Box-Behnken design application in the leaching of roasted chalcopyrite concentrate using imidazolium-based ionic liquid

Chad Goodman, Antoine F Mulaba-Bafubiandi and Elvis Fosso-Kankeu

***Abstract*—** The box-Behnken design was used to layout the runs and conditions used to recover copper during leaching from chalcopyrite which was subjected to different roasting temperatures. The lixiviants sulphuric acid and the imidazolium ionic liquid 1-Butyl-3-methyl Imidazolium Hydrogen Sulphate [Bmim]HSO4 and sodium bromide were used and recoveries compared.

The methodology followed included leaching in a beaker with a solid liquid ratio of 10% with the two lixiviants, one being the sulphuric acid and the other the imidazolium based ionic liquid with the different parameters which were roasting temperature, leaching temperature, concentration and time.

With the addition of the bromide oxidant the imidazolium ionic liquid provided higher copper recoveries than the sulfuric acid alone. The ionic liquid has the advantage of being environmental friendly, while the sulfuric acid provides a simpler and cheaper option.

***Keywords*—** Chalcopyrite, imidazolium ionic liquid, bromide, sulfuric acid, box-Behnken design, roasting, leaching.

I. Introduction

In the world today there has been a rapid increase in the "green movement", this being that the world is moving in an eco-friendly direction. This trend is evident in rising investment in renewable energy, a shift towards eco-friendly construction practices, and a greater emphasis on reducing waste and carbon footprints [1]. There was also a study conducted by creative research platform Visual GPS, in conjunction with market research firm YouGov, also indicates a shift during the pandemic. The survey indicated that 81% of people polled expect companies to be environmentally conscious in their advertising and communications, and 69% of respondents said they were doing everything possible to minimize their carbon footprint [2]. This movement as one can guess can also be seen in the mining industry by making processes more sustainable and techniques greener. This movement is achieved by a combination of factors, including environmental regulations, growing consumer and investor pressure for sustainability, and the recognition that sustainable mining can also offer economic benefits [3].

Chalcopyrite, which is shown by the chemical formula CuFeS2 is a yellow crystalline mineral that contains Sulphur, Copper and Iron. 70% of copper deposits world-wide are owed to chalcopyrite since is the most common iron bearing [4]. Chalcopyrite can be found in areas where igneous or metamorphic rocks are formed. Although mainly used for its copper content there are many other reasons this ore is still mined to this day and that is due to its use in the jewelry sector, its use in copper smelting and for its use in the making of sculptures [5].

There are however challenges with the processing of chalcopyrite, and this is due to passivation. Passivation is the formation of a surface layer that inhibits further dissolution and copper extraction, often due to the accumulation of sulfur-containing compounds like polysulfides or elemental sulfur [6]. Chalcopyrite is often roasted before it is sent to the subsequent stages of extraction (leaching) as this is to make these stages easier and recoveries higher. Roasting is a process where sulfide ores are heated in the presence of oxygen at high temperatures which results in them being converted into metal oxides and impurities being removed [7].

This copper metal can be extracted using either hydrometallurgy or pyrometallurgy depending on if certain criteria are met. Predominantly copper is extracted using pyrometallurgy, however it is only pursued if seen as economical. Moreover, this process results in SO2 being emitted. Alternatively, hydrometallurgy is used for when the grades of copper are low because its costs for operation are lower [8]. Due to the previous stated points, chalcopyrite extraction is done using hydrometallurgical leaching, wherein lixiviants are usually used in conjunction with various oxidizing agents [9].

The use of acids, however, does come with various drawbacks with the most prominent being the emission of SO2, this can however be avoided by using different reagents for the extraction. There has been intense research going into the use of ionic liquids as leaching reagents. Ionic liquids illustrated by the acronym (ILs), are salts in liquid form when the temperature is that of room temperature or low

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temperatures in general [10]. The reason for the sudden interest is they can be directly used as electrolytes for the electrowinning of dissolved metal ions, high potential for selectivity in metal extraction, possibility for recycling and reuse and produce little to no vapor pressure therefore does not produce air pollution. An example of these liquids and also quite a popular ionic liquid in the leaching field is Imidazolium-based liquids. The structure is that of a imidazole ring which forms the core of the cation, with alkyl groups often attached to the nitrogen atoms, allowing for tailoring of the ionic liquid's properties. Thus, allowing it to have unique properties and is often used in chemical reactions due to its ability to dissolve a wide range of substances [11].

