



Sulphuric acid leaching of zinc and copper from Nigerian Complex Sulphide Ore in the presence of hydrogen peroxide

by P.A. Olubambi*†, J.O. Borode†, and S. Ndlovu*

Synopsis

The leaching of zinc and copper from Nigerian bulk sulphide ore with sulphuric acid in the presence of hydrogen peroxide was studied. The aim of the study was to investigate the effectiveness of hydrogen peroxide as an oxidant for the sulphuric acid leaching of zinc and copper from a complex sulphide ore. Distribution of elements within the various particle sizes was initially determined by ICP-OES and XRF to provide a basis for explaining the dissolution process. The effects of concentration of hydrogen peroxide, particle size, stirring speed, and leaching time at a constant temperature of 298K were studied. There were variations in elemental and mineralogical composition within the different sizes. The concentrations of zinc and copper reduced as particle size decreased while silica, sulphur, iron and lead contents increased. Dissolution results showed that the leaching rate of copper was lower than that of zinc. The highest recoveries of zinc and copper were obtained at a leaching time of 180 minutes, stirring speed of 160 rpm, particle size of 75 μm and a concentration of 1M H_2SO_4 /1M H_2O_2 . It was observed that the leaching rate of zinc and copper increased with increasing hydrogen peroxide concentration. Increased stirring speed had a negative leaching effect as it promotes hydrogen peroxide decomposition.

Keywords: Nigerian sulphide ore, zinc, copper, hydrometallurgy, sulphuric acid, hydrogen peroxide.

Introduction

In recent times, attention has been drawn to low-grade complex sulphide ores due to the decline in the world deposits of high-grade ores. Complex sulphide ores are in many cases difficult to treat with conventional mineral processing methods and the concentrates produced are often not clean enough. This seriously limits their commercial value (Ortega and Bonilla, 1983). Differential flotation does not release all the constituents' phases (Majima, 1969) and so the different concentrates obtained are of poor quality with a low metal recovery. This makes further pyrometallurgical processing of these ores very difficult and costly (Rubio and Frutos, 2002) and renders them difficult to commercialize (Sandstrom and Peterson, 1997). As a result, the metal value is preferably extracted directly from the low-grade ores through a hydromet-

allurgical process. However, sulphide ores do not allow the recovery of metal by direct chemical leaching (Hiskey and Wadsworth, 1975; Dutrizac, 1989), due to their low solubility in most leaching reagents.

In such cases, ore must be pre-treated in order to release the desired metals physically and to make the ore amenable to leaching reagents (Chiacchiarini *et al.*, 2003). Processes available for the treatment of complex sulphide ores involve sulphide oxidation to form water-soluble sulphates or oxides. The sulphur oxidation processes include roasting, pressure oxidation, chemical oxidation and bioleaching. The processes that are traditionally used are complex and expensive (Gomez *et al.*, 1997, Han and Meng, 2003). Hence attention has been drawn to the use of easily available and cheap reagents like sulphuric acid for base metal recovery (Antonijevic and Bogdanovic, 2004) and means of optimizing the dissolution kinetics. Although sulphuric acid may not be as effective as other stronger oxidizing acids like nitric acid (Çopur, 2001, 2002; Prasad and Pandey, 1998), it is, however, preferred to all other reagents in terms of cost, corrosion wear and the ease of regeneration during electrowinning (Biswas and Davenport, 1980).

As a means of optimizing sulphuric acid leaching, there is the possibility of aiding its dissolution kinetics or oxidation through the use of hydrogen peroxide. Hydrogen peroxide is a strong oxidizing agent and promotes the oxidation and leaching potential of sulphuric acid. Besides its high oxidizing potential, it is easier to handle than gaseous oxidants. It can

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also be catalyzed into hydroxyl radicals, which are extremely reactive. The dissolution of ores using sulphuric acid in conjunction with hydrogen peroxide has been studied and successful results were obtained. Jiang *et al.* (2002) studied the dissolution kinetics of manganese-silver associated ores, and found that hydrogen peroxide greatly increased sulphuric acid recovery of silver. The reaction orders of H_2SO_4 and H_2O_2 were determined as 0.80 and 0.68, respectively. Silver leaching was found to be diffusion-controlled and follows the kinetic model: $1 - 2x/3 - (1 - x)^{2/3} = Kt$ (x is the molar fraction, t is the time (min), and k is mass transfer coefficient). Antonijevic *et al.*, (1997 and 2004), on the other hand, studied the dissolution kinetics of chalcopyrite by hydrogen peroxide in sulphuric acid. Copper recoveries were found to be significantly increased as the concentrations of sulphuric acid and hydrogen peroxide increased. Nevertheless, all these research studies have been centred on high grade ores and none on low-grade, complex Nigerian sulphide ores.

