

Investigation of feldspar flocculation characteristics using anionic, cationic and non-ionic flocculants in single and two-step flocculation systems

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This study investigates the flocculation behaviour of feldspar suspension using a variety of anionic, cationic, and non-ionic flocculants (A.336, A.338, A.1011, A.5250, A.110, A.120, A.1858, C.573, N.333 and N.351) in single and two-step flocculation systems. Experiments have been performed at various dosages ranging from 0.2 to 12.5 mg/L. Flocculation performance is evaluated based on turbidity, settling rate, and efficiency. Of the flocculants tested, non-ionic flocculant N.351 demonstrated the most effective flocculation performance in terms of both turbidity reduction and flocculation efficiency. N.351 achieved a flocculation efficiency of 98.5% at a dosage of 0.8 mg/L, achieving the lowest turbidity value of 6.4 NTU (Nephelometric Turbidity Unit). Although N.351 demonstrated turbidity reduction and flocculation efficiency, the highest settling rates are observed in the two-step system employing the cationic-anionic flocculation combinations of C.573-A.338 and C.573-A.1011. These combinations achieved the highest settling rate of 2700 mm/min at dosages of 8.5 mg/L and 12.5 mg/L, respectively. This experimental study demonstrates that optimum flocculant dosages can vary significantly depending on the type of flocculant, the combination used, and the performance parameters. These results provide valuable information to optimize feldspar flocculation in industrial applications, which could improve process efficiency and product quality in feldspar using industries.

Keywords: Feldspar, Flocculation efficiency, Flocculation mechanism, Flocculation systems, Settling rate, Turbidity

Introduction

Feldspar is a versatile and widely used mineral in many industries including ceramics, glass, paint, paper, and plastics. In these industries, approximately 60% of feldspar is used in glass production, 35% in ceramics, and 5% in other applications. In glass production, feldspar acts as a flux, lowering the melting temperature of glass and increasing its chemical resistance and thermal durability. In the ceramics industry, it reduces the vitrification temperature, facilitates the formation of a glassy phase and increases the product's mechanical strength. In the paint industry, it is used as a filler to provide paint with functional properties such as gloss, viscosity control, and uniform spreading. In the paper industry, it is used as an additive to improve physical properties such as surface smoothness and opacity^{1,2,3}.

Flocculation is a widely used method in many industrial fields such as mining and ore processing, wastewater treatment, the paper industry, and biotechnology. As a fundamental step in mineral processing, flocculation plays an important role in solid-liquid separation and dewatering processes,

particularly in fine-grained and colloidal systems. Compared with alternative separation methods, flocculation offers significant advantages, such as ease of application, low energy requirements, cost-effectiveness, and environmental sustainability^{4,5}.

Flocculation is a complex process involving several mechanisms that significantly affect the floc structure. Electrostatic repulsion reduction and van der Waals attraction play a role in particle destabilization, while bridge flocculation and patch neutralization are considered to be the dominant mechanisms. Bridge flocculation (Fig. 1), associated with high molecular weight polymers, occurs when polymer tails and loops form links between particles. This bridging can occur in two ways: a single polymer chain connecting two particles or association of separately adsorbed polymer molecules on different particles. On the other hand, patch neutralization produces flocs similar to those formed by inorganic coagulants^{6,7}.

Anionic, cationic, and non-ionic flocculants are widely used in the flocculation process due to their distinct chemical properties and mechanisms. Anionic flocculants, which are usually polyacrylamide (PAM)

based, contain weakly acidic carboxylic acid groups that make their charge density dependent on the pH. They are most effective under neutral to alkaline pH conditions and are suitable for the flocculation positively charged particles. Cationic flocculants, on the other hand, typically contain quaternary ammonium groups that provide a permanent positive charge, independent of pH. These are known as strong electrolyte polymers. However, weak electrolyte polymers that acquire cationic characteristics in acidic environments are also available. Cationic flocculants are particularly effective at neutralizing negatively charged particles in acidic to neutral pH environments. Non-ionic flocculants, such as polyethylene oxide, are less sensitive to changes in pH and ionic strength, enabling them to be used across a wide range of conditions⁸⁻¹⁰.

