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Specific Ion Effects on the Behaviour of Mixtures of Sodium Iso-Butyl Xanthate and Sodium Di-Ethyl Dithiophosphate during the Flotation of a Cu-Ni-PGM Ore: Effects of CaCl₂ and NaCl

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Abstract: Inorganic electrolytes present in the process water used during froth flotation may have both beneficial and detrimental effects. These effects are said to be ion specific as some ions may result in enhanced froth stability, increased mineral recoveries and decreased concentrate grades while others may bring the opposite effects. Onsite process water quality variations have intensified the need to understand the relationship between inorganic electrolytes and flotation reagents on flotation performance. The use of mixtures of thiol collectors in sulfide flotation is a common practice across the globe, however very little investigations have considered these in process waters of varying compositions. This study considers the effect of common cations, Na⁺ and Ca²⁺ in process water on the behaviour of mixtures of thiol collectors. Single salt solutions of NaCl and CaCl₂ at an ionic strength of 0.0213 mol.dm⁻³ were used to investigate the behaviour of mixtures of two thiol collectors. These were carefully selected to understand how mixtures of thiol collectors behave in the presence of a monovalent cation versus a polyvalent cation. Bench-scale froth flotation tests were conducted using a Cu-Ni-PGM ore from the Merensky Reef. The results have shown that the divalent cation, Ca²⁺, resulted in higher %Cu and %Ni recoveries at all collector mixtures compared to the monovalent cation, Na⁺. The concentrate grades were however slightly compromised as slightly more gangue reported to the concentrate in the presence of Ca²⁺. This behaviour is attributed to the effect of polyvalent cations on bubble coalescence and froth stability.

Keywords: Electrolytes; Froth Stability; Process Water; Thiol Collector Mixtures; Water quality

1. Introduction

Mixtures of collectors are widely used in the flotation of sulfide minerals and are believed to have performance benefits. An increase in valuable metal recovery, increase in flotation kinetics and reduction in reagent consumption has been reported [1–3]. A fundamental study on the synergistic effect of mixtures of dithiocarbonates with different radical groups and dithiocarbonates mixed with xanthates on pyrite flotation was conducted by Bradshaw and O'Connor [1], they observed that these thiol collector mixtures resulted in higher recoveries and grades than single thiol collectors. This was attributed to better surface coverage with mixed thiol collectors and therefore better

hydrophobization. Thiol collectors such as dithiophosphates (DTP) and xanthates are said to have an ability to increase hydrophobicity and are also classified as good froth stabilising agents [1–4]. These attributes of most thiol collectors result in recoveries of heterogeneous minerals such as base metal sulfides, and may also result in high solids recoveries of both fine and coarse interlocked particles owing to a possible increase in froth stability [2,4]. Roy et al. [5] concluded that the xanthates are able to adsorb selectively on fine particles with co-collectors such as dithiophosphates and hydroxamate playing an important role in adsorbing onto coarse particles. Also, McFadzean et al. [3] showed that collector mixtures had a beneficial effect in recovering heterogeneous ores such as PGMs, where many different valuable minerals were targeted by a specific collector within the collector mixture.

