

ECO-FRIENDLY REAGENTS UPSCALING AND OPTIMIZATION IN FROTH FLOTATION: CASE STUDIES ON HF AND NASH SUBSTITUTION.

Borhane Ben Said¹⁾, Martin Rudolph¹⁾ and Lucas Pereira¹⁾

¹ Helmholtz Institute Freiberg for Resource Technology,
Helmholtz-Zentrum Dresden-Rossendorf, Chemnitz Str. 40, 09599 Freiberg, Germany
(*Corresponding author: b.ben-said@hzdr.de)

ABSTRACT

This study validates a developed workflow for the upscaling and optimization of flotation reagent systems in froth flotation, recently demonstrated on low-grade European scheelite ore with two further industrial case studies. The workflow integrates the Design of Experiments (DoE) at batch scale and numerical optimization at continuous operation (mini pilot, pilot, plant scale) to identify optimal reagent conditions and ensure reliable scale-up into industrial applications by continuously refining the parameter space. In the first case, sodium fluoride (NaF) is investigated as a replacement for hydrofluoric acid (HF) as an activator in feldspar flotation from quartz. The second case addressed substituting sodium hydrosulfide (NaSH) with a lignin-based biopolymer to separate chalcopryrite and molybdenite selectively. The workflow validates its industrial relevance and potentials as a fast and systematic approach for introducing safer and more environmentally friendly reagents into flotation practice.

KEYWORDS

Reagent optimization, Design of Experiments (DoE), Numerical optimization, Upscaling, Feldspar flotation, Chalcopryrite–molybdenite separation.

1 Introduction

The development and implementation of new reagent systems remain among the most critical yet challenging aspects of froth flotation. Despite the wide range of available collectors, frothers, and depressants, industrial operations still rely heavily on empirical reagent selection and incremental adjustments based on operator experience. This practice often results in inconsistent selectivity, excessive reagent consumption, and limited adaptability to variations in feed mineralogy or water chemistry. At the same time, increasingly stringent environmental regulations and societal expectations for sustainable mineral processing are driving the replacement of hazardous reagents with safer, biodegradable alternatives.

To address these challenges, the present work investigates two industrial case studies that exemplify the transition from conventional to environmentally responsible flotation reagents in both oxide and sulfide systems. The first case study focuses on feldspar flotation from quartz-bearing pegmatitic ores, where hydrofluoric acid (HF) - traditionally used as a surface activator for feldspar [1] - is targeted for substitution by sodium fluoride (NaF). Although HF provides excellent selectivity by modifying silicate surfaces, it is highly corrosive, toxic, and difficult to handle, posing severe risks to workers and the environment [2]. Its volatility and persistence also create significant challenges for wastewater treatment and process safety.

The second case study examines the selective separation of chalcopryrite (CuFeS₂) and molybdenite (MoS₂), where sodium hydrosulfide (NaSH) is commonly employed as a depressant for copper minerals [3]. While effective, NaSH is strongly alkaline and releases hydrogen sulfide gas (H₂S), which is toxic, malodorous, and hazardous under industrial operating conditions [4]. In this study, a lignin-

based biopolymer was evaluated as a green alternative to NaSH, aiming to achieve comparable selectivity while eliminating the associated safety and environmental concerns.

Together, these case studies highlight practical strategies for introducing safer, sustainable reagent chemistries into industrial flotation, demonstrating that environmental compatibility and process efficiency can be achieved simultaneously through rational reagent substitution and optimization.

2 Material and methods

2.1 Reagents

In the feldspar–quartz flotation system, sodium fluoride (NaF, Merck/Sigma-Aldrich, Germany) was used as a feldspar activator. A primary aliphatic amine collector supplied by Clariant AG (Switzerland) was employed to render the activated feldspar surfaces hydrophobic, while 4-methyl-2-pentanol (MIBC, Alfa Aesar, Germany) served as a frother to enhance froth stability. Sulfuric acid (H₂SO₄, Merck/Sigma-Aldrich, Germany) and sodium hydroxide (NaOH, Carl Roth GmbH, Germany) were used as pH regulators.

In the Cu–Mo flotation system, calcium oxide (CaO, Merck/Sigma-Aldrich, Germany) was used as a pH regulator. Lignin-based biopolymer depressants supplied by Borregaard AS (Norway) were tested. Ekofol (Mining and Water Solutions GmbH, Germany) was used as a molybdenite collector, while potassium amyl xanthate (PAX) and sodium isobutyl xanthate (SIBX), both supplied by AECI Mining Chemicals (South Africa), were employed as chalcopyrite collectors.

2.2 Design of experiments

Design of Experiments (DoE) is a structured, statistical approach used to systematically investigate the influence of multiple process variables on one or more measured responses. Rather than varying one factor at a time, DoE enables simultaneous variation of several factors, allowing interactions between variables to be detected and quantified. This approach provides a comprehensive understanding of the system under study while significantly reducing the number of experimental runs required compared to conventional trial-and-error methods [5]. The different types of DOE allow to investigate and optimize the process under study with an efficient and methodical way of working.

The fundamental objective of DoE is to establish statistically validated relationships between input parameters (factors) and process outputs (responses). These relationships can then be expressed as mathematical models—typically regression equations—that describe how the process behaves within the studied experimental region. By analyzing these models, it becomes possible to identify significant main effects, two-way or higher-order interactions, and quadratic (curvature) effects, which together define the response surface of the process.

DoE is particularly valuable in flotation process development, where numerous interacting variables (e.g., reagent dosage, pH, pulp density, air flow, and frother type) influence metallurgical performance indicators such as recovery, grade, or selectivity. Through DoE, these factors can be optimized efficiently to maximize the desired response while maintaining robustness against natural process variability.

2.3 Micro and batch flotation experiments

2.3.1 Sodium Fluoride as a Substitute for Hydrofluoric Acid in Feldspar Flotation

The feldspar–quartz ore used in this study contains approximately 65 wt% feldspar and 35 wt% quartz. The ore sample exhibited a d_{80} of about 190 μm , with approximately 70 % of particles larger than 100 μm , reflecting its coarse-grained character.

Aliquots of 250 g were prepared using a Retsch PT100 rotary sample divider and used as feed material for each flotation test. Each batch test was carried out at a 25 wt% solid content in a Magotteaux® bottom-driven flotation cell. The impeller speed was kept constant at 700 rpm, and the air flow rate was set to 5 L/min to ensure stable froth formation.

Prior to each flotation test, the pulp pH was adjusted using H_2SO_4 or NaOH , and the slurry was conditioned for 5 minutes to establish the target pH. Sodium fluoride (NaF) was then added as the feldspar activator and conditioned for 3 minutes. Following activation, the amine collector was introduced and conditioned for 3 minutes in the rougher stage and in the scavenger stage. In selected scenarios, methyl isobutyl carbinol (MIBC) was added as frother at 50 g/t.

Flotation was performed with froth scraping every five seconds, and three successive concentrates were collected from the rougher stage at cumulative flotation intervals of 30 s, 60 s, and 90 s, followed by a scavenger stage collected after an additional 120 s of flotation. The flotation procedure is illustrated in Figure 1, and the tested parameters along with their respective levels are summarized in Table 1. After each test, the flotation products were filtered and dried overnight at 50–60 °C, and the dry weights were used to calculate mass recoveries. The chemical and mineralogical compositions of the concentrates and tailings were determined using a portable X-ray fluorescence spectrometer (pXRF) SciAps X-555 to quantify the distribution of feldspar and quartz across the flotation products.

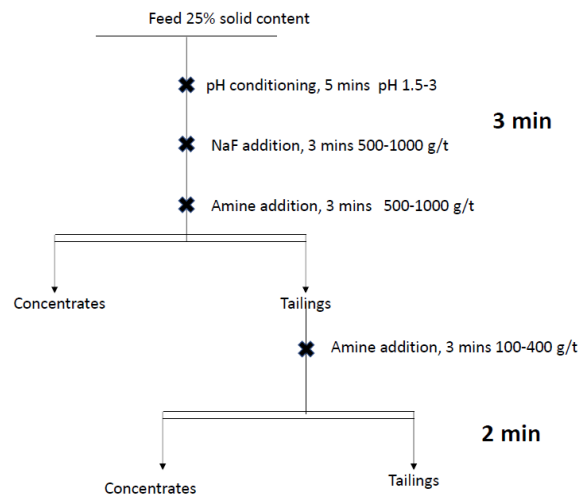


Figure 1 Schematic representation of the batch flotation procedure for feldspar–quartz separation

Table 1 Levels of variables investigated (2^3 full factorial design).

Parameter	Unit	Factor level	
		-	+
pH	-	1.5	3

dodecylamine dosage	g/t	500	1000
NaF dosage	g/t	500	1000

Once the optimal parameter ranges were identified, a series of targeted flotation scenarios was developed to examine the effect of reagent addition sequence, multiple dosing, and extended conditioning on flotation selectivity. These scenarios were systematically tested at batch scale under controlled conditions, as summarized in Table 2.

