



RESEARCH ARTICLE

Effect of stirring device on CuO dissolution by glycine

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ABSTRACT

In this study the leaching of CuO particles by glycine, an environmentally friendly leachant, was investigated under different conditions. It was found that the variation of glycine and NaOH concentrations as well as CuO loading influenced slightly the dissolution of CuO while temperature, the volume of solution and the nature of stirring impacted greatly the dissolution process. Magnetic and mechanical stirring registered inverse effects. In fact, increasing the volume of solution from 105 to 405 mL decreased the dissolution of CuO from 100% to 26.2% registered after 180 min respectively under magnetic stirring, while it increased from 43.5% to 91.7% respectively under the same conditions with mechanical stirring. Thus, the nature of stirring is a crucial parameter that may radically change the dissolution results. The dissolution was found to be controlled by chemical reaction.

Keywords: CuO particles, glycine, dissolution, hydrodynamics, stirring device

1. INTRODUCTION

Copper is one of the most studied metals in the literature because of its large utilization in domestic and industrial fields [1, 2]. In hydrometallurgy, lot of research is conducted to optimize copper extraction from ores or secondary resources using different leaching reagents. However, many of these reagents pose potential environmental risks [3, 4]. Nowadays, efforts are made for the implementation of sustainable processes through the use of environmentally benign and renewable products such as organic acids and amino acids [5-8].

Glycine is a natural product which is non-toxic, non-volatile and non-flammable. In human body, glycine acted as precursor to proteins and is found in high concentrations in the skin, connective tissues of the joints and muscle tissue and in bone broth. Glycine is produced relatively cheaply. It is used in food, metal plating, animal feed, pharmaceutical industries, for the production of glyphosate herbicide, fertilizers and explosives [9, 10].

In the literature, several studies were reported dealing with the leaching of copper from different sources using amino acids. Tanda et al. [9] studied the

leaching of copper oxide from different ores. They found that the optimum leaching conditions are pH 11 and glycine to copper ratio of 4:1 thereby they extracted 95%, 91%, 83.8%, and 17.4% of copper after 24 h from azurite, malachite, cuprite and chrysocolla respectively. Oraby and Eksteen [11] studied the selective leaching of copper from a gold-copper concentrate in glycine solutions in the presence and absence of peroxide. They found that 98 % of copper can be selectively leached in glycine-peroxide solution in 48 h at ambient conditions and a pH of 10.5-11. Feng and Deventer [12] studied the role of amino acids such as glycine, L-valine, DL- α -alanine, and L-histidine in the ammoniacal thiosulphate leaching of a pyrite concentrate. They found that amino acids enhanced the leaching of copper species from the pyrite concentrate and that thiosulphate consumption decreased from 12.6 kg t⁻¹ to 6.6 kg t⁻¹, 6.3 kg t⁻¹, 5.2 kg t⁻¹ and 4.5 kg t⁻¹ after 24 h with the addition of 10 mM L-valine, glycine, DL- α -alanine and L-histidine, respectively.

Mixing operations are encountered widely throughout productive industry in processes involving physical and chemical change. Many sectors carry out mixing operations on a large scale such as in food,

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pharmaceutical, paper, and plastics industries [13]. As a result, the financial investment in both the capital and running costs of mixing processes is considerable. In fact, it was estimated that the cost to the process industries due to an inadequate understanding of mixing varied from 1 to 10 billion dollars per year [14].

Mixing is carried out usually to increase the homogeneities, and to enhance the rate processes. Understanding how the solids and liquids move to achieve these requirements is an essential prerequisite for a successful mixer design [13, 15, 16]. In the field of hydrometallurgy, metal dissolution testing is a fundamental part of mineral extraction and manufacturing. Tests are usually focused on the effect of parameters such as concentration, liquid/solid ratio, temperature and stirring speed. These parameters are often investigated with only one mixing device. Testing the nature of mixing may bring useful data that can be further exploited for any leaching system. Only limited information on the impact of mixing equipment on leaching process was generated to date in hydrometallurgical field [17].

The work described in this paper is an attempt to address the factors governing the dissolution process of pure CuO particles by glycine with a particular attention given to the effect of stirring device.

