# STATISTICAL ANALYSIS AND OPTIMISATION OF ZINC DUST PURIFICATION OF ZINC SULPHATE ELECTROLYTE USING DESIGN-EXPERT SOFTWARE



#### **EXPLORATORY PROJECT**

IN

## METALLURGICAL ENGINEERING

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lot. Than	aks a lot, sir!
lot. Than	

# **CERTIFICATE**

This is to certify that the exploratory project work entitled "Statistical Analysis and Optimisation of Zinc Dust Purification of Zinc Sulphate Electrolyte using Design-Expert Software" embodies the bonafide work carried out by

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## 1. ABSTRACT

To analyse and optimise the effect of various parameters, based on the data set obtained by the experiment, on the result using Desing-Expert software. We require a tool that can comprehend the data and do a statistical operation, viz. to investigate a hypothesis by the f-value method, to provide graph and 3D representation, and so forth. We deal with a set of data while performing the cementation experiment such as time, concentrations, and so forth. To analyse these data, the experimental data set is fed in the software, and analysis and optimisation are accomplished.

## 2. INTRODUCTION

Design-Expert software is used in many streams such as industry and educational institute because of impactful statistical tools inherited into it. Design of experiments is a method by which we make purposeful changes to input factors of our process in order to observe the effects on the output. This software can and have been performed in virtually every industry on the planet; agriculture, chemical, pharmaceutical, electronics, automotive, hard goods manufacturing, etc. Service industries have also benefited by obtaining data from their process and analyzing it appropriately.

We have made use of this software for analysis of data set obtain from Zinc (Zn) dust purification of the solution, i.e. cementation. Cementation process for extraction of metals from its fused salt solution has widely accepted because of its simplicity and ease of production. The metal with higher electronegative reduction potential is used to precipitate metal present in the solution.

In this particular experiment, we have used Zn dust with different mesh size to precipitate impurity metals such as Copper (Cu), Nickel (Ni), Cadmium (Cd) and Cobalt (Co) from Zinc Sulphate solution to get purified Zinc Sulphate solution. We have used Zinc dust of mesh size of 100, 200 and 300, and its effect on the concentration of impurities metals with time. All impurity metals' concentration is determined in each interval with the help of UV Spectrophotometry. A list of data tables came as the response of experiment, and these set of data is fed into the Design-Expert software. First, we proceeded for Copper, and the series of analysis is done in the software and final optimised parameters are accomplished. Similarly, analysis and optimisation is carried out for all impurity metal.

# 3.1 A GLIMPSE ON DESIGN EXPERT SOFTWARE

The software is a based on complex statistical mathematics. It encompasses many impactful statistical tools, such as

- Two-level factorial screening designs: Identify the vital factors that affect the process so that one can make breakthrough improvements.
- General factorial studies: Discover the best combination of categorical factors, such as source versus the type of raw material supply.
- Response surface methods (RSM): Find the optimal process settings to achieve peak performance.

Design-Expert program offers rotatable 3D plots to easily view response surfaces from all angles. By using mouse we can set flags and explore the contours on interactive 2D graphs.

## 3.2 CEMENTATION USING ZINC DUST

In Cementation, a more reactive metal dissolves in a solution to precipitate a less reactive metal from the solution. Here the electrochemical series can be used to determine the reactivity of a metal.

Zinc can be added to a solution consisting metals such as Copper, Cobalt, Cadmium and Nickel which are having lower reactivity than Zinc to precipitate these metals. Zinc dissolves because the conversion of the metal to metal ions involves a significant free energy decrease than that in the case of metals having higher electropositive reduction potential. It should, however, be noted that elements in the electrochemical series have been arranged for a specific temperature, 25°C and the solution concentration.

Here, for the purification of Zinc sulphate solution from impurity metals like Copper, Cobalt, Cadmium and Nickel, cementation process is carried out using Zinc Dust.

# 4. Cementation of Copper

# 4.1 Experimental Dataset

Precipitation of Copper metal for different percentage excess of amount of Zinc Dust, viz. 100, 200, 300 and 400 with different mesh size, i.e. -100, -200 and -300 were studied. The concentration of the impurity metal viz. Copper is measured in each interval with UV Spectrophotometry, and a list of data table is obtained.

