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Minimisation of Cobalt Coprecipitation During the Removal of Iron, Aluminium, and Manganese in the Cobalt Circuit (FAM) by Oxidising the Bath with Hydrogen Peroxide, Case Study: The METALKOL Hydrometallurgical Plant in the Democratic Republic of Congo

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Abstract: The removal of impurities from cobalt-bearing solutions is an important step in the processing of cobalt to obtain a high-quality product. The main problem at this stage is the loss of cobalt that occurs through coprecipitation. A sample of cobalt-rich solution was taken from the feed to the Iron, Aluminium and Manganese (IAM) circuit at the Metalkol hydrometallurgical plant in the Democratic Republic of Congo. The pH and potential were the parameters studied to assess their effect on the overall process. After contact with 20 ml of a 30 g/l concentrated hydrogen peroxide solution and 27.3 g of calcium carbonate, the results showed a significant reduction in cobalt loss and a high level of impurity removal, with the exception of manganese, which could not be removed.

Keywords*: Coprecipitation, Hydrogen Peroxide, Oxidation, Potential, Ph, Calcium Carbonate, Impurities***.**

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I. INTRODUCTION

Although less well known to the general public than other metals such as copper or iron, cobalt plays a crucial role in many industries. Since 2016, demand for cobalt has accelerated thanks to the rechargeable battery industry, growing demand for superalloys, the arms industry and promising sectors such as hybrid and electric cars (Rumbu, 2018; Darton et al., 2020). Its properties, such as corrosion resistance, hardness, and magnetic properties, make it an indispensable element in the manufacture of high-tech products, including lithium-ion batteries, metal alloys, the chemical industry, the medical industry, and pigments (U.S. Geological Survey, 2022; Shedd, 2017).

Cobalt production is generally secondary to that of other metals such as copper, nickel, zinc, iron and silver. This is because cobalt is often only present in small proportions in polymetallic ores, making its metallurgical extraction complex (Rumbu, 2018; Mudd & Jowitt, 2018). This complexity stems

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from the fact that hydrometallurgical or pyrometallurgical processes must be carefully optimised to separate cobalt from other associated metals, minimising losses and coprecipitation.

The efficient recovery of cobalt from aqueous solutions is therefore a major challenge for the metallurgical industry. One of the main challenges lies in the selectivity of processes for removing impurities such as iron, aluminium, manganese and copper, which tend to coprecipitate with cobalt (Zhao et al., 2019). The use of hydrogen peroxide as an oxidising agent appears to be a promising approach for improving the selectivity of these operations (Rumbu, 2018; Kazmierczak & Vicot, 2014). This study aims to deepen our understanding of the mechanisms of cobalt coprecipitation during the oxidation and precipitation of impurities, as well as to evaluate the effectiveness of hydrogen peroxide in minimising this phenomenon.

Table 1 Results of the Analysis of a Low-Grade Raffinate Sample

Eléments	**Acidité**	**Cu**	**Co**	**Fe**	**Mn**	**Al**	**Cd**	**Zn**	**U**	**Mg**	**Volume**	**pH**
Units	g/l	g/l	g/l	g/l	g/l	[ppm]	[ppm]	[ppm]	[ppm]	[g/l]	[mL]	
Raffinate	8.07	0,07	3,29	0,64	0,78	304,13	2,29	364,04	15,52	4,17	1000	1,55

The precipitation reagent used is calcium carbonate CaCO3. Its chemical characterisation is as follows

Table 2 Chemical Characterisation of the Calcium Carbonate Sample

Eléments	**Cu**	**Co**	**Fe**	**Mn**	**Al**	**Cd**	**Zn**	**U**	**Mg**
Unités	[%]	[%]	[%]	[%]	[%]	[ppm]	[ppm]	[ppm]	[%]
CaCO3	0,002	0,004	0,849	0,042	0,833	0,91	16	8	1,184

Hydrogen peroxide has long been considered unstable, due to numerous attempts to separate it from water. This instability is attributed to the presence of transition metal impurities in solution, even at very low concentrations, which catalyse its decomposition (Kazmierczak & Vicot, 2014; Jones, 2015). It is a chemical compound consisting of two hydrogen atoms and two oxygen atoms (H_2O_2), with a molar mass of 34.0147 g/mol. Hydrogen peroxide is a colourless liquid, slightly more viscous than water, with a pungent odour that increases with concentration. It decomposes through an exothermic dismutation reaction into water and oxygen, in proportions that depend on the presence of impurities and stabilisers (Kazmierczak & Vicot, 2014; Bockris & Reddy, 2000).

II. MATERIALS AND METHODS

➢ *Sampling and Preparation*

60 litres of solution from solvent extraction (low-grade raffinate) were collected to facilitate the work. This solution, rich in cobalt, nevertheless contains some significant impurities such as copper, zinc, iron, manganese, cadmium, aluminium, etc.

The table below describes the proportions of the elements dissolved in the low-grade raffinate after analysis by atomic absorption and inductively coupled plasma atomic emission spectrometry (ICP).

➢ *Tests*

A series of oxidation tests enabled us to find the ideal precipitation zone for impurities without reaching the cobalt co-precipitation zone. The results showed us that at a volume of 20 ml of hydrogen peroxide, the potential is 470.2 mV.

