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Optimal analysis for leaching of lead from processed anode slime

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Abstract

The leaching of lead from the treated anode slime with triethanolamine (TEA) was investigated by using full factorial and orthogonal design techniques. The effects of TEA concentration, solid/liquid ratio, temperature, and contact time on the leaching process were evaluated to determine the interactions between the parameters. The major factors for the system were found to be TEA concentration and reaction temperature. The highest recovery is obtained at process conditions of 3.5 M TEA concentration, reaction time of 150 min, solid/liquid ratio of 1/10, and a temperature of 313 K. A polynomial model between the important parameters and response was deduced using variance analysis at 95 % confidence level. The analytical performance for measurement at the point of optimum by this technique was found to be superior and more proper than that of one variable at a time.

Keywords

Optimal analysis, processed anode slime; recovery of lead, leaching; statistical design.

Introduction

In nature, the lead element is usually found in association with zinc, silver, and copper-containing minerals and is extracted together with these metals. The lead-based minerals are galena, cerussite, and anglesite. Nevertheless, more than half of the lead is currently supplied by recycling (Li et al., 2018; Więcek et al., 2019; Ding et al., 2018). Recycled elements are becoming progressively important as the industry responds to public demand that resources be conserved and surroundings be secured. For this reason, the recovery of valuable metals, including gold, silver, and lead, from secondary resources such as anode slime and scraps is now being studied and put to practical use (Laubertová et al., 2019; Xu et al., 2016; Ruşen et al., 2018).

Valid methods for the earnings of precious elements in the anode slimes can be usually individuated into pyrometallurgical, which contains roasting by an oxidizing chemical, soda-ash roasting processes (Wang et al.,2014; Han et al., 2017; Khanlarian et al., 2019; He et al., 2017), and hydrometallurgical, where which the anode slimes are processed by various leachants like sulfuric acid, hydrochloric acid and chlorine (Xiao et al., 2018; Xing et al., 2017; Li et al., 2017; Dönmez et al., 2001).

Currently, the hydrometallurgical treatment for copper anode sludges is generally preferred because of its advantages such as its applicability to small and large processes, relatively low capital costs compared to a smelter, and relatively prevention of air pollution from sulfur dioxide.

The chemistry and composition of the anode slime are based on the composition of the anodes and the refining conditions. According to the morphology and composition of the anode slimes, various processes such as the pyro-, hydropyro-, pyrohydro-, and hydrometallurgical methods are evolved for the recovery of precious elements present in this slime (Xing et al., 2020; Mohanty et al., 2019; Mikoda et al., 2019).

Various works on the anode slimes were done (Havuz et al.,2010; Xing et al., 2018; Ruşen et al., 2019). Dönmez et al. (1998) investigated the leaching in H_2SO_4 solutions of copper from anode slime. Optimum conditions were determined in which Cu conversion was achieved as 99.7 %. The recovery of selenium from Iranian sar-cheshmeh copper anode slime was made by Pasdar et al. (2019). Arsenic and antimony from anode slimes were dissolved in 0.4 M KOH. Then, selenium was solubilized with the alkaline roasting of anode slimes in the presence of Na_2CO_3. Finally, the recovery of selenium was determined as 98 %.

The experimental simulation technique is properly applied for search on industrial settings and for the analysis of findings in the physical, chemical, medical, social, economic, engineering, or sciences spheres. The primary goal of scientific research is to show the statistical significance of an effect that a particular factor exerts on the dependent variable. In addition, poorly designed experiments may yield the wrong results, losing both time and scientific resources. The other advantages of the experimental simulation approach are cost reduction, increased efficiency of the experiment, and often, the elucidation of the essential nature of a process (Yang et al., 2019; Lesňák et al., 2020; Tokkan et al., 2013).

The purpose of the present work is to detect the optimal conditions for leaching of lead in treated anode slime by aqueous triethanolamine (TEA) solutions using the factorial experimental design. In addition, a regression model is obtained such that, for the chemical process, one reaction parameter defines the adjustment of another and the resulting interactive impacts.