2. MATERIALS & METHOD

Powder of CuO (black color) with a mean size of 3 μ m was purchased from Fluka ($\geq 98\%$). Glycine (99%, Fluka) and NaOH (99%, Biochem) were used as received. Distilled water was used for the preparation and washing in all experiments. In a typical experiment 0.05 g of copper oxide (CuO) was contacted at 25 °C with a solution containing glycine and NaOH. The mixture was magnetically stirred. During CuO dissolution a deep blue solution is produced indicating the formation of copper (II)-glycinate complexes which are stable over a broad pH range (2.8-12) [9, 18, 19] and can be analyzed by molecular spectrometry. In this study a JENWAY 6705UV/VIS spectrometer was used at 640 nm found as the maximum absorbance of the complexes formed. Cupric sulphate pentahydrate (98%, Fluka) was used at different concentrations to generate a calibration curve (Fig.1). In this work, different parameters were investigated such as the concentration of glycine and NaOH, CuO loading, temperature, and stirring device (magnetic and mechanical). The results are presented below.

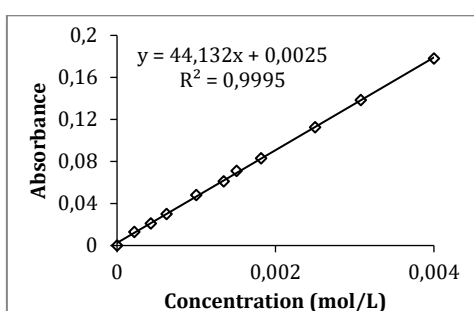
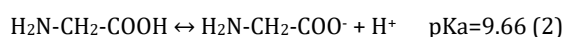
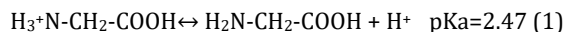


Fig 1. Calibration curve

3. RESULTS & DISCUSSION

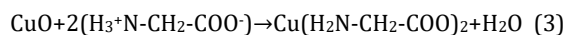
3.1. Effect of the operation conditions on CuO dissolution

The glycine molecule ($\text{H}_2\text{N}-\text{CH}_2-\text{COOH}$) contains carboxylic and amino functional groups and is itself amphoteric. In aqueous solutions it can be in two forms depending on the pH [9]:



The amine group is protonated at pH below 2.47 and glycine is in an anionic form at pH above 9.66. In the intermediate interval the neutral form of glycine is in equilibrium with the zwitterion $\text{H}_3^+\text{N}-\text{CH}_2-\text{COO}^-$.

The effect of glycine concentration was studied by contacting 0.05 g of CuO with 200 mL of glycine at different concentrations: 0.1-0.5-1 and 1.5 M at 25 °C and 250 rpm. To each solution 5 mL of NaOH at 0.5 M were added ($V_{\text{total}}=205$ mL) thereby the pH of the solutions was 8.9. At this pH value it is the zwitterion which is the predominant specie. Thus, it can be supposed that the reaction of CuO dissolution occurred as follows:



The results show that the extent of CuO dissolution increased slightly with increasing glycine concentration. It attained 12.3-17.6 -23.3 and 29% after 1 min of contact and increased to 70.9- 79.7- 92.2 and 96.8% after 180 min with 0.1-0.5-1 and 1.5 M respectively (Fig.2a).

The effect of CuO loading was studied by testing 3 values 0.03-0.05 and 0.07 g. The other parameters were kept constant ($C_{0(\text{glycine})} = 0.1$ M, 25 °C and 250 rpm). The results (Fig. 2b) show three curves fused together up to 90 min beyond which the effect was more visible. The dissolution rate decreased with increasing CuO loading attaining 58.1-70.9 and 77.6 % with 0.07 g-0.05 g and 0.03 g respectively after 180 min.

The same slight effect was also observed when the concentration of NaOH was varied (Fig.2c) while maintaining the other parameters constant ($C_{0(\text{glycine})}=0.1$ M, $m_{\text{CuO}}=0.05$ g, 25 °C and 250 rpm) reaching 66.8 % with 0.1 M and 83.4 % with 2M after 180 min.

Figure 2d shows the effect of temperature (25-30-40-50 and 60 °C) tested under the conditions ($C_{0(\text{glycine})}=0.1$ M and $m_{\text{CuO}}=0.05$ g and 250 rpm). On the contrary of the previous parameters, temperature had an important effect on the dissolution of copper oxide. At 25 °C it reached 70.9% after 180 min and at 60 °C total dissolution was obtained after only 20 min of reaction.