The efficiency of flocculation depends on chemical parameters such as the type and dosage of flocculant used, its molecular weight and the type and concentration of ions present in the medium. Operating conditions, such as the pH of suspension, solid ratio, particle size, stirring time and speed,

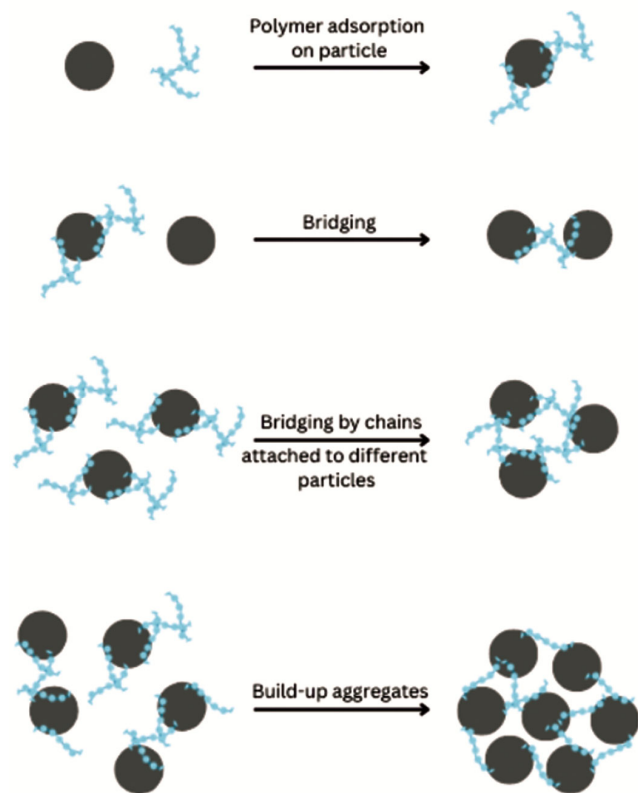


Fig. 1 — The effect of polymer on particle adsorption and subsequent steps leading to flocculation

temperature, and settling rate, also play an important role¹⁰⁻¹².

In recent years, there has been increasing interest in multi-component flocculants, particularly in two-step flocculation systems. These systems have been shown to have advantages over single flocculant applications and potentially offer improved particle agglomeration and settling efficiencies^{7,9,11,13-16}. The synergistic effects of combining different flocculants can lead to more robust floc formation, faster settling rates, and improved water clarity^{14,15}.

In two-step flocculation systems, the sequence of flocculant addition can significantly influence the flocculation process and particle interactions. If a cationic flocculant is added first, it neutralizes the negatively charged surfaces of the particles, thereby reducing their repulsive forces. After charge neutralization, the addition of an anionic flocculant causes the neutralized particles to form larger, more stable flocs via bridging mechanism^{8,16,17}. When a non-ionic flocculant is first added, it forms a loose particle network by bridging between particles, primarily through hydrogen bonding, van der Waals forces or other weak interactions. When an anionic flocculant is added after a non-ionic flocculant, it interacts with the positively charged or weakly neutralized regions of the particle surface or aggregates, thereby increasing the bridging and stability of the flocs. This combination typically results in denser flocs with higher settling rates.

The efficiency of the flocculation is usually evaluated based on parameters such as turbidity, settling rate, and flocculation efficiency. Turbidity, which is a measure of water clarity, is important for determining the effectiveness of solid-liquid separation. The settling rate indicates the speed at which flocs descend in the suspension, which is essential for process efficiency. Flocculation efficiency provides an overall measure of the process's success in aggregating and removing suspended particles¹⁸.

Optimization of flocculant type and dosage is critical to achieving high flocculation efficiency, as these parameters directly affect floc size, settling rate and selective separation of desired minerals. Although flocculation processes have been widely studied for various mineral systems, the flocculation behaviour of feldspar, particularly in relation to different flocculant types and their combinations, remain insufficiently investigated. This study investigated the flocculation properties of feldspar using different types of flocculant

(anionic, cationic, and non-ionic) in single and two-step flocculation systems, using key parameters such as turbidity, settling rate, and flocculation efficiency.

Experimental Section

Materials

This study used a feldspar sample from Muğla/Yatağan in Türkiye. The elemental composition of the sample, as determined by X-ray fluorescence (XRF) analysis, is given in Table 1. The feldspar sample was dry-ground to below 90 µm using a ceramic ball mill. Wet sieving analysis indicated that 95.2% of the ground feldspar passed through a 75 µm sieve.