Table 3 Results on Filtrates from Tests Without H_2O_2 Addition

Elements	***$CaCO_3$***	**Cu**	**Co**	**Fe**	**Mn**	**Al**	**Cd**	**Zn**	**U**	**Mg**	**Volume**	**pH**
Units	[g]	[g]	[g]	[g]	[g]	[g/l]	[ppm]	[ppm]	[ppm]	[g/l]	[ml]	
Test 1	27,3	0,00	3,03	0,00	0,77	0,00	1,70	161,80	0,40	3,04	1000	5,90
Test 2	27,3	0,00	3,00	0,00	0,75	0,00	1,60	160,70	0,40	2,98	1000	5,82
Test 3	34,3	0,00	3,00	0,00	0,76	0,00	1,50	163,80	0,04	3,10	1000	5,59
Test 4	37,8	0,00	2,99	0,00	0,77	0,00	1,40	154,76	0,30	3,19	1000	5,57

Table 4 Results on Filtrates from Tests with H2O2 Added

Element	**H_2O_2**	***$CaCO_3$***	**Cu**	**Co**	**Fe**	**Mn**	**Al**	**Cd**	**Zn**	**U**	**Mg**	**pH**	**filtrat**
Units	ml	*g*	g/l	g/l	g/l	g/l	[g/l]	[ppm]	[ppm]	[ppm]	[g/l]		ml
Test 1	20,00	27,3	0,00	3,16	0,00	0,72	0,81	2,03	149,86	0,28	2,86	5,53	970
Test 2	40,00	27,3	0,00	3,02	0,00	0,70	0,79	1,91	144,50	0,28	2,74	5,31	920
Test 3	45,00	34,3	0,00	2,94	0,00	0,71	0,78	1,84	138,62	0,25	2,74	5,27	880
Test 4	50,00	37,8	0,00	2,99	0,00	0,70	0,78	1,62	138,02	0,19	2,65	5,16	850
Test 5	55,00	48,8	0,00	2,98	0,00	0,63	0,78	1,61	136,53	0,10	2,23	5,12	800

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- *The Conditions Under Which this Test was Carried are:*

✓ Volume of the solution: 1000 ml;
✓ Working temperature: 30°C ;
✓ Reaction time : 6 hours.

➢ *Precipitation Yields of Impurities*

$$Rdt\ de\ precipitation(\%) = \frac{Initial\ concentration - final\ concentration}{Initial\ concentration} \times 100 \quad (1)$$

Table 5 Precipitation Yields of Impurities and Co-Precipitation of Cobalt Without the Influence of Hydrogen Peroxide

Elements	Cu	Co	Fe	Mn	Al	Cd	Zn	U	Mg
Test 1	100,0%	16,4%	100,0%	13,3%	100,0%	43,2%	29,6%	97,6%	33,3%
Test 2	100,0%	19,1%	100,0%	15,5%	100,0%	49,5%	31,3%	98,1%	34,6%
Test 3	100,0%	19,1%	100,0%	14,0%	100,0%	52,7%	30,0%	99,8%	31,8%
Test 4	100,0%	19,4%	100,0%	12,7%	100,0%	55,8%	33,8%	98,5%	30,0%

Deux types d'essais ont été effectués dans le cadre de cette recherche : un lot d'essais d'orientation dans les conditions de l'usine et un lot d'essais avec ajout du peroxyde d'hydrogène.

Where :

- Initial concentration is the concentration of impurities in the solution before precipitation;
- Final concentration is the concentration of impurities in the solution after precipitation.

Table 6 Precipitation Yields of Impurities and Cobalt Coprecipitation as a Function of Hydrogen Peroxide Dose

Eléments	H_2O_2 (ml)	Cu	Co	Fe	Mn	Al	Cd	Zn	U	Mg
Test 1	20,00	100,00%	6,83%	98,48%	10,46%	99,74%	14,01%	60,07%	98,25%	33,47%
Test 2	40,00	100,00%	10,96%	98,48%	12,95%	99,75%	19,10%	61,50%	98,25%	36,26%
Test 3	45,00	100,00%	13,32%	98,48%	11,71%	99,75%	22,06%	63,06%	98,44%	36,26%
Test 4	50,00	100,00%	11,84%	98,48%	12,95%	99,75%	31,38%	63,22%	98,81%	38,36%
Test 5	55,00	100,00%	12,14%	98,48%	21,65%	99,75%	31,80%	63,62%	99,38%	48,13%

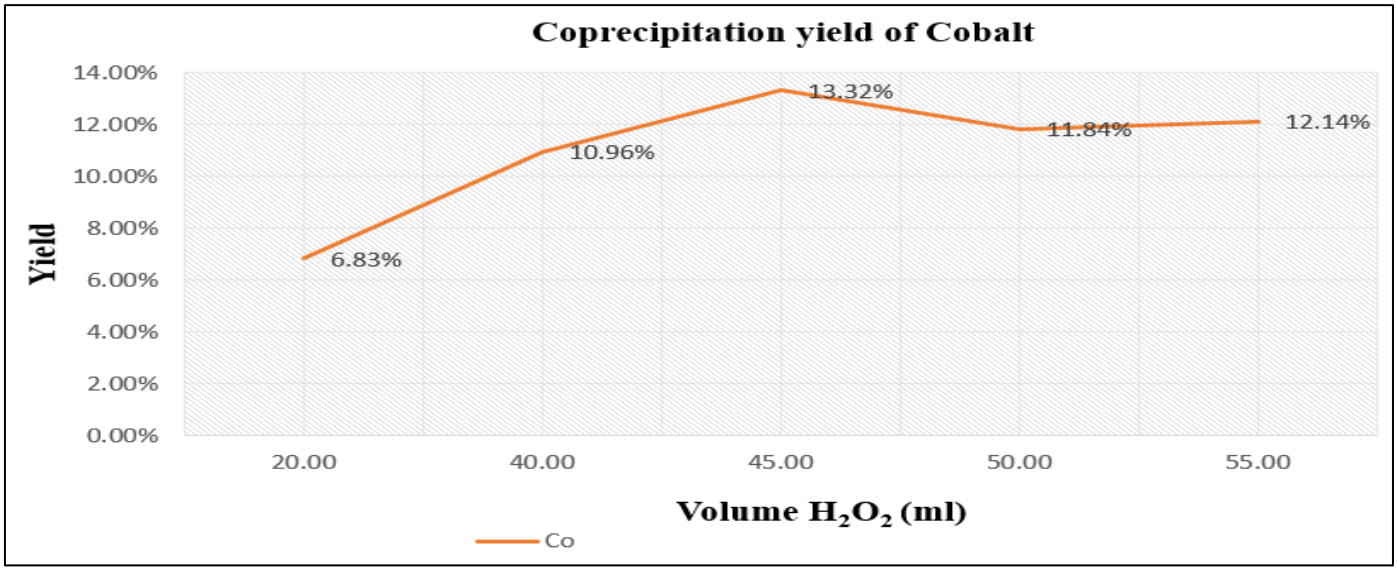

Fig 1 Cobalt Coprecipitation Yield as a Function of Hydrogen Peroxide Dose

By analysing Figure 1 and Table 6, it is clear that cobalt co-precipitation increases with the dose of H_2O_2. The higher the H2O2 dose, the more cobalt is also carried away, and at a dose of 20 ml of H2O2, less cobalt is carried away. After adding the peroxide, the precipitating agent, CaCO3, is added until the pH reaches 5.53 according to the plant conditions

