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The Impact of Different Initial Concentrations of Co, Fe, Mn, and KMnO4 on the Efficiency of Cobalt Removal from Zinc Electrolyte Solution

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Abstract

Removal of cobalt from zinc electrolyte solution is one of the most important and difficult steps in zinc production using hydrometallurgy method. The impact of initial concentrations of cobalt, manganese, and Fe and amounts of potassium permanganate on the efficiency of cobalt removal by potassium permanganate from zinc electrolyte solution was investigated in this research. The results indicated that the higher the initial concentration of cobalt, manganese, and Fe is, the lower the cobalt removal amount will be; however, as the amount of potassium permanganate increases, the efficiency of cobalt removal will enhance. It was also found that, in order to make a permissible level of the dissolved cobalt, the consumption of potassium permanganate should be increased as the concentration of cobalt, Fe, and manganese increases. If the concentration of manganese is more than 500 mg/L, it can impact the reduction of the efficiency of cobalt removal to a great extent; but when the initial concentration of cobalt is high, the significance of the impact of the initial amounts of manganese would decrease. Additionally, if the manganese concentration is less than 200 mg/L, the optimal removal of cobalt (less than 2 ppm) will not occur under any circumstances. The results also indicate that if the potassium permanganate concentration is 1 g/L or lower, the Fe ions in the solution will drastically reduce the cobalt removal efficiency.

Keywords
Synthetic Solution, Cobalt Removal, Manganometry Method, Design Expert Method, Optimization.

1. INTRODUCTION

In addition to reducing the quality and purity of the final product, metal impurities such as cobalt existing in the zinc electrolyte solution can prevent the formation of zinc sheet on the cathode surface [1- 4]. Therefore, the cobalt purification process is highly significant. The zinc industry currently applies two types of Cu-Sb and Cu-As additive combinations to activate the cobalt cementation process across the world [4- 7]. In general, most of the zinc factories dealing with high concentrations of cobalt in zinc electrolyte solution (above 15-20 mg/L) apply Cu-As as an additive which seems more appropriate for high concentrations of cobalt [8- 10]. Furthermore, compared to the Cu-Sb activation system, the zinc powder consumption in Cu-As activation system is lower [3,9-11]. However, the Cu-Sb is preferred to Cu-As in usage due to its lower environmental risks. Arsenic trioxide was previously used in Iran to remove cobalt, but the project of using arsenic trioxide failed due to the production of arsenic gas (AsH3) which is extremely toxic and deadly and also because the Iranian factories are unable to properly ventilate it. Potassium permanganate is currently used to remove cobalt in Iran [12, 13]. In this method, at the temperature of 85°C and based on the reactions 1 to 3, permanganate ions ($MnO4^-$) are used and the cobalt ion (II) is oxidized to cobalt (III). Then, cobalt (III) is precipitated as cobalt hydroxide (Co(OH)3) by adding some lime in a heat treatment reactor [14, 15].

$$MnO_4^- + 3Mn^{2+} + 2H_2O \rightarrow 4MnO_2 + 4H^+ \quad (1)$$
$$MnO_4^- + 4H^+ + 3Co^{2+} \rightarrow MnO_2 + 3Co^{3+} + 2H_2O \quad (2)$$
$$2Co^{3+} + 3Ca(OH)_2 \rightarrow 2Co(OH)_3 + 2Ca^{2+} \quad (3)$$

Zinc producing plants in Iran are supplied from various sources that have different percentages of impurities such as cobalt, nickel, Fe, cadmium, manganese, etc. For this reason, the concentration of impurities in zinc electrolyte solution input to the process may be different several times during the year and each time the appropriate operating circumstances must be provided for cobalt removal in the heat treatment reactor by trial and error, which may lead to waste of time and money. Important ions, which affect the efficiency of the manganometric method, include the concentrations of cobalt, manganese, permanganate, and Fe [16-19].

Unfortunately, the process of using potassium permanganate has been conducted in a completely experimental way in Iran and no serious research has ever been carried out in this field. Hence, the impact of the concentration of cobalt, manganese,

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and iron ions as well as the added amount of potassium permanganate is investigated in this research. For a proper statistical analysis and appropriate reduction of experiments in this paper, the Design Expert software was used to determine the dependence of the efficiency of cobalt removal process on the initial concentration of cobalt, manganese, and iron and its relationship with the required amount of potassium permanganate.

