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Optimization of Acid Leaching of Rutile Containing Slag using Factorial Based Response Surface Modeling

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Abstract

In this study the leaching of a high rutile containing slag in sulphuric acid has been investigated to find optimum leaching conditions. Using "Fractional Factorial" technique it was deduced that leaching temperature and duration, acid concentration and particle size are the most prominent parameters in the leaching process. The optimization of the leaching process was investigated using the "Rotatable Central Composite Design" technique. The four prominent parameters were studied at 5 different test values each. 3D response surface was employed to illustrate interaction of the parameters. The optimum leaching conditions for the ilmenite concentrate are thus recognized as: T = 123.9°C, time = 323 min, acid conc. = 8.73 mol/ltr, particle size = 33–60 µm. Under these conditions the theoretical and experimental recovery of titanium for the concentrate are found to be 58.2% and 56.1% respectively. Using the same technique the optimum conditions for the slag are found as: T = 122.2°C, time = 255 min, acid conc. = 11.84 mol/ltr, particle size = 54–95 µm. Under these conditions the theoretical and experimental recovery of titanium for the slag are found to be 67.4% and 66.2% respectively.

Keywords: Factorial–Based Methods, Ilmenite, Leaching Optimization, Slag, Sulphuric Leaching

1. Introduction

Titanium dioxide TiO_2 is extensively used in paint and paper industries. There is also great interest in the semi conductive and light sensitive behaviour of this material. As a result, titanium has found use in photo voltaic cells.

Titanium dioxide pigments may be produced by two different methods. In the chlorination method, chlorine gas is used to extract titanium tetrachloride from titanium rich concentrates/slag[1]. Carbon is used to enhance the process[2,3]. Titanium tetrachloride is subsequently distilled for purification. Pure titanium tetrachloride $TiCl_4$ may be oxidized to form anatase or rutile with the bulk of the chlorine recycled[4]. This process involves many stages and is therefore costly. Chlorine gas is considered a hazardous material and ultimate care and safety regulations are needed in this process.

Direct leaching processes such as the sulphate process have been developed to lower production costs and reduce environmental risks associated with the chlorination process[5–7]. Up to 50% of world titanium production is via the sulphate process[8].

Products such as $TiOSO_4$ and $FeSO_4$ will dissolve during the leaching stage, according to following reaction[8–10]:

$$FeTiO_3 + 2H^+(aq) \rightarrow TiO^{2+}(aq) + Fe^{2+}(aq) + 2OH^-(aq) \qquad (1)$$

Using DOE technique on red mud revealed that the temperature and acid concentration are dominant parameters in the leaching process[11]. In[12] author and co–workers studied the leaching of red mud in dilute sulphuric acid. They identified the temperature, acid concentration and pulp density as significant parameters in the leaching process and the optimum leaching conditions obtained at 60˚C with a pulp density of 5% and acid concentration of 6N and recovery was 64.5%.

Mechanical activation accelerated the leaching process due to phase transformation, structural defects, strain

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and changes in the surface characterization of minerals, lattice disorder and decreased particle size[13–16]. Addition of a particular amount of iron powder after a certain time improved the dissolution of iron and titanium[17]. The rate of dissolution of ilmenite is higher under reducing conditions[18]. SO_2 and Ti^{3+} as reducing agent can enhance leaching only if Fe^{2+} ions are present and the positively effect of Ti^{3+} more than SO_2 [19].

Using preoxidation strengthen the rate of iron removal from ilmenite due to produced phase transformation. In[20] author have reported more recovery of titanium using preoxidation step. This process produced Pseudobrookite phase that highly dissolute during leaching step.

According to conventional method, increments of temperature and sulphuric acid concentration have increased the rate of titanium dissolution[21].

The optimizations of the leaching procedures are usually based on one–variable–at–a–time methods, which facilitate the interpretation of the results obtained, but interactions between variables are not taken into consideration. Consequently, a false minimum or maximum may be attained, leading to the use of certain conditions in which the combination of the variables is not that which provides the best analytical response. Experimental design methods have been recently applied extensively to optimize the extraction and leaching procedures[11,12,22–24]. One of the most used multivariate tools is two–level factorial design (full or fractional)[25–27]. It is used to check the preliminary significance of the variables in the system under study. In this method, the main effects of the variables and their interactions are estimated. This is one of the greatest advantages of multivariate optimization compared to conventional optimization. Another advantage is that the number of experiments is considerably reduced particularly in the case with many factors. Additionally, optimum values of factors can be determined with higher degree of reliability using Central Composite Design (CCD)[25,26].