There are several million tons of complex sulphide ores occurring in Nigeria (Onah, 2001; NIPC Nigeria) comprising galena, sphalerite, pyrite, siderite, quartz, and chalcopyrite with traces of silver and gold. No attempts have been made to develop a suitable process for the optimal extraction of the zinc and copper present in these ores. Previous research investigations on these ores have only centred on flotation studies (Onyemaobi, 1987), with little work on direct leaching. Adebayo *et al.*, (2003) investigated only hydrogen peroxide dissolution of a high grade Nigerian chalcopyrite in the presence of sulphuric acid, and demonstrated that copper recovery was greatly enhanced by sulphuric acid leaching in conjunction with hydrogen peroxide. High grade chalcopyrite deposits in Nigeria are very small, and it would not be economically justified to set up a hydrometallurgical plant for copper recovery from these deposits only. Concerted efforts are therefore being made by the Nigerian government to invite and encourage private sector participation and joint ventures to develop, exploit and stake concessions on these complex sulphide ore deposits (Ekaette, 2003).

This study therefore investigates the effectiveness of a hydrometallurgical route for processing these ores using sulphuric acid for the leaching of zinc and copper in the presence of hydrogen peroxide, considering influencing parameters such as concentration of hydrogen peroxide, leaching time, particle diameter and stirring speed.

Experimental mode

Materials

The complex sulphide ore used in this study was obtained from Ishiagu in Ebonyi State, South Eastern region of Nigeria. Detailed characterization studies of the ore using SEM/EDX, XRD, XRF, and ICP-OES have been reported in Olubambi *et al.* (2006). XRD analysis identified the presence of siderite (42%), sphalerite (35%), galena (11%), quartz (8%), and traces of chalcopyrite and pyrite. Sphalerite occurred as ferrous sphalerite with variations in the percentages of Fe and Zn within the various particle sizes, with finer particles having lower percentages of iron.

Experimental technique

Leaching experiments were carried out in 250 ml Erlenmeyer flasks. These flasks were placed in a thermostatic heated water bath shaker to provide the necessary stirring and keep the temperature constant at 25°C. Analytical grade sulphuric acid (98 vol% H_2SO_4), hydrogen peroxide (34 vol% H_2O_2) and distilled water were used. Leaching experiments were conducted in two stages, namely sulphuric acid leaching first, followed by sulphuric acid leaching in conjunction with hydrogen peroxide.

Sulphuric acid leaching

Sulphuric acid solution (1M, 100 ml) was introduced into the reactor. At the desired temperature, 2 g of the concentrate at a particle size of 100% passing 75 μm was added to the acid solution. The contents were stirred at a constant speed of 160 rpm for 45 minutes. Thereafter, 2 ml of pregnant solution were taken from the reactor every 45 minutes, filtered and the filtrate were diluted to 100 ml with distilled water in a volumetric flask for copper and zinc analysis using atomic absorption spectrometry (AAS). This procedure was repeated for varying particle sizes of +106 -75 and +150-106 μm respectively and stirring speeds of 180, 200 and 220 rpm.

Sulphuric acid with hydrogen peroxide leaching

These experiments were conducted to determine the effect of hydrogen peroxide concentration on the dissolution rates in sulphuric acid. Equal volumes of 50 ml each of sulphuric acid and hydrogen peroxide were introduced into the reactor vessel. At the reaction temperature, 2 g of ore with a particle size of 100% passing 75 μm were added and the stirring speed was kept at 160 rpm. A sample was drawn from the reactor every 45 minutes, filtered, and the filtrate were diluted to 100 ml with distilled water in a volumetric flask and subjected to elemental analysis. The procedure was repeated for varying concentrations of hydrogen peroxide (0.25, 0.5, 0.75 and 1.0 M), particle sizes of +106 -75 and +150-106 μm respectively and stirring speeds of 180, 200 and 220 rpm.

