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# Enhancing Solid–Liquid Separation In Nitric Acid Leaching Of Low-Grade Alumina Ores

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***Abstract**: This study investigates the enhancement of solid–liquid phase separation in nitric acid-leached pulps derived from low-grade alumina-containing raw materials, including kaolin and substandard bauxites. The application of polyelectrolyte flocculants (K-4, K-6, K-9, and AKS) was assessed under both laboratory and pilot-scale conditions. Key influencing parameters such as pH, flocculant dosage, particle size distribution, and liquid-to-solid ratios were systematically evaluated. Results indicate that flocculants significantly accelerate sedimentation and improve filtration efficiency, with K-4 showing the highest performance. Optimal clarification occurred within 20 minutes, achieving up to a 4-fold increase in sedimentation rate and a 2.7-fold increase in filtration rate compared to untreated pulps. Experimental findings were supported by chemical and mineralogical analysis of Vorukhsky, Turgaysky, and Tikhvinsky bauxites, revealing distinct responses to flocculation based on composition and dispersion behavior. Additionally, the residual flocculant was found to enhance the efficiency of sludge washing across multiple stages. The outcomes provide practical insights for optimizing the hydrometallurgical processing of low-grade aluminum ores, contributing to more efficient and sustainable alumina recovery via nitric acid technology.*

**Keywords:** *alumina extraction, nitric acid leaching, solid-liquid separation, polyelectrolyte flocculants, kaolin and bauxite pulp, filtration efficiency.*

## INTRODUCTION

The growing demand for alumina as a primary raw material for the aluminum industry has intensified research on the efficient processing of low-grade ores and secondary resources. Conventional methods, particularly the Bayer process, often face significant limitations when applied to kaolin and substandard bauxites due to their high silica content, fine dispersion of residues, and the formation of complex by-products [1]. As a result, alternative hydrometallurgical approaches, such as nitric acid leaching, have gained increasing attention for their ability to handle diverse alumina-bearing raw materials with improved recovery rates.

One of the major challenges associated with nitric acid technology lies in the separation of solid and liquid phases following autoclave decomposition [2]. The fine particle size distribution, elevated viscosity, and density of nitrate-based solutions hinder sedimentation and filtration, leading to reduced process efficiency and increased operational costs. To overcome these limitations, the use of synthetic polyelectrolyte flocculants has emerged as a promising solution [3]. By promoting aggregation of suspended particles, flocculants accelerate sedimentation, enhance clarification, and significantly improve filtration performance. Recent advances in polymer chemistry have introduced a range of flocculants, such as K-4, K-6, K-9, and AKS, which differ in molecular structure, functional groups, and physicochemical properties [4]. Their performance is strongly influenced by pulp characteristics, including pH, liquid-to-solid ratio, and mineralogical composition of the raw material. Laboratory and pilot-scale studies have demonstrated that properly selected flocculants can improve sedimentation and filtration rates by more than two-fold, thereby offering a viable pathway for industrial-scale implementation [5].

## METHODOLOGY

In this study, kaolin and substandard bauxite ores obtained from the Vorukhsky, Turgaysky, and Tikhvinsky deposits were used as primary raw materials. Prior to the leaching process, kaolin samples were subjected to thermal dehydration at 630–650 °C in order to enhance their reactivity, while the bauxite samples were analyzed for their chemical and mineralogical composition, particle size distribution, and dispersion characteristics [6]. Nitric acid leaching was performed under autoclave conditions at 70 °C with liquid-to-solid ratios ranging from 4:1 to 8:1, which provided slurries of densities between 1.31 and 1.37 g/cm³ and pH values within the range of 0.1 to 1.0. The pulps contained up to 70% of finely dispersed particles smaller than 0.063 mm, which made solid–liquid separation particularly challenging [7].

To address this issue, synthetic polyelectrolyte flocculants including K-4, K-6, K-9, and AKS were applied to the nitric acid pulps at dosages between 38 and 113 g/m³. The flocculants were introduced during continuous stirring, and their influence on sedimentation and clarification was monitored by measuring the height of the clarified liquid layer at regular time intervals ranging from 5 to 60 minutes [8]. Comparative tests were carried out using pulps without additives to establish baseline clarification rates. The performance of the flocculants was evaluated by assessing the settling rate and the degree of supernatant transparency [9].

