



Research article

Magnesium recovery from ferrochrome slag: kinetics and possible use in a circular economy

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ABSTRACT

The ever-increasing demand for ferrochrome alloys has resulted in a substantial accumulation of ferrochrome slag by-products in many mining areas. On the other hand, the ferrochrome slag has been identified as one waste material that is rich in magnesium (Mg) and has not been effectively exploited. Beneficiating of ferrochrome slag (FCS) waste material is envisaged as a means of achieving sustainable recovery of Mg. Previous studies have used sulphuric acid as a lixiviant for leaching FCS at moderate temperatures to recover Mg. In this study, the recovery of Mg from ferrochrome slag was investigated using hydrochloric acid (HCl) as the lixiviant at low temperatures. Previous studies have shown that various metal oxides have been proven to be more amenable to leaching using HCl. This study examined the effects of acid content, leaching temperature, and reaction time on the recovery of Mg from FCS. Kinetic and thermodynamic analysis of the leaching process were also investigated as these are critical factors for maximum extraction of the Mg. The results showed that the highest recovery of Mg of 88.2% was obtained from FCS using 5 M HCl with a solid to liquid ratio of 1:10, mixing intensity of 250 rpm, reaction time and temperature of 150 min and 70 °C, respectively. The shrinking core model (SCM) was used in kinetic analysis to find the experimental data's best fit. A linear relationship was obtained with the coefficient of determination for the chemical reaction model (K_c) of >0.9 which indicates a good fit. The activation energy obtained for the diffusion and chemical reaction models were 95.44 and 41.45 kJ/mol, respectively, demonstrating that the rate-limiting phase is the one involving the chemical reaction.

1. Introduction

Magnesium (Mg) is a critical metal for highly specialized fields that include aerospace, military, and other technological applications. It is the lightest known structural metal which makes it cost-competitive for its use in the aforementioned applications (Zhang et al., 2019). The Mg has also found wide applications in agriculture specifically in the synthesis of fertilizers as it is a secondary nutrient critical for chlorophyll synthesis (Ostara, 2022). At the moment, population growth and technological advancement have propagated the demand with a perennial increase in the need for Mg. Consequently, in addition to primary Mg sources such as magnesite ($MgCO_3$), dolomite $CaMg(CO_3)_2$, and seawater, it is imperative to exploit waste sources that are rich in Mg to meet the growing demand (Özdemir et al., 2009; Raza et al., 2013). Ferrochrome slag (FCS) is a potential source of Mg that is abundant in Southern Africa. It has

entrained valuable metals which include 20–25wt% Mg which makes it a viable secondary source of the metal (Dube et al., 2018).

It is actually noted that Southern Africa has the largest resource of metallurgical grade chromite ore in the world. A substantial amount of these reserves lie along the Great Dyke in Zimbabwe (Mashanyare and Guest, 1997). A smelting technique is used to create ferrochrome alloys from chromite ores, and for every tonne of ferrochrome alloy produced, about 1.2 tonnes of FCS are typically created. The chemical makeup of the chromite ore, feedstock, amount of ash in the coke, and other reducing agents used typically determine this, though (Dube et al., 2018). As a result of its strength, durability, and chemistry, FCS is no longer seen as a waste residue merely to be discarded, but as a potential economic secondary source (Binici and Aksogan, 2011; Bidari and Aghazadeh, 2015). Inherently, FCS has drawn interest in the fields of construction, glass ceramics, and recovery of valuable metals. However, there have

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been an array of quality concerns in its application in construction activities, particularly, as a concrete aggregate due to the presence of magnesia which makes the material susceptible to swelling problems and results in cracking of structures (Zhang et al., 2019). Consequently, pre-treatment of the FCS in order to remove the magnesia may be vital to improving the use of the FCS for construction purposes. The disadvantage of using FCS in the production of glass ceramics has been the need to add numerous additives, which raises the process's complexity and economic viability. To the best of the authors' knowledge, there are relatively few investigations on the recovery of magnesium from FCS. Most recent studies have concentrated on recovering Fe, Ni, Co, and Cr from FCS (Shen and Forssberg, 2003; Dube et al., 2018). Recovery of metals such as Mg from FCS allows producers to enter the lucrative metal markets and improve the bottom line for mining companies.

