

Received: April 23, 2016; reviewed; accepted: June 28, 2016

LEACHING KINETICS OF NEAR INFRARED SENSOR-BASED PRE-CONCENTRATED COPPER ORES BY SULPHURIC ACID

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Abstract: Leaching kinetics of near infrared sensor-based preconcentrated copper ore was investigated to understand its leaching behaviour in a sulphuric acid solution. The leaching process was studied by monitoring the effect of some parameters under optimum conditions. The process kinetics was determined by fitting the experimental data into the shrinking core model. Contrasting results were obtained for the preconcentrated ore. The results indicated that increasing the acid concentration and temperature as well as decreasing the particle size and stirring speed enhanced the leaching rate of copper above 80%. The leaching efficiencies of 95.5 and 83.7% were obtained after 2 h leaching under the studied experimental conditions. Good correlation coefficients were obtained with the experimental data when the data were fitted into the chemical reaction controlled model. The activation energy was calculated to be 39.8 and 50.03 kJ/mol for the preconcentrated ore indicating that dissolution of copper occurred through the chemical reaction on the particle surface. Characterization of the residue by a X-ray diffraction (XRD) technique revealed abundant peaks of quartz and other associated minerals which were unreactive toward the lixiviant. A inductively coupled plasma spectroscopy (ICP-MS) analysis showed reduction in metal concentration in comparison to the raw ore which evidently conformed to the ore reactivity in the acid media.

Keywords: leaching, copper ore, sensor-based, pre-concentration, kinetics

Introduction

Most of the world high-grade copper resources are dwindling due to growing exploitation to meet global demand owing to technological advancement (Haque et al., 2014). With most copper ore body increasingly becoming complex due to high

concentrations of undesirable metals and gangues coupled with the renewed pressure on environmental protection has led to development of ways of improving the quality of low copper ore grades for efficient leaching operations (Baba et al., 2015). New approaches such as sensor-based sorting and pre-concentration are currently employed to preconcentrate ores containing high gangue materials prior to leaching. Upgrading of ores enhances copper recovery and reduction in waste reporting to the downstream concentration as well as reducing the leaching of unwanted materials (Murphy et al., 2012; Iyakwari et al., 2013). Application of the technique in combination with leaching is believed to have huge benefits such as minimising the cost of chemical requirement for ore leaching. Besides, it offers an improved and sustainable mineral processing operation by reducing the energy consumption (Wills, 2011).

Due to the increasing pressure on environmental protection to mitigate pollution arising from the traditional pyrometallurgical method of processing of copper ores, premium is now placed on the use of hydrometallurgical leaching method (Liu et al., 2012). There has been growing interest in development of the hydrometallurgical leaching method in recent years involving application of a wide range of techniques such as chemical and biological methods for copper recovery from concentrates (Gibas et al., 2015). Application of these methods is acclaimed to have many advantages especially in processing of low-grade copper ores (Baba et al., 2014; Gibas et al., 2015). However, the ability of mineral to float depends upon its surface chemistry and properties (Kelly and Spottiswood, 1982). Since most copper oxidized ores are readily soluble in acids, conventional flotation methods are not effective for treating such ores in view of their ease of solubility (Liu et al., 2011). Consequently, copper minerals such as chrysocolla, malachite and azurite, which are soluble in acids, are commonly treated by sulphuric acid (Wei et al., 2009, Liu et al., 2011, Seo et al., 2013). It is important to note that the copper ores used in this study contained varying composition of hydrated calcite and silicate with variable composition and properties. These properties make them difficult for the conventional copper flotation techniques to be successfully carried out. For efficient leaching and recovery of copper the ores were upgraded using near infrared sensor-based pre-concentration and accordingly classified. Therefore, this study is concerned with the basic understanding of the variation in dissolution behaviour and the process kinetics to obtain useful information in development of useful extraction parameters in treatment of pre-concentrated copper ores by the hydrometallurgical process.