With the enhancements in technology, it's no surprise that instead of running these experiments to compare the results of leaching via the two different methods and wasting valuable time, minerals, money and equipment, the results of these tests can be run using various programs. An example of this software is the Box-Behnken design, named after its creators, George E.P Box and Donald Behnken. This design works by placing experimental points at the midpoints of the edges of the experimental space, along with center points allowing for efficient exploration of a process or system to identify optimal conditions while avoiding extreme combinations of factors [12]. There are various platforms that can run this design and examples include Minitab, Design-Expert, R, MATLAB, and Excel [13].

There are many issues at hand that come when leaching using acidic lixiviants. Examples of issues encountered include slow dissolution rates, passivation of the chalcopyrite surface, and the formation of secondary products that impede leaching efficiency. Contrary to this however, there have been various studies showing the effects of ionic liquid leaching, which indicate good recoveries, more efficient "circular "process and less environmental impacts. This is due to the fact ionic liquids are green solvents that provide lower flammability, volatility, and toxicity, while also enabling selective metal extraction and recyclability [14]. Therefore, the leaching of roasted chalcopyrite shall be carried out using ionic liquids, with the optimal recovery conditions gathered using software to interpret the results from the leaching process. Said results will be achieved using the Box-Behnken design, which is able to compare the different variables and indicate the best recovery conditions.

The main challenge in leaching chalcopyrite will be to minimize the effect of passivation [15-22].

II. METHODOLOGY

A. Box-Behnken design

The Box-Behnken design was set up using Minitab software with the four variables of temperature, roasting temperature, time and concentration being used. Each variable had three points which were the low, centre and high points resulting in a total of 28 runs taking place. This provided the matrix conditions for the entire experiment.

B. Sample

First approximately 500g of concentrated chalcopyrite was weighed and collected for the whole experiment

C. XRF

Approximately 20g of sample was used for XRF analysis where 10g of sample was weighed then mixed with 5g of Sasol wax. This mixture was then placed in an aluminium cup and thereafter placed in the pellet press die where it was compacted using the hydraulic pellet press machine. After the pellet was gotten, the sample was taken to the Rigaku ZSX primus II XRF analyser for semi-quantitative analysis and the chemical composition obtained.

D. XRD

Approximately 10g was taken for XRD where it was placed in the sample container and filled to the maximum. A glass side was placed on top of the sample to hold down and flatten the surface of the sample making it homogeneous while being equally spread over the glass container. The finished sample was placed into the XRD spectrometer and the sample analysis obtained.

E. Roasting

The rest of the sample was then taken and split for roasting where it underwent 3 different roasting temperatures namely, 500℃, 700℃ and 900℃. The different samples were then allowed to cool before 30g was taken again and made into powder for the XRD and XRF procedures to be done again but now on the roasted samples.

F. Leaching

Leaching was carried out practically with two different lixiviants, the first was sulphuric acid as a control and the other was the imidazolium based ionic liquid (1-Butyl-3-methylimidazolium Hydrogen Sulphate [Bmim]HSO4) combined with sodium bromide which was a complexing agent at an amount of 100g/t to aid in copper extraction. The roasted samples were also first taken for pulverization to create a fine powdered sample.

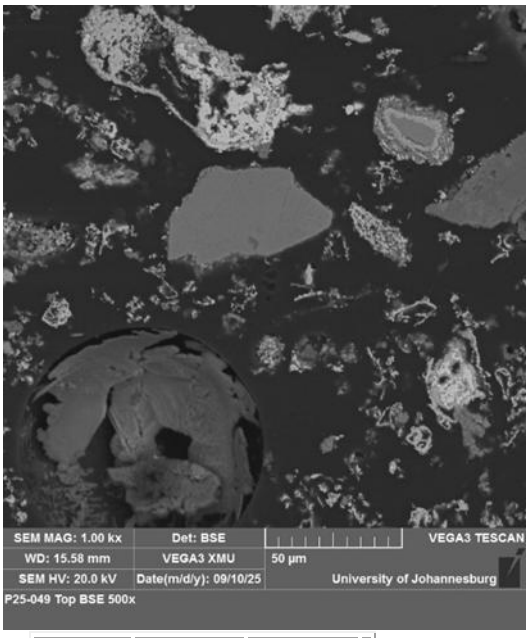
5g of sample was placed in a beaker, deionized water and sodium bromide added as well as magnetic bar and placed on a magnetic stirrer and ran for the time, concentration, roasting temperature and temperature depending on the run number. After the time was completed, the leachate was taken and filtered in a conical flask and added into a collection tube and then ICP-AAS was done on the leachate to get results from the runs. The table below was followed for the 28 runs needed to be carried out.