Results and discussion

Effect of particle size

The effect of particle size on the dissolution of zinc and copper from the complex ore was studied using three different particle sizes (100% passing 75, +106 -75 and +150-106 μm). The results shown in Figures 1 and 2 revealed that the leaching efficiencies of zinc and copper increased with a decrease in particle size for the 75 and 106 μm materials. This is in agreement with the findings of Akcil, (2002) and Antonijevic *et al.*, (2004) who demonstrated that dissolution the rate increases with decreasing particle sizes. Larger particle sizes promote the decomposition of hydrogen peroxide to gaseous oxygen and water (Adebayo *et al.*, 2003), which led to a reduction in the oxidizing power of hydrogen peroxide. Since the intermolecular bond between hydrogen peroxide is a relatively weak hydrogen bond, larger particles possessing higher kinetic energies easily break the intermolecular bond when in contact with hydrogen peroxide.

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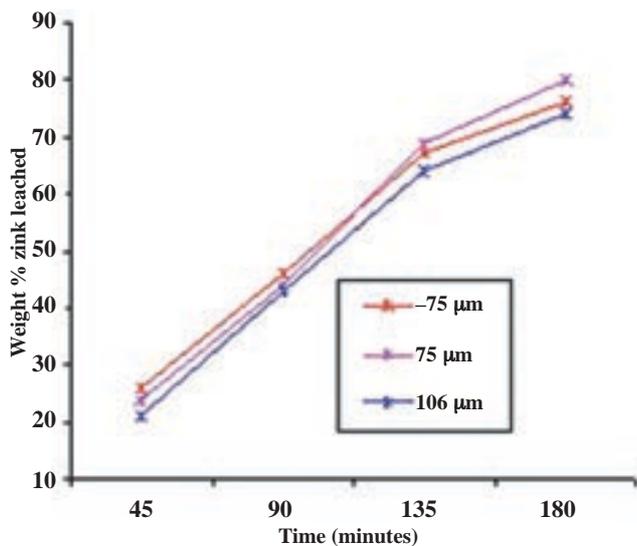


Figure 1—Dissolution of Zinc in 1M H₂O₂ and 1M H₂SO₄ at a stirring speed of 160 rpm

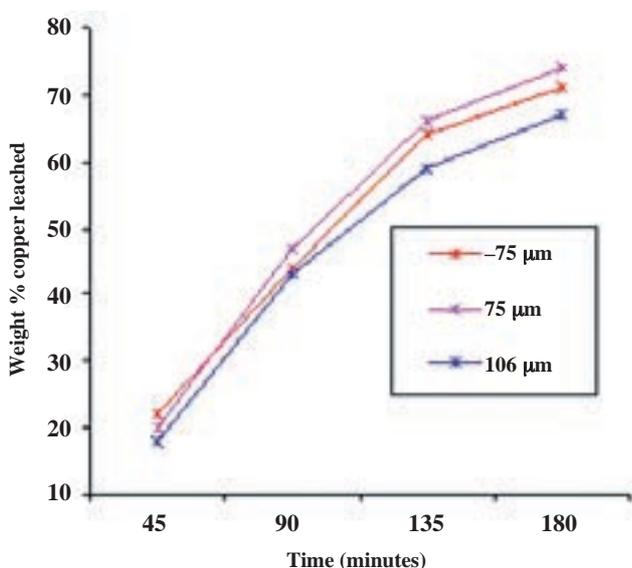


Figure 2—Dissolution of Copper in 1M H₂O₂ and 1M H₂SO₄ at a stirring speed of 160 rpm

The lower dissolution rates at larger particle sizes could also be attributed to the decrease in the surface area of the reacting ore. At larger particle sizes, less metal content is exposed to the reacting solution, thereby reducing contact between the leaching reagent and metal content of the ore. Antonijevic *et al.* (1997) also found that the decomposition of hydrogen peroxide to oxygen and water was not only accelerated by particle size but also to the presence of iron, where iron within the ore acted as a catalyst.

A particle size of 75 μm gave the maximum zinc and copper dissolutions of 80% and 74% respectively after 180 minutes of leaching. One would have expected highest recoveries at the smaller particle size (-75 μm). However, the lower leaching efficiencies of 68% and 62% for zinc and copper dissolution respectively obtained at a particle size of -75 μm could probably be attributed to the fact that there was a significant amount of SiO₂ (Table II, Olubambi *et al.*, 2006)