Experimental

Material and characterization

The ore samples were obtained from Los Pozos mining district in Chile, South America. Samples were pre-concentrated and classified into product, middling and waste using near infrared (NIR) sensor-based method (Iyakwari et al., 2013). The surface analysis on the uncrushed/crushed sample particles by X-ray fluorescence

(XRF) and NIR was carried out prior to mineralogical and chemical analyses. Each category of the NIR classified samples was subjected to various characterization processes. Bulk samples of each category were divided into two during the mineralogical and chemical analyses. In each case, samples were crushed, ground, homogenised and sieved separately into different size fractions using American Standard Test Sieve Series before subjecting same to analyses. A flow-diagram for the mineralogical and chemical analyses of the classified ore is shown in Fig. 1.

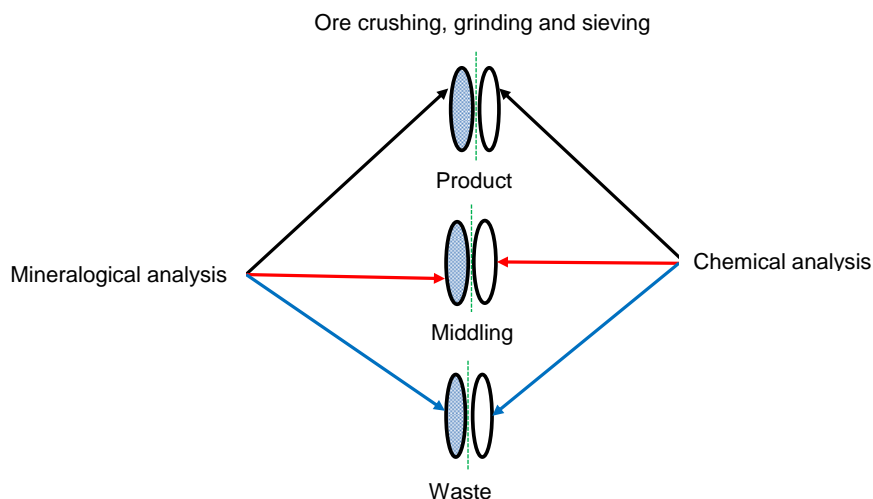


Fig. 1. Flow-sheet for mineralogical and chemical analyses of NIR pre-concentrated copper ore

The mineralogical analysis of the classified ore was carried out using a QEMSCAN[®] 4300 system, which is based on a Zeiss scanning electron microscope. The results are given in Table 1. Also a scanning electron microscope (SEM) system (Fig. 2) was used to determine the distribution of copper within the ore matrix and its texture. A particle size fraction of $-63 + 45 \mu\text{m}$, due to its large surface area, was used to prepare polish blocks of 30 mm which were then coated with carbon to improve imagery and enhance the surface conductivity. X-Ray diffraction (XRD) of ground samples (raw and leached fractions) were analysed for mineralogical content using Siemens/Bruker D5000. The XRD measurements were matched with known signatures using the EVA software.

Elemental compositions of the different size fractions were determined using a portable desktop Thermo Scientific Niton FXL 950 FM X-ray Fluorescence spectrometer. The instrument employs energy dispersive spectrometry (EDX) method. The measuring window covers a diameter of 8 mm, and X-rays penetrates approximately 1 to 2 mm into the sample.

The chemical analysis of the ore fractions was carried out using Inductively Coupled Plasma Spectrometry (ICP-MS, Agilent Technology Model 7700).

Experiments were mostly carried out using – 63+45 μm size fraction except otherwise stated. Selected size fractions were first digested in aqua regia and determined for concentration of Cu and other selected elements such as Mn, Zn, Co and Ni. Deionised water and reagent grade chemicals obtained from VWR Chemical Company, UK were used to prepare all solutions. Due to high copper content only two categories of the NIR classified ore, that is product and middling, were subjected for leaching in this study.