The results from the experiment were then compared with the results from the box Behnken design and compared therefore acknowledging the application of the design

III. RESULTS AND DISCUSSION

CHARACTERIZATION

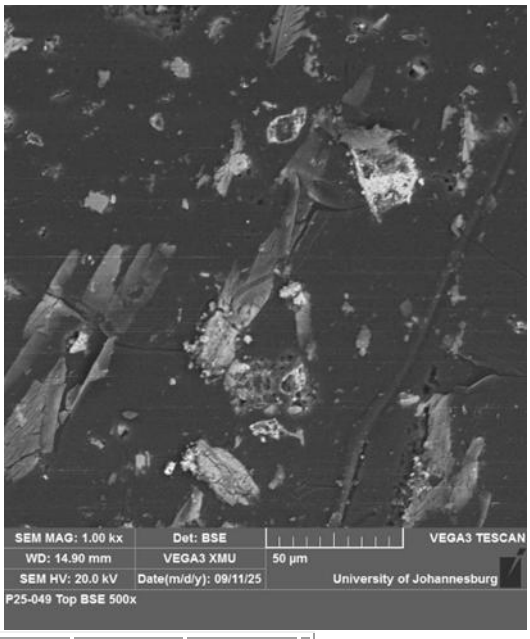
SEM and EDS



Element	Weight%	Atomic%
O K	18.68	39.87
Mg K	2.35	3.30
Si K	0.27	0.33
S K	25.58	27.25
K K	0.77	0.67
Ca K	0.70	0.60
Fe K	3.05	1.86
Cu K	48.59	26.11
Totals	100.00	

Fig. 1: SEM and EDS for 500 roasting

Figure 1 above shows both the SEM and EDS results for the roasted chalcopyrite sample at 500℃. It can be seen from the SEM that there are various contrasts between the surrounding elements and the chalcopyrite. Chalcopyrite usually appears dark on SEM but it can also be seen that the impurities present are presented by the lighter particles which could mean pyrite is present. From the EDS its seen that the element sulphur makes up a large amount of the SEM this means that there are still a lot of sulphides present and that more heat should be added for better conversion into oxides. There is also low levels of iron which can also lead one to believe that the ore is of low grade.

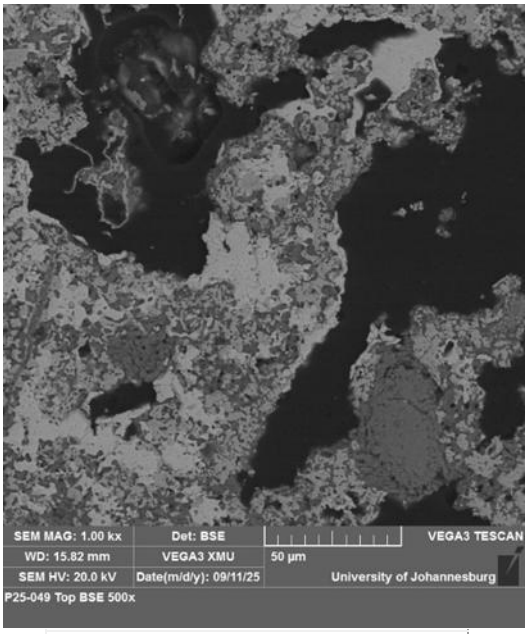


Element	Weight%	Atomic%
O K	54.29	76.13
Mg K	0.84	0.78
S K	16.62	11.63
Cl K	4.71	2.98
Ca K	0.52	0.29
Fe K	1.37	0.55
Cu K	21.65	7.64
Totals	100.00	

Fig. 2: SEM and EDS for 700 Roasting

temperature

Figure 2 illustrates the SEM and EDS results for the roasted chalcopyrite sample at 700℃. From the SEM as well as the EDS it can be seen there are a lot of disseminated particles present. In this sample, however, the sulphur level has dropped, and oxygen levels have increased, indicating that the sample is becoming more oxidized at this temperature.



Element	Weight%	Atomic%
O K	22.68	52.18
Mg K	1.24	1.88
Si K	0.34	0.45
Fe K	19.82	13.07
Ni K	0.41	0.26
Cu K	55.51	32.16
Totals	100.00	

Fig. 3: SEM and EDS for 900 roasting

Figure 5 above shows the SEM and EDS results for the chalcopyrite sample which was roasted at 900℃. In this image there are less disseminated particles present, and the overall image appears lighter. This lighter image can be due to the impurities present. Sulphur is completely gone meaning the sample is fully oxidized.