The extraction methods used in recovering metals such as Mg from slag materials are cheap compared to technologies adopted to recover Mg from primary sources which include the Pidgeon process and thermal reduction. This is due to the fact that primary recovery methods for recovering Mg in a pure state are energy-intensive as they are carried out at high temperatures. Recovering Mg using the leaching process and utilizing the leachate in its impure state for processes such as struvite precipitation is envisaged to improve the overall socioeconomics of the process. Struvite ($\text{NH}_4\text{MgPO}_4\cdot\text{H}_2\text{O}$) is a slow release mineral fertilizer with commercial value and contains Mg as a critical component. Chemical expenses, particularly Mg, which can account for up to 75% of total expenditures, have a significant impact on the price of struvite manufacturing (Bradford-Hartke et al., 2021). Raw seawater that has an estimated concentration of 1300 mg/L of Mg has been successfully utilized for struvite precipitation despite having other cations such as calcium which tend to result in unwanted side reactions (Shaddel et al., 2020). As shown in Table 1, FCS used in the current study has an average weight of 26.4 wt% of MgO in which the concentration of Mg is likely to surpass that of seawater if the leaching process is effective. In addition, it is imperative that locally Mg is easily accessible locally. Countries such as Zimbabwe are landlocked, but have vast FCS in landfills which are rich in Mg as shown in Table 1. As a result it will not be cost effective utilizing alternatives such as seawater as Mg source considering transportation costs. Therefore, readily available sources at low cost, should be investigated such as FCS for use in Zimbabwe.

The Mg bearing compounds have proven to be amenable to leaching using inorganic acids such as H_2SO_4 and HCl at low temperatures (Du et al., 2022). One of the uttermost challenges of leaching with inorganic acids has been high cost. However, this can be minimised by utilizing acids that can be regenerated. Recent research work has shown that pyrohydrolysis and oxyhydrolysis of chloride rich solutions can be effectively used to regenerate HCl (Mckinley and Ghahreman, 2017). The leached solution from FCS will be a rich chloride solution, therefore, has potential to be regenerated and significantly curtail the chemical costs. It has also been shown that silicate minerals which are a huge component of FCS are difficult to dissolve compared to oxides and hydroxides using inorganic acids (Engström et al., 2013). This is critical towards ensuring selective recovery of Mg through leaching. Leaching is a heterogeneous

reaction that happens at the boundary between the solid and liquid phases, to put it simply. The dissolution of mineral ore occurs at the diffusion layer that forms at the interface between the two phases (Mashifana et al., 2019). Studying the reaction kinetics and mechanisms of dissolution to predict the behavior of Mg in solution is essential to rationalize the leaching process (Özdemir et al., 2009). In this study, the effects of variation of lixiviant concentration, time, and temperature were also investigated. In order to identify the rate-limiting phase, it was crucial to study the activation energy needed for the leaching of FCS. Typically, a value below 40 kJ/mol indicates a diffusion-controlled process, while a value above 40 kJ/mol indicates a chemical reaction-controlled process (Teimouri et al., 2020).

2. Materials and methods

2.1. Slag raw material

The FCS was collected from Zimasco, Kwekwe in Zimbabwe. It was initially pulverized and sieved to 80% passing through 75 μm . The mineralogy of the FCS was determined using X-Ray diffraction and is shown in Table 1.

2.2. Leaching experiments

The temperature was kept constant with a difference of 0.5 °C during the leaching studies in a 1-liter tank with an overhead variable-speed mixer submerged in a water bath. The system was heated to the desired temperature while being constantly stirred until the temperature reached and remained at the specified degree. A fixed amount of FCS was used in all experiments thus maintaining a solid to liquid ratio of 1:10 with 500 ml of either 5 M or 6 M HCl used as the leaching solution. The HCl was chosen because previous studies have shown that various metal oxides have been proven to be more amenable to leaching using HCl as compared to sulfuric acid. The HCl was added gradually with the contents stirred at a constant rate of 250 rpm. A 5 ml sample was periodically withdrawn during a time interval of 30–180 min. The sample was diluted by 100 ml of deionized water and thereafter filtered. Thereafter, the filtrate was analyzed for Mg concentration using atomic absorption spectrometer (AA-GBC Dual). All experiments were done in duplicate and average values are represented. The Al and Cr recovered were analysed at the deduced optimal conditions.

The calculation for the recovery of metals (M) from FCS is given in Eq. (1).

$$\text{Metal recovery (M) (wt\%)} = \frac{\text{mass of M extracted}}{\text{Mass of M in ferrochrome slag}} \times 100\% \quad (1)$$

3. Results and discussion

3.1. Effect of stirring speed on magnesium recovery

Using a 14:1 liquid to solid ratio and a 6 M concentration of HCl, an experiment was conducted at 80 °C for 180 min to examine the effect of mixing on the recovery of magnesium. Figure 1 clearly shows that changing the stirring speed had a favorable impact on the Mg recovery, with a significant increase occurring between 150 and 250 rpm. After that, the Mg recovered stayed largely steady with no statistically significant fluctuation. The results show that the solid particles were adequately suspended and the H^+ ions were distributed appropriately at stirring speeds above 250 rpm. Therefore, a 250 rpm stirring speed was used in all future trials. To exclude stirring speed from the study's variables.

3.2. Effect of liquid to solid ratio on magnesium recovery

At 80 °C, 350 rpm of stirring, and 6 M of HCl, an experiment was conducted to determine the effect of the liquid to solid ratio on the

Table 1. Mineralogy of ferrochrome slag.