Ten different polyacrylamide-based flocculants were used in this study, including the non-ionic (Magnafloc 333 and Magnafloc 351), the anionic (Magnafloc 336, Magnafloc 338, Magnafloc 1011, Magnafloc 5250, Cyanamid 110, Cyanamid 120, and SNF 1858 U), and the cationic (Cyanamid 573) flocculants. The flocculants used in the experiments were procured from BASF SE, SNF and Cyanamid Company. All flocculants used were of analytical grade. Their properties are given in Table 2. Stock solutions of each flocculant were prepared at a concentration of 0.5% (w/w) using distilled water. Working solutions at a concentration of 0.05% (w/w) were then prepared from these stock solutions for use in the experiments.

Methods

In the experiments, the flocculants were tested individually. Successful flocculation was observed only with the non-ionic flocculants N.333 and N.351. Therefore, these non-ionic flocculants were used both

individually and in combination with anionic flocculants (A.336, A.338, A.1011, and A.5250). Additionally, cationic flocculant C.573 was combined with various anionic flocculants (A.336, A.338, A.1011, A.5250, A.110, A.120, and A.1858) to form two-step flocculation systems.

The experiments were carried out at the natural pH (6.1) of the suspension. The flocculant dosages ranged from 0.2 to 12.5 mg/L. For each test, a suspension was prepared by adding 1 g of feldspar to 600 mL of distilled water in a 750 mL glass beaker. The suspension was stirred at 500 rpm for five minutes to ensure homogeneity. In the single flocculation tests, the desired dosage of flocculant was added to the suspension and stirred at 500 rpm for one minute. In two-step flocculation tests, the first flocculant was added and stirred for one minute. Then, the second flocculant was added to the suspension at the desired dosage and stirred at the same speed for an additional one minute. Following flocculant addition, the stirring speed was reduced to 200 rpm in both single and two-step flocculation tests and the suspension was stirred for two minutes to promote floc formation. After this, the system was stopped and the flocs were allowed to settle for two minutes.

After settling, a 10 mL supernatant sample was carefully collected 4 cm below the air-liquid interface. Turbidity was measured using a MicroTPI turbidimeter, and the results were expressed in Nephelometric Turbidity Units (NTU).

The settling rate of the flocs was determined by observing changes in the height of the suspension in the beaker over time. The 600 mL and 300 mL marks on the beaker were used as reference points for this measurement.

Table 1 — Elemental analysis of the feldspar sample

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	Na ₂ O	K ₂ O	CaO	MgO	P ₂ O ₅
Wt. (%)	70.07	17.34	0.12	0.34	10.64	0.40	0.61	<0.01	0.24

Table 2 — Properties of the flocculants used in the study

Flocculant Name	Flocculant Code	Flocculant Type	Physical Form	Molecular Weight
Magnafloc 333	N.333	Non-ionic	Powder – off white	Medium
Magnafloc 351	N.351	Non-ionic	Powder – off white	Medium
Magnafloc 336	A.336	Anionic	Powder – off white	High
Magnafloc 338	A.338	Anionic	Powder – off white	High
Magnafloc 1011	A.1011	Anionic	Powder – of white	High
Magnafloc 5250	A.5250	Anionic	Powder – off white	High
Cyanamid 110	A.110	Anionic	Powder – off white	Low
Cyanamid 120	A.120	Anionic	Powder – off white	Medium
Cyanamid 573	C.573	Cationic	Liquid – yellow	Low
SNF 1858 U	A.1858	Anionic	Powder – off white	High

The flocculation efficiency was calculated using the following equation¹⁹:

$$\text{Flocculation efficiency (\%)} = \left[\frac{(T_0 - T_f)}{T_0} \right] * 100 \dots (1)$$

Where T_0 is the initial suspension turbidity and T_f is the final turbidity after flocculation.

Results and Discussion

Effect of Flocculant Type and Dosage on Turbidity

Turbidity is a critical parameter for assessing the effectiveness of flocculation, as it directly indicates the clarity of the supernatant after solid-liquid separation. In this study, the initial turbidity of the feldspar suspension was 430.1 NTU, indicating a high concentration of suspended particles.

Fig. 2a shows the effect of non-ionic flocculant N.333 and its combinations with anionic flocculants (A.336, A.338, A.1011, and A.5250) on turbidity.