Filtration experiments were conducted on both laboratory and pilot scales. In the laboratory, a nutsch filter equipped with "PU" brand filter fabric was employed under a vacuum of 0.45–0.50 kg/cm², with parameters such as specific output of the solid phase, solution flow rate, and cake humidity being recorded [10]. For validation purposes, pilot-scale experiments were carried out with pulp preheated to 75–80 °C, where filtration performance was compared to laboratory results in order to account for process-specific and operational variations.

Additional experiments were devoted to sludge washing under countercurrent conditions in order to determine the effect of residual flocculants adsorbed on the solid phase [11]. Filtration rates were measured across three consecutive washing stages, while the chemical composition of the wash water was analyzed, particularly focusing on $Al_2O_3$ and $Fe_2O_3$ concentrations [12]. The presence of residual flocculant was observed to enhance washing efficiency and reduce the loss of valuable components. All experimental results were processed statistically to identify key operational trends, and comparative analysis was performed to rank the effectiveness of individual flocculants. Mineralogical and chemical data of the ores were further used to interpret the observed differences in clarification and filtration performance between kaolin and various bauxite types [13].

In the processing of kaolin and low-grade bauxite ores for alumina production via the nitric acid method, the separation of solid and liquid phases following autoclave decomposition presents significant operational challenges [14]. These difficulties arise from the fine dispersion of solid residues and the elevated viscosity and density of nitrate-based aluminum solutions. To enhance the efficiency of this phase separation, the application of flocculant precipitants has been identified as one of the most effective approaches [15].

The effectiveness of synthetic polyelectrolyte flocculants largely stems from the characteristics of their aqueous solutions, which are influenced by multiple parameters such as the flocculant's working concentration, the acidity (pH) of the system, the surface charge and size distribution of suspended solids, the pulp temperature, and other environmental factors [16]. Additionally, the selection of an appropriate synthetic flocculant must consider its molecular composition, the presence and density of functional groups, as well as its intrinsic physicochemical properties.

Recent studies indicate that polyelectrolytes such as K-4, K-6, K-9, and ACS exhibit microheterogeneous structures composed of diverse aggregates, including coiled macromolecules and extended fibrillar elements, which associate through parallel alignment into bundle-like formations [17]. As reported by K. S. Akhmedov and S. Zaynutdinov, the polymer backbone of K-4 incorporates various functional groups, including amide ($-CONH_2$), carboxyl groups (in both $-COOH$ and $-COO^-$ forms), as well as cyclic imides. The presence of imide and amide functionalities arises as intermediate species during the alkaline saponification of polyacrylonitrile [18].

**Table 1. Dependence of the clarification efficiency of nitric acid-treated kaolin slurry on the type of flocculant used.**

| Settling time, min. | Height of the clarified slurry layer | | | | | |
|---|---|---|---|---|---|---|
| | without additives | | with an additive | | | |
| | ml | % | AKC – 4 | | K – 4 | |
| | | | ml | % | ml | % |
| 10 | 2 | 4 | 12.6 | 25.3 | 12.8 | 25.6 |
| 20 | 4.1 | 8.2 | 16.05 | 32.1 | 15.9 | 31.8 |
| 30 | 5.3 | 12.7 | 19.8 | 39.6 | 18.05 | 36.1 |
| 40 | 6.5 | 13.1 | 20.08 | 40 | 18.23 | 36.7 |
| 50 | 6.8 | 13.6 | 20.11 | 40.2 | 18.34 | 36.9 |
| 60 | 7.1 | 14.1 | 20.15 | 40.3 | 18.6 | 37.2 |

This study investigates a method for accelerating the separation of solid and liquid phases in nitric acid pulps by introducing flocculants—specifically, polyelectrolytes K-4, K-6, K-9, and AKS. Experimental work was carried out to enhance the efficiency of phase separation in nitric acid pulps formed during the leaching of thermally dehydrated kaolin clay (calcined at 630–650°C) with nitric acid. The resulting slurry exhibited the following characteristics at 70°C: slurry density ranged from 1.3614 to 1.367 g/cm³; supernatant density (urea-containing solution) was between 1.240 and 1.243 g/sm³; pH of the slurry ranged from 0.94 to 1.05. The mass fraction of solid particles smaller than 0.063 mm accounted for 60–70%, and the dynamic viscosity of the solution was in the range of 1.104 to 1.120 mPa·s.