At this pH, the apparent potential was maintained at 470.2 mV. According to the Pourbaix diagram below, cobalt remains in the form of Co^{2+} under these conditions and cannot precipitate. Otherwise, the existence of other species and other uncontrolled chemical reactions may give rise to certain poorly

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soluble cobalt compounds, which explains the small amount of cobalt carried over in the precipitates from FAM.

III. RESULTS AND DISCUSSION

➢ *The Parameters Monitored Were pH and Potential*

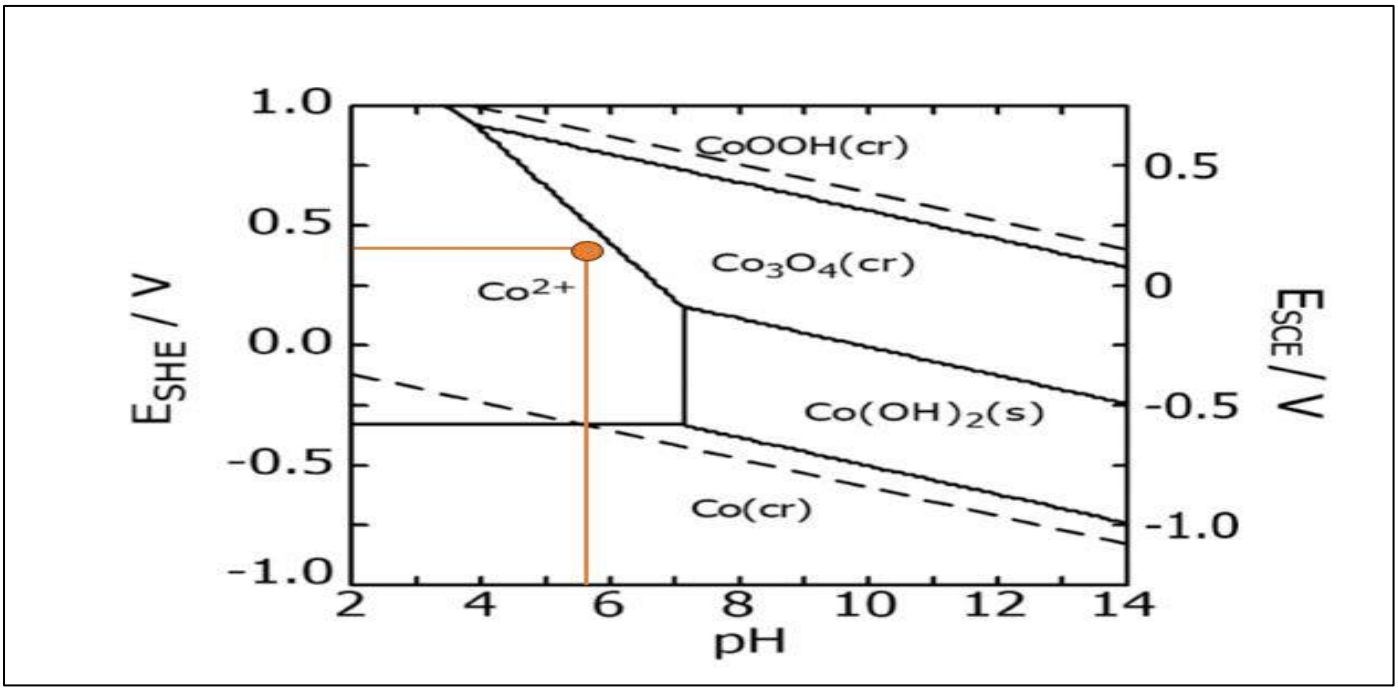

Fig 2 Potential-pH Diagram for Cobalt at pH=5.53 and E=470.2 mV

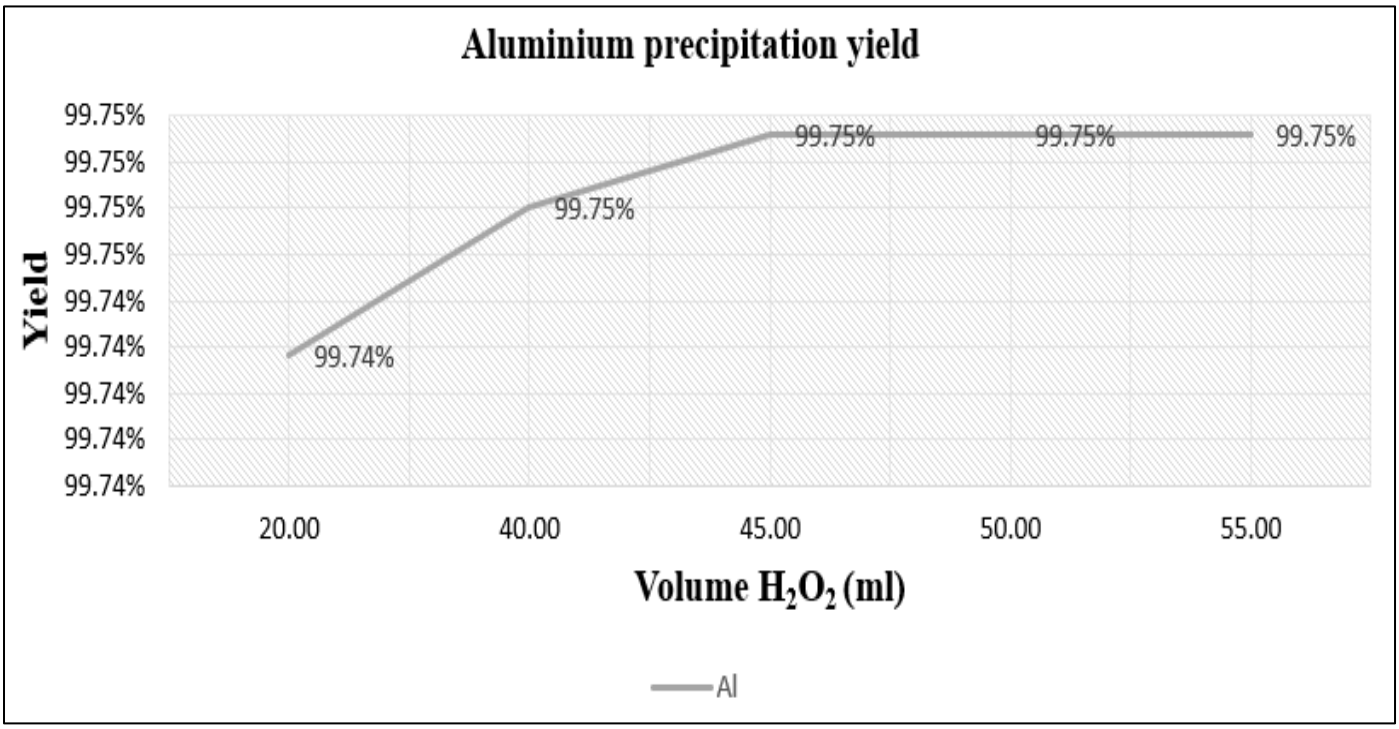

Fig 3 Aluminium Precipitation Yield as a Function of Hydrogen Peroxide Dose

Analysing Figure 2 and Tables 4 and 6, the results show that aluminium precipitation increases with the dose of H2O2; the higher the dose of H2O2, the more aluminium is also carried away. At pH 5.53 and a potential of 470.2 mV (relative to the standard hydrogen electrode, SHE), aluminium only precipitates as Al $(OH)_3$ hydroxide for the following reasons:

- At this pH and potential, aluminium is more stable in the form of hydroxide than in the form of Al^{3+} ions or metal. The Pourbaix diagram shows the stability ranges of the different species. At pH 5.53, Al $(OH)_3$ hydroxide is stable due to the pH and potential conditions. Below this pH or above this potential, other species such as Al^{3+} or Al metal may be more stable.

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- Al $(OH)_3$ hydroxide forms when the concentration of Al^{3+} ions exceeds a certain value, which is reached when the solubility product of the hydroxide is exceeded. At pH 5.53, the concentration of OH^- ions is sufficient to allow the formation of Al $(OH)_3$ precipitates.

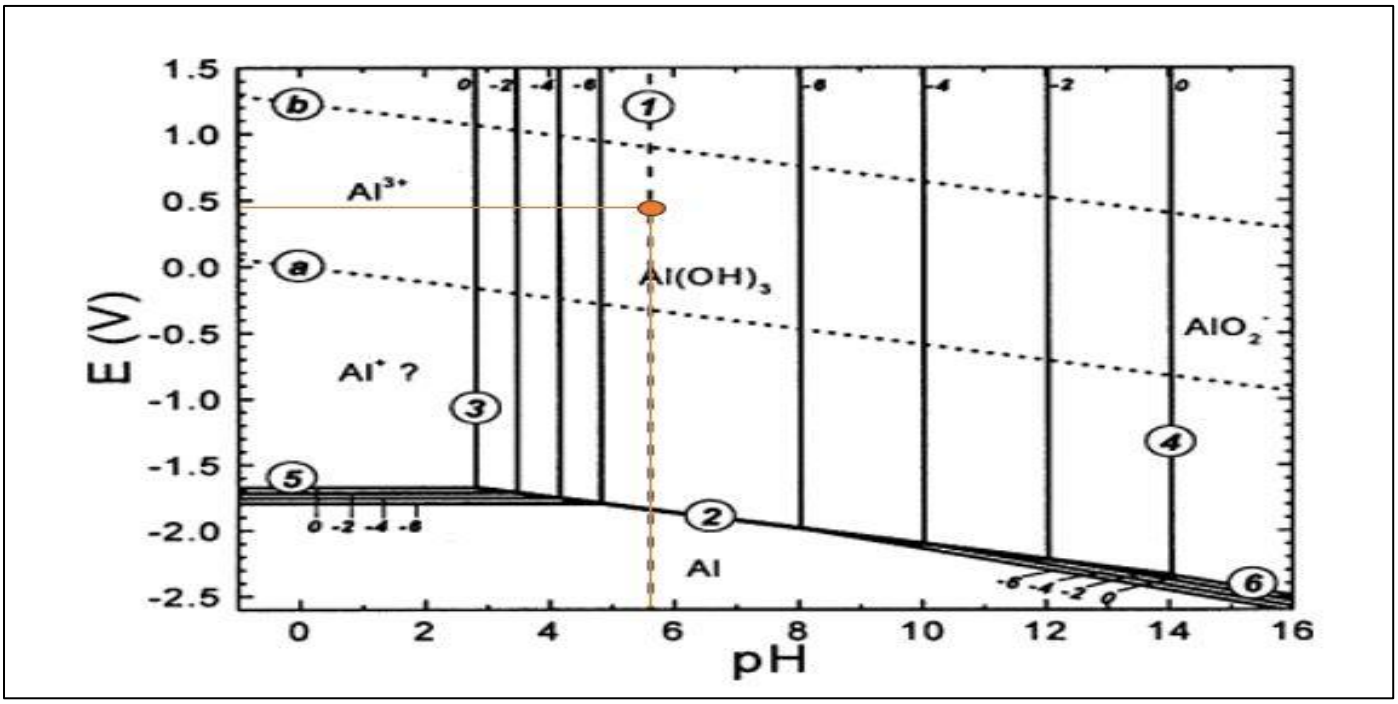

Fig 4 Potential-pH Diagram for Aluminium at pH=5.53 and E=470.2 mV

- The Pourbaix diagram for aluminium shows the stability zones for different species such as metallic aluminium, Al^{3+} ions in solution, and Al $(OH)_3$ hydroxide. The curves in the diagram indicate the boundaries between these zones. At pH 5.53 and potential 470.2 mV, the zone where Al $(OH)_3$ is stable is where the pH and potential are compatible with the formation of this hydroxide and not other forms.

