MODELING AND EVALUATING THE IMPORTANT FACTORS AFFECTING THE SOLVENT EXTRACTION OF LANTHANUM AND NEODYMIUM FROM NITRIC ACID SOLUTION IN D2EHPA+CYANEX272+LACTIC ACID SYSTEM

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ABSTRACT

This study was aimed to investigate the separation performance of La and Nd from nitric acid solution by D2EHPA-Cyanex272-actic acid system. Response surface methodology (RSM) was employed to evaluate and optimize the effects of pH, Cyanex272 to D2EHPA volume ratio, extraction time and solvents concentration on the extraction efficiency of La and Nd. The quadratic (i.e. $E_{Nd}^{0.25} = +2.898 + 0.884A - 0.179B + 0.151C + 0.039D - 0.070E - 0.457A^2 - 0.219B^2$*) and 2F1 (i.e.* $E_{La} = +32.281 + 31.402A - 5.065B + 9.913C + 4.277D - 0.388E + 10.144AC$*) models were proposed for the relationship between the extraction efficiency of Nd and La and the influential factors, respectively. The results showed also that the linear effects of pH, Cyanex272 to D2EHPA volume ratios, solvents concentration and the quadratic effects of pH and Cyanex272 to D2EHPA volume ratios were significant in Nd extraction. The significant parameters referring to La extraction were the linear effects of pH, solvents concentration, and Cyanex272 to D2EHPA volume ratio, the interaction effect of pH and solvents concentration. It was also found that within the range of parameters investigated, the maximum extraction efficiency of neodymium and lanthanum was ca 74.43 and 22.83 %, respectively.*

Keywords: lanthanum, neodymium, solvent extraction, extraction efficiency, RSM.

INTRODUCTION

The rare earth elements (REEs) are an unusual group of seventeen elements belonging to lanthanides. They have unique properties: magnetic, catalytic, chemical, metallurgical and phosphorescent. That is why REEs are very important elements from an industrial viewpoint. They are extensively used in metallurgy, lasers, batteries, magnets and hi-technology [1]. With the increasing demand for REEs, the purification and separation of REEs from each other have gained significant importance. The separation of mixtures of rare earths into the individual elements using fractional precipitation, ion exchange, fractional crystallization and solvent extraction methods is very difficult because of the small differences in their physical and chemical properties [2]. Among all of these, solvent extraction is the most successful method used for industrial separation and purification of rare-earths [3]. The separation and purification of REEs from different acid solutions are carried out using acidic, basic, and neutral extracting agents as well as combinations of them [4 - 8].

Solvent extraction is extensively employed as REEs flexible separation and purification method in various fields such as hydrometallurgy because of its simplicity [9 - 10]. Various extracting agents are used. As a chelating agent, 8-hydroxyquioline (HQ) is a good extractant in respect to REEs acting under favorable experimental conditions – a high extraction efficiency and a large separation ability can be achieved. HQ and its derivatives have been extensively investigated [11 – 22] in REEs extraction and purification. Sun et al. (2005) report that the mixture of CA12 and bis (2,4,4-trimethylpentyl) phosphinic acid (Cyanex272) in n-heptane has a synergistic effect in case of separating Y (yttrium) of HRE (Ho, Er, Tm, Yb and Lu) from chloride solutions [23]. Sun et al. [24] report that a mixture of Cyanex272 and sec-nonyl phenoxy acetic acid (CA-100) exhibits a significant synergistic effect in the extraction of Sc, Y, La, Ga, Yb from chloride solutions. Wang et al. [25] show that separation coefficient of yttrium from heavy rare earth is higher in the double solvent (CA12-Cyanex272-TBP) extraction system than that in the single (CA12-TBP) one. Tri-n-butyl phosphate (TBP) is introduced to the latter system as a phase modifier to achieve fast phase separation and to improve the stability of the organic phase. Much works has been performed in the field of separation and extraction of rare earth elements but there are a few reports on the separation and extraction of La and Nd using mixture systems of Cyanex272, D2EHPA and lactic acid. Hence, this research is aimed to investigate and optimize the effects of important factors determining the separation efficiency of neodymium (Nd) and lanthanum (La) from nitric acid solution using Cyanex272/D2EHPA/lactic acid system.