Compound	Average composition %
SiO_2	31.4
MgO	26.4
Al_2O_3	18.0
Cr_2O_3	14.3
Fe_2O_3	4.4
CaO	3.3
TiO_2	0.6
Other	1.6

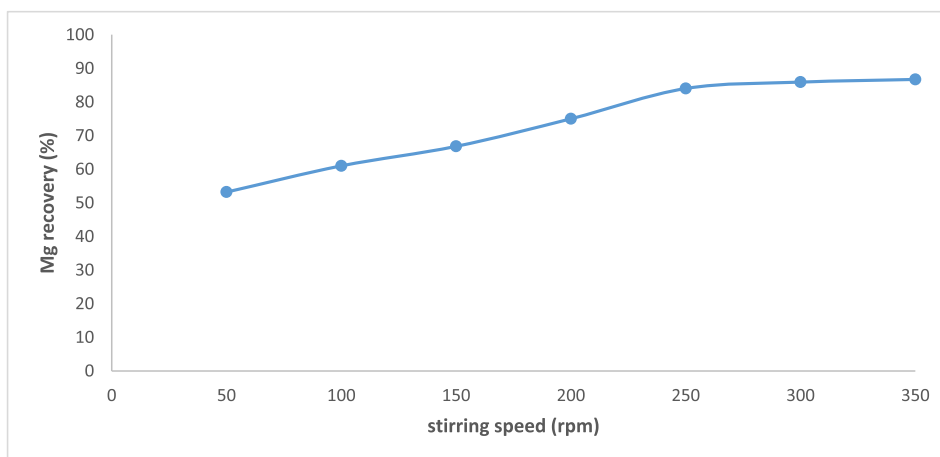


Figure 1. Effect of stirring speed on Mg recovery when leaching with HCl.

recovery of magnesium. Figure 2 demonstrates that the Mg recovery increased as the liquid to solid ratio rose. It was implied that a reduced mass transfer resistance at the liquid-solid interface due to a larger liquid to solid ratio would lead to an increase in the rate of leaching. In heterogeneous solid liquid reactions, the soluble reactants spread across the interface or through the porous solid layer. Then come chemical processes. The rate of the reaction is controlled by the reactant's diffusion through the solid product layer and solution boundary layer.

3.3. Effect of reaction time on magnesium recovery

Figure 3 illustrates statistically significant differences for both various reaction durations for the same concentration and for different concentrations for the same reaction time, demonstrating the importance of those conditions in the leaching process. Additionally, Figure 3 demonstrates that the Mg extracted increased significantly between 30 and 150 min, strongly indicating that the Mg recovered was dependent on the reaction time. The figure also demonstrates that from 30 to 90 min, there was initially a noticeable variation in the Mg recovered utilizing the two different doses of 5 and 6 M of HCl. More significantly, when leaching duration rose, the amount of Mg recovered increased as well. Mg recovery reached 88.2% after 150 min; an additional 15 min of leaching

time (bringing the total to 180 min) had no discernible impact on the amount of Mg extracted. As a result, 150 min was determined to be the ideal reaction time. In a study by Dube et al. (2018), Mg recovery was 66.9% under similar working conditions using sulphuric acid as the lixiviant, which is lower than when using HCl. Given that H^+ ions should be primarily responsible for the dissolution process, sulphuric acid containing H^+ ions should have a faster dissolution rate at the same acid concentration, however this was not the case according to the data given in Figure 3 when compared to a study by Dube et al. (2018). This could be explained by anions' propensity to form complexes on solid surfaces with varying stabilities, which alter the dissolution efficiency. In this instance, the chemical differences between sulphate and chloride ions may have been the deciding factor (Habbache et al., 2009).

3.4. Effect of temperature on Mg recovery

Figure 4 shows the dependency of Mg recovery on reaction temperature for a leaching time of 150 min. The graph shows that Mg does not easily dissolve at low temperatures and an increase in temperature increased the rate of dissolution of Mg. This is attributed to the exponential dependency of the rate constant in the Arrhenius equation with temperature. In addition, at higher temperatures, the collision of