At this pH of 5.53 and potential of 470.2 mV, conditions are such that aluminium hydroxide Al $(OH)_3$ and carbonate $Al_2(CO_3)_3$ are the most stable forms. These compounds are stable in this specific region of the Pourbaix diagram, explaining why aluminium precipitates in these forms. Other forms such as Al^{3+} or Al metal are not favoured under these specific conditions.

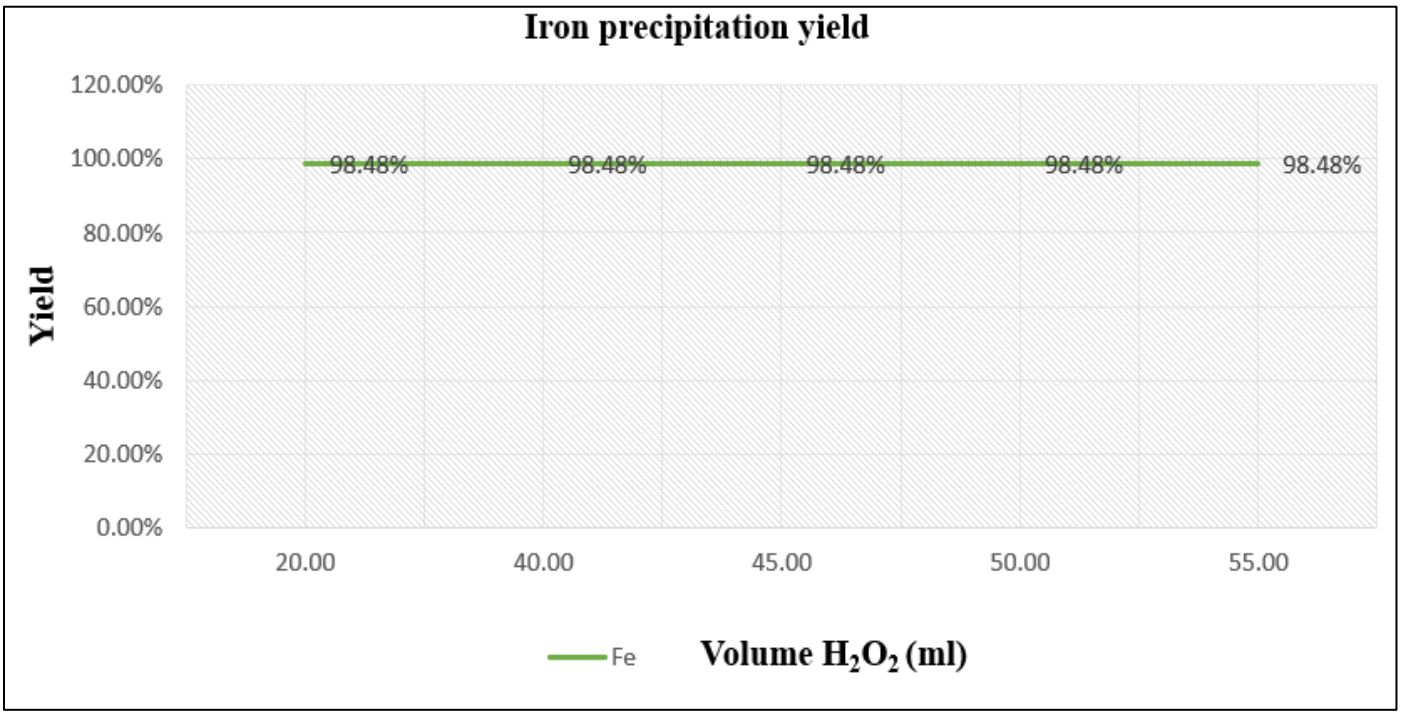

Fig 5 Iron Precipitation Yield as a Function of Hydrogen Peroxide Dosage

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By analysing Figure 5 and Tables 4 and 6, the results show that iron precipitation increases with the dose of H_2O_2, i.e. the higher the dose of H_2O_2, the more iron is carried away. The Pourbaix diagram below shows that at this pH of 5.53, which is relatively acidic, and with this potential of 470.2 mV, Fe^{3+} is stable in the form of ions in solution at higher potential values. However, for $Fe(OH)_3$ to precipitate, the pH must be high enough to promote the formation of iron hydroxide. If the potential is low enough for the iron to be in the form of Fe^{3+} but the pH is slightly acidic, it is possible that iron hydroxide precipitate will begin to form if the OH^- concentration is high enough.

