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# Optimization of Smithsonite Ore Solubility with Lactic Acid Leaching Reagent

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**Research Article**

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## Abstract

In this study, optimum parameters for leaching performance of lactic acid reagent in smithsonite ore were determined using L16 (45) experimental plan in the Taguchi Statistical Method. Since maximum smithsonite ore extraction was desired for the result, Taguchi's 'bigger is better' performance formula was used. In the experiments; parameters affecting dissolution such as temperature, reaction time, particle size, acid concentration and solid/liquid ratio were investigated. When ANOVA analysis was applied to the parameters, it was determined that the most effective parameters according to F values were acid concentration, solid/liquid ratio and particle size, respectively. In addition, it was observed that zinc conversion increased with increasing temperature and decreasing solid/liquid ratio and particle size. The parameter levels that maximized the S/N value were determined to be 30ºC temperature, 0.01 $g.mL^{-1}$ solid/liquid ratio, 1 M lactic acid concentration, -160.5 µm particle size and 20 min reaction time. Under these conditions, it was found that smithsonite ore was dissolved by 98.85%.

**Keywords:** Smithsonite ore, organic acid, optimization, Taguchi Method, dissolution

# Laktik Asit Liç Reaktifi ile Smithsonit Cevherinin Çözünürlüğünün Optimizasyonu

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## Öz

Bu çalışmada, laktik asit reaktifinin smithsonit cevherindeki liç performansı için optimum parametreler Taguchi İstatistiksel Yönteminde L16 (45) deney planı kullanılarak belirlenmiştir. Sonuç için maksimum smithsonite cevheri ekstraksiyonu istendiğinden Taguchi'nin 'büyük daha iyidir' performans formülü kullanılmıştır. Deneylerde; sıcaklık, reaksiyon süresi, partikül boyutu, asit konsantrasyonu ve katı/sıvı oranı gibi çözünmeyi etkileyen parametreler incelenmiştir. Parametrelere ANOVA analizi uygulandığında, F değerlerine göre en etkili parametrelerin sırasıyla asit konsantrasyonu, katı/sıvı oranı ve partikül boyutu olduğu belirlenmiştir. Ayrıca, çinko dönüşümünün artan sıcaklık ve azalan katı/sıvı oranı ve partikül boyutu ile arttığı gözlemlenmiştir. S/N değerini maksimize eden parametre seviyeleri 30ºC sıcaklık, 0,01 $g.mL^{-1}$ katı/sıvı oranı, 1 M laktik asit konsantrasyonu, -160.5 µm partikül boyutu ve 20 dk reaksiyon süresi olarak belirlenmiştir. Bu koşullar altında, smithsonite cevherinin %98.85 oranında çözündüğü bulunmuştur.

**Anahtar Kelimeler:** Smithsonit cevheri, organik asit, optimizasyon, Taguchi Yöntemi, çözünme

## Introduction

As the amount of consumption increases and technological developments accelerate, the need for raw materials has also increased. At the same time, it is clear that the processing of these resources will contribute to the development of countries. One of the most effective factors in developed countries reaching the level of technology and prosperity has been the mining sector. Although ores are commonly obtained by pyrometallurgical methods in the mining sector, the recovery of low-grade ores by hydrometallurgical methods involves less energy, higher yields and fewer environmental problems [1-4]. In the hydrometallurgical method, the impurities in the ore content pass into solution along with the desired product, making the process difficult. In order to minimize this negativity, organic leaching reagents that provide more selective dissolution have been widely used in recent years. Despite these advantages of using organic acids as leaching reagents, they are not preferred in leaching processes that require high temperatures because they have very low boiling points and can sometimes decompose at the boiling point. They are also not suitable reagents for dissolving poorly soluble solids [5-9]. Today, the most consumed metals and compounds are zinc after steel, aluminum and copper. Zinc, is estimated to constitute 0.13% of the earth's crust. Zinc is usually produced hydrometallurgically from minerals such as sphalerite, marmatite or smithsonite ore [10-12]. Although sulfur-zinc compounds obtained from these mines are widely used worldwide, the use of these compounds has begun to be restricted due to the harmful effects of sulfur formed during processing. Therefore, due to increasing environmental awareness, the use of carbonated zinc ores has been widely preferred in the production of biodegradable materials, especially in recent years [13, 14]. Lactic acid, one of the organic acids, can be used both to increase selectivity and to reduce the damage of inorganic acids. Lactic acid, with the chemical formula $C_3H_6O_3$, is one of the oldest organic acids widely known in industrial and biotechnological applications worldwide. Lactic acid is used in the production of products such as bread, cheese in the food industry, and cosmetics, medicine, chemical synthesis, biodegradable plastics [15-16]. Zinc lactate is a lactic acid-derived zinc salt and is an important multifunctional ingredient used in toothpaste, chewing gum, deodorant, mouthwash and many other oral care products. The supply of zinc lactate has been steadily increasing worldwide in the growing healthcare sector [17-18]. The Taguchi method used here is an optimization technique that reduces the number of experiments by using orthogonal array (OA) experimental design. The Taguchi method is a simple, effective and systematic method used to optimize the system in terms of quality and cost [19-20]. This method is used to scan the parameters of a process and produce the process information necessary to determine its optimal conditions using the least number of experiments possible. The most important advantage of orthogonal experimental design is that fewer factors are used and their levels are changed simultaneously [21-22]. This technique allows the determination of a variance index by converting the obtained data into a signal/noise (S/N) ratio. There are three types of S/N ratios, depending on the characteristics of the experiment in which they are used. These are “the largest is the best”, “the smallest is the best”, “nominal is the best”. In addition, the