Leaching procedure

The leaching experiments were performed in a 500 cm^3 four-neck split flask reactor in a thermostatically heating mantle with a temperature control unit. The reactor was equipped with an overhead mechanical stirrer for agitation of contents and a rubber stopper for sampling from the leaching solution. The desired temperature was attained by adjusting a thermostatically-controlled electric heating mantle. For each experimental run, 250 cm^3 of freshly prepared sulphuric acid of predetermined molarity was charged into the reactor and heated to the required temperature before adding the solid copper ore of known weight (Ekmekyapar et al. 2012; Baba et al., 2013). After the desired stirring speed and reaction temperature were attained, 5 cm^3 of leachate solution was withdrawn at time intervals of 15, 30, 45, 60, and 120 minutes, filtered using a Whatman 540 grade filter paper and then analysed for dissolved metals. The fraction of copper extracted was calculated as a difference in amount dissolved or undissolved at various time intervals of 2 h. The kinetic analysis of the leaching process was investigated using the shrinking core model (Levenspiel, 1998).

Results and discussion

Characterization studies

Mineralogical assessment

Mineralogical analysis of the classified ore revealed different mineral compositions as depicted in Table 1. Based on the NIR and QEMSCAN[®] correlated results, the major copper-bearing mineral in the classified ore was chrysocolla. Other copper-bearing minerals such as cuprite and malachite occurred in trace amount below 0.2 wt. %. The product samples contained higher content of these minerals than the middling and waste. The gangue content in the classified ore included silicate and calcite with the waste having more concentrations of the gangues than middling and product. The XRD analysis showed that the raw ore also contained Fe_2O_3 , SiO_2 , KAlSi_3O_8 and $\text{KAl}_3\text{SiO}_{10}(\text{OH})_2$ minerals.

Table 1. Mineralogical analysis of NIR sensor-based sorted ore samples with QEMSCAN[®], in wt.%

NIR Sample category	Chrysocolla	Hematite	Cuprite	Muscovite	Biotite	Kaolinite	Malachite	Chlorite
Products	3.93	23.92	0.01	2.89	10.92	0.11	0.17	11.67
Middlings	2.70	48.75	0.00	3.20	4.98	0.54	0.00	6.63
Waste	0.73	1.47	0.02	4.06	7.38	0.01	0.17	20.66

NIR Sample category	Calcite	Ankerite	Quartz	K-feldspar	P-feldspar	Others	Total
Products	2.87	0.50	14.11	27.23	0.25	1.41	100.00
Middlings	1.73	0.69	15.23	14.11	0.39	1.05	100.00
Waste	12.10	0.64	34.08	15.63	0.84	2.21	100.00

Morphological assessment

The SEM image (Fig. 2) indicated that the copper particles in the crushed ore were finely disseminated with intergrown on a micron scale. The ore was morphologically characterised by intergrowth with chrysocolla minerals hosting copper particles residing within the hematite rich iron source and K-feldspars. This appeared to be in line with the characteristic porous nature of the chrysocolla minerals. The analysis of revealed spatial variability within the crushed ore with copper ubiquitously distributed within the crushed grain sizes of the chrysocolla, K-feldspar and hematite (Fig. 2). The observed EDS and X-ray peaks indicated abundant peaks of Fe with short peaks of Cu peak in the middling with these corresponding elements (Al, Si, K, Ca). On the other hand, the product contained lower values of these metals with abundant concentration of Cu compared to that of the middling. The Fe and Cu peaks appeared to correlate with their mineralogical concentrations according to classification.