2. EXPERIMENTAL

2.1. Design of Experiments

Table 1 illustrates the variables and their levels by the central composite design method and specifies the type of points. Increasing the number of points ramps up the estimation power and defines the curvature properties better. Each factor is varied in five levels in the central composite design method: lower axial, lower factorial, central, upper factorial and upper axial. It should be noted that this method is different from the five-level factorial one because 25 (5^2) tests are required for two factors in the five-level method. However, the central composite design method is a two-level factorial method to which the central and axial points are added so that the curvature behavior in the model can be well-defined. Similarly, if the factorial approach is used in this experiment to find the optimal point, the number of levels, 5, to the 5rd power (4 factors) will be equal to 1,024 tests. This number drops to 30 tests based on the response surface model, which is very important in terms of economic cost and reduced time of experiments.

2.2. Materials and Methods

One-time-distilled water was used for solubilization and dilution. The potassium permanganate industrial grade used in this paper was prepared by Kimia Manganese Factory, Zanjan. Sulfuric acid (Merck) and industrial lime were used to adjust the pH. Cobalt sulfate, Manganese, Fe and Zinc (Merck) were also used to make a synthetic solution in this research.

2.3. Machinery Applied

All experiments were performed in a two-liter glass reactor. Optimal temperature conditions were provided for the experiments using a Heater-Stirrer (Heidloph MR 3001 K). The pH of the make-up solution and its temperature were adjusted by a pH meter by (WTW multi 9310 model) and they were controlled during the experiments. Finally, the amount of the desired metals were analyzed and measured by the AA 240 atomic absorption spectrometer (Varian, Australia) in this research.

2.4. Methodology

Designing experiments was based on initial concentrations of 0-140 mg/L for cobalt, 0-100 mg/L for Fe, 0-1000 mg/L for manganese and 0-2 g/L for potassium permanganate in the zinc sulfate solution at concentration of 85 g/L. The analyses designed by the software are presented in Table 2. According to Table 2, solubilization was conducted for each experiment and one liter of synthetic solution with an initial pH of 4.5 was poured into a glass reactor by creating a concentration of each of the parameters and heated by a heater to reach the desired temperature. Then, certain amounts of potassium permanganate were added to the solution and it was mixed as required by the stirrer and adjusting pH was made by lime-water. After the process time and a proper mixing, the mixture was filtered. Then, the sub-filtration solution was sampled and the final amount of cobalt was measured by an atomic absorption spectroscopy. The removal results by atomic absorption are also presented in Table 2. The removal percentage for each sample was calculated according to the initial concentration of cobalt in that sample in accordance with Equation 4.

$$\text{Removal percentage} = \frac{\text{Initial concentration of cobalt } - \text{secondary concentration of cobalt}}{\text{Initial concentration of cobalt}} \times 100 \tag{4}$$

Table1. Independent variables and their levels for the central composite design

Code	Variable	Units	Type	Low actual	High actual	Low coded	High coded
A	Co	mg/L	Numeric	35	105	-1.000	1.000
B	Fe	mg/L	Numeric	25	75	-1.000	1.000
C	Mn	mg/L	Numeric	250	750	-1.000	1.000
D	MnO_4	g/L	Numeric	0.5	1.5	-1.000	1.000

Table 2 . Analyses designed by Design Expert software

No	Co	Fe	Mn	$KMnO_4$	Co removal percentage
1	70	50	500	1	82
2	105	25	250	1.5	98
3	0	50	500	1	100
4	70	50	500	2	98.34
5	70	50	500	1	81

6	35	25	250	1.5	96.46
7	35	25	250	0.5	72.86
8	70	50	500	0	0
9	70	50	500	1	83.2
10	70	50	1000	1	49.57
11	35	75	750	0.5	30.57
12	70	50	500	1	82.64
13	105	25	250	0.5	55.9
14	70	50	0	1	79.75
15	35	75	250	0.5	68
16	105	75	250	1.5	89
17	35	75	750	1.5	99.5
18	105	25	750	1.5	96.47
19	35	25	750	1.5	99.5
20	105	25	750	0.5	53.52
21	105	75	750	0.5	24.2
22	105	75	750	1.5	77
23	140	50	500	1	42.71
24	70	50	500	1	82.71
25	35	25	750	0.5	39.14
26	35	75	250	1.5	99.5
27	70	50	500	1	82.57
28	70	100	500	1	76.14
29	70	0	500	1	92
30	105	75	250	0.5	27.42