Table 2 Overview of the batch flotation scenarios conducted for feldspar–quartz separation

Scenario	Procedure
Scenario 2	NaF added also in scavenger stage
Scenario 3	pH 2.4, NaF 1000 g/t
Scenario 4:	same as 3 + frother (50 g/t)
Scenario 5	conditioning at pH 5 with NaOH
Scenario 6	dual conditioning pH 5 (NaOH) + pH 3 (H ₂ SO ₄)
Scenario 7	as 6 + NaF in Scavenger
Scenario 8	amine addition before NaF
Scenario 10a	amine addition before rougher 3 (500 g/t)
Scenario 10b	NaF addition before rougher 3
Scenario 11a	NaF conditioning → partial amine addition
Scenario 11b	amine conditioning → partial NaF addition
Scenario 12a	NaF conditioning + partial amine addition
Scenario 12b:	amine conditioning + partial NaF addition
Scenario 13	no NaF in rougher 1, only in rougher 2
Scenario 14a–c	longer conditioning times (NaF 6 min, amine 3–6 min)

2.3.2 Case Study II: Lignin-Based Biopolymer as a Substitute for Sodium Hydrosulfide in Cu–Mo Separation

2.3.2.1 Microflotation

Microflotation tests were conducted on chalcopyrite to evaluate the performance of various lignin-based biopolymer depressants and to assess the influence of pH on mineral floatability. Each test used 1 g of chalcopyrite suspended in 160 mL of 10^{−2} M KCl background solution, with the pH adjusted between 9 and 11 using CaO. The PAX concentration was fixed at 10^{−3} M, and the biopolymer depressant dosage was maintained at 200 mg/L. The depressants tested included F-100, F-200, F-250, F-500, DP-51756, DP-51757, and DP-51790, each representing distinct molecular architectures and functional group compositions.

The slurry was stirred at 400 rpm using a magnetic stirrer and conditioned for 5 minutes after pH adjustment, followed by 3 minutes of conditioning with the depressant and 3 minutes with the collector. The conditioned suspension was then transferred to a Hallimond tube, stirred for 2 minutes at 800 rpm, and floated for 2 minutes at an airflow rate of 20 cm³/min. The resulting concentrates and tailings were filtered, oven-dried at 50–60 °C overnight, and weighed to determine flotation recovery.

2.3.2.2 Batch flotation

Batch flotation tests were conducted to investigate the selective separation of molybdenite and chalcopyrite using lignin-based biopolymer depressants. The initial series of tests focused on

molybdenite flotation, employing Ekofol as the molybdenite collector and targeting the depression of chalcopyrite. The pulp pH was adjusted to 10.5 using CaO, and flotation was carried out in a Magotteaux® bottom-driven flotation cell at 25 wt% solids. Three successive concentrates were collected from the rougher stage at cumulative flotation intervals of 30 s, 60 s, and 90 s.

In a second series of batch tests, the strategy was reversed to float chalcopyrite while depressing molybdenite. In these tests, PAX or SIBX were used as the chalcopyrite collector, while the same lignin-based depressants were evaluated for their molybdenite depressing efficiency. The pH was again maintained at 10.5 using CaO, and the rougher stage followed the same cumulative collection intervals of 30 s, 60 s, and 90 s, followed by a scavenger stage collected after an additional 2 minutes of flotation. The flotation products were filtered, dried at 50–60 °C, and analyzed by a SciAps X-555 pXRF to determine Cu and Mo grades.

2.4 Pilot scale

Pilot-scale flotation tests were carried out using a Continuous Flotation Machine (CFM) integrated into the CPT Mini Pilot Plant from Eriez®. Each test was operated for approximately 2 to 2.5 hours to achieve steady-state conditions. During operation, the masses of the final concentrate and tailings were continuously monitored, and steady state was assumed to be reached when the time-dependent mass increase in both streams became nearly constant. At this point, samples were collected simultaneously from each stream over a one-minute interval and subsequently analyzed by a SciAps X-555 pXRF to determine the Cu and Mo grades. Depending on the ore processed and the circuit flowsheet configuration, the pH was measured and controlled at selected cells. The same reagent systems as used in the batch flotation experiments were applied at pilot scale.

3 Results

3.1 Sodium Fluoride as a Substitute for Hydrofluoric Acid in Feldspar Flotation

3.1.1 Batch flotation

Selected results of the experimental plan, carried out according to **Table 1**, are presented in Figure X. The Design of Experiments (DoE) for feldspar–quartz flotation revealed that the condition at pH 3, with 1000 g/t NaF and amine dosages of 2100 g/t in the rougher stage and 400 g/t in the scavenger stage, produced the most favorable separation line. Run 1 was therefore selected as the baseline condition for all subsequent scenario tests, in which the effects of conditioning time, reagent addition sequence, and multi-stage dosing were systematically investigated.

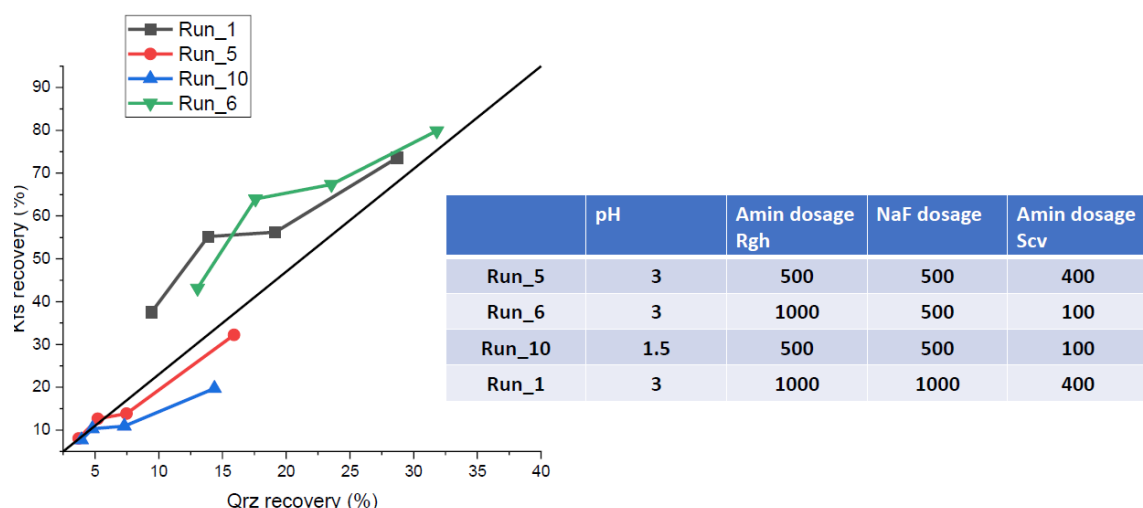
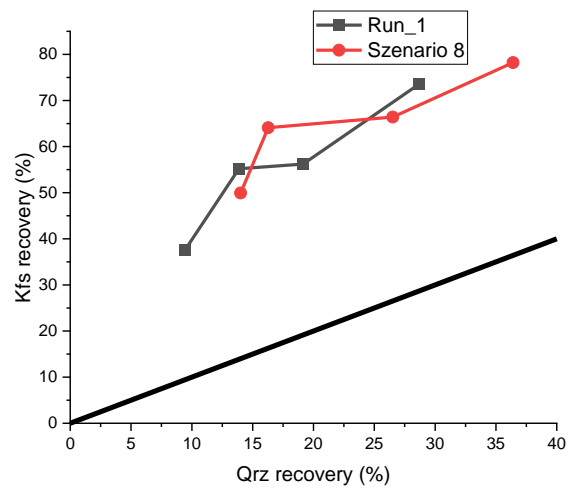
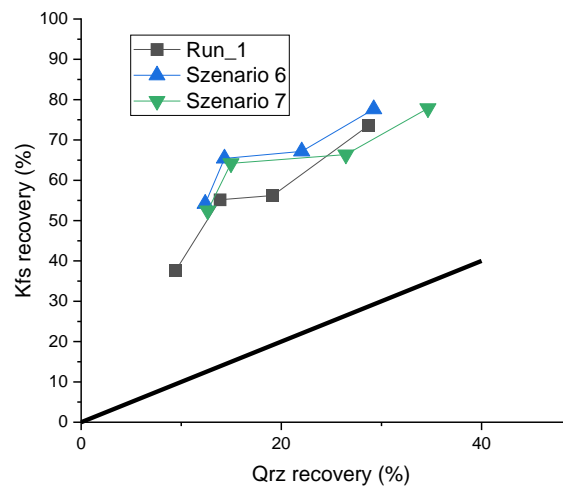
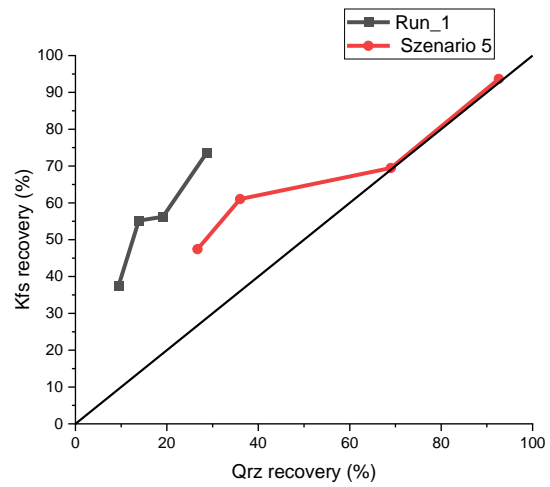
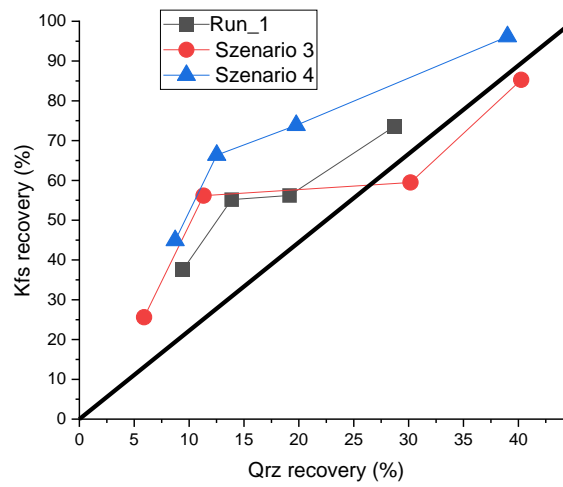
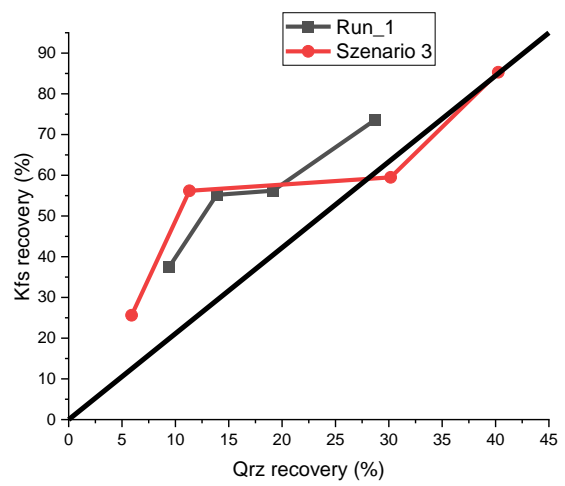
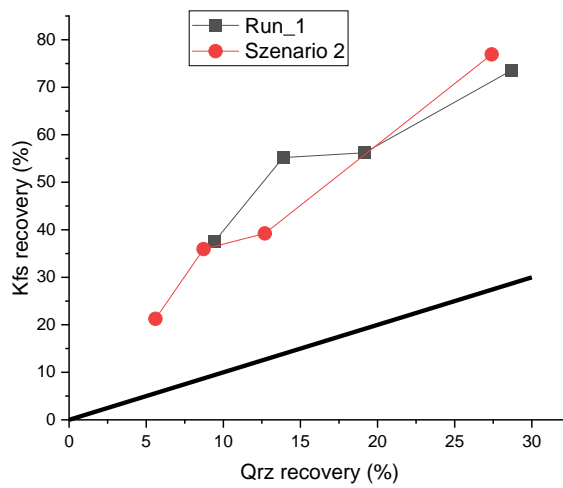


