INFLUENCE OF ULTRASOUND ON NITRIC ACID LEACHING OF ALUMINA FROM KAOLIN CLAYS.

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Abstract: *This article presents the results of a comprehensive study on the application of ultrasound to intensify the nitric acid leaching of alumina from calcined kaolin clays of the Angren deposit. The propagation of ultrasonic waves in liquid media induces cavitation phenomena that enhance mass transfer and reaction kinetics, especially at the solid–liquid interface. A specially designed ultrasonic bath equipped with a PMS-6M magnetostrictive transducer powered by a UZG-2.5A generator was used to investigate process intensification under controlled conditions. The experiments were conducted using a statistical design of experiments (DoE), specifically a fractional factorial design (2^{4-1}), to assess the influence of key parameters: temperature, leaching time, stoichiometric acid dosage, and nitric acid concentration. The optimization criterion was the yield of Al_2O_3 in solution. A regression model was developed, and the method of steepest ascent was applied to identify optimal process conditions. At the optimal point, alumina recovery reached 93% within a significantly reduced leaching time.*

Keywords: *ultrasonic cavitation, alumina leaching, kaolin clay, nitric acid, Angren deposit, hydrometallurgy, process intensification, magnetostrictive transducer, factorial design, aluminum extraction.*

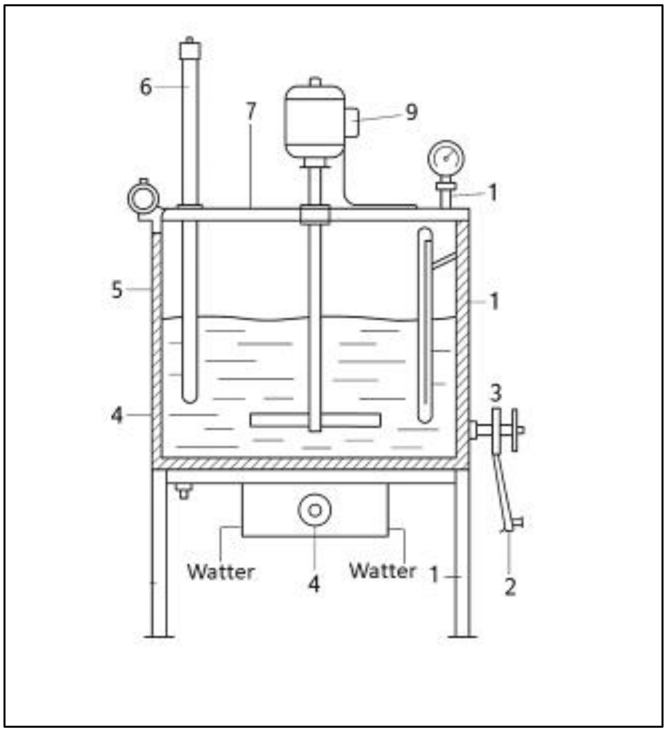
Introduction. The application of ultrasound to enhance various processes and address specific technological challenges in hydrometallurgy has garnered significant interest among researchers. This growing attention is largely attributed to the distinctive effects produced by the propagation of ultrasonic waves through liquid media. A central mechanism in this context is the formation of cavitation zones, along with a range of chemical and physicochemical transformations that occur within the liquid phase. These effects are particularly pronounced at the interfaces between different phases in heterogeneous, fluid-based technological systems, where ultrasound can significantly influence reaction kinetics and mass transfer. Cavitation phenomena — specifically the formation of gas-filled voids or discontinuities within a liquid — arise from the inherent asymmetry in a liquid's mechanical response: while liquids can resist high compressive forces, they are highly susceptible to rupture when subjected to tensile (negative) pressures, leading to the emergence of vapor-filled cavities. Building upon this physical property, B.A. Agranat [2] proposed and provided a theoretical foundation for the controllability of ultrasonic cavitation processes.

In recent years, a wide range of innovative ultrasonic equipment has been developed both domestically and internationally. Among the most widely used configurations are systems incorporating vacuum tube-based generators coupled with magnetostrictive transducers, which remain prevalent due to their reliability and effectiveness in industrial applications.