Previous studies have demonstrated that the addition of flocculating agents significantly enhances the sedimentation and filtration rates of nitric acid pulps, with the efficiency being dependent on the dosage of K-4 and K-6 polyelectrolytes [20]. Table 1 and Figure 1 present data on the sedimentation behavior of autoclaved kaolin suspensions treated with AKS and K-4 flocculants. The experimental conditions were as follows: flocculant dosage – 60 g/m³, temperature – 70°C, and a liquid-to-solid ratio (L:S) of 4:1. The introduction of flocculants promotes aggregation of suspended particles, thereby improving phase separation. It was also observed that extending the settling time beyond 30 minutes produced no substantial improvement in clarification efficiency (see Table 1).

In subsequent experiments, the filtration behavior of autoclaved pulp and the optimal conditions for sludge washing were examined. The presence of polymer-based flocculants was found to enhance filtration productivity by a factor of 2.5 (as shown in Table 2). Furthermore, the amount of flocculant adsorbed onto the solid phase was sufficient to accelerate the washing of the sediment, without causing any measurable loss of the target components.

**Table 2. Effect of flocculant type on the filtration rate of nitric acid-leached kaolin slurry**

| Pulp | Specific output | | |
|---|---|---|---|

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| | for the solid phase, kg/m²·h | solution flow rate, m³/m²·h | Humidity, % | Increase in filtration rate, relative units |
|---|---|---|---|---|
| Without additives | 130 – 150 | 0.25 – 0.30 | 40 – 45 | – |
| With an additive | | | | |
| K – 4 | 340 – 405 | 0.65 – 0.72 | 40 – 42 | 2.60 – 2.70 |
| AKC | 340 – 400 | 0.63 – 0.75 | 40 – 42 | 2.60 – 2.70 |

**The flocculant consumption rate is 60 g/m³.*

Large-scale industrial tests on the sedimentation and filtration of nitric acid-leached kaolin suspensions with flocculant additives confirmed the findings previously established in laboratory experiments (Table 3).

Filtration of the pulp was performed using a nutsch filter equipped with a "PU" brand filter fabric. The process involved feeding the pulp, preheated to 75–80°C, from an intermediate tank through a cork valve into the filter. A vacuum was generated using an RMK-3 water jet pump and maintained within the range of 0.45–0.50 kg/sm² throughout the operation.

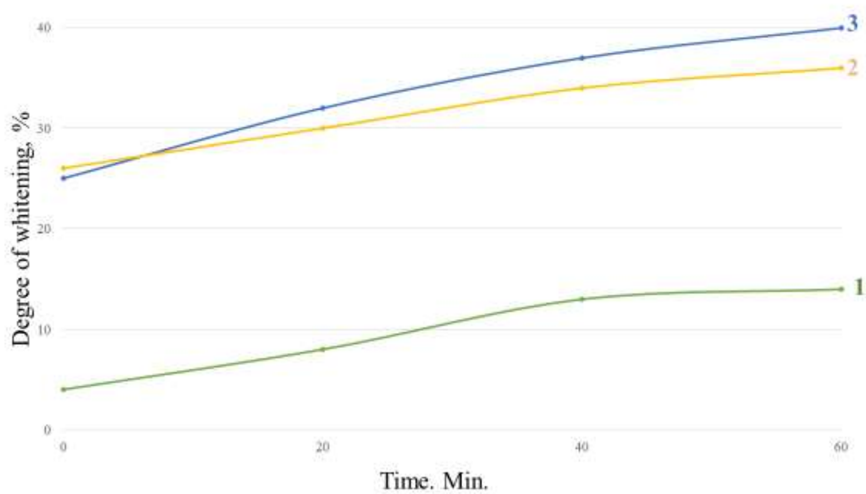


**Fig. 1. Dependence of the clarification efficiency of nitric acid-leached kaolin pulp on the flocculant type used 1 - pulp without additives; 2 - with preparation K-4;3 - with preparation AKS.**

In the enlarged-scale experiments, the pulp filtration rate demonstrated an increase by a factor of 2.2 to 2.3, which, although substantial, remained slightly below the values obtained under controlled laboratory conditions. This discrepancy may be attributed to process-specific variables and operational limitations inherent to large-scale systems.

