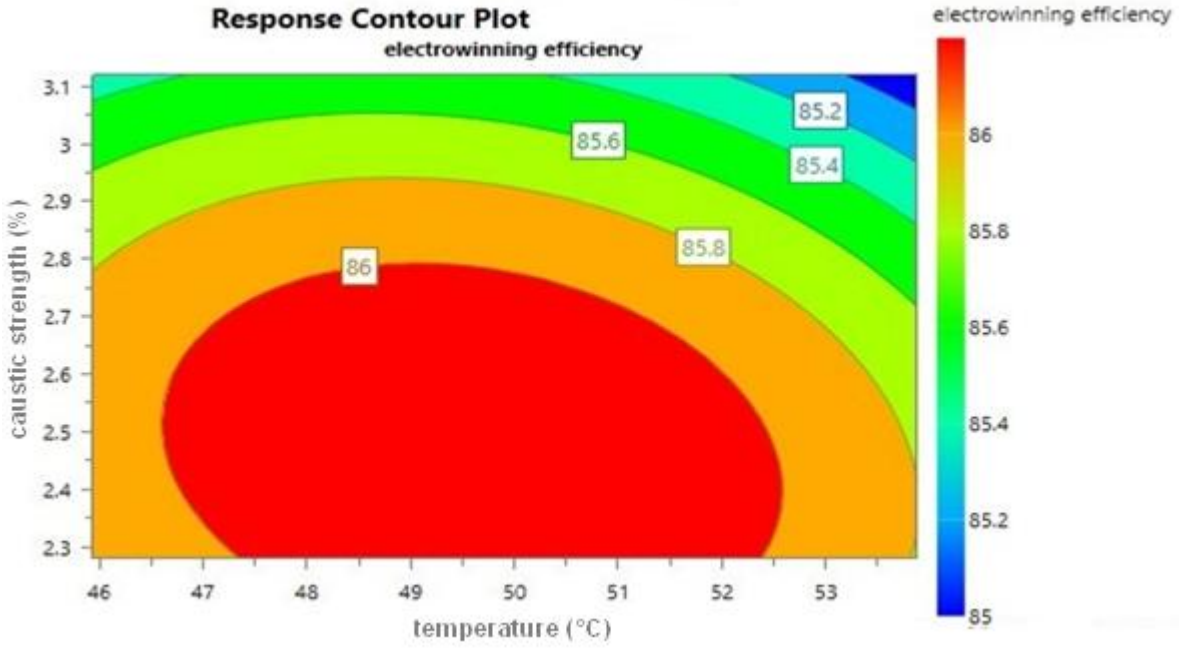

Figure 13: Contour plot of electrowinning efficiency showing interaction between caustic strength and temperature at current density of 788.24 Am^{-2}

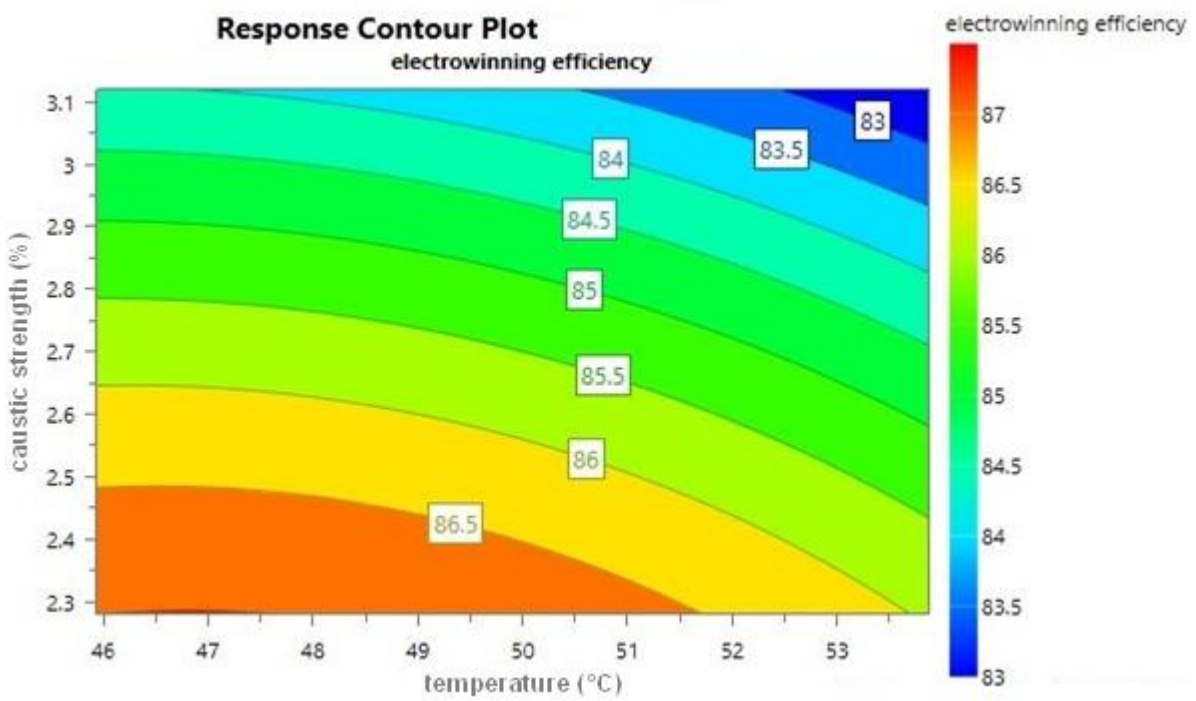

Figure 14: Contour plot of electrowinning efficiency showing interaction between caustic strength and temperature at current density of 797.06 Am^{-2}