Figure 2 Selected results from the Design of Experiments (DoE) plan for feldspar–quartz flotation

The series of batch flotation scenarios developed from the baseline condition (Run 1) aimed to investigate how reagent addition sequence, conditioning time, and multi-stage dosing affect feldspar–quartz separation. Starting from the optimized setting, successive tests introduced controlled variations to assess selectivity trends (cf. Table 2). When NaF was added in both the rougher and scavenger stages (Scenario 2), feldspar recovery increased slightly, while quartz recovery remained nearly constant—showing no significant difference compared with the baseline Run 1. Lowering the pH to 2.4 (Scenario 3) led to an increase in quartz recovery, resulting in poorer selectivity. The addition of MIBC (35 g/t) as a frother (Scenario 4) improved froth stability and produced one of the best feldspar–quartz separation performances. Conditioning under mildly alkaline conditions (pH 5, Scenario 5) resulted in poor selectivity, with both feldspar and quartz recoveries exceeding 90%. In contrast, dual pH conditioning (Scenario 6, pH 5 → 3) produced results comparable to the baseline Run 1.

Subsequent tests varying the order of reagent addition revealed that amine pre-conditioning (Scenario 8) favoured higher overall recovery but lower selectivity, whereas NaF pre-conditioning (Scenario 10a) yielded high feldspar recovery and reduced quartz flotation, representing the best overall selectivity. Extended NaF conditioning times (Scenarios 14a–c) generally enhanced feldspar activation; however, excessively long conditioning or multiple amine additions tended to increase quartz recovery and deteriorate selectivity.

Overall, the results demonstrated that feldspar recovery benefits from sufficient NaF activation time and optimized reagent addition sequences, while maintaining selectivity requires controlled froth stability and pH conditions.



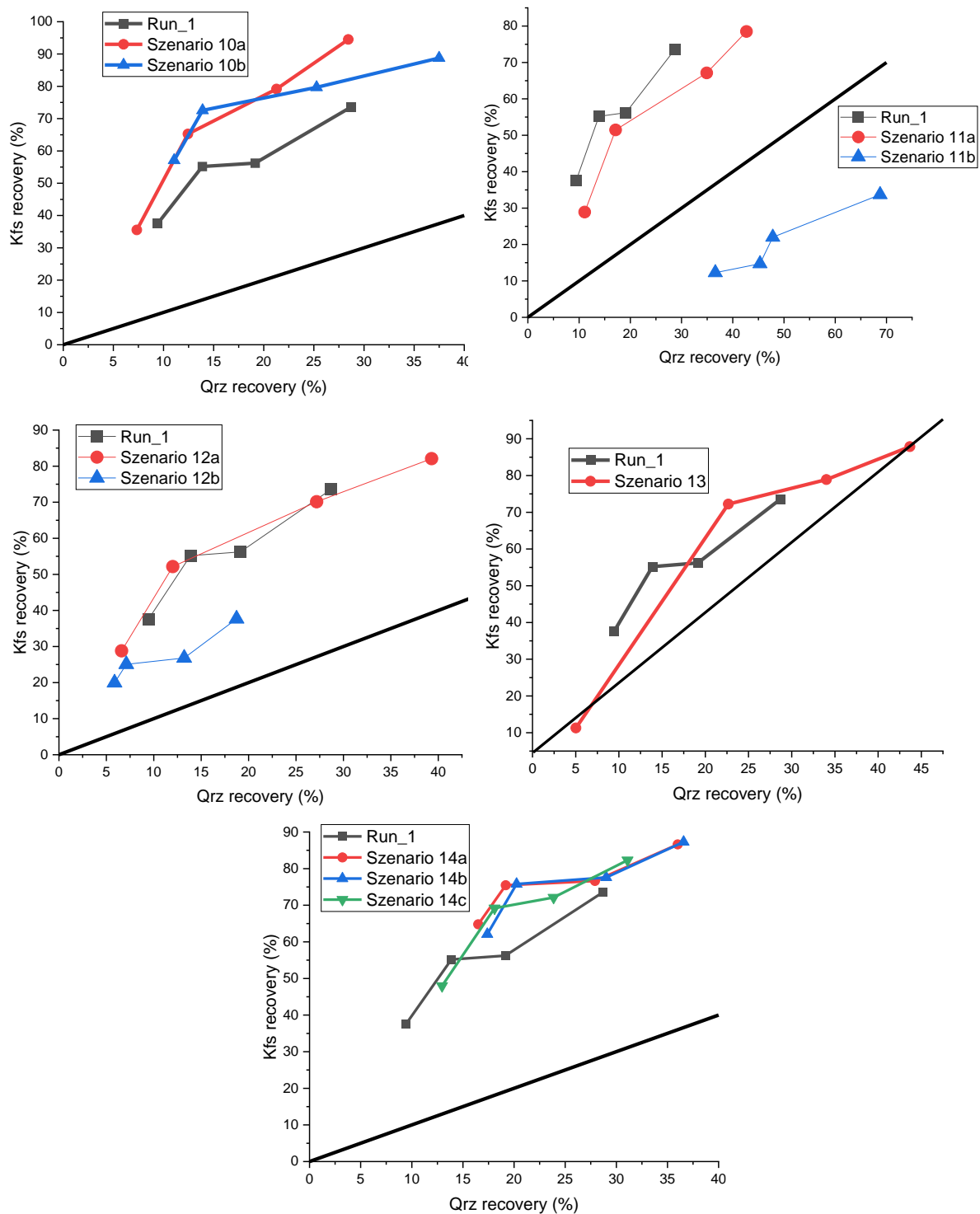


Figure 3 Results of the batch flotation scenarios conducted for feldspar–quartz separation, showing the influence of pH, NaF dosage, amine dosage, and reagent addition sequence on feldspar (Kfs) and quartz (Qrz) recoveries. The tests were derived from the baseline condition (Run 1: pH 3, NaF 1000 g/t, amine 1000 + 400 g/t) and designed to assess the effects of conditioning time, reagent order, and multiple reagent additions on flotation selectivity.

3.1.2 Pilot scale

Pilot-scale flotation tests were conducted in a continuous circuit consisting of three rougher cells, two cleaner cells, and two scavenger cells (**Figure 4**). The circuit was designed to replicate industrial feldspar flotation under controlled hydrodynamic conditions. Each test was operated until steady-state was achieved, during which the mass flows of the final concentrate and scavenger tailings were continuously recorded.