3. RESULT AND DISCUSSION

Statistical evaluation of Equation 5 was made by performing F-test and ANOVA analysis of variance and the result is presented in Table 3. Enova data confirms the accuracy of this quadratic model. With regard to the first-order model, it can be seen that the p value of the quadratic model is less than 0.0001, indicating that the model is significant (generally, the p values less than 0.05 in the model indicate that the model is significant). The F-parameter is also a criterion of the deviation of the data from the mean value. In general, for a model that successfully predicts the test results, the F-value is typically high. The F is 34.27 for cobalt in this model which indicates that the model is completely significant. Furthermore, a great R^2 of 0.9697 confirms the high accuracy of the established model (it is recommended to determine the regression coefficient of determination - R^2 should be higher than 0.80). In addition, the predicted correlation coefficient (Pred- R^2) and the adjusted correlation coefficient (Adj- R^2) are also within a reasonable range (if the difference between the Pred-R^2 and Adj-R^2 values is less than 20%, the developed model can be considered authentic).

$$Co = +12.33 +16.05A +4.84B +3.58C -16.82D +5.44AB -0.2 AC -9.02AD +0.91BC - 2.31BD - 1.12CD+6.13A2 -1.11B2 +2.29C2 +4.47D2 \quad (5)$$

Table 3. The results of the analysis of variance (ANOVA) table of the model fitted to the response data

Source	Sum of squares	Mean square	F value	P-value
Model	2394.52	119.73	40.84	0.0001
Model	17345.76	1238.98	34.27	0.0001
A-Co	6183.42	6183.42	171.04	0.0001
B-Fe	561.54	561.54	15.53	0.0013
C-Mn	307.81	307.81	8.51	0.0106
D-MnO_4	6792.93	6792.93	187.90	0.0001
AB	472.74	472.74	13.08	0.0025
AC	0.82	0.82	0.023	0.8820
AD	1302.67	1302.67	36.03	0.0001
BC	13.16	13.16	0.36	0.5553
BD	85.15	85.15	2.36	0.1457
CD	20.05	20.05	0.55	0.4680
A^2	1030.58	1030.58	28.51	0.0001
B^2	33.66	33.66	0.93	0.3499
C^2	143.64	143.64	3.97	0.0647
D^2	549.20	549.20	15.19	0.0014
R-Squared	0.9697			
Adj R-Squared	0.9414			
Pred R-Square	0.8257			
Std. Dev.	6.01			
Mean	21.76			
C.V.%	27.63			

Fig. 1 illustrates the interaction of initial concentrations of cobalt and manganese and its effect on the efficiency of cobalt removal from zinc sulfate solution by potassium permanganate. As shown in Fig. 1, the percentage of cobalt removal will decrease as the initial concentration of cobalt and manganese increases in the presence of 1 g/L of potassium permanganate. Due to the reaction of permanganate with cobalt and manganese, as the number of these ions increases in the solution, higher amount of permanganate will be required for optimal removal of cobalt. When the concentration of cobalt is high in zinc sulfate solution, the initial concentration of manganese would have little impact on the cobalt removal percentage. Additionally, when the concentration of manganese is higher than 500 mg/L, the percentage of cobalt removal will be low even in the initial low concentrations of cobalt; however, at concentrations below 500 mg/L of manganese ions, the cobalt removal will be well performed. In fact, high concentrations of manganese are a disturbing factor for the optimal removal of cobalt. As the concentration of manganese increases, the permanganate ions will react more with the manganese and the oxidation of cobalt will not be conducted well by the permanganate ions. For this reason, the overall percentage of cobalt removal will reduce.