**Table 3. Effect of flocculant type on the clarification degree of autoclave pulp**

| Settling time, min. | Degree of pulp clarification, % | | | The increase in clarification rate (multiplicity) in the case | |
|---|---|---|---|---|---|
| | without additives | with an additive | | | |
| | | K – 4 | K – 6 | K – 4 | K – 6 |
| 10 | 4 | 19.1 | 18.1 | 4.7 | 4.5 |
| 20 | 6 | 26.2 | 25.1 | 4.3 | 4.1 |
| 30 | 11 | 33.5 | 33.4 | 3 | 3 |
| 40 | 12.8 | 36.6 | 35.2 | 2.9 | 2.8 |
| 50 | 14.2 | 39.1 | 37.5 | 2.6 | 2.4 |
| 60 | 15.2 | 41.3 | 40.2 | 2.4 | 2.3 |

**The flocculant dosage is 60 g/m³, and the temperature is 70°C.*

Verification of the filtration behavior of solid phases (shtof) formed during the countercurrent washing process (see Fig. 2) revealed that the presence of residual flocculant across all three washing stages contributed to a noticeable enhancement in filtration efficiency. The data presented in Table 5 confirm the acceleration of the filtration process as a result of this residual flocculant effect.

**Table 4. Filtration performance of acidic kaolin slurry under pilot-scale conditions**

| Pulp | Specific output | | Increase in filtration rate, relative units |
|---|---|---|---|
| | for the solid phase, kg/m²·h | solution flow rate, m³/m²·h | |

| | | | |
|---|---|---|---|
| Without additives | 130 – 150 | 0.25 – 0.30 | – |
| K – 4 | 340 – 405 | 0.65 – 0.72 | 2.60 – 2.70 |
| AKC | 340 – 400 | 0.63 – 0.75 | 2.60 – 2.70 |

Thus, the application of flocculating agents enhances the solid–liquid separation process in nitric acid-treated kaolin suspensions. Furthermore, the adsorption of the flocculant onto the sludge contributes to a more efficient filtration process during the treatment of industrial wastewater.

The influence of flocculating additives on the separation of solid phases from pulp generated during the leaching of low-grade bauxites from the Vorukhsky, Turgaysky, and Tikhvinsky deposits was investigated. These bauxite samples exhibit notable differences in both chemical composition and mineralogical structure.

**Table 5. The chemical composition of substandard bauxites (in %) is as follows:**

| Content of components, % | Vorukhsky | Turgaysky | Tikhvinsky |
|---|---|---|---|
| $SiO_2$ | 41.48 | 13.39 | 14.32 |
| $SiO_3$ | 0.08 | 0.33 | 0.03 |
| $Al_2O_3$ | 43.50 | 46.66 | 44.76 |
| $Fe_2O_3$ | 11.84 | 11.89 | 15.39 |
| CaO | 0.33 | 1.12 | 4.65 |
| MgO | 0.04 | 0.04 | 0.04 |
| $K_2O$ | 0.20 | 0.07 | 0.15 |
| $Na_2O$ | 0.20 | 0.30 | 0.11 |
| ∑ | 100.02 | 100.52 | 99.53 |
| O.O.C. | 2.35 | 26.95 | 20.10 |

The particle size distribution data are presented in Table 7.

**Table 6. Effect of adsorbed flocculant amount on sishtof washing efficiency and composition of resulting wash water**

| Mash washing stage | Mortar application rate: $m^3/m^2 \cdot h$ | Concentration in solution, g/l | | pH of the solution |
|---|---|---|---|---|
| | | $Al_2O_3$ | $Fe_2O_3$ | |
| Without additives | | | | |
| III | 0.40 – 0.50 | 35 – 40 | 0.15 – 0.22 | 1.0 – 2.3 |
| II | 0.50 – 0.61 | 17 – 20 | 0.05 – 0.06 | 3.5 – 4.0 |
| I | 0.70 – 0.80 | 1.5 – 2.0 | – | 4.5 – 5.2 |
| With the addition of K – 4 | | | | |
| III | 0.85 – 0,95 | 32 – 35 | 0.15 – 0.22 | 2.0 – 2.4 |
| II | 1.0 – 1.1 | 13 – 16 | 0.05 – 0.06 | 3.5 – 4.0 |
| I | 1.1 – 1.3 | 1.5 – 2.0 | – | 4.5 – 5.0 |
| With the addition of K – 6 | | | | |
| III | 0.8 – 0.9 | 32 – 35 | 0.15 – 0.23 | 2.0 – 2.4 |
| II | 0.95 – 1.05 | 15 – 17 | 0.05 – 0.06 | 3.5 – 4.0 |
| I | 1.05 – 1.2 | 1.5 – 2.0 | – | 4.5 – 5.0 |