METHODOLOGY

The optimization of the process variables by a conventional approach requires the performance of a very large number of experiments, which would be very expensive and time consuming. Furthermore, it does not reveal the influence of the interactions between the process variables on the dependent variable. The experiments design is a statistical technique for planning, conducting, analyzing, interpreting, and optimizing the experiments that has advantages with regard to the requirement of less resources (time, reagents, and experimental work). Moreover, the designs where the levels of all variables are changed simultaneously allow the development of mathematical models to assess the statistical significance of the main and interaction effects of the independent variables [26]. The experiments design has also proven to be a powerful tool in many areas [27 - 31]. Response surface methodology (RSM) is a collection of statistical and mathematical methods that are useful for modeling and analyzing engineering problems. The RSM is now considered a standard statistical approach to designing experiments, building models, evaluating the effects of many factors, finding the optimal conditions for desirable responses, and reducing the number of required experiments [28, 32 - 33]. The most popular RSM method is the CCD [32 - 33]. Hence, we use CCD in this study to design the experiments. Generally, this design consists of the following parts: (i) a full or fractional factorial point, (ii) star or axial points at a specific distance from the center, and (iii) a center point.

EXPERIMENTAL

Reagents and preparation

The commercial extractants, Cyanex272 (bis(2,4,4-trimethylpentyl) phosphinic acid) ($C_{16}H_{35}O_2P$) of a purity of 90 % and D2EHPA (bis (2-ethyhexyl) phosphate) ($C_{16}H_{35}O_4P$) of a purity of over 95 % were purchased from Merck (Germany) and these reagents were used without further purification.

Kerosene was applied as a diluent in the solvent extraction. Different amounts of extractants were dissolved in kerosene to achieve the concentration required.

The oxides of rare earth elements such as lanthanum, neodymium, gadolinium, and dysprosium of a purity higher than 99 % were purchased from Merck (Germany). The stock solutions of REEs were prepared from their oxides (up to 99) by dissolving them in a concentrated nitric acid (10 ml) and distilled water. Also, for some of the insoluble rare earth oxides, the stock solution was prepared using a hot plate and magnetic stirrer (SHIN SAENO). The stock solutions obtained were analyzed by using the ICP (ICP-Varian, Geological Survey of IRAN). Their chemical composition is shown in Table 1.

Solutions of HNO_3 and NaOH were also used to

Table 1. Chemical composition of the stock solution.

Element	Dy	Gd	Nd	La
Chemical composition (ppm)	184	176	166	153

Table 2. Selected parameters, their coded and real values in the center composite design.

Factors	Symbol	Low axial Level (-1.5)	Low factorial Level (-1)	Medium level (0)	High factorial Level (+1)	High axial Level (+1.5)
pH	A	0.5	1	2	3	3.5
Cyanex272/D2EHPA volume Ratio	B	0.25	1	2.5	4	4.75
Solvent concentration (molar)	C	0.025	0.05	0.1	0.15	0.175
Leaching time (min)	D	2.5	5	10	15	17.5
Lactic acid concentration (molar)	E	0.2	0.4	0.8	1.2	1.4

adjust the pH of the aqueous phase. The pH meter used in experiments was RS323 interface AZ8601 model.

Solvent extraction procedure

The experiments were carried out using different volumes of solvent extractions of La, Nd, Dy, and Gd of a concentration of 0.05 ml L^{-1} and pH = 2. 10 ml of the aqueous phase (stock solution) were stirred with 10 ml of the organic phase for 10 min with a magnetic stirrer at room temperature (298 K). When mixing was completed, the mixture was transferred to a conical separating funnel for two-phase separation. Metal ion concentrations in the aqueous phase prior to and after the extraction were determined by ICP. The metal contents in the organic phase were also obtained by mass balance.

The solvent performance was evaluated by using three factors including the distribution ratio (D), and the extraction efficiency (E) determined on the ground of Eqs. (1) to (2), respectively.

$$D = \frac{[M]_t - [M]_a}{[M]_a} \quad (1)$$

$$E = \frac{D}{D + (\frac{V_{aq}}{V_{org}})} \times 100 \quad (2)$$

where $[M]_t$ and $[M]_a$ represented the initial and final concentration of the metal ions in the aqueous phase, while V_{aq} and V_{org} were the volumes of the aqueous and organic phases.

