

Enhancement of nitric acid leaching of alumina from kaolin by ultrasonic treatment

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Abstract: This study investigates the effect of ultrasonic cavitation on the nitric acid leaching of alumina from calcined kaolin clays of the Angren deposit. Ultrasonic treatment was applied to intensify mass transfer and improve alumina extraction efficiency. Experiments were performed using a laboratory ultrasonic bath based on a fractional factorial design to evaluate the effects of temperature, leaching time, nitric acid concentration, and acid dosage. A regression model was developed, and the optimum process conditions were determined using the method of steepest ascent. The results showed that ultrasonic activation significantly accelerated the leaching process and increased alumina recovery compared with conventional leaching. Under optimal conditions, the alumina recovery reached 93%, demonstrating the effectiveness of ultrasound-assisted nitric acid leaching. The proposed approach offers an efficient method for improving the hydrometallurgical processing of kaolin raw materials.

Keywords: ultrasonic cavitation; alumina; kaolin clay; nitric acid leaching; hydrometallurgy; process intensification.

INTRODUCTION

Ultrasound has become an important tool for intensifying hydrometallurgical processes because of its ability to improve mass transfer and accelerate chemical reactions in heterogeneous systems. When ultrasonic waves propagate through a liquid medium, cavitation bubbles are formed and subsequently collapse, creating localized regions of high temperature and pressure. These phenomena enhance the interaction between solid particles and the leaching solution, thereby increasing the dissolution rate of valuable components. Based on these principles, Agranat [2] established the theoretical foundations for the controlled application of ultrasonic cavitation in technological processes.

Continuous advances in ultrasonic equipment have expanded its application in extractive metallurgy. Magnetostrictive transducers combined with ultrasonic generators are widely used due to their operational stability, high energy efficiency, and suitability for laboratory and industrial research. Ultrasonic frequencies above 18 kHz have been successfully applied to improve numerous metallurgical operations, including leaching, extraction, crystallization, and purification.

Leaching is the key stage in the recovery of alumina from low-grade aluminosilicate raw materials. However, conventional nitric acid leaching often requires prolonged processing times and considerable acid consumption while providing limited alumina recovery, particularly from iron-bearing kaolin. These limitations increase operating costs and reduce process efficiency.

Consequently, the development of methods capable of intensifying the leaching process and improving alumina extraction remains an important research objective.

The present study investigates the effect of ultrasonic cavitation on the nitric acid leaching of calcined kaolin from the Angren deposit. Particular attention is given to evaluating the influence of operating parameters on alumina recovery and identifying optimal processing conditions using a statistical design of experiments and regression analysis.

RESEARCH METHODOLOGY

Calcined kaolin clay obtained from the Angren deposit (Uzbekistan) was used as the raw material for the experimental investigations. Nitric acid was employed as the leaching reagent, while ultrasonic treatment was applied to intensify the dissolution process. The experiments were carried out in a laboratory-scale ultrasonic bath fabricated from 1X18H9T stainless steel and equipped with a PMS-6M magnetostrictive transducer powered by a UZG-2.5A ultrasonic generator. The reactor was fitted with a mechanical stirrer, electric heater, thermometer, and sampling device to ensure controlled operating conditions throughout the experiments.

The influence of ultrasonic cavitation on alumina leaching was evaluated using a statistical design of experiments. A fractional factorial design (2^{4-1}) was adopted to investigate the effects of four independent variables: leaching temperature (x_1), leaching time (x_2), stoichiometric nitric acid dosage relative to Al_2O_3 content (x_3), and nitric acid concentration (x_4). Alumina recovery in the leach solution was selected as the response variable. Regression coefficients were calculated by the least-squares method, and the method of steepest ascent was applied to determine the optimum operating conditions for the leaching process.

To assess the effectiveness of ultrasonic activation, comparative experiments were conducted under identical conditions with and without ultrasonic exposure. The concentration of dissolved alumina was determined by complexometric titration using EDTA (Trilon B), and the analytical results were verified by solid-phase analysis. The obtained experimental data were processed using regression analysis to evaluate the significance of the investigated parameters and identify the conditions that maximize alumina recovery.

