

DETERMINATION OF OPTIMAL CONDITIONS FOR AUTOCLAVE DESILICONIZATION OF A ZINC PRODUCT

Received – Primljeno: 2020-08-24

Accepted – Prihvaćeno: 2020-10-30

Preliminary Note – Prethodno priopćenje

The article presents the results of a study to determine the optimal conditions for an autoclave desiliconization of a zinc product. According to the plan of a six-factor experiment at 5 levels, the optimal conditions for the process of leaching of silicon dioxide from a zinc product were determined. Based on significant partial dependencies, a generalized multivariate equation was compiled for the degree of extraction of silica in solution. The generalized multi-factor equations make it possible to determine the optimal conditions for the desiliconization of a silica-containing zinc product with a combination of different temperatures, the duration of the process, and the initial alkali concentration in the solution.

Key words: zinc product, autoclave, leaching, silicon dioxide, desiliconization

INTRODUCTION

The main process of autoclave leaching of high-silicon sulfide materials is the process of interaction of silicon dioxide with alkaline solutions. However, selective dissolution of SiO_2 in the alkaline medium from zinc-containing products at elevated temperatures has hardly been studied on a practical level.

The first studies of the leaching of silicon dioxide from zinc high-silicon raw material were carried out using of a probabilistic-deterministic experiment planning method (PDP) of based on the well-known formula M. Protodjakonov for processing of statistical data [1-2] and which was improved by V. P. Malyshev, Professor [3-5].

The use of PDP allows to explore the process of desiliconization of zinc product under different combinations of influencing factors, in order to determine the optimal process mode, to study the kinetic parameters of dissolution of silicon dioxide from zinc alkaline solutions product [6]. Although this empirical method does not provide a rigorous description of the kinetics of the process of leaching silicon dioxide of from the zinc product, nevertheless, the information obtained by this method is sufficient to predict the chemical technology processes.

EXPERIMENTAL PART AND DISCUSSION OF RESULTS

In order to determine the optimal conditions for the process of leaching silicon dioxide from the zinc product, a plan for a six-factor experiment on 5 levels was drawn up. The independent factors were considered:

temperature (T/K), duration of leaching (τ /min), initial concentration of alkali (C/kg/m³) and the ratio of liquid to solid (L : S) in the original pulp. The limits of changes in independent factors are shown in Table 1.

The zinc flotation product of the current output from Karagaily MPP was used for these experiments, which contains always the following mass%: Zn – 41,46; SiO_2 – 17,60; S – 19,78; Pb – 3,46; Fe – 3,36; BaSO_4 – 1,92; Al_2O_3 – 2,44; CaO – 0,77.

The experiments were performed in autoclaves from stainless steel IXI8H9T. The pulp of a certain volume for each experiment was prepared immediately before the experiment in the autoclave itself.

Autoclaves were fixed in a rotating frame placed in a heating furnace after that the frame drive and heating were switched on. The start of leaching was taken into account from the moment when the set temperature was reached, and this temperature was maintained automatically with an accuracy of ± 275 K during the whole experiment. After the experiment, the autoclaves were cooled under a stream of cold water and the pulp was opened and filtered. The products were subjected to chemical analysis and calculated the rate of extraction of silica into the solution, expressed in fractions of a unit (α) are shown in Table 2.

Considering the rate of silica extraction into solution (α /fr.u.) as a research function, we sampled the experimental array by factors and found the average values of the function by factor levels in accordance with the recommendations set out in [1-2] and plotted point diagrams of the dependencies of silica extraction into solution on the studied factors (Figure 1). The equations of partial dependences of the extraction rate of silicon dioxide into solution and approximating functions are shown in Table 3.

The analysis of partial dependencies showed that in matrix experiments, the temperature and duration of

K.S. Turebekova (kakosh-94@mail.ru), G. Burkitseterkyzy, G.L. Katkeyeva, Ye.M. Zhunussov Chemical-Metallurgical Institute, Karaganda, Kazakhstan

Table 1 Factors and levels of their variation within the study of a zinc product desiliconization process

Factor	Level				
	1	2	3	4	5
(T-273)/K	100	125	150	175	200
τ /min	15	30	60	120	240
C/kg/m ³	80	120	160	200	240
L : S	4	6	8	10	12

Table 2 Matrix plan and results of a six-factor experiment for leaching silicon dioxide from a silica-containing zinc product