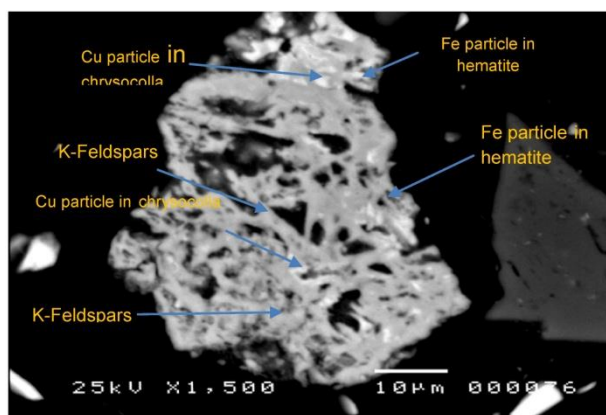


Fig. 2. SEM image of polished copper block

Chemical composition

The chemical composition of the classified ore by ICP-MS showed that the products had 1.79 wt. % copper content while the middling 1.32 wt. %. The concentration of Zn, Mn, Co, and Ni in the classified ore was as low as 0.02, 0.06, 0.03 and 0.01 ppm, respectively in the product and 0.03, 0.06, 0.04, and 0.02 ppm, respectively in the middling (Table 2). It is evident that the concentration of those elements determined in the middling was higher than in the product. This may be associated to the element-bearing minerals as was found to be more significant in the middling samples.

Table 2. Chemical composition of available copper used for leaching by ICP-MS (%)

Ore category	Cu	Zn	Mn	Co	Ni
Product	1.79	0.34	1.83	0.57	0.23
Middling	1.33	0.41	2.63	0.61	0.31

Leaching studies

Effect of H₂SO₄ concentrations

The effect of H₂SO₄ concentration on leaching was investigated by performing a number of experiments at various concentrations from 0.5 to 3.0 M at 80 °C using the particle size fraction of -63+45µm and stirring speed of 300 rpm. The results shown in Fig. 3 indicated that dissolution of copper from the near infrared pre-concentrated and classified ore was strongly dependent on the acid concentration. The observation suggested that increasing the concentration of acid led to the increase in the leaching rate for both product and middling. The extraction of copper in the product increased from 65.5 to 86.02% and from 58.16 % to 80.57% with the acid concentration from 0.5 M to 3.0 M. The variation in the rate of copper extraction in two fractions correlated well with the amount of copper in accordance with the pre-concentration process. The observed trend of increasing copper leaching from the classified ore was related to the increasing H⁺ activity as a result of acid concentration effect. Linearity in the rate of copper dissolution with time was observed during the experiment after 15 minutes. Obviously, the reaction was faster with high dissolution rate during the first 15 minutest of time in the two categories of the ore after which the reaction starts slowing down with less rate of dissolution as a result less content of copper were extracted. Since increasing acid concentration have positive correlation with rate of dissolution, 3.0 M of acid concentration was selected for investigating the effect of other leaching parameters. This observation is in a good agreement with the previous works (Habbache et al., 2009; Seo et al., 2013) on separation of copper ore of different origins.

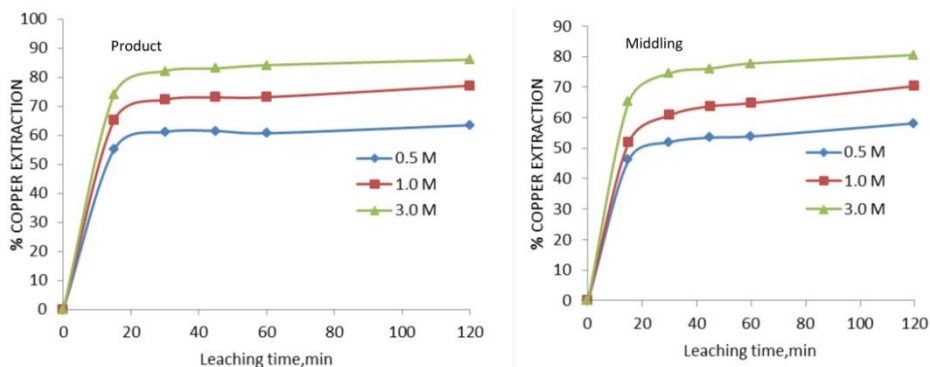


Fig. 3. Effect of H_2SO_4 concentration on dissolution of sensor-based pre-concentrated copper ore (80°C , 300 rpm, and particle size $-63+45\ \mu\text{m}$)