RESULTS

In order to optimize leaching efficiency, minimize nitric acid consumption, and reduce processing time, a series of experiments were carried out involving the ultrasonic-assisted leaching of alumina from kaolin clays using nitric acid. The application of ultrasound was intended to intensify

the dissolution process, enhance phase contact at the solid-liquid interface, and thereby improve the overall recovery of aluminum from the raw material.

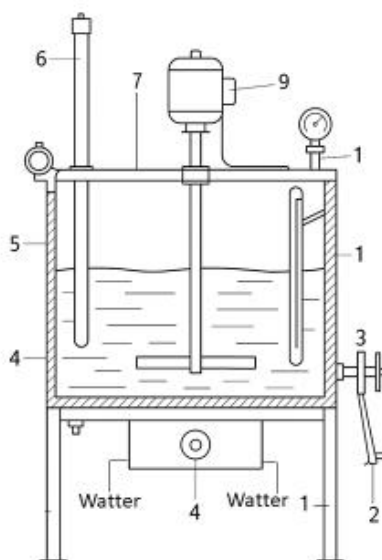


Figure 1. Ultrasonic leaching bath apparatus

1 – ultrasonic bath body, 2 – structural frame, 3 – sampler, 4 – gasket, 5 – magnetostrictive transducer, 6 – control thermometer, 7 – heater, 8 – electric motor with mechanical stirrer, 9 – hinged lid.

As the source of ultrasonic energy, a PMS-6M type magnetostrictive transducer was employed. This transducer was powered by an UZG-2.5A ultrasonic generator, which provided the required high-frequency oscillations to induce cavitation and enhance mass transfer during the leaching process.

The experimental studies were carried out in a custom-designed ultrasonic bath fabricated from 1X18H9T-grade stainless steel, where the bottom of the bath also functioned as a vibrating diaphragm for the magnetostrictive transducer (see Fig. 1). The apparatus was engineered to ensure continuous mechanical agitation of the slurry, enable real-time temperature regulation, and allow sampling during operation without interrupting the leaching process. The frequency of ultrasonic oscillations was held constant throughout the experiments, in accordance with the operational specifications of the UZG-2.5A generator. Maximum anode current was used as a reference point, and oscillation frequency was monitored using an ICH-7 frequency meter to ensure process stability and reproducibility.

The initial raw material used in the experiments was calcined kaolin clay sourced from the Angren deposit. The chemical composition of the material was as follows (in mass %): Al_2O_3 – 24.00; Fe_2O_3 – 2.50; SiO_2 – 73.36; MgO – 0.17; SO_3 – 0.17; K_2O – 0.85; Na_2O – 0.21; loss on ignition –

0.45. The average particle size of the clay fraction was 0.5 mm, ensuring sufficient surface area for interaction with leaching reagents during ultrasonic treatment.

The primary objective of this research was to intensify the leaching processes through the application of ultrasonic cavitation. The study was specifically directed at elucidating the influence of ultrasonic energy on the leaching rate of alumina from kaolin-based raw materials. To identify the optimal operational parameters, the investigation employed a statistical experimental design methodology.

The following process variables were selected as independent (input) factors within the experimental matrix:

x_1 – Temperature (°C);

x_2 – Leaching time (hours);

x_3 – Stoichiometric dosage of nitric acid relative to Al_2O_3 content (%);

x_4 – Concentration of nitric acid in the leaching solution (%).

This structured approach allowed for a comprehensive assessment of both individual and interactive effects of process variables on alumina extraction efficiency under ultrasonic activation.

The response variable used for process optimization in this study was the yield of Al_2O_3 in the leachate (y). In constructing the experimental design, it was assumed that second-order interactions among the factors would have negligible influence on the outcome. Based on this assumption, a fractional factorial design of type 2^{4-1} was employed, which represents a half-replicate of a full 2^4 factorial experiment. The design was generated using the defining relation $x_4 = x_1x_2x_3$, allowing for an efficient yet informative exploration of the main effects and primary interactions among the selected variables.