XRD

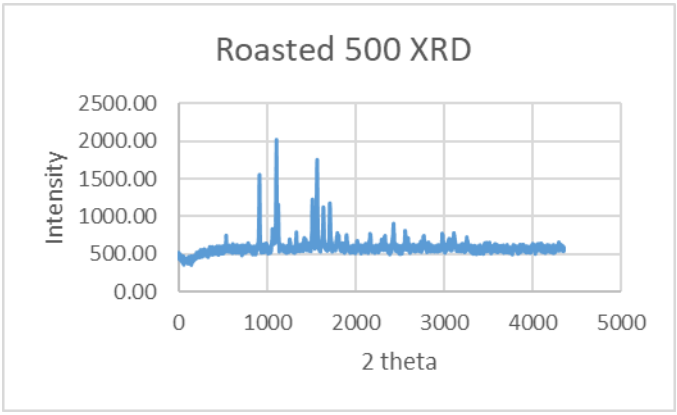


Fig. 4: XRD plot for chalcopyrite roasted at 500

The figure above illustrates the XRD results for the roasted chalcopyrite at 500℃, as seen in the previous results of SEM and EDS for the same sample the highest peaks present belong to that of copper sulphate concluding that at this stage copper sulfate is the most present phase at this stage.

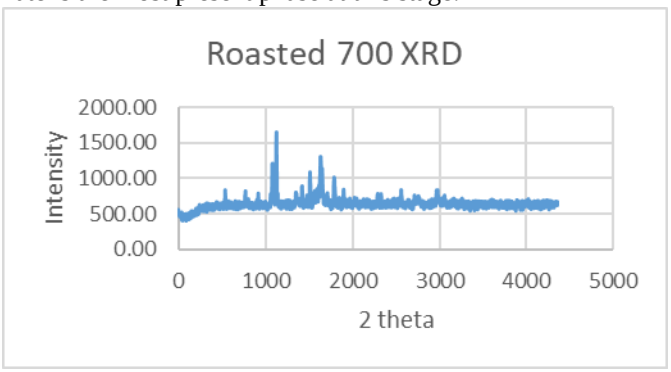


Fig.5: XRD plot for chalcopyrite roasted at 700

Figure 5 shows the XRD plot for chalcopyrite roasted at 700℃, the phase has changed now due to the sulphur content decreasing and oxygen content increasing. Now the highest peak belongs to copper iron oxide, indicating that the sample is more oxidized and the sulphur content has decreased.

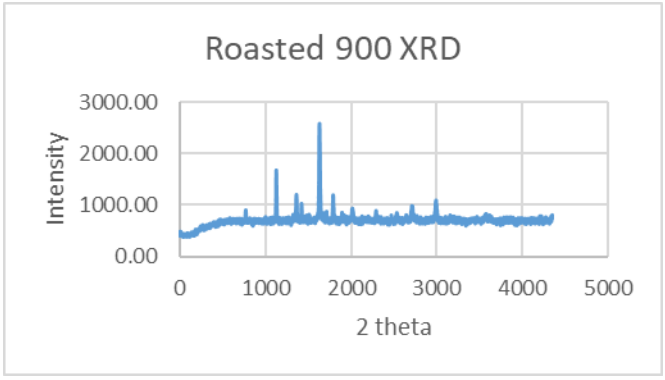


Fig. 6: XRD plot for chalcopyrite roasted at 900

Figure 8 indicated the XRD plot for 900℃ roasted chalcopyrite, it is similar to the phases present in 700℃. Once again the highest peak present is copper iron oxide, once again

meaning that this is the most abundant phase present. According to SEM and EDS there is even less sulphur present meaning as heat increased over the three roasting procedures the oxidation increased.

ANOVA

Ionic liquid

Pareto chart for iron recoveries

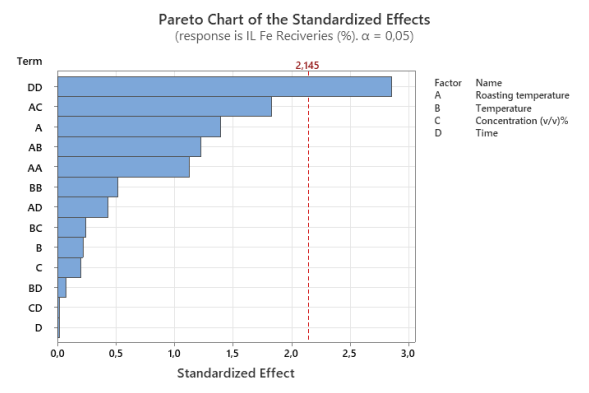


Figure 7: Response surface regression for ionic liquid iron recoveries vs roasting temperature vs temperature vs concentration (v/v)% vs time

