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Optimization of Pure Copper Powders Production from Leach Solutions Containing Copper

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In this study, in first step, oxidized copper ore was dissolved under optimum conditions in an aqueous NH_3-$(NH_4)_2SO_4$ solution. In the second step, Chevreul's salt, $Cu_2SO_3{\cdot}CuSO_3{\cdot}2H_2O$, was precipitated under optimum conditions by passing SO_2 from the leach solutions obtained. In the third step, Chevreul's salt was dissolved in an acetonitrile-water (AN/H_2O) system in a nitrogen atmosphere. The optimum dissolution of Chevreul's salt at reaction temperature 87 °C, pH 2.75, stirring speed 700 rpm, solid-to-liquid ratio 0.225-g mL^{-1} and AN/H_2O ratio 41 % (v/v =). Initial concentration of $CuSO_4$ solution was chosen as 0.25 M at a fixed value. In the fourth step, the copper powders were produced under optimum conditions by thermal disproportionation of copper(I) sulphate solutions in a nitrogen atmosphere. The best onditions on the dissolution and the precipitation were determined by using 2^n factorial experimental design and orthogonal central composite experimental design methods. The optimum conditions on the maximum copper precipitation at the reaction temperature 70 °C, the reaction time 35 min, the stirring speed 550 rpm, inner pressure (vacuum pressure) 201.3 mm Hg. The fixed parameter chosen at the precipitation reaction was the initial concentration of $CuSO_4$ solution, 0.25 M. Under these optimum conditions, the best precipitation yield was 99.33 %.

Keywords: Copper powder, Chevreul's salt, Factorial experimental design, Orthogonal central composite experiment design.

INTRODUCTION

Production of metals from aqueous solutions is generally chemical process. Production of copper powder from leach solutions is of special importance in hydrometallurgical processes[1-5]. The precipitation of mixed valence compounds from aqueous solutions are of considerable interest in production. One of the mixed valence sulphite compounds is Chevreul's salt[2,5,6]. Chevreul's salt has a highly stable structure. It is an intermediate stage product in copper production. At the same time, the precipitation of this compound from aqueous solutions forms a key stage in hydrometallurgical processes[6,7].

Studies related to precipitation of Chevreul's salt or copper metal by using hydrometallurgical systems have intensified in recent years. Çolak *et al.*[8] precipitated Chevreul's salt from ammonia solution and sulphur dioxide gas. Silva *et al.*[9] synthesized double sulphites such as $Cu_2SO_3{\cdot}MSO_3{\cdot}2H_2O$ (M=Cu, Fe, Mn or Cd). They reported that these salts were thermally stable up to 200 °C. Parker and Muir[6] determined the conditions for precipitation of Chevreul's salt from impure leach solutions. Innoue *et al.*[7] obtained Chevreul's salt by a reaction between $CuSO_4$ and $NaHSO_3$ and characterized by X-ray photoelectron spectroscopy, magnetic susceptibility, electron paramagnetic resonance and electronic spectroscopy. Andrade *et al.*[5] investigated isomorphic series of double sulphites such as the $Cu_2SO_3{\cdot}MSO_3{\cdot}2H_2O$ (M=Cu, Fe, Mn or Cd). They determined that these mixed valence systems can be used as a model to identify intermediates in atmospheric conditions. Hoffmann *et al.*[10,11] proved that transition metal ions such as Fe(III), Cu(II), Co(II), Co(III) and Mn(II) are effective homogeneous catalysts for the autoxidation of sulphur dioxide in an aqueous solution. More recent studies have indicated that metal sulphites and sulphite complexes in solution may play an important role as intermediate products in various chemical processes[11].

In this study, Chevreul's salt was dissolved in an acetonitrile-water (AN/H_2O) system in a nitrogen atmosphere. The copper powders were produced by thermal disproportionation of copper(I) sulphate solutions under optimum conditions in nitrogen atmosphere. 2^n factorial experimental design and orthogonal central composite design methods were used to determine the optimum conditions.