Effect of stirring speed

Figure 4 shows that increasing the stirring speed from 250 to 300 rpm led to improved dissolution efficiency of copper in the classified ore. However, further increase in stirring speed from 300 to 800 rpm revealed the low extraction rate for 3M H_2SO_4 acid, particle size $-63+45\ \mu\text{m}$, and temperature 80°C . Thus, increasing the agitation speed above 300 rpm did not have the profound effect on the rate of copper dissolution. It indicated that high stirring above 300 rpm was not required for leaching studies, because it only led to dispersion of the ore and poor contact time between the solid material and lixiviant. The stirring speeds of 250, 300, 500 and 800 rpm resulted in extraction efficiencies of 83.2, 93.3, 90.8 and 88.7%, respectively, for product and 60.1, 74.5, 72.1 and 64.5%, respectively, for middling. The result shown in Fig. 4 suggests that higher copper content in the product than in the middling is a result of pre-concentration of the ore and subsequent classification. In the study, the intermediate stirring rate of 300 rpm, which gave higher extraction rate, was chosen for investigation of other parameters.

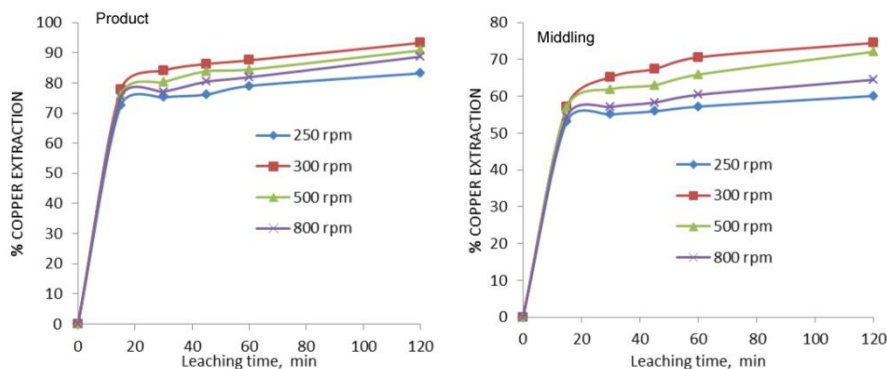


Fig. 4. Effect of stirring speed on dissolution of sensor-based pre-concentrated copper ore (80°C , 3M H_2SO_4 , particle size $-63+45\ \mu\text{m}$)

Effect of particle sizes

Investigation of the effect of particle size on leaching was investigated by considering three different particle sizes (-63+45, -90+63 and -125+90 μm). During the leaching experiment the concentration of H_2SO_4 , temperature, solid-to-liquid ratio and stirring speed were kept constant and were equal to 3 M H_2SO_4 , 80 $^\circ\text{C}$, 5g/250 cm^3 and 300 rpm, respectively. The analysis was carried out using the product fractions as shown in Fig. 5. It was found that particle size had influence on leaching of copper. The rate of copper extraction increased with decrease in the particle size. Extraction of copper for the -125+90 μm fraction increased from 68.5 to 84.4 % and from 72.6 to 90.1% for the size fraction of -90+63 μm after 120 minutes of leaching. However, when finer size fraction of -63+45 μm was used a more profound increase was obtained with extraction of 97.3%. It indicated that the finer particle size had faster extraction rate. This may be due to the greater geometric surface area and degree of available surface area for acid contact with the solid copper material. It should be noted that the finer the particle size, the more the surface area per unit weight of the material was exposed for reaction due to liberation from the ore matrix (Gharabaghi et al., 2013; Baba et al., 2013; Awe et al., 2013).