Regression equation for iron recoveries

IL Fe Recoveries (%)= -10,6+0,0092 A+0,261 B-1,58 C+3,35 D-0,000009 A^2-0,00161 B^2-0,2374 D^2-0,000198 AB+0,00194 AC-0,000424 AD+0,0067 BC-0,0015 BD-0,004 CD

Figure 7 above indicates the pareto graph for the ionic liquids used to recover iron. From the graph it is seen that time is the most significant effect on the recoveries followed by the combination of roasting temperature and concentration (v/v)% and in third place was roasting temperature. The regression equation above shows the equation giving the relationship between the dependent and more than one independent variable.

Pareto chart for copper recoveries

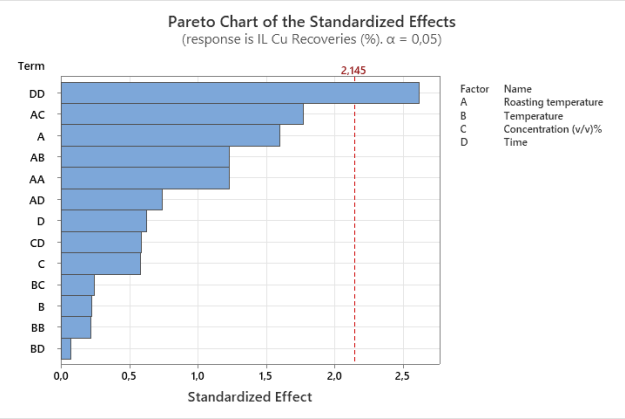


Figure 8: Response surface regression for ionic liquid copper recoveries vs roasting temperature vs temperature vs concentration (v/v)% vs time

Regression equation for copper recoveries

IL Cu Recoveries=254-0,462A-1,97B-18,8C+21,5D+0,000123A^2+0,0084B^2 -2,66D^2+0,00244 AB+0,0230AC+0,0089AD-0,083BC-0,018BD+1,39 CD

Figure 9 above indicates the pareto chart for copper recoveries when using the ionic liquid. From this chart it is seen that once again time is the most significant followed by the combination of roasting temperature and concentration (v/v)% and the third most significant effect is the roasting temperature. The least significant effects is shown by the combination of temperature and time. Above is also the regression equation indicating the relationship between all the variables.

Sulphuric acid

Pareto chart for iron recoveries

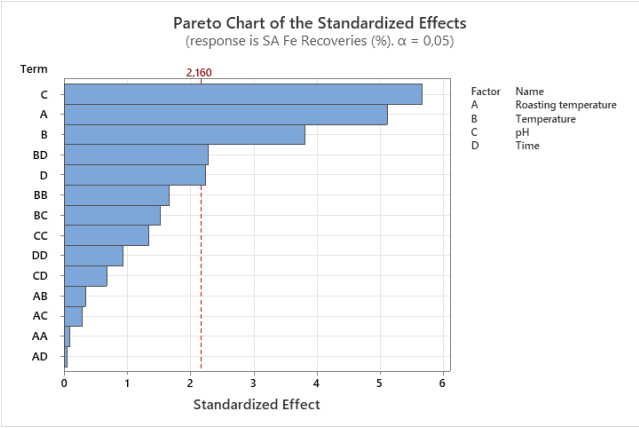


Figure 9: Response surface regression for sulphuric acid iron recoveries vs roasting temperature vs temperature vs pH vs time

Regression equation for iron recoveries

SA Fe Recoveries (%)=31,6-0,0081A-0,361B-6,6C-3,03D-0,000002A^2+0,00465B^2+3,36C^2+0,146D^2-0,000088AB+0,00223AC-0,00010AD-0,156BC+0,0584BD-0,520CD

Figure 10 indicated the pareto graph, it indicates the regression surface regression for iron recoveries using sulphuric acid. For these responses the most significant impacts are observed from pH followed by roasting temperature then temperature. The least impcts are seen by combinations of roasting temperature and pH as well as the combination of roasting temperature and time. Lastly the regression equation above indicates the relationships between all the variables.