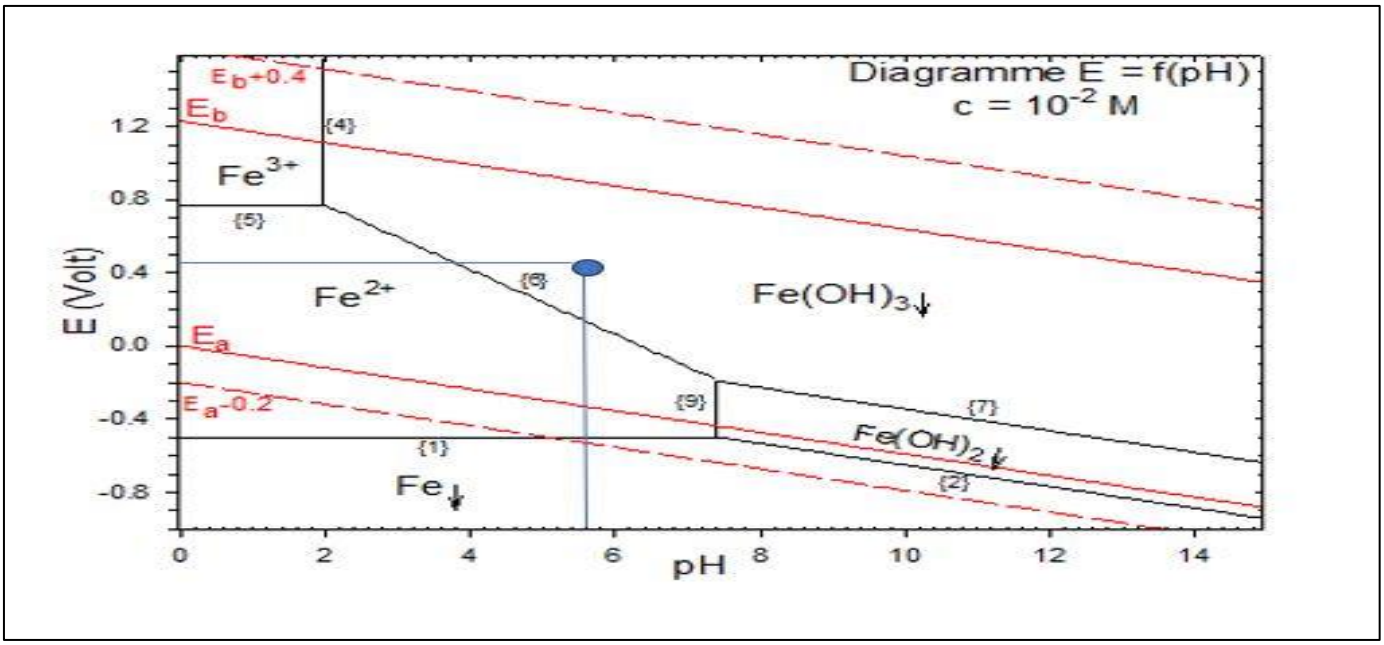

Fig 6 Potential-pH Diagram for Iron at pH=5.53 and E=470.2

Iron (III) carbonate precipitate, often represented by $Fe_2(CO_3)_3$, forms when iron is in solution in the form of Fe^{3+} ions and in the presence of carbonate (CO_3^{2-}). At a pH of 5.53, the presence of CO_3^{2-} is necessary for the formation of iron carbonate. Carbon in the form of CO_2 dissolves in water to form bicarbonate (HCO_3^-) and carbonate (CO_3^{2-}) ions. At a pH of 5.53, the system is slightly acidic, which means that the concentration of CO_3^{2-} is relatively low compared to a higher pH. If the potential is low, there may be greater stability of iron in the form of Fe^{2+} rather than Fe^{3+}, and the formation of $Fe_2(CO_3)_3$ would be possible if CO_3^{2-} is sufficiently present.

At a pH of 5.53 and a potential of 470.2 mV, conditions could favour the formation of iron hydroxide $Fe(OH)_3$, if the pH is high enough to provide a sufficient concentration of OH^- ions, even if the potential is high enough to keep Fe^{3+} in solution; whereas the formation of iron carbonate $Fe_2(CO_3)_3$ will depend on the concentration of CO_3^{2-}, which is also sufficiently present. This indicates that the iron has indeed precipitated in the form of carbonate.

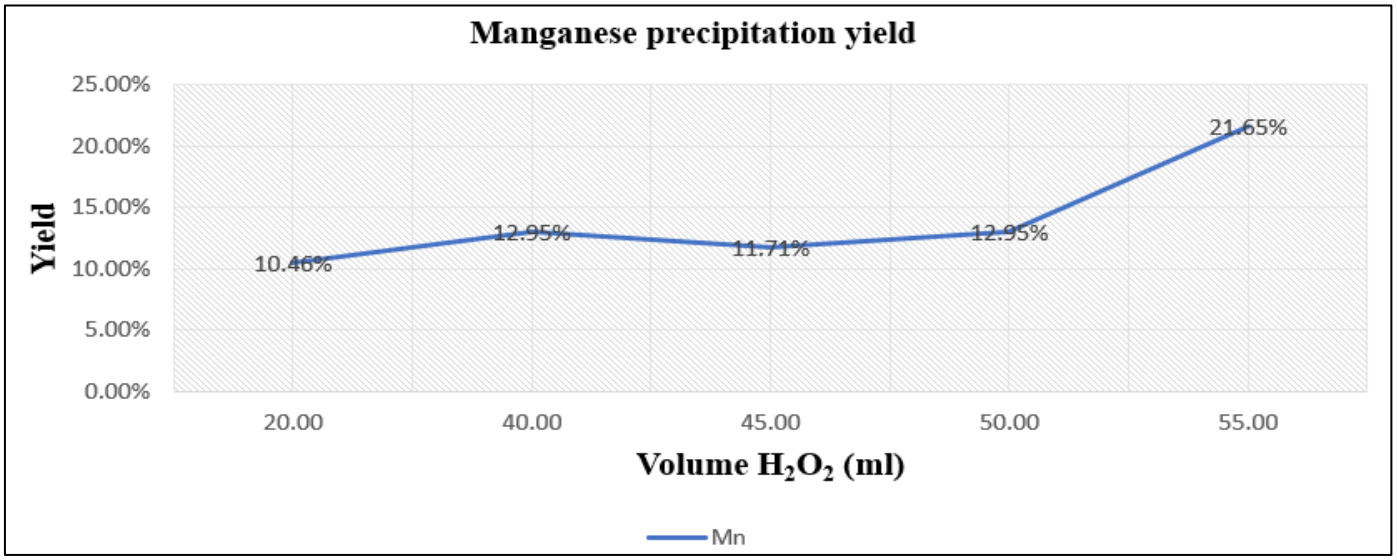

Fig 7 Manganese Precipitation Yield as a Function of Hydrogen Peroxide Dose

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By analysing Figure 7 and Tables 4 and 6, the results show that manganese precipitation is not as significant at any dose of H_2O_2, regardless of the increase in the dose of H_2O_2. We found that the concentration of manganese in the filtrate did not change significantly after the FAM operation.