RESULTS AND DISCUSSION

Statistical analysis

The design of the experiments is performed using the response surface methodology (RSM) for modeling and optimizing the operational parameters of the separation performance of lanthanum (La) and neodymium (Nd) from nitric acid solution by solvent extraction. Its usage decreases the number of experiments, time and material resources. Furthermore, the analysis of the results is easily performed and the experimental errors are minimized. In this study, pH, the concentrations of the solvents, the volume ratio of Cyanex272 to D2EHPA, the concentration of lactic acid, the solvent extraction time were studied as operating parameters of separation Nd from La. The selected levels of the parameters are shown in Table 2. Table 3 shows the calculated extraction efficiency in respect to Nd and La based on the central composite design used.

The statistical software package Design-Expert 7 is used for regression and graphical analysis of the obtained data during the experiments. An analysis of the variance (ANOVA) is applied to estimate the statistical parameters. Tables 4 and 5 list the statistical analysis results aiming to select the models of 95 % confidence level (p-value < 0.05). The small probability values ($p < 0.05$) indicate the model validity.

The statistical analysis shows that the quadratic model and 2F1 fit to the experimental results referring to Nd and La, respectively. The fitness of the model is determined by the correlation coefficient (R^2) value. The latter provides a measure of the observed response

Table 3. A central composite design of the experiments aiming to evaluate the effects of the operating parameters on the separation of La from Nd.

Run	A	B	C	D	E	Extraction efficiency of La (%)	Extraction efficiency of Nd (%)
1	1	4	0.15	15	0.4	0.6	1.67
2	1	4	0.15	5	1.2	0.45	0.79
3	1	1	0.15	5	0.4	0.16	9.09
4	3	4	0.05	5	1.2	46.52	99.25
5	2	2.5	0.175	10	0.8	84.71	48.93
6	3	4	0.05	15	0.4	57.13	91.30
7	2	4.75	0.1	10	0.8	11.57	20.63
8	2	2.5	0.1	10	0.8	24.9	71.41
9	2	2.5	0.1	10	1.4	16.82	84.02
10	1	1	0.05	5	1.2	1.63	0.20
11	0.5	2.5	0.1	10	0.8	0.37	1.28
12	1	4	0.05	5	0.4	5.85	2.91
13	3	1	0.15	5	1.2	89.74	99.77
14	1	1	0.15	15	1.2	0.26	13.04
15	3	1	0.15	15	0.4	89.79	99.85
16	3	1	0.05	15	1.2	70.14	98.35
17	2	2.5	0.1	10	0.8	24.85	71.75
18	2	2.5	0.1	10	0.8	24.80	71.65
19	3	1	0.05	5	0.4	31.78	84.47
20	1	4	0.05	15	1.2	1.15	0.10
21	3.5	2.5	0.1	10	0.8	86.11	99.68
22	1	1	0.05	15	0.4	2.82	4.76
23	3	4	0.15	15	1.2	83.77	96.38
24	2	2.5	0.025	10	0.8	29.82	62.82
25	2	2.5	0.1	17.5	0.8	37.89	85.75
26	2	2.5	0.025	10	0.8	5.93	38.27
27	2	2.5	0.1	2.5	0.8	26.23	83.14
28	3	4	0.15	5	0.4	76.89	99.98
29	2	0.25	0.1	10	0.8	59.69	99.76

values variability explained by the experimental factors and their interactions. A greater fitness between the empirical model and the actual data is obtained in case the correlation coefficient approaches unity, while its lower values refer to lower relevance of the dependent variables of the model which explaining the behavior variations. The correlation coefficient values calculated for Nd and La are 0.92 and 0.91, respectively. The values of the adjusted determination coefficient (adjusted R^2 = 0.895 for Nd and adjusted R^2= 0.881 for La) are also acceptable indicating a significance of the model. The values of the predicted correlation coefficient for Nd and La are also acceptable. This supports the significance of the model and the coefficient. The proposed regression models including important parameters (in the coded units) are as follow:

$$E_{Nd}^{0.25} = +2.898 + 0.884A - 0.179B + 0.151C + \\ +0.039D - 0.070E - 0.457A^2 - 0.219B^2 \quad (3)$$

$$E_{La} = +32.281 + 31.402A - 5.065B + \\ +9.913C + 4.277D - 0.388E + 10.144AC \quad (4)$$

Table 4. Analysis of the variance of the response surface reduced quadratic model predicting La extraction efficiency.