EXPERIMENTAL

Statistical methods measure the effects of operating variables and their mutual interactions on the process *via* experimental design. Today, the most widely used experimental design method is the 2^n factorial design. According to this method, the principal steps of experiments are designed: determination of response variables, choice of factor levels, statistical analysis of data. Consequently, in the final step is obtained a statistical regression model. Two-level experimental design requires 2^n runs in 2 levels of each factor. Coded values of variables are high level = +1 and low level = -1. The coding of variables is extremely important for the analysis of data. Between factor levels, the central coordinate of the design is zero and this value coincides with the origin of coordinates[12].

The variance analysis table (ANOVA) shows the effects of all variables and their mutual interactions[13]. Experimental data based on the design are fitted to a second-order polynominal equation as follows:

$$\hat{Y} = b_o + \sum_{i=1}^{n} b_i X_i + \sum_{i=1}^{n} b_{ii}(X_i^2 - \bar{X}_i^2) + \sum_{i=1}^{n}\sum_{j=1}^{n} b_{ij} X_i X_j \quad (1)$$

where, the coefficient b_i shows the main effects of the factors (X_i); b_{ii} and b_{ij} coefficients represent second-order and mutual interaction terms. The independent term bo represents the response at zero level of every factor ($X_1=X_2=X_3=X_i=0$)[15,16].

If the variance analysis indicates that second-order effects are significant, auxiliary experiments are carried out. Among various second-order designs, the orthogonal central composite design is the most popular which requires 2^n auxiliary runs conducted at two new factor levels, -β , +β. They are calculated by the following relations:

$$\beta = \left(\frac{QF}{4}\right)^{1/4} \quad (2)$$

$$Q = [(N)^{1/2} - (F)^{1/2}]^2 \quad (3)$$

$$N = 2n + F + m_o \quad (4)$$

where F: the number of main experiments in factorial design; N: total number of experiments and m_o: the number of central replicates.

In the planning of experimental designs, the coded values are usually used instead of absolute values of the variables. The relationship between coded value (X) and absolute value (Z) is as follows:

$$X = \frac{2(Z - Z_o)}{(Z_2 - Z_1)} \quad (5)$$

where Z_1 is the low level; Z_2 is the high level and Z_0 is the medium level of the factor[2,13,15].

Oxidized copper ore was dissolved under optimum conditions in an aqueous NH_3-$(NH_4)_2SO_4$ solution. Chevreul's salt was precipitated under optimum conditions by passing SO_2 from these leach solutions obtained by Çalban *et al*[1,2]. The dissolution process in AN/H_2O system of Chevreul's salt was carried out in a glass reactor (250 mL) with three-necks. The reactor was submerged in a Memmert™ (GmbH & Co. KG, Germany) water bath with digital temperature controller. The content of reactor was stirred with a Cole-Parmer™ (COLE-PARMER International, USA) water-resistant magnetic stirrer with digital rate controller. The probe of WTW™ (GmbH & Co. KG, Germany) pH-meter, which simultaneously measures the pH and the temperature was immersed into the solution. The reactor was fitted with a back-cooler to prevent loss by evaporation. All of the experiments were performed under a nitrogen atmosphere. Chevreul's salt in a certain amount was put into the reactor. $CuSO_4$ solution (0.25 M) was added by taking into consideration the solid to liquid ratio. When the content of the reactor reached the desired temperature, the stirring operation was started at a stable speed. AN-H_2O mixture was added drop by drop to the reactor. Experimental parameters and their values for the dissolution process are presented in Table-1. Experimental results were presented in Table-2. The optimum dissolution conditions were determined to resolve all of Chevreul's salt. A vacuum pump was attached to the reactor. The inner pressure was measured by means of a gauge on the vacuum pump. When the Cu_2SO_4 solution reached the desired temperature, the stirring was started at a stable speed in a nitrogen atmosphere. Finally, the copper powders were precipitated in several minutes by thermal disproportionation under vacuum. At the end of the experiment, the nitrogen gas was stopped and the suspension was filtered through filter paper. In order to prevent discoloration of the copper powders in the air, the precipitate was washed with an ether+alcohol mixture. The copper powders were then dried in a desiccator and copper analysis was performed[16]. The chosen experimental parameters and their values for the precipitation process are presented in Table-3. The experimental results were presented in Table-4.