However, most of these studies were conducted in fresh or normal process water without considering variations in inorganic electrolytes resulting from onsite process water recirculation; the presence of electrolytes in process water may impact these reported beneficial effects of collector mixtures. Fundamental studies on water quality effects on flotation have shown that increases in the ionic strength of process water and specific inorganic electrolytes in process water can result in higher water and solids recoveries, and most importantly affecting mineral grades and recoveries [6–9]. Recently, for a study that considered a Cu-Ni-PGM ore, mixtures of isobutyl xanthate (SIBX) and sodium diethyl dithiophosphate (SEDTP) in increasing ionic strength of plant water, it was shown that an increase in the ionic strength of plant water increased water recoveries and therefore froth stability in parallel with SEDTP's froth stabilizing effect, thus suggesting an additive interaction on the froth stabilisation effect of increasing ionic strength and SEDTP ratio. The presence of cationic electrolytes such as Ca^{2+} and Mg^{2+} has been found to have an ability to enhance froth stability, these electrolytes are known to decrease repulsive forces between collector ions and mineral surface and thereby enhancing bubble particle attachment and mineral recoveries [10–14]. This may be resulting from the exothermic heat evolved when aqueous reagents are added; indicating that there is an interaction with negative thiol collector ions which causes differences in the dilution enthalpies because of the attraction which occurs between the ions and water [15]. However, other researchers have found that the collector adsorption may be hindered by metal ions which hydrolyse in alkaline conditions [16,17]. Literature also suggests that inorganic electrolytes can improve bubble-particle attachment efficiency through the compression of the electrical double layer which in turn causes a reduction of the electrostatic repulsion between particles and bubbles and therefore resulting in increased floatability [11]. It has been postulated that xanthate adsorbs onto the mineral surface via charge transfer between the collector and the mineral surface while dithiophosphates adsorb via the formation of metal thiolates on the mineral surface [18,19]. These adsorption reactions can therefore be affected by the presence of ions that are regarded as either structure makers or structure breakers [14].

There is, therefore, a growing need to understand whether the presence of common inorganic electrolytes in process water affect the behaviour of mixtures of thiol collectors during the flotation of a Cu-Ni-PGM ore. Therefore, this study investigates the effect of ion type on the behaviour and performance of thiol collector mixtures during the flotation of a sulfidic Cu-Ni-PGM ore using NaCl and CaCl_2 in the presence of SIBX (sodium isobutyl xanthate) and SEDTP (sodium di-ethyl dithiophosphate).

2. Materials and Methods

1 kg of a Merensky ore sample was milled at 66% solids in single salt solutions, in order to obtain a grind of 60% passing $75\mu\text{m}$, for 13.5 minutes as determined by the milling curve for each experimental run. The milled sample was transferred into a 3 L Barker flotation cell. The volume of the cell was made up to generate 35% solids using single salt solutions (for the solution being investigated). The cell was fitted with a variable speed drive and the pulp level was controlled manually. The impeller speed was set at 1200 rpm. An air flow rate of 7 L/min was maintained for all flotation experiments and a constant froth height of 2 cm was sustained throughout. The cell height was constantly corrected to 2 cm by the addition of the single salt solution being tested. Collectors were dosed into the mill and therefore had a conditioning time of 13.5 minutes. The depressant and

frother were dosed into the flotation cell and were allowed to condition for 2 minutes and 1 minute respectively. Table 1 shows the suite of reagents used. Concentrates were collected at 1, 3, 7 and 20 minutes respectively by scraping the froth into concentrate trays every 15 seconds. A feed sample was taken before flotation and tails sample after each flotation test. Feeds, concentrates and tails were filtered, dried in an oven and weighed before Cu, Ni, and S analyses.

A total collector dosage of 150 g/t of the selected thiol collector mixtures was used and these were corrected for their active content or purity. The mixtures were based on molar ratios as shown in Table 1. The characteristics of the chosen SIBX and SEDTP are shown in Table 2. 40 g/t of frother (Betachem modified DOW 200) was used throughout the flotation tests. The pH was kept constant at ~9.18. In order to induce the hydrophilicity of naturally floatable gangue such as talc, a polysaccharide depressant used throughout the test work was Depramin 267 (Modified CMC) at a dosage of 100 g/t. Reagents were supplied by Senmin and ore sample was obtained from the Bushveld complex in SA. Two types of single salts were tested, namely sodium chloride (NaCl) and calcium chloride (CaCl₂) and these were of analytical grade. Table 3 shows the chemistry of the single salt solutions tested throughout this study.

Table 1. A summary of experimental conditions.

Description	Condition
Single salt solution type	NaCl, ionic strength of 0.0213 M CaCl ₂ , ionic strength of 0.0213 M
Depressant type and dosage	CMC – Depramin 267 100 g/t
Collector and dosage	Fixed at 150 g/t SIBX: SEDTP Molar ratios (%): 100:0, 90:10, 50:50, 10:90, 0:100
Frother and dosage	Dow 200 at 40 g/t

Table 2. Characteristics of thiol collectors used in the experimental tests.