at particle sizes below 75 μm. The presence of silica has been observed to cause a shift in iron mobilization (Davis *et al.* (2001), and high levels of silica alter the mineralogy of ferrous oxidation products in natural systems, leading to the precipitation of a more stable solid precipitate (Mayer and Jarrell, 1996). Phoenix *et al.* (2003) noted that iron may precipitate in the presence of silica as either an amorphous Fe(OH)_{3(s)} phase (which might not occur at high acidity of leaching) or as poorly ordered hydrous iron-silicates e.g. ((Fe,Mn)SiO₃,Fe₂³⁺Si₂O₇·2(H₂O)) and ((Fe,Mg)₃Si₄O₁₀(OH)₂). The precipitate form therefore alters the reactivity and redox behaviour of ferrous/ferric ions in the system. Rushing (2002) confirmed that an increase in silica content decreases the rate of oxidation of Fe²⁺ to Fe³⁺. The rate of copper and zinc dissolutions would therefore decrease since the amount of ferric/ferrous ions needed to oxidize sulphide to sulphate is reduced. In all the experiments, the percentage of copper leached was found to be lower than that of zinc. This poorer dissolution of chalcopyrite could be attributed to a galvanic reaction between chalcopyrite and sphalerite (Cupor, 2001). Hence, zinc tends to be preferentially dissolved from sphalerite over copper from chalcopyrite.

Concentration of hydrogen peroxide

The effect of the concentration of hydrogen peroxide on the leaching of zinc and copper from the bulk ore is shown in Figures 3 and 4. The percentages of zinc and copper dissolved in all the experiments were found to increase with the addition of hydrogen peroxide. The observation that the rate of leaching increases with increasing concentration of the leaching agent (Habashi, 1970) suggests that hydrogen peroxide increases the rate and extent of reaction. It also confirms that hydrogen peroxide has a significant effect on oxidation of sulphide in order to release the zinc and copper ion. This may be due to the high oxidation potential of hydrogen peroxide, which increases the dissolution of the ore by partially converting the sulphide to elemental sulphur and subsequently to sulphate.

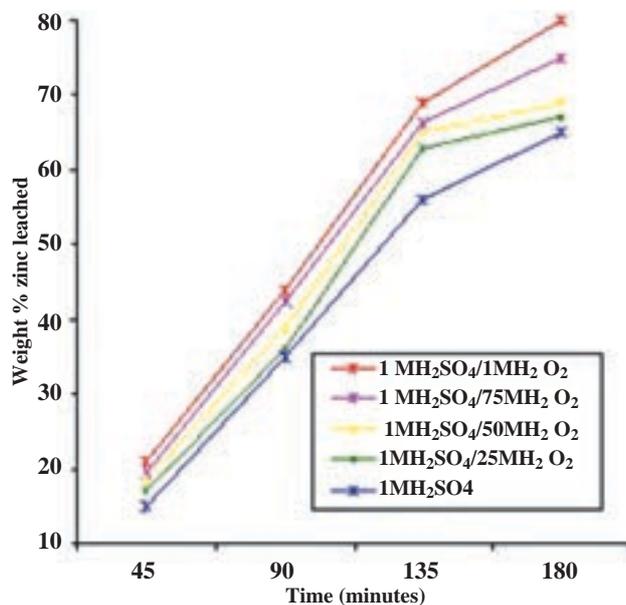


Figure 3—Leaching of zinc at a stirring speed of 160 rpm and particle size of 75 μm

Sulphuric acid leaching of zinc and copper from Nigerian Complex sulphide ore

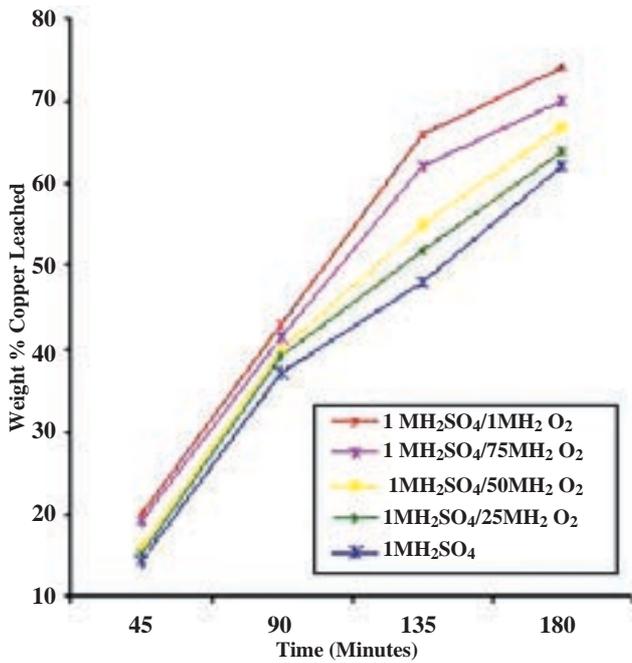
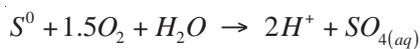