#### · 100% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>				
No.		Ni (ppm)				
	(min)	Mesh	Mesh	Mesh		
		-100	-200	-300		
1	0	21	21	21		
2	2	18.7	18.5	17.8		
3	5	17.8	17.3	16.2		
4	10	17	16.3	15.7		
5	15	16.3	14.8	13.7		
6	30	13.2	12.8	12.3		
7	45	12.5	8.9	8.4		
8	60	7	6.6	6.5		
9	90	6.1	5.6	4.8		
10	120	5	3.48	3.2		

#### · 200% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>				
No.		Cu (ppm)				
	(min)	Mesh	Mesh	Mesh		
		-100	-200	-300		
1	0	495	495	495		
2	2	420	401	215		
3	5	419	390	95		
4	10	408	342	33		
5	15	313	294	3.5		
6	30	282	40	0		
7	45	73	5.6	0		
8	60	61	0	0		
9	90	16	0	0		
10	120	0	0	0		

#### ·300% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>				
No.		Cu (ppm)				
	(min)	Mesh	Mesh	Mesh		
		-100	-200	-300		
1	0	495	495	495		
2	2	366	352	137		
3	5	364	331	44.5		
4	10	350	288	0		
5	15	252	242	0		
6	30	207	10.5	0		
7	45	49.5	0	0		
8	60	38	0	0		
9	90	0	0	0		
10	120	0	0	0		

#### ·400% Excess Zinc Dust

Sl. No.	Time	Concentration of Cu (ppm)				
	(min)	Mesh -100	Mesh -200	Mesh -300		
1	0	495	495	495		
2	2	415	396	156		
3	5	413	369	48		
4	10	406	322	12		
5	15	298	255	0		
6	30	268	32	0		
7	45	55	0	0		
8	60	47	0	0		
9	90	0	0	0		
10	120	0	0	0		

# 4.2 Analysis using Design-Expert

Three parameters taken into consideration during the Zinc dust purification experiment and their corresponding number of levels are as shown in table.

Parameters	Number of levels
Time	10
Percentage excess Zn dust	4
Size of Zinc dust	3

Multilevel Categoric Design for purification of Copper was made and fed the data into it.

## 4.2.1 Fit Summary

Linear, 2FI (2-Factorial interaction), Quadratic, Cubic and Quartic model can be generated from the data fed into the software. p-values and R<sup>2</sup> values are considered to find out the significant terms to be considered for the model and thus the best model for the analysis of the experiment is determined.

Source	Sequential p-value	Adjusted R <sup>2</sup>	Predicted R <sup>2</sup>	
Linear	< 0.0001	0.6115	0.5906	
2FI	0.0109	0.6445	0.6058	
Quadratic	< 0.0001	0.8274	0.8091	
Cubic	0.0005	0.8601	0.8352	Suggested
Quartic	0.0382	0.8748	0.8307	

From the above table, we can see that the Cubic model is the suggested model for Analysis based on the p-values and R<sup>2</sup> values.

#### 4.2.2 ANOVA for Cubic model

Source	F-value	p-value
Model	35.83	< 0.0001
A-Time	418.04	< 0.0001
B-Percentage Excess of Zinc	3.67	0.0584
C-Size of Zinc Dust	54.73	< 0.0001
AB	0.8983	0.3456
AC	19.49	< 0.0001
BC	0.0108	0.9893
$A^2$	144.60	< 0.0001
$B^2$	1.62	0.2065
ABC	0.1146	0.8919
A <sup>2</sup> B	0.0013	0.9710
A <sup>2</sup> C	2.64	0.0761
$AB^2$	0.4684	0.4953
B <sup>2</sup> C	0.0950	0.9094
$A^3$	28.32	< 0.0001
$B^3$	0.7508	0.3884

The **Model F-value** of 35.83 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

**P-values** less than 0.0500 indicate model terms are significant. In this case A, C, AC, A<sup>2</sup>, A<sup>3</sup> are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

#### **Fit Statistics**

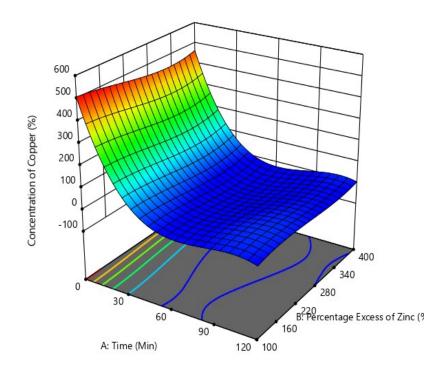
Std. Dev.	70.64	R <sup>2</sup>	0.8848
Mean	171.70	Adjusted R <sup>2</sup>	0.8601
C.V. %	41.14	Predicted R <sup>2</sup>	0.8352
		Adeq Precision	19.7480

The **Predicted R**<sup>2</sup> of 0.8352 is in reasonable agreement with the **Adjusted R**<sup>2</sup> of 0.8601; i.e. the difference is less than 0.2.

**Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 19.748 indicates an adequate signal. This model can be used to navigate the design space.