RESULTS AND DISCUSSION

Dissolution and precipitation reactions: When Chevreul's salt is dissolved in an acetonitrile-water solution, the reactions taking place in the medium can be written as follows[6,8].

$$3\,[Cu_2SO_3 \cdot CuSO_3 \cdot 2H_2O](s) + 5\,CuSO_4(aq) + 56\,CH_3CN(aq) \rightarrow 14\,Cu(CH_3CN)_4^{+}(aq) + 4\,H^{+}(aq) + 9\,SO_4^{-2}(aq) + 2\,SO_2(aq) + 4\,H_2O(l) \quad (6)$$

$$SO_2(aq) \rightarrow SO_2(g) \quad (7)$$

when the copper powders are precipitated from Cu_2SO_4 solutions, the reactions taking place in the medium can be written as follows[6,8]:

$$2\,Cu(CH_3CN)_4^{+}(aq)\ Cu(s) + Cu^{+2}(aq) + 8\,CH_3CN(g) \quad (8)$$

Effects of parameters: The effects of parameters on the dissolution of Chevreul's salt were investigated using the levels of parameters given in Table-1. The statistical tests'graphichs were plotted in Fig. 1. The effects of parameters on the precipitation

TABLE-1
PARAMETERS AND THEIR LEVELS ON THE DISSOLUTION OF CHEVREUL'S SALT

Factors	Low level (-)		High level (+)		Medium level (0)
	First order	Second order	First order	Second order	First and second order
X_1: Temperature (°C)	60	53	80	87	70
X_2: pH	2.5	2.33	3	3.17	2.75
X_3: Stirring speed (rpm)	550	450	750	850	650
X_4: Solid to liquid ratio (g.mL^{-1})	1/10	0.092	1/8	0.133	9/80
X_5: AN/H_2O ratio (%)	38	36	44	46	41

TABLE-2
FACTORIAL EXPERIMENTAL DESIGN MATRIX ON THE DISSOLUTION OF CHEVREUL'S SALT

Exp. No.	X_1	X_2	X_3	X_4	X_5	Y_t(min.)	$\hat{Y}_i$
1	-	-	-	-	-	14.23	14.16
2	-	+	-	+	+	29.37	40.20
3	-	-	+	+	+	12.37	9.06
4	-	+	+	-	-	27.05	30.84
5	+	-	-	-	-	6.87	3.16
6	+	+	-	+	+	9.84	16.83
7	+	-	+	+	+	9.03	3.53
8	+	+	+	-	-	9.11	10.51
9	+	+	+	+	-	8.42	9.93
10	+	-	+	-	+	7.37	0.77
11	+	+	-	-	+	12.68	20.22
12	+	-	-	+	-	5.42	3.08
13	-	+	+	+	-	25.63	30.33
14	-	-	+	-	+	9.45	6.23
15	-	+	-	-	+	34.25	43.52
16	-	-	-	+	-	14.56	14.15
17	-1.6647	0	0	0	0	41.37	30.90
18	+1.6647	0	0	0	0	2.92	5.84
19	0	-1.6647	0	0	0	10.17	12.73
20	0	+1.6647	0	0	0	29.54	27.54
21	0	0	-1.6647	0	0	34.15	25.42
22	0	0	+1.6647	0	0	16.88	18.07
23	0	0	0	-1.6647	0	10.82	8.27
24	0	0	0	+1.6647	0	12.77	7.78
25	0	0	0	0	-1.6647	14.72	10.37
26	0	0	0	0	+1.6647	15.87	12.68
1	0	0	0	0	0	9.12	15.87
2	0	0	0	0	0	10.25	15.87
3	0	0	0	0	0	11.42	15.87

TABLE-3
PARAMETERS AND THEIR LEVELS ON THE PRECIPITATION OF COPPER POWDERS

Factors	Low level (-)		High level (+)		Medium level (0)
	First order	Second order	First order	Second order	First and second order
X_1: Temperature (°C)	60	55	80	85	70
X_2: Reaction time (min.)	30	27.65	40	42.35	35
X_3: Stirring speed (rpm)	500	450	600	650	550
X_4: Inner pressure (mm Hg)	212.60	201.30	260.80	272.10	236.70

