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Sulfidization-Free Potassium Amyl Xanthate for Tenorite Recovery in Flotation of Copper-Quartz Systems

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Abstract

Copper oxide (Cu(II)O) and quartz are critical minerals in industrial applications, and their efficient separation is essential for optimizing resource recovery and waste management. This study investigates the use of Potassium Amyl Xanthate (PAX) as a collector for tenorite (CuO) flotation without prior sulfidization, a traditional but complex and costly process. Microflotation experiments demonstrated that CuO recovery reached approximately 80% at pH 10 with a PAX dosage of 600 g/ton, while quartz recovery remained negligible under the same conditions. Binary microflotation tests revealed the intricate dynamics between quartz and CuO. At a 25:75 Quartz to CuO ratio, quartz recovery in the concentrate was around 60%, while CuO recovery reached approximately 75%. When the proportion of quartz increased to 33:67, quartz recovery dropped slightly to 55%, while CuO recovery remained high at 80%. Remarkably, at a 5:95 ratio, quartz recovery significantly decreased to 35%, and CuO recovery was maximized at nearly 85%. These results demonstrate the selective effectiveness of PAX for CuO flotation, especially at higher CuO concentrations. Sodium silicate effectively suppressed quartz recovery, reducing it to as low as 20% at lower quartz-to-CuO ratios, particularly at pH 7 with a dosage of 400 g/ton. X-ray diffraction (XRD) analysis confirmed the effectiveness of separation and identified mineral transformations in concentrates and tailings. The results underscore the potential of PAX as an efficient and cost-effective collector for CuO flotation, offering a simplified and robust approach for improving mineral recovery in complex systems.

Keywords

Copper oxide, Microflotation, Copper oxides flotation, Silicates, Froth flotation

1. Introduction

Municipal solid waste incineration (MSWI) bottom ashes, shortly incineration bottom ashes (IBA), represent one of the largest mineral waste streams with an annual production of 19 million tons in the European Union (EU) [1]. It contains about 8 % ferrous and 2 % non-ferrous metals as elements or alloys which are recovered by magnetic and eddy-current separation, respectively [2]. Non-ferrous metals are the most valuable fraction of IBA, so despite their low share their recovery usually covers the entire costs of IBA processing [3]. However, copper, as the most important non-ferrous metal, occurs not only as element or alloy, but also in mineral phases such as tenorite (CuO) or cuprite (Cu_2O) [4] and to a lesser extent as covellite (CuS) [5] which may weather to secondary phases such as atacamite ($Cu_2Cl(OH)_3$) connellite ($Cu_{19}(SO_4)(OH)_{32}Cl_4 \cdot 3H_2O$) [6], nantokite (CuCl) or spertiniite ($Cu(OH)_2$) [7]. Since these compounds are not electrically conductive, they cannot be recovered by eddy-current separation, and remain in the mineral fraction of the

IBA. This not only corresponds to a resource loss, but also poses environmental threats due to the leaching of Cu into the environment when the mineral fraction is used as construction material [4].

In order to tackle this problem, froth flotation has been investigated to recover not only metallic Cu, but also Cu compounds from IBA, using dithiophosphates and thiocarbamates as collectors. By this approach 50 % of the Cu was recovered with Cu concentrations of about 5 % in the froth fraction [8]. For an increased yield and purity, a more fundamental understanding of the froth flotation process is required.

Copper oxides present in IBA, are more challenging with respect to froth flotation than copper sulphides present since they interact weaker with traditionally used copper sulphide collectors, are prone to dissolution in the process water during flotation and are characterized by strongly hydrophilic properties [9]. Fatty acids have been used as collectors for copper oxides without any pre-treatment [10], but in most cases a sulphidization of the oxides is applied prior to froth flotation [11]. Potassium amyl xanthate (PAX) has been used successful as collector for copper minerals, but in that case sulphidization of the originally present cuprite and tenorite was conducted using sodium sulphide (Na_2S) as a sulphidising agent [12].

