DEVELOPMENT OF AUTOCLAVE TECHNOLOGY FOR NITRIC ACID TREATMENT OF ALUMINA FROM KAOLIN CLAYS

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Abstract: The article presents the development of an autoclave technology for nitric acid leaching of alumina from kaolin clays. The research was motivated by the limitations of conventional agitation leaching, including high nitric acid consumption, significant dissolution of iron impurities, and low aluminum extraction efficiency. Laboratory-scale experiments were conducted using raw, calcined, and enriched kaolin clays in a titanium autoclave under varying temperature, acid concentration, and leaching duration conditions. Calcined non-enriched clay was identified as the most suitable material for autoclave treatment. The optimal parameters—temperature of 200°C, 85–90% acid dosage, and 3-hour leaching time—were validated in a 5-liter setup, achieving up to 87% Al_2O_3 extraction with minimal iron co-dissolution.

Keywords: autoclave leaching, alumina extraction, kaolin clay, nitric acid, continuous process, temperature effect

The increased consumption of nitric acid, significant dissolution of iron impurities contained in the raw material, and relatively low extraction of aluminum oxide during the agitation leaching of calcined kaolin clays with 40% nitric acid [1] prompted a shift towards the most advanced method of mineral raw material processing - the autoclave method [2].

On a laboratory scale, we studied the leaching of raw, calcined, and enriched kaolin clays [3] in a one-liter titanium autoclave at temperatures of 150-225°C, with nitric acid dosages of 80-125% and process durations of 1-5 hours. The use of calcined non-enriched material proved to be the most promising. Subsequently, experiments were conducted in a five-liter apparatus, and the optimal process parameters were recommended: temperature - 200°C, dosage of 35% nitric acid - 85-90%, leaching time - 3 hours. The extraction of Al_2O_3 into solution was approximately 87%, while the transfer of iron, due to its hydrolytic precipitation, decreased to 0.5 g/l of Al_2O_3. The application of autoclave technology allowed for a reduction in nitric acid consumption by more than 35%, increased aluminum extraction by 2-3%, and yielded nitrate solutions with an iron modulus exceeding 200.

The aim of this study is to develop the optimal mode for a periodic process in a 25-liter titanium autoclave (Fig. 1) and to investigate the possibility of its implementation in a continuously operating autoclave battery. The effects of process temperature, nitric acid concentration and dosage, and the firing temperature of kaolin clays on the kinetics and degree of aluminum oxide extraction into the solution were examined (Table 1). The process balance indicators for the optimal mode were determined, and consumption coefficients and losses of raw materials, nitric acid, and auxiliary materials were calculated. The balance data revealed that nitric acid losses are significantly lower (by 2-3%) compared to agitation leaching, and these losses can be further reduced by using optimal methods for bringing the autoclave to operating conditions (Fig. 2).

A more in-depth investigation of the periodic (batch) leaching stage enabled the refinement of optimal process parameters and facilitated the acquisition of preliminary data for techno-economic assessments. The analysis demonstrated that scaling up from laboratory to pilot or industrial scales does not significantly impact the fundamental performance indicators of the leaching process. Nevertheless, the batch mode exhibits several critical limitations, foremost among them being the substantial time losses associated with the repeated loading and unloading of material, as well as the cyclical heating and cooling of the leaching apparatus, which negatively affects both operational efficiency and energy consumption.

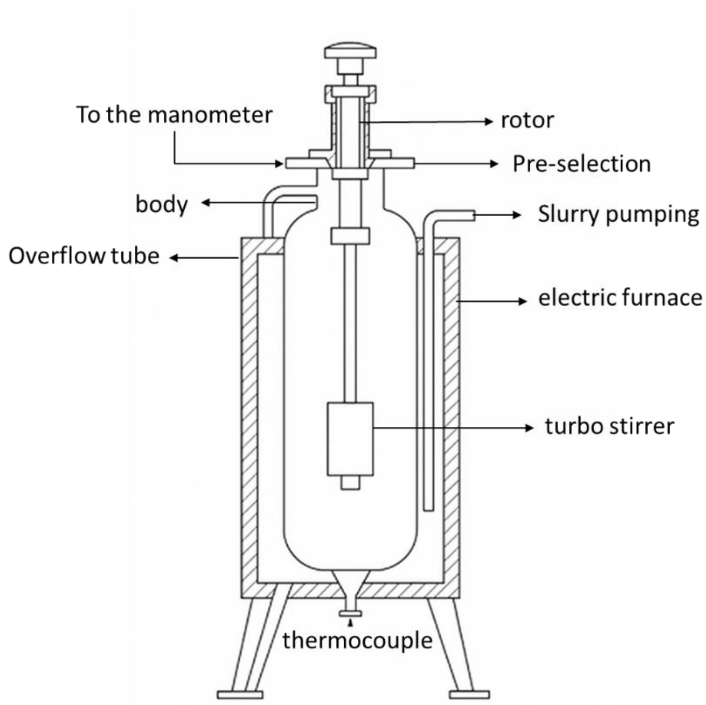

Fig. 1. Batch autoclave with a capacity of 25 l.