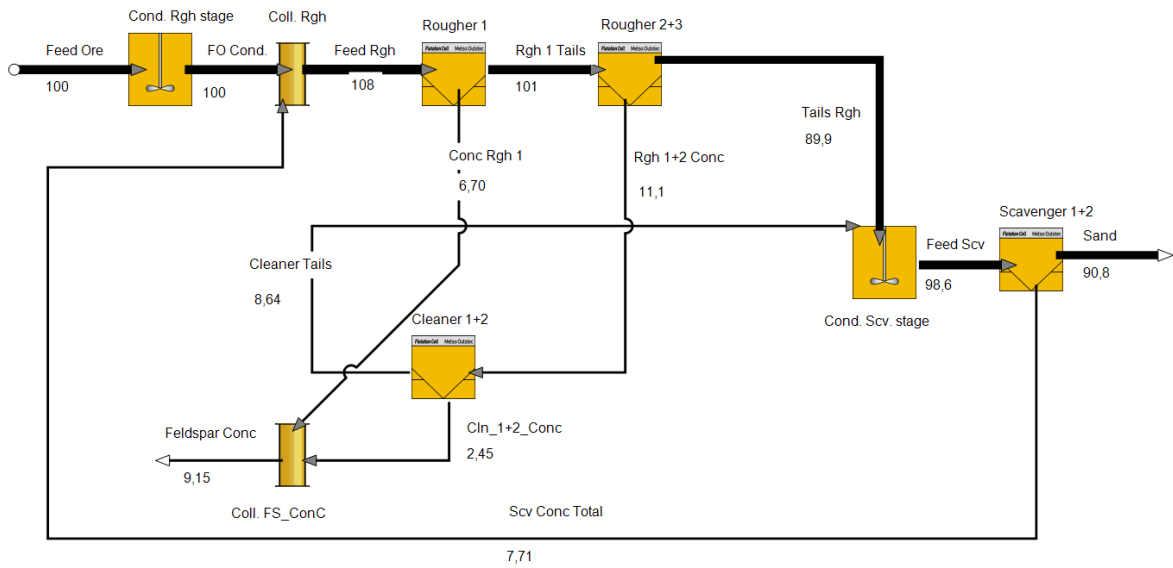


Figure 4 Process flowsheet of the pilot-scale feldspar flotation circuit consisting of three rougher, two cleaner, and two scavenger cells.

The overall feldspar recovery reached a maximum of approximately 57%, a value primarily constrained by the geometry of the pilot-scale cells. The cells were tall and narrow, requiring high turbulence to maintain suspension of the coarse feldspar particles ($d_{80} \approx 190 \mu\text{m}$). However, this elevated turbulence also increased the probability of particle–bubble detachment. As a result, despite effective feldspar activation by NaF, a considerable fraction of coarse feldspar remained in the scavenger tails, limiting the overall recovery.

3.2 Case Study II: Lignin-Based Biopolymer as a Substitute for Sodium Hydrosulfide in Cu–Mo Separation

3.2.1 Microflotation

The microflotation results presented in Figure 5 compare the performance of various lignin-based biopolymer depressants on chalcopyrite at pH 9, 10, and 11. Overall, most Pionera and DP-series reagents exhibited negligible depressing effect on chalcopyrite, as reflected by the consistently high flotation recoveries across the tested pH range. Only the F-250 product showed a noticeable reduction in chalcopyrite recovery.

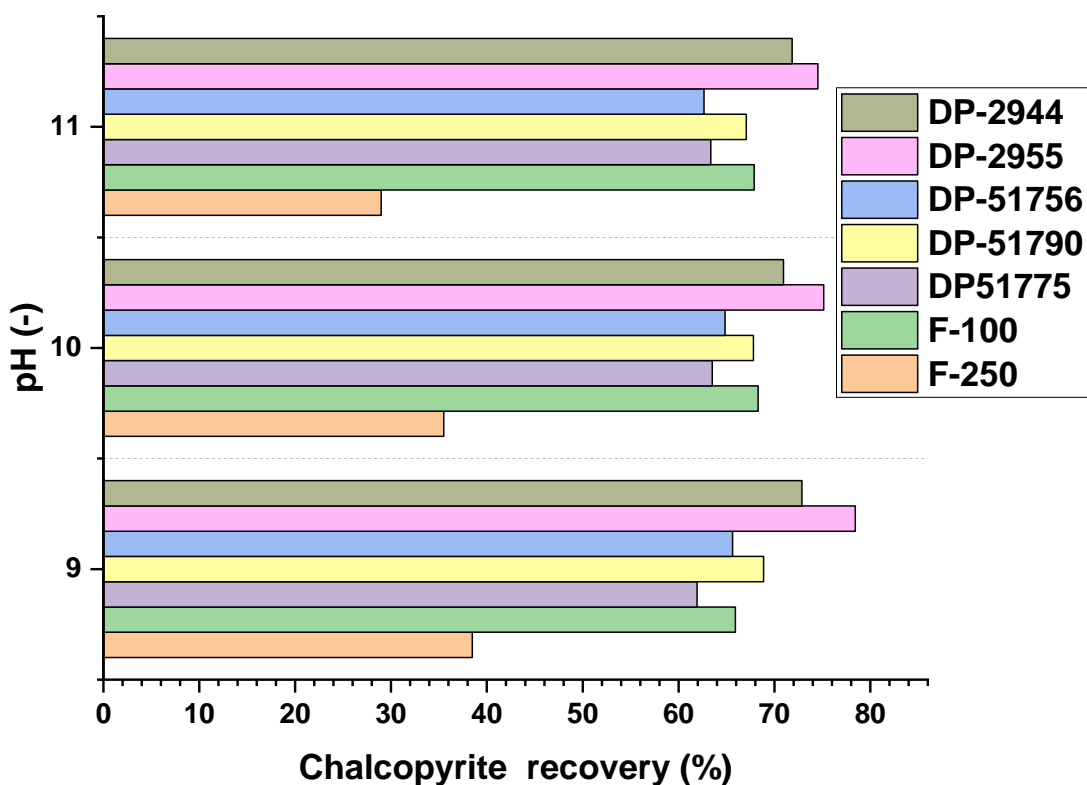


Figure 5 Microflotation results of chalcopyrite at pH 9, 10, and 11 in the presence of different lignin-based biopolymer depressants

3.2.2 Molybdenite flotation

3.2.2.1 Batch flotation

The direct molybdenite flotation tests were designed to evaluate whether lignin-based biopolymer depressants could selectively depress chalcopyrite while allowing molybdenite to float. Ekofol was used as the molybdenite collector, and the Pionera products were added as potential chalcopyrite depressants. Contrary to the intended objective, the results showed that the Pionera products strongly depressed molybdenite with a moderate depressing effect on chalcopyrite, leading to significantly lower Mo recoveries compared to the blank tests as shown in Figure 6. The degree of depression varied across products. Consequently, the initial flotation approach—targeting molybdenite recovery with concurrent chalcopyrite depression—was unsuccessful, prompting a reversal of the separation strategy in subsequent tests to depress molybdenite and float chalcopyrite.

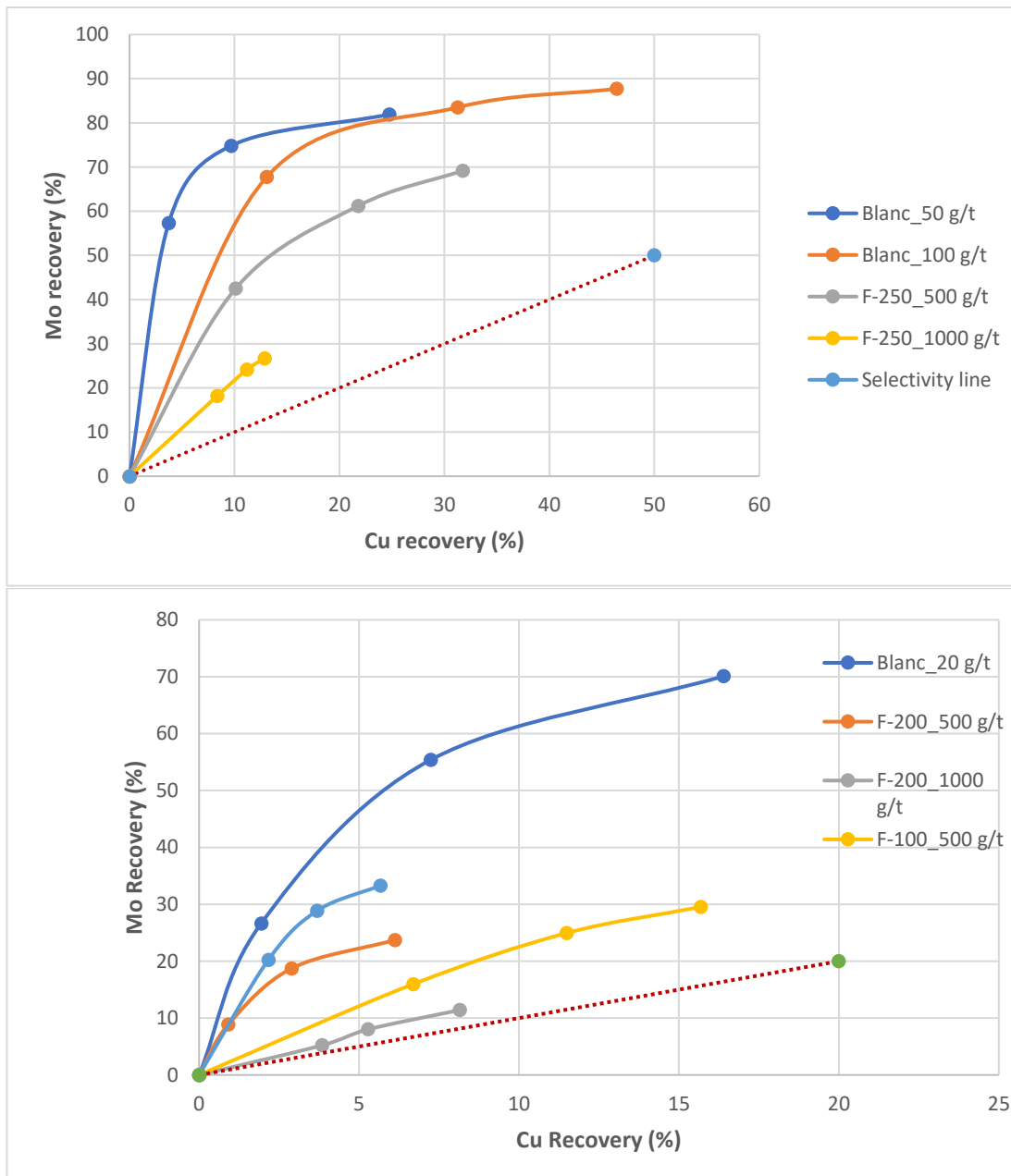


Figure 6 Molybdenite flotation results obtained with Ekofol collector at 20, 50, and 100 g/t in the presence of Pionera biopolymer depressants F-100, F-200, and F-250 (500–1000 g/t).