Recently, investigations on single minerals and synthetic IBA components have been conducted. A mixture of thiocarbamate and thiophosphates, AERO MX-5160, was proved to be the most suitable collector to separate synthetic tenorite and covellite from individual slag constituents (quartz, glass, cement, gypsum) [5]. In that study, also the Zeta potential and the contact angle of individual phases were determined and the suitability of four different thiocarbamate, thiophosphate and thiourea collectors was tested in a Hallimond tube. Their results indicate that the Zeta potential of copper oxide varies between commercially available and self-synthesized tenorite. For pH ≤10, the former is in the range of -20 to -25 mV, the latter mostly around -40 mV. However, for pH ≥11 a similar Zeta potential of about -35 mV was obtained. They obtained recovery rates around 80 % for a broad pH range from 7 to 10 for thiourea, AERO 3473 and AERO MX-5160 as collectors. The contact angle of tenorite was found to be 42 ° without collector and more than 90° with any of the four collectors, whereas the contact angle of quartz was 0° without collector and with AERO 3473 and AERO MX5160, 24° with Danafloat 245 and >90° with Thiourea [5]. In the froth flotation experiments, in contrast to the Hallimond tube, AERO 3473 showed best results and about 95 % of all Cu could be recovered from a tenorite quartz mixture (1:24 mixing ratio).

In comparison to previous studies, novel results on the use of PAX as a collector for tenorite without prior sulfidization are presented. Furthermore, the effect of different mixing ratios between tenorite and quartz, ranging from 5% to 95%, was investigated.

2. Methodology and Materials

2.1 Materials

The study utilized high-purity copper oxide (tenorite “CuO”), and quartz as primary minerals, both prepared with an average particle size of approximately 150 microns. To systematically analyze their adsorption and flotation behaviors, “PAX” was employed as a collector to enhance the hydrophobicity of CuO. Quartz, on the other hand, was selectively depressed using sodium silicate to mitigate its flotation activity. Sodium hydroxide (NaOH) and hydrochloric acid (HCl) were used as pH modifiers to examine the influence of pH on flotation efficiency and surface interactions.

2.2 X-Ray Diffraction (XRD) Analysis

Representative samples were finely ground in a clean agate mortar to ensure homogeneity and analyzed using a G670 Diffractometer (HUBER Diffraktionstechnik GmbH & Co. KG, Germany) in transmission mode with a Guinier camera setup. The X-ray source was a copper cathode operating at 40 kV and 30 mA, with Cu Kα1 radiation filtered through a germanium monochromator crystal. Data collection for each sample was conducted over one hour.

The resulting XRD patterns were processed using Match! 4.0 software (CRYSTAL IMPACT, Germany) in semi-automatic mode with manual corrections. Final refinements were performed using the Rietveld method in the FullProf subroutine. Despite the known artifact introduced by the fixation gel at approximately $2\vartheta = 20°$, all diffraction peaks were successfully identified and analyzed.

For mixtures with uneven phase ratios (e.g., 95% to 5%), the Rietveld refinement approach yielded inconsistent results. To address this, a correction function was developed based on a comparison of samples with known ratios and their respective peak intensity ratios. The correction function is expressed as:

$$R_{\text{real}} = f_{\text{corr}}(R_{\text{int}})$$
$$f_{\text{corr}}(R_{\text{int}}) = 0.0188R_{\text{int}} + 2.25R_{\text{int}} + 0.309$$

Where R_{real} is the weighed in atomic ratio of each mixture, R_{int} is the ratio of the intensities measured at the respective highest peak of each phase and f_{corr} is the correction function to correlate based on maximizing R^2 figure of merit and visualized in **Figure 1** and **Table 2** .Using this correction, the real phase ratios in the binary microflotation experiments were accurately approximated based on the main peak intensity ratios in the XRD patterns.