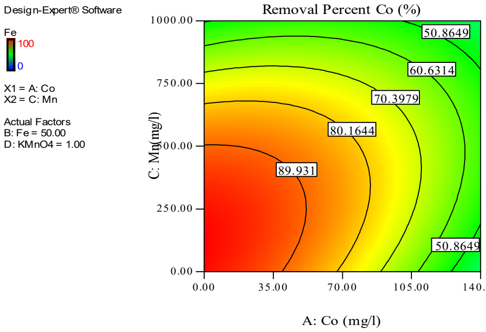

Fig. 1. Mutual effect of initial Co and Mn concentrations on cobalt removal efficiency.

Fig. 2 illustrates the interaction of initial concentrations of cobalt and iron and its effect on the efficiency of cobalt removal from zinc sulfate solution. According to Fig. 2, the percentage of cobalt removal from the solution will decrease as the iron and cobalt increase simultaneously. The lower the cobalt concentration is, the lower the impact of iron ions on the efficiency of cobalt removal will be. In fact, different concentrations of iron will not disturb the cobalt removal when the initial concentration of cobalt in solution is less than 70 mg/L in the presence of 1 g/L potassium permanganate. But when the cobalt ions concentration in the solution exceeds 70 mg/L, the percentage of cobalt removal will decrease as the iron ions concentration in the solution increases, and part of the permanganate used is applied only to remove the iron ions. According to Figure 2, it has a direct impact on the cobalt removal and can lead to a reduction in the process efficiency. Iron ions have no impact on cobalt removal at concentrations lower than 25 mg/L and proper removal also takes place at the highest initial concentration of cobalt ions.

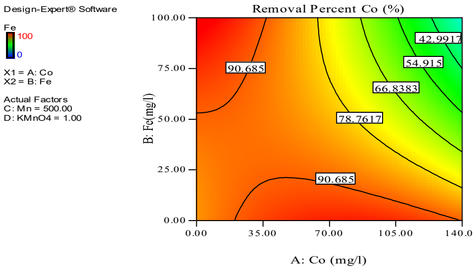

Fig. 2. Mutual effect of initial cobalt and Fe concentrations on cobalt removal efficiency.

According to Fig. 3, the interaction of the initial concentration of cobalt ions and potassium permanganate ions and its effect on the efficiency of cobalt removal from zinc sulfate solution is presented. As seen in Figure 3, with the increase of the concentration of potassium permanganate, the cobalt removal efficiency will sharply increase; however, by increasing the initial concentration of cobalt ions in the feed the cobalt removal efficiency would decrease. The results indicate that the increase in potassium permanganate has a direct effect while the initial cobalt ions have an inverse impact on increasing the percentage of removal of cobalt in the solution. In fact, the higher the concentration of cobalt ions and the lower the concentration of potassium permanganate in the solution are, the lower the percentage of cobalt removal will be. When 2 g/L of potassium permanganate is added to the solution, the increase in the initial concentration of cobalt has little impact on the percentage of its final removal, and the optimal removal takes place in almost all concentrations; however, when the concentration of potassium permanganate added to the solution is less than 1 g/L, no optimal removal can be observed even in the initial low concentrations of cobalt which indicates the concentration of potassium permanganate is the most important factor in removing the cobalt. Forming manganese dioxide, oxidation of cobalt and absorbing it in the structure of manganese dioxide, permanganate leads to a high analytical grade of cobalt-free zinc sulfate solution.

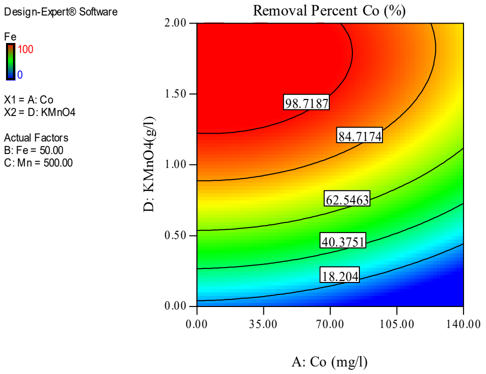

Fig. 3. Mutual effect of initial concentrations of cobalt and potassium permanganate on cobalt removal efficiency.