optimum operating conditions obtained from laboratory studies with this method can also be used in real production environments [23-26]. The aim of this study is to examine the effects and optimization of various parameters such as acid concentration, reaction time, solid/liquid ratio, temperature and particle size in the dissolution of smithsonite ore with lactic acid for the production of zinc lactate.

## Materials and Method

The smithsonite ore used in the study was obtained from Kayseri in Türkiye. The ore was initially crushed in a jaw crusher, subsequently processed in a grinder, and then sieved using ASTM standard sieves to achieve particle size fractions of -160.5, -283.5, -477.5 and -890 μm. Then, using conventional volumetric and gravimetric techniques, chemical analyses were performed [27]. Table 1 presents the analytical findings. The X-ray diffraction (XRD) analysis of smithsonite ore was obtained from a Philips PW 3710 BASED model device. The XRD showing the contents of the smithsonite ore was illustrated in Figure. 1.

***Table 1.*** *The chemical composition of smithsonite ore*

| **Component** | **(%)** |
|---|---|
| ZnO | 59.12 |
| $Al_2O_3$ | 1.52 |
| $SiO_2$ | 1.8 |
| CaO | 0.9 |
| $Fe_2O_3$ | 1.6 |
| Impurities | 1.46 |
| Ignition loss | 33.6 |

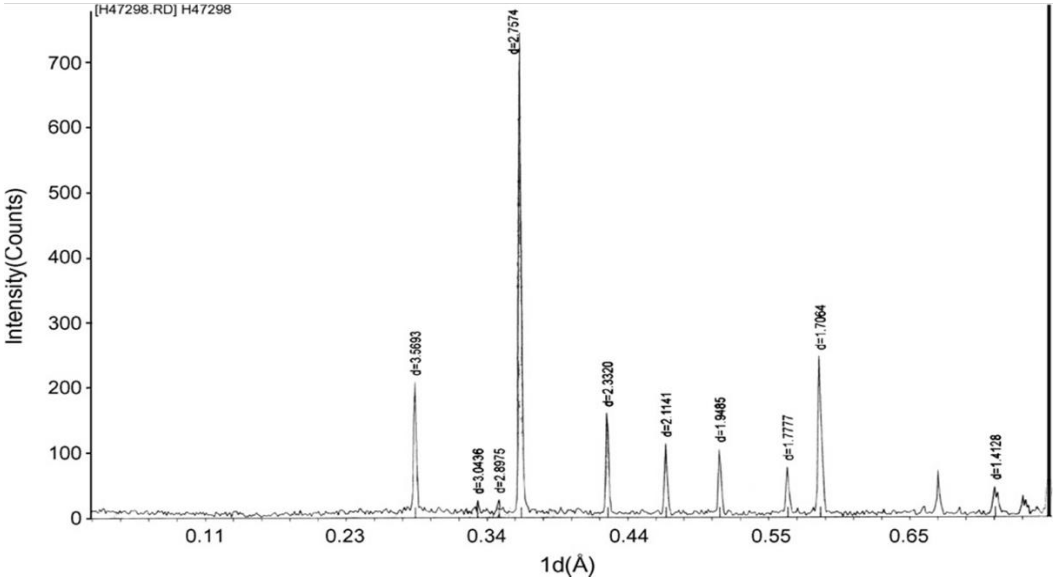


***Figure 1.*** *XRD graph of smithsonite ore*

Lactic acid, a fruit acid that is used as a reagent a carbon hydrate substrate is fermented by microorganisms to produce [28]. The ethylenediamine tetra-acetic acid (EDTA) reagent used in the analysis is high purity. Leaching experiments were carried out in a 500 mL cylindrical glass reactor