Material and Methods

The traditional ‘step-by-step’ or ‘one variable at a time’ optimization strategy is widely used. The method is simple and easy. Nevertheless, because it does not serve any information about the optimum situation and can cause only a local optimum of the process, it is not an effective and economic tactic. The optimization also neglects interactions between parameters and demands unnecessarily numerous runs. For rapidly rising costs of tests, the development and optimization of any analytical method must be done with as few experiments and as low costs as possible (Mokmeli et al., 2015; Janič et al., 2019).

Whereas statistical experiment design provides an effective and efficient approach to optimization strategy. An experimental design aims to supply as much knowledge as possible from few experiments and define the optimum conditions for the different parameters that influence a system. In other words, the design is especially important if experiments are expensive or require huge resources of time and material.

The most used experimental design to guess the primary parameters, besides the interaction effect, is the 2^n factorial design. Each of the variables is examined at two levels, high and low. As the number of factors, n, increases, the number of experiments increases proportionally. A full factorial design is one in which all possible combinations of the factors at all levels involved in the experiments are applied. The first step in the 2^n factorial experimental design method is the choice of the variables and their levels. The second step in the method is the determination and statistical analysis of data. To occur a statistical model is the last stage for the experimental design (Achilli et al., 1997).

The leaching of lead from anode slime in different triethanolamine concentrations using microwave effect is studied by Tokkan et al. (2012). A statistical model on variance analysis has been established using the Matlab computer software for various parameters. The optimum conditions for the work are determined as leaching temperature 313 K, the concentration of triethanolamine 3.5 M, reaction period 150 minutes and solid-liquid ratio 0.1.

Guo et al. (2017) offered a process to convert and separate selenium and arsenic from copper anode slime (CAS) by an alkali fusion process. Second-order polynomial models and 3D response surface plots were set to establish the relationship between the responses and the variables. By the overlaid contours at NaOH/CAS mass ratio of 0.65-0.75, fusion time of 20-30 min and fusion temperature of 803-823 K, optimum area of > 90% selenium conversion ratio and > 90% arsenic conversion ratio was supplied.

For this study, the anode slime was obtained from Sarkuysan Co., Turkey, and the slime was processed to remove copper. The samples of the anode slime were generally analyzed by gravimetric and volumetric methods. Again, the analysis of some elements was done by X-ray diffractogram. Trace elements also were determined by using atomic absorption spectroscopy. The chemicals used were of analytical level. The test results are presented in Tab. 1.

Tab. 1. The chemical composition for processed anode slime

Component	Composition (%, w/w)
Ag	2.05
Au	0.13
Pb	29.0
SO_4^{-2}	28.7
Sb	17.16
As	0.93
Cu	0.33
Sn	15.95
SiO_2	1.72
Zn	0.28
Fe	0.16
Ni	0.03
The others	2.86

Leaching experiments were carried out in a glass reactor of 500 mL volume, equipped with a mechanical stirrer with a digital controller unit, a water-bath with a digital temperature controller, and a back cooler (Fig. 1). After the temperature was adjusted at the wished value, the TEA solution was poured into the reactor. Later, an amount of solid was added to the reactor, and reactor content was started to be mixed. For each test, the reactor slurry was quickly filtered. Eventually, the lead amount was tested in an atomic absorption spectrophotometer.

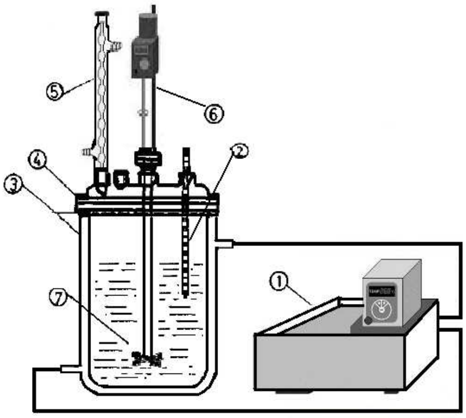


Fig. 1. Experimental setup. 1. Water bath, 2. Termometer, 3. Glass reactor, 4. Cover fastener, 5.Back cooler, 6.Mechanical stirrer, 7. Stirrer blade.