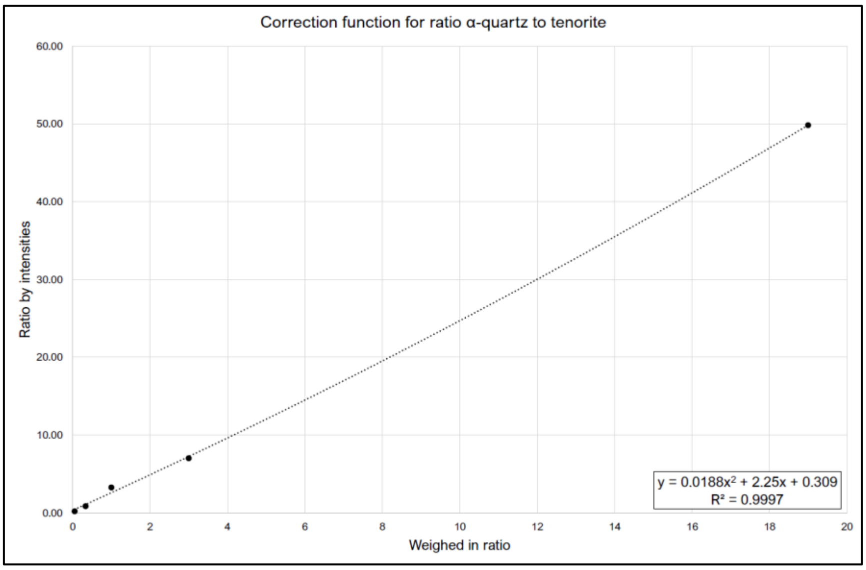


Fig. 1 Applied correction function with figure of merit based on mixtures with known fractions of quartz and tenorite.

Table 1 Parameters for the correction function. All ratios R are given as amount of quartz divided by tenorite.

	5%Q+95%T	**25%Q+75%T**	**50%Q+50%T**	**75%Q+25%T**	**95%Q+5%T**
$\boldsymbol{R}_{\textbf{int}}$	0.18	0.85	3.25	7.01	49.84
$\boldsymbol{R}_{\textbf{real}}$	0.05	0.34	1.00	3.00	19.00

2.3 Zeta Potential

Zeta potential measurements were conducted using a Zetasizer Nano ZS instrument (Malvern Instruments, Inc., Westborough, MA, USA) to assess the surface charge behavior of pure quartz and CuO, as well as their interactions with flotation reagents, across a pH range of 7 to 10. The reagents used were sodium silicate (acting as a quartz dispersant) and PAX (a CuO collector).

To prepare the samples, stock suspensions were made by dispersing 0.1 wt.% of each pure mineral into deionized water. The suspensions were sonicated for 5 minutes to ensure thorough dispersion and left to settle overnight to reach equilibrium. The pH of the suspensions was adjusted using a combination of 0.25M HCl as a strong acid, 0.1M HCl as a weak acid, and 0.25% NaOH as a strong base. These adjustments were precisely controlled using the MPT-3 Autotitrator (Malvern Instruments, Inc.) to achieve the desired pH conditions.

2.4 Microflotation Process

A Hallimond micro-flotation tube with an internal volume of 150 mL as shown in **Figure 2** was employed to investigate the flotation behavior of CuO and quartz using PAX as a collector and sodium silicate as a dispersant. The micro-flotation tests were conducted at room temperature to evaluate the selectivity of PAX in collecting CuO and the effectiveness of sodium silicate in dispersing quartz.

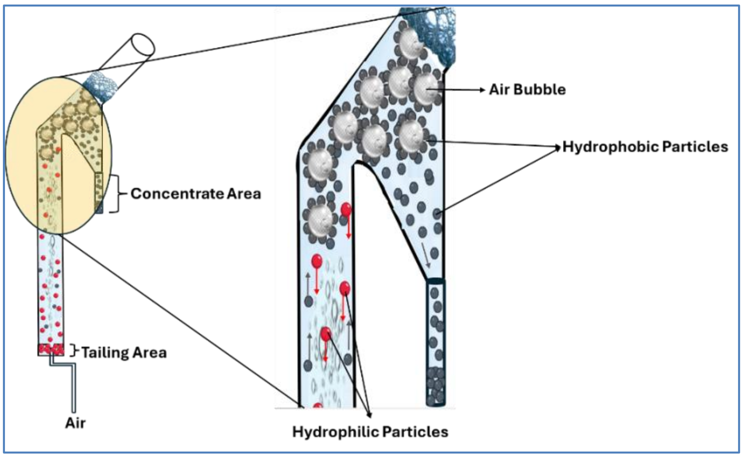


Fig. 2 Concept of Hallimond Micro-flotation tube

2.4.1 Single Mineral Microflotation Tests

In the single mineral experiments, CuO or quartz was tested individually to assess their specific responses to the flotation reagents. Nitrogen gas was introduced as the flotation medium at a flow rate of 40 cm³/min, with the pressure maintained at 10 psi. The particle size of the minerals was controlled within the range of +75 µm to -150 µm. Each flotation test used three grams of either CuO or quartz. The pulp was conditioned for 10 minutes before adding reagents, and the pH was adjusted using HCl and NaOH to the desired levels of 7, 8, 9, or 10, depending on the experimental setup. PAX was added as a collector for CuO at dosages of 200, 400, and 600 g/ton across the different pH levels. Similarly, sodium silicate was used as a dispersant for quartz at identical dosages and pH conditions. Following conditioning, the pulp was transferred into the Hallimond tube for flotation. The recovery of CuO and quartz was calculated based on the net dry weight of the concentrates, assuming a mineral grade of 100%. These experiments aimed to determine the optimal dosage and pH for the selective flotation of CuO with PAX and quartz with sodium silicate under various conditions.