According to Fig. 4, the removal of cobalt in the presence of manganese and Fe by potassium permanganate was investigated. As illustrated in Fig. 4, increasing the concentration of manganese and Fe would lead to a decrease in the percentage of cobalt removed by potassium permanganate. The results indicate that, at a constant concentration of manganese, the efficiency of cobalt removal reaction decreases as Fe concentration increases; at a constant concentration of Fe, however, as the initial concentration of manganese ions increases, it can be observed that first the percentage of cobalt removal has an increasing trend and then it decreases. As the initial concentration of Fe increases, the decreasing trend of cobalt removal efficiency at low concentrations of manganese will be quite evident. This indicates that when the concentration of Fe ions in the environment is low, the cobalt removal efficiency will decrease less as the manganese concentration increases.

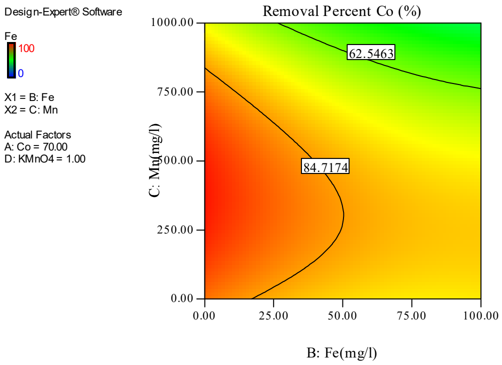

Fig. 4. Mutual effect of initial concentrations of Fe and manganese on cobalt removal efficiency.

As illustrated in Fig. 5, the interaction of initial concentrations of Fe and potassium permanganate on the removal of cobalt ions is presented. The initial concentration of cobalt was 70 mg/L in this study. According to Figure 5, the removal of cobalt increases sharply at all concentrations of Fe in the solution and by increasing the concentration of potassium permanganate. In fact, the Fe ions have no impact on the process efficiency at concentrations above 1 g/L of potassium permanganate and the optimal removal of cobalt is conducted even at the highest concentration of Fe; but the cobalt removal efficiency would decrease as concentration of Fe in solution increases at concentrations below 1 g/L potassium permanganate.

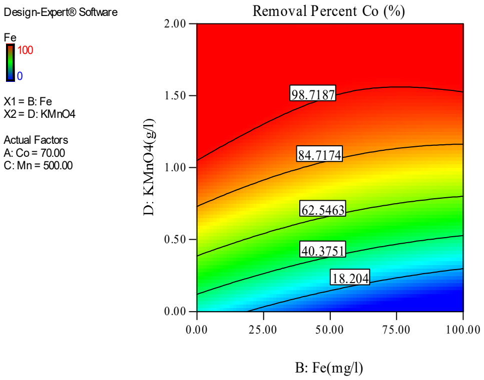

Fig. 5. Mutual effect of initial concentrations of Fe and potassium permanganate on cobalt removal efficiency

Fig. 6 indicates the interaction of the initial concentration of manganese ions with potassium permanganate and its effect on the efficiency of cobalt removal in zinc sulfate solution. According to Figure 6, as the concentration of manganese is below 250 mg/L or over 750 mg/L, there will be a slight decrease in cobalt removal efficiency with an increase in the potassium permanganate concentration. In fact, the optimal limit for manganese to remove the cobalt is in the range of 250-750 mg/L; however, the cobalt removal efficiency will decrease as the manganese concentration increases at low concentrations of potassium permanganate. Figure 6 illustrates that cobalt removal occurs even in the absence of manganese due to the direct reaction of permanganate with cobalt which leads to the production of manganese dioxide; the maximum removal percentage of cobalt, however, is about 80% in the absence of manganese that indicates the effective role of manganese for an optimal and close to 100% removal of cobalt. Figure 6 also illustrates that if the concentration of manganese is lower than about 200 mg/L, the removal of cobalt becomes close to 100 at no concentration of potassium permanganate and it is necessary to add manganese ions to the solution for the thorough removal of cobalt.

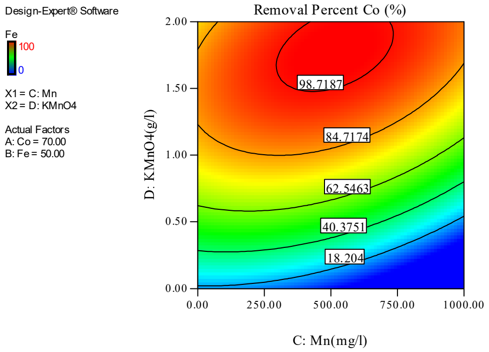

Fig. 6. Mutual effect of initial concentrations of manganese and potassium permanganate on the efficiency of cobalt removal.