In the current study, optimization of the variables affecting the sulphuric acid leaching of "Kahnooj" ilmenite concentrate and a rutile containing slag such as Temperature, Acid concentration, Leaching time, Solid to liquid ratio, Particle size and Stirring speed, was performed by a fractional factorial design and subsequent CCD. To the best of our knowledge, this is the first study that applies DOE for the sulphuric leaching of titanium from Ilmenite and slag.

2. Experimental

2.1 Ilmenite and Slag Samples and Chemicals

Ilmernite concentrate supplied by Kahnooj complex, southern Iran and a rutile containing slag obtained from reduction of the concentrate was used in the initial screening tests and final optimization tests. The slag was obtained by reducing ilmenite concentrate in a graphite crucible at 1500˚C. Table 1 exhibits the analysis of the concentrate and the slag.

2.2 Apparatus and Experimental Procedure

The leaching experiments were carried out in a 500 ml three necked glass fitted with a reflux condenser to minimize volatilization of water. A magnetic heater stirrer was used for agitation. The reactor had two entrances and each of them used for specific purpose, one of them for entrance feeding and the other for temperature measurement.

In each run 400 ml of sulphuric acid with the specified concentration was heated to the predetermined temperature on a hot plate. Subsequently, the predetermined amount of solids (ilmenite or slag) was added to the bath. Samples were taken from the system at specific time intervals. Each sample was filtered prior to analysis. Titanium recovery was calculated using Equation (2):

$$R_{Ti} = \frac{C_{Ti}}{D_{Ti}.m}.100 \qquad (2)$$

Where, R_{Ti}(%), C_{Ti}(g/lit), D_{Ti}(g/lit) and m(wt. %) indicate titanium recovery, titanium in the solution, pulp density and titanium content of the solid respectively. The sample was subsequently analysed by atomic absorption spectroscopy.

2.3 Experimental Plan

Design of experiments includes identification of important variables and determining their values,

Table 1. Chemical composition of Kahnooj ilmenite concentrate and slag

Oxides	TiO_2	$FeO+Fe_2O_3$	SiO_2	CaO	MgO	Al_2O_3
Ilmenite (Wt. %)	43.37	40.08	8.15	2.44	2.32	1.87
Slag (Wt. %)	67.8	3.74	8.97	7.53	6.42	2.57

conducting required experiments, and analysing results. An important technique in experiment design is the "2 factorial" method, where, each variable is studied at two different levels[27]. In this method, the number of required runs increases exponentially as the number of variables increase. The variable (factor) is studied using "fractional factor" technique. Based on analysis results, additional runs might be required. Among "2 level" methods, the "Central Composite" technique is favoured by many scientists. This method requires "2n" additional runs at 2 levels of $-\alpha$ and $+\alpha$[28]. Using this method, the number of required runs will amount to $N = F + 2n + m_0$, where, F indicate the number of experiments in factorial design, and 2n and m_0 represent number of auxiliary runs and number of central replicates respectively. The statistical computer package "Design–Expert 7.0.0 Trial" (DX7) is used in this research.

3. Results and Discussions

3.1 Screening of the Parameters using Fractional Factorial Design

The effects of six important experimental variables, namely, temperature, time, acid concentration, particle size, pulp density, and stirring speed, the "one quarter fractional factorial" design has been employed. The total number of runs amount to $2^{(6-2)} = 16$ runs. These factors together with their levels have been illustrated in Table 2.

Table 3 indicates screening results for ilmenite and the slag. To study the interaction of experimental parameters, normal probability diagrams have been employed (Figure 1(a), (b)). In these diagrams, variables with significant effects are located away from the straight line, while, other parameters lie on the line. The further a specific variable is from the line, the more significant its effect will be. It can therefore be deduced that variables A, B, C, and the interaction between variables AB are the most influential parameters in the leaching process. The effects of these parameters on ilmenite and slag are not identical.