3.2.2.2 Pilot scale

The pilot-scale flotation tests were conducted in a continuous circuit comprising four rougher cells and two cleaner cells, as shown in Figure X, and followed the same reagent regime established during the batch experiments. A series of tests, carried out according to Table X, were performed to systematically vary the pH, Ekofol dosage in the rougher and cleaner stages, as well as the type and dosage of depressant, including a blank test without any depressant addition.

Table 3 Summary of pilot-scale flotation test conditions for the Cu–Mo ore. Parameters varied include pH, Ekofol collector dosage, depressant type and dosage, and the presence or absence of depressant (blank test).

	pH (-)	Ekofol dosage (g/t)	Depressant dosage (g/t)	Depressant type (-)
Blank	10.5	50	—	—
Test 1	11	30	1000	F-250
Test 2	10	70	2000	F_100

Test 3	9	90	500	F-200
Test 4	9.5	20	1500	F-500

The results confirmed the trends observed at batch scale as illustrated in figures Figure 7 to Figure 11. The Pionera lignin-based products consistently depressed molybdenite, leading to high Mo recoveries in the tailings and only limited molybdenite reporting to the final concentrate.

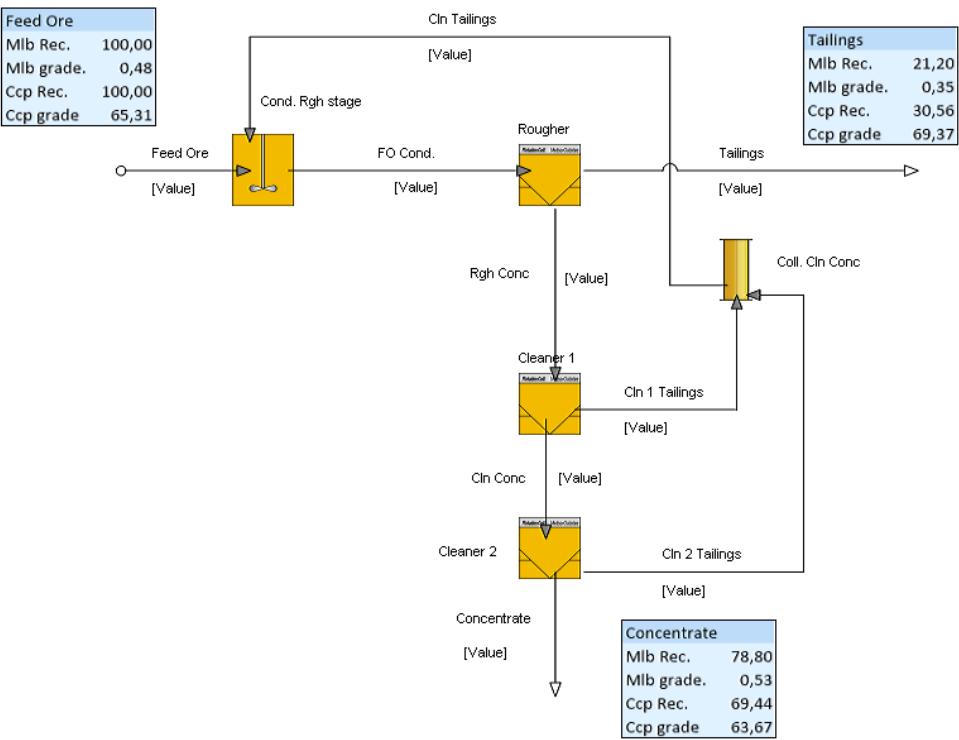


Figure 7 Schematic of the pilot-scale Cu–Mo flotation circuit and corresponding results for the blank test conducted without depressant addition.

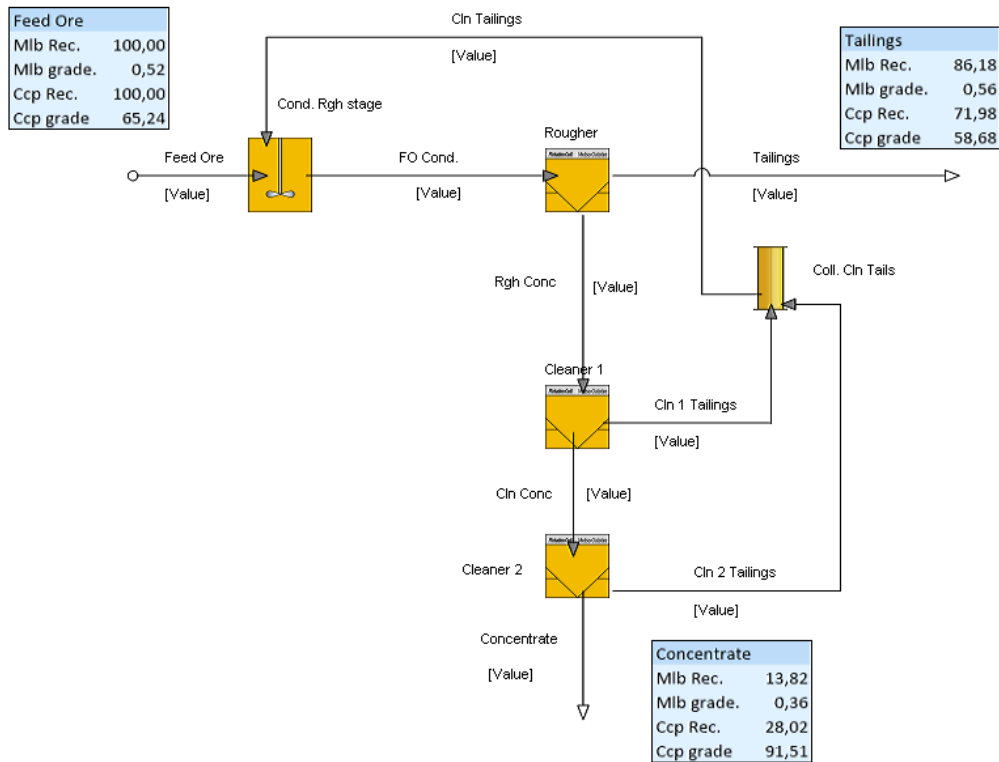


Figure 8 Schematic of the pilot-scale Cu–Mo flotation circuit and corresponding results for test 1

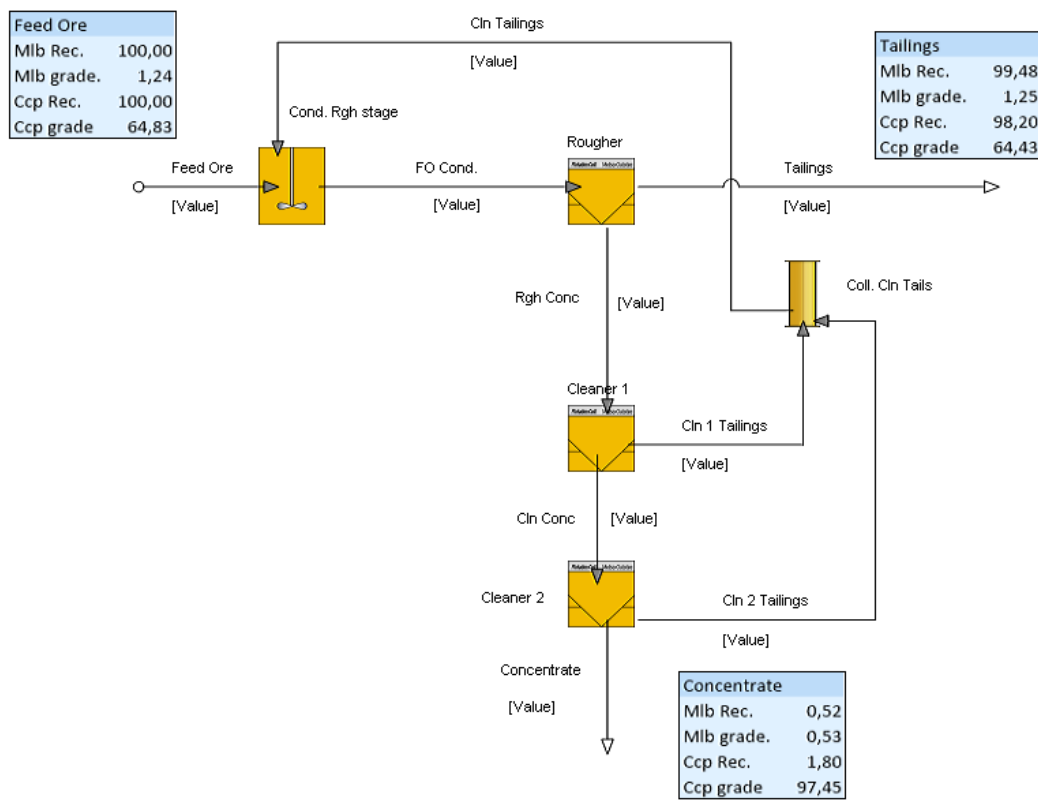


Figure 9 Schematic of the pilot-scale Cu–Mo flotation circuit and corresponding results for test 2

