Peer review history for:

El-Bacha R, Salhi A, Abderrafia H, Rabi S. Optimising water use in copper flotation with design of experiments and machine learning. S Afr J Sci. 2025;121(3/4), Art. #16239. https://doi.org/10.17159/sajs.2025/16239

HOW TO CITE:
Optimising water use in copper flotation with design of experiments and machine learning [peer review history]. S Afr J Sci. 2025;121(3/4), Art. #16239. https://doi.org/10.17159/sajs.2025/16239/peerreview

The original manuscript for review is appended below for reference.

Reviewer 2: Round 1
Date completed: 03 March 2024
Recommendation: Accept / Revisions required / Resubmit for review / Resubmit elsewhere / **Decline** / See comments
Conflicts of interest: None

Does the manuscript fall within the scope of SAJS?
Yes/No
Is the manuscript written in a style suitable for a non-specialist and is it of wider interest than to specialists alone?
Yes/No
Does the manuscript contain sufficient novel and significant information to justify publication?
Yes/**No**
Do the Title and Abstract clearly and accurately reflect the content of the manuscript?
Yes/No
Is the research problem significant and concisely stated?
Yes/No
Are the methods described comprehensively?
Yes/**No**
Is the statistical treatment appropriate?
Yes/**No**/Not applicable/Not qualified to judge
Are the interpretations and conclusions justified by the research results?
Yes/**Partly**/No
Please rate the manuscript on overall contribution to the field
Excellent/Good/Average/**Below average**/Poor
Please rate the manuscript on language, grammar and tone
Excellent/Good/**Average**/Below average/Poor
Is the manuscript succinct and free of repetition and redundancies?
Yes/No
Are the results and discussion confined to relevance to the objective(s)?
Yes/No
The number of tables in the manuscript is
Too few/Adequate/Too many/Not applicable
The number of figures in the manuscript is
Too few/**Adequate**/Too many/Not applicable
Is the supplementary material relevant and separated appropriately from the main document?
Yes/No/**Not applicable**

Please rate the manuscript on overall quality
Excellent/Good/Average/Below average/**Poor**
Is appropriate and adequate reference made to other work in the field?
Yes/**No**
Is it stated that ethical approval was granted by an institutional ethics committee for studies involving human subjects and non-human vertebrates?
Yes/No/**Not applicable**
If accepted, would you recommend that the article receives priority publication?
Yes/**No**
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Yes/No

Comments to the Author:

In general, the language used in the manuscript is free of unnecessary jargon. This potentially makes the manuscript one that can be easily understood by a wide audience. Unfortunately, the dearth of technical details and specificity makes some sections of the manuscript almost unintelligible.

The abstract is unbalanced and is missing a lot of key information. Conventionally, an abstract of a journal manuscript is supposed to provide a concise, but full, picture of the manuscript; with brief sections (about one to three sentences each); introducing the problem that the study was designed to address, delineating study aim(s) and objective(s), outlining the methodology used, highlighting key results and what they mean and summarising key conclusions.

Overall, the literature review is seriously wanting. The author(s) merely cite the core findings of four (4) studies, mainly that the four studies disagree on the impact of using recycled water on copper flotation: one set agrees with what appears to be the authors hypothesis-though not stated in black and white- and the other set disagrees with the author(s) implied hypothesis. There is not enough context given about any of the four studies, which makes it impossible for one to assess if the comparison made reasonable, and/or critically think of possible causes of the differences in the results that were obtained. It is hard not to wonder if this weakness is partly due to the extremely limited range of literature that the author(s) relied upon to ground this study (the reference list is only seven items long). Also, it is standard to conclude the literature review by clearly articulating the aims and objectives of the study being reported on? The authors have not done so. Adding to this confusion is the aim stated in lines 58-59, which appears to be the aim of the literature review section rather than the whole manuscript, and what is stated at the beginning of the materials and methods sections, lines 96-99, which is too vague to be of much value.

The way in which the materials and methods section is written makes it impossible for anyone, who might be interested, to replicate the study. The section is incredibly non-specific for a methodology section. Also, lots of key information is missing. For example, how many times were the experiments run, how many samples were collected each time, were the samples collected in multiples, make and model of different equipment used, reagents used, etc. What do(es) the author(s) mean(s) by statistical graphics?

Figures and tables are better introduced in text before they appear on the manuscript. Otherwise, the reader does not know what they are supposed to be looking at and/or how to look at it, if these items come before the text in which they are introduced. Generally, the results are not contextualised in terms of the existing literature and what they mean. It is not clear if Tables 1 and 2 are supposed to be part of the results and discussion section or part of the materials and methods section.

Finally, it is hard to comment on the accuracy, meaning and value of the conclusion since the main sections in the paper are missing so much vital information.