Source	Sum of Squares	Degree of freedom	Mean Square	F Value	p-value Prob > F
Model	24780.35	6	4130.05	35.56	< 0.0001
A-pH	20214.69	1	20214.69	174.07	< 0.0001
B- Cyanex272/D2EHPA volume Ratio	526.08	1	526.08	4.53	0.0447
C- Solvent concentration (molar)	2014.85	1	2014.85	17.35	0.0004
D- Leaching time (min)	375.09	1	375.09	3.23	0.0860
E- Lactic acid concentration (molar)	3.08	1	3.08	0.026	0.8720
A×C	1646.53	1	1646.53	14.17	0.0011
Residual	2554.81	22	116.12		
Pure Error	0.005	2	0.0025		
Cor Total	27335.17	28			
R-Squared	0.906537				
Adj R-Squared	0.881048				
Pred R-Squared	0.83365				
Adeq Precision	20.45447				

Table 5. Analysis of the variance of the response surface reduced quadratic model predicting Nd extraction efficiency.

Source	Sum of Squares	Degree of freedom	Mean Square	F Value	p-value Prob > F
Model	20.59	7	2.94	35.18	< 0.0001
A-pH	16.03	1	16.03	191.78	< 0.0001
B- Cyanex272/D2EHPA volume Ratio	0.65	1	0.65	7.83	0.0107
C- Solvent concentration (molar)	0.47	1	0.47	5.64	0.0271
D- Leaching time (min)	0.03	1	0.03	0.38	0.5423
E- Lactic acid concentration (molar)	0.10	1	0.10	1.20	0.2845
A^2	2.38	1	2.38	28.57	< 0.0001
B^2	0.55	1	0.55	6.60	0.0179
Residual	1.75	21	0.08		
Pure Error	6.3E-06	2	3.15E-06		
Cor Total	22.34	28			
R-Squared	0.92				
Adj R-Squared	0.89				
Pred R-Squared	0.82				
Adeq Precision	19.77				

Eqs. 3 and 4 express the relasionship between the important terms and the extraction efficiency of Nd and La, respectively.

Effect of the factors and their interactions

The effects of the variable parameters on the extraction Nd and La are investigated. The parameters that increase and/or decrease the extraction efficieny are further appraised and the results obtained are presented in Figs. 1-3. Fig. 1a indicates that the increase of pH and the volume ratios of Cyanex272 to D2EHPA increases and decreases, correspondingly, the extraction efficiency of Nd in the quadratic form. According to Fig. 1b, the extraction efficiency of La decreases with increase of