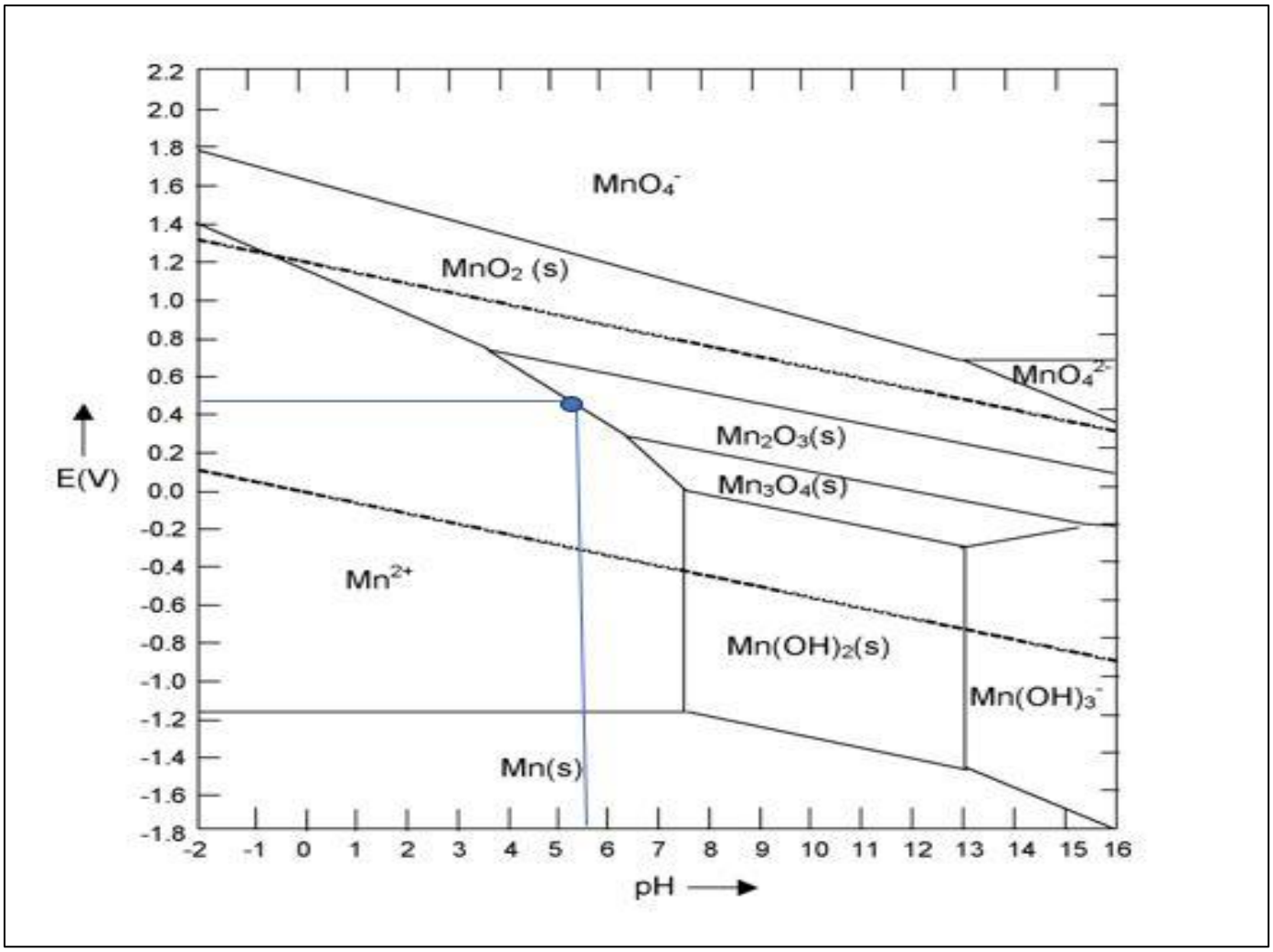

Fig 8 Potential-pH Diagram for Manganese at pH=5.53 and E=470.2

The Pourbaix diagram for manganese shows the stability zones of the different manganese species as a function of pH and redox potential. Here are the main manganese species and their stability conditions:

- Mn^{2+} (manganese(II) ion) ;
- MnO_2 (manganese dioxide) ;
- MnO_4^- (permanganate ion) ;

Manganese does not precipitate as carbonate ($MnCO_3$) at pH 5.53 and a potential of 470.2 mV due to ion concentration and potential conditions that favour oxidised forms of manganese and insufficient carbonate concentrations to allow $MnCO_3$ precipitation. It is true that 20 ml of $CaCO_3$ is not sufficient to bring manganese into a zone where it would precipitate sufficiently in carbonate form.

➢ *$MnCO_3$ (Manganese Carbonate).*

With such a high potential (470.2 mV), manganese is more likely to be in oxidised form (MnO_2 or MnO_4^-) than in Mn^{2+} form. The high potential favours the presence of Mn^{3+} or Mn^{4+} in solution rather than Mn^{2+}. At a pH of 5.53, carbonate ions (CO_3^{2-}) are present in low concentrations, as bicarbonate (HCO_3^-) is the predominant form in a slightly acidic solution. The low concentration of CO_3^{2-} reduces the possibility of $MnCO_3$ formation. Since this research focuses on the removal of cobalt in FAM precipitates, analysis of Figure 5 and Tables 4 and 6 shows that cobalt was minimised at a dose of 20 ml of hydrogen peroxide. At this same dose, iron and aluminium were significantly eliminated, while manganese did not follow suit. The other doses of peroxide, which significantly reduced cobalt, are not taken into account, although impurities such as copper, iron, zinc, uranium, magnesium and aluminium were significantly eliminated.