Also, gold electrowinning efficiency increases up to a limiting amount with high current density as shown in figure 12, 13 and 14 respectively. This could be that current is consumed by other side reactions such as the evolution of hydrogen and the deposition of other metals such as copper, and does not contribute to further gold deposition. Similar trend was observed by Costello (2005) in electrowinning of gold ore[20].The corners in the left bottom of the above graphs represent a minimum amount of the caustic strength and temperature.

3.3 Model Validation

In order to test the validity of the model with respects to the response variable of gold electrowinning efficiency, a separate simulationtest was performed at the conditions predicted by the model, as shown in table 8. The results of table 8 indicate a close agreement with the values predicted by the model. Therefore, the model from a response surface methodology is considered to be exact and consistent, for predicting the electrowinning efficiency of gold from solution.

Table 8: Output of optimized parameter

Temperature (°C)	Caustic strength (%)	Current density (Am^{-2})	Electrowinning efficiency (%)	Iterations	Log (D)	DPMO	Cpk (electrowinning efficiency)
46.734	2.369	784.492	86.917	0	-4.1062	5600	0.979366
46.999	2.458	786.324	86.953	11	-4.5945	2700	1.13777
48.322	2.399	782.921	86.648	7	-2.8537	2400	1.12238
53.317	2.311	786.649	85.773	22	-1.7693	2000	1.17786
47.131	2.636	796.275	87.198	8	-2.3590	10900	0.836248
46.999	2.769	796.798	86.459	7	-2.4810	5100	0.962633
53.086	2.369	792.347	86.623	0	-2.7935	4400	1.06795
53.579	2.431	796.958	86.826	19	-3.4645	11000	0.805516
49.910	2.725	792.347	85.887	0	-1.8545	0	1.79263
46.734	2.280	784.492	86.997	0	-6.9419	12700	0.808779
46.734	2.725	792.347	86.433	0	-2.4394	2000	1.24856
49.910	2.725	797.060	86.328	0	-2.2924	5400	0.966049

3.4 Process Optimization

Optimization of the factors affecting the electrowinning process can be carried out depending on the outcome from the process. For high electrowinning efficiency of gold, the option is for minimum caustic strength and temperature as displayed in figure 12, 13 and 14.Conversely, for practical and economic reasons, low production cost due toreagent cost and less elution problems due to low caustic strength are required. An agreement must be made among these factors in order to have desirable electrowinning conditions. Literature available indicates that the elution of all metal cyanide species could be improved by adding less hydroxide to the pre-treatment.However, too much caustic strength would result in elution of base metals from the carbon, which will consequently lower the electrowinning efficiency

and fineness of the gold bullion [1].Therefore, a level conditionmust be found based on the mineralogy of the ore, solventand also energyrequirement so as not to create problems during onward processing.

The three factors considered for the study affect the economics of the process in various ways.For high current density,the electric field increases implying that it increases the gold deposition on the surface of the cathode. Temperature influences many parameters in solutions such as dissolved oxygen, activity coefficients, oxidation rates and corrosion. For temperature above 45 to 50°Cthe effect of the ions decline dramaticallyand thereby enhance gold deposition. In practice, caustic is added to the pre-treatment, but mainly to stabilize the cyanide and to improve conductivity to enhance cell performance. Therefore, the desirable condition for gold electrowinning are caustic strength of 2.28 - 2.43%, current density of 784.50– 797.00 Am^{-2} and temperature of 46.70 – 49.50°C to obtain an electrowinning efficiency of about 86.9% (close to the target value of 87%). The merit of lesser base metals is the higher fineness of the gold bullion and hencereduction of the refining costs. This is due to the relationship between the fineness and the refining costs. Another potential means of reducingthe base metals would be to add cyanide and caustic after the elution.This will allow to increase the cyanide strength and run the electrowinning at the recommended condition.This could be interesting to explore in the future.

4. Conclusions

Gold electrowinning is relatively a simple process. However, using a proper analytical method to accurately evaluate the outcome of the process is a much challenging step. The use of mathematical modelling in optimization of the process has been ascertained.From the experiment run on the plant scale, three findings could be highlighted.

1) Optimization of influential factors in gold electrowinning is conducted using response surface methodology-central composite face-centered design (RSM-CCF).
2) The three factors considered affect the electrowinning process in various ways. The caustic strength was found to have the most influential effect with current density being less significant on electrowinning efficiency. Temperature along with the other two factors had a negative effect on electrowinning efficiency.
3) A strong mathematical model with no lack of fit was developed and the validity of the model evaluated experimentally. The result shows that the model is reliable and accurate for predicting the electrowinning efficiency.

5. Acknowledgement

The researcher would like to appreciate the management of Goldfields Ghana Limitedthe mine where data was obtained for the study.

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