

INVESTIGATION OF CONTINUOUS AUTOCLAVE TECHNOLOGY FOR ALUMINA PRODUCTION FROM KAOLIN CLAY

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Abstract: This study investigates the development of an autoclave nitric acid leaching technology for the extraction of alumina from kaolin clays as an alternative non-bauxite aluminum resource. The research was motivated by the limitations of conventional atmospheric leaching, including high nitric acid consumption, low alumina recovery, and significant dissolution of iron impurities. Laboratory-scale experiments were conducted using raw, calcined, and enriched kaolin samples in titanium autoclaves under various temperatures, acid dosages, and processing times. The results showed that calcined non-enriched kaolin exhibited the highest leaching performance. The optimal process parameters were determined to be a temperature of 200°C, nitric acid dosage of 85–90%, and a leaching duration of 3 hours, achieving alumina extraction of up to 87% with minimal iron contamination. A multi-stage autoclave battery operating under quasi-continuous conditions was also evaluated. The developed technology reduced nitric acid consumption by more than 35%, improved reagent utilization, and demonstrated strong potential for industrial-scale alumina production from kaolin clay.

Keywords: alumina extraction, kaolin clay, autoclave leaching, nitric acid, continuous processing, aluminum recovery, hydrometallurgy.

Introduction

The growing global demand for aluminum and the gradual depletion of high-grade bauxite reserves have intensified the search for alternative alumina-bearing raw materials. Among these alternatives, kaolin clays have attracted considerable attention due to their widespread availability and significant aluminum content. The development of efficient technologies for alumina extraction from kaolin is therefore of great scientific and industrial importance. In recent years, increasing emphasis has been placed on resource conservation, reduction of reagent consumption, and the utilization of non-traditional mineral resources for sustainable alumina production.

Conventional nitric acid leaching of calcined kaolin under atmospheric agitation conditions is associated with several technological challenges, including high nitric

acid consumption, substantial dissolution of iron impurities, and relatively low alumina recovery. These limitations reduce process efficiency and increase production costs. Consequently, the application of autoclave leaching technology, which operates under elevated temperature and pressure conditions, has emerged as a promising approach for enhancing the extraction of alumina while minimizing reagent losses and impurity dissolution.

The article investigates the development of an autoclave nitric acid leaching process for alumina recovery from kaolin clays. Laboratory and pilot-scale experiments were conducted using raw, calcined, and enriched kaolin samples under various temperature, acid dosage, and leaching time conditions. Particular attention was given to determining the optimal technological parameters and evaluating the feasibility of scaling the process from batch operation to a quasi-continuous autoclave system. The influence of operating conditions on alumina extraction efficiency, nitric acid consumption, and iron dissolution was systematically analyzed.

Furthermore, the study explores the implementation of a multi-stage autoclave battery designed to achieve continuous processing. Continuous operation offers significant advantages, including improved automation, reduced labor intensity, lower energy consumption, and enhanced overall process efficiency. The findings demonstrate that autoclave leaching at elevated temperatures can substantially improve alumina recovery while reducing nitric acid consumption, thereby providing a technologically and economically viable route for alumina production from kaolin clay.

Therefore, the research contributes to the advancement of alternative alumina production technologies and provides a scientific basis for the industrial application of nitric acid autoclave leaching processes using kaolin as a non-bauxite aluminum source.

Research Methodology

The experimental studies were conducted to develop an efficient autoclave nitric acid leaching technology for alumina extraction from kaolin clays. The research involved the investigation of raw, calcined, and enriched kaolin samples under various processing conditions to determine the optimal technological parameters for maximum aluminum recovery and minimum impurity dissolution.

Initially, laboratory-scale experiments were carried out using a 1-liter titanium autoclave. The effects of temperature (150–225°C), nitric acid dosage (80–125% of the stoichiometric requirement), and leaching duration (1–5 hours) on alumina extraction were systematically examined. Prior to leaching, selected kaolin samples were

subjected to thermal activation through calcination in order to enhance their reactivity toward nitric acid treatment.