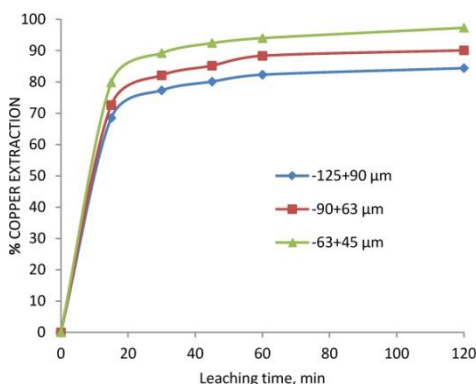


Fig. 5. Effect of particle size on dissolution of sensor-based pre-concentrated copper ore (80 $^\circ\text{C}$, 3M H_2SO_4 , 300 rpm, 5g/250 cm^3)

Effect of temperature

The effect of temperature on dissolution of sensor-based pre-concentrated and classified ore was determined by maintaining the stirring speed of 300 rpm, H_2SO_4 acid concentration of 3 M, particles size of fraction -63+45 μm and temperature range of 60 to 80 $^\circ\text{C}$. Figure 6 shows that the rate of copper dissolution increased with increasing the reaction temperature. Expectedly, the strong influence of temperature led to increase in the leaching rate. Copper extraction increased in the pre-concentrated-classified ore from 87.45 to 98.82 % in the product and 80.57 to 88.98 % in the middling when temperature increased from 60 to 80 $^\circ\text{C}$. The variation in the copper content between the product and middling was also noticed by

significant increases in copper extraction in the product (Fig. 6). The observation in Fig. 6 is believed to be as a result of increase in reaction velocity constant k between the reactants. It is important to note that the decrease in the time required for maximum copper extraction to occur in Fig. 6 when compared with Fig. 3 is as a response to temperature increase as was observed where over 80 % copper was extracted during 30 minutes leaching time. Similar results were obtained in previous studies (Gharabaghi et al., 2013; Parada et al., 2015).

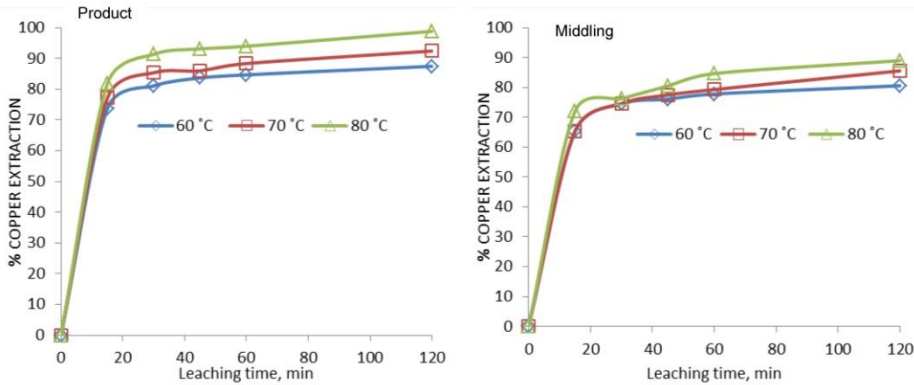
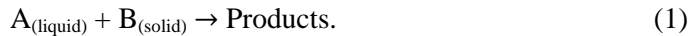


Fig. 6. Effect of temperature on dissolution of sensor-based pre-concentrated copper ores

Discussion

Kinetic analysis

The shrinking core model was applied to understand the mechanism controlling dissolution during leaching of the classified copper ore in the H_2SO_4 solution. The reaction model between the solid leaching copper particle and H_2SO_4 solution as earlier studied by Baba et al. (2013) and Gharabaghi et al. (2013) is:



According to the shrinking core model, the leaching process is controlled by several steps. Thus, for any particular liquid/solid reaction system, the reaction rate is dependent on any of the following: diffusion through the liquid film, diffusion through the ash/product layer, chemical reaction at the surface of the solid particle and or mixed of diffusion and chemical reaction are found to be consistent with the stoichiometry (Levenspiel, 1998; Gharabaghi et al., 2013; Baba et al., 2013). The equations for the three models are expressed as follows:

$$1 - (1 - \alpha)^{1/3} = \frac{K_c M_B C_A t}{\rho_s \beta r_0} = k_r t \quad (2)$$

$$\frac{2}{3}\alpha - (1 - \alpha)^{2/3} = \frac{2M_S DC_A t}{\rho_s \beta r^2 O} = k_d t \quad (3)$$

$$\alpha = k_f t = \frac{3bk_c C_A}{\rho_s r O} \quad (4)$$

where α is the fraction reacted, K_C reaction rate constant, M_S weight of the solid, C_A concentration of the dissolved lixiviant A in the bulk of the solution, ρ_s density of the classified ore, β stoichiometric coefficient of the reagent in the leaching reaction, r radius of the solid particle, t reaction time, D diffusion coefficient in the porous product layer, k_r and k_d are rate constant.

The experimental data presented in Fig. 6 were fitted into the three models to determine the effect of temperature and the mechanism controlling the process. A good correlation coefficient was obtained between the experimental data and chemical reaction model presented by Eq. (2) and this model was subsequently used to analyse the data. The plot of $1 - (1 - \alpha)^{1/3}$ versus time t for the experimental data was obtained (Fig. 7).

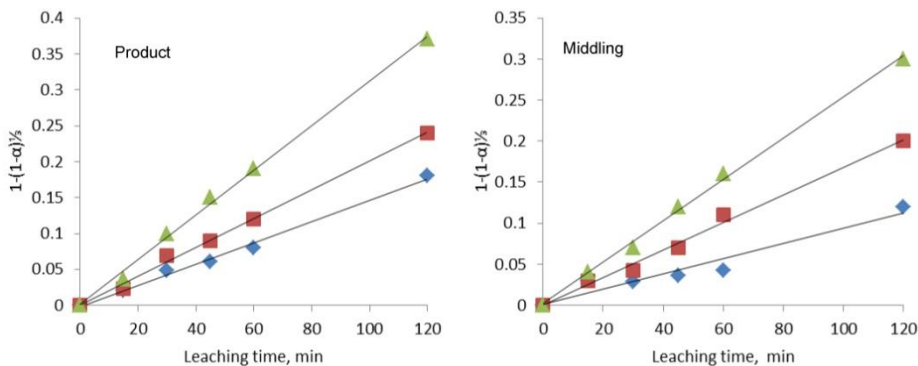


Fig. 7. Plot of $1 - (1 - \alpha)^{1/3}$ versus time t for experimental data taken from Fig. 6

The activation energy was determined using the Arrhenius relation:

$$k_r = A \exp\left[-E_a / (RT)\right]. \quad (5)$$

Figure 8 is the Arrhenius plot constructed by carrying out a plot of $\ln k$ versus $1/T$, the k values fitted from Eq. (2) and $(-E_a/R)$ is the slope. The calculated activated energy was found to be 39.8 for product and 50.03 kJ/mol for middling. The variation in the value of activation energy between the product and middling could probably be due to the variation in amount of gangue and other minerals thus suggesting that leaching of the middling is more sensitive to the reaction temperature. Conversely, with values of activation energy of greater than 12 kJ/mol obtained it clearly indicates

that the examined leaching process of the ores is controlled by chemical reaction on the particle surface (Awe, 2013; Baba et al., 2013).