Collector Name	Abbreviation	Mr, g/mol	% Purity
Sodium Iso – butyl Xanthate	SIBX	200	97
Sodium di – ethyl dithiophosphate	SEDTP	208.21	45

Table 3. The concentration of ions presents in the prepared single salts solutions.

Water type	Ca ²⁺ (mg/L)	Mg ²⁺ (mg/L)	Na ⁺ (mg/L)	Cl ⁻ (mg/L)	SO ₄ ²⁺ (mg/L)	NO ₃ ⁻ (mg/L)	NO ₂ ⁻ (mg/L)	CO ₃ ²⁻ (mg/L)	TDS (mg/L)	IS [M]
NaCl	-	-	554	854	-	-	-	-	1408	0.0213
CaCl ₂	322	-	-	570	-	-	-	-	892	0.0213

3. Results and Discussion

Figure 1 shows the total cumulative solids and water recoveries for all conditions tested. It is evident that both solids and water recoveries generally increased with an increase in the molar ratio of SEDTP for both single salt solutions. A dramatic increase in both solids and water recoveries is seen with SEDTP ratios above or equal to 50% with 100% SEDTP pulling the most water and solids to the concentrate compared to 100% SIBX. It can also be seen that at 100% and 90% SIBX molar ratios, CaCl₂ resulted in similar solids and water recoveries compared to NaCl, while, CaCl₂ resulted in higher solids and water recoveries at higher SEDTP molar ratio ($\geq 50\%$) as compared to NaCl, except that at 100% SEDTP molar ratio, NaCl pulled more water compared to CaCl₂.

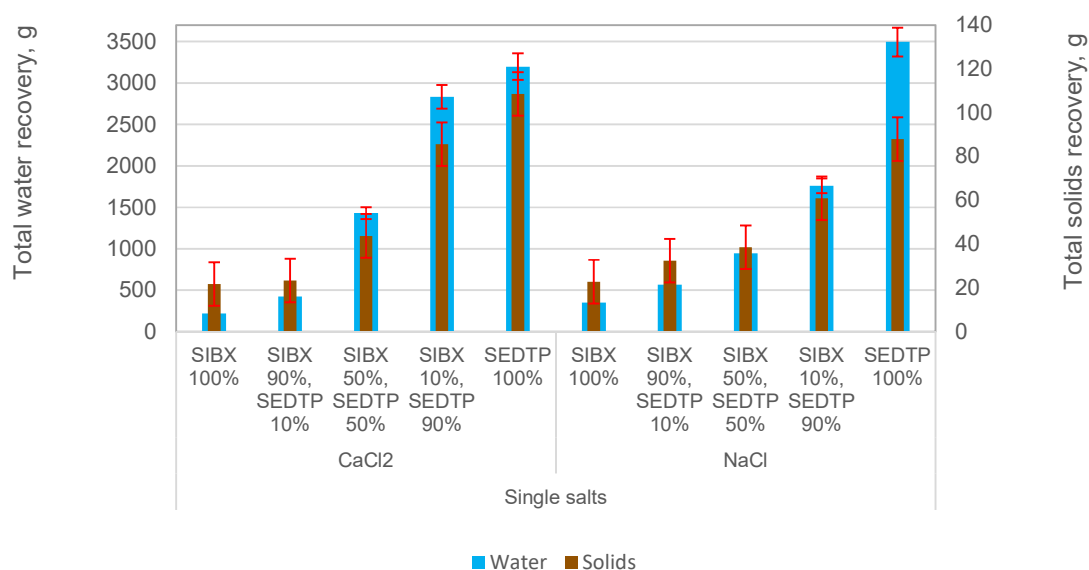


Figure 1. Total water and solids recoveries using solutions of CaCl₂ and NaCl at various thiol collector mixtures.