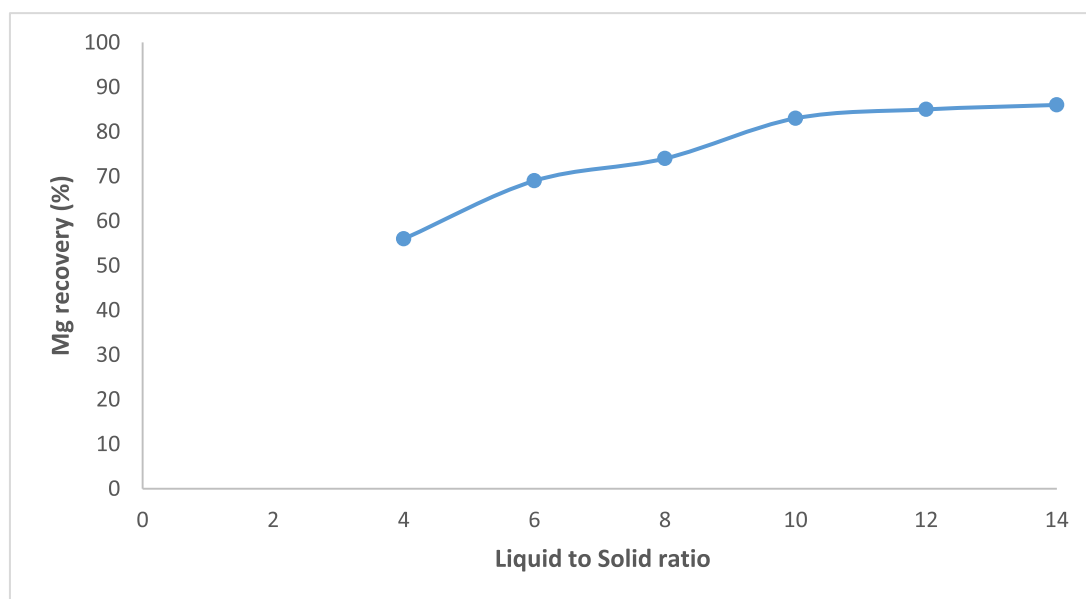


Figure 2. Effect of liquid to solid ratio on Mg recovery when leaching with HCl.

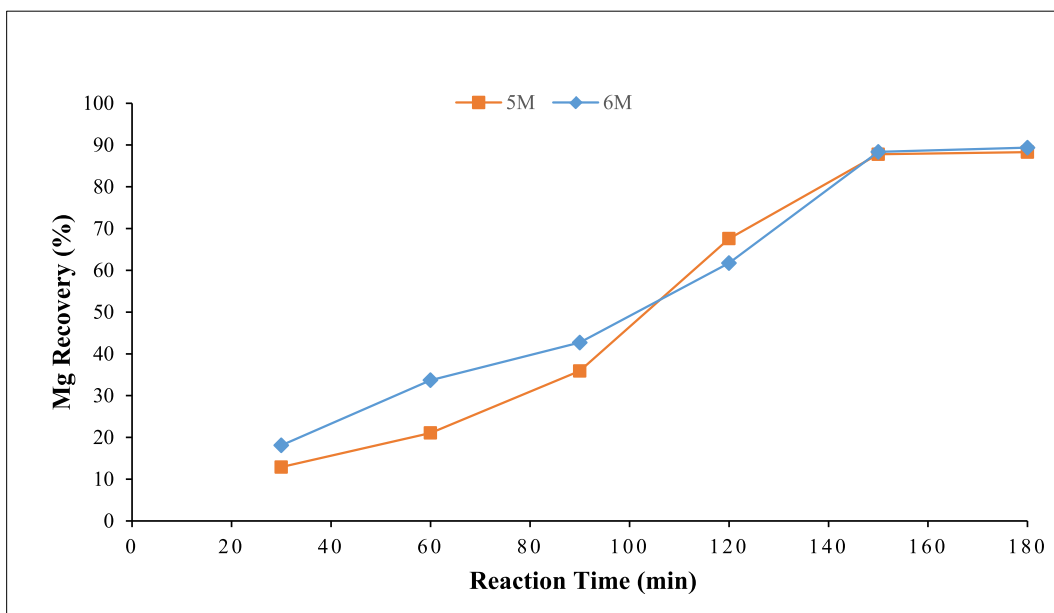


Figure 3. Effect of reaction time and hydrochloric acid concentration on magnesium extraction.

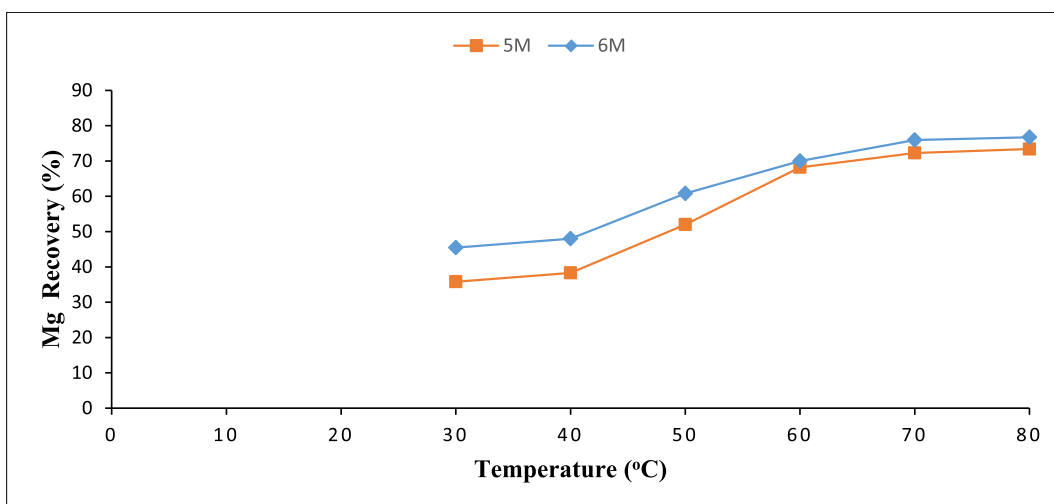


Figure 4. Effect of temperature on magnesium recovery.

reactants is more intensive and results in a faster reaction rate between the Mg bearing compounds and the leaching solution. At 80 °C formation of a gelatinous substance was observed which could be due to precipitation of silica as the FCS has a high silica content. The dissolution of the silica increases with temperature, but since silica is sparingly soluble it precipitates from solution forming the gel. According to Mubarok and Adi Kurniawan (2015), the gel has a tendency of adsorbing Mg in the solution and, therefore, reducing the Mg recovered. Consequently, the desired operating temperature was taken as 70 °C.