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Pareto chart for copper recoveries

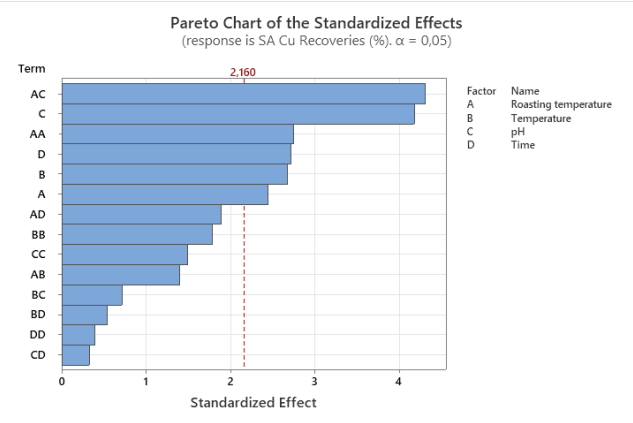


Figure 10: Response surface regression for sulphuric acid copper recoveries vs roasting temperature vs temperature vs pH vs time

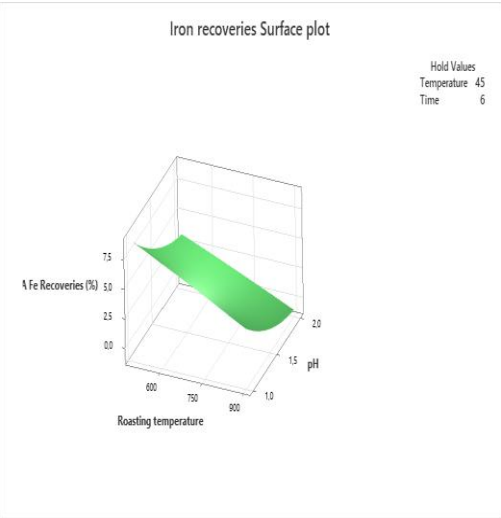
Regression equation for copper recoveries

SA Cu Recoveries (%)=153-0,313A+1,75B+57,9C-21,3D+0,000250A^2-0,0287B^2+21,7C^2+0,359D^2+0,00208AB-0,1913AC+0,0210AD-0,423BC+0,080BD+1,44CD

Figure 10 above is the pareto graph for sulphuric acid recoveries against rosting temperature, temperature, pH and time. It is seen that the lesser effects are faced from combinations of temperature and time and combination of pH and time. The higher effects are from the combination of roasting temperature and pH, followed by pH and then time. Above is also the regression equation indicating the relationship between sulphuric acid copper recoveries and time, roasting temperature, temperature, pH and time.

Surface plots

Sulphuric acid iron recovery surface plots



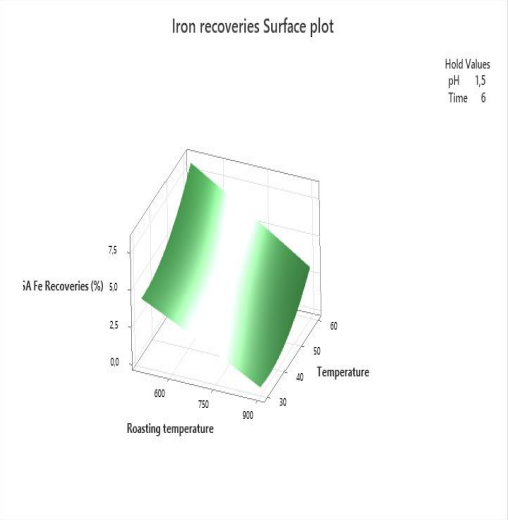
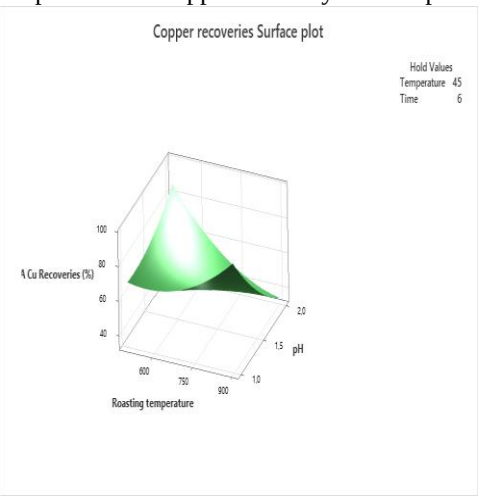


Figure 11: Surface plots for roasting temperature vs pH and roasting temperature vs temperature

Figure 11 above are the surface plots of roasting temperature vs pH and roasting temperature vs temperature respectively. From the plot of roasting temperature vs pH temperature and time was held constant at 45℃ and 6 hours. While for the roasting temperature vs temperature plot the pH and time were held constant at 1,5 and 6 hours. For the plot on the left higher recoveries occur at lower pH of 1 and lower roasting temperature of 500℃. Whereas for the other plot on the right-hand side higher recoveries occurred at higher temperatures (60℃) and lower roasting temperatures (500℃).