TABLE-4
FACTORIAL EXPERIMENTAL DESIGN MATRIX ON THE PRECIPITATION OF COPPER POWDERS

Exp. No.	X_1	X_2	X_3	X_4	Y_i (%)	$\hat{Y}_i$
1	-	-	-	-	86.79	84.16
2	+	+	-	+	82.56	89.11
3	-	+	+	+	79.83	74.12
4	+	-	+	-	89.32	95.92
5	-	-	-	+	75.25	71.06
6	+	+	-	-	91.02	99.14
7	-	+	+	-	73.90	69.75
8	+	-	+	+	85.54	90.58
9	-1.47	0	0	0	76.15	77.43
10	+1.47	0	0	0	98.97	93.24
11	0	-1.47	0	0	83.44	84.47
12	0	+1.47	0	0	89.95	84.47
13	0	0	-1.47	0	77.11	75.21
14	0	0	+1.47	0	75.53	72.98
15	0	0	0	-1.47	99.33	94.98
16	0	0	0	+1.47	86.21	86.11
1	0	0	0	0	79.94	82.88
2	0	0	0	0	79.52	82.88
3	0	0	0	0	81.00	82.88

of the copper powders were investigated using the levels of parameters presented in Table-3. The statistical tests'graphichs were plotted in Fig. 2.

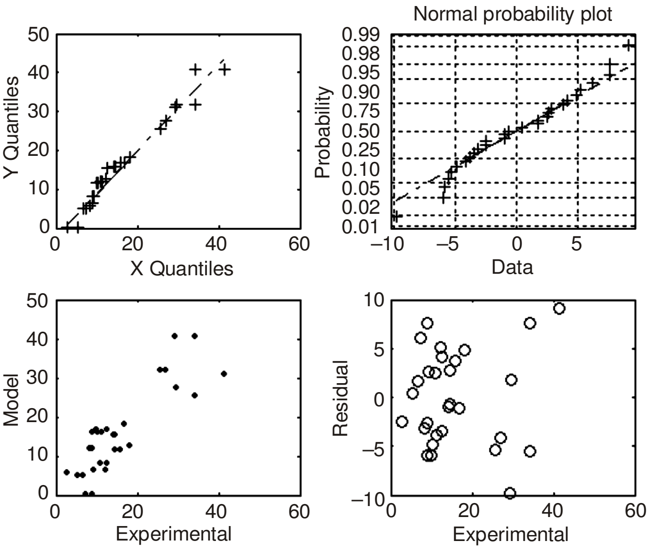

Fig. 1. Statistical tests'graphics (the dissolution of Chevreul's salt in AN/H_2O solutions)

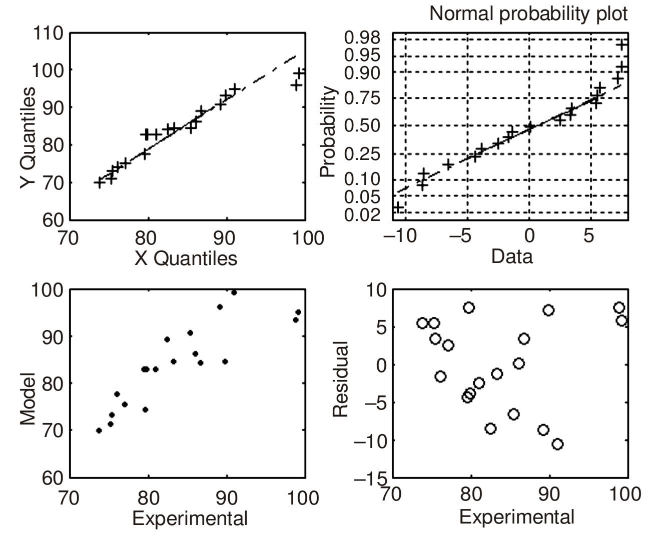

Fig. 2. Statistical tests'graphics (the precipitation of copper powders in Cu_2SO_4 solutions)

Dissolution tests of Chevreul's salt: The effect of the temperature was studied in the range of 53-87 °C. Chevreul's salt was completely dissolved in AN/H_2O system. In all experiments, the other parameters were rigorously controlled. The results are presented in Table-1. The dissolution times of the Chevreul's salt for 53, 70 and 87 °C temperatures were found to be 41.37, 10.26 (the mean value of three central replicates) and 2.92 min, respectively. The dissolution rates increased significantly with increasing of the temperature. This situation can be explained as follows. The reaction rates at homogeneous and heterogeneous reactions increase usually with increasing of temperature. Therefore, the reaction occurs faster.