Table 4 indicates the effect of each parameter on each material. It can be observed that leaching temperature has a more profound effect on the slag compared to ilmenite.

As expected, results reveal that titanium recovery into the solution increases with increasing temperature, acid concentration, and time allowed for leaching. Titanium recovery also increases with finer particle size. This result is in accordance with previous researches[19,21]. Pulp density and steering speed don't have a significant effect on titanium recovery in the chosen range of experiments.

It can therefore be deduced that a more precise model can be obtained by optimizing the four parameters of temperature, time, acid concentration and particle size.

Table 2. Experimental parameters and levels

Factor	Notation	Levels		
		–1	0	+1
Temperature (^{0}C)	A	80	95	110
Time (min)	B	180	240	300
Acid concentration (M)	C	6	8	10
Pulp density (g/lit)	D	5	15	25
Particle size (μm)	E	38–90	106–200	106–200
Stirring speed (rpm)	F	400	500	600

Table 3. Design matrix and responses for fractional factorial design

Run order	A	B	C	D	E	F	Ilmenite recovery (%)	Slag recovery (%)
1	–	–	–	–	–	–	4	6.9
2	–	–	–	+	–	+	1	3.8
3	–	+	–	–	+	+	4	12.2
4	–	–	+	–	+	+	3.8	9.8
5	+	+	+	–	+	–	43.6	53.2
6	–	+	–	+	+	–	3.8	7.4
7	+	–	–	–	+	–	11.2	29.6
8	+	–	+	–	–	+	27.8	40.9
9	+	+	–	–	–	+	38.6	51
10	–	+	+	–	–	–	10.6	19
11	+	–	–	+	+	+	17	30.4
12	+	+	+	+	+	+	48.8	61
13	+	+	–	+	–	–	44.6	52.7
14	+	–	+	+	–	–	35	44.4
15	–	+	+	+	–	+	11.4	18.9
16	–	–	+	+	+	–	4.6	7.7
17	0	0	0	0	0	0	23	30.7
18	0	0	0	0	0	0	24	25.3
19	0	0	0	0	0	0	25.6	33

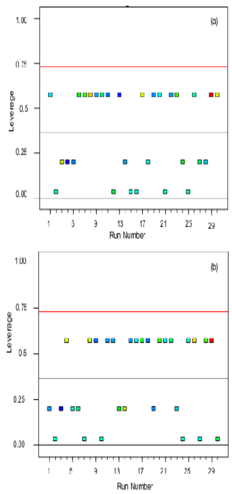

Figure 1. Normal probability plots of the effects for the fractional factorial design. (a) Ilmenite. (b) Slag.

Table 4. Rate effect of effective parameter on ilmenite and slag leaching

Effects	A	B	C	E	AB
Ilmenite rate percentage (%)	65.16	17.99	4.92	1.71	6.07
Slag rate percentage (%)	79.46	12.11	4.02	0.81	2.09

3.2 Development of a Model by "Central Composite Design" (CCD) Method

The "Central Composite Design" was introduced by Box and Wilson[29]. When applied to "k" parameters which have been coded as x_1 to x_k, CCD comprises of 3 parts. The first part is a factorial design, including "2^k" experiments. Each parameter is assigned two experimental codes. For example, experiments relating to the first parameter, that is x_1, are assigned $x_i = +1$ or $x_i = -1$. In general the experiments are represented as $x_i = +1$ or $x_i = -1$ for i = 1 to k, hence 2^k runs. The second part, the axial, comprises of "2k" runs. In each run, one parameter may be at level +α or −α (α > 0) while the other parameters are kept at their zero level. The final part is related to the central part. In this part all the parameters are at their zero level.

In this study, the rotatable CCD technique has been employed to find optimum conditions. It is assumed that α = 2. Tables 5 and 6 exhibit parameter levels and experiment design method respectively. Recovery values for titanium are indicated for each run.