Preliminary tests were done to see the effect of relevant parameters and determine the experimental field. With the information gathered from pre-experiment and literature search, the parameter levels (high and low) were determined. As parameters in the process, TEA concentration (X_1), temperature of reaction (X_2), solid/liquid ratio (X_3), and contact period (X_4) were examined. In addition, the tests were designed with the above-mentioned methodology. To simplify the calculations, high, low, and mean levels for the parameters were coded as (+), (-), and (0), as shown in Table 2.

Tab. 2. Parameter and levels belong to 24 factorial plan

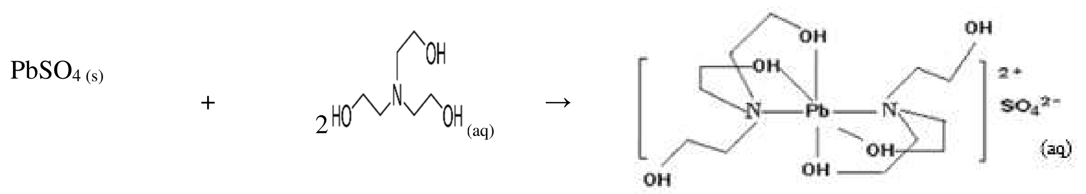
Parameter	Variant	Low Level (-)	High level (+)	Average level (0)
Concentration of TEA (M)	X_1	2.5	3.5	3.0
Ambience temperature (K)	X_2	303	313	308
Solid-liquid ratio (g.mL^{-1})	X_3	0.10	0.20	0.15
Contact period (min.)	X_4	90	150	120

The centre point was equalized to the mean worth of a parameter. From the test results, the wanted data were compiled and tested by the MATLAB software package for interpreting the worth of each factor for the optimization.

Results and Discussion

Based on thermodynamic data, it is shown that both Pb^{+1} and Pb^{2+} complexes have the largest negative enthalpy and negative entropy changes compared to many transition metals. This result reveals that the Pb^{+2} complex, including TEA, forms a more stable complex than many other structures. Still, it shows that the complexation of transition metals and metal complexes with amino alcohols requires further investigation (Majid et al., 2008; Ashram et., 2010).

The fundamental reaction for dissolution in the lead from unprocessed anode slime in a TEA solution may be represented as follows:



Leadsulphate Triethanolamine Lead triethanolamine sulphate complex

Initially, the 24 factorial design is used to develop the first-order model with interaction terms as follows:

$$Y = b_o + \sum_{i=1}^{4} b_i X_i + \sum_{i=1}^{4} \sum_{j\rangle 1}^{4} b_{ij} X_i X_j \qquad (1)$$

where Y is the predicted yield, n is the number of variables, X_i represents the factors, b_o is the response at the zero level for the parameters, b_1 is the individual responses for each factor effect, b_{ij} is the responses for the interactions between parameters.

With four factors, a 24 factorial plan requires 16 runs for the present work. To reduce the effects of unchecked factors and systematic errors, all tests are carried out in a randomized order. Moreover, three central repeats are made to consider the test errors. The test plan is shown in Table 3, together with the coded values of the parameters and the response values. Each trial is conducted only once.

Tab. 3. Test plan and consequents

Test No	Standard order	X_1	X_2	X_3	X_4	Y_{Pb}
16	1	+	-	-	-	50.13
12	2	-	-	-	-	39.12
2	3	+	+	-	-	61.93
6	4	-	+	-	-	42.85
13	5	+	-	+	-	59.66
9	6	-	-	+	-	44.41
1	7	+	+	+	-	71.16
5	8	-	+	+	-	51.30
4	9	+	-	-	+	56.52
8	10	-	-	-	+	46.65
10	11	+	+	-	+	69.46
11	12	-	+	-	+	54.93
3	13	+	-	+	+	62.69
7	14	-	-	+	+	50.46
14	15	+	+	+	+	77.02
15	16	-	+	+	+	58.78
1*	1*	0	0	0	0	59.97
2*	2*	0	0	0	0	60.46
3*	3*	0	0	0	0	61.30

** Central points replicates.*

The regression expression for lead recovery from processed anode slime, founded with first-order design by variance analysis done at 95 % confidence interval, is as follows:

$$Y_{Pb} = 56.78 + 7.50X_1 + 4.86X_2 - 3.37X_3 + 3.50X_4 + 1.46X_1X_2 \tag{2}$$

In the variance analysis, if the influence of one of the parameters is significant as checked with the testing mistake, it is determined that the changes in the selected response cannot occur by chance. Those changes in the response can only be the effects of the factors. The parameters causing the variation in the response are called important.