3.5. Kinetic analysis

The shrinking core model (SCM) was used so as to understand the kinetics of the leaching process. This is an effective model for the analysis of heterogeneous reactions which involves the leaching of metal from a solid-liquid interface. The model approximates real particles during the dissolution process more clearly than other conversion models (Mashifana et al., 2019; Habbache et al., 2009). As a result, the shrinking core

model was used to examine the kinetic data of the leaching of FCS while applying the usual Eqs. (2), (3), and (4) (Teimouri et al., 2020) for diffusion, chemical reaction, and mixed control, respectively. The chemical reaction model is as follows;

$$1 - (1 - X)^{\frac{1}{3}} = k_c t \tag{2}$$

where X is the fraction reacted; k_c is the chemical rate constant, and t is the reaction time.

If diffusion through the solid layer made up of an insoluble core encircling the unreacted core controls the reaction rate, the integral rate expression for the diffusion control model can be written as:

$$1 - \frac{2X}{3} - (1 - X)^{\frac{2}{3}} = k_d t \tag{3}$$

where k_d is the pore diffusion rate constant.

A variation based on both the chemical reaction and diffusion (mixed control model) across the interface was used to further examine the

leaching kinetics and determine if any other model might have a better fit. The integral rate expression for the mixed control model is represented as:

$$1 - \frac{2X}{3} - (1 - X)^{\frac{2}{3}} + Bw(1 - (1 - X))^{\frac{1}{3}} = k_m t \tag{4}$$

where k_m is the pore diffusion rate constant, B is the ratio of k_c / k_d .

The coefficient of determination, R^2 for the SCM for the chemical reaction model was above 0.90 for all the plots shown in Figure 5. This shows that the model is a good fit. Figure 6 shows the plots for the pore diffusion-controlled model with R^2 values ranging between 0.82 - 0.94. However, most of the correlation coefficients except one are less than those for the chemical reaction model plot. As for Figure 7, only plots at 30 °C, 40 °C and 50 °C have correlation coefficients above 0.90 with the coefficient of determination at 70 °C being the lowest at 0.5645. This implies that the mixed control model cannot be adequately used to describe the leaching of Mg from FCS at temperatures above 70 °C. It is noted from Figures 6 and 7 that the correlation coefficients of the chemical reaction and diffusion model plots have values closer to 1 which depicts a linear relationship and also entails the plots are a good fit. However, in this study, chemical reaction model can be considered as the most appropriate fit of the leaching process of FCS. It should also be mentioned that the magnesium that is leaving behind creates a porous structure that may prevent the exposure of further layers. The fluid combination and the development of inert solid patches that passivate a portion of the mineral surface may be rate-limiting factors in the kinetics.

Therefore, the chemical reaction model was shown as the best model at varying temperatures against the other two models.

3.6. Activation energy

A chemical reaction's pace and activation energy are closely connected. In this study, the chemical reaction model was determined to be

the most appropriate fit of the leaching process of FCS. In other words, the chemical reaction model was found to be the rate limiting step. The Arrhenius equation was used to calculate the activation energy because the slower the chemical reaction, the larger the activation energy. The Arrhenius equation describes the interdependence of the rate of reaction (k) on temperature (T). A common form of the equation is given by Eq. (5)

$$k = k_o e^{-\frac{E_a}{RT}} \tag{5}$$

where k_o , E_a , R and T are the pre-exponential factors, activation energy, gas constant, and the reaction temperature, respectively. A more useful form of the relation is obtained by taking the logarithmic of both sides of the equation to give Eq. (6).

$$\ln k = \ln k_o - E_a/RT \tag{6}$$

For the diffusion and chemical reaction models in this investigation, the logarithm of the apparent rate constant ($\ln k$) was plotted against the inverse of the corresponding temperature ($1/T$), as indicated in Figure 8.

The slopes shown in Figure 8 are steep and negative, implying high activation energy for the chemical reaction model. The magnitude of the activation energy (E_a) deduced from the gradient of the plots shown in Figure 8 were 95.44 and 52.05 kJ/mol for diffusion and chemical reaction processes, respectively. Typically a value above 40 kJ/mol indicate a chemical reaction controlled process (Simate, 2009; Teimouri et al., 2020).