Table 7 is an "Analysis of Variance" (ANOVA) table. The significance of each parameter has been illustrated in

Table 5. Factors and their levels for Central Composite Design (CCD)

Factor	No	Level				
		−α	−1	0	+1	+α
Temperature (°C)	A	65	80	95	110	125
Time (min)	B	120	180	240	300	360
Concentration (M)	C	4	6	8	10	12
Particle size (μm)	D	25–38	38–90	90–106	106–200	200–250

Table 6. Design matrix and the responses for Central Composite Design (CCD)

Run order	Point type	A	B	C	D	Ilmenite recovery (%)	Slag recovery (%)
1	fact	−1	−1	−1	−1	11	13.1
2	fact	+1	−1	−1	−1	28.7	38.9
3	fact	−1	+1	−1	−1	15.6	20.2
4	fact	+1	+1	−1	−1	35.5	42.5
5	fact	−1	−1	+1	−1	11.5	13.6
6	fact	+1	−1	+1	−1	38.5	46.5
7	fact	−1	+1	+1	−1	14.5	17.6
8	fact	+1	+1	+1	−1	47.2	58.6
9	fact	−1	−1	−1	+1	10.7	12.4
10	fact	+1	−1	−1	+1	22.1	25.7
11	fact	−1	+1	−1	+1	18	21.8
12	fact	+1	+1	−1	+1	28.6	34.4
13	fact	−1	−1	+1	+1	9.5	10.6
14	fact	+1	−1	+1	+1	25.4	30.7
15	fact	−1	+1	+1	+1	8.7	10.2
16	fact	+1	+1	+1	+1	34	40.8
17	axial	−α	0	0	0	4.6	5.5
18	axial	+α	0	0	0	33.8	41
19	axial	0	−α	0	0	10	12.6
20	axial	0	+α	0	0	20.2	24.2
21	axial	0	0	−α	0	11.8	14.7
22	axial	0	0	+α	0	16.6	20
23	axial	0	0	0	−α	25	30.2
24	axial	0	0	0	+α	14.1	16.5
25	center	0	0	0	0	18	19.2
26	center	0	0	0	0	17.6	27.2
27	center	0	0	0	0	17.7	21
28	center	0	0	0	0	16	20.4
29	center	0	0	0	0	22.7	21.8
30	center	0	0	0	0	17.1	21.5

Table 7. Analysis of variance (ANOVA) table for the response surface model

Source	Df		F Value		p–value Prob>F	
	Ilmenite	Ilmenite	Ilmenite	Slag	Ilmenite	Slag
Model	10	10	18.08	16.09	0.0001	0.0001
A	1	1	134.32	39.77	0.0001	0.0001
B	1	1	13.68	11.89	0.0015	0.0027
C	1	1	1.98	4.83	0.020	0.0097
D	1	1	1.38	2.16	0.0312	0.0401
AB	1	1	4.86	4.14	0.0098	0.0091
AC	1	1	0.89	0.94	0.0877	0.0719
AD	1	1	1.06	0.91	0.0673	0.0795
BC	1	1	0.031	0.042	0.766	0.6451
BD	1	1	0.018	0.004	0.8941	0.9462
CD	1	1	0.62	0.57	0.1087	0.1645
Residual	19	19				
Lack of fit	14	14	3.46	3.62	0.089	0.0816
Pur error	5	5				
Cor total	29	29				

this table. Value of "prob>f" less than 0.050 for a factor indicates that its effect is significant. So, the temperature, time, concentration particle size and interaction between temperature and time are the significant parameters.

The F–value of 18.08 and 16.09 for Ilmenite and slag implies that the model is significant. The F–values of 3.46 and 3.62 for the lack of fit of ilmenite and slag imply that they are not significant relative to the pure experimental error and confirm the validity of the model. A quadratic response surface model based on a higher F–value to fit the experimental data was selected.

The employed model for recovery of titanium consists of 4 main parameters and 1 interaction factors. Equation 3 and 4 indicate the codes employed for calculating titanium recovery (R_1) from ilmenite and slag respectively (high coefficient for each code resulted more effect on recovery):

$$R_1 = +19.93 + 9.21 * A + 2.94 * B + 1.02 * C - 0.81 * D + 1.29 * A * B \quad (3)$$

$$R_1 = +20.70 + 9.68 * A + 3.35 * B + 1.18 * C - 0.88 * D + 1.37 * A * B \quad (4)$$

Figure 2 (a), (b) exhibit leverage in the model. Leverage indicates effectiveness of the model with respect to a specific run[28]. If a point has a leverage one, then the model must go through that point. High values of leverage are not desirable. Any error will result in great amounts of divergence if the leverage value is high. Figure 2 (a), (b) indicate acceptable (< 0.75) values for leverage. Therefore, errors are not likely to affect experiment results significantly.