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Given the relatively high $SiO_2$ content (41.8%) in Vorukh bauxite, this material was selected to study the effect of flocculating additives on the pulp separation process. The nitric acid pulp, prepared by leaching Vorukh bauxite at a specific gravity of 1.310–1.321, with a pH range of 0.1 to 0.3 and a liquid-to-solid ratio of 8:1, was subjected to separation experiments conducted both in the presence and absence of flocculants.

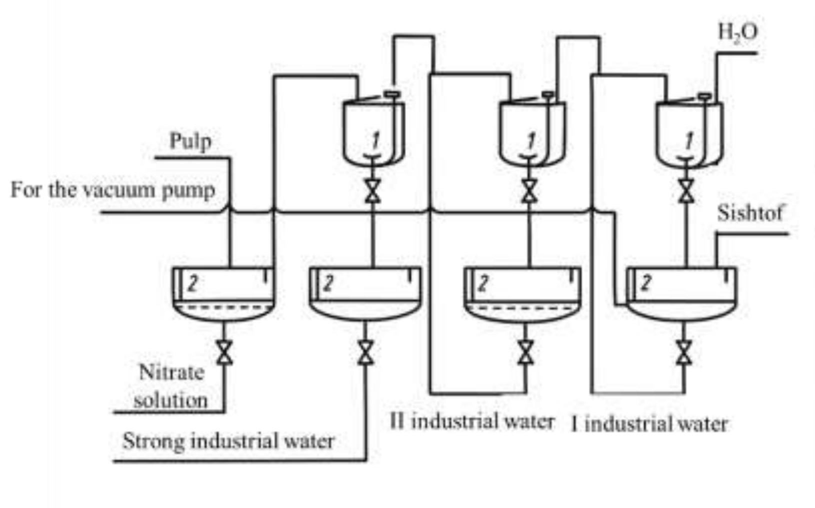


**Fig. 2. Schematic representation of the equipment and technological line used for pulp filtration and subsequent washing of the filter sishtof. 1 - repulpator, 2 - nutsch filter.**

The data in Table 8 and the graphical representation in Figure 3 demonstrate how the effectiveness of pulp clarification is influenced by the specific type of flocculant and its consumption rate.

**Table 7. Particle size distribution of substandard bauxites**

| Bauxite | Fractional composition, % | | | | | | |
|---|---|---|---|---|---|---|---|
| | <0.20 mm | <0.18 mm | <0.10 mm | <0.083 mm | <0.05 mm | 0.05 mm or less | ∑ |
| Vorukhsky | 12.1 | 9.3 | 16.5 | 8.7 | 37.1 | 14.8 | 99.7 |
| Turgaysky | 0.5 | 11.1 | 27.8 | 11.2 | 21.6 | 26.7 | 99.5 |
| Tychvinsky | 0.6 | 12.2 | 27.3 | 12.1 | 12.1 | 22.9 | 99.9 |

In terms of efficiency, the tested flocculants can be ranked in the following order: K-4, K-9, and K-6. The majority of pulp clarification—up to 65%—occurred within the first 20 minutes after flocculant addition. Extending the settling time or increasing the flocculant dosage beyond this point did not result in a significant enhancement of the clarification index; rather, it only contributed to the further compaction of the thickened pulp. In all cases, the use of flocculants accelerated the clarification process by approximately 3.5 to 4 times. As a result, the supernatant solution became visually transparent.

The experimental procedure was carried out as follows: after the leaching stage, the resulting pulp was subjected to solid-liquid separation via filtration. The solid residue was then washed, dried, and fractionated using a standard set of sieves. The size-classified solid fractions were subsequently introduced into an aluminum nitrate solution preheated to 70 °C at a liquid-to-solid ratio of 8:1, followed by mechanical stirring. Flocculating additives were then added to the prepared pulp, and their influence on the clarification rate was systematically investigated.