Based on the laboratory results, the most promising material, namely calcined non-enriched kaolin clay, was selected for further investigation. Pilot-scale experiments were then performed in a 5-liter and subsequently a 25-liter titanium autoclave to evaluate process scalability and to verify the optimal operating conditions. The influence of process temperature, nitric acid concentration, acid consumption, and treatment duration on the extraction efficiency of Al_2O_3 was analyzed. The experimental studies were conducted using a 25-L titanium autoclave. The schematic representation of the autoclave system is shown in Figure 1.

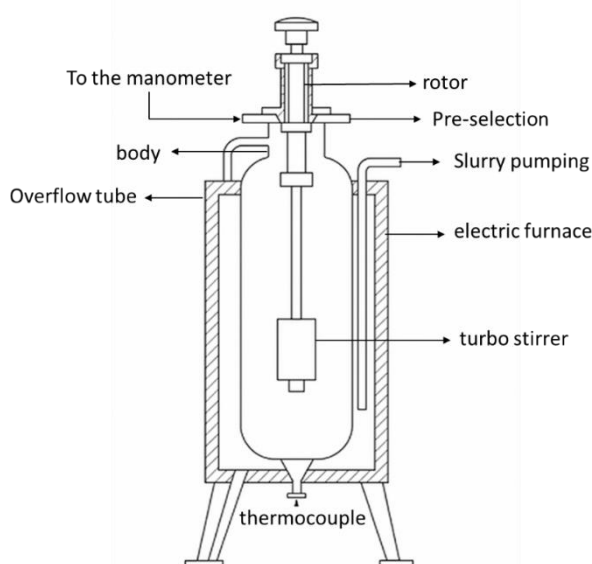


Figure 1. Batch autoclave with a capacity of 25 l.

To assess the feasibility of continuous industrial operation, a multi-stage autoclave battery consisting of four interconnected autoclaves, a centrifugal pump, a repulpator, a tubular heat exchanger, and a receiving tank was employed. The system was operated under quasi-continuous conditions, where pulp transfer between stages was achieved using compressed air pressure. Temperature and pressure were continuously monitored using a potentiometric control system equipped with chromel-copel thermocouples and pressure gauges.

Samples of the liquid and solid phases were collected at regular intervals throughout the experiments. The concentrations of aluminum, iron, and free nitric acid in the leach solutions were determined using standard analytical procedures. The filter cakes obtained after leaching were analyzed for residual aluminum content to calculate extraction efficiency. Process performance was evaluated based on alumina recovery,

nitric acid consumption, iron dissolution, material balance indicators, and reagent losses.

The obtained experimental data were used to establish the optimal technological parameters and to evaluate the technical feasibility of implementing continuous autoclave leaching for industrial alumina production from kaolin clay.

Results and discussion

The experimental investigation demonstrated that the efficiency of alumina extraction from kaolin clay strongly depends on the leaching temperature, nitric acid dosage, and processing time. Comparative tests performed on raw, enriched, and calcined kaolin samples revealed that calcined non-enriched kaolin exhibited the highest reactivity during nitric acid leaching. Thermal activation of kaolin significantly improved the accessibility of aluminum-bearing phases, resulting in higher extraction efficiencies compared with untreated materials.

The effect of temperature on the leaching process was particularly pronounced. Increasing the temperature from 150°C to 200°C led to a substantial increase in Al_2O_3 dissolution, whereas further temperature elevation produced only marginal improvements while increasing energy consumption. Under optimal conditions, namely a temperature of 200°C, nitric acid dosage of 85–90%, and a leaching duration of 3 hours, the extraction of alumina reached approximately 87%. Simultaneously, the concentration of dissolved iron remained low due to hydrolytic precipitation under autoclave conditions.

Pilot-scale experiments conducted in 5-liter and 25-liter titanium autoclaves confirmed the reproducibility of laboratory results. Material balance calculations showed that nitric acid losses during autoclave leaching were significantly lower than those observed in conventional agitation leaching processes. The reduction in acid consumption exceeded 35%, indicating improved reagent utilization and lower operational costs. The performance indicators obtained under different operating modes are summarized in Table 1. The results demonstrate that Mode III provided the highest alumina extraction efficiency (87.5%) while minimizing nitric acid losses (1.6%).