As illustrated in Figure 1, water and solids recoveries increased with an increase in SEDTP collector molar ratio for both single salts. This increase can be attributed to the frothing effect that dithiophosphate collectors are known to have [2,4]. It was also shown that, for 50% SEDTP and 90% SEDTP molar ratios, the divalent cation Ca²⁺ resulted in higher solids and water recoveries for all thiol collector mixtures when compared to the monovalent cation, Na⁺. This finding is in agreement with the froth stabilizing nature of polyvalent cations compared to monovalent cations which are less stabilizing on the froth [10–13,20,21]. Although it was expected that CaCl₂ would result in higher solids and water recoveries as compared to NaCl for all thiol mixtures, at 100% SIBX and 10% SEDTP molar ratios, NaCl resulted in similar water and solids recoveries.

This similarity in the behaviour of the thiol collector mixtures in two different salts could be due to a different mechanism than that of froth stabilization. As it is expected that Ca²⁺ would be more froth stabilizing and therefore would result in higher solids and water recoveries than Na⁺. The results on solids and water recoveries can be attributed to either the froth stabilising or destabilising action of the selected inorganic electrolytes in parallel with the collector combinations' action on hydrophobicity and froth stability. These mechanisms should be investigated further.

Figure 2 shows the total cumulative Cu recoveries and grades for all tested conditions. It is evident that there was an increase in Cu recoveries and decrease in Cu grades with an increase in SEDTP molar ratios in both single salts. For 100% SIBX, 90% SIBX:10% SEDTP, 50% SIBX:50% SEDTP and 10% SIBX:90% SEDTP, CaCl₂ resulted in higher Cu recoveries compared to NaCl. CaCl₂ and NaCl resulted in similar Cu recoveries when using 100% SEDTP. 100% SIBX molar ratio resulted in fairly similar Cu grades in both CaCl₂ and NaCl. However, at 90% SIBX:10% SEDTP, CaCl₂ resulted in

higher Cu grades than NaCl while flotation with 50%, 90% and 100% SEDTP molar ratios in NaCl also resulted in similar Cu grades to those obtained in CaCl₂.

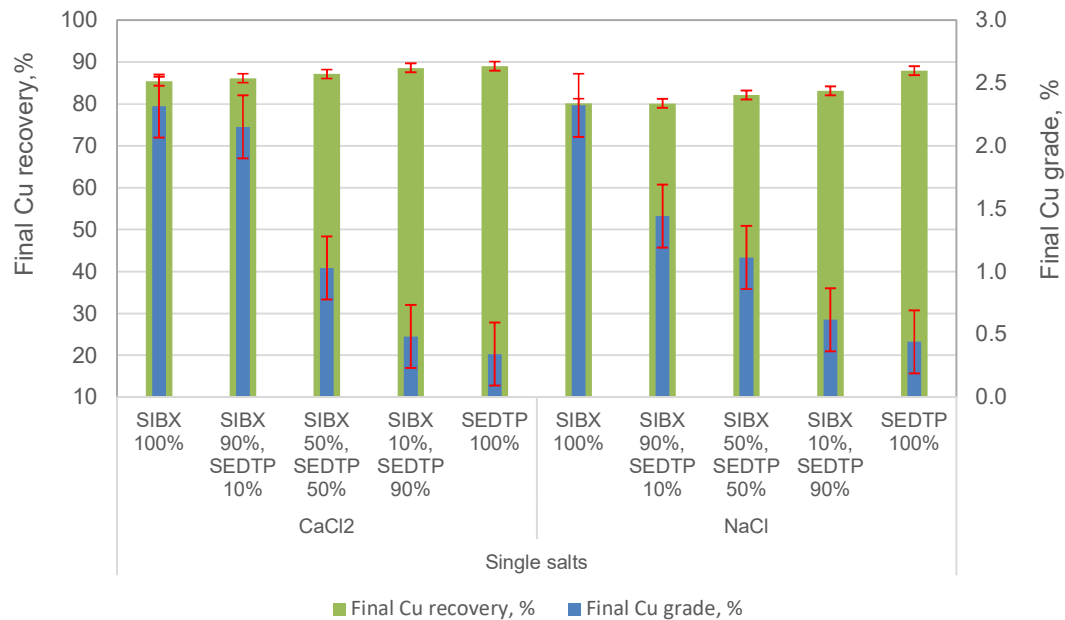


Figure 2. Final Cu recoveries and grades using solutions of CaCl₂ and NaCl at various thiol collector mixtures.