equipped with a temperature-controlled circulator to keep the reactor content at constant temperature during the reaction, a digitally controlled mechanical stirrer to adjust the stirring speed, a thermometer for temperature control, and a condenser to prevent losses by evaporation. Since the stirring speed had no effect in the kinetic study conducted by Ilhan [29], the stirring speed was kept constant at 600 rpm. For each experiment, 200 mL of lactic acid with a certain concentration was added to the reactor at the desired temperatures. When the temperature in the reactor content reached equilibrium, the reactor content was stirred at 600 rpm, while 2.0 g of smithsonite ore with the desired grain size was added to the reactor. Samples were taken from the reaction mixture at predetermined time intervals from the leaching solution, filtered, and zinc was determined [27]. To observe the solubilities of noise, such as the temperature and humidity of the laboratory environment, each experiment was performed twice at various times under the same conditions. Zinc can be determined by complexometric analysis using EDTA (ethylenediaminetetraacetic acid) in the presence of an ammonia-ammonium chloride buffer (pH ≈ 10). Here, free $Zn^{2+}$ ions form a stable 1:1 complex with EDTA and the effects of other metal ions can be masked. A suitable metallochromic indicator, most commonly Eriochrome Black T (EBT), is added to the solution. In the absence of EDTA, EBT forms a poorly stable, red-coloured complex with $Zn^{2+}$. During titration, EDTA displaces the indicator from this complex because of its stronger binding affinity for $Zn^{2+}$, resulting in a distinct colour change at the endpoint from red (metal-indicator complex) to blue (free indicator). Thus, complete deprotonation with EDTA is achieved and the stable $[ZnY]^{2-}$ complex ($\log K_f \approx 16.5$) is formed as in Equation 1.

$$Zn^{2+}\,(aq) + Y^{4-}\,(aq) \rightarrow [ZnY]^{2-}(aq) \quad (1)$$

In light of this information, the determination of zinc was made by following the procedure below. During the dissolution process, 10 mL of the samples were withdrawn and filtered at certain time periods and approximately 50 mL of pure water, 10 mL of buffer solution and a few drops of Eriochromblack T indicator were added. $Zn^{2+}$ was then determined using the volumetric method using a 0.1 molar solution of EDTA. Zinc content was calculated from EDTA consumption.

$$Zn = 65.40 \times M_{EDTA} \times V_{EDTA}\ (mL) \quad (2)$$

$M_{EDTA}$ = molarity of EDTA solution ($moL.L^{-1}$)

$V_{EDTA}$ = EDTA solution used (mL)

Conversion fraction,

$X_{Zn}$ = Amount of $Zn^{2+}$ in sample / Amount of $Zn^{2+}$ in solid

Basically, the following reactions can occur in the dissolution of lactic acid in water:

$$2C_3H_6O_{3\,(aq)} + 2H_2O_{\,(l)} \longleftrightarrow 2H_3O^+_{\,(aq)} + 2C_3H_5O_3^-{}_{\,(aq)} \quad (3)$$

$$ZnCO_{3\,(s)} + 2H_3O^+_{\,(aq)} \longleftrightarrow Zn^{+2}{}_{(aq)} + CO_{2\,(g)} + 3H_2O_{\,(l)} \quad (4)$$

The following are the smithsonite ore dissolving reactions:

$$ZnCO_{3\,(s)} + 2C_3H_6O_{3\,(aq)} \longrightarrow Zn^{+2}{}_{(aq)} + 2C_3H_5O_3{}^{-1}{}_{(aq)} + H_2O_{\,(l)} + CO_{2\,(g)} \quad (5)$$

As described previously, in a design process, the Taguchi approach can successfully cut down on the number of tests needed. Therefore, in this study, the Taguchi approach was chosen to design the experimental system. For the experimental design, parameters were chosen that are thought to affect the reaction efficiency, such as temperature, solid/liquid ratio, particle size, lactic acid concentration and reaction time [30]. Preliminary experiments were used to determine experimental parameters and their levels, as indicated in Table 2.