3.7. Chemical costs of leaching ferrochrome slag and using it as a magnesium source

A key portion of the operating costs is the cost of the chemicals. It is deterministic of the procedure's economic viability and makes up a significant portion of the whole leaching process. In order to do an economic analysis, the chemical costs based on the stoichiometric amounts required for leaching FCS as stated in Table 2 were taken into account. The total cost for each case was calculated per kg of FCS.

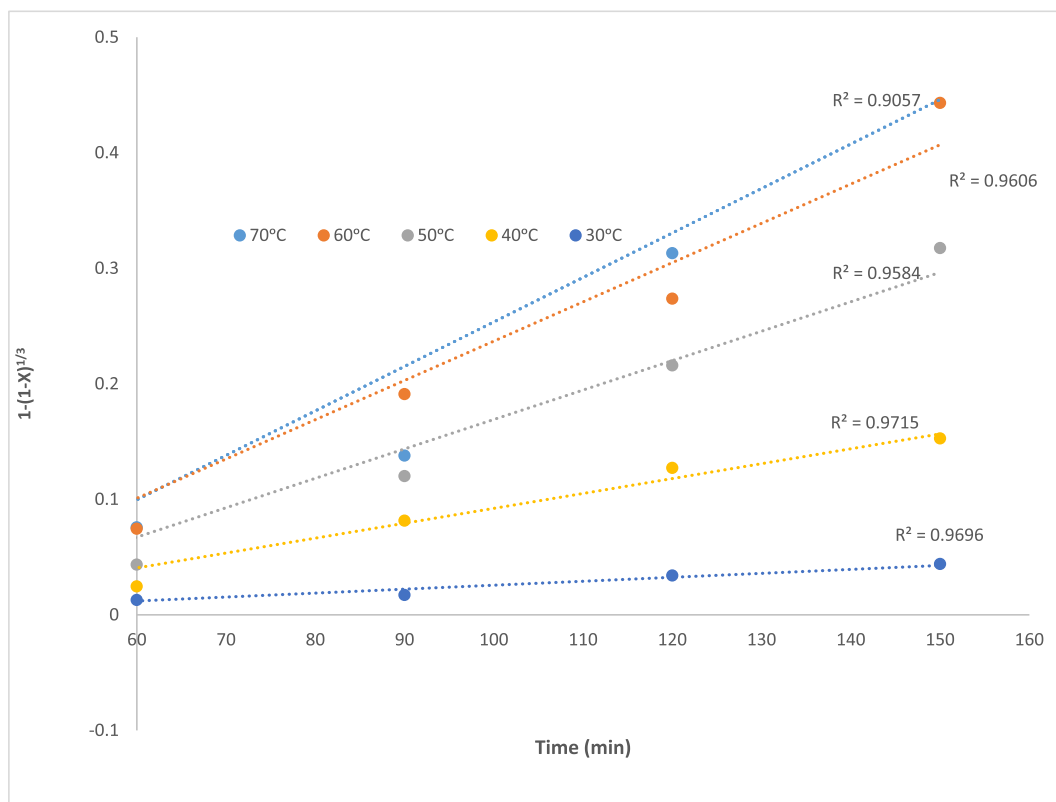


Figure 5. Kinetic plot showing the rate constant, k_c , at different temperatures.