In addition, the impacts of the quadratic expressions are tested by the following statistical expression:

$$LOF_{curv.} = \frac{m_O F.(\bar{y}_1 - \bar{y}_O)^2}{m_O + F} \tag{3}$$

where, m_o is the quantity for centrical point test, $LOF_{curv.}$ is the curvature, F is the number of factorial tests, $\bar{y}_1$ is the average of the factorial tests, and $\bar{y}_0$ is the average of central copies. As observed from Table 4, the analysis of variance shows curvature effect. As the $LOF_{curv.}$ expression is effective, efficacies of quadratic expressions are regarded.

Tab. 4. Variance analysis

Variation source	Sum of squares	d.f.	Mean squares	F ratio	Decision (α=0,05)
X_1	901.05	1	901.05	1991.57	Effective
X_2	378.21	1	378.21	835.94	Effective
X_3	181.51	1	181.51	401.18	Effective
X_4	195.65	1	195.65	432.44	Effective
$X_1 X_2$	34.08	1	34.08	75.32	Effective
$X_1 X_3$	7.69	1	7.69	16.99	Ineffective
$X_1 X_4$	6.67	1	6.67	14.74	Inactive
$X_2 X_3$	1.15	1	1.15	2.54	Inactive
$X_2 X_4$	6.19	1	6.19	13.68	Inactive
$X_3 X_4$	7.72	1	7.72	17.05	Inactive
Curvate	51.38	1	51.38	113.57	Effective
Model lack of fit	2.17	5	0.44	0.96	Inactive
Test error	0.91	2	0.46		
Total	1774.36	18			

F0.05(1,2) = 18.51; F0.05(5,2) = 19.30

Also, adjunct tests are done. In the various second-order designs, the orthogonal central-composite design is the most proper, which needs 2^n auxiliary runs performed at two new factor levels; the factorial design calls for $-\beta$, $+\beta$, β, calculated by the following equation:

$$\boldsymbol{\beta} = (\frac{QF}{4})^{1/4} \tag{4}$$

where

$$Q = \left[N^{1/2} - F^{1/2}\right]^2 \tag{5}$$

$$N = F + 2n + m_o \tag{6}$$

F is the number of tests for factorial design
N is the total number of tests and m_o is the number of central replicates.

The new factor levels are shown in Table 5, where some variable levels are rounded off based on the sensitivity of the equipment given.

Tab. 5. Auxiliary parameter levels for the central composite plan

Parameter	Variant	Low Level (-)	High level (+)
Concentration of TEA (M)	X_1	2.2	3.8
Ambience temperature (K)	X_2	300	316
Solid/liquid ratio (g.mL^{-1})	X_3	0.07	0.23
Contact time (min.)	X_4	74	166

For the auxiliary works, the design matrix and results are shown in Table 6.

Tab. 6. Experimental design for second-order model and dissolution yield

Experiment No	X_1	X_2	X_3	X_4	Y_{Pb}
17	-1.546	0	0	0	51.06
18	+1.546	0	0	0	70.89
19	0	-1.546	0	0	50.36
20	0	+1.546	0	0	73.57
21	0	0	-1.546	0	49.13
22	0	0	+1.546	0	63.16
23	0	0	0	-1.546	56.72
24	0	0	0	+1.546	65.21

To make easy calculations, the second-order equation is described as follows:

$$Y = b_o + \sum_{i=1}^{4} b_i X_i + \sum_{i=1}^{4} b_{ii}\left(X_i^{2} - \overline{X_1^{2}}\right) + \sum_{i=1}^{4}\sum_{j\rangle 1}^{4} b_{ij} X_i X_j \tag{7}$$

That is

$$\overline{X_1^{2}} = \frac{1}{N}\sum_{i=1}^{N} X_i^2 = \frac{F+2\beta^2}{N} \tag{8}$$