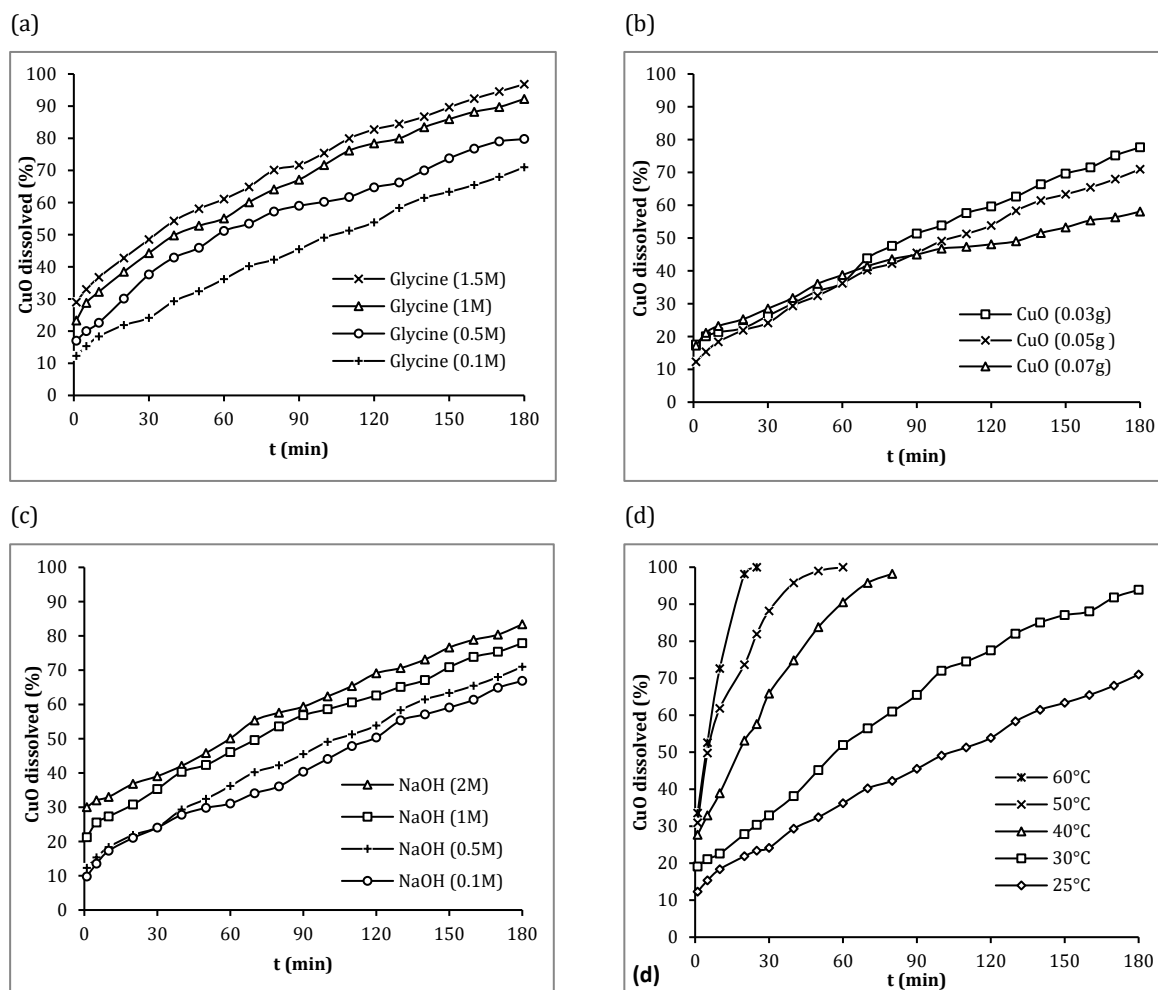


Fig 2. Effect of glycine concentration (a), the amount of CuO (b), NaOH concentration (c) and temperature (d) on the rate of CuO dissolution by glycine

In a previous study we investigated the leaching of CuO from CuO/ α -Al₂O₃ catalyst in the presence of different acids (HCl, HNO₃, H₂SO₄, and C₆H₈O₇) [20]. It was found that hydrochloric acid yields the highest CuO dissolution efficiency, followed by sulfuric acid, nitric acid and citric acid. Citric acid was the weakest leachant because of the absence of aggressive anions in the systems (Cl⁻, SO₄²⁻, NO₃⁻) which were found to be effective in the dissolution process. The time for total dissolution was about 14 min with inorganic acids at ambient temperature using an acid concentration of 0.5 M at 25 °C while 2 h at 80 °C were necessary to reach the same dissolution efficiency with citric acid solution. In this study the dissolution of CuO by glycine at 0.1 M was total after 25 min at 60 °C but took up to 3 h at ambient temperature. However, despite their lower efficiencies compared to mineral acids, the use of organic leachants such as glycine or citric acid remains more interesting from economical and environmental point of views.