***Table 2.*** *Parameters and levels used in optimization*

| | Parameters | Levels | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 |
| A | Acid concentration (M) | 0.25 | 0.5 | 0.75 | 1 |
| B | Reaction time (min) | 10 | 20 | 30 | 50 |
| C | Temperature (°C) | 20 | 30 | 40 | 50 |
| D | Solid/liquid ratio (g $mL^{-1}$) | 0.01 | 0.02 | 0.04 | 0.06 |
| E | Particle size (μm) | -160.5 | -283.5 | -477.5 | -890 |

The orthogonal array experimental design proposed by Taguchi can be used to investigate the effects of numerous parameters on the performance characteristics in a series of experiments. In Taguchi method, five different parameters were under investigation, and for each parameter, four different levels had been determined. The experiments were carried out using the $L_{16}(4^5)$ OA experimental design plan. The order of the experiments was placed in columns OA, $L_{16}$ $(4^5)$, which were selected as the experimental plan of the parameters, and the average Zn (%) values obtained from the experiments were given in Table 3. To prevent unanticipated noise sources that could emerge during the experiment and negatively impact the results, the order of the experiments was randomly assigned. The internal effects of the parameters with each other are not considered in the theoretical analysis because some initiative experiments show that they can be unregarded. The validity of this supposition was controlled by validation experiments under optimal conditions.

***Table 3.*** *$L_{16}$ $(4^5)$ experimental plan and average Zn (%)*

| Experiment No | Parameters and levels | | | | | Average Zn (%) |
|---|---|---|---|---|---|---|
| | A | B | C | D | E | |
| **1** | 1 | 1 | 1 | 1 | 1 | 51.32 |
| **2** | 1 | 2 | 2 | 2 | 2 | 48.28 |
| **3** | 1 | 3 | 3 | 3 | 3 | 24.14 |
| **4** | 1 | 4 | 4 | 4 | 4 | 16.49 |
| **5** | 2 | 1 | 2 | 3 | 4 | 38.28 |
| **6** | 2 | 2 | 1 | 4 | 3 | 28.66 |
| **7** | 2 | 3 | 4 | 1 | 2 | 77.11 |
| **8** | 2 | 4 | 3 | 2 | 1 | 78.15 |

*Table 3. continued…*

| | | | | | | |
|---|---|---|---|---|---|---|
| **9** | 3 | 1 | 3 | 4 | 2 | 42.79 |
| **10** | 3 | 2 | 4 | 3 | 1 | 65.96 |
| **11** | 3 | 3 | 1 | 2 | 4 | 53.81 |
| **12** | 3 | 4 | 2 | 1 | 3 | 69.87 |
| **13** | 4 | 1 | 4 | 2 | 3 | 79.45 |
| **14** | 4 | 2 | 3 | 1 | 4 | 81.57 |
| **15** | 4 | 3 | 2 | 4 | 1 | 72.31 |
| **16** | 4 | 4 | 1 | 3 | 2 | 71.65 |

The performance statistic is selected as the optimisation criterion. The S/N calculation was performed to maximise the zinc lactate contents in solution to use the S/N ratio (highest, best performance characteristic) for optimum performances. Using the following Eq. 6, the S/N performance statistics were assessed. [31]:

$$S/N = -10\log(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{Y_i^2}) \quad (6)$$

Here, S/N is the performance statistics, n is the number of repetitions done for an experimental combination, and $Y_i$ is the performance value of ith experiment. It's possible that the Taguchi method may not have been performed the experimental circumstances that corresponded to the optimum working conditions throughout the whole experimental phase. In these situations, the balanced characteristic of the OA can be utilized to determine the performance value corresponding to the ideal operating conditions using the $Y_i$ equation. For this, the $Y_i$ equation is given in Eq. 7. [32]:

$$Y_i = \mu + X_i + e_i \quad (7)$$

In equation 5, $e_i$ represents the experiment's random error, $X_i$ represents the constant influence of the parameter combination employed, and μ represents the overall average of the performance values. The confidence interval must be assessed because this equation is a point estimate that is computed using experimental data to assess the significance of validation experiment outcomes. Equation 6 can be used to get the chosen error level's confidence interval. [33]:

$$CI = Y_i \pm \sqrt{F_{\alpha;1,DF_{MSe}}\, MSe(\frac{1+m}{N}+\frac{1}{n_i})} \quad (8)$$