The effect of the pH was investigated for the range of 2.33-3.17. Chevreul's salt was completely dissolved in AN/H_2O solutions. The experimental results are presented in Table-2. As seen from the Table, the dissolution times were obtained as 10.17, 10.26 (the mean value of three central replicates), 29.54 minutes, respectively. The reaction time increased with increasing of the pH. This effect of pH on the dissolution can be explained as follows. When the pH increases, the dissolution time increases due to decreasing of acidity of the solution medium.

The effect of the stirring speed was examined in the range of 450-850 rpm. The dissolution times of Chevreul's salt for 450, 650 and 850 rpms were found as 34.15, 10.26 (the mean value of three central replicates) and 16.88 min, respectively. The results are presented in Table-2. The dissolution rate increased with increasing of the stirring speed. After 650 rpm, it decreased due to the vortex effect of stirring.

The effect of the solid to liquid ratio was investigated in the range of 0.092-0.133 g mL^{-1}. The dissolution times for 0.092, 0.1125 and 0.133 g mL^{-1} were found to be 10.82, 10.26 (the mean value of three central replicates) and 12.77 min, respectively. The experimental results are presented in Table-2. The dissolution times were almost identical for the solid to liquid ratios chosen.

The effect of the AN/H_2O ratio was studied in the range of 36-46 %. The dissolution times for 36, 41 and 46 % were found to be 14.72, 10.26 (the mean value of three central replicates) and 15.87 min, respectively. The experimental results are presented in Table-2. The reaction time decreased in the range of 36-41 %. It increased in the range of 41-46 %. This situation can be explained as follows. When AN/H_2O ratio increases, complex formation among AN and Cu^+ ions increases with increasing of AN in the solution. In addition, the dissolution time increases with decreasing of water in the solution.

Precipitation tests of pure copper powders: The effect of the temperature was studied in the range of 55-85 °C. The copper powders were precipitated from Cu_2SO_4 solutions obtained from the previous stage. The results are presented in Table-4. The percentages of the copper powders precipitated for 55, 70 and 85 °C were found to be 76.15, 80.16 (the mean value of three central replicates) and 98.97 %, respectively. The obtained copper powders increased significantly with increasing of the temperature.

The effect of the reaction time was investigated for the range of 27.65-42.35 min. Copper powders were precipitated from the Cu_2SO_4 solutions. The experimental results are presented in Table-4. The copper powders were obtained as 83.44, 80.16 (the mean value of three central replicates) and 89.95 %, respectively. As seen from the values, copper powders increased little with increasing of the reaction time.

The effect of the stirring speed was examined in the range of 450-650 rpm. The copper powders obtained for 450, 550 and 650 rpms were found to be 77.11, 80.16 (the mean value of three central replicates) and 75.53 %, respectively. The results are presented in Table-4. The amounts of the copper powders were almost identical in the stirring speeds chosen.

The effect of the inner pressure (vacuum pressure) was studied in the range of 201.3-272.1 mm Hg pressures. The amounts of the copper powders precipitated for 201.3, 236.7 and 272.1 mm Hg vacuum pressures were found to be 99.33, 80.16 (the mean value of three central replicates) and 86.21 %, respectively. The experimental results are presented in Table-4. The produced copper powders decreased with increasing of inner pressure. Therefore, when the vacuum pressure increases, the amounts of the optained copper powders increase.

Statistical analysis of dissolution and precipitation: The collected data on the dissolution and the precipitation were analyzed by an MATLAB compatible PC using ANOVA computer software package for the evaluation of the effect of each parameter on the optimisation criteria. First, a first order model was chosen to fit the experiment data:

$$\hat{Y} = b_o + \sum_{i=1}^{4} b_i X_i + \sum_{i=1}^{n}\sum_{jñ1}^{n} b_{ij} X_i X_j \qquad (9)$$