Figure 3 shows the final Ni recoveries and grades for all tested conditions. It can be seen that increases in SEDTP molar ratios resulted in a decrease in Ni grades in both CaCl₂ and NaCl. For each SIBX:SEDTP molar ratio, Ni grades in CaCl₂ were similar to those obtained in NaCl except for the 90% SIBX:10% SEDTP as the use of CaCl₂ resulted in higher Ni grades than NaCl. In CaCl₂, an increase in SEDTP results in a slight increase in Ni recoveries. However, in NaCl, a slight decrease in Ni recoveries is seen with increasing SEDTP molar ratio; the lowest recovery is at 100% SEDTP molar ratio and is similar to the Ni recovery with 100% SIBX in CaCl₂. Therefore, in CaCl₂, the highest Ni recovery is obtained with 100% SEDTP and this is fairly comparable with the highest Ni recovery in NaCl which was obtained at 100% SIBX.

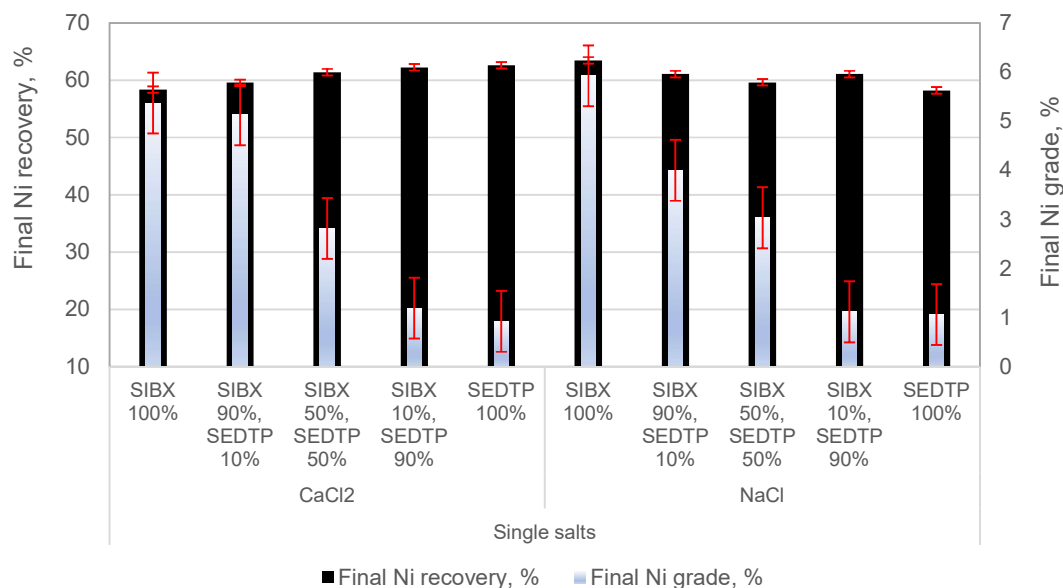


Figure 3. Final Ni recoveries and grades using solutions of CaCl₂ and NaCl at all thiol collector mixtures.

Figure 2 and 3 showed that Cu recoveries were increasing with an increase in SEDTP molar ratio for both single salts. It was also shown that Ni recoveries followed a similar trend to that of Cu recoveries in CaCl₂. A trend of a decrease in Ni recoveries with increasing SEDTP was seen in NaCl. These results were in line with the observations on solids and water recoveries and in agreement with reported improvements in mineral recoveries when using thiol collector mixtures [2]. Cu and Ni grades were decreasing with an increased SEDTP molar ratio in solutions of CaCl₂ and NaCl. This can be attributed to an enhancement in froth stability resulting in more gangue being pulled to the concentrate [22,23]. It was also shown that the divalent cation, Ca²⁺ resulted in higher Cu recoveries at all collector mixtures as compared to the monovalent cation, Na⁺. This illustrates that divalent cations can enhance hydrophobicity of thiol collectors better than monovalent cations as indicated by high Cu and Ni recoveries.