Continuity is a fundamental requirement in the development of industrial technological processes, as it enables full automation and mechanization of production operations. This, in turn, leads to significant reductions in labor intensity, processing time, and resource consumption, thereby enhancing overall operational efficiency and economic viability.

We carried out experimental studies on the continuous leaching of alumina-containing materials using an autoclave battery setup, which comprised four sequentially connected autoclaves, a centrifugal pump, a repulpator, a tubular heat exchanger, and a receiving tank. Process temperature control was maintained via a PSR-04 multipoint potentiometric system, with a chromel–copel thermocouple serving as the temperature sensor. Each autoclave was outfitted with a sampling port, a safety relief valve, and a pressure gauge rated for 50 atm, connected through an oil-filled separator to ensure accurate pressure readings under high-temperature conditions. Prior to initiating the leaching trials, the entire unit underwent hydraulic pressure testing at 30 atm to verify system integrity. Due to the absence of a high-pressure acid dosing pump, the first autoclave functioned as an intermediate reservoir, from which the leach pulp was successively transferred to the remaining three operational autoclaves, enabling a quasi-continuous flow mode within the experimental circuit.

Table 1. Effect of Treatment Modes on Nitrate Removal Efficiency from Aqueous Solutions

Nitrate solution	Mode I	Mode II	Mode III
volume, L content, g	10.6	10.5	10.45
Al_2O_3	955	960	962
HNO_3	3645	3690	3705
Rinse water			
volume, L content, g	15.2	14.8	14.9
Al_2O_3	477	480	481
HNO_3	1825	1840	1855
Sludge			

dry weight in grams	5570	5560	5558
Al_2O_3 content in grams	218	210	208
humidity, %	35.2	34.7	35.1
Losses, %			
Al_2O_3	13.3	12.8	12.5
HNO_3	3.1	2.5	1.6
Al_2O_3 Extraction, %	86.7	87.2	87.5

The experiment was conducted according to the following protocol. A centrifugal pump was used to charge pulp into all autoclaves, with the acid dosage pre-calculated to ensure a 90% nitric acid concentration relative to the mass of solid phase. Initially, all three autoclaves operated in batch mode for a duration of two hours. Following this period, the pulp was discharged from the third (final) autoclave, while simultaneously, pulp from the zero (preliminary) autoclave was introduced into the first autoclave. This transfer was driven by excess pressure created by compressed air, allowing us to simulate a quasi-continuous leaching process. Every 30 minutes, the zero autoclave was disconnected from the system, its internal pressure was relieved, and a fresh pulp portion was introduced from the repulpator. During this charging cycle, the discharge from the third autoclave was paused to maintain system balance.

Throughout the experiment, temperature and pressure parameters were continuously monitored in each operational autoclave. Working samples were collected every 30 minutes and analyzed to determine the concentrations of aluminum, iron, and free nitric acid in the liquid phase. In addition, the resulting filter cakes were analyzed for residual aluminum content, providing insight into the leaching efficiency at each stage.

During the initial two hours of operation in periodic (batch) mode, the system approached a transitional concentration regime. This preparatory phase ensured that, upon switching the autoclave battery to continuous operation, a stable concentration of Al_2O_3 and free HNO_3 was rapidly established across all three stages of the system.

In the first stage, following the initiation of fresh pulp loading, a temporary decrease in the alumina concentration in the solution was observed, coinciding with an increase in the free acid content. After approximately 1.5 hours of continuous operation, the concentrations of both Al_2O_3 and HNO_3 stabilized, indicating the onset of steady-state conditions.

In the second stage, the system reached dynamic equilibrium slightly later. Notably, an initial increase in Al_2O_3 concentration up to 88 g/l was recorded, followed by a gradual decline. By the sixth hour of operation, the alumina concentration had stabilized, confirming system equilibrium.

In the third stage, the concentration of Al_2O_3 initially rose to 89.5 g/l, after which a gradual decrease was observed, with the concentration stabilizing in the range of 85–87 g/l. Upon increasing the operating temperature to 200 °C, a significant enhancement in leaching efficiency was observed, with Al_2O_3 concentrations in the third stage exceeding 100 g/l. This result underscores the positive temperature dependence of the leaching kinetics and the potential for process intensification under elevated thermal conditions.

CONCLUSION

The conducted research has demonstrated the technological and economic feasibility of using autoclave leaching with nitric acid for the extraction of alumina from kaolin clays. Laboratory and pilot-scale experiments confirmed that calcined, non-enriched kaolin is the most suitable raw material for this process. The optimal leaching parameters—temperature of 200°C, 85–90% acid dosage, and 3-hour duration—ensured high alumina recovery (up to 87%) with minimal co-dissolution of iron impurities. Transitioning from batch to quasi-continuous operation using a multi-stage autoclave battery enabled more efficient pulp handling and rapid stabilization of Al_2O_3 and HNO_3 concentrations at each stage. Increasing the process temperature further enhanced aluminum recovery, with concentrations exceeding 100 g/L in the final stage. The implementation of continuous autoclave leaching reduces nitric acid consumption by over 35%, improves reagent utilization, and offers a scalable, energy-efficient solution for alumina production from alternative raw materials such as kaolin clay.

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