2.4.2 Binary Mineral Microflotation Tests

After optimizing the conditions in the single mineral microflotation tests, binary microflotation experiments were conducted using the optimal reagent dosages and flotation conditions determined previously. Binary mixtures of CuO and quartz were tested at different ratios, including 25%-75%, 50%-50%, 75%-25%, 95%-5%, 5%-95%, 33%-67%, and 67%-33%.

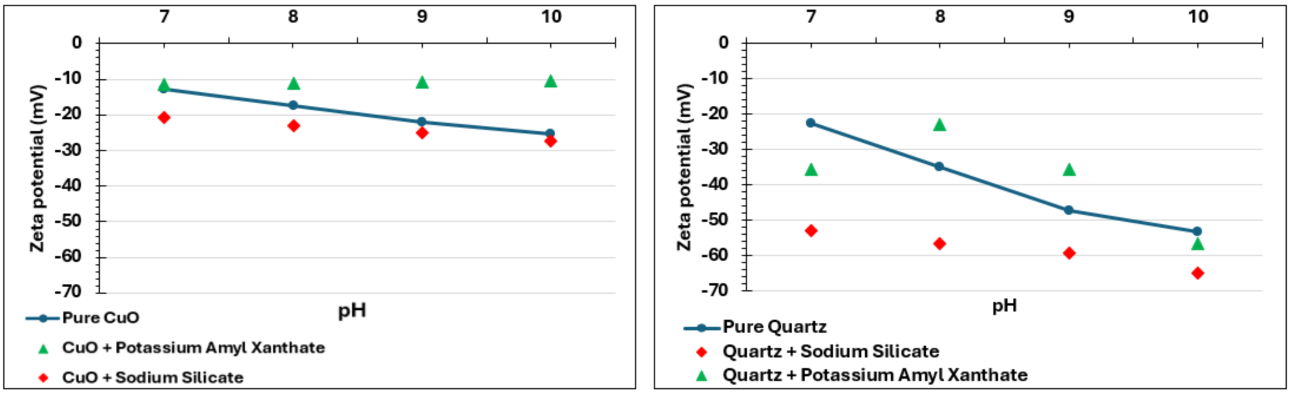
In these binary tests, a total of 3 grams of the mineral mixture was used, reflecting the specific proportions of CuO and quartz. The same optimized pH levels and reagent dosages from the single mineral tests were applied. The pulp was conditioned according to the optimized protocol before being introduced into the Hallimond tube for flotation. Recovery rates were calculated based on the net dry weight of the concentrates, and XRD analysis was performed to determine the mineral composition in the concentrates. These binary flotation experiments were designed to assess the selective separation capability of PAX and sodium silicate under varying mineral ratios, offering insights into their performance in complex mineral systems.

3. Results and Discussion

3.1 Zeta Potential Measurements

The zeta potential measurements were conducted on both CuO and quartz under varying pH levels (7, 8, 9, and 10), before and after treatment with reagents— PAX and sodium silicate. As seen in **Figure 3** the zeta potential values of the pure minerals without reagents demonstrated typical pH-dependent trends, with a more negative potential observed at higher pH values. The zeta potential of pure CuO showed a linear decrease from -11 mV at pH 7 to approximately -40 mV at pH 10, which indicates a typical negative surface charge as pH increases. After the addition of PAX, a notable shift in the zeta potential values was observed. The surface charge became less negative across all pH values, indicating adsorption of the collector on the CuO surface, with the magnitude of the zeta potential change (Δξ) increasing from +1.4 mV at pH 7 to +15 mV at pH 10. The positive shift in zeta potential after treatment suggests the adsorption of the collector molecules onto the mineral surface, likely due to electrostatic interactions or chemical bonding between CuO and the xanthate ions. In contrast, treatment with sodium silicate resulted in a more negative zeta potential, especially at lower pH values. The magnitude of the change (Δξ) reached a peak of -8.5 mV at pH 8 and gradually reduced to -1.8 mV at pH 10. This suggests that sodium silicate acts as a dispersant, preventing aggregation by increasing the repulsion between particles due to the more negative surface charge.