When used alone and in combination with anionic flocculants, N.333 shows a significant increase in turbidity beyond a flocculant dosage of 1.6 mg/L. This is due to steric stabilization and destabilization effects that occur when excess polymer saturates particle surfaces. These effects prevent interparticle bridging and cause previously formed flocs to disintegrate, resulting in continuous increase in turbidity⁸.

Fig. 2b shows that the turbidity of the second non-ionic flocculant N.351 and its combinations with anionic flocculants (A.336, A.338, A.1011, and A.5250) exhibit similar trends to those of the non-ionic flocculant N.333. Turbidity values for both the N.351 flocculant and its combinations reach a minimum at a dosage of 1.6 mg/L, after which they increase sharply. Fig. 2a and Fig. 2b demonstrate that dual systems consistently exhibit higher turbidity values than single flocculant systems. This is interpreted as the addition of anionic flocculants potentially inhibiting the

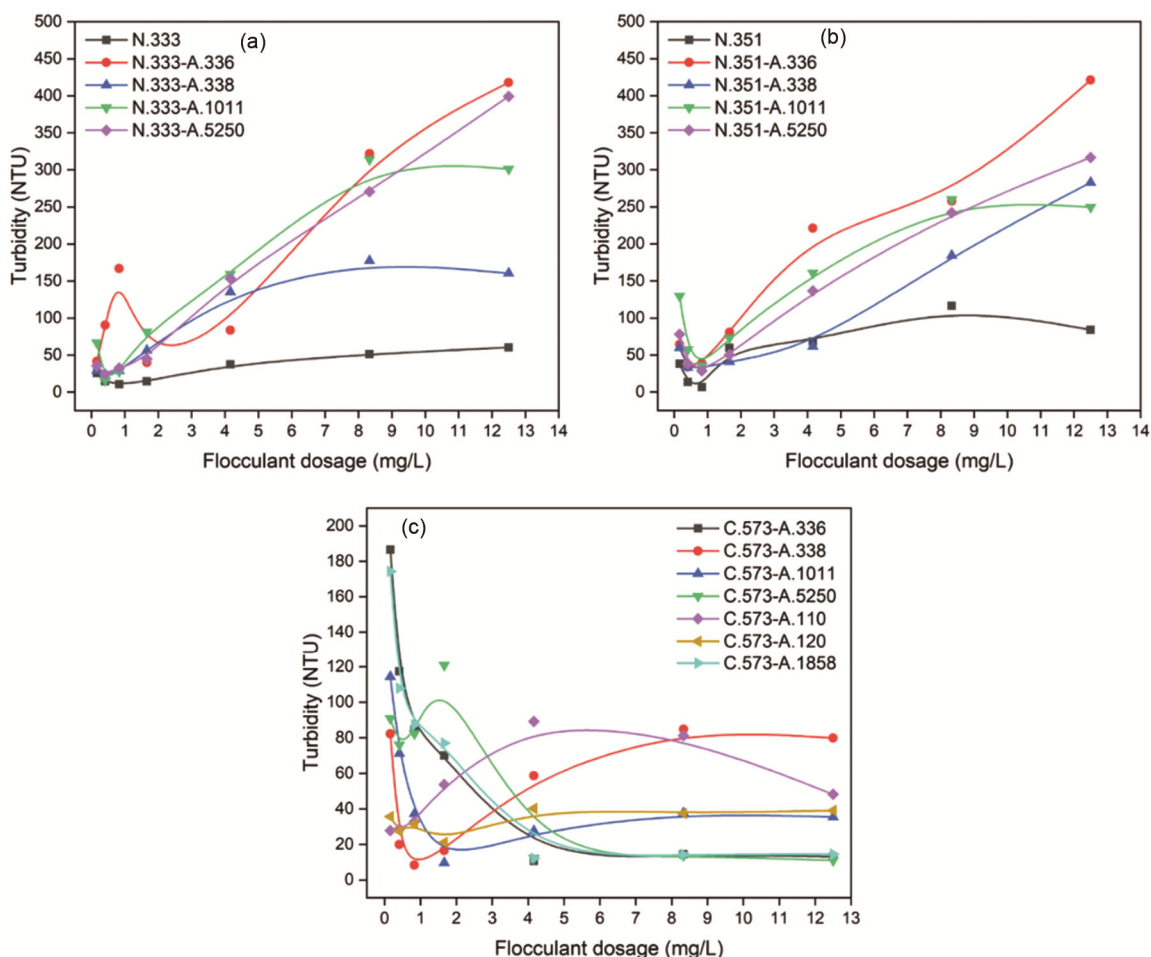


Fig. 2 — Effect of flocculant type and dosage on feldspar suspension turbidity: (a) N.333 and its combinations with anionic flocculants, (b) N.351 and its combinations with anionic flocculants and (c) C.573 in combined with anionic flocculants

optimal bridging mechanism formed by non-ionic flocculants alone in these systems.