## 4.2.3 Model Graphs: 3D Surface





# Design-Expert® Software Factor Coding: Actual

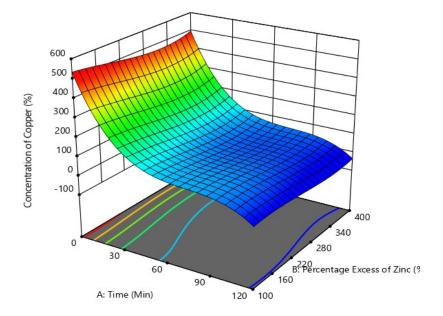
#### Concentration of Copper (%)



X1 = A: Time X2 = B: Percentage Excess of Zinc

#### **Actual Factor**

C: Size of Zinc Dust = -100



# **Design-Expert® Software** Factor Coding: Actual

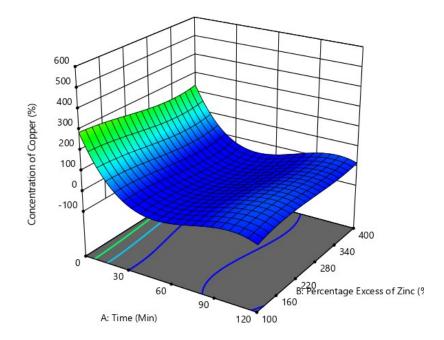
#### Concentration of Copper (%)



X1 = A: Time X2 = B: Percentage Excess of Zinc

#### **Actual Factor**

C: Size of Zinc Dust = -300

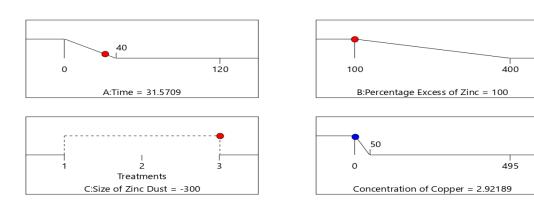


# 4.3 Numerical optimization for the Experiment

## Copper

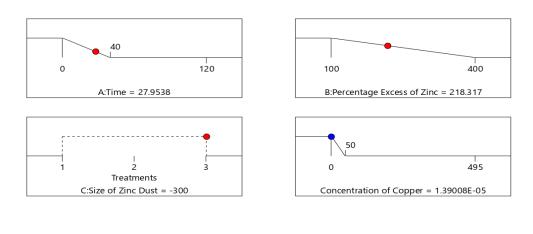
Our objective is to minimize the time, percentage excess Zinc dust and concentration of Copper in the solution (0-50 ppm). We come up with three solutions.

#### Solution 1:



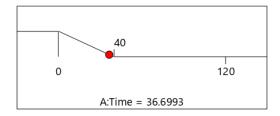
Desirability = 0.612 Solution 1 out of 3

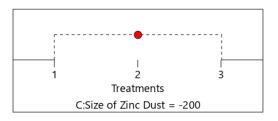
#### Solution 2:

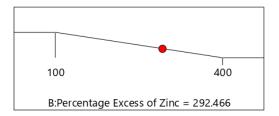


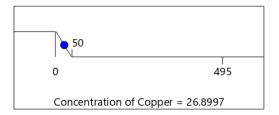
Desirability = 0.600 Solution 2 out of 3

## Solution 3:









Desirability = 0.255 Solution 3 out of 3

# 5. Purification of Cobalt

## **5.1 Experimental Dataset**

Precipitation of Copper metal for different percentage excess of amount of Zinc Dust, viz. 100, 200, 300 and 400 with different mesh size, i.e. -100, -200 and -300 were studied. The concentration of the impurity metal viz. Copper is measured in each interval with UV Spectrophotometry, and a list of data table is obtained.

## • 100% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>				
No.		Co (ppm)				
	(min)	-100	-200	-300		
1	0	22	22	22		
2	2	21.8	21	20.5		
3	5	21	17.6	17.6		
4	10	19.4	16	15.2		
5	15	19	15.6	15		
6	30	18.2	15.1	14.8		
7	45	18.7	16	16		
8	60	21	17.8	17		
9	90	21.6	20.4	18.9		
10	120	22.4	21	19.4		

#### · 200% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>			
No.		Co (ppm)			
	(min)	-100	-200	-300	
1	0	22	22	22	
2	2	21.3	20.7	20.1	
3	5	20	17.9	17.9	
4	10	19.2	19	18.3	
5	15	18	16.7	16.1	
6	30	17.2	16.2	15.8	
7	45	16.7	15.7	15.3	
8	60	17	16.8	16.5	
9	90	18.4	17.8	17.3	
10	120	20.7	18.4	18	

#### · 300% Excess Zinc Dust

Sl.	Time	Concentration of			
No.		Co (ppm)			
	(min)	-100	-200	-300	
1	0	22	22	22	
2	2	21.7	21	19.8	
3	5	21	18.6	18.6	
4	10	18.4	17.3	17	
5	15	17.2	16.8	16	
6	30	16.7	16.5	15.9	
7	45	16.5	16.3	15.6	
8	60	17	16.5	16	
9	90	19.4	20	18.1	
10	120	20	19	18.4	

#### · 400% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>				
No.		Co (ppm)				
	(min)	-100	-200	-300		
1	0	22	22	22		
2	2	19.6	19.1	18.3		
3	5	19	17.3	17.2		
4	10	17.6	16	15.2		
5	15	16	15.6	15		
6	30	15.8	15.8	14.8		
7	45	15.3	15.2	15		
8	60	16.4	16	15.7		
9	90	17.8	17.1	16.4		
10	120	18.5	17.6	17.2		

## 5.2 Analysis using Design-Expert

Linear, 2FI (2-Factorial interaction), Quadratic, Cubic and Quartic model can be generated from the data fed into the software. p-values and R<sup>2</sup> values are considered to find out the significant terms to be considered for the model and thus the best model for the analysis of the experiment is determined.