F is the value obtained from table F, $DF_{MSe}$ is the mean squared error's degree of freedom, m is the number of degrees of freedom used to estimate $Y_i$, N is the total number of experiments, $n_i$ is the verification experiment's number of repeats, and α is the error level. Before assessing Eqs. (7) and (8), the following equation should be used to apply the omega transformation of percentage data when the experimental data are expressed as a percentage (%). Equation 9 is then used to carry out the inverse transformation and identify the pertinent values. [34]:

$$\Omega(\text{db}) = -\log(\frac{1}{P} - 1) \quad (9)$$

where the decibel value of the percentage value that is subject to omega transformation is represented by Ω (db), and P is the proportion of the product determined experimentally.

## Results and Discussion

## Definition of the Optimum Conditions

The optimization experiments of $L_{16}(4^5)$ were performed according to the Taguchi method and the effect of each parameter was determined using the Minitab 18 package program. For example, Table 3 demonstrates that the experiments for which column A equals 1 are those with experiment numbers 1, 2, 3, and 4. As a result, the first data point's performance statistics value is the mean of the results from experiments 1, 2, 3, and 4. The conditions of the experiments for which column A is 2 (i.e., experiments with numbers 5, 6, 7, and 8), show the experimental conditions for the second data point. The value in number of the highest point on each graph in Table 4 indicates the optimal value of each parameter. The obtained results are plotted in Figures 2-6. The degree to which parameters affect performance statistics is determined by selecting the highest points from the graphs. In Figure 2, for instance, which illustrates how performance increases with acid concentration, the highest value of the acid concentration is seen to be 1 M and level 4. The experiment that corresponds to optimal conditions (A: 4, B: 2, C: 2, D: 1, E: 1, in Table 4) is not conducted throughout the experimental study, as can be shown from the experimental plan provided in Table 3. Therefore it should be mentioned that the dissolution percentage in Table 6 is an anticipated result by using on equations (6-9).

***Table 4.*** *Response table for signal/noise ratios*

| **Levels** | **Acid Concentration** | **Reaction Time** | **Temperature** | **Solid/Liquid Ratio** | **Particle Size** |
|---|---|---|---|---|---|
| 1 | 29.96 | 34.12 | 33.76 | 36.77 | 36.41 |
| 2 | 34.10 | 34.36 | 34.85 | 36.03 | 35.28 |
| 3 | 35.12 | 34.29 | 34.09 | 33.20 | 32.92 |
| 4 | 37.63 | 34.04 | 34.11 | 30.82 | 32.20 |
| Delta | 7.67 | 0.32 | 1.08 | 5.95 | 4.20 |
| Rank | 1 | 5 | 4 | 2 | 3 |