Fig. 2c shows the turbidity results for cationic flocculant C.573 in combination with anionic flocculants (A.336, A.338, A.1011, A.5250, A.110, A.120, and A.1858). These combinations showed a different pattern from that observed with of non-ionic flocculants. An initial increase in turbidity was observed at low dosages, followed by a sharp decline at a dosage of 1.6 mg/L. This initial increase in turbidity could be due to the charge neutralization effects. As the C.573 neutralizes the negative surface charge of the feldspar particles, it may cause destabilization before effective flocculation occurs. Above 4.0 mg/L dosage, turbidity remains relatively constant for most combinations, suggesting that a saturation point has been reached where the addition of a second flocculant does not significantly improve turbidity reduction. However, the C.573-A.110 and C.573-A.338 combinations showed slight increases in turbidity after 4 mg/L dosage, possibly due to

overdosing effects. Overall, the results emphasize the importance of dosage in the performance of C.573 with anionic flocculants.

Effect of flocculant type and dosage on settling rate

The settling rate directly affects the efficiency of the solid-liquid separation and the overall processing time. A higher settling rate indicates more effective flocculation, resulting in the formation of larger, denser flocs that settle more quickly.

Fig 3a shows the settling rates for non-ionic flocculant N.333 and its combinations with anionic flocculants (A.336, A.338, A.1011, and A.5250). The settling rates were initially high up to a dosage of 1.6 mg/L; after which a decrease or stabilization was observed at higher dosages. This behaviour can be explained by polymer bridging mechanism. At lower dosages, the flocculant molecules effectively bridge the particles, creating large flocs that settle rapidly. However, increasing the dosage beyond the optimum