The presence of divalent cation, Ca²⁺, resulted in higher Cu grades at higher SIBX molar ratios compared to Na⁺, which resulted in higher Cu and Ni grades at higher SEDTP molar ratios than Ca²⁺ ion. These results could indicate that the presence of these inorganic electrolytes influences the mineral surface chemistry and floatability differently. The increase in Ni recoveries with low Ni grades in the presence of the divalent cation, Ca²⁺, shown in Figure 3 could very well emanate from how Ca²⁺ interacts and impact the surface chemistry of pentlandite particles. This could have resulted in nickel middling's being pulled into the concentrate and also increasing the froth stability at higher SEDTP molar ratios of 90 and 100% as shown in Figure 3. These findings are in agreement with Wiese et al. [24,25] who showed that increasing froth stability results in an increase in mineral recoveries but also with a decrease in mineral grades. It is also illustrated that Ca²⁺ resulted in higher Cu and Ni recoveries and lower mineral grades at mixed thiol collector molar ratio of 50:50 as compared to Na⁺ shown in Figure 2 and 3.

A fundamental study by Wang et al. [26] showed that divalent cations such as Mg²⁺ and Ca²⁺ are more effective than monovalent cations such as Na⁺ and K⁺ in reducing the viscosity and therefore increasing copper flotation due to a stronger compression of the electrical double layer. The higher mineral recoveries and lower mineral grades at molar ratios of 50% SIBX: 50% SEDTP and 10% SIBX: 90% SEDTP in the presence of Ca²⁺ shown in Figure 2-3 suggest that the presence of ions in process water could enhance collector selectivity. This is in agreement with McFadzean et al. [3] who showed that collector mixtures were able to recover heterogeneous ores such as PGMs, where many different

valuable minerals could be targeted by a specific collector in the mixture. They also found that some collectors effect an optimum hydrophobicity on the particles, making them good froth stabilisers which is in line with the lower mineral grades and higher recoveries. The decrease in mineral grades with an increase in SEDTP molar ratio suggests that unwanted gangue minerals could have been pulled to the concentrates leading to higher solids recoveries. Further investigative work needs to be conducted in order to understand the underlying mechanisms of inorganics electrolytes on the adsorption of thiol collector mixtures.

5. Conclusions

Water and solids recoveries increased with an increase in SEDTP collector molar ratio for both single salts. This increase was attributed to the frothing effect that dithiophosphate collectors are known to have. Ca^{2+} resulted in higher solids and water recoveries in thiol collector mixtures containing more than 50% SEDTP molar ratio compared to Na^+ . However, Na^+ resulted in higher water at molar ratio of 100% SEDTP. This study showed an increase in Cu recoveries and a decrease in Cu grades in increasing SEDTP molar ratios in both single salts. The divalent cation, Ca^{2+} resulted in higher Cu and Ni recoveries at all collector mixtures than the monovalent cation, Na^+ owing to the froth stabilizing nature of Ca^{2+} and its possible effects on the hydrophobization of sulfide minerals. In CaCl_2 , the highest Ni recovery was obtained using 100% SEDTP molar ratio and this was fairly comparable with the highest Ni recovery in NaCl which was obtained at 100% SIBX molar ratio.

Ca^{2+} resulted in lower mineral grades at higher SEDTP molar ratios compared to Na^+ . Ca^{2+} may have aided thiol collector selectivity at high SIBX molar ratio but selectivity reduced with the increase in SEDTP molar ratio. This should however be a topic for further investigative test work.

The decrease in mineral grades with an increase in SEDTP molar ratio suggests that unwanted gangue minerals was pulled to the concentrate leading to higher solids recoveries. Further investigative work, however, needs to be conducted in order to understand the underlying mechanisms of inorganics electrolytes on the adsorption of thiol collector mixtures.

The results of this study have shown that specific ions in process water may affect the behaviour of mixtures of thiol collectors differently depending on the chemistry of the ions and their interfacial effects on the pulp phase.

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