## **5.2.1** Fit Summary

Source	Sequential p-value	Adjusted R <sup>2</sup>	Predicted R <sup>2</sup>	
Linear	0.0048	0.0902	0.0421	
2FI	0.5896	0.0801	-0.0268	
Quadratic	< 0.0001	0.5395	0.4812	
Cubic	< 0.0001	0.8185	0.7830	Suggested
Quartic	< 0.0001	0.8834	0.8220	

From the above table, we can see that the Cubic model is the suggested model for Analysis based on the p-values and R<sup>2</sup> values.

#### 5.2.2 ANOVA for Cubic model

Source	F-value	p-value
Model	26.55	< 0.0001
A-Time	8.12	0.0053
B-Percentage Excess of Zinc	9.19	0.0031
C-Size of Zinc Dust	30.95	< 0.0001
AB	8.19	0.0052
AC	0.3910	0.6774
ВС	4.99	0.0087
A <sup>2</sup>	260.55	< 0.0001
B <sup>2</sup>	22.92	< 0.0001
ABC	0.1518	0.8593
A <sup>2</sup> B	0.1256	0.7238
A <sup>2</sup> C	0.0127	0.9874
AB <sup>2</sup>	10.24	0.0018
B <sup>2</sup> C	2.14	0.1237
A <sup>3</sup>	147.78	< 0.0001
B <sup>3</sup>	13.26	0.0004

The **Model F-value** of 26.55 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

**P-values** less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, BC, A<sup>2</sup>, B<sup>2</sup>, AB<sup>2</sup>, A<sup>3</sup>, B<sup>3</sup> are significant model terms. Values greater than 0.1000 indicate the model terms are not significant

#### **Fit Statistics**

Std. Dev.	0.9507	R <sup>2</sup>	0.8505
Mean	18.17	Adjusted R <sup>2</sup>	0.8185
C.V. %	5.23	Predicted R <sup>2</sup>	0.7830
		<b>Adeq Precision</b>	21.4508

The **Predicted R**<sup>2</sup> of 0.7830 is in reasonable agreement with the **Adjusted R**<sup>2</sup> of 0.8185; i.e. the difference is less than 0.2.

**Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 21.451 indicates an adequate signal. This model can be used to navigate the design space.

# 5.2.3 Model Graphs: 3D Surface

# **Design-Expert® Software** Factor Coding: Actual

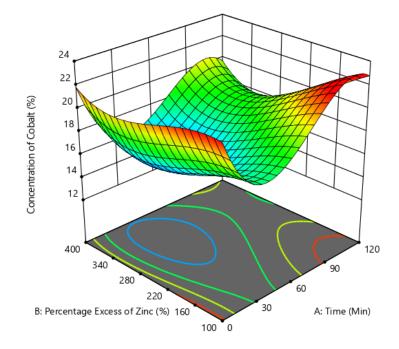
#### Concentration of Cobalt (%)

14.78 22.4

X1 = A: Time X2 = B: Percentage Excess of Zinc

#### **Actual Factor**

C: Size of Zinc Dust = -100



#### Design-Expert® Software

Factor Coding: Actual

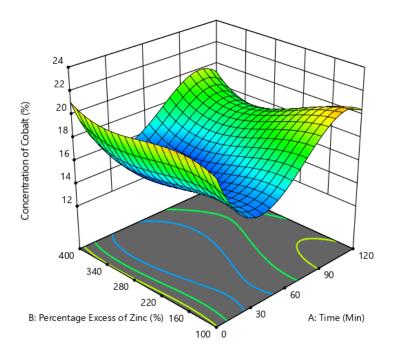
#### Concentration of Cobalt (%)

14.78

X1 = A: Time X2 = B: Percentage Excess of Zinc

#### **Actual Factor**

C: Size of Zinc Dust = -200



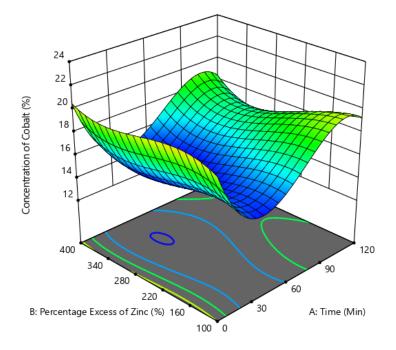
# **Design-Expert® Software** Factor Coding: Actual