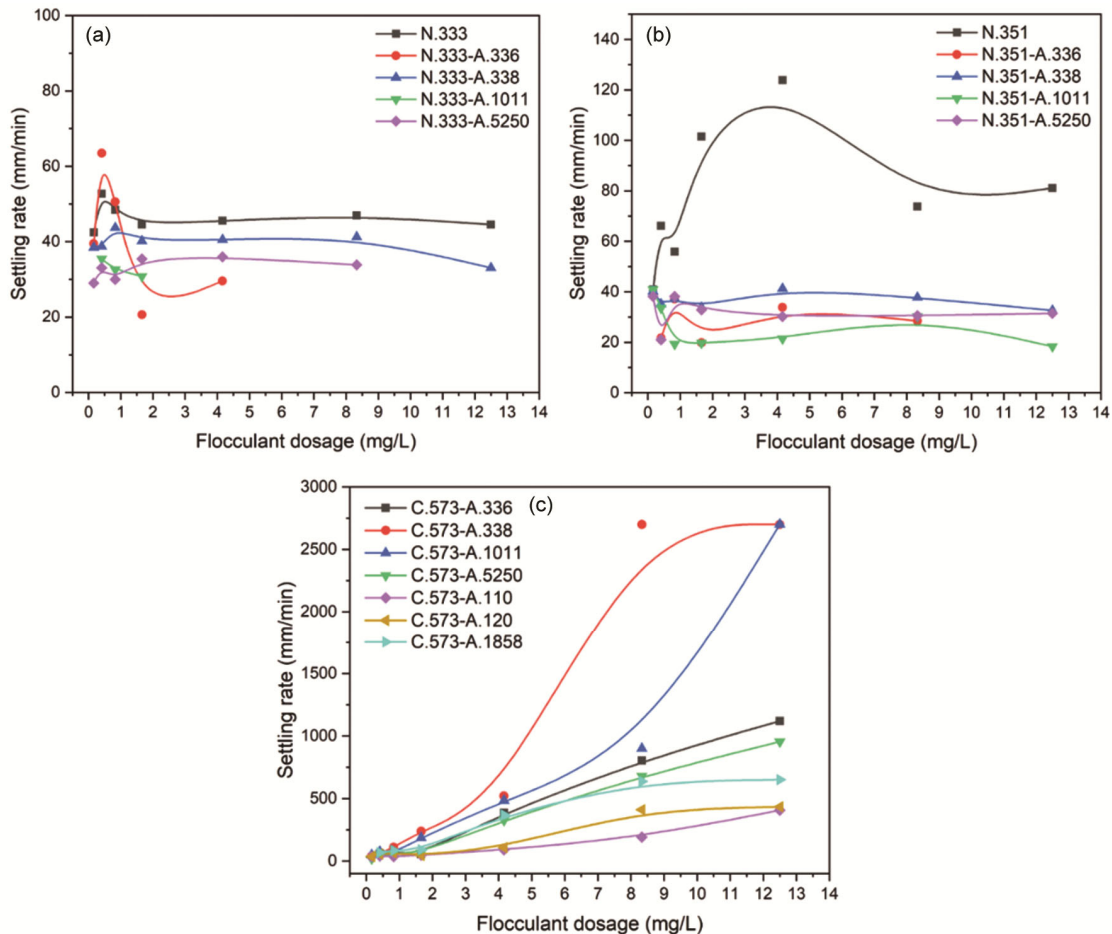


Fig. 3 — Effect of flocculant type and dosage on feldspar suspension settling rate: (a) N.333 and its combinations with anionic flocculants, (b) N.351 and its combinations with anionic flocculants and (c) C.573 in combined with anionic flocculants

can cause in steric stabilization or overcrowding of the polymer chains on particle surfaces. This creates smaller, less dense flocs that settle more slowly, thereby reducing the settling rate²⁰. Of all combinations, the N.333-A.336 combination achieved the highest settling rate of 63.5 mm/min at a dosage of 0.4 mg/L. This synergistic effect between non-ionic and anionic flocculants indicates that larger, more settleable flocs are formed at lower dosages compared to single flocculant system²¹. However, beyond certain dosages, settling rates became unmeasurable due to increased turbidity: above a dosage of 4.0 mg/L for the N.333-A.336 combination, above 1.6 mg/L dosage for the N.333-A.1011 combination and above a dosage of 8.5 mg/L for the N.333-A.5250 combination. These results highlight the importance of dosage optimization, suggesting a narrow optimum dosage range.

Fig. 3b shows the settling rates for non-ionic flocculant N.351 and its combinations with anionic flocculants (A.336, A.338, A.1011, and A.5250). Of all the flocculants tested, only N.351 produced a distinct bell-shaped curve when used on its own. It achieved a maximum settling rate of 123.8 mm/min at a dosage of 4.0 mg/L. This curve is characteristic of polymer flocculants and represents the transition from bridging flocculation to steric stabilization as dosage increases²². In contrast, the combinations of N.351 with anionic flocculants exhibited more consistent and stable settling rates with lower rates across the tested dosages. These flatter curves suggest that the addition of anionic flocculants alters the flocculation mechanism by introducing electrostatic interactions that compete with the bridging mechanism of the non-ionic flocculant N.351.

Fig. 3c shows the settling rates for combinations of the cationic flocculant C.573 with anionic flocculants (A.336, A.338, A.1011, A.5250, A.110, A.120, and A.1858). These combinations exhibited markedly different trends to those observed with non-ionic flocculants and their combinations. All cationic-anionic combinations demonstrate high settling rates, which increase with flocculant dosage, rising sharply above 4.0 mg/L. Among these, the C.573-A.338 and C.573-A.1011 combinations achieved the highest settling rates of 2700 mm/min at dosages of 8.5 mg/L and 12.5 mg/L, respectively. The enhanced settling rates observed for the cationic-anionic flocculant systems results from a combination of charge neutralization and polymer-bridging, achieved through the sequential addition of flocculants. At the natural pH of the suspension, feldspar

particles possess negatively charged surfaces. In the first step, the cationic flocculant C.573 partially neutralizes these surface charges, significantly reducing interparticle electrostatic repulsion and allowing closer particle approach. In the second step, the subsequent addition of an anionic flocculant (A.338 or A.1011) promotes effective polymer bridging between the charge-neutralized particles, resulting in formation of larger, denser and more compact flocs. These flocs exhibit higher effective mass and reduced hydrodynamic resistance, leading to markedly increased settling velocities. This combined mechanism provides a clear explanation for the significantly higher settling rates observed in two-step flocculation systems²³.