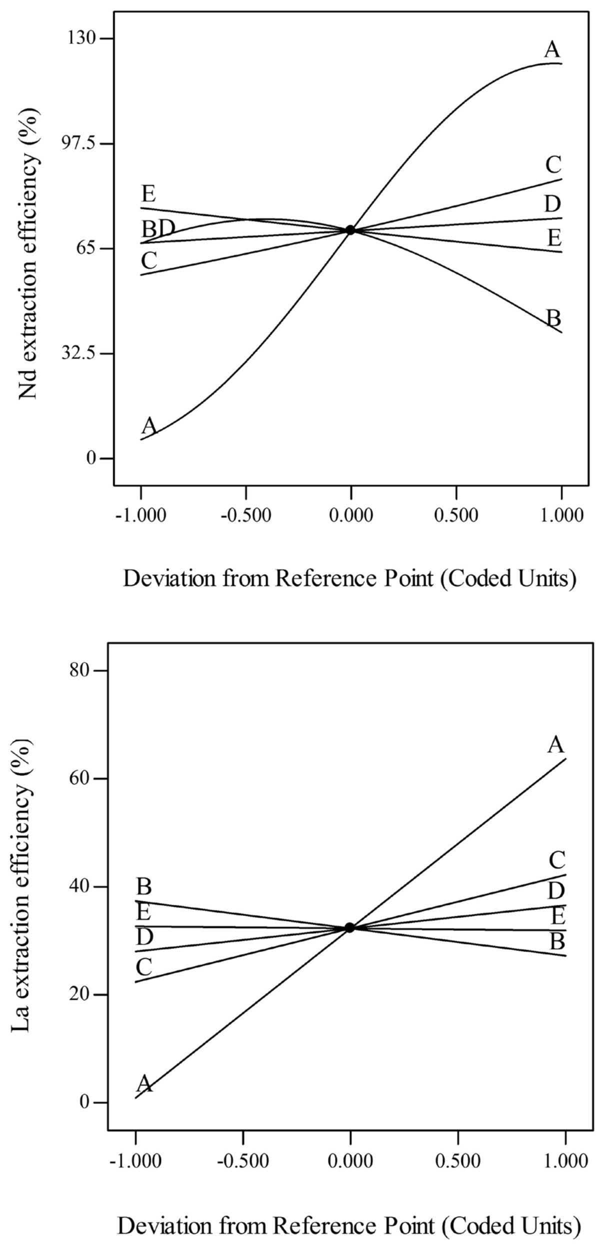

Fig. 1. The effect of the main parameters including pH (A), the volume ratio of Cyanex272/D2EHPA (B), the solvents concentration (C), the leaching time (D) and the lactic acid concentration (E) on the extraction efficiency of Nd (a) and La (b).

the volume ratios of Cyanex272 to D2EHPA. Also, Fig. 1a-b shows that both the extraction efficiency of Nd and La increases linearly with increase of the solvents concentration, lactic acid concentration and time. It is also observed that the lactic acid concentration has the least effectt on the extraction efficiency of La.

The interactions effects of the parameters are also investigated. The results obtained are shown in Fig. 2.

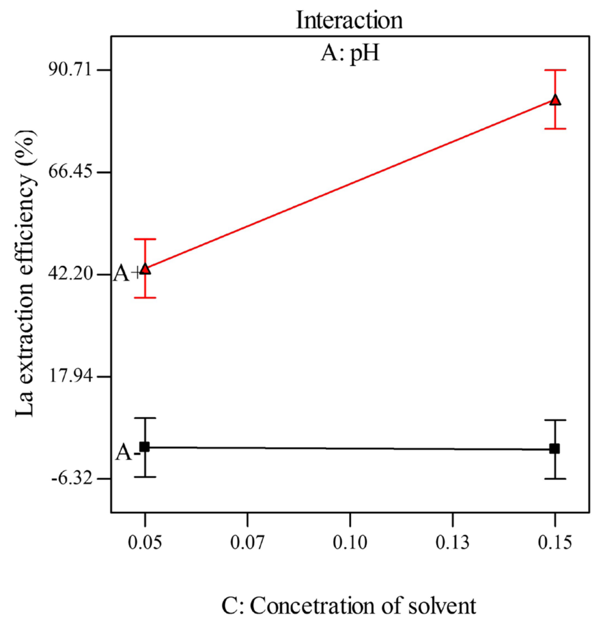

Fig. 2. Interaction effect between the solvents concentration and pH on the extraction efficiency of La.

The effect of the interaction between pH and the solvents concentration on the extraction efficiency of La is outlined. It is seen that the increase of the solvents concentration has no effect on La extraction efficiency at lower pH values, while at higher pH the extraction efficiency of La increases with the increase of the solvents concentration. The extraction efficiency of La grows from 42 % to 91 % in the latter case. The values of pH and the solvents concentration have the greatest effect on the extraction efficiency of Nd and La. The relationship between pH and the concentration of the solvents is presented in Fig. 3.

Fig. 3 is a summarization of Eqs. 3 and 4 as well as Tables 4 and 5. The graph presented there confirms that the effect of pH is greater than that of the solvents concentration in the course of extraction of Nd and La.