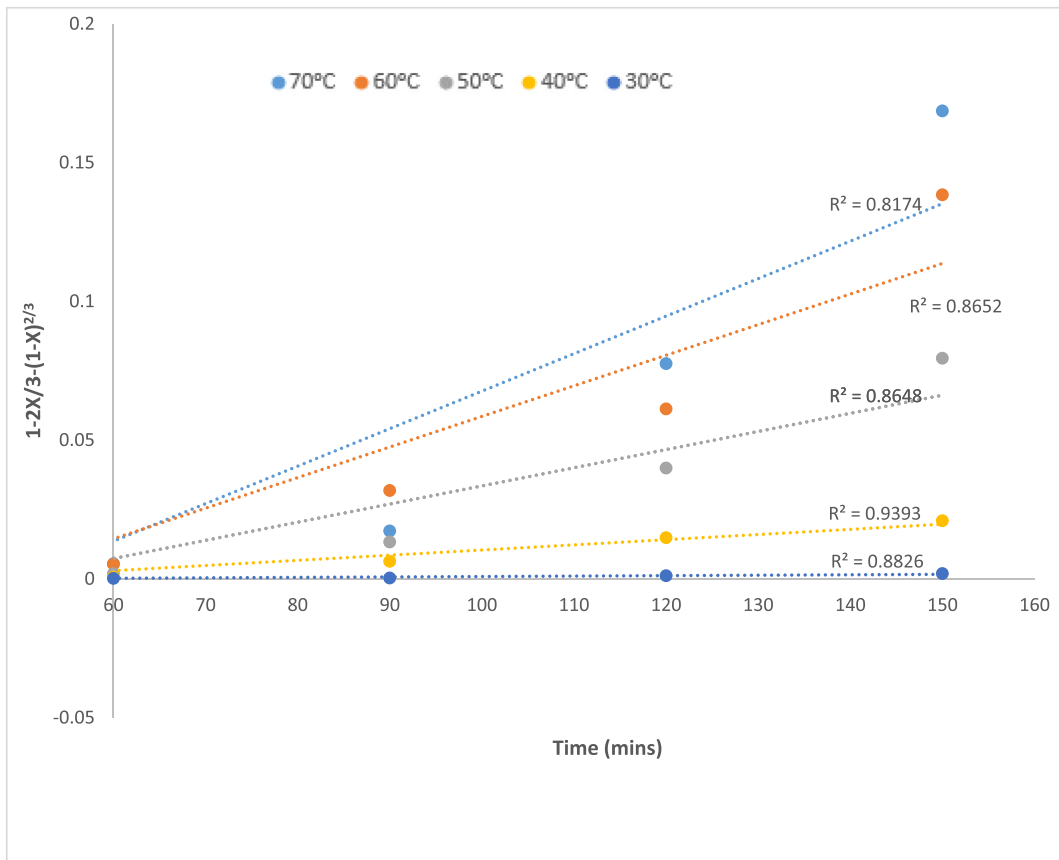


Figure 6. Kinetic plot showing the rate constant, k_d , at different temperatures.

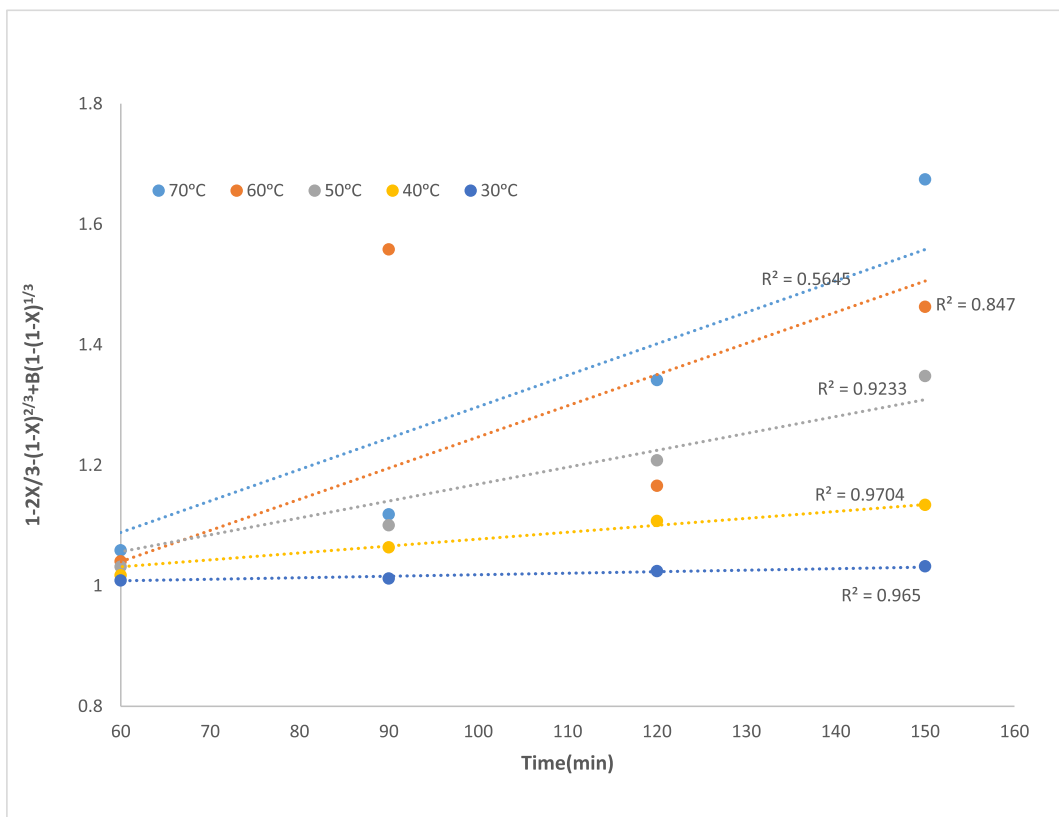


Figure 7. Kinetic plot showing the rate constant, k_m , at different temperatures.