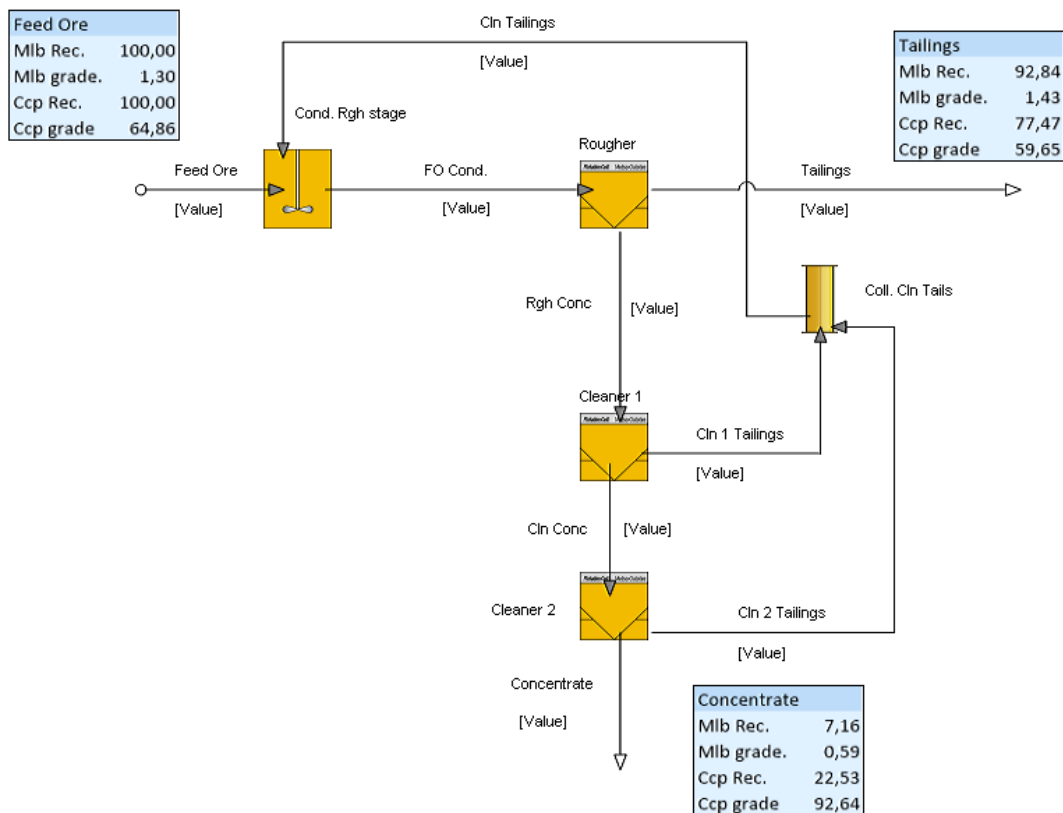


Figure 10 Schematic of the pilot-scale Cu–Mo flotation circuit and corresponding results for test 3

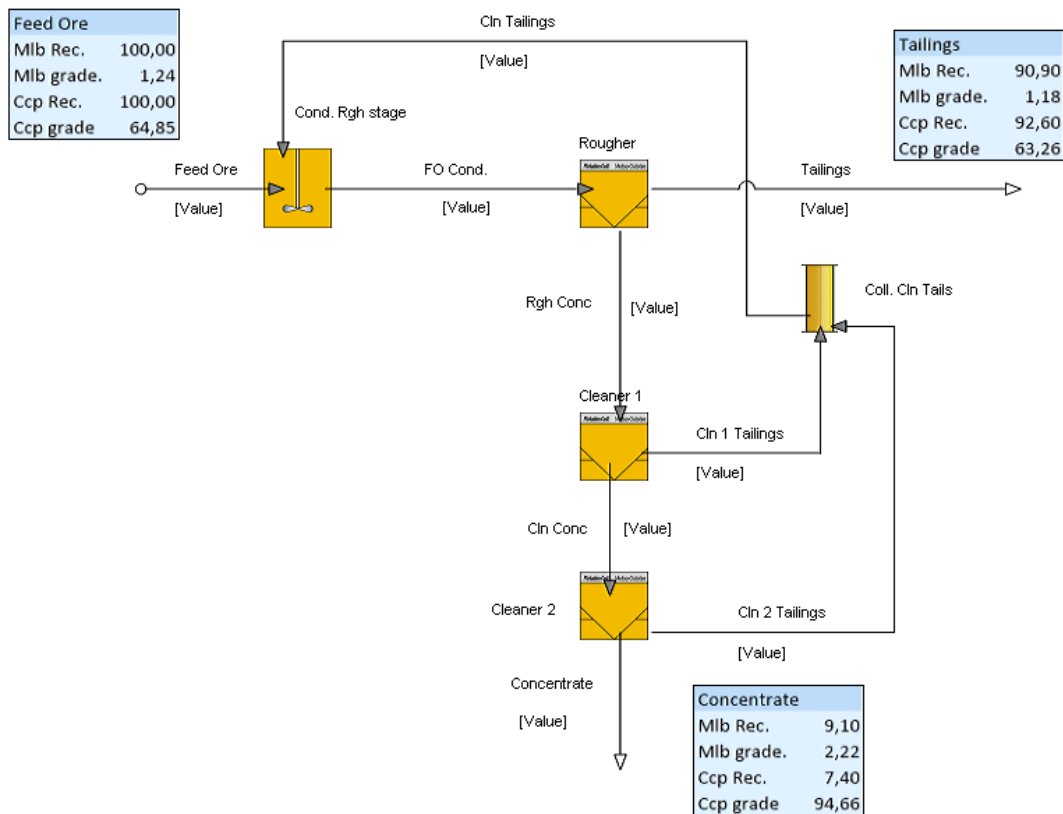


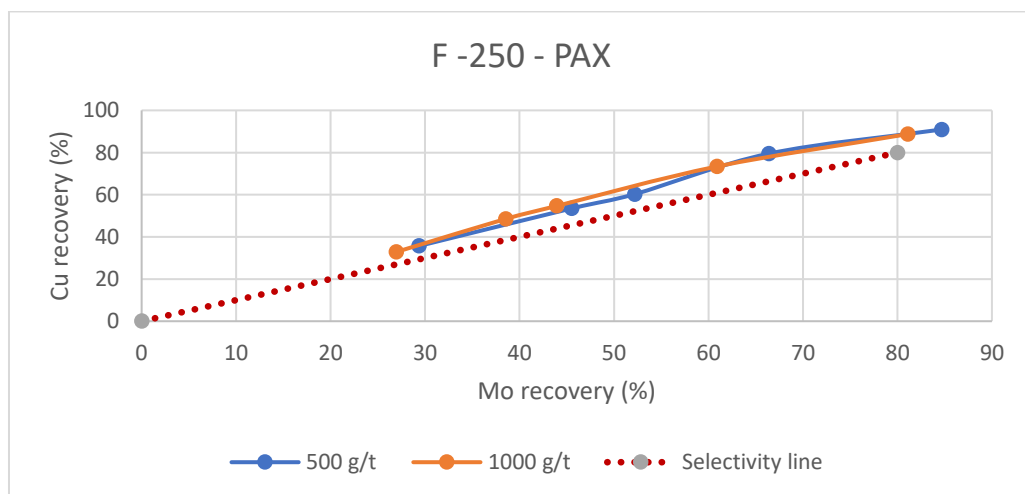
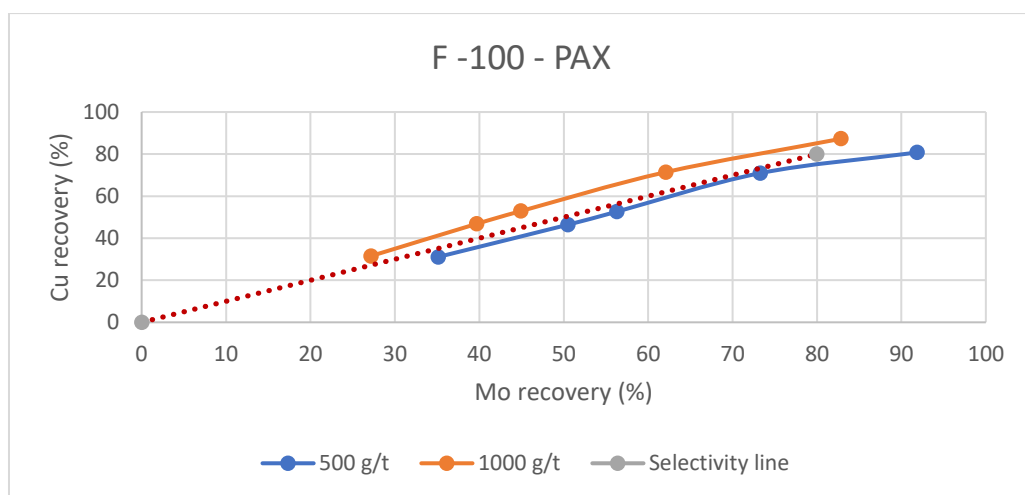
Figure 11 Schematic of the pilot-scale Cu–Mo flotation circuit and corresponding results for test 4

3.2.3 Chalcopyrite flotation

3.2.3.1 Batch flotation

The direct chalcopyrite flotation tests were performed to reverse the initial flotation strategy, aiming to selectively depress molybdenite while floating chalcopyrite. Experiments were conducted at pH 10.5, adjusted with CaO, using PAX (Figure 12) or SIBX (Figure 13) as collectors, while lignin-based biopolymers were evaluated as potential molybdenite depressants. The results showed that most Pionera products exerted only a moderate influence on selectivity, yielding comparable recoveries of Cu and Mo. Among the standard Pionera series, F-500 combined with PAX at 1000 g/t produced the best selectivity, achieving high chalcopyrite recovery while substantially depressing molybdenite flotation.