Author response to Reviewers: Round 1

First, I thank you for your feedback and your relevant remarks.
Following this, I have made changes to my article concerning the form and content.
in terms of form:

- I included the bibliography in the introduction to have a coherent text showing the previous studies and the role of my contribution
- I gathered some figures having the same role in the article
- I added references (in total 24 references)
- I added Data Availability and Code Availability statement to put the data as well as the python codes to obtain the figures available for the reader

in terms of content:

- I integrated Machine Learning (ML) in the Design of Experiment process to have a complete and efficient analysis of the phenomenon (effect of recycled water on copper recovery) in particular, ML allowed to choose the most adequate optimization model which explains much better the relationship between the factors and the response
- I added a figure showing the flowsheet followed during the laboratory tests and which presents the real flowsheet of the plant
- I plotted the response surface to determine the optimal proportion of fresh and recycled water allowing to reach a metal recovery of 80%
- I confirmed the results of the experimental plans (water proportions) by real tests in the laboratory (for this, I added a figure of the results)

Reviewer 1 (round 1) and Reviewer 3 (rounds 1 and 2)

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Appendix 1: Original manuscript for review

Optimizing Copper Flotation with Recycled Water using experimental design: Impacts on Recovery and Grade while Minimizing Freshwater Use

Abstract

This study investigates the impact of recycled water on copper flotation and aims to determine the optimal proportion of recycled water that can be used in the mining process in SOMIFER BLEIDA. The experimental results demonstrate that the use of recycled water has a significant impact on the copper flotation process, affecting both the recovery and grade. Through experiments involving varying proportions of water, an optimal proportion of recycled water was identified. This proportion maximizes the recovery and grade while minimizing the consumption of freshwater resources. After obtaining the optimal proportion of water, the reaction environment was changed, which required a re-optimization of the quantities of reagents used in copper flotation. In particular, the quantities of AXK, NaHS and pH were reoptimized using a design experiment to further improve the efficiency of the process.

Significance

The use of 100% fresh water can lead to higher efficiency, but its high cost makes it economically unfeasible. However, using a mixture of 50% fresh water and 50% recycled water can be both cost-effective and efficient. Furthermore, the use of experimental designs to optimize reagents can help companies achieve the best possible efficiency despite using recycled water. Therefore, this approach can be a sustainable and cost-effective solution for companies looking to maximize their profits while reducing their environmental impact.

Keywords: Recycled water, copper flotation, optimal proportion, recovery and grade, reagent quantities, sustainable water management.

*AXK: collector from xanthates family

*NaHS: sodium hydrosulfide

*pH: potential of hydrogen

*SOMIFER: Société Minière du Bougafer

1. Introduction

Copper flotation is an important process used in the extraction of copper from its ores. One important factor that affects the efficiency of this process is the quality of water used in the flotation process. In recent years, there has been a growing interest in using recycled water in copper flotation due to the increasing scarcity of freshwater resources.

The impact of recycled water on copper flotation has been extensively studied in scientific research. Several studies have shown that the use of recycled water can have a negative impact on copper flotation, as it may contain contaminants that can interfere with the flotation process. These contaminants may include dissolved salts, organic compounds, and other minerals.[1]

To address this issue, researchers have focused on determining the optimal proportion of fresh water that should be used in copper flotation to achieve maximum efficiency. This involves studying the effect of different proportions of fresh and recycled water on the flotation process, and identifying the optimum ratio that provides the best results and. As the optimum ratio is determined, the reaction environment will change so reagents used in copper flotation are then reoptimized.[2]

Overall, the impact of recycled water on copper flotation is an important area of research that has significant implications for the mining industry. By understanding the effect of recycled water on the process and optimizing the use of fresh water, researchers can help to improve the efficiency and sustainability of copper extraction processes.

2. Literature Review

In recent years, there has been growing interest in the use of recycled water in copper flotation due to the increasing scarcity of freshwater resources. However, the impact of recycled water on copper flotation recovery has been a subject of much debate in the scientific community. This literature review aims to provide an overview of the research conducted in this area and to identify the key findings and conclusions.[3]

Several studies have been conducted to investigate the impact of recycled water on copper flotation recovery. In one study by Xu et al. (2017), the authors used simulated seawater and recycled water in copper flotation experiments and found that the use of recycled water resulted in a decrease in copper recovery compared to fresh water. The authors attributed this to the presence of dissolved salts and other impurities in the recycled water, which interfered with the flotation process.[4]

Similarly, in a study by Valderrama et al. (2017), the authors found that the use of recycled water in copper flotation resulted in a decrease in copper recovery and an increase in the consumption of reagents. The authors attributed this to the presence of impurities such as calcium, magnesium, and sulfate ions in the recycled water, which affected the pH and chemical composition of the flotation pulp.[5]

However, not all studies have found a negative impact of recycled water on copper flotation recovery. In a study by Wang et al. (2018), the authors found that the use of recycled water had no significant impact on the recovery of copper in a low-grade copper ore flotation process. The authors attributed this to the fact that the impurities in the recycled water were at a low concentration and did not significantly affect the flotation process.[6]

Another study by Gomez et al. (2019) investigated the use of recycled water in copper flotation at a copper mine in Chile. The authors found that the use of recycled water did not significantly impact copper recovery, but did result in a decrease in the consumption of fresh water and reagents. The authors attributed this to the high quality of the recycled water used in the process.[7]

Overall, the research conducted in this area suggests that the impact of recycled water on copper flotation recovery can vary depending on the quality of the recycled water and the specific conditions of the flotation process. While some studies have found a negative impact of recycled water on copper recovery, others have found no significant impact or even a positive impact on the consumption of fresh water and reagents. Therefore, further research is needed to fully understand the impact of recycled water on copper flotation recovery and to develop strategies for optimizing its use in the copper mining industry.