The factorial design matrix and the experimental results are presented in Tables 2 and 4. To test the significance of the factor effects, an analysis of variance has been conducted at 95 and 99 % confidence intervals. The statistical results are presented in Table-5. As seen from the Table, at 95 and 99 % confidence levels, the temperature (X_1) and the pH (X_2) on the dissolution of Chevreul's salt were found to be effective. Also, the stirring speed (X_3), the AN/H_2O ratio (X_4) and the solid to liquid ratio (X_5) were found to be ineffective. As seen from Table-6, at 95 % confidence level, the temperature (X_1), the reaction time (X_2) and the inner pressure (vacuum pressure) (X_3) on precipitation were effective. Whereas, the stirring speed (X_3) was ineffective. At 99 % confidence level, the temperature (X_1) was only effective. Furthermore, the effects of pure quadratic terms were controlled by means of the following statistic:

$$LOF_{curv.} = \frac{m_o F(\bar{Y}_1 - \bar{Y}_0)^2}{m_o + F} \qquad (10)$$

where m_o is the number of central point experiments, F is the number of factorial experiments, $\bar{Y}_1$ is the mean of factorial experiments and $\bar{Y}_0$ is the mean of central replicates.

TABLE-5
ANALYSIS OF VARIANCE ON THE DISSOLUTION OF CHEVREUL'S SALT (ANOVA)

Source of variation	Sum of squares	df	Mean squares	F_O ratio	Decision (α = 0.1)
X_1	1218.44	1	1218.44	923.06	Effective
X_2	425.28	1	425.28	322.18	Effective
X_3	104.70	1	104.70	79.31	Effective
X_4	0.45	1	0.45	0.34	Ineffective
X_5	10.40	1	10.40	7.88	Ineffective
X_1^2	12.53	1	12.53	9.49	Effective
X_2^2	36.31	1	36.31	27.51	Effective
X_3^2	68.90	1	68.90	52.20	Effective
X_4^2	122.83	1	122.83	93.05	Effective
X_5^2	37.67	1	37.67	28.54	Effective
X_1X_2	184.11	1	184.11	139.48	Effective
X_1X_3	18.08	1	18.08	13.70	Effective
X_1X_4	0.004	1	0.004	0.003	Ineffective
X_1X_5	1.64	1	1.64	1.24	Ineffective
X_2X_3	10.65	1	10.65	8.07	Ineffective
X_2X_4	11.01	1	11.01	8.34	Ineffective
X_2X_5	22.02	1	22.02	16.68	Effective
X_3X_4	7.97	1	7.97	6.04	Ineffective
X_3X_5	370.30	1	370.30	280.53	Effective
X_4X_5	0.0005	1	0.0005	0.0004	Ineffective
Model lack of fit	62.58	6	10.43	7.90	Ineffective
Experimental error	2.64	2	1.32	–	–
Total	2734.00	28	–	–	–

$F_{1; 2; 0.9}$= 8.53; $F_{6; 2; 0.9}$= 9.37

TABLE-6
ANALYSIS OF VARIANCE ON THE PRECIPITATION OF COPPER POWDERS (ANOVA)

Source of variation	Sum of squares	df	Mean squares	F_O Ratio	Decision (α = 0.05)
X_1	354.86	1	354.86	611.83	Effective
X_2	0.00	1	0.00	0.00	Ineffective
X_3	7.08	1	7.08	12.20	Effective
X_4	111.62	1	111.62	192.45	Effective
X_1^2	12.03	1	12.03	20.74	Effective
X_2^2	5.05	1	5.05	8.70	Effective
X_3^2	153.86	1	153.86	265.27	Effective
X_4^2	117.18	1	117.18	202.04	Effective
X_1X_2	6.16	1	6.16	10.62	Effective
X_1X_3	11.86	1	11.86	19.76	Effective
X_1X_4	5.47	1	5.47	9.44	Effective
X_2X_3	133.02	1	133.02	229.34	Effective
X_2X_4	20.39	1	20.39	35.15	Effective
X_3X_4	61.14	1	61.14	105.42	Effective
Model lack of fit	7.26	2	3.63	6.25	Ineffective
Experimental error	1.19	2	0.58	–	–
Total	1011	18	–	–	–

$F_{1; 2; 0.9}$= 8.53; $F_{2; 2; 0.9}$= 9.00

As seen from Tables 4 and 5, $LOF_{curvature}$ is effective. For this reason, the second-order experimental design matrix is created. The second-order design matrix and the experimental results are presented in Tables 2 and 4. According to the experimental results, the regression models were obtained as follows: The dissolution time eqn. (Yt) of Chevreul's salt in AN/H_2O solutions.