Improvement of process parameters

One of the main aims of this study is to find the optimum process parameters to maximize and minimize the extraction efficiency of Nd and La, respectively. Numerical optimization by using Design Experts software made it possible to find the levels of parameters determining the utmost separation of Nd from La. Table 6 shows the result of this optimization (based on coded and actual values). It is observed that the maximum extraction ef-

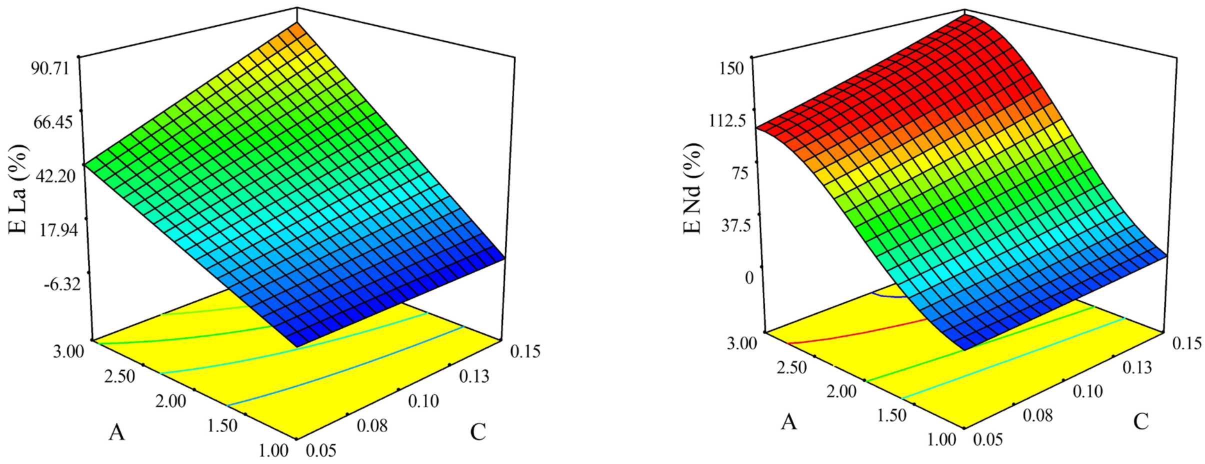

Fig. 3. Response surface plot showing the effect of pH (A) and the solvents concentration (C) on the extraction efficiency (E) of La (a) and Nd (b) when all other variables were kept at their center level.

ficiency of Nd and La refers to ca 74.43 and 22.83 %, respectively. In addition, three extra solvent extraction experiments were conducted at the predicted optimum conditions to confirm the validity of the model. The results obtained are listed in Table 6. As considered, these values are in a good agreement with the values obtained by modeling of the process studied.

CONCLUSIONS

The aim of this study was to evaluate, model and optimize the factors influencing the extraction efficiency of La and Nd. Response surface methodology was employed to assess the main and interactive effects of the parameters and also to optimize the extraction efficiency of Nd from La. The parameters invesigated referred to pH, the volume ratio of Cyanex272 to D2EHPA, the solvents concentration, the leachnig time and the lactic acid concentration. The quadratic and 2F1 models were fitted by RSM to the experimental data. The suggested regression models based on coded factors were:

$$E_{Nd}^{0.25} = +2.898 + 0.884A - 0.179B + 0.151C + \\ +0.039D - 0.070E - 0.457A^2 - 0.219B^2$$

$$E_{La} = +32.281 + 31.402A - 5.065B + \\ +9.913C + 4.277D - 0.388E + 10.144AC$$

The results showed that the values predicted by the model equations were in very good agreement with the observed one. It was found that the model could explain about 92 % and 90 % of the variability of the extractive efficiency of Nd and La, respectively. The factors influence degree showed that the linear effects of pH, the volume ratio of Cyanex272 to D2EHPA, the solvents concentration as well as the quadratic effects of pH and volume ratios of Cyanex272 to D2EHPA were statistically significant for the extraction of Nd. Also, it was distinguished that the linear effects of pH, the solvents concentration, and the volume ratio of Cyanex272 to D2EHPA as well as the interaction effect of pH and the solvents concentration were important parameters in the extraction of La.

Table 6. The parameters proposed levels validating laboratory experiments.

Factors	A	B	C	D	E	Nd Extraction efficiency (%)	La Extraction efficiency (%)
Model projections	2.19	2.42	0.05	5	0.4	74.43	22.83
Model validation	2.2	2.5	0.05	5	0.4	73.7	21.23

Moreover, it was found that the optimum values of operational parameters for the maximum extraction of Nd and La referred to pH of 2.19, a volume ratio of Cyanex272 to D2EHPA of 2.42, 0.05 molar solvent concentration, extraction time of 5 min and 0.4 molar lactic acid concentration. The extraction efficiency of Nd was 74.43 % , while that of La was 22.83 % under these conditions.

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