Table 1. Planning matrix and results of the first series of experiments.

	x_1	x_2	x_3	x_4	y
Basic level	80	1.5	80	20	-
Range of variation	10	0.5	20	5	-
Upper level	90	2	100	25	-
Lower level	70	1	60	15	-
Experience1	+	+	-	-	68.7
2	-	-	-	-	52.5
3	+	-	-	+	85.4
4	-	+	-	+	93.2
5	+	=	+	+	79.3
6	-	-	+	+	59.2
7	+	-	+	-	80.6
8	-	+	+	-	74.6

Based on the data obtained from the first series of experiments, the regression equation describing the relationship between the response variable (y – Al_2O_3 yield in solution) and the coded independent variables (x_1, x_2, x_3, x_4) was constructed using the method of least squares. The following estimates of the regression coefficients were calculated:

$b_0 = 72.93$ – intercept (mean response), $b_1 = -5.56$ – effect of temperature, $b_2 = +3.51$ – effect of leaching time, $b_3 = +0.49$ – effect of stoichiometric dosage, $b_4 = +3.84$ – effect of acid concentration, $b_{12} = -8.01$ – interaction between temperature and time, $b_{13} = +0.96$ – interaction between temperature and dosage, $b_{14} = +0.13$ – interaction between temperature and acid concentration.

The residual standard deviation was calculated as:

$$S\left\{\frac{2}{y}\right\} = 0.687, S^2_{\{b\}} = 0.086, f = 8; F_{ek} = 0.184; F_{0.05}(3; 8) = 4.1$$

The resulting equation for the response function (y – Al_2O_3 yield in solution, %) is as follows:

$$y = 72.93 + 5.45x_1 + 3.51x_2 + 0.49x_3 + 3.48x_4 - 8.01x_1x_2 + 0.96x_1x_3 + 0.13x_1x_4$$

Since the linear effect estimates in the applied experimental design are not confounded by interaction terms, it is valid to apply the method of steepest ascent (also known as the gradient ascent approach) within the linear approximation region. This method enables the identification of the most efficient direction in the factor space to increase the response variable-in this case, the Al_2O_3 yield in solution.

Table 2. Indicators related to determining the direction of vertical lift.

	x_1	x_2	x_3	x_4	y
Initial level	80	1.5	80	20	-
Range of variation	10	0.5	20	5	-
Upper level	90	2	100	25	-
Lower level	70	1	60	15	-
Coefficient b_1	5.65	3.51	0.49	3.84	-
The product of b_1 and the interval of variation	55.6	1.76	9.8	17.4	-
Step size of 5 when measuring x_1	5	0.168	0.88	1.565	-
Rounding	5	0.2	1	2	-
Experience 9 (Beginner Level)	80	1.5	80	20	90
10	85	1.7	81	22	93
11	90	1.9	82	24	88
12	95	2.1	83	25	83
13	100	2.3	84	28	-

At the zero (center) level of the experimental design, the leaching of alumina from Angren kaolin clay was conducted under baseline conditions without deviation along the steepest ascent path. Under these conditions, the Al_2O_3 yield in solution reached 93%, indicating a relatively high extraction efficiency.

The quantitative determination of extracted alumina was carried out using complexometric titration with Trilon B (EDTA). To ensure accuracy, the results were cross-validated by solid phase analysis, confirming the consistency of Al_2O_3 recovery measurements.

To evaluate the specific impact of ultrasonic cavitation on the leaching process, a comparative analysis was performed between experiments conducted with and without ultrasonic exposure. The results demonstrated a notable increase in the leaching rate and shortening of the processing time under the influence of ultrasonic vibrations, confirming the positive intensification effect of ultrasound on alumina dissolution kinetics.

During the experiments, the pulp volume was kept constant across all trials, regardless of the variation in nitric acid concentrations. The mass of kaolin clay samples ranged from 500 to 960 grams, depending on the specific experiment.

In each case, the pulp was first heated to the target process temperature, after which ultrasonic exposure was initiated. From this moment, the leaching time was recorded to ensure consistency in comparing kinetic behaviors under identical thermal conditions.

The results are illustrated in Figure 2, which clearly demonstrates the acceleration of the leaching process and enhanced alumina recovery rates under ultrasonic activation compared to conventional agitation-based leaching.

Experiments have established that 30 minutes after the start of ultrasound exposure on the pulp, an intense reaction occurs in the bath, which almost concludes after 1.5 hours.

In the case of leaching without ultrasonic activation, a comparable increase in alumina yield becomes noticeable only after approximately 2.5 hours of processing. However, despite extending the total leaching time to 3–4 hours, the overall extraction efficiency of Al_2O_3 under these conventional conditions remains limited to 35–40%. This contrast highlights the kinetic advantage provided by ultrasonic cavitation, where enhanced mass transfer and intensified solid–liquid interactions significantly accelerate the leaching process and improve overall recovery.