#### Concentration of Cobalt (%)

14.78 22.4

X1 = A: Time X2 = B: Percentage Excess of Zinc

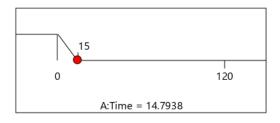
Actual Factor C: Size of Zinc Dust = -300

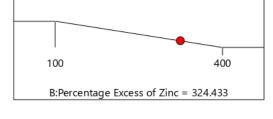


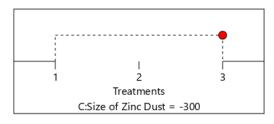
# 5.3 Numerical optimization for the Experiment

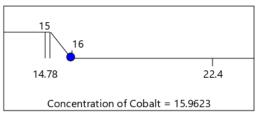
Optimisation of parameters for Cobalt purification considering criteria as to minimize the time, percentage excess Zinc dust and limiting Cobalt Concentration (15-16 ppm) was done, and the following solution was obtained.

#### Solution:









Desirability = 0.051 Solution 1 out of 1

# 6. Purification of Cadmium

# **6.1 Experimental Dataset**

Precipitation of Cadmium metal for different percentage excess of amount of Zinc Dust, viz. 100, 200, 300 and 400 with different mesh size, i.e. -100, -200 and -300 were studied. The concentration of the impurity metal viz. Cadmium is measured in each interval with UV Spectrophotometry, and a list of data table is obtained.

#### • 100% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>				
No.		Cd (ppm)				
	(min)	Mesh	Mesh	Mesh		
		-100	-200	-300		
1	0	400	400	400		
2	2	397	362	240		
3	5	350	331	123		
4	10	294	290	53.9		
5	15	213	178	28.3		
6	30	168	49	23		
7	45	103	26	17		
8	60	43.8	25	16.4		
9	90	37	23	13		
10	120	25.2	20	11		

#### · 200% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>					
No.			Cd (ppm)				
	(min)	Mesh	Mesh	Mesh			
		-100	-200	-300			
1	0	400	400	400			
2	2	392	359	239			
3	5	345	327	120			
4	10	288	286	52.8			
5	15	208	172	16.4			
6	30	157	42	19			
7	45	97	23	11			
8	60	40.2	21	9			
9	90	36	20	15			
10	120	21	18	12.9			

#### · 300% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>				
No.			Cd (ppm)			
	(min)	Mesh	Mesh	Mesh		
		-100	-200	-300		
1	0	400	400	400		
2	2	390	353	238		
3	5	339	320	116		
4	10	282	282	49		
5	15	200	165	15.5		
6	30	147	38.5	15		
7	45	89	18	13		
8	60	36.5	15	10		
9	90	31.5	13	9.5		
10	120	18	11	8.6		

#### • 400% Excess Zinc Dust

Sl.	Time	<b>Concentration of</b>				
No.		Cd (ppm)				
	(min)	Mesh	Mesh	Mesh		
		-100	-200	-300		
1	0	400	400	400		
2	2	392	357	238		
3	5	342	325	118		
4	10	286	284	51		
5	15	205	170	17		
6	30	152	40	16		
7	45	93	24	12		
8	60	38.5	19	11.4		
9	90	34	18	9.2		
10	120	21	14	8.6		

## 6.2 Analysis using Design-Expert

## **6.2.1 Fit Summary**

Linear, 2FI (2-Factorial interaction), Quadratic, Cubic and Quartic model can be generated from the data fed into the software. p-values and R<sup>2</sup> values are considered to find out the significant terms to be considered for the model and thus the best model for the analysis of the experiment is determined.

Source	Sequential p-value	Adjusted R <sup>2</sup>	Predicted R <sup>2</sup>	
Linear	< 0.0001	0.5993	0.5781	
2FI	0.1371	0.6113	0.5690	
Quadratic	< 0.0001	0.8389	0.8214	
Cubic	< 0.0001	0.9027	0.8861	Suggested
Quartic	0.0002	0.9268	0.9039	

From the above table, we can see that the Cubic model is the suggested model for Analysis based on the p-values and R<sup>2</sup> values.

#### 6.2.2 ANOVA for Cubic model

Source	F-value	p-value
Model	53.59	< 0.0001
A-Time	636.19	< 0.0001
B-Percentage Excess of Zinc	0.2120	0.6462
C-Size of Zinc Dust	56.59	< 0.0001
AB	0.0055	0.9410
AC	17.12	< 0.0001
BC	0.0025	0.9976
A <sup>2</sup>	260.29	< 0.0001
B <sup>2</sup>	0.3228	0.5713
ABC	0.0024	0.9976
A <sup>2</sup> B	0.0273	0.8690
A <sup>2</sup> C	2.56	0.0825
AB <sup>2</sup>	0.0455	0.8316
B <sup>2</sup> C	0.0612	0.9406
A <sup>3</sup>	75.57	< 0.0001
B <sup>3</sup>	0.0051	0.9431