3.2. Effect of mixing device

Two kinds of stirrers were used to homogenize the solutions (magnetic and mechanical). The mechanical stirrer was formed by a pitched blade turbine (5 cm of diameter and inclined with 45°) while magnetic stirring was carried out with a magnetic bar with 2 cm

in size. Mechanical stirrers or impellers are often used in industry. They are classified in two general types; axial flow and radial flow. Axial flow impellers impose essentially bulk motion to the fluid. They are very useful in mixing solid-liquid suspensions. They promote a downward velocity profile and are characterized by low shear and high axial dispersion, they create good top to-bottom motion in a tank, which results in good mixing. Radial impellers discharge fluid radially outward to the vessel walls and impose essentially shear stress to the fluid. Mechanically agitated tanks have been studied over a wide range of laboratory and industrial conditions, in order to improve the agitation energy efficiency [21-27] but less in hydrometallurgical field to optimize the extraction efficiency of minerals. In this study a pitched four blade turbine (axial flow) was used. A pitched blade turbine consists of a hub with an even number of blades bolted and tack-welded on it [13]. The blades can be at any angle between 10 and 90° from the horizontal [28-30], but the most common blade angle is 45° as that used in this study.

3.2.1. Effect of stirring speed

The effect of stirring speed was studied with both mixing devices by testing three values: 100-250 and 400 rpm while maintaining the other conditions

constant ($C_{0(\text{glycine})}=0.1 \text{ M}$, $m_{\text{CuO}}=0.05 \text{ g}$, $25 \text{ }^\circ\text{C}$ and 205 mL). In both cases, increasing stirring speed led to improve the dissolution rate. It attained 55.8-70.9 and 92.7 % with magnetic stirrer (Fig. 3a) and 50.2-57.2 and 80 % with mechanical one (Fig. 3b) at 100-250 and 400 rpm respectively after 180 min.

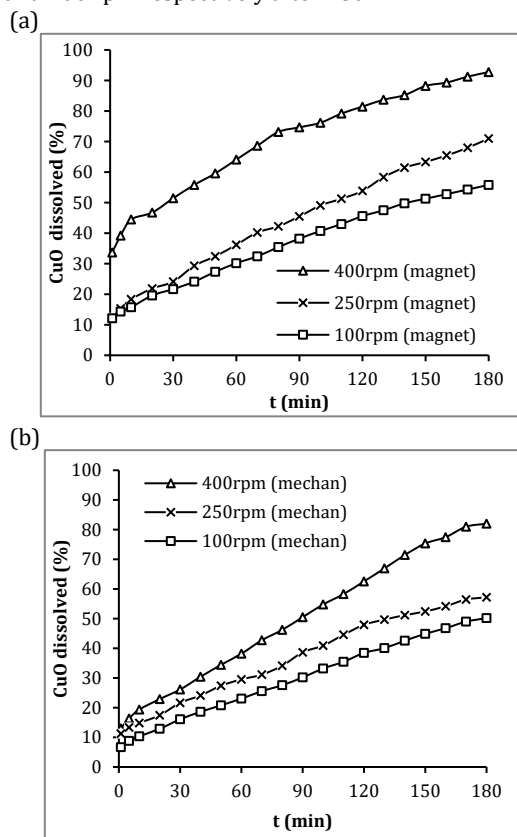


Fig 3. Effect of magnetic (a) and mechanical (b) stirring on CuO dissolution by glycine

3.2.2. Effect of the volume of solution

The volume of solution inside which the reagents react together may influence the rate of the reaction. In this work, the effect of the volume of solution was studied by maintaining the mol numbers of the reagents constant (0.02 mol of glycine and $6.28 \cdot 10^{-4} \text{ mol}$ of CuO (0.05 g)) and varying the volume of water ($100\text{-}200\text{-}300$ and 400 mL). To each volume of solution 5 mL of NaOH at 0.5 M were added ($2.5 \cdot 10^{-3} \text{ mol}$). The dissolution reaction was studied with both mixing devices at a constant stirring speed of 250 rpm and 25°C .

a. Magnetic stirring

A magnetic bar of 2 cm in size was used. The results show that the rate of dissolution increased when the volume of solution decreased (Fig.4a). In fact, total dissolution was achieved with 105 mL after 120 min while only 26.2% were dissolved with 405 mL after 180 min . These results can be explained by the increase in the frequency of reactive collisions between the reagents occurring in small volumes resulting in the acceleration of the dissolution reaction. An induction period (where no dissolution occurred) of 30 min was observed with 405 mL .