3. Materials and Methods

To investigate the impact of different proportions of fresh and recycled water on copper flotation recovery and to re-optimize the quantities of reagents used in copper flotation, a series of laboratory experiments can be conducted using the following experimental methodology:

Sample preparation: A representative sample of the copper ore is collected and prepared for the flotation experiments. The sample is crushed and ground to a specific particle size range to ensure consistency in the tests.

Flotation tests: Flotation tests are conducted using a laboratory flotation cell or a bench-top flotation machine. The tests are carried out by adding a predetermined amount of the prepared sample to the flotation cell, along with fresh water, recycled water, or different proportions of both.

Experimental design: This involves selecting a range of different proportions of fresh and recycled water to be used in the experiments and systematically varying these proportions to identify the optimal ratio that provides the best results. After determining the optimal ratio, a design experiment was applied to re-optimize the quantities of reagents used in the new environment.

Data collection: During each experiment, data is collected on the recovery of copper, the grade of the concentrate, and the consumption of reagents. The data is recorded and used to analyze the impact of different proportions of fresh and recycled water and reoptimized reagent quantities on the copper flotation recovery.

Data analysis: The data collected is analyzed using statistical graphics to identify the impact of different proportions of fresh and recycled water on the copper flotation recovery. This involves comparing the recovery and grade of the concentrate obtained using fresh water, recycled water, and different proportions of both.

Results interpretation: The results obtained from the experiments and the data analysis are interpreted to draw conclusions about the optimal proportion of fresh and recycled water for copper flotation recovery. These conclusions are used to optimize the use of water and reagents in copper flotation processes.

Overall, conducting a series of laboratory experiments with different proportions of fresh and recycled water using DOE approach is an effective way to investigate the impact of water quality and reagents on copper flotation recovery. By systematically varying the proportion of fresh and recycled water, researchers can identify the optimal ratio that provides the best results and optimize the use of water in copper flotation processes.

4. Results and Discussion

After conducting several copper flotation tests with different types of water, we obtained the following results.