By defining,

$$b_o^l = b_o - \sum_{i=1}^{4} b_{ij}\overline{X_1^{2}} \tag{9}$$

Eq. (8) may be rewritten in the classical shape as follows:

$$Y = b^l{}_o + \sum_{i=1}^{4} b_i X_i + \sum_{i=1}^{4} b_{ii} X_i^{2} + \sum_{i=1}^{4}\sum_{j\rangle 1}^{4} b_{ij} X_i X_j \tag{10}$$

The model tested by regression analysis for 95 % confidence range is as follows:

$$Y_{Pb} = 57.75 + 7.25X_1 + 5.49X_2 - 3.63X_3 + 3.32X_4 - 1.00X_1^2 - 1.53X_2^2 - 3.02X_3^2 - 1.02X_4^2 + 1.46X_1X_2 - 0.69X_1X_3 - 0.65X_1X_4 - 0.27X_2X_3 + 0.62X_2X_4 + 0.69X_3X_4 \tag{11}$$

At a 95 % confidence level, it is shown that the correlation coefficient belonging to the second-order model is 0.94. This implies that this model nearly fitted the experimental data. Moreover, for fewer factors, the simpler model might also be founded using variance analysis made for 95 % confidence level as follows. As ascertained from Table 4, the analysis of variance shows a curvature effect.

$$Y_{Pb} = 57.75 + 7.25X_1 + 5.49X_2 - 3.63X_3 + 3.32X_4 - 1.00X_1^2 - 3.02X_3^2 - 0.65X_4^2 + 1.46X_1X_2 \tag{12}$$

Eq. (13) shows that the main parameters influencing lead leaching are TEA concentration and reaction temperature; the other variants have proportionally smaller effects.

In contrast, the terms of second-order and the interaction terms affect the system. From Table 7, it is possible to identify that the interactions between stirring speed, TEA concentration, and reaction period do not have important effects on the metal recovery, except for the effectiveness of TEA concentration and temperature.

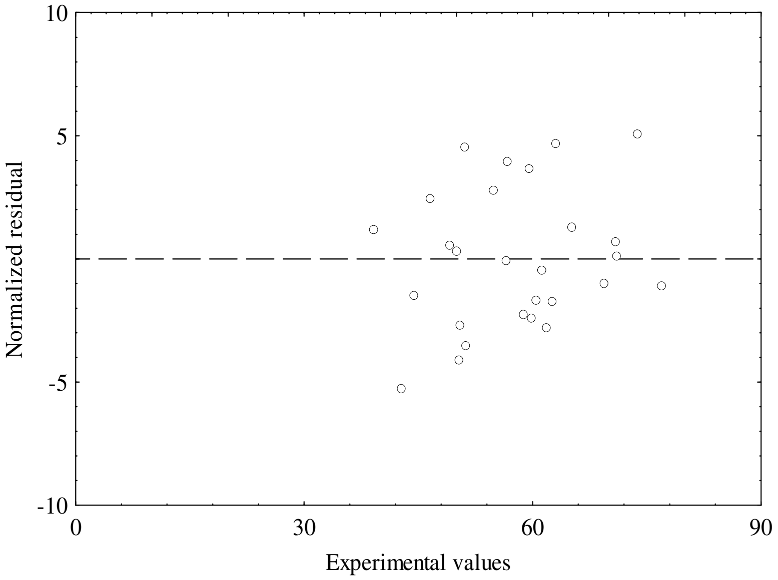
Tab. 7. Variance analysis belong to the second-order model

Variation source	Sum of squares	d.f.	Mean of squares	F ratio	Decision (α=0,05)
X_1	1093	1	1093	2416.4	Effective
X_2	622	1	622	1374.5	Effective
X_3	275	1	275	607.6	Effective
X_4	230	1	230	507.5	Effective
X_1^2	11	1	11	24.4	Effective
X_2^2	4	1	4	8.1	Ineffective
X_3^2	103	1	103	227.7	Effective
X_4^2	11	1	11	24.6	Effective
$X_1 X_2$	34	1	34	75.3	Effective