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 ₂ O ₃	955	960	962
HNO ₃	3645	3690	3705
Rinse water			
volume, L	15.2	14.8	14.9
content, g			
Al ₂ O ₃	477	480	481
HNO ₃	1825	1840	1855
Sludge			
dry weight in grams	5570	5560	5558
Al ₂ O ₃ content in grams	218	210	208
humidity, %	35.2	34.7	35.1
Losses, %			
Al ₂ O ₃	13.3	12.8	12.5
HNO ₃	3.1	2.5	1.6
Al ₂ O ₃ Extraction, %	86.7	87.2	87.5

The losses of Al₂O₃ and HNO₃ obtained under different operating modes are illustrated in Figure 2. As shown, the transition from Mode I to Mode III resulted in a gradual decrease in both alumina and nitric acid losses. Al₂O₃ losses decreased from 13.3% to 12.5%, while HNO₃ losses were reduced from 3.1% to 1.6%. These results indicate improved process efficiency and reagent utilization under Mode III conditions, confirming the advantages of optimized autoclave operation.

The transition from batch to quasi-continuous operation using a multi-stage autoclave battery resulted in improved process stability and productivity. During continuous operation, stable concentrations of Al₂O₃ and free HNO₃ were established throughout the system after an initial adjustment period. In the first stage of operation, a temporary decrease in alumina concentration was observed due to the introduction of fresh pulp; however, steady-state conditions were achieved within approximately 1.5 hours.

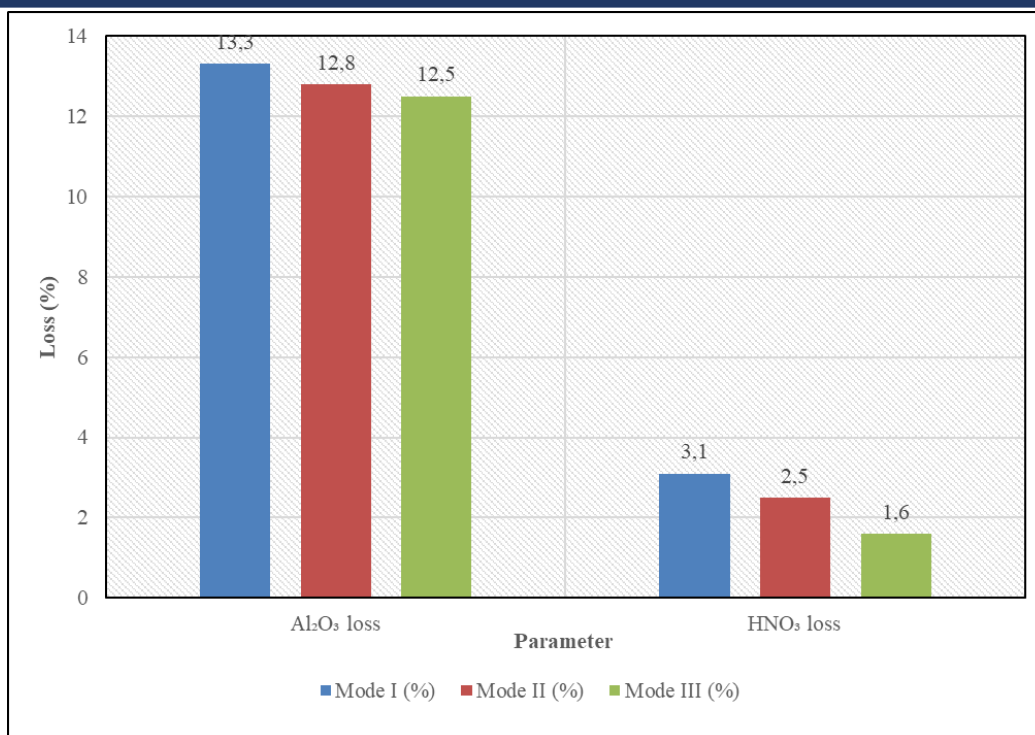


Figure 2. Effect of operating mode on Al₂O₃ and HNO₃ losses during autoclave nitric acid leaching of kaolin clay.