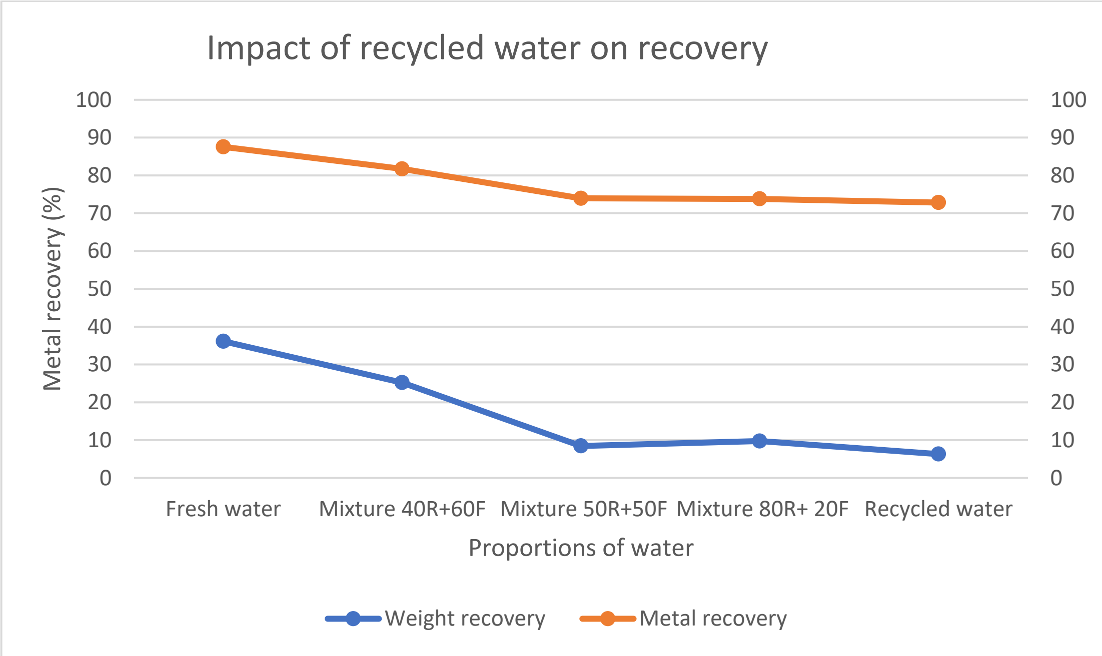

Figure 1 : Impact of recycled water on metal recovery

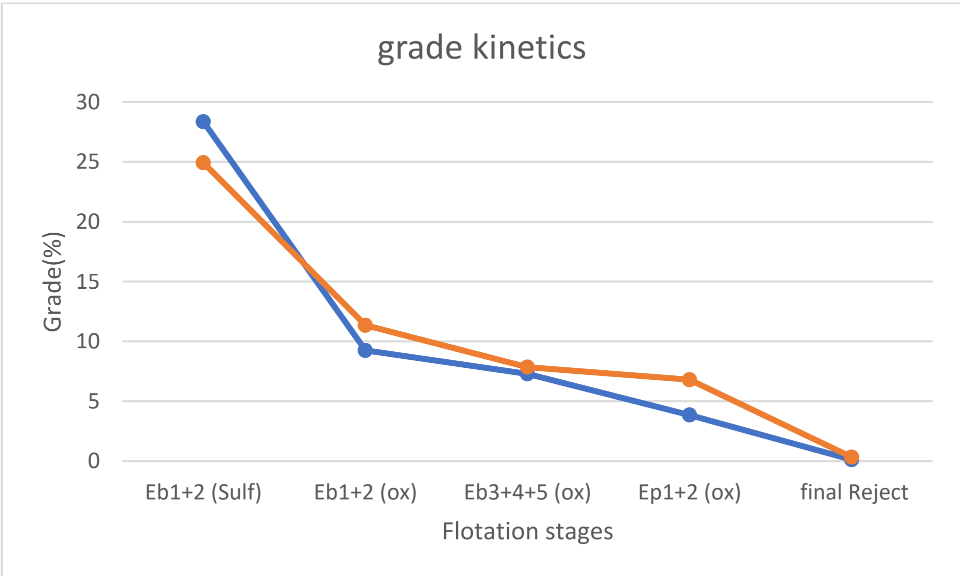

Figure 2 : Grade kinetics as a function of flotation stages

Using fresh water maximizes the recovery due to the low quantity of penalizing elements such as Ca, Mg, sulfates, and carbonates. However, introducing recycled water in quantities greater than 50% is detrimental to recovery due to the high number of penalizing elements that are recycled and disrupt the flotation process.

Ca and Mg consume reagents, especially the frother, which causes the foam to explode during the collection of useful copper elements, leading to their release into the pulp instead of flotation. Recycling water, which already contains a quantity of reagents AXK and NaHS, adds to the quantity added during flotation, causing the phenomenon of the double layer, which depresses oxidized and sulfured copper elements.

On the other hand, recycling at 50% is acceptable because it allows for moderate recovery. This is due to the recycling of an acceptable amount of pre-conditioned and dissolved reagents, which converges towards an optimal quantity when added to the reagents added during flotation (**Figure 1**)

We notice that sulfide copper is better recovered using fresh water than using recycled water, while oxidized copper is better recovered using recycled water than fresh water. This can be explained by the fact that recycled water already contains a quantity of sulfates resulting from the decomposition of NaHS, which forms a first layer on the sulfide copper. When the collector is added, a double layer is formed, which depresses the sulfide copper. On the other hand, using fresh water forms a single layer and flotation takes place under favorable conditions. The good content of oxidized copper using recycled water can be explained by the fact that it contains a quantity of decomposed sulfates, which add to the amount of NaHS added, surrounding the oxidized copper with a sulfur layer, which leads to its activation, since the oxidation rate is around 75% (**Figure 2**)

Once the optimal ratio of fresh and recycled water has been determined, the composition of the medium will change, and therefore, a new optimization of the flotation reagents is required. Specifically, the quantities of AXK, NaHS, and pH levels need to be re-evaluated. To achieve this, a complete factorial design experiment with two levels was carried out, which allowed for the identification of the optimal quantities of each of the three factors in the new mixture. The reagents and the test matrix used in the experiment are presented in tables below:

Table 1: Calculation of the number of tests

Number of factors	Number of levels	Number of tests
3	2	2^3=8

Table 2: Low and high levels of the factors

Factor	Low level (-1)	High level (1)
AXK	60g/t.	100g/t.
NaHS	900g/t.	1100g/t.
pH	8	10

After using Two-level factorial design on the quantities of reagents used in copper flotation such as AXK, NaHS, and pH, we obtained these results:

Table 3: Matrix of tests

AXK	NaHS	pH	Metal recovery (%)
-1	-1	-1	59,3
1	-1	-1	59,51
-1	1	-1	72,73
1	1	-1	67
-1	-1	1	73,26
1	-1	1	85,42
-1	1	1	63,52
1	1	1	75

Regression equation in non-coded units:

Metal recovery (%) = -584,8 + 1,845 AXK + 0,7329 NaHS + 67,90 pH - 0,003372 AXK*NaHS - 0,1465 AXK*pH - 0,07765 NaHS*pH + 0,000329 AXK*NaHS*pH