Effect of flocculant type and dosage on flocculation efficiency

The flocculation efficiency is a comprehensive parameter that indicates the overall effectiveness of the flocculation process. It considers both the initial and final turbidity of the suspension, providing an insight into the percentage of particles removed through flocculation.

Fig. 4a shows the flocculation efficiency for non-ionic flocculant N.333 and its combinations with anionic flocculants (A.336, A.338, A.1011, and A.5250). The N.333 alone achieved the highest efficiency of 97.5% at a dosage of 0.8 mg/L. For the combinations, the initial efficiency values were similarly high at low dosages, but then decreased gradually. Most of the combinations experienced a rapid decline in efficiency at dosages beyond the optimum. This behaviour is consistent with the polymer-bridging flocculation mechanism, whereby polymer oversaturation on the particle surface can result in steric stabilization and decreased flocculation efficiency due to reduced bridging ability⁸. Notably, the N.333-A.338 combination showed better stability at higher dosages than the other combinations, indicating a potential synergistic effect or a wider effective dosage range. In contrast, the efficiency of combinations such as N.333-A.336 and N.333-A.1011 decreased sharply. This may be due to sensitivity to overdosing, which could lead to floc breakage or destabilization.

Fig. 4b presents the flocculation efficiency for non-ionic flocculant N.351 and its combinations with anionic flocculants (A.336, A.338, A.1011, and A.5250). The efficiency curve of N.351 and its combinations follows a similar trend to that of N.333 and its combinations shown in Fig. 4a; a rapid increase in efficiency up to a dosage of 0.8 mg/L, at which