Sulphuric acid copper recovery surface plots



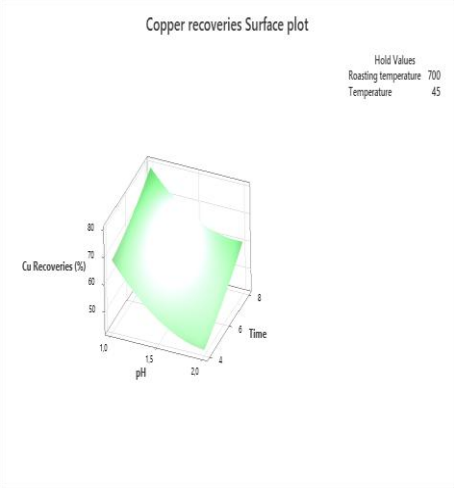
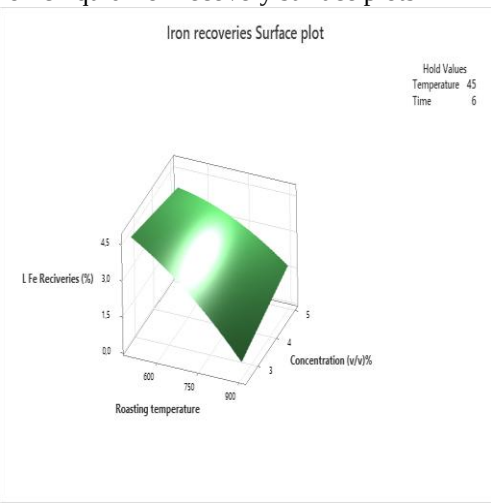


Figure 12: Surface plots for roasting temperature vs pH and pH vs time

The surface plots for roasting temperature vs pH and pH vs time is shown in Figure 12 above. For the plot on the left which is the roasting temperature vs pH temperature was held constant at 45℃ and time at 6 hours. Higher recoveries are seen at high roasting temperatures and low pH as well as with low roasting temperatures and a pH at 2. For the plot on the right which is pH vs time higher recoveries are seen when the pH is low (around 1) while time is a duration of 8 hours.

Ionic liquid iron recovery surface plots



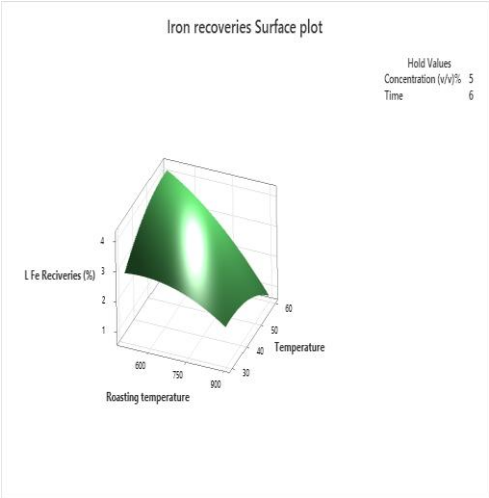
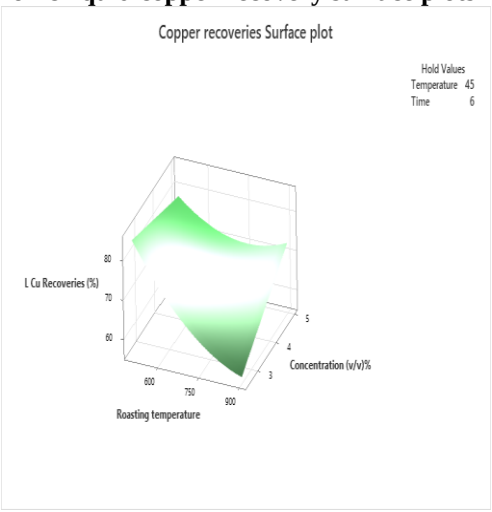


Figure 13: Surface plots for roasting temperature vs concentration (v/v)% and roasting temperature vs temperature

Figure 13 illustrates the surface plots of roasting temperature vs concentration (v/v)% and roasting temperature vs temperature. The first plot (the plot on the left) temperature was held constant at 45℃ and time at 6 hours. Results illustrated that the highest iron recoveries are seen when the roasting temperature is lower (500℃) and concentration lower (2,5%). Recoveries were still high however for high concentrations and low roasting temperatures. The second plot on the right where concentration was held constant at 5% and time at 6 hours shows that iron recoveries are highest when the roasting temperature is lower and temperatures higher. Higher recoveries can also be seen for low roasting temperatures and lower temperatures and higher roasting temperatures and lower temperatures.