These results may be verified by plotting Cook's distance (Figure 3 (a), (b)) and "difference in the fit", DEFITS diagrams (Figure 4 (a), (b)). Cook's distance diagrams indicate the change in the regression if one of the data points is omitted[30]. This plot also used for estimation of the influence of a data point when performing least squares regression analysis. Relatively large values are associated with cases with high leverage and may be an outlier and should be investigated. All values for cook's distance are less than allowed value (less than 1).

DEFITS exhibit the the change in the predicted value for a point, obtained when that point is left out of the regression[30]. The desirable values for DEFITS plot show the correlation of model.

The validity of optimization experiments was confirmed due to allowed values for all leverage, cook's distance and DEFITS plot. Furthermore, Leverage, Cook's distance and DEFITS plots were constructed by DX7 _software.

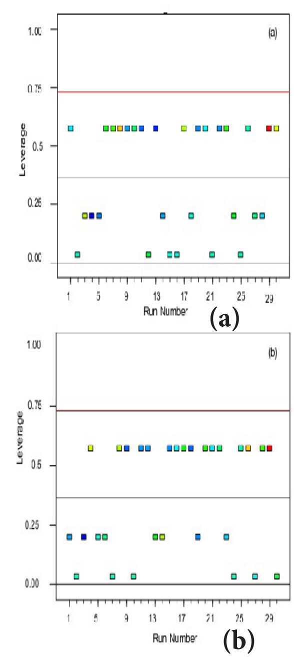

Figure 2. Leverage plot for quadratic model obtained from central composite design. **(a)** Ilmenite. **(b)** Slag.

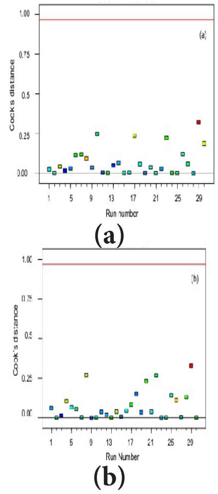

Figure 3. Cook's distance plot for model. **(a)** Ilmenite. **(b)** Slag.

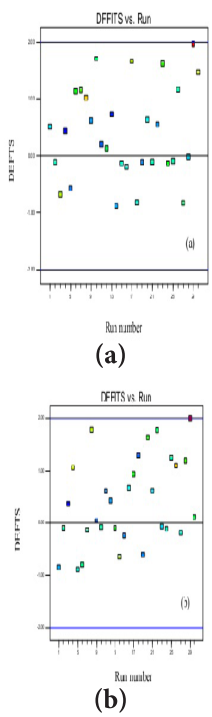

Figure 4. Difference in Fits (DEFITS) plot for evaluating of the quadratic model. **(a)** Ilmenite. **(b)** Slag.

In order to gain a better understanding of the interaction effects of variables on recovery, Three–Dimensional (3D) plots for the measured responses were formed based on the model equations (Equations 3 and 4). Also the relationship between the variables and responses can be further understood by these plots. The response model is mapped against two experimental factors while the two other factors are held constant at their central levels. Figure 5 (a), (b) indicate interaction of AC on the response surface for ilmenite and slag respectively. It may be observed that increasing temperature and acid concentration results in an increase in the recovery for both ilmenite and slag. In many researches based on conventional method, increments of recovery with increase of temperature and concentration have been reported[8, 9,19,20].

Figure 6 (a), (b) indicate interaction of AD on the response surface for ilmenite and slag respectively. It may be observed that increasing temperature results in an increase in the recovery while increasing particle size reduces recovery. Fine particles resulted in more dissolution surface and so, recovery increased by decrement of particle size. Using high temperature and low particle size enhanced titanium recovery from concentrate. Figures 5 and 6 indicate that optimum conditions do not occur in the centre part of the design.

The main aim of this study has been to indicate optimum leaching conditions for ilmenite and slag using experimental data. The response surface methodology can be used to find desirable location in the design space. Variable can be minimum or maximum in this location. In this research, the response surface methodology was used by DX7 software to find the optimum condition of ilmenite and slag leaching process. To maximize the leaching process, temperature, acid concentration and time set on their maximum value and particle size set on its minimum value.