The **Model F-value** of 53.59 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

**P-values** less than 0.0500 indicate model terms are significant. In this case A, C, AC, A<sup>2</sup>, A<sup>3</sup> are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

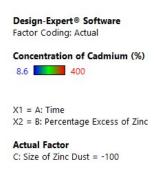
#### **Fit Statistics**

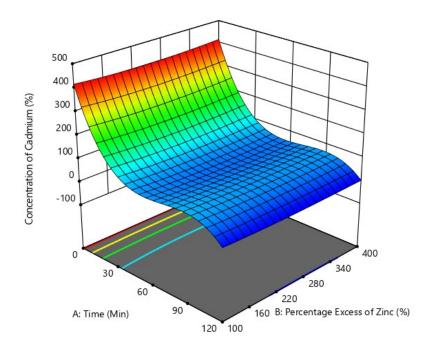
Std. Dev.	45.97	R <sup>2</sup>	0.9199
Mean	150.23	Adjusted R <sup>2</sup>	0.9027
C.V. %	30.60	Predicted R <sup>2</sup>	0.8861
		Adeq Precision	22.8028

The **Predicted R<sup>2</sup>** of 0.8861 is in reasonable agreement with the **Adjusted R<sup>2</sup>** of 0.9027; i.e. the difference is less than 0.2.

**Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 22.803 indicates an adequate signal. This model can be used to navigate the design space.

## 6.2.3 Model Graphs: 3D Surface





# Design-Expert® Software Factor Coding: Actual

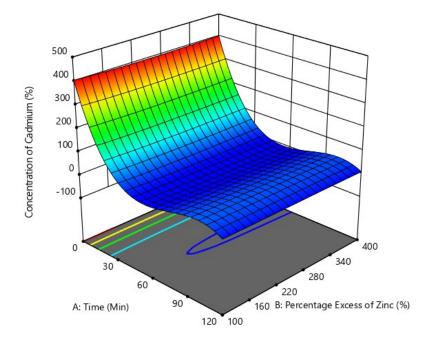
#### Concentration of Cadmium (%)



X1 = A: Time X2 = B: Percentage Excess of Zinc

#### **Actual Factor**

C: Size of Zinc Dust = -200



# **Design-Expert® Software** Factor Coding: Actual

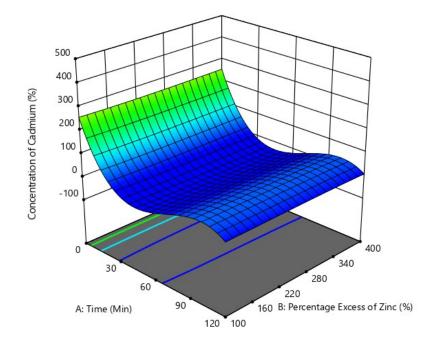
#### Concentration of Cadmium (%)

8.6

X1 = A: Time X2 = B: Percentage Excess of Zinc

#### Actual Factor

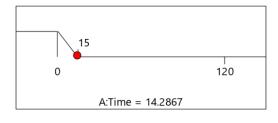
C: Size of Zinc Dust = -300

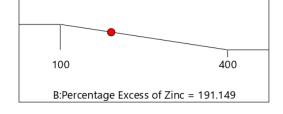


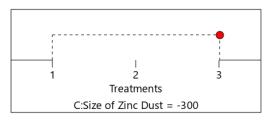
# 6.3 Numerical optimization for the Experiment

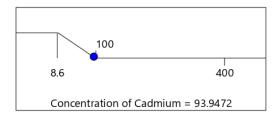
Optimisation of parameters for Cadmium purification considering criteria as to minimize the time, percentage excess Zinc dust and limiting Cadmium Concentration (8.6-100 ppm) was done, and the following solution was obtained.

#### Solution:









Desirability = 0.130 Solution 1 out of 1

# 7. Purification of Nickel

## 7.1 Experimental Dataset

Precipitation of Nickel metal for different percentage excess of amount of Zinc Dust, viz. 100, 200, 300 and 400 with different mesh size, i.e. -100, -200 and -300 were studied. The concentration of the impurity metal viz. Nickel is measured in each interval with UV Spectrophotometry, and a list of data table is obtained

#### · 100% Excess Zinc Dust

Sl.	Time	Concentration of				
No.		N	Ni (ppm)			
	(min)	Mesh	Mesh	Mesh		
		-100	-200	3200		
1	0	21	21	21		
2	2	18.7	18.5	17.8		
3	5	17.8	17.3	16.2		
4	10	17	16.3	15.7		
5	15	16.3	14.8	13.7		
6	30	13.2	12.8	12.3		
7	45	12.5	8.9	8.4		
8	60	7	6.6	6.5		
9	90	6.1	5.6	4.8		
10	120	5	3.48	3.2		