To evaluate the stability of the continuous autoclave process, the concentration of dissolved alumina was monitored at different stages of the autoclave battery. The obtained results are presented in Figure 3. The concentration profiles indicate that after an initial transition period, the system gradually approached steady-state conditions. Stage 1 stabilized at approximately 84 g/L, whereas Stages 2 and 3 reached equilibrium concentrations of about 85 g/L, confirming efficient mass transfer and stable process operation.

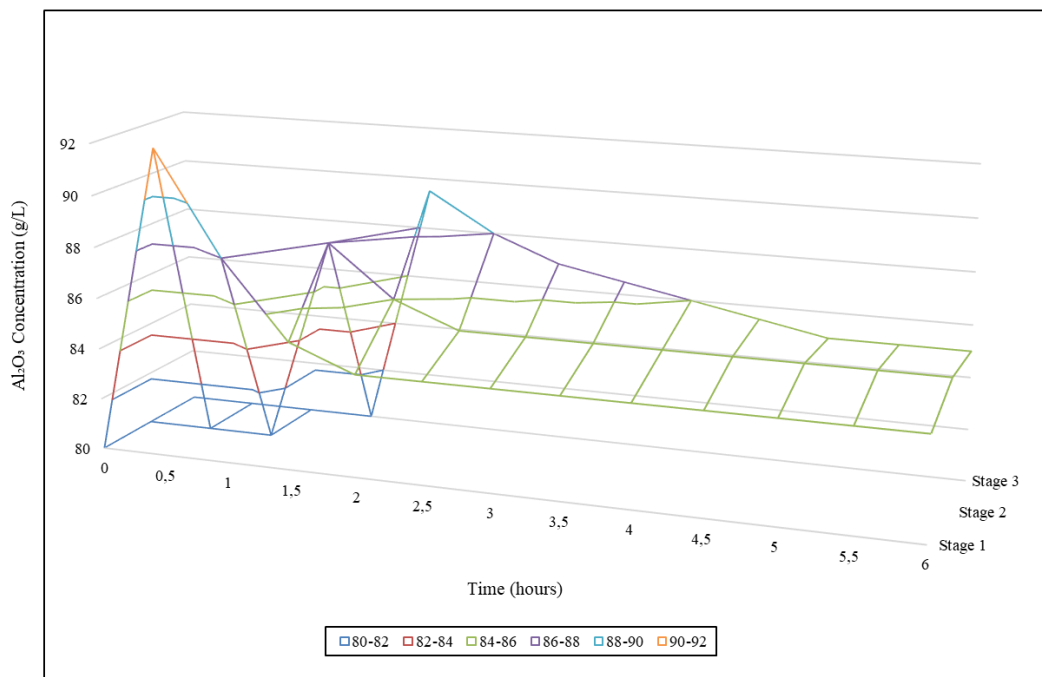


Figure 3. Changes in Al_2O_3 concentration with time in the three-stage continuous autoclave leaching system.

As shown in Figure 3, the alumina concentration in all stages gradually approached equilibrium values during continuous operation. The highest concentration was observed in Stage 3, reaching approximately 89.5 g/L during the initial period before stabilizing near 85 g/L. These results demonstrate the effectiveness of the multi-stage autoclave configuration in maintaining stable alumina extraction and ensuring continuous process performance. In the second and third stages, alumina concentrations gradually increased and subsequently stabilized. The maximum Al_2O_3 concentration reached approximately 89.5 g/L under standard operating conditions. When the process temperature was increased to 200°C, the concentration of dissolved alumina exceeded 100 g/L in the final stage of the autoclave battery. These results confirm that elevated temperature positively influences leaching kinetics and enhances aluminum recovery.

The obtained results demonstrate that autoclave nitric acid leaching is an effective method for processing kaolin clay and producing alumina-rich nitrate solutions. The combination of high alumina recovery, low iron contamination, and reduced acid consumption highlights the technological advantages of the proposed process. Furthermore, the successful implementation of a quasi-continuous autoclave system indicates strong potential for industrial-scale application and contributes to the development of alternative alumina production technologies based on non-bauxite raw materials.

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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