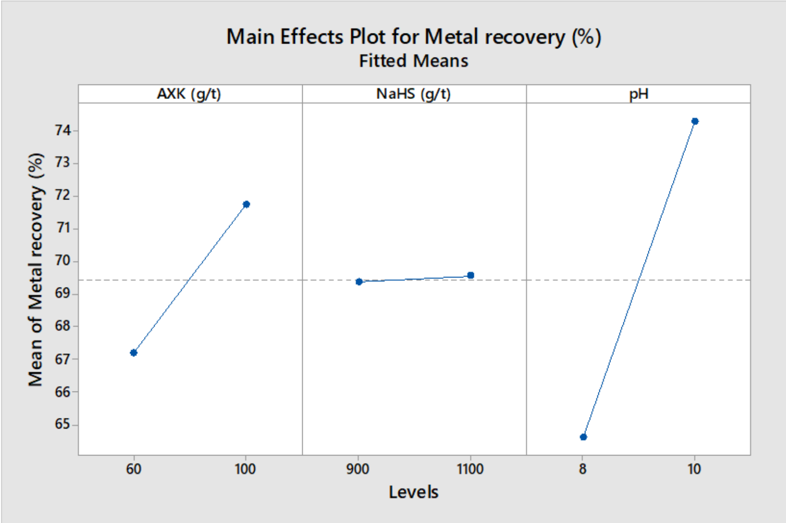

Figure 3 : Main effects plot for metal recovery

The main effects plot (**Figure 3**) confirms that pH is the most influential factor since small variations in this factor have a high effect on metal yield. AXK comes in second place in terms of its effect, and NaHS comes in last.

A line plot of mean metal recovery (%) with two factors on the x-axis can provide valuable information about how the factors interact and affect the metal recovery as shown below:

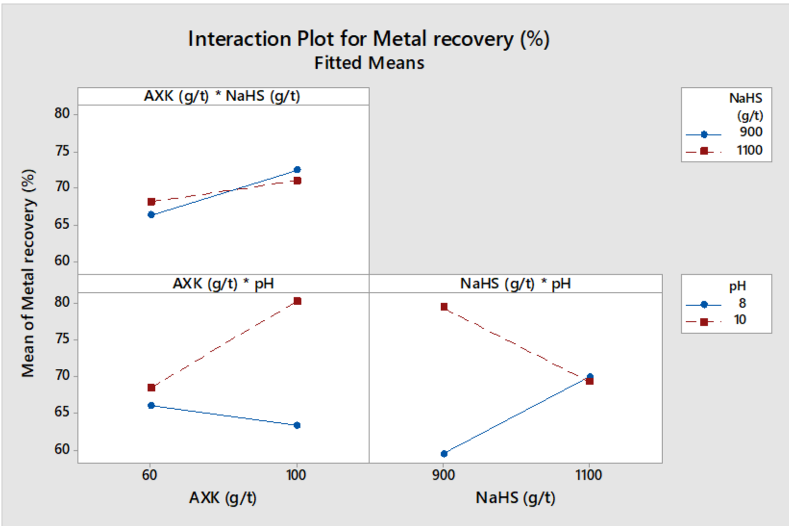

Figure 4 : Interaction plot for metal recovery

The steep slope of each factor suggests that both NaHS and AXK have a significant effect on the mean metal recovery. This means that changes in the levels of NaHS and AXK can result in a considerable change in the mean metal recovery.

The non-parallel lines suggest that there is an interaction between NaHS and AXK. This means that the effect of NaHS on the mean metal recovery depends on the level of AXK, and vice versa. The combined effect of the two factors is greater than the sum of their individual effects. This suggests that an optimal combination of NaHS and AXK can maximize the mean metal recovery.

The intercept at a mean metal recovery of 69.5% indicates the maximum mean metal recovery achievable with the current levels of NaHS and AXK. This suggests that further increasing the levels of NaHS and AXK beyond the current levels may not result in a higher mean metal recovery.

Also, the fact that the lines are not parallel indicates that the effect of NaHS on metal recovery is not constant across all levels of pH. Instead, the effect of NaHS on metal recovery depends on the level of pH, and vice versa. The fact that the lines intersect at the end of the NaHS domain suggests that the optimal combination of NaHS and pH for maximum metal recovery lies at this point.

There is an interaction between AXK and pH, which is indicated by the observed behavior of intersection. Specifically, at the estimated intersection point of AXK and pH, the mean metal recovery is 65%.

Overall, the non-parallel lines and intersections of the mean metal recovery with (NaHS,pH), (AXK,pH) and (AXK, NaHS) suggest that there may be opportunities to optimize the levels of NaHS, AXK and pH to achieve higher mean metal recovery, while taking into account any potential trade-offs or constraints associated with operating the process at these optimal levels.