## · 200% Excess Zinc Dust

Sl. No.	Time	Concentration of Ni (ppm)		
	(min)	Mesh -100	Mesh -200	Mesh -300
1	0	21	21	21
2	2	18.2	17.3	17
3	5	17	16.1	15.3
4	10	15.2	15.1	14.8
5	15	14.4	14.2	12.5
6	30	11.6	9.78	9.6
7	45	8.1	6	5.95
8	60	5.9	5.1	4.78
9	90	4.9	3.6	3.35
10	120	4.2	2.95	2.85

#### · 300% Excess Zinc Dust

Sl.	Time	Concentration of		
No.		Ni (ppm)		
	(min)	Mesh	Mesh	Mesh
		-100	-200	-300
1	0	21	21	21
2	2	17	15.9	15.2
3	5	15	14.2	13.3
4	10	13.6	13.5	12.8
5	15	13	12.3	10
6	30	9	8.52	7.68
7	45	6.32	4.77	4.11
8	60	4.62	4	3.1
9	90	4.08	2.84	2.42
10	120	3.52	1.88	1.5

#### · 400% Excess Zinc Dust

Sl.	Time	Concentration of		
No.		Ni (ppm)		
	(min)	Mesh	Mesh	Mesh
		-100	-200	-300
1	0	21	21	21
2	2	17.9	16.5	16.4
3	5	16.2	15.5	14
4	10	14.5	14.3	13.5
5	15	13.8	13.5	11
6	30	11	9	8.65
7	45	7.5	5.23	5.11
8	60	5	4.56	4.1
9	90	4.5	3.2	3
10	120	3.98	2.21	2.12

## 7.2 Analysis using Design-Expert

## 7.2.1 Fit Summary

Linear, 2FI (2-Factorial interaction), Quadratic, Cubic and Quartic model can be generated from the data fed into the software. p-values and R<sup>2</sup> values are considered to find out the significant terms to be considered for the model and thus the best model for the analysis of the experiment is determined.

Source	Sequential p- value	Adjusted R <sup>2</sup>	Predicted R <sup>2</sup>	
Linear	< 0.0001	0.6115	0.5906	
2FI	0.0109	0.6445	0.6058	
Quadratic	< 0.0001	0.8274	0.8091	
Cubic	0.0005	0.8601	0.8352	Suggested
Quartic	0.0382	0.8748	0.8307	

From the above table, we can see that the Cubic model is the suggested model for Analysis based on the p-values and R<sup>2</sup> values.

#### 7.2.2 ANOVA for Cubic model

Source	F-value	p-value
Model	35.83	< 0.0001
A-Time	418.04	< 0.0001
B-Percentage Excess of Zinc	3.67	0.0584
C-Size of Zinc Dust	54.73	< 0.0001
AB	0.8983	0.3456
AC	19.49	< 0.0001
BC	0.0108	0.9893
A <sup>2</sup>	144.60	< 0.0001
B <sup>2</sup>	1.62	0.2065
ABC	0.1146	0.8919
A <sup>2</sup> B	0.0013	0.9710
A <sup>2</sup> C	2.64	0.0761
AB <sup>2</sup>	0.4684	0.4953
B <sup>2</sup> C	0.0950	0.9094
A <sup>3</sup>	28.32	< 0.0001
B <sup>3</sup>	0.7508	0.3884

The **Model F-value** of 35.83 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

**P-values** less than 0.0500 indicate model terms are significant. In this case A, C, AC, A<sup>2</sup>, A<sup>3</sup> are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

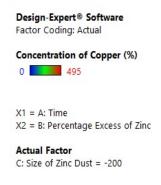
#### **Fit Statistics**

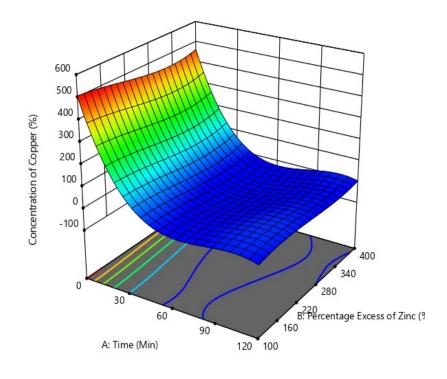
Std. Dev.	70.64	R <sup>2</sup>	0.8848
Mean	171.70	Adjusted R <sup>2</sup>	0.8601
C.V. %	41.14	Predicted R <sup>2</sup>	0.8352
		Adeq Precision	19.7480

The **Predicted R** $^2$  of 0.8352 is in reasonable agreement with the **Adjusted R** $^2$  of 0.8601; i.e. the difference is less than 0.2.

**Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 19.748 indicates an adequate signal. This model can be used to navigate the design space.