- Effect of the size of the magnetic bar

In magnetic stirring, the size of the bar may influence the rate of the chemical reaction. To investigate this parameter, a second magnetic bar with 6 cm in size was tested and the results were compared to those of 2 cm . The longer bar obviously gave the best results (Fig. 4b). With the volume of 105 mL , total dissolution was obtained after 75 min with bar 6 cm and after 120 min with bar 2 cm . With 405 mL , 72.5% and 26.2% were reached with bars 6 cm and 2 cm respectively after 180 min . On the other hand, it can be observed that plots obtained with bar 6 cm were closer to each other than those obtained with 2 cm and no induction period was observed in this case. The bar 6 cm involved higher turbulence than bar 2 cm so that the decrease in the volume of solution impacted less the dissolution results.

b. Mechanical stirring

The effect of the volume of solution was investigated with mechanical stirrer under the same conditions used for magnetic one. The mechanical stirrer was centrally located and placed in the middle height of the solutions.

The results show that on the contrary of magnetic stirring, increasing the volume of the solution led to increase the dissolution of copper oxide (Fig. 5). In fact, after 180 min , 91.6% of CuO were dissolved with 405 mL and 43.47% with 105 mL . During the experiments, it was observed that CuO particles settled at the bottom of the vessel and that increasing the volume of the solution led to partially disperse the particles into the bulk. In the literature, it was reported that there is a location under the paddle in the central position of the vessel where the velocity of the fluid is minimal leading to accumulate the particles in the form of aggregate [31]. The formation of agglomerate reduced the surface of the particles exposed to the fluid and hence decreased the dissolution rate. On the other hand, CuO density (6.31 g cm^{-3}) is important and needs high turbulence to maintain the particles in suspension.

The increase in the dissolution rate of CuO with increasing the volume of solution under mechanical stirring may be explained by the fact that an important volume of water may remove more efficiently the aggregate located under the paddles and drag the particles to the main stream where the turbulence is higher increasing consequently the dissolution rate. In a reduced volume, the capacity to move the same amount of solid is lowered leading to settle the particles as aggregate and decrease the dissolution rate.

- Effect of the position of the impeller inside the vessel

The impeller positioning is an important factor impacting hydrodynamics. In order to study this parameter, the impeller was disposed at different heights measured from the base of the vessel: 1 cm , 4.5 cm and 7.5 cm (Fig.7). The experiments were carried out with 205 mL (total height of the liquid $=8.5 \text{ cm}$).

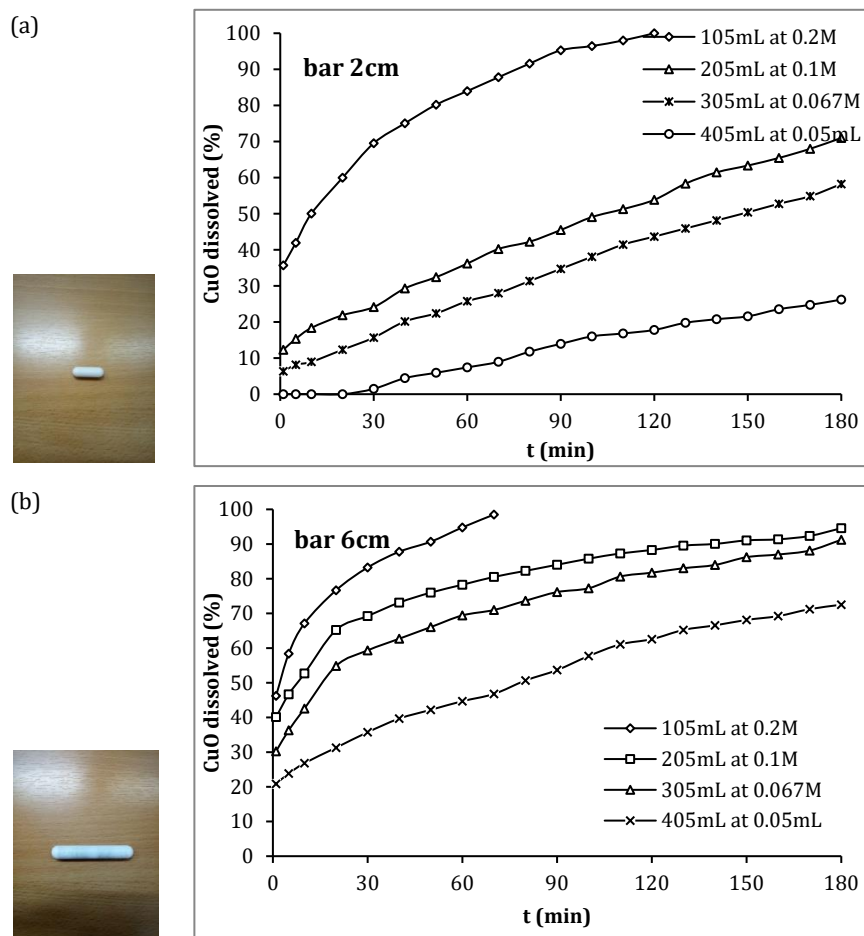


Fig 4. Effect of magnetic bar size 2 cm (a) and 6 cm (b) on CuO dissolution by glycine