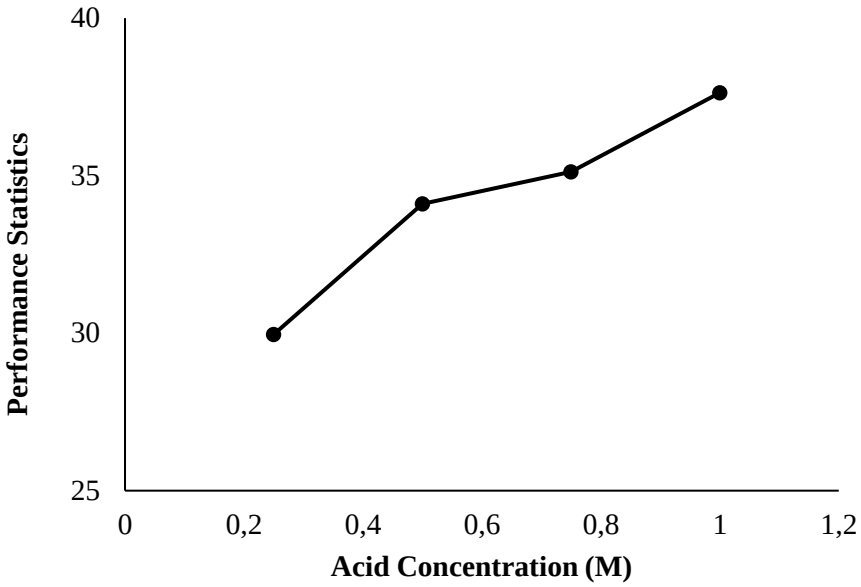



***Figure 2.*** *The effect of acid concentration on the optimization*

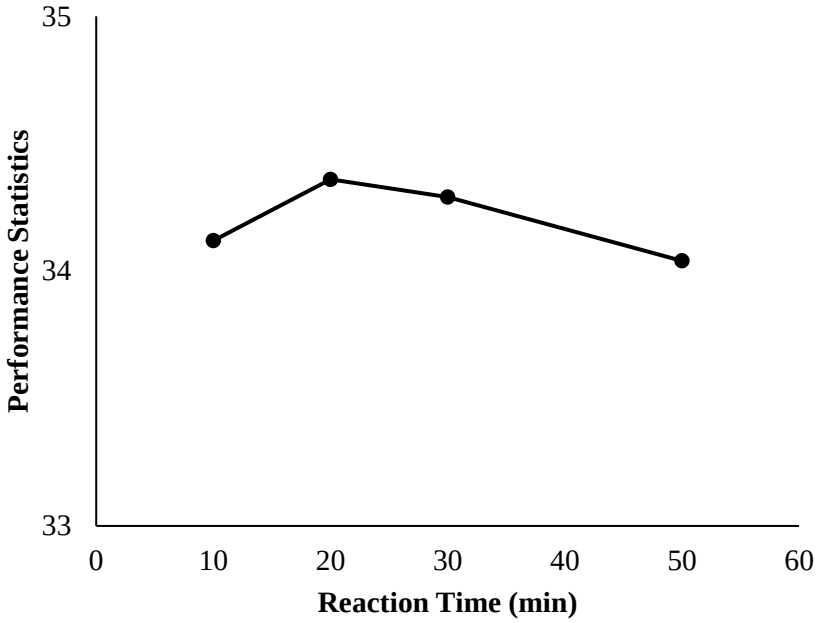



***Figure 3****. The effect of reaction time on the optimization*

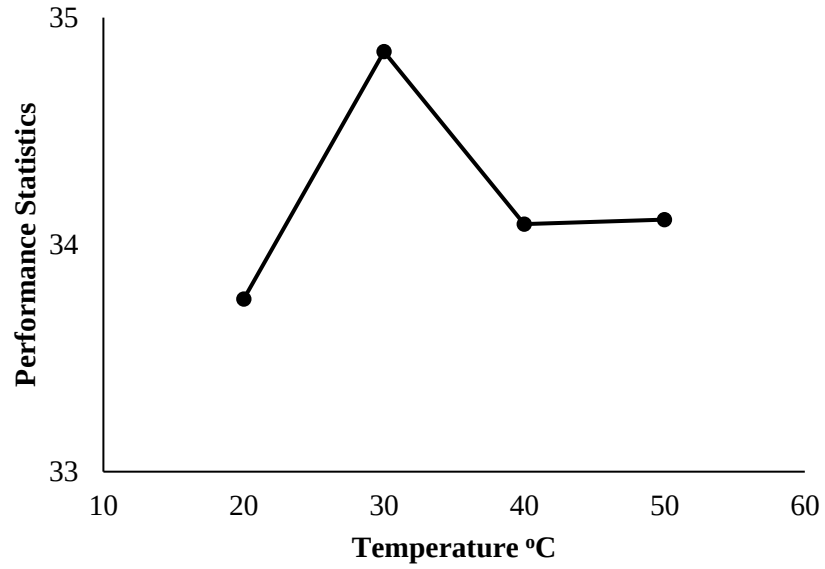



***Figure 4.*** *The effect of temperature on the optimization*

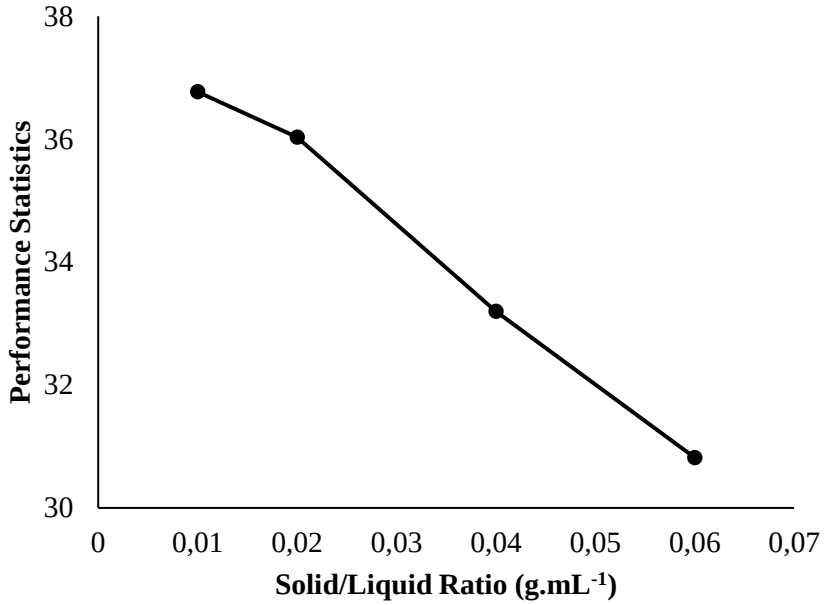



***Figure 5****. The effect of solid/liquid ratio on the optimization*

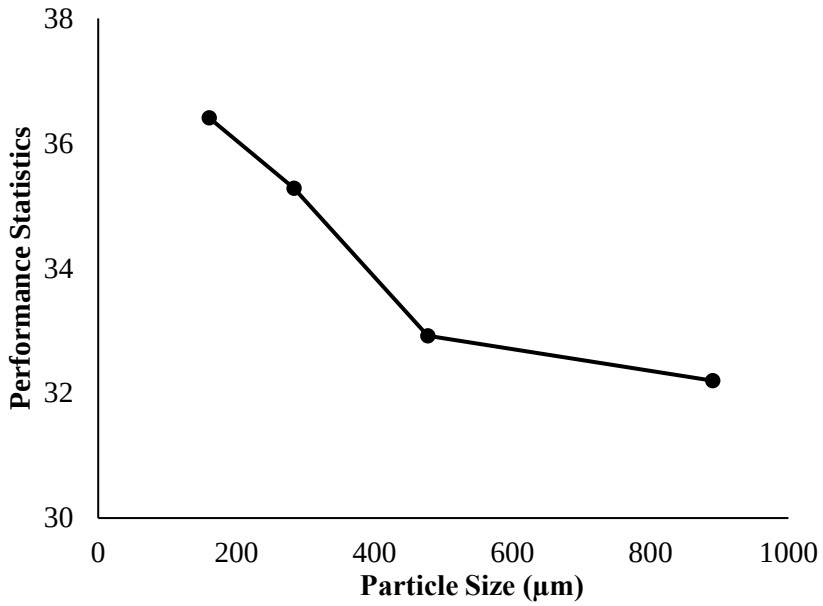



***Figure 6.*** *The particle size effect on the optimization*

## Analysis of Variance

The performance characteristics and the statistical analysis of variance (ANOVA) analysis can be used to predict the optimum set of process parameters. ANOVA examines effects such as degrees of freedom, sum of squares, variance and percentages. Table 5 shows the findings of the ANOVA analysis for the relative probability of the parameters. Each process parameter's F-value reveals which parameter significantly affects the results. Parameter examples are determined by comparing the experimentally found $F_{exp}$ value with the values in the $F_{Tab}$ table. If $F_{Exp}>F_{Tab}$, the relevant parameter is active, otherwise the parameter is inactive. The F ratio was found to be 9.28 by reading from the 95% confidence interval table. The experimental results can be considered to be within ±5% error