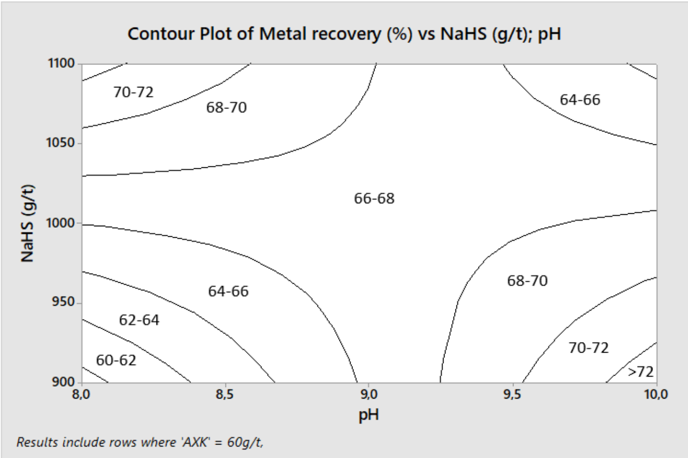

Figure 5 : Contour plot of metal recovery vs NaHS; pH with AXK=60g/t

By fixing AXK at 60g/t, the yield can reach its maximum potential of over 72% in two ways: by maximizing NaHS and maintaining a pH around 8, or by adopting a pH close to 10 while keeping NaHS levels to a minimum (**Figure 5**).

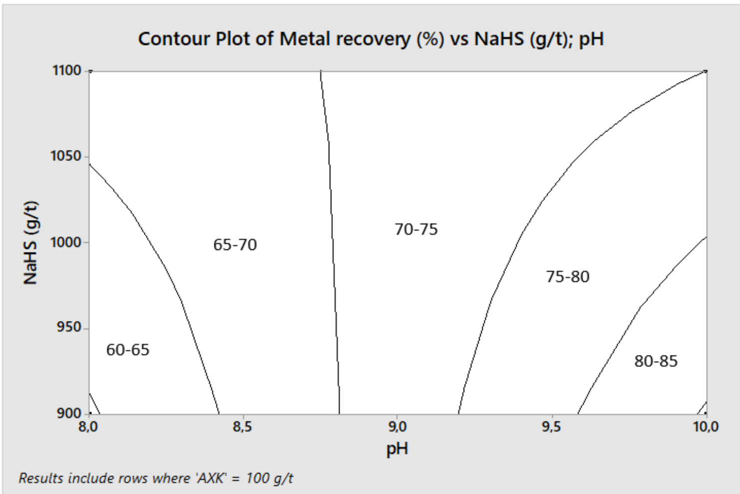

Figure 6 : Contour plot of metal recovery vs NaHS; pH with AXK=100g/t

By fixing AXK at 100g/t, the yield can reach its maximum potential of over 85% with a minimum NaHS level of 900g/t and a pH of 10. This results in significant cost savings for NaHS (**Figure 6**).

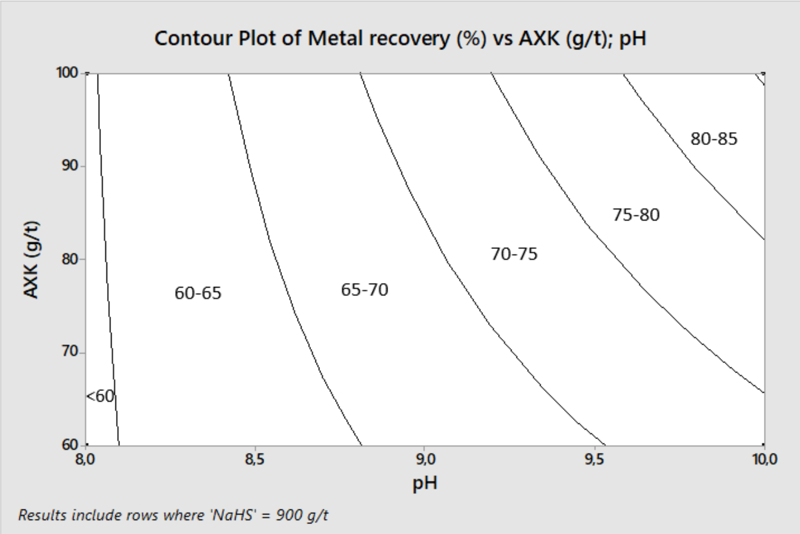

Figure 7 : Contour plot of metal recovery vs AXK; pH with NaHS=900g/t

By fixing NaHS at 900g/t, the yield is favorable when using an amount of AXK exceeding 80g/t and maintaining a pH between 9.7-10, and can reach up to 85%. However, by using the maximum amount of AXK and maintaining a pH of 10, the yield can exceed 85% (**Figure 7**).