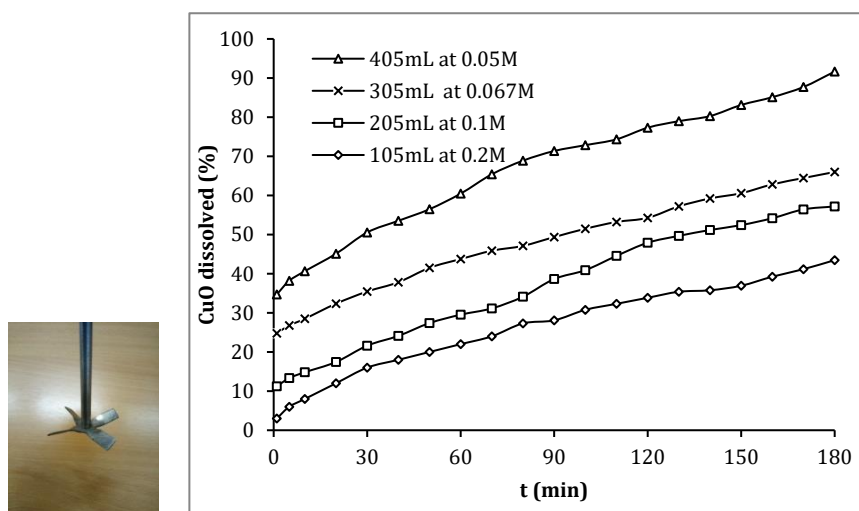


Fig 5. Effect of mechanical stirring on the dissolution of CuO by glycine

The results show that the lowest dissolution efficiency (50.3%) was obtained when the paddles were close to the upper surface (7.5 cm). The dissolution was slightly improved (57.2%) when the paddles were placed very close to the bottom of the vessel (1 cm) while the best results were obtained at middle height (4.5 cm) reaching 76.7% after 180 min (Fig. 6).

In the literature, the suspension of particles in tanks stirred by different types of impeller was investigated. It was reported that axial flow impeller generates an axial jet that sweeps the vessel bottom of settled solids and that the flow at the bottom is mainly directed outward [27]. When the distance of the impeller-bottom increased, a flow transition occurs. The outflow of the impeller becomes more radial and

the jet from the impeller is directed towards the vessel wall. The flow direction at the vessel bottom is reversed and is directed inward, rather than outward as was the case with the axial flow pattern [32]. Furthermore, the velocities at the bottom are lower than with the axial flow pattern. As a result, complete solids suspension is much more difficult to obtain in this case and the solids mainly move around at the tank bottom, instead of being suspended throughout the vessel [31]. The settling of particles when the impeller was positioned at 7.5 cm height was visually observed confirming the explanation given above. Thus, there is an optimal position of the impeller inside the vessel where the suspension of particles is maintained and their dissolution favored.

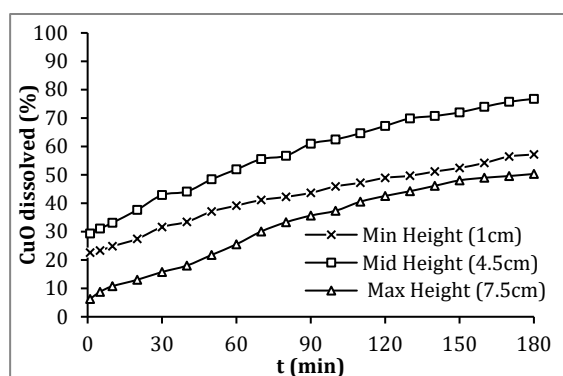


Fig 6. Effect of the position of paddles inside the reactor on the dissolution of CuO by glycine

It is worth noting that the position of the impeller at 1 cm height (very close to the bottom) approaches the conditions of magnetic stirring. A comparison of the results between magnetic and mechanical stirring under the same conditions (200 mL of glycine at 0.1 M+5 mL of NaOH at 0.5 M, 0.05 g of CuO, 25 °C, 250 rpm) shows that magnetic stirring was more efficient in CuO dissolution despite the small size of the magnetic bar used (2 cm) compared to that of the impeller (diameter 5 cm) dissolving 70.9 % versus 57.2 % respectively after 180 min. It can be supposed that since the magnetic bar is disposed at the bottom of the vessel, it enters into contact with the powder and disperses the particles more efficiently during rotation, while the region of low velocity still exists with mechanical stirring leading to agglomerate the particles and hinders the dissolution rate. The settling of particles at 1cm height of impeller was also visually observed. Thus, stirring device may cause a radical change in flow dynamic and consequently in dissolution rate. This was highlighted by the effect of the volume where opposite effects were observed between magnetic and mechanical stirrers.