N ^o test/ experiment	Factor levels actual estimated				Extraction ratio of SiO ₂ into solution/fr.u.		
	(T-273)/K	τ /min	C/kg/m ³	L : S			
					α	α II (1)	α K (3)
1	100	15	80	4	0,007	0,033	0,001
2	100	30	120	6	0,023	0,039	0,003
3	100	60	160	8	0,057	0,050	0,011
4	100	120	200	10	0,117	0,067	0,032
5	100	240	240	12	0,168	0,093	0,082
6	125	15	120	12	0,060	0,085	0,065
7	125	30	200	4	0,048	0,116	0,133
8	125	60	240	10	0,149	0,162	0,231
9	125	120	160	6	0,124	0,164	0,236
10	125	240	80	8	0,197	0,204	0,310
11	150	15	160	10	0,083	0,178	0,263
12	150	30	240	8	0,210	0,269	0,408
13	150	60	120	4	0,114	0,266	0,404
14	150	120	80	12	0,210	0,340	0,489
15	150	240	200	6	0,766	0,451	0,580
16	175	15	200	8	0,262	0,335	0,484
17	175	30	160	12	0,445	0,382	0,528
18	175	60	80	6	0,661	0,475	0,596
19	175	120	240	4	0,805	0,714	0,705
20	175	240	120	10	0,790	0,704	0,702
21	200	15	240	6	0,753	0,585	0,655
22	200	30	80	10	0,650	0,596	0,660
23	200	60	200	12	0,824	0,791	0,729
24	200	120	120	8	0,810	0,882	0,752
25	200	240	160	4	0,774	1,115	0,796
Average value					0,364	0,364	0,394

Table 3 Equations of partial dependences of silica extraction rate from zinc product due to studied factors

Equations	Correlation coefficient (R) and its significance as $t_n > 2$	
	R	Significance
$\alpha_1 = 33,91 \cdot 10^{-2} - 8,02 \cdot 10^{-3} (T-273) + 5,17 \cdot 10^{-5} (T-273)^2$	0,98	significant
$\alpha_2 = 9,95 \cdot 10^{-2} \tau, 0,306$	0,99	significant
$\alpha_3 = 43,295 \cdot 10^{-2} - 1,56 \cdot 10^{-3} C + 6,32 \cdot 10^{-6} C^2$	0,69	significant
$\alpha_4 = 40,574 \cdot 10^{-2} - 5,18 \cdot 10^{-3} L$	0	insignificant

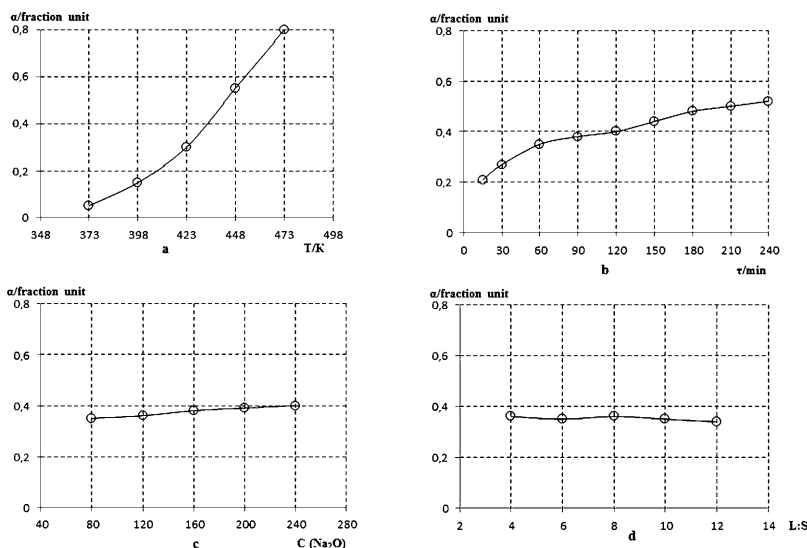
leaching have the greatest influence on the extraction rate of silicon dioxide into solution [7-8]. Thus, the extraction rate of increases by 6 times when the temperature increases from 398 to 473 K (Figure 1, a). With increasing duration of leaching, the curve of silica extraction rate into the solution (Figure 1, b) increases throughout the research interval, without reaching saturation. An increase in the concentration of caustic soda (Figure 1, b) does not significantly affect the extraction rate of silicon dioxide into solution, which is probably due to a change in the properties of the alkali solution itself. It is known that with increasing concentration of caustic soda, the viscosity of the solution increases, and

the solubility of oxygen decreases [9-10]. The partial dependence of the ratio of liquid to solid in the pulp was insignificant (Figure 1, d).