In modern metallurgical practices aimed at extracting metals and other valuable components from various types of ores, a broad spectrum of mechanical vibrations—spanning diverse frequencies and intensities—is actively employed. In particular, high-energy elastic oscillations with frequencies exceeding 18 kHz (i.e., in the ultrasonic range) are increasingly applied to enhance and accelerate a number of critical technological operations. Among the equipment capable of generating such oscillations, vacuum tube-based ultrasonic generators equipped with appropriate transducers are considered highly suitable, as they fulfill key performance criteria for industrial implementation in terms of power stability, frequency range, and operational reliability.

In the processing of low-grade alumina-bearing raw materials, leaching represents the central stage in the recovery of the target component — alumina. However, when employing nitric acid leaching techniques, particularly agitation or autoclave methods, the treatment of iron-rich alumina-containing feedstocks often results in relatively low alumina recovery rates, accompanied by significantly increased consumption of acid reagents. This not only reduces process efficiency but also raises concerns regarding economic viability and environmental impact.

Methods and 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.

Fig. 1. Ultrasonic leaching bath apparatus

1 – Ultrasonic bath body (housing)

2 – Structural frame (support)

3 – Sampler (sampling valve or port)

4 – Gasket (sealing element)

5 – Magnetostrictive transducer (ultrasonic source)

6 – Control thermometer (manual monitoring)

7 – Heater (thermal unit for solution temperature control)

8 – Electric motor with mechanical stirrer

9 – Hinged lid (top cover for sample insertion/removal)

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
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	-
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.

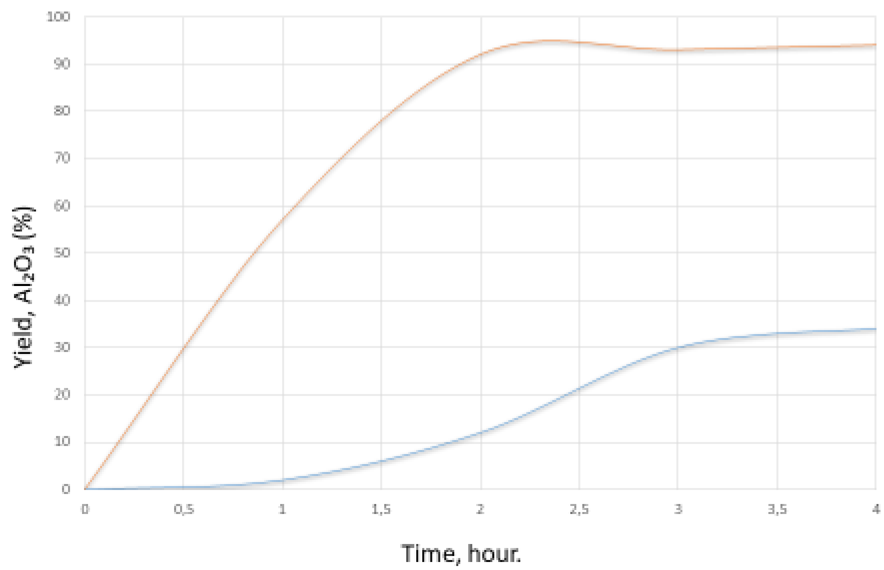
During the experiments, the pulp volume was kept constant across all trials, regardless of the variation in nitric acid concentrations. The mass of kaolin clay samples ranged from 500 to 960 grams, depending on the specific experiment.

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.

Fig. 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 conducted research confirms the high potential of using ultrasonic cavitation to intensify the nitric acid leaching process of alumina from kaolin clays. The application of ultrasound significantly accelerates the dissolution of Al_2O_3, enhances mass transfer, and increases the yield of the target component. Experimental results showed that under optimized conditions, alumina recovery reached up to 93%, while conventional leaching without ultrasound yielded only 35–40% after a longer processing time.

The developed regression model and gradient ascent method enabled the identification of optimal parameter combinations, and the effectiveness of ultrasonic leaching was confirmed by titration and solid-phase analyses. Despite the positive laboratory-scale results, current ultrasonic equipment (generators and transducers) requires further adaptation for industrial-scale operations.

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