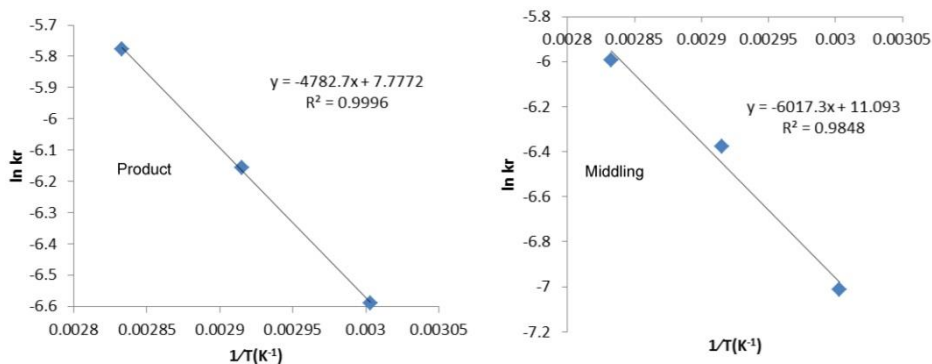


Fig. 8. The Arrhenius plot for dissolution kinetics of sensor-based pre-concentrated copper ore

Residual product characterization

The residue obtained after optimum leaching by 3 M H₂SO₄ solution was subjected to mineralogical and chemical analyses using XRD and ICP-MS. The XRD result obtained revealed reduction in peaks of most of the major minerals when compared to profiles of raw ore, which was an indication that most metals associated with them were depleted, while abundant peaks of quartz suggested that this mineral was unreactive during the leaching process. Disappearance of calcite peaks was a strong indication of its reactivity and acid consumption behaviour. This observation was also reported by Bingol and Canbazoglu (2004). On the other hand, the ICP-MS analysis of Cu, Zn, Mn, Co and Ni is summarized in Table 3. The result indicated that the concentration of these elements were affected probably due to activity of acid which was apparently mostly responsible for their co-dissolution with Cu. These elements found in the ore mineral matrix reacted effectively with the acid. The decreased concentration of copper in the residue after leaching suggests that the metal was effectively leached. The extraction efficiency of copper was determined using the following relationship (Liu et al., 2011):

$$A (\%) = \frac{W_m \times Cu_a - W_r \times Cu_r}{W_m \times Cu_a} \times 100 \quad (5)$$

where A is the percentage recovery of Cu, W_m weight of ore material before leaching, Cu_a copper content of the raw ore material before leaching, W_r weight of the residue after leaching and Cu_r is the copper content of the residue after leaching. The extraction efficiency of copper was found to be 95.5% (product) and 83.7% (middling), while the efficiency of Zn, Mn, Co and Ni metals was insignificant (< 10 %) when compared to that of Cu. Expectedly, the high efficiency in the product than

the middling follows an earlier pattern shown from the NIR pre-concentration and subsequent leaching results obtained with the acid.

Table 3. ICP-MS residue analysis after 2 h leaching

	Cu, %	Zn, %	Mn, %	Co, %	Ni, %
Product	0.15	0.20	0.50	0.14	0.10
Middling	0.13	0.31	0.62	0.14	0.14

Conclusions

Leaching kinetics of a near infrared sensor-based pre-concentrated copper oxide ore in H_2SO_4 solution was investigated. The pre-concentration process produced three fractions classified as either the product, middling or waste depending on the copper and gangue content. The product fraction was determined to contained more copper than the middling and waste. Contrasting results were found for leaching of two fractions that is product and middling. The rate of copper extraction was found to be in accordance with pre-concentration with better leaching results obtained for the product than middling. The rate of copper dissolution and extraction was enhanced with increase in the sulphuric acid concentration and temperature. Moreover, with decreasing particle size and stirring speed the rate of copper extraction increased. The leaching efficiency was determined to be 95.5% in the product fraction and 83.7% in the middling fraction. The kinetic analysis of the process was evaluated using the shrinking core model for chemical control reactions. The activation energy of 39.8 and 50.03 kJ/mol for the product and middling supported the proposed dissolution kinetics model through the particle surface. The XRD and ICP-MS analyses of the residue suggested that during the leaching process, only easy-to-leach particles were dissolved leaving an unreactive ore matrix. In terms of leaching application, the pre-concentrated ore can be treated separately on the basis of copper content for economic benefits of the leaching process.

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