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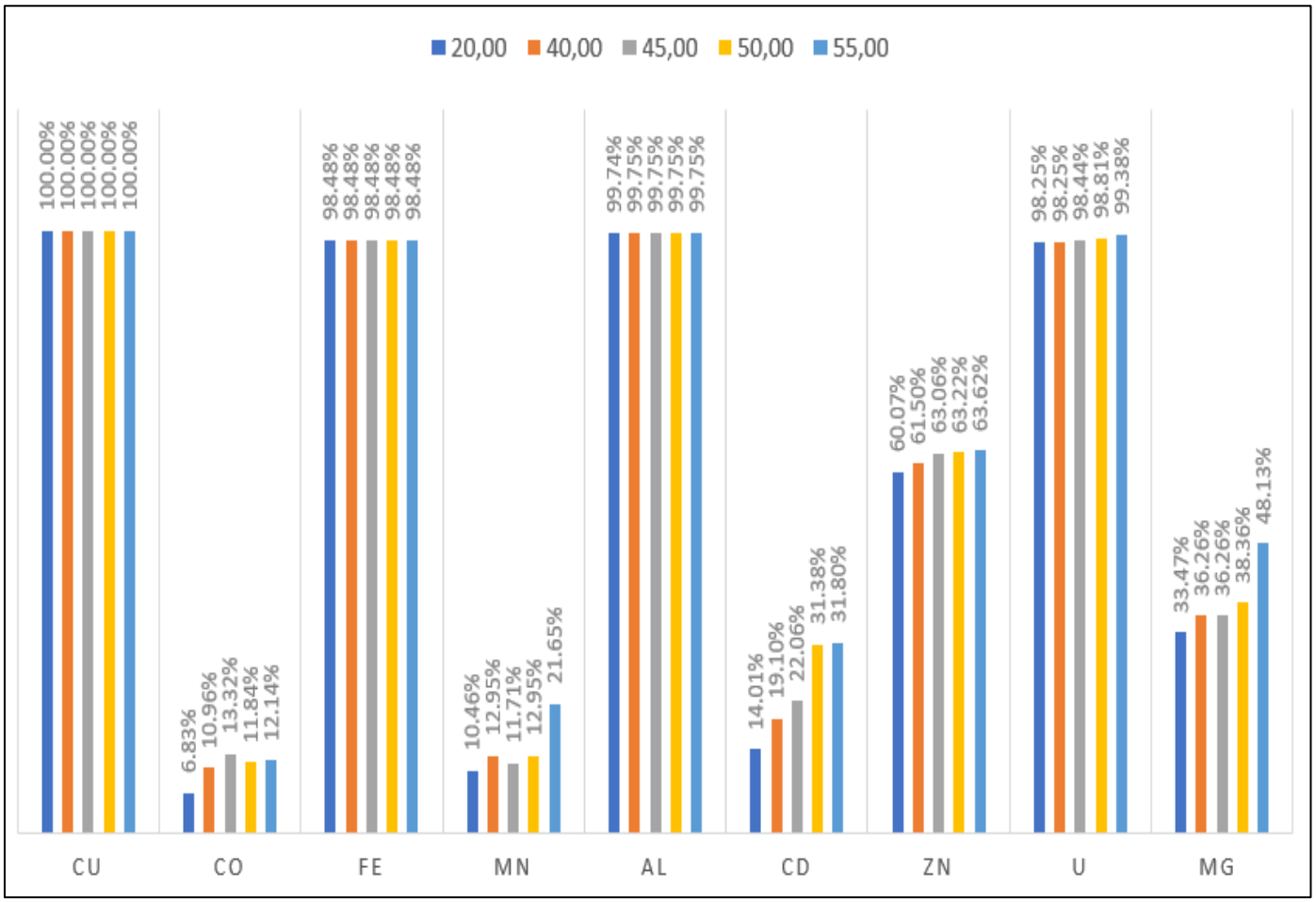

Fig 9 Coprecipitation Yields of Cobalt and Precipitation of All Impurities as a Function of Hydrogen Peroxide Dose

IV. CONCLUSION

The study conducted as part of this research aims to minimise cobalt coprecipitation in the FAM process at the Metalkol plant's cobalt circuit. The cobalt circuit is fed by a solution with the following characteristics: Acidity 8.07 g/l; pH 1.55; concentrations of Cu, Co, Fe, Mn, Al, Cd, Zn, U and Mg are respectively 0.007 g/l; 3.29 g/l; 0.64 g/l; 0.78 g/l; 304.13 ppm; 2.29 ppm; 364.04 ppm; 15.52 ppm and 4.17 g/l in a volume of 1000 ml. A series of oxidation tests were carried out to find the ideal zone for precipitating impurities without reaching the cobalt coprecipitation zone. The results showed that at a volume of 20 ml of hydrogen peroxide, the potential is 470.2 mV.

After precipitation with calcium carbonate as the precipitating agent, for 27.3 g of CaCO3, the pH at the end of the reaction was 5.53, which proves, according to the various potential-pH diagrams, that the cobalt remained in its Co^{2+} form and did not coprecipitate under these conditions, but that all impurities except manganese were completely eliminated.

The results after chemical analysis of the filtrate are as follows: concentrations of Cu, Co, Fe, Mn, Al, Cd, Zn, U and Mg are respectively 0.00 g/l; 3.16 g/l; 0.00 g/l; 0.72 g/l; 0.81 g/l; 2.03 ppm; 149.86 ppm; 0.28 ppm and 2.86 g/l. with impurity precipitation yields and coprecipitation of cobalt in the order of Cu 100%; Co 6.83%; Fe 98.48%; Mn 10.4%; Al 99.74%; Cd 14.01%; Zn 60.07%; U 98.25% and Mg 33.47%. Referring to Tables 5 and 6, a minimisation of cobalt co-precipitation of around 9.57% was observed.

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