3.3. Kinetic study

Leaching kinetic is often described by the Shrinking Core Model where the reaction between solid and fluid reactants occurs on the outer surface of the solid [33]. The solid reactant is initially surrounded by a fluid film through which mass transfer occurs between the solid and the bulk fluid. As the reaction proceeds, the unreacted core of the solid shrinks toward the center of the solid and a porous product layer forms around the unreacted core. This

heterogeneous model stipulates that the rate of a reaction can be controlled either by diffusion through the liquid film or by diffusion through a layer of products or by the chemical reaction. Thus, for a spherical, nonporous particle, the fraction x of the solid dissolved as a function of time t is given as follows:

$$x = k.t \quad (4)$$

for control by diffusion through the liquid film

$$(1-x)^{2/3} + 2(1-x) = k.t \quad (5)$$

for diffusion control through the product layer

$$1 - (1-x)^{1/3} = k.t \quad (6)$$

for control by the chemical reaction

where k is the rate constant (min^{-1}). The rate of dissolution is controlled by the slowest process.

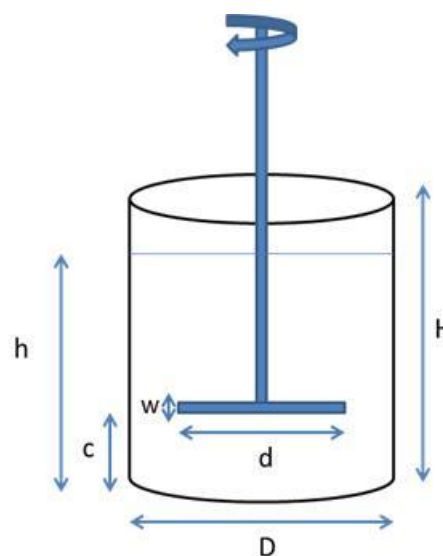


Fig 7. Characteristics of the mixed system. It consists of a flat bottomed cylindrical vessel of diameter $D = 6$ cm and height $H = 12$ cm. The liquid height is $h = 8.5$ cm. Mixing is ensured by a four-bladed impeller of diameter $d = 5$ cm and height $w = 7$ mm with blade attack angle of 45° . The clearance between the vessel base bottom and the middle of the impeller blades (variable) $c = 1$ cm, 4.5 cm and 7.5 cm

Levenspiel model was applied for the dissolution of copper oxide by glycine at different temperatures under magnetic stirring (results of figure 2d) in order to determine the rate constants and the activation energy.

The rate constants were determined by plotting the left side of equations (4-6) versus reaction time. Thus, the three expressions $[(x), (1 - 3(1-x)^{2/3} + 2(1-x))$ and $(1 - (1-x)^{1/3})]$ were reported on the y axis as a function of time (t) which was reported on the x axis. The slopes of the straight lines were equal to the rate constants (k). The application of the model reveals that equation (6) gave straight lines with linear regression coefficients close to 1 indicating that the dissolution of copper oxide was controlled by the chemical reaction (Fig. 8a).