In conclusion, it should be mentioned that many zinc factories in Iran suffer from a high concentration of impurities, including cobalt, due to the low quality of mine soil. Unfortunately, in many important Iranian zinc industry companies, the filtrate solution prepared for the hot refining stage usually contains about 100 ppm cobalt. Calcimin company, the largest zinc factory in Iran, which is fed from the low grade soil of Angoran mine, has not experienced cobalt less than 100 ppm in recent years. A review of sources shows that zinc factories in the world experience a much lower concentration of cobalt in the hot refining stage [20-24]; Therefore, it is possible that common methods of cobalt purification, such as cementation with arsenic trioxide or antimony trioxide as surface activators in addition to zinc powder, will not be effective in removing cobalt from leach filtrate solution. Numerous practical experiences also confirm this [25]. Also, regarding the optimal use of potassium permanganate to remove cobalt and its relationship with other impurities, no related article has been presented at the domestic and international level until now; Although each industrial unit may have a special strategy based on its own experiences; So that the use of potassium permanganate is optimized as much as possible.

4. CONCLUSION

The impact of initial concentrations of effective ions in the zinc electrolyte solution on cobalt removal process by potassium permanganate was investigated in this study. Cobalt and potassium permanganate concentrations have the greatest impact and the initial concentration of Fe and manganese ions has the least impact on cobalt removal from zinc sulfate solution. The presence of high concentrations of cobalt, manganese, and Fe greatly reduces the process efficiency, but low concentrations of Fe have less impact on cobalt removal and amounts less than 200 mg/L of manganese cannot help the optimal removal of cobalt. Therefore, the best concentration of manganese for an optimal removal of cobalt is between 200-500 mg/L. Furthermore, the lower is the initial amounts of cobalt and Fe, the better it will be, and the higher is the initial amount of potassium permanganate added to the process, the higher the cobalt removal efficiency would be.

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تأثیر غلظت‌های اولیه مختلف کبالت، آهن، منگنز و پرمنگنات پتاسیم بر راندمان حذف کبالت از محلول الکترولیت روی

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چکیده

حذف کبالت از محلول الکترولیت روی یکی از مهمترین و دشوارترین مراحل تولید روی با استفاده از روش هیدرومتالورژی است. در این تحقیق، تاثیر غلظت اولیه کبالت، منگنز و آهن و مقادیر پرمنگنات پتاسیم بر راندمان حذف کبالت توسط پرمنگنات پتاسیم از محلول الکترولیت روی، مورد بررسی قرار گرفت. نتایج نشان داد که هر چه غلظت اولیه کبالت، منگنز و آهن بیشتر باشد، میزان حذف کبالت کمتر خواهد بود. با این حال، با افزایش میزان پرمنگنات پتاسیم، راندمان حذف کبالت افزایش می یابد.

همچنین مشخص شد که برای رسیدن به حد مجاز کبالت در محلول، مصرف پرمنگنات پتاسیم باید با افزایش غلظت کبالت، آهن و منگنز افزایش یابد. اگر غلظت منگنز بیش از ۵۰۰ میلی گرم در لیتر باشد، می تواند تا حد زیادی بر کاهش راندمان حذف کبالت تأثیر بگذارد؛ اما زمانی که غلظت اولیه کبالت بالا باشد، اهمیت تاثیر مقادیر اولیه منگنز کاهش می یابد. علاوه بر این، اگر غلظت منگنز کمتر از ۲۰۰ میلی گرم در لیتر باشد، حذف بهینه کبالت (کمتر از ۲ پی پی ام) تحت هیچ شرایطی اتفاق نمی افتد. نتایج همچنین نشان می دهد که اگر غلظت پرمنگنات پتاسیم ۱ گرم در لیتر یا کمتر باشد، یون آهن موجود در محلول راندمان حذف کبالت را به شدت کاهش می دهد.

واژه‌های کلیدی

محلول مصنوعی سولفاته، حذف کبالت، روش منگانومتری، دیزاین اکسپرت، بهینه‌سازی.