The zeta potential of pure quartz also exhibited a decreasing trend with increasing pH, from approximately -30 mV at pH 7 to -60 mV at pH 10. The addition of sodium silicate resulted in a pronounced shift towards more negative values, with the greatest effect seen at pH 7, where the magnitude of the zeta potential change (Δξ) reached -30.39 mV, indicating strong adsorption of the dispersant. The dispersant's ability to impart a highly negative surface charge on quartz particles plays a key role in preventing their flotation by promoting dispersion. The addition of PAX had a milder effect, particularly at lower pH levels, with Δξ being -13.11 mV at pH 7 and gradually reducing to -3.52 mV at pH 10. This suggests a limited interaction of PAX with quartz, consistent with its role as a collector for CuO.



Condition	The magnitude of the zeta potential Δξ			
	pH 7	pH 8	pH 9	pH 10
CuO + Sodium Silicate	-8	-5.5	-2.9	-1.8
CuO + Potassium Amyl Xanthate	+1.4	+6.2	+11.4	+15

Condition	The magnitude of the zeta potential Δξ			
	pH 7	pH 8	pH 9	pH 10
Quartz+ Sodium Silicate	-30.39	-21.505	-11.79	-11.8
Quartz+ Potassium Amyl Xanthate	-13.11	+11.875	+11.82	-3.52

Fig. 3 Zeta potential measurement of CuO (Left) and Quartz (Right) as a function of pH.

3.2 Microflotation Experiments

The microflotation tests were conducted in two phases to investigate the flotation recovery of CuO with PAX and quartz with sodium silicate. The aim was to identify the optimum reagent doses and pH levels for each mineral, as these conditions were used in the second phase of the study for binary flotation.

3.2.1 Single Minerals Microflotation

As shown in **Figure 4**, the recovery of CuO increased progressively with the dosage of PAX across all pH levels. At a dose of 200 g/ton, the CuO recovery ranged from about 50% at pH 7 to approximately 55% at pH 10. However, when the dosage was increased to 400 g/ton, a significant improvement in recovery was observed, with values reaching around 70-75% at pH 9 and 10. The highest recovery rates were achieved at the dose of 600 g/ton, particularly at pH 10, where recovery approached 80%, indicating that this combination of dose and pH was the most effective for maximizing CuO flotation. The results reveal that the optimum dose was found to be 600 g/ton of PAX, with pH 10 as the most favorable condition. Under these conditions, CuO recovery reached approximately 80%, highlighting the strong interaction between PAX and the CuO surface in an alkaline environment. The improved recovery at pH 10 can be attributed to the enhanced adsorption of PAX, which increases the hydrophobicity of CuO particles, thereby facilitating their flotation.

The quartz recovery was consistently low across all doses of sodium silicate, demonstrating the effectiveness of the reagent as a depressant. At a dose of 200 g/ton, quartz recovery decreased from around 40% at pH 7 to 30% at pH 10, indicating that the depressant effect of sodium silicate strengthened with increasing pH. At the higher dose of 300 g/ton, quartz recovery further decreased to around 25-30% across all pH levels, demonstrating that increasing the sodium silicate dose enhanced its depressant effect on quartz flotation. These results show that pH 7 with 400 g/ton of sodium silicate was more effective in depressing quartz, which is desirable for the binary flotation process. While the zeta potential became more negative with increasing pH, the microflotation results indicated that pH 7 was more effective at suppressing quartz recovery.

The zeta potential results align with the single mineral flotation results by demonstrating that less negative zeta potentials at pH 10 for CuO correlate with higher recovery using 600 g/ton of PAX, while more negative zeta potentials for quartz at pH 7 correspond with stronger depression by 400 g/ton of sodium silicate and lower quartz recovery. These conditions were selected based on the outcomes of the single-phase tests to ensure optimal performance in the binary flotation system.