When the DP-51757 formulation was introduced (Figure 14), a marked improvement in Cu–Mo selectivity was observed at a dosage of 500 g/t. This product yielded high Cu recoveries with minimal Mo recovery, indicating a stronger and more specific interaction with molybdenite surfaces. Consequently, DP-51757 in combination with PAX was identified as the most promising reagent system and selected for preliminary pilot-scale tests to evaluate its performance under continuous operating conditions.



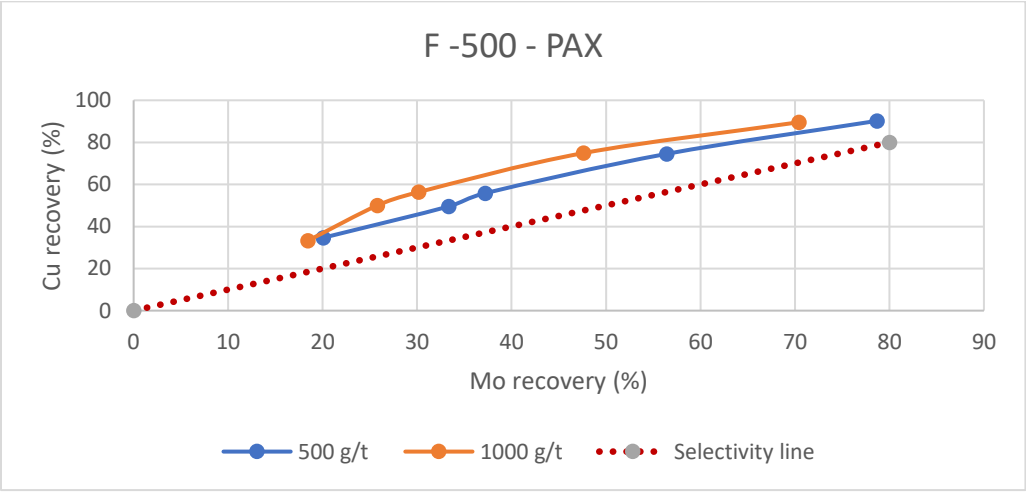
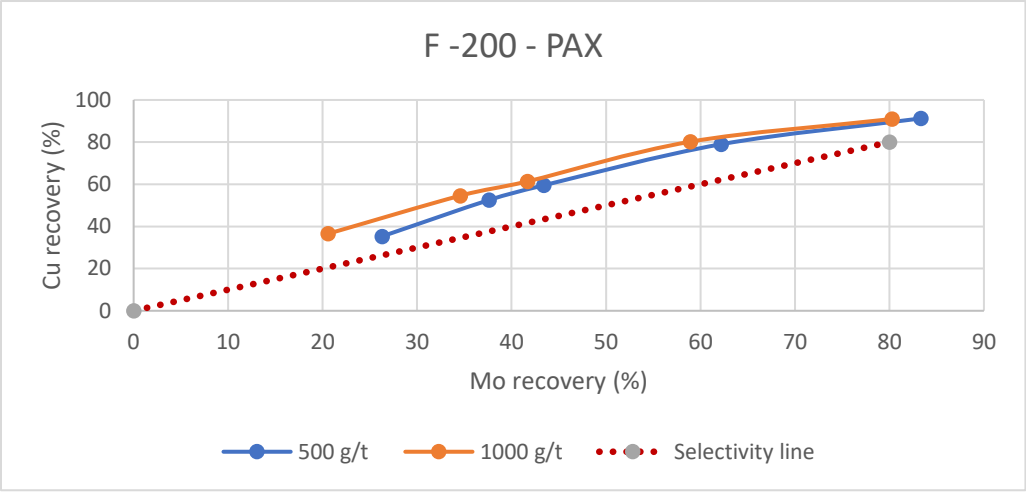
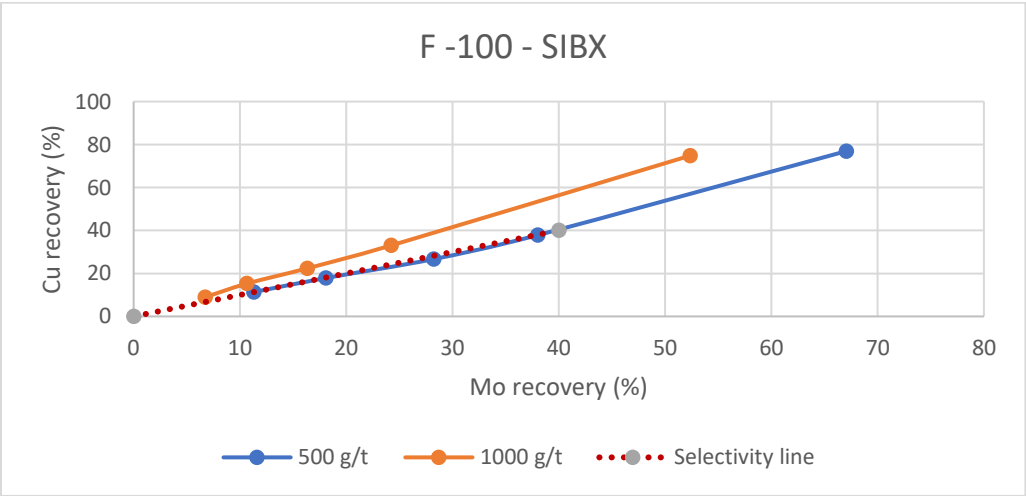


Figure 12 Effect of PAX dosage and depressant type on Cu–Mo separation during direct chalcopyrite flotation at pH 10.5. Tests were conducted using PAX and SIBX collectors with various Pionera lignin-based depressants.



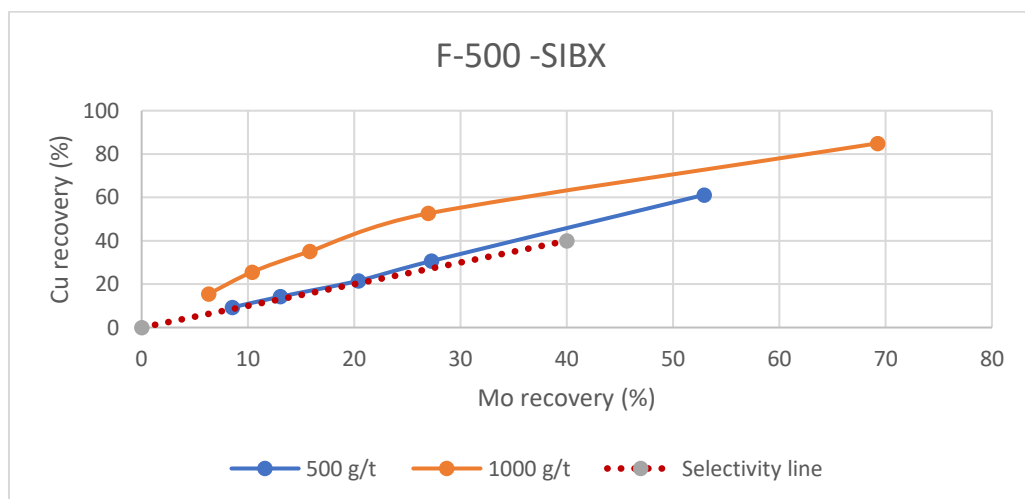
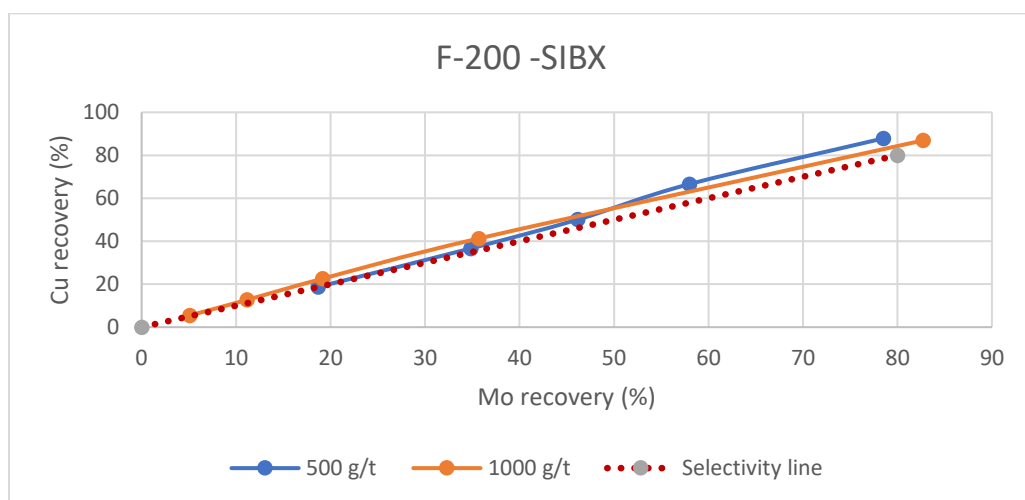
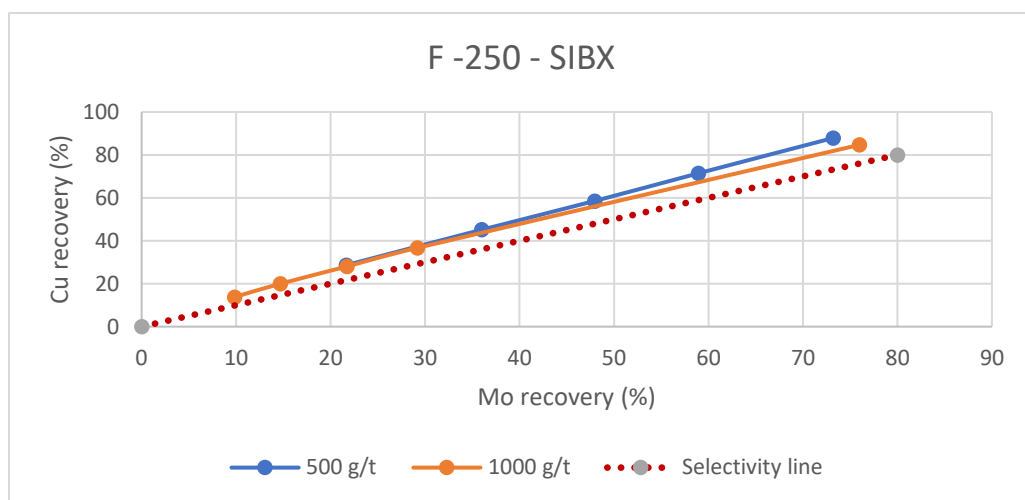