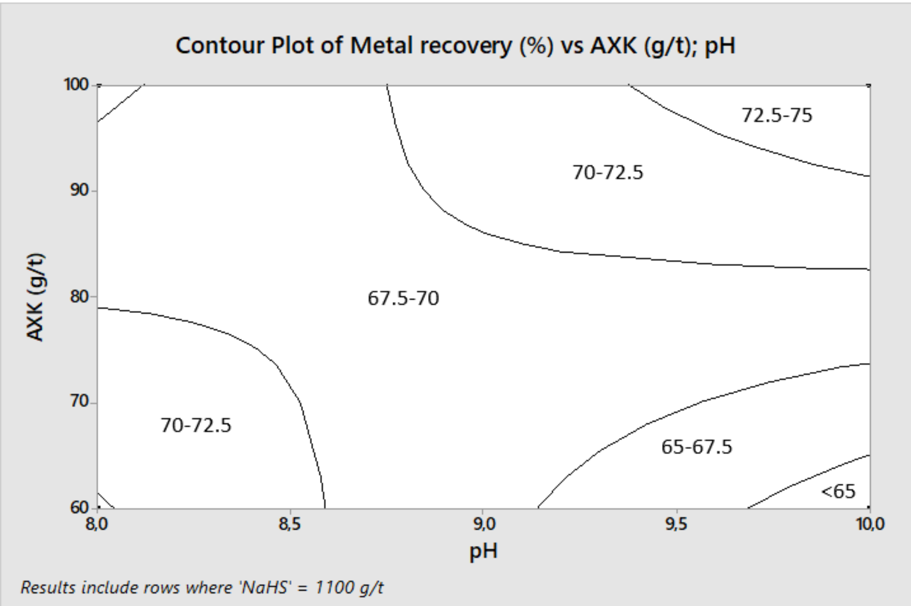

Figure 8 : Contour plot of metal recovery vs AXK; pH with NaHS=1100g/t

When NaHS is fixed at 1100g/t, two zones can be identified where the yield is maximal and exceeds 75%: the first zone is achieved by using around 60g/t of AXK and maintaining a pH of 8, while the second zone is obtained by using an amount of AXK exceeding 90g/t and maintaining a pH above 9.5 (**Figure 9**).

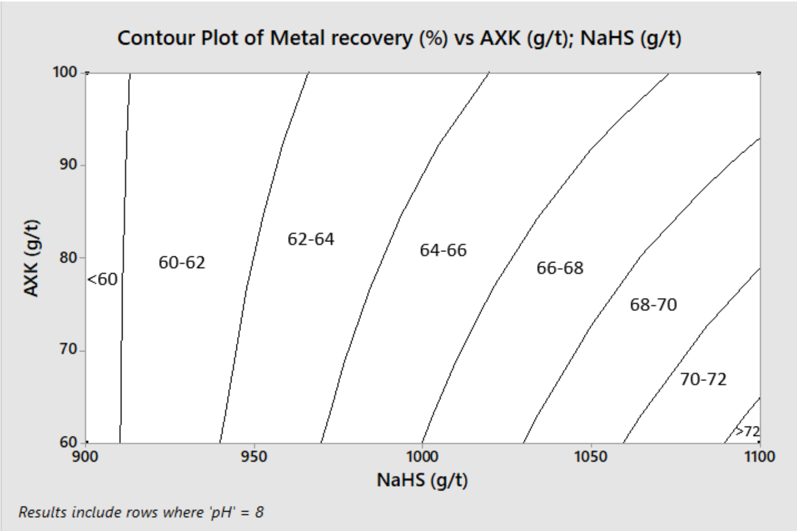

Figure 10 : Contour plot of metal recovery vs AXK; NaHS with pH=8

When the pH is fixed at 8, the yield can reach its maximum potential but only up to approximately 72% for doses of AXK ranging from 60-65g/t and NaHS levels close to 1100g/t (**Figure 11**).

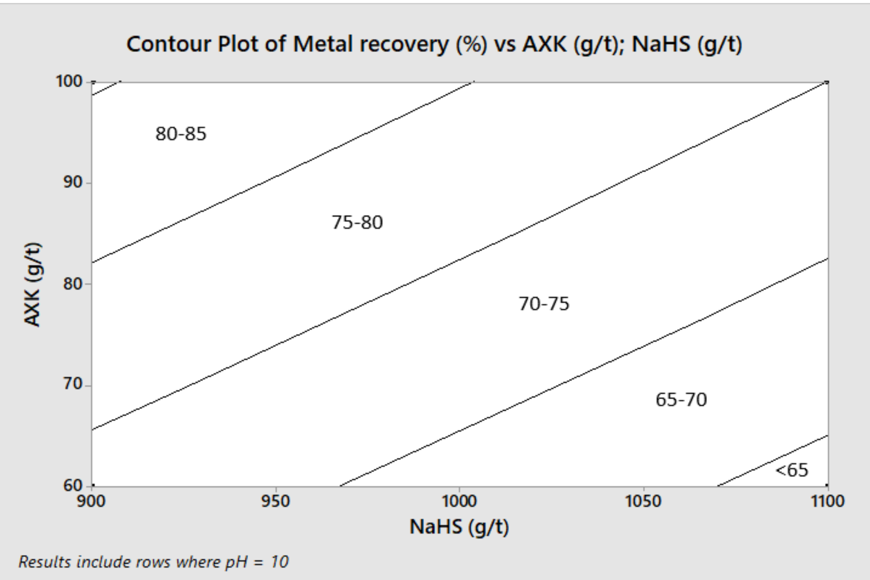

Figure 12 : Contour plot of metal recovery vs AXK; NaHS with pH=10

By fixing the pH at 10, the yield can reach its maximum potential and can exceed 85% for doses of AXK at 100g/t and NaHS at 900g/t (**Figure 10**).