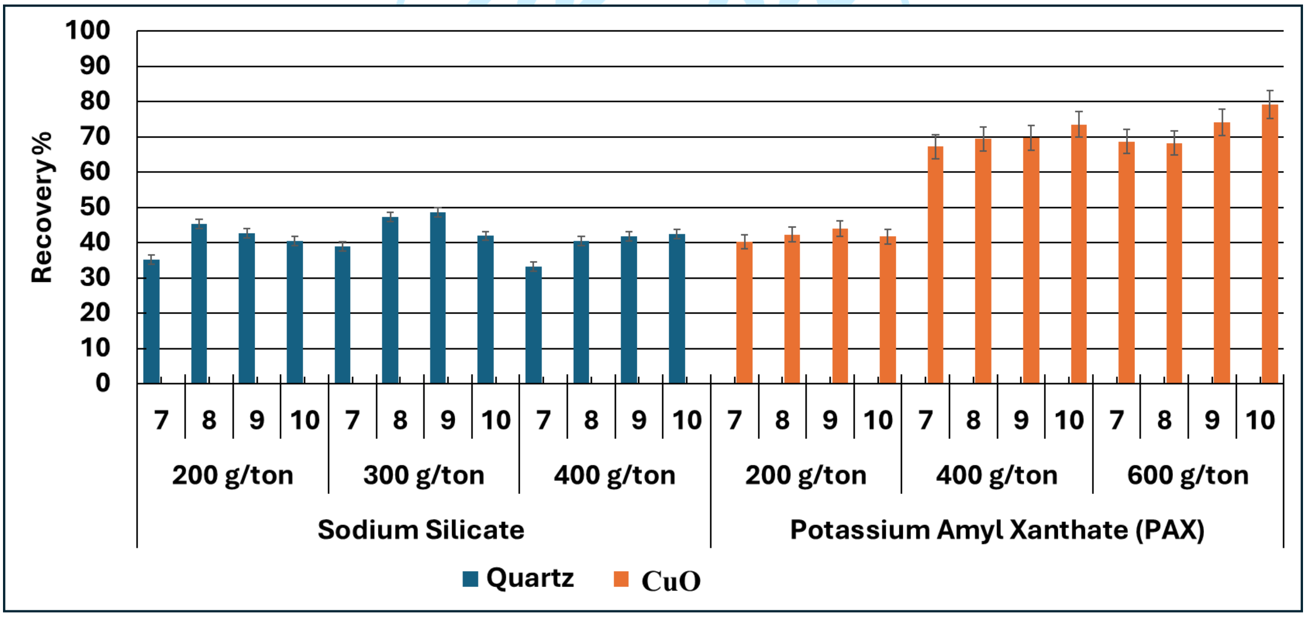


Fig. 4 Recovery (%) of Quartz and CuO as a function of pH and reagent dosage.

3.2.2 Binary Minerals Microflotation

The binary microflotation tests aimed to evaluate the selective recovery of CuO and quartz across various quartz and CuO ratios. The results, as shown in **Figure 5**, demonstrate significant variation in the recovery of both minerals based on their relative concentrations in the system.

At a 25:75 Quartz to CuO ratio, the recovery of quartz in the concentrate was around 60%, while the recovery of CuO reached approximately 75%. As the proportion of quartz increased to a 33:67 ratio, the recovery of quartz dropped slightly to around 55%, while CuO recovery remained high at around 80%. Interestingly, when the system was dominated by CuO at a 5:95 ratio, the recovery of quartz significantly decreased to around 35%, while CuO recovery was maximized, reaching close to 85%. The binary microflotation results reveal the intricate dynamics between quartz and CuO during flotation. The recoveries of both minerals are highly dependent on their respective concentrations in the system. Notably, CuO recovery remained consistently high across most Quartz to CuO ratios, which highlights the effectiveness of PAX as a collector for CuO, especially at higher concentrations of CuO. This aligns well with the findings from the single-phase flotation and zeta potential results, where pH 10 and a 600 g/ton dose of PAX showed optimal performance in recovering CuO.

On the other hand, quartz recovery was most effectively suppressed as the CuO content increased in the system. This can be attributed to the efficient depression of quartz by sodium silicate at pH 7 and a 400 g/ton dose, which was selected as the optimal condition for quartz depression. The selective depression of quartz is more evident at lower Quartz to CuO ratios, where quartz recovery was reduced to as low as 20%. This demonstrates that the dosage and pH conditions selected for sodium silicate are highly effective in minimizing quartz flotation, even in the presence of CuO. Interestingly, the results show that as the proportion of quartz in the system increased (e.g., 50:50, 67:33, 75:25 ratios), quartz recovery became more competitive with CuO recovery. This suggests that while the depressant (SS) is effective at lower quartz concentrations, higher quartz proportions make it more challenging to fully suppress quartz flotation. However, even in systems with a high quartz content, CuO recovery remained relatively high, indicating that PAX continues to act selectively on CuO under these conditions.