More than 50 optimum conditions are thus suggested. One of these optimum conditions has been schematically

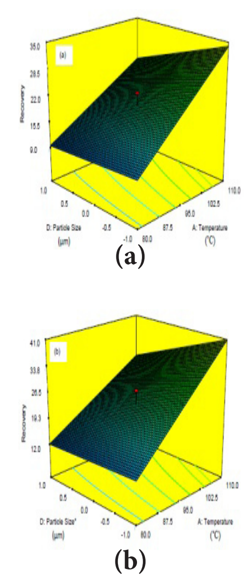

Figure 5. Three–Dimensional (3D) response surface for A: temperature – C: acid concentration. **(a)** Ilmenite. **(b)** Slag.

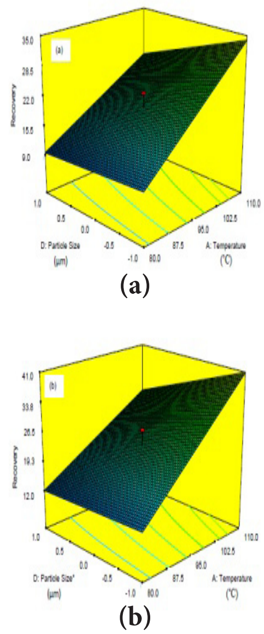

Figure 6. Three–dimensional (3D) response surface for A: temperature – D: particle size. **(a)** Ilmenite. **(b)** Slag. [* coded as Table 5].

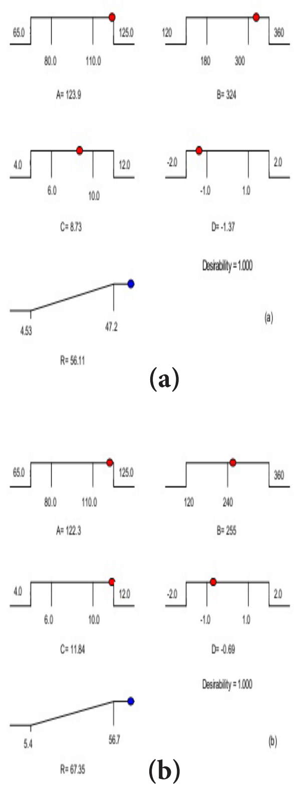

Figure 7. Schematic representation of the optimum values of factors, responses and the corresponding levels for. **(a)** Ilmenite. **(b)** Slag.

illustrated in Figure 7 (a), (b) for ilmenite and slag respectively. Based on this observation, the optimum leaching conditions for ilmenite are achieved at a temperature of 123.9˚C, acid concentration of 8.73 M, particle size of 33–60 µm (coded –1.37 in Figure 7(a)), and leaching time of 324 min. Under these conditions the theoretical and experimental titanium recovery values are 58.2% and 56.1% respectively. The optimum conditions for leaching of the slag are predicted at a temperature of 122.2˚C, acid concentration of 11.84 M, particle size of 54–95 µm (coded –0.69 in Figure 7 (b)), and leaching time of 255 min. Under these conditions the theoretical and experimental titanium recovery values are 67.4% and 66.2% respectively.

4. Conclusions

- Using response surface model based on factorial design it is found that increasing temperature, acid concentration, and time of leaching results in higher titanium recovery, while increasing particle size reduces titanium recovery. In previous researches, the temperature, time, acid concentration and particle size were reported as the effective parameters and the importance of parameters were not discussed. In this research, it was deduced that the temperature is the most effective parameter in both slag and ilmenite leaching.
- DX7 software has ability to specify the effective interaction parameter. In this research the interaction between temperature and time has a profound effect on the leaching of ilmenite and high rutile containing slag. The impact of Temperature–Time parameter is more than effect of acid concentration and particle size on titanium recovery.
- Titanium recovery from slag exceeds titanium recovery from ilmenite. As the software suggested, the maximum theoretical recoveries due to the range of parameters are 60.7% and 73.5 for ilmenite and slag respectively. At the same conditions, the maximum experimental recoveries are 59.2% and 73.1 for ilmenite and slag respectively. So the leaching can be a useful method for extraction of titanium from ilmenite.

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