Summary:

The above results lead us to conclude that the doses of reagents that give a yield of over 80% are: 100 g/t of AXK, 900 g/t of NaHS, and a pH of 10.

So let's confirm all this by using curves of optimization:

Choice 1: maximization of metal recovery

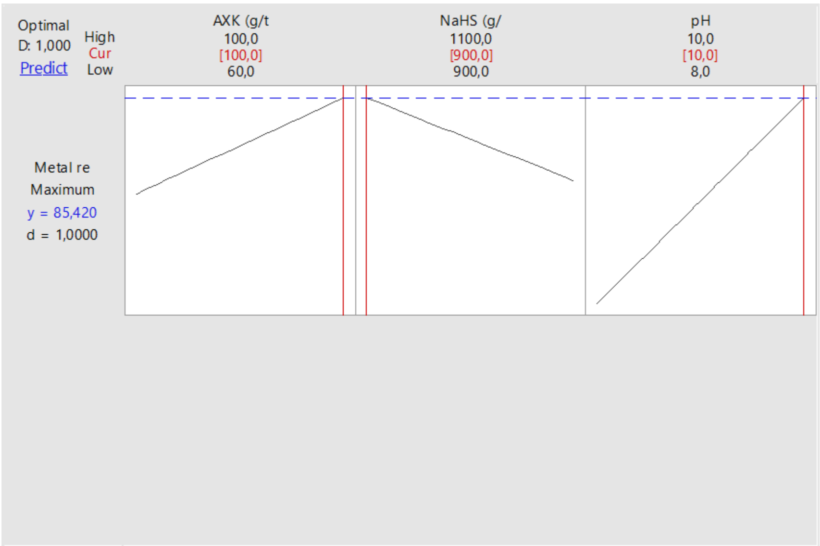

Figure 13: Doses of reagents to maximize metal recovery

As is clear from the figure above, to maximize metal recovery, we will need 100 g/t of AXK, 900 g/t of NaHS, and a pH of 10 (**Figure 11**)

Choice 2: metal recovery=80% (which is sufficient)

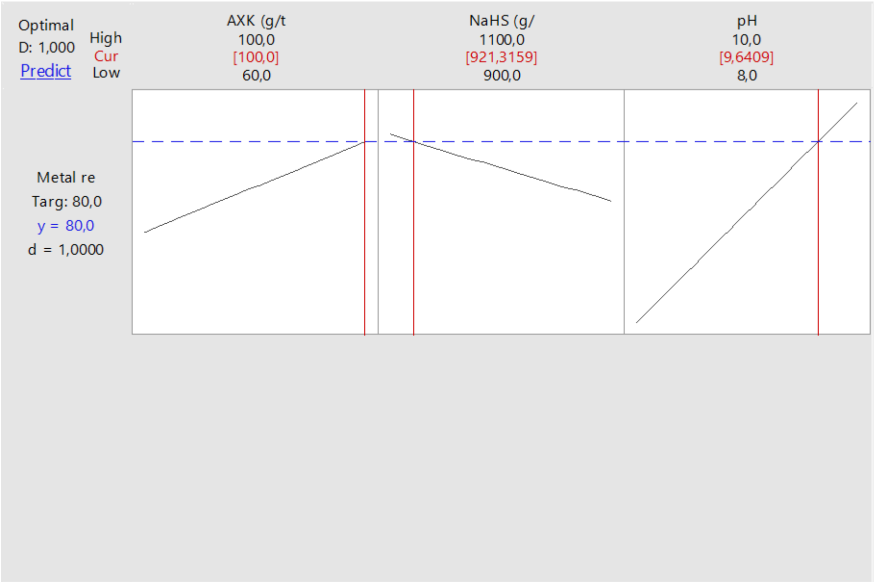

Figure 14: Doses of reagents to achieve a metal recovery of 80%

To achieve a metal recovery of about 80%, which is widely sufficient and meets the customers' requirements, we will need 100 g/t of AXK, 921.31 g/t of NaHS, and a pH of 9.64 (**Figure 12**)

So, it is recommended to use these doses since they meet the customers' requirements and at the same time save the cost of the reagents used.

5. Conclusion

In conclusion, using 100% fresh water may lead to higher yield, but its high cost makes it economically unfeasible. However, using a mixture of 50% fresh water and 50% recycled water can be both profitable in terms of yield and economical, as recycled water from the dam can be utilized, reducing the overall cost. Moreover, using design experiments to optimize reagents can help companies achieve the best possible yield despite using recycled water. Thus, this approach can be a sustainable and cost-effective solution for companies looking to maximize their profits while minimizing their environmental impact.

Acknowledgement: Anonymised

Disclosure statement: Conflict of Interest: there are no conflicts of interest about this work. Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

References

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