range. This shows that the interaction effects of the parameters are actually minimal. F values are calculated as 469.63 for acid concentration, 9.64 for temperature, 342.99 for solid/liquid ratio and 178.8 for particle size. Since F values are greater than 9.28, it can be said that acid concentration, temperature, solid/liquid ratio and particle size are effective on dissolution. Since there is not much change in the reaction time, it is not effective. When the F values among the effective parameters are compared according to their sizes, the most effective parameters are acid concentration and solid/liquid ratio. In addition, the P value being below 0.05 is statistically significant and being zero strengthens the meaning that it is very effective on the process.

***Table 5.*** *The analysis of variance (ANOVA)*

| **Parameters** | **df (SD)** | **% Contribution** | **SS** | **MSS** | **F-ratio** | ***P*-Value** |
|---|---|---|---|---|---|---|
| Acid concentration (M) | 3 | 46.81 | 122.436 | 40.8119 | 468.63 | 0.000 |
| Reaction time (min) | - | - | - | - | - | - |
| Temperature (°C) | 3 | 0.96 | 2.519 | 0.8398 | 9.64 | 0.047 |
| Solid/liquid ratio ($g.mL^{-1}$) | 3 | 34.26 | 89.613 | 29.8708 | 342.99 | 0.000 |
| Particle size (μm) | 3 | 17.86 | 46.715 | 15.5718 | 178.80 | 0.001 |
| Error | 3 | 0.10 | 0.261 | 0.0871 | | |
| Total | 15 | 100.00 | 261.54 | | | |