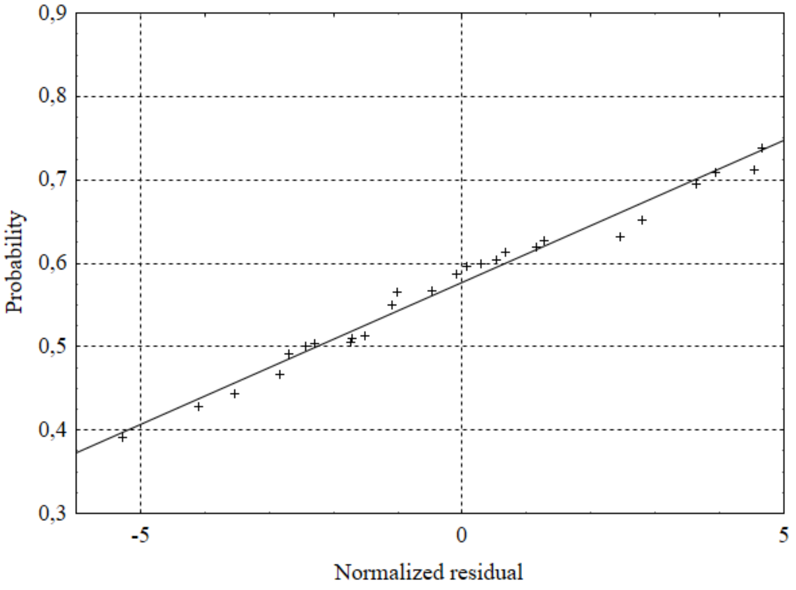
$X_1 X_3$	8	1	8	17	Ineffective
$X_1 X_4$	7	1	7	14.7	Ineffective
$X_2 X_3$	1	1	1	2.5	Inactive
$X_2 X_4$	6	1	6	13.7	Ineffective
$X_3 X_4$	8	1	8	17.1	Ineffective
Model lack of fit	61,99	10	6,20	13.70	Ineffective
Test mistake	0,91	2	0,45		
Total	2474,9	26			

F *0.95(1; 2)= 18.531;*F*0.95(10; 2)= 19.40*

The graphical representations of the analytical tests are given in Figures 2a and 2b, which assists that the model is realistic.



(a)



(b)

Fig. 2. Test graphics for statistical (a and b).

Conclusions

The leaching of lead from processed anode slime in TEA solutions has been investigated in this study. From the pre-experiments, particle size and agitation rate are observed to be negligible factors; temperature, TEA concentration, solid-liquid ratio, and reaction period, in addition to their quadratic effects, are determined to be the effective parameters. On the basis of these results, whereas the solid-to-liquid ratio provides a negative effect on the response, the TEA concentration, reaction temperature, and contact period have been concluded to have a favourable efficacy. The R^2 value provided from the variance analysis is 0.94, which implies that the model can explain that a variation of 94 % for lead leaching is attributed to the factors; furthermore, it also shows that the model does not express only 6 % of the total variation.

As shown in Table 3, the highest recovery is obtained at process conditions of 3.5 M TEA concentration, the reaction time of 150 min, the solid/liquid ratio of 1/10, and the temperature of 313 K. The mutual effects of TEA concentration and reaction temperature are found to be the most significant effects among the effects of all the factors.

The graphical residual test is a significant and efficient model-validation test method and has been utilized to control the adequacy of the established models in this investigation. In Fig. 2a, a normal graph of the residuals has been presented. The figure shows all residuals lying on a straight line, which explains that the residuals have been distributed normally. The values of the normalized residues depend on the test mistakes. Therefore, it may be said that systematic mistakes belonging to a well-established model are unavailable. In Fig. 2b, the experimental values are plotted versus the residuals to check the assumption that the random errors are distributed with constant variance and mean zero. All residuals are distributed between -5 and +5.

It can be concluded that, under complex experimental conditions, this experimental design is of great practical technique for investigating the maximum value. In addition, the use of such a design method reduces the number of experiments, data analyzed, and testing errors. Moreover, the design can be important before detailed kinetics and optimization can be carried out; also, it can provide the background for pilot and industrial-scale applications.

ORCID :

Nomenclature

R^2 multiple correlation coefficient
X_1 codified value of TEA concentration (M)
X_2 codified value of reaction temperature (K)
X_3 codified value of solid-to-liquid ratio (g.mL^{-1})
X_4 codified value of contact time (h)
b_i regression coefficient
Y leaching yield (%)

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