Ionic liquid copper recovery surface plots



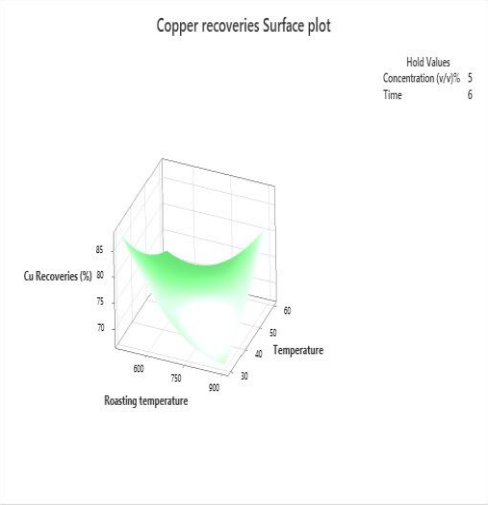


Figure 14: Surface plots for roasting temperature vs concentration (v/v)% and roasting temperature vs temperature

Figure 14 indicates the surface plot of roasting temperature vs concentration (v/v)% and roasting temperature vs temperature. For the plot on the left temperature and time was held constant at 45℃ and 6 hours respectively. Results from this plot indicate that highest copper recoveries are seen when the roasting temperature is on the lower side and concentration from 2,5% up to 5%. Lower recoveries are seen when roasting temperatures are high and concentration is on the lower side. For the plot on the right concentration was held at 5% and time at 6 hours. From the plot highest recoveries occurs as both extremes; where roasting temperature was low and temperature low as well as when roasting temperature was high and temperature high.

Recoveries

Iron Recoveries using sulphuric acid

The recoveries of iron when using sulphuric acid at the different parameters followed in the methodology. The highest iron recoveries are seen at run 24 (5,92%) when the temperature was 700℃, pH was 1,5. temperature 60℃ and time 8 hours. While the lowest recovery (0,38%) came from run 20 where the roasted temperature was 900℃, pH 2, time 6 hours, and temperature 45℃.

Sulphuric acid copper recoveries

The copper recoveries when leaching with sulphuric acid while adhering to the parameters set in the methodology. The highest recovery was seen in run 19 where recovery was 92% and conditions were a pH of 2, time 6 hours, temperature of 45℃ and roasting temperature of 500℃. The lowest recovery of 28,03% in run 20 occurred when temperature was 45℃, roasting temperature was 900℃, time was 6 hours, and pH was 2.

Ionic liquid iron recoveries

The recoveries of iron when leaching with the ionic liquid, the highest recovery occurred on run 3 (5,36%) when the roasting temperature was 500℃ and the temperature was 60℃, concentration was 5% and time 6 hours. In contrast the lowest recovery happened at run 4 (0,32%) when the roasting temperature was 900℃ where temperature was 60℃, concentration 2,5% and time 6 hours.

Ionic liquid copper recoveries

The recoveries of copper when the ionic liquid was used as the lixiviant. It can be seen that the highest recovery occurred at run 17 (95,9%) when the temperature was 45℃, time 6 hours, concentration 2,5% and roasting temperature 500℃. The lowest recovery in comparison was achieved in run 18 when roasting temperature was 900℃, time 6, concentration 2,5% and temperature were 45℃ where recovery was 45,24%.

According to the article by Rodríguez and others in 2020 [14] the recoveries obtained fall in the realm of possibility. In the article it was seen that when using the ionic liquid at a concentration of 20% (v/v), temperatures of 90℃ and sodium bromide is added at 100g/ton the recovery gotten was 83,6% when the chalcopyrite was leached for 48 hours.

IV. Conclusion

In conclusion it can be deduced that ionic liquids can be substituted for the leaching of roasted chalcopyrite. It must be noted, however, that the leaching was aided by a complexing agent (sodium bromide) which adds to the overall cost this route will take. The results can then be extrapolated upon using the box Behnken design to identify various relationships and optimal conditions. Therefore, one must weigh out all the variables, these being that ionic liquids have higher costs and often require complex synthesis, they are very viscous which can hinder large scale operations and often if not pure results achieved can alter. If adhered to, however, and process seen as feasible then the route of leaching with the ionic liquid can be used offering its own advantages than that of common lixiviants.

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