F (0.05, 2, 9) 0.95 = 9.28

According to the optimization results of the dissolution of smithsonite ore in lactic acid solutions, the optimum working conditions obtained by considering the largest and best cases from the graphs drawn were found as A: 4, B: 2, C: 2, D: 1 and E: 1 (These conditions; Acid concentration 1 molar (A), Reaction time 20 minutes (B), Temperature 30 °C (C), Solid/Liquid ratio 2/200 (D), Particle size-160.5 μm (E). The optimum levels of the parameters and the numerical values of these levels were given in Table 6. After determining the optimum working conditions, it was determined that 98.85% of the smithsonite ore obtained under these conditions was dissolved.

- These results obtained from the ANOVA analysis clearly reveal the effects of the parameters examined in the study (acid concentration, temperature, solid/liquid ratio, grain size) on the dissolution efficiency of smithsonite ore in lactic acid. On the other hand, the fact that the interactions between the parameters are minimal in the experimental system is supported by the following explanations.
- Approximately 99.9% of the total variance is explained by the main effects, and the error term makes only a 0.1% contribution. If there were significant binary or multiple interactions in the system, it would be expected to appear as an increasing error variance since these effects are not reflected in the model. However, the fact that the error variance is this low shows that the interaction terms are small enough to be ignored on the dissolution efficiency.
- The fact that only the main effects are included in the ANOVA table within the scope of the analysis shows that the model is not established in a direct factorial or full interaction structure. However, the high explanatory power of the model and the fact that the predicted dissolution rate (close to 100%) is very close to the experimental value (98.85%) indicate that this modeling provides sufficiently reliable results. In this context, even if the interaction terms were not included in the model, there was no significant gap in the results. This confirms that the interactions play a secondary role in the system behavior.

- The chemical effects of the dissolution parameters are quite distinct and are not complementary to each other, but rather independent contributors. This situation also supports chemically why the interactions are weak.
- The fact that all *P*-values of the parameters are below the value of 0.05, which is considered statistically significant, shows that the effect of the parameters on the dissolution reaction is revealed with high confidence. If the effects between the parameters were significant, the significance of the parameters in the model would decrease and the *P*-values would increase. However, the fact that such a situation is not observed again supports that the interactions between the parameters are minimal.

***Table 6.*** *Optimum operating conditions and dissolution percentage*

| **Parameters** | | **Optimum Levels** | **Optimum Values** |
|---|---|---|---|
| A | Acid concentration (M) | 4 | 1 |
| B | Reaction time (min) | 2 | 20 |
| C | Temperature ($^oC$) | 2 | 30 |
| D | Solid/liquid ratio ($g.mL^{-1}$) | 1 | 2/200 |
| E | Particle size (μm) | 1 | -160.5 |
| Estimated dissolution quantity (%) | | 100 | |
| Estimated Confidence Interval (%) | | 96.95 – 100 | |
| Dissolution percentage (%) | | 98.85 | |

## Conclusion

According to the Taguchi approach, a product or process should be designed in a way that is applicable to noise factors or variables that may occur before, during or after production. Therefore, the Taguchi method is widely used in determining optimum operating conditions. The optimum conditions for dissolving smithsonite ore in lactic acid solutions were found using the Taguchi $L_{16}$ design. Particle size, solid/liquid ratio, temperature, reaction time and acid concentration were selected as the factors influencing dissolution. The parameter levels that maximized the S/N value were determined to be acid concentration 1 molar, reaction time 20 minutes, temperature 30$^oC$, solid/liquid ratio 2/200 $g.mL^{-1}$, particle size -160.5 μm. It was determined that 98.85% of the smithsonite ore obtained under these conditions was dissolved. When ANOVA analysis was performed for the selected parameters, it was determined that the most effective parameters according to F values were acid concentration, solid/liquid ratio and particle size, respectively. It was found that the solubility increased with the increase in temperature and with the decrease in solid/liquid ratio and particle size. In this study, optimum conditions in the laboratory environment produced by the Taguchi method can also be applied to real production conditions. Therefore, these results obtained can also contribute to the industrial production of zinc lactate from smithsonite ore.

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***Authors Contribution*** Authors contributed equally to the study. All authors read and approved the final manuscript

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