These binary flotation results confirm the effectiveness of the selected flotation conditions (pH and reagent dosages) in achieving the desired selective separation of CuO from quartz. This lays the foundation for future studies focused on further optimizing reagent conditions and investigating the performance of this binary system in a more complex mineral matrix.

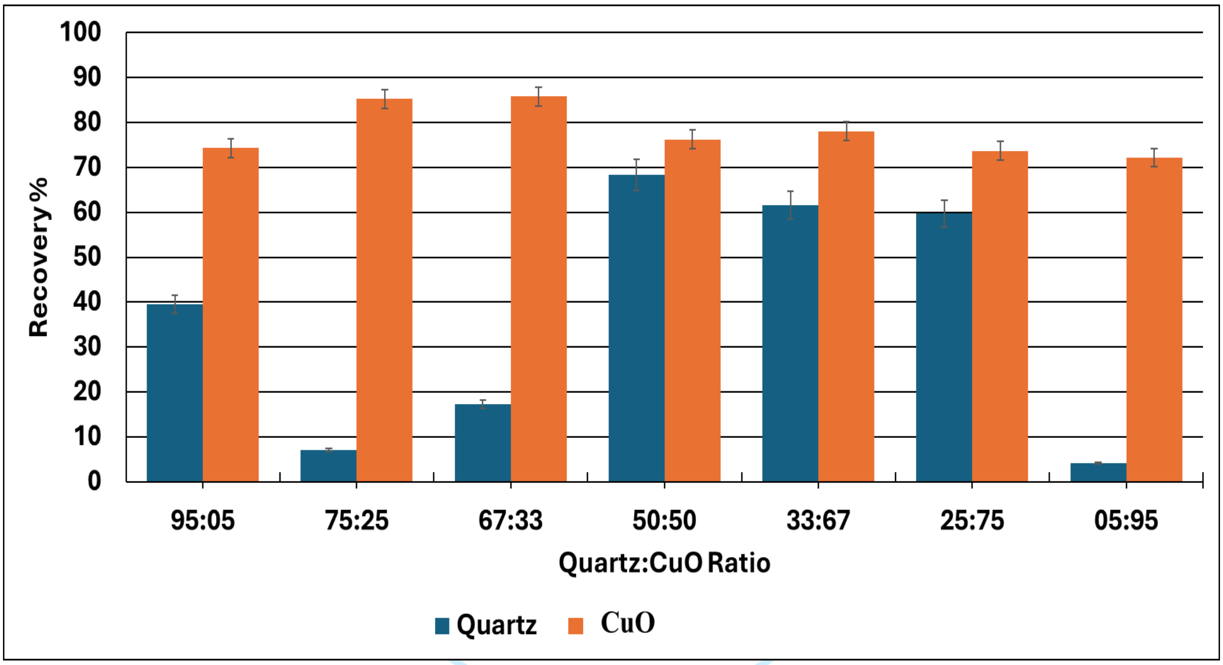


Fig. 5 Microflotation performance of Quartz: CuO mixture at the optimum flotation parameters.

The XRD analysis of binary mixtures revealed significant variations in the phase composition of the collected samples and tailings. **Figure 6** illustrates the diffractograms of these mixtures, with dominant peaks attributed to α-Quartz and Tenorite. Using a correction function based on the intensity ratios of the primary peaks, the phase ratios were determined with improved accuracy, as summarized in **Table 2**.

The XRD results (**Table 2**) indicate that α-Quartz consistently dominates the phase composition across all collections and tailings, with its proportion ranging from 67% to 99% in the collected samples and 55% to 99% in the tailings. Tenorite was present in smaller amounts, varying between 1% and 33% in the collections and 1% to 45% in the tailings.

Table 2 Ratios of the binary constituents by correction of intensity ratios by "XRD'' (Quartz: CuO).