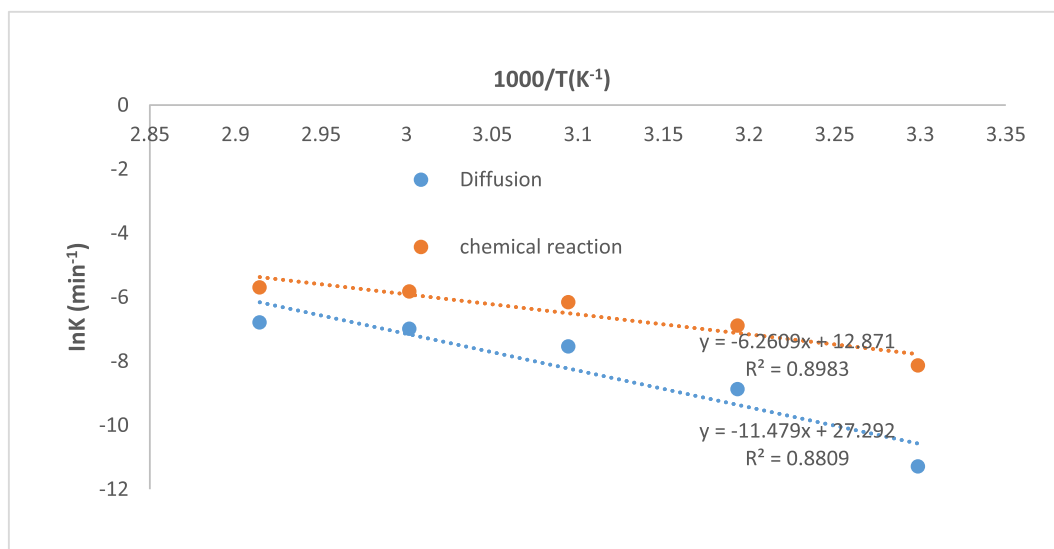


Figure 8. Arrhenius plot for leaching of magnesium from ferrochrome slag.

Table 2. Chemical costs for leaching ferrochrome slag using HCl.

The unit price of magnesium source (US\$) per kg ^a	HCl: 1.03 FCS: Free
Magnesium source requirement for 1 m ³ wastewater ^b (kg·m ⁻³ wastewater)	FCS:1 kg
	HCl: 0.48 kg
Chemical cost of HCl (US\$ per kg of FCS leached)	0.4944

^a Transportation and capital costs are not included.

^b Chemical costs were obtained from ACOL Chemicals [Holdings.com](https://www.acolchemicals.com) in Zimbabwe.

The chemical costs for leaching 1 kg of ferrochrome slag were deduced to be US\$0.4944. The chemical costs of leaching ferrochrome slag are less than those of recovering magnesium from ores as ferrochrome slag is assumed to be free. However, magnesium ores will have to be mined and undergo processes such as milling thermal reduction mainly the pigeon process. As both alternatives use high temperatures to operate, these thermal processes are mostly criticized for their hazardous emissions and large energy requirements.

3.8. Potential use of ferrochrome slag as magnesium source

The leached solution is envisaged to have impurity metal ions, based on the characterization of the solid samples oxides of Si, Al, Cr which are also amenable to leaching with HCl. Si will mostly be present in solution as silicic acid compounds, but as the pH rises, Mg(OH)₂ can be used to precipitate Si as insoluble silicates. The efficiency, rate constant, purity, and shape of struvite are changed when sea water is used as an alternate magnesium source for struvite precipitation (Bradford-Hartke et al., 2021). However, under constant pH conditions these variations can be minimised. On the other hand, the 88.2% of Mg recovered from FCS at optimal conditions translates to 13.94 g/L of magnesium in solution which is more than the average sea water concentration of 1.3 g/L utilized as an alternative for struvite precipitation. Similar to seawater, the leached solution from FCS will have ions such as calcium which can hinder the formation of struvite. The impact of such ionic impurities on the quality and yield of struvite will have to be investigated in future studies. On the contrary, sea water has a higher alkalinity whereas the FCS leached solution is acidic and struvite precipitation requires alkaline conditions, which means there will be additional chemical costs for pH adjustment. However, ferronickel slag was used as a source of magnesium in a work by Kwon et al. (2019) for the precipitation of struvite. It

was concluded that employing magnesium chloride as the magnesium source for struvite precipitation was more expensive than using the leached solution from ferronickel slag. Therefore, it is anticipated that when FCS leached solution is utilized for struvite precipitation, comparable findings will be obtained. Most notably, FCS can be utilized as a substitute source of magnesium for the precipitation of struvite because it is susceptible to hydrochloric acid leaching and because enough magnesium is present in the leached solution. Even though it can be utilized right away as a building material, the leached slag residue has a lower potential value.

4. Conclusions

The study revealed that it is feasible to recover a substantial amount of Mg from FCS through leaching with HCl at moderate temperatures. With an increase in reaction time and temperature, Mg recovery from FCS increased. The kinetic analysis of the leaching process was easily understood by the SCM. In the leaching of FCS using HCl, it was discovered that the chemical reaction model with a linear connection for the plot and correlation coefficients above 0.90 for all temperatures studied was an appropriate model. The apparent activation energies for the chemical reaction and diffusion-controlled processes were 95.44 and 52.04 kJ/mol, respectively, demonstrating that the chemical reaction step was what constrained the leaching process. The chemical costs of leaching ferrochrome slag and using it as a source of magnesium for struvite precipitation are lower than using magnesium chloride indicating the process may be more economically feasible if capital costs are also investigated.

Declarations

Author contribution statement

L.B. Moyo: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

G.S. Simate; T.A. Mamvura: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

Data will be made available on request.

Declaration of interest's statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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