# 7.2.3 Model Graphs: 3D Surface





# Design-Expert® Software Factor Coding: Actual

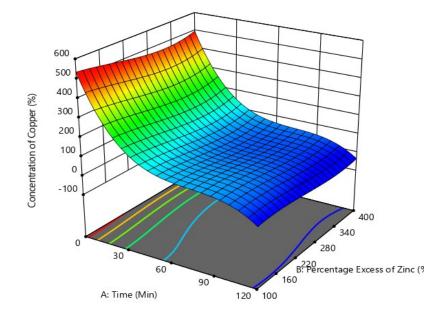
#### Concentration of Copper (%)



X1 = A: Time X2 = B: Percentage Excess of Zinc

#### **Actual Factor**

C: Size of Zinc Dust = -100



# Design-Expert® Software Factor Coding: Actual

#### Concentration of Copper (%)

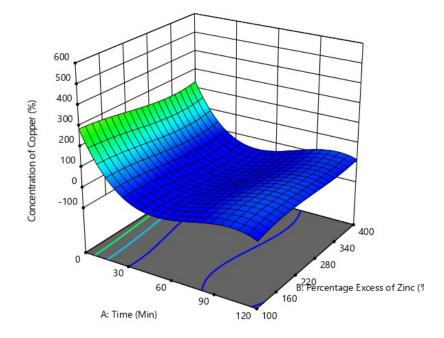


X1 = A: Time

X2 = B: Percentage Excess of Zinc

#### **Actual Factor**

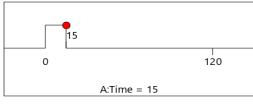
C: Size of Zinc Dust = -300

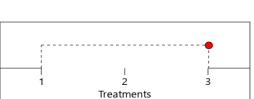


# 7.3 Numerical optimization for the Experiment

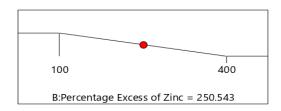
Optimisation of parameters for Nickel purification considering criteria as to minimize the time, percentage excess Zinc dust and limiting Nickel Concentration (1.5-12 ppm) was done, and the following solution was obtained.

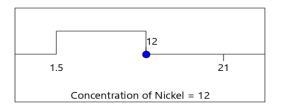
#### Solution 1:





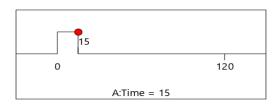
C:Size of Zinc Dust = -300

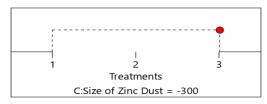


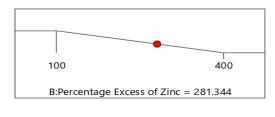


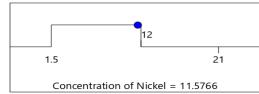
Desirability = 0.498 Solution 1 out of 2

#### Solution 2:









Desirability = 0.396 Solution 2 out of 2

## 8. RESULTS AND DISCUSSION

• Concentration of impurity decreased with time as impurity metals got precipitated out by Zinc but concentration of Cd increases after 1 h cementation, due to re-dissolution.

$$M + 2H^+ + 1/2 O_2 = M^{2+} + H_2O$$

- -300 mesh zinc dust has given better cementation results than -100 and -200 mesh due to large surface area per unit volume.
- Better Cementation results were observed for an excess Zinc Dust of 300% compared to that of 100%, 200% and 400%.
- Multilevel Categoric design of the Cementation Experiment was made considering Time, Percentage Excess Zinc Dust and Size of Zinc Dust as factors.
- The expected change in response per unit change in factor value when all remaining factors are held constant was found and thus the response for any factor value can be deduced.

## 9. CONCLUSIONS

With the set of experimental data, we can predict the response from the randomly known values of the parameters by analysing data set in the Design-Expert software. Furthermore, we can optimise the parameters based on our required criteria of parameters. For instance, Numerical optimisation showed, for example, Copper with particular criteria of parameters is optimised and gave the solution which is having Desirability 0.130. Also, we can estimate the relative influence of the parameters on the response. Here, the main parameters investigated were time, percentage excess Zinc and Zinc dust size.

## 10. References

- [1] https://www.statease.com/docs/v11/contents/optimization/optimization-overview.html
- [2] Principles of extraction of non-ferrous metals by H.S. Ray
- [3] Abrasheva, B., Karoleva, V., 1984. Classification of technological schemes for purification of solutions by cementation in the zinc production plants. Metallurgy 8, 39-41 (in Bulgarian).
- [4] Panão, A.S.I., Carvalho, J.M.R. De, Correia, M.J.N., n.d. Copper Removal from Sulphuric Leaching Solutions by Cementation 1–12.
- [5] Boyanov, B.S., Konareva, V. V., Kolev, N.K., 2004. Purification of zinc sulfate solutions from cobalt and nickel through activated cementation. Hydrometallurgy 73, 163–168. doi:10.1016/j.hydromet.2003.09.002
- [6] van der Pas, V., Dreisinger, D.B., 1996. A fundamental study of cobalt cementation by zinc dust in the presence of copper and antimony additives. Hydrometallurgy 43, 187–205. doi:10.1016/0304-386X(95)00107-R