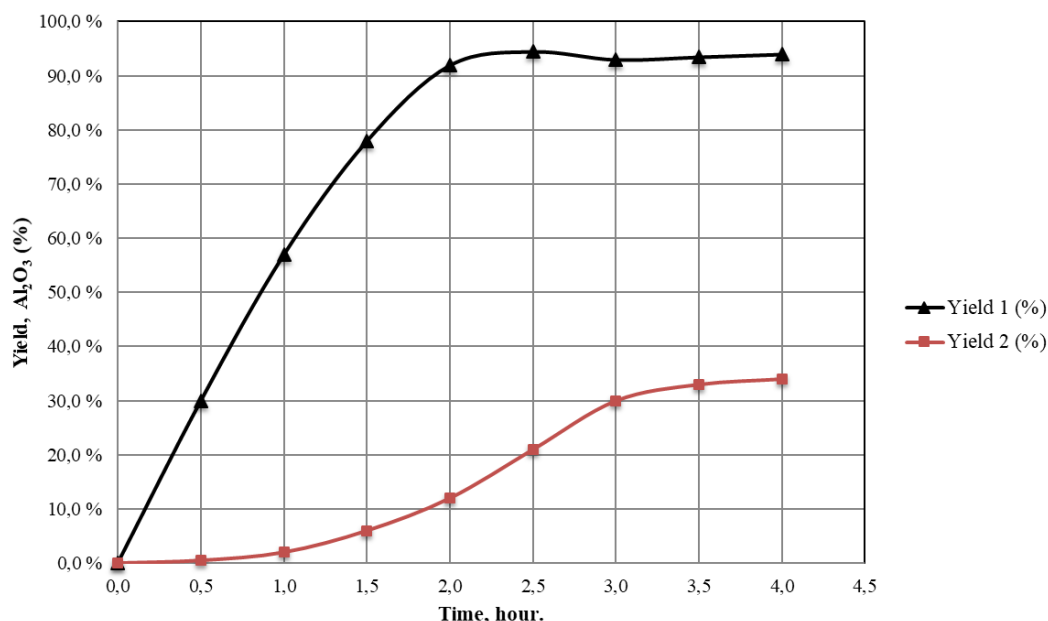


Figure 2. Dependence of Al_2O_3 yield on leaching duration with ultrasonic exposure (curve 1) and without ultrasonic exposure (curve 2); process temperature: 85 °C; nitric acid concentration: 22%.

The results of the present study demonstrate that the application of ultrasonic cavitation significantly enhances the leaching efficiency of alumina from Angren kaolin clays when treated with nitric acid. The observed intensification of the process—manifested in higher Al_2O_3 yields and reduced processing times—clearly underscores the technological potential of ultrasound-assisted hydrometallurgical methods.

Given these promising findings, it is scientifically and practically justified to expand and deepen future investigations in this field. However, it should be noted that the currently available ultrasonic generators and magnetostrictive transducers are not yet sufficiently robust or scalable to fully meet the operational requirements of large-scale industrial alumina production.

Therefore, future research efforts will be directed toward studying the decomposition of kaolin clays in an ultrasonic field under near-industrial conditions, with special attention to the application of a rotary-pulsation apparatus—a promising alternative for intensifying solid–liquid processes in continuous-flow systems.

CONCLUSION

The present study demonstrated that ultrasonic cavitation is an effective method for intensifying the nitric acid leaching of alumina from calcined kaolin clays of the Angren deposit. The application of ultrasonic energy significantly enhanced mass transfer, accelerated the dissolution process, and reduced the leaching time compared with conventional treatment.

Statistical analysis based on a fractional factorial design and regression modeling enabled the evaluation of the effects of temperature, leaching time, nitric acid concentration, and acid dosage on alumina recovery. The optimum operating conditions were determined using the method of steepest ascent, under which the alumina recovery reached 93%.

The obtained results confirm that ultrasonic-assisted nitric acid leaching is a promising approach for improving the hydrometallurgical processing of kaolin raw materials. The proposed technology provides higher extraction efficiency while reducing processing time and has considerable potential for further development and application in industrial alumina production.

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