Under the optimal conditions previously established for bauxite from the Vorukhsky deposit, further studies were carried out using pulps obtained from the leaching of bauxites from the Turgaysky and Tikhvinsky deposits. These pulps were characterized by low pH values, specific liquid-to-solid ratios, and particular specific gravities. The influence of flocculating agents on the settling rate of these pulps is summarized in Table 9. A comparison of clarification data indicates that, in the absence of flocculant additives, the settling rate remains low. However, the introduction of equal quantities of flocculating reagents significantly accelerates the phase separation process. The effectiveness of the reagents varies, likely due to differences in the dispersion characteristics, chemical composition, and mineralogical features of the raw materials.

Thus, it has been established that the rate of solid-liquid phase separation in pulps formed during autoclave leaching with nitric acid, in the presence of flocculants, is influenced by several factors, including reagent dosage, pH of the medium, liquid-to-solid ratio, particle size distribution, and the chemical and mineralogical composition of the original raw material.

**Table 8. Effect of flocculant consumption on the clarification rate of autoclave pulp**

| Measurement time, min. | Pulp clarification degree, % | | | |
|---|---|---|---|---|
| | Without additives | with an additive | | |
| | | K – 9 | K – 4 | K – 6 |
| The flocculant consumption rate is 38.0 g/m$^3$ | | | | |
| 5 | 4 | 40.40 | 37.60 | 12.20 |
| 10 | 8 | 57.50 | 51.40 | 26.50 |
| 15 | 12 | 61.50 | 60.30 | 39.90 |
| 20 | 14 | 61.70 | 64.90 | 50.00 |
| 25 | 18 | 64.00 | 66.20 | 58.10 |
| 30 | 21 | 64.00 | 66.20 | 62.10 |
| The flocculant consumption rate is 63.0 g/m$^3$ | | | | |
| 5 | 4 | 47.00 | 52.50 | 20.80 |
| 10 | 8 | 59.00 | 61.80 | 39.50 |
| 15 | 12 | 61.20 | 67.00 | 47.90 |
| 20 | 14 | 63.20 | 69.00 | 54.20 |
| 25 | 18 | 64.10 | 69.00 | 59.30 |
| 30 | 21 | 64.10 | 69.60 | 62.40 |
| The flocculant consumption rate is 88.0 g/m$^3$ | | | | |
| 5 | 4 | 37.20 | 56.50 | 45.10 |
| 10 | 8 | 55.00 | 63.40 | 60.80 |
| 15 | 12 | 57.00 | 66.30 | 64.70 |
| 20 | 14 | 64.70 | 68.30 | 67.60 |
| 25 | 18 | 66.60 | 68.30 | 68.60 |
| 30 | 22 | 66.60 | 71.80 | 70.60 |
| The flocculant consumption rate is 113.0 g/m$^3$ | | | | |
| 5 | 4 | 49.00 | 65.60 | 44.20 |
| 10 | 8 | 66.00 | 70.40 | 61.50 |
| 15 | 12 | 68.00 | 72.20 | 66.30 |
| 20 | 14 | 70.00 | 73.30 | 69.20 |
| 25 | 18 | 71.70 | 73.30 | 71.10 |
| 30 | 22 | 71.70 | 74.30 | 72.10 |