$$Y_t = 15,87 - 7.53X_1 + 4.45X_2 - 2.21X_3 - 0.14X_4 + 0.69X_5 + 0.90X_1^2 + 1.54X_2^2 + 2.12X_3^2 - 2.83X_4^2 - 1.57X_5^2 - 3.39X_1X_2 + 1.06X_1X_3 - 0.017X_1X_4 + 0.32X_1X_5 - 0.82X_2X_3 - 0.83X_2X_4 + 1.17X_2X_5 + 0.71X_3X_4 - 4.81X_3X_5 - 0.006X_4X_5 \text{ (at 90 \% confidence interval-full model)} \quad (11)$$

The percentage (Y_p) of pure copper powders precipitated from the Cu_2SO_4 solutions.

$$Y_p = 82.87 + 5.37X_1 - 0.01X_2 - 0.76X_3 - 3.01X_4 + 1.13X_1^2 + 0.73X_2^2 - 4.05X_3^2 + 3.54X_4^2 + 0.88X_1X_2 + 1.20X_1X_3 - 0.83X_1X_4 - 4.08X_2X_3 + 1.60X_2X_4 + 21.77X_3X_4 \text{ (at 90 \% confidence interval-full model)} \quad (12)$$

As seen from the ANOVA Table of dissolution tests, the quadratic terms are effective. Therefore, the orthogonal central composite design is planned to estimate the effect of quadratic terms. With F = 16, $m_o = 3$ and n = 5, β is calculated as 1.6647 according to eqn. 2. Factor levels for the second-order model are presented in Table-1. As seen from the ANOVA Table of precipitation tests, the quadratic terms are effective. With F = 8, $m_o = 3$ and n = 4, β is calculated as 1.47 according to eqn. 2. Factor levels for the second-order model are presented in Table-3. Also, the second-order model is defined as at eqns. 1 and 13.

$$\bar{X}_i^2 = \frac{1}{N}\sum_{i}^{N} X_i^2 = \frac{F + 2\beta^2}{N} \quad (13)$$

The second-order model eqn. 11 obtained at 90 % confidence level was obtained as 20 factors. It fitted the experimental results very well. The multiple correlation coefficient, r^2, was found to be 0.9917. The other second-order model eqn. 12 was obtained at 90 % confidence level. It was 14 factors and fitted the experimental results very well. The multiple correlation coefficient, r^2, was found to be 0.9761.

Systematic errors in a well-established model are absent. Normalised residuals depend on experimental errors and these values exhibit a normal distribution[12-15]. The statistical tests' graphics are presented in Fig. 1 and 2.

Conclusions

- The optimum dissolution conditions of Chevreul's salt were determined as the reaction temperature 87 °C, the pH 2.75, the stirring speed 700 rpm, the solid to liquid ratio 0.225 g mL^{-1} and the AN/H_2O ratio 41 % (v/v). The concentration of $CuSO_4$ solution was chosen as a fixed value, 0.25 M.
- The optimum conditions found for the amount of maximum copper powder were: the reaction temperature 70 °C, the reaction time 35 min, the stirring speed 550 rpm, the inner pressure (vacuum pressure) 201.3 mm Hg. The fixed parameter chosen at precipitation reaction was concentration of $CuSO_4$ solution, 0.25 M.
- The copper powders produced are pure at 99.33 %.
- Under optimum conditions, the percentage of copper precipitating in copper powder form was 99.33
- Chevreul's salt completely dissolved all of the experiments in an AN/H_2O mixture in a nitrogen atmosphere.
- The temperature is the most predominant parameter on both the dissolution of Chevreul's salt and the production of copper powders.
- Chevreul's salt dissolution time decreased with increasing of the temperature, the stirring speed and the solid to liquid ratio.
- The temperature, the pH and the stirring speed are highly effective on the dissolution rate of Chevreul's salt.
- The solid to liquid and the AN/H_2O ratios are barely effective on the dissolution of Chevreul's salt.
- The copper powders were produced from the Cu_2SO_4 solutions in a nitrogen atmosphere.
- The copper powder precipitation yield increased with increasing of the temperature.
- The amounts of copper powders decreased with increasing of the reaction time, the stirring speed and the inner pressure (vacuum pressure).
- The most effective parameters on the precipitation of copper powders are the temperature and the inner pressure (vacuum pressure).

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