Figure 13 Effect of SIBX dosage and depressant type on Cu–Mo separation during direct chalcopyrite flotation at pH 10.5. Tests were conducted using PAX and SIBX collectors with various Pionera lignin-based depressants.

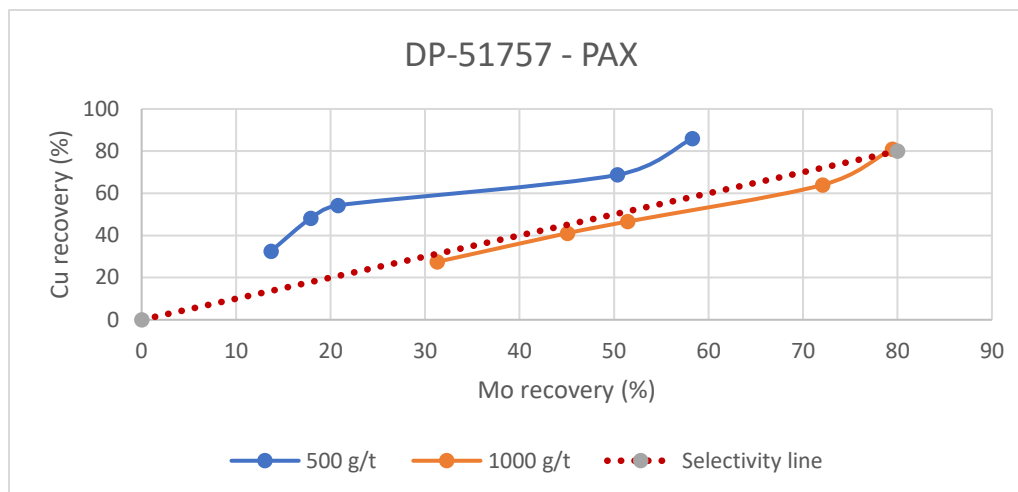


Figure 14 Effect of the DP-51757 lignin-based depressant dosage on Cu–Mo selectivity during direct chalcopyrite flotation at pH 10.5. The introduction of DP-51757 resulted in a marked improvement in selectivity at a dosage of 500 g/t, with high chalcopyrite recovery and minimal molybdenite flotation especially in the first stages, confirming its superior performance compared with standard Pionera products.

3.2.3.2 Pilot scale

The pilot-scale direct chalcopyrite flotation test was operated at pH 10.5, adjusted with CaO, using PAX as the collector and DP-51757 as the primary molybdenite depressant (Figure 15). The circuit reached a stable steady state after approximately 2.5 hours of operation. The results demonstrated a clear improvement in Cu–Mo selectivity compared with earlier pilot tests targeting molybdenite flotation. Chalcopyrite recovery reached about 93% Cu, while molybdenite recovery in the Cu concentrate remained relatively high at around 60%. This test is considered highly promising, serving primarily as a proof of concept to validate the selective depression behavior of DP-51757 under continuous operating conditions. Nevertheless, the reagent dosages still require further optimization to enhance the depression efficiency of molybdenite while maintaining high Cu recoveries. The circuit performance confirms the potential of this reagent system, and numerical optimized pilot tests are planned to maximize Cu recovery and improve molybdenite depression prior to industrial-scale validation.

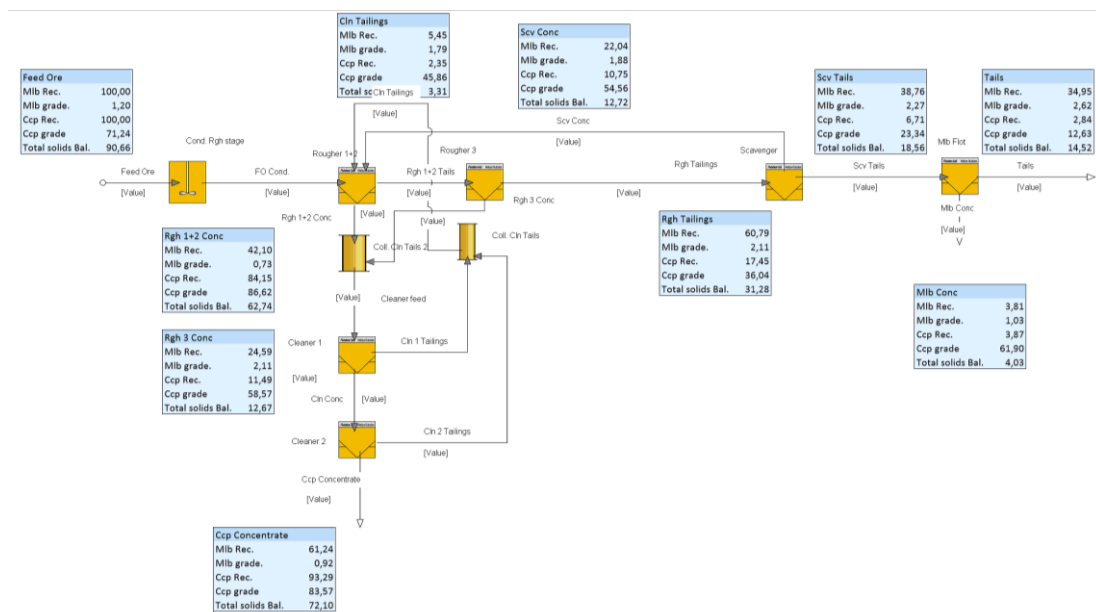


Figure 15 Pilot-scale direct chalcopyrite flotation using PAX (collector) and DP-51757 (molybdenite depressant) at pH 10.5

4 Conclusion

Both case studies demonstrate the potential of safer and more sustainable reagent systems to replace conventional chemicals in industrial flotation without compromising metallurgical performance.

In the feldspar–quartz system, sodium fluoride (NaF) proved to be a technically viable and much safer substitute for hydrofluoric acid (HF). Extended NaF conditioning and optimized reagent sequencing enhanced feldspar activation while minimizing quartz flotation. Pilot-scale tests confirmed the selectivity trends observed at laboratory scale, though the overall feldspar recovery (approx. 57%) was limited by the tall and narrow geometry of the pilot cells, which induced strong turbulence and particle–bubble detachment of coarse feldspar particles. Despite these hydrodynamic constraints, the results clearly validated NaF as an industrial alternative to HF.

In the Cu–Mo system, the flotation strategy was reversed to float chalcopyrite while selectively depressing molybdenite using lignin-based biopolymers as environmentally benign alternatives to sodium hydrosulfide (NaSH). The DP-51757 formulation, in combination with PAX at pH 10.5, achieved high Cu recovery and strong molybdenite depression within a shorter residence time compared to NaSH-based systems. Pilot-scale continuous tests confirmed this behavior, producing high-grade Cu concentrates with limited Mo contamination, demonstrating a safer and technically viable substitute for NaSH in Cu–Mo separation.

Overall, the two studies highlight the technical feasibility and industrial relevance of replacing hazardous reagents such as HF and NaSH with environmentally benign alternatives, showing that proper control of reagent chemistry, dosing, and process hydrodynamics can achieve sustainable mineral beneficiation without sacrificing selectivity or recovery.

5 References

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