**Raw material: Vorukhsky bauxite; temperature: -70°C.*

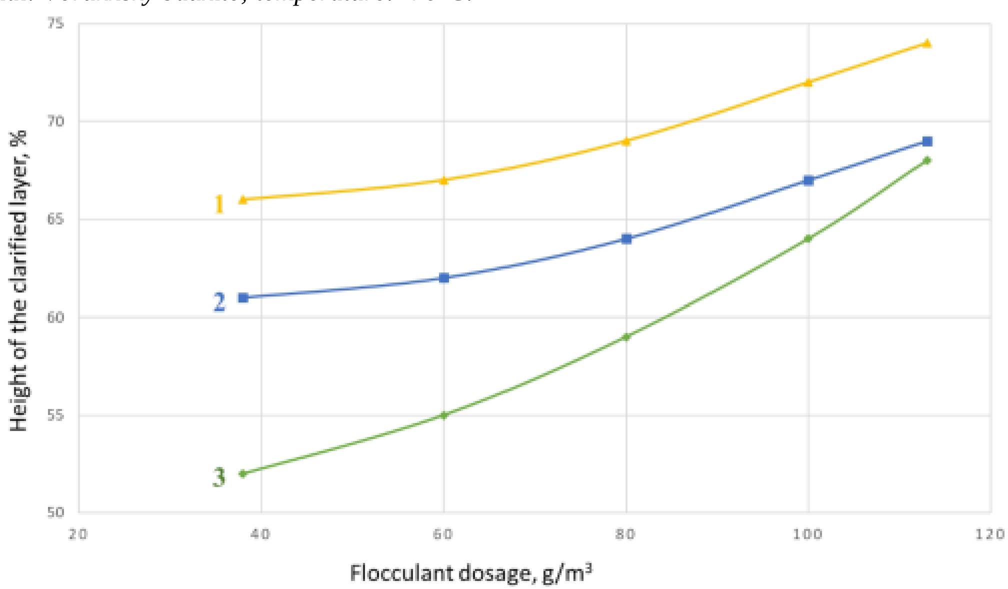


**Fig. 3. Relationship between the height of the clarified layer of autoclave pulp and the flocculant consumption rate (clarification time - 20 min) 1 - K - 4; 2 - K - 9; 3 - K - 6.**

**Results**

The experimental investigations revealed that the application of synthetic polyelectrolyte flocculants significantly improved the efficiency of solid–liquid separation in nitric acid-leached pulps derived from kaolin and substandard bauxites. In the absence of additives, the sedimentation process was notably slow, resulting in poor clarification even after extended settling times. The introduction of flocculants such as K-4, K-6, K-9, and AKS accelerated sedimentation by a factor of 3.5–4.0, with up to 65% clarification achieved within the first 20 minutes of settling. Among the tested reagents, K-4 consistently demonstrated the highest performance, producing a clarified supernatant of high transparency and stable sediment compaction.

Filtration studies further confirmed the positive influence of flocculant addition. Compared to untreated pulps, the presence of K-4 and AKS increased the specific output of the solid phase by more than 2.5 times, while simultaneously reducing cake moisture and enhancing solution flow rates. Laboratory experiments using nutsch filtration under vacuum conditions showed that filtration productivity improved from an average of 130–150 $kg/m^2 \cdot h$ without additives to 340–405 $kg/m^2 \cdot h$ with K-4. Pilot-scale experiments confirmed these results, although absolute values were slightly lower due to operational constraints, demonstrating increases in filtration rate by approximately 2.2–2.3 times relative to the untreated pulp.

The residual effect of adsorbed flocculants also played an important role during the countercurrent washing process of filter cakes. Analysis of wash waters indicated that the presence of residual K-4 and K-6 improved filtration rates across all three washing stages, while maintaining the recovery of valuable components such as $Al_2O_3$ and minimizing $Fe_2O_3$ losses. These findings suggest that flocculants not only enhance primary separation but also contribute to greater efficiency in subsequent washing operations.

Comparative experiments with bauxite samples from different deposits confirmed that mineralogical and chemical compositions strongly affect flocculant performance. Vorukhsky bauxite, characterized by its relatively high $SiO_2$ content (41.8%), displayed the most pronounced improvements when treated with K-4 and K-9, while Turgaysky and Tikhvinsky bauxites showed more moderate enhancements. Particle size distribution was also identified as a key factor, with finely dispersed fractions responding more effectively to flocculant treatment.

**Conclusion**

This study has demonstrated that the use of synthetic polyelectrolyte flocculants represents an effective approach to overcoming the major challenges of solid–liquid separation in nitric acid-leached pulps obtained from kaolin and substandard bauxites. Experimental results confirmed that the introduction of flocculants, particularly K-4, significantly accelerates sedimentation and improves clarification efficiency, achieving up to a four-fold increase in settling rate compared with untreated pulps. Filtration studies further established that flocculant addition enhances productivity by more than 2.5 times while reducing cake moisture and increasing solution flow rates.

The residual adsorption of flocculants on the solid phase was also found to improve countercurrent washing performance, reducing the loss of valuable components and maintaining the quality of the recovered alumina. Comparative tests on bauxite samples from different deposits indicated that the effectiveness of flocculants is strongly influenced by mineralogical and chemical characteristics, with high-silica materials showing the greatest improvement. Overall, the findings highlight the importance of flocculant selection and dosage optimization in nitric acid-based hydrometallurgical systems. By improving the efficiency of sedimentation, clarification, and filtration, synthetic flocculants contribute to more sustainable and economically feasible processing of low-grade alumina raw materials. These outcomes provide a practical basis for industrial implementation and suggest promising directions for further research on advanced polymer systems with enhanced environmental compatibility.

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