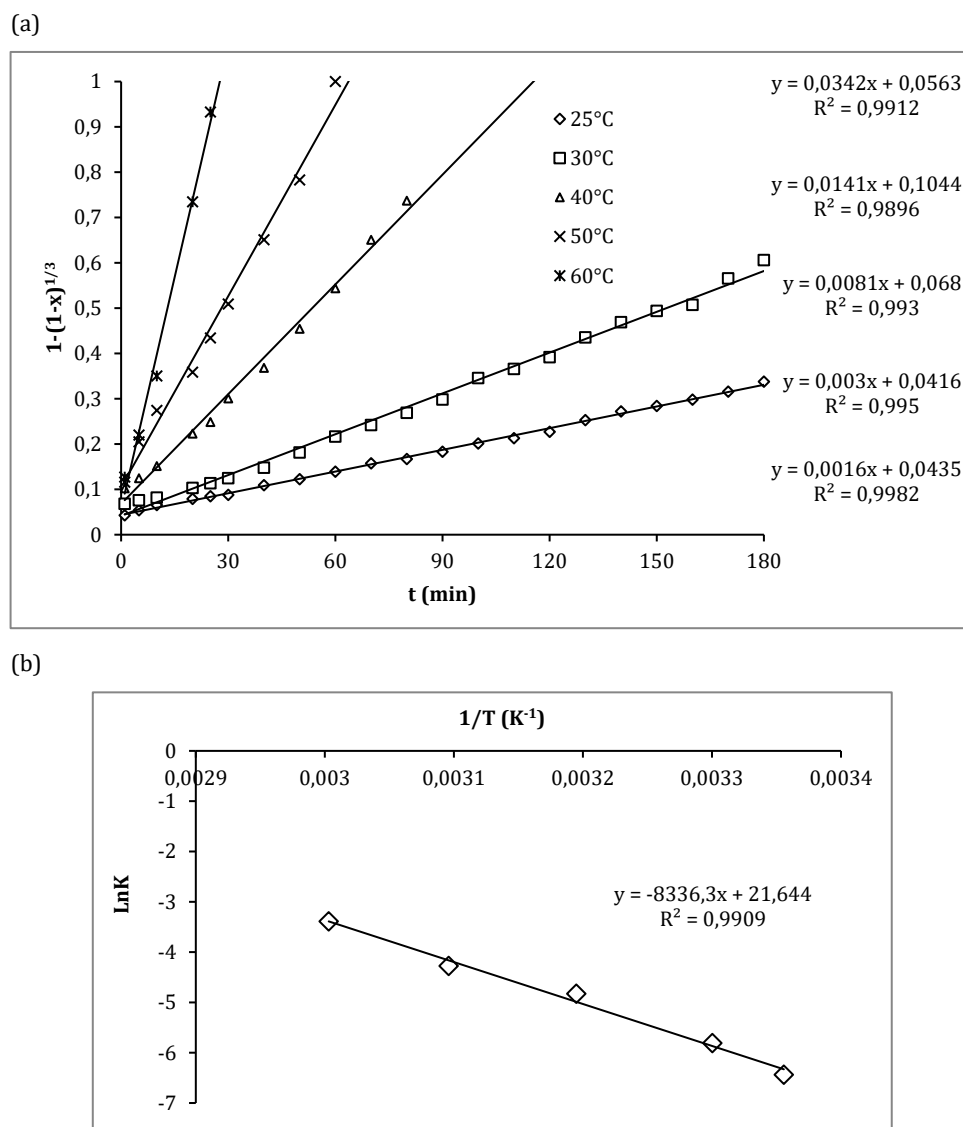


Fig 8. Fitting the experimental data obtained at different temperatures by Levenspiel Model (a) and determination of the activation energy for the dissolution reaction of CuO by glycine (b)

The values of the rate constants were used to calculate the activation energy by using the Arrhenius equation ($k = A \cdot e^{-E_a/RT}$). By taking the logarithm of both sides the equation became: $\text{Ln}k = \text{Ln}A - (E_a/R)T$ and it is possible by plotting $\text{Ln}k$ as a function of $1/T$ to determine the value of slope E_a/R and calculate the activation energy (E_a) of the reaction (Fig. 8b). Under the conditions used in this study E_a value was found to be equal to $69.31 \text{ kJ mol}^{-1}$. A chemically controlled reaction is generally a temperature dependent process and has activation energy higher than 40 kJ mol^{-1} . Our results have shown that the dissolution of CuO by glycine was greatly affected by temperature which agrees with this assumption.

4. CONCLUSIONS

This article is intended to share the preliminary results of a study that was conducted to gain a basic understanding of the role of hydrodynamics in dissolution testing for a pure component. The results for pure components are important as they may be used to evaluate leaching of more complex systems

containing several mineral constituents such as in ores and mineral wastes.

The results obtained in this study show that glycine may be used as an eco-friendly reagent as it dissolved easily CuO under mild conditions and produced a non-pollutant effluent. Noticeable but small effects of reagent concentrations were observed while temperature, the volume of solution and the nature of stirring impacted greatly the dissolution process. The effect of the volume of solution depended on the nature of mixing device. In fact, the dissolution of CuO was enhanced by reducing the volume of solution under magnetic stirring and by increasing it under mechanical stirring. These differences in results were due to differences in fluid movements created by magnetic bar and the paddles inside the vessel. The results obtained underline the crucial effect of hydrodynamics created by mixing devices on the rate of chemical reactions. This should be more routinely monitored as a part of dissolution tests when projecting to design a reactor for leaching process.

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