The consensus multivariate equation for the degree of silica extraction into solution, based on significant partial dependencies, has the following form:

$$\alpha_{II} = \frac{[33,91 \cdot 10^{-2} - 8,02 \cdot 10^{-3} (T-273) + 5,17 \cdot 10^{-5} (T-273)^2] [9,95 \cdot 10^{-2} \tau^{0,306}]}{[43,295 \cdot 10^{-2} - 1,56 \cdot 10^{-3} C + 6,32 \cdot 10^{-6} C^2]^{-1} \cdot 0,1325} \quad (1)$$

The calculation using equation 1 gives results (Table 2) that significantly correlates with experimental data ($R = 0,91$, $tR = 24,26 > 2$). However, when substituting the experimental data of the active factors into equation (1), the α II function exceeded the unit one time. Taking



a – temperature; b – duration of leaching; c – initial concentration of alkali; d – ratio L : S

Figure 1 Partial dependences of extraction rate of silicon dioxide (a) into solution on

into account that the multivariate dependence will be used further to determine the optimal conditions and kinetic characteristics of the process of silicon dioxide leaching from the zinc product, equation (1) was introduced into the exponent with an upper limit of one unit

$$\alpha_K = \exp[-0,2522\alpha_{11}^{-0,9659}] \quad (2)$$

or in expanded form

$$\alpha_K = \exp\left\{-0,2522 \left[\frac{[33,91 \cdot 10^{-2} - 8,02 \cdot 10^{-3}(T-273) + 5,17 \cdot 10^{-5}(T-273)^2] [9,95 \cdot 10^{-2} \tau^{0,306}]}{[43,295 \cdot 10^{-2} - 1,56 \cdot 10^{-3}C + 6,32 \cdot 10^{-6}C^2]^{-0,1325}} \right]^{-0,9659} \right\} \quad (3)$$

Coefficients “0,2522” and “0,9659” exponents are determined using the least squares method. High values of $R = 0,90$ and $tR = 21,71$ indicate the adequacy of the multi-factor dependence (3) to desiliconization process of zinc product.

The calculation using equation (3) showed that at a temperature of 443 K, the process duration of 2,0 – 2,5 hours, the initial concentration of sodium oxide of 120 kg/m³, the extraction rate of silicon dioxide into solution from a silica-containing zinc product was 0,62 and 0,64 fr. units.

Since in matrix experiments, the conditions for pulp mixing in autoclaves did not provide a suspended solid state in the pulp, in order to specify the optimal leaching conditions, experiments were performed on desiliconization of zinc product containing Zn – 41,5 and SiO₂ – 17,6 % in a three-liter autoclave equipped with a stirrer and electric heating. Heating and cooling of the pulp was carried out through a metal case. At a temperature of 443 K, the leaching duration of 2 hours, the initial concentration of sodium hydroxide 160 kg/m³ and L : S = 3 : 1, the initial extraction of silicon dioxide from the zinc product into the solution was 60,5 %.

CONCLUSION

Therefore, it is shown that the mathematical model of the process obtained in the form of a generalized

multi-factor equation allows to determine the optimal conditions for desiliconization a silica-containing zinc product when combining different values of temperature, duration of the process and the initial concentration of alkali in the solution.

Paper was performed within grant No. AP05130454.

REFERENCES

- [1] Omarov K. B., Absat Z. B., Aldabergenova S. K., Rakhimzhanova N. Z., Muzapparov A. A. “Use of the chrome (VI) oxide as a sorbent of arsenic from sour copper-containing solutions”, Bulletin of the University of Karaganda-Chemistry, 2017.
- [2] Markova Ye. V., Adler Yu. P., Granovskiy Yu. V. “Chemistry experiment desing”, Journal of the All-Union Chemical Society after named D. I. Mendeleev, 1980.
- [3] Malyshev V. P., Katkeyeva G. L., Zubrina Yu. S., Oskembekov I. M., Gizatullina D. R. “Development of a complex probabilistic-deterministic model of grinding and flotation processes”, Complex Use of Mineral Resources, (2017) 1, 47-53.
- [4] Malyshev V. P. The probabilistic determined planning of experiment. – Alma-Ata: Nauka, 1981, 116.
- [5] Malyshev V. P. Mathematical planning of metallurgical and chemical experiment, Alma-Ata, 1977, 37.
- [6] Kolodziejczak-Radzimska A., Jesionowski T. “Zinc Oxide-From Synthesis to Application: A Review”, Materials, 2014.
- [7] Turebekova K. S., Oskembekov I. M., Bekturganov N. S., Oskembekova Zh. S., Katkeyeva G. L. “On the possibility of fluorine ammonium sulfate opening of batite raw materials”, Complex Use of Mineral Resources, (2017) 1, 58-62.
- [8] Turebekova K. S., Oskembekov I. M., Oskembekova Zh. S., Katkeyeva G. L., Zhunussov E. M. “Research and development of the process scheme of leaching of barytic raw material”, Bulletin of the University of Karaganda-Chemistry, (2018) 1, 92-98.
- [9] Handbook of a chemist, 1964.
- [10] New reference chemist and technologist, 2002, 964.

Note: The responsible translator for English languages is Folmer Yelena Pavlovna, Karaganda, Kazakhstan.