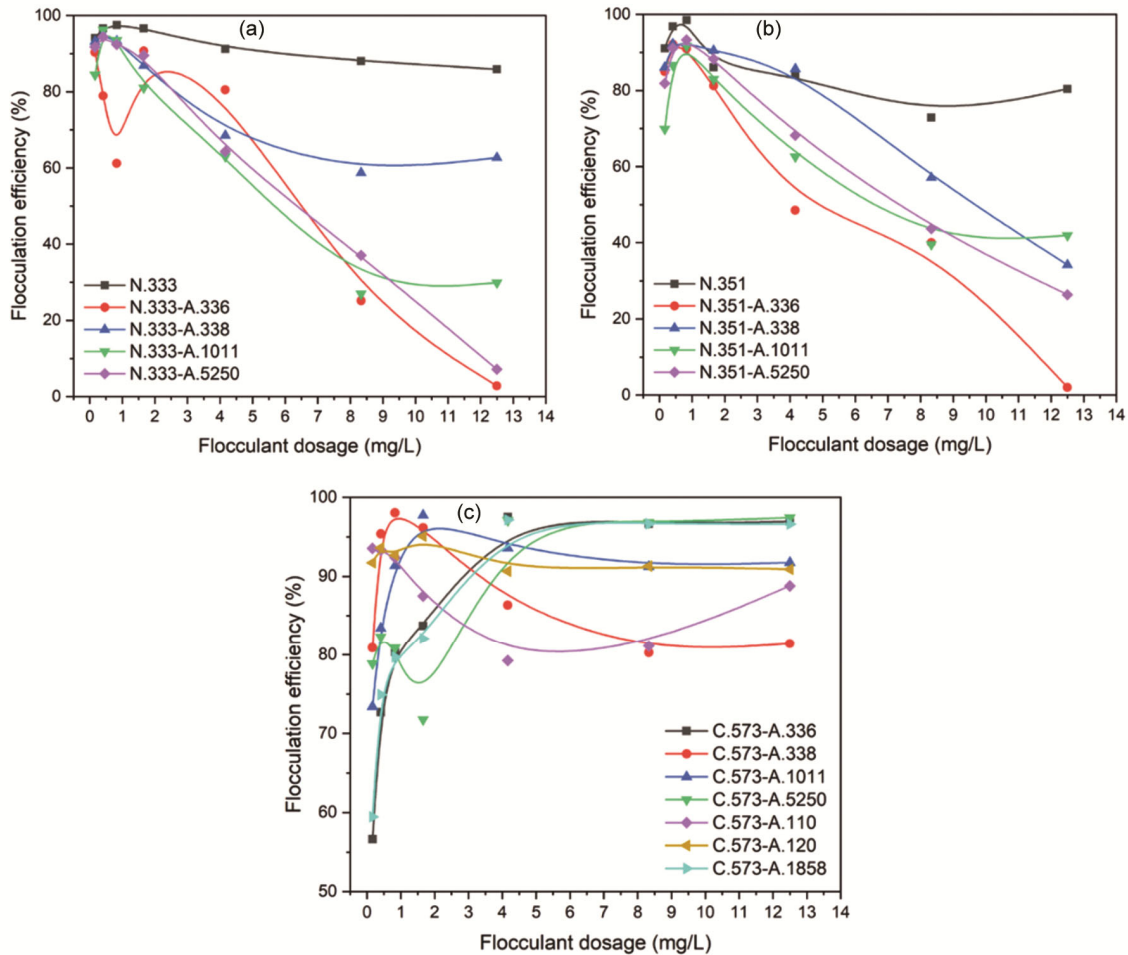


Fig. 4 — Effect of flocculant type and dosage on feldspar flocculation efficiency: (a) N.333 and its combinations with anionic flocculants, (b) N.351 and its combinations with anionic flocculants and (c) C.573 in combination with anionic flocculants

maximum efficiency is achieved, followed by a gradual decrease with further dosage increase. At this optimum dosage, N.351 reached efficiency of 98.5%, slightly higher than N.333's 97.5%. Despite this, N.333 displays a more stable and consistent efficiency across the entire dosage range, whereas N.351 shows a steeper decline beyond the optimum dosage. The superior overall performance of N.333 may be attributed to its higher molecular weight and greater polymer chain flexibility, allowing for more effective particle bridging and the formation of strong, stable flocs²⁰.

Fig. 4c shows the flocculation efficiency of cationic flocculant C.573 in combination with anionic flocculants (A.336, A.338, A.1011, A.5250, A.110, A.120, and A.1858). Among the tested combinations, C.573-A.338 combination exhibited the highest flocculation efficiency of 98.1% at a low dosage of 0.8 mg/L. The efficiency curves for these combinations showed a sharp rise in flocculation efficiency at lower

dosage compared with non-ionic flocculants, followed by a stable plateau or mild fluctuation at higher dosages. This behaviour can be attributed to synergistic effects between the charge neutralization provided by the cationic flocculant and polymer bridging facilitated by the anionic flocculants¹⁷. These results demonstrate the effectiveness of two-step flocculation systems, indicating that dual-flocculant system can match or outperform the efficiency single flocculants.

Conclusion

This comprehensive study investigated the flocculation characteristics of feldspar, examining different types of flocculants and their combinations. Among the tested flocculants, the superior turbidity reduction and flocculation efficiency achieved with the non-ionic flocculant N.351 can be attributed mainly to its molecular structure and charge-independent adsorption mechanism. As a non-ionic polyacrylamide,

N.351 interacts with negatively charged feldspar surfaces through hydrogen bonding and van der Waals forces rather than electrostatic attraction, allowing uniform adsorption across the particle surface. Additionally, the flexible polymer chains of N.351 promote effective interparticle bridging via extended loops and tails, resulting in the formation of compact and stable flocs that efficiently capture fine particles. Consequently, N.351 exhibited the lowest turbidity value (6.4 NTU) and the highest flocculation efficiency (98.5%) at an optimum dosage of 0.8 mg/L, demonstrating its strong potential for efficient feldspar flocculation. Combinations of cationic C.573 with anionic flocculants showed excellent performance in improving the settling rates in two-step flocculation. Notably, the C.573-A.338 and C.573-A.1011 combinations achieved the highest settling rates of 2700 mm/min at dosages of 8.5 mg/L and 12.5 mg/L, respectively. This demonstrates the potential for synergistic effects in two-step flocculation systems by combining charge neutralization and bridging mechanisms. The study demonstrated that optimum dosages vary considerably depending on the type of flocculant used, the combinations applied, and the specific performance parameters evaluated. This highlights the critical importance of precise dosage optimization in industrial applications, in order to strike an effective balance between turbidity reduction, settling rate, and flocculation efficiency. The results provide a robust basis for optimizing solid-liquid separation, offering the potential to improve product quality, enhance process performance, and reduce environmental impact in feldspar-using industries. In addition to technical performance, optimized flocculation strategies may also offer economic and environmental benefits by reducing reagent consumption, improving water recycling, and lowering energy demand in downstream dewatering processes.

Conflicts of Interest

The authors declare no conflicts of interest.

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