Collection	**C1 95:05_2**	**C2 75:25_2**	**C3 67:33_2**	**C4 50:50_2**	**C5 33:67_2**	**C6 25:75_2**	**C7 05:95_2**
α Quartz	78%	94%	88%	96%	97%	67%	99%
Tenorite	22%	6%	12%	4%	3%	33%	1%
Tailing	**T1 95:05_2**	**T2 75:25_2**	**T3 67:33_2**	**T4 50:50_2**	**T5 33:67_2**	**T6 25:75_2**	**T7 05:95_2**
α Quartz	84%	92%	86%	95%	96%	55%	99%
Tenorite	16%	8%	14%	5%	4%	45%	1%
Relative differences of the collection compared to tailing fraction							
α Quartz	-8%	+2%	+2%	+1%	+1%	+21%	+/-0%
Tenorite	+41%	-27%	-14%	-25%	-17%	-26%	-24%

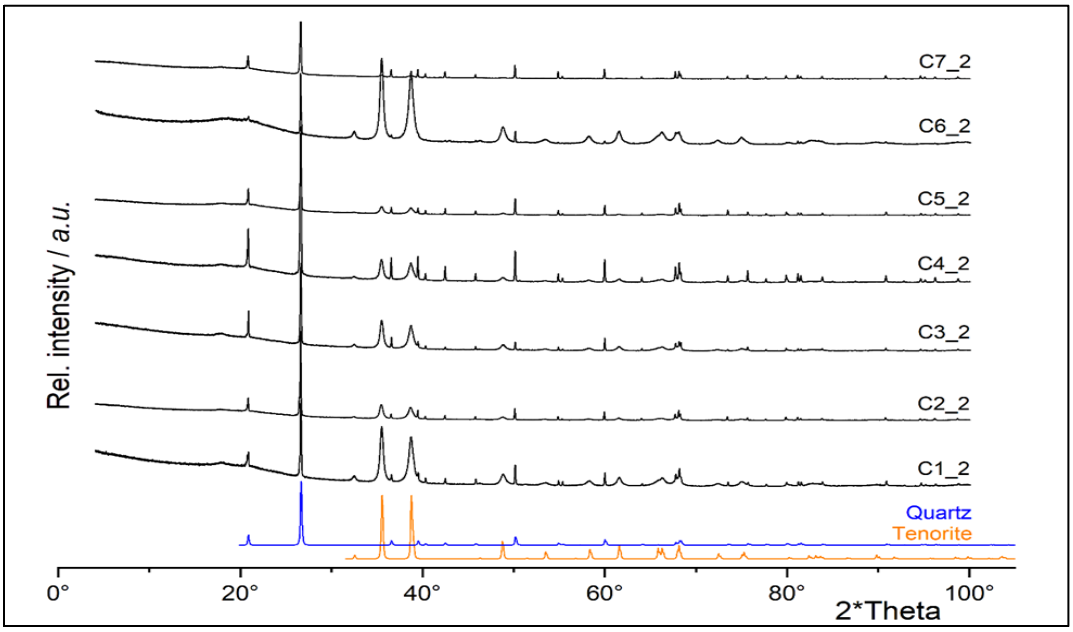


Fig. 6 XRD pattern of collected quatz+tenorite fractions, compared to calculated diffractograms based on COD entries

These XRD findings align with the microflotation results, where the dosage of PAX and sodium silicate played crucial roles. At higher PAX dosages and pH 10, Tenorite recovery improved significantly, achieving up to 80%, while sodium silicate effectively suppressed quartz flotation, particularly at pH 7.

The observed trends in phase composition suggest that reagent optimization is critical to achieving selective separation. Lower quartz recovery in tailing fractions at higher Tenorite concentrations reflects the efficiency of the flotation conditions in enhancing Tenorite hydrophobicity while minimizing quartz flotation.

4. Conclusion

This study presents a significant advancement in the flotation of tenorite (CuO) by demonstrating the feasibility of using PAX as a collector without the need for prior sulfidization. Traditionally, sulfidization has been considered essential for enhancing the flotation of copper oxide minerals; however, our findings in XRD analysis and zeta potential measurements confirmed the successful identification of mineral compositions and elucidated the surface charge interactions at varying pH levels. Specifically, CuO exhibited a notable increase in recovery, reaching approximately 80% at pH 10 with a PAX dosage of 600 g/ton. In contrast, quartz recovery was minimized, demonstrating the effectiveness of sodium silicate, particularly at pH 7 with a dosage of 400 g/ton, where recovery was reduced to around 25-30%.

The binary flotation tests highlighted the selective recovery capabilities of PAX, revealing that CuO recovery remained high across various quartz to CuO ratios, while quartz recovery was significantly suppressed as the concentration of CuO increased. These results illustrate the intricate balance between collector and depressant interactions, emphasizing the potential for tailored reagent strategies to enhance flotation selectivity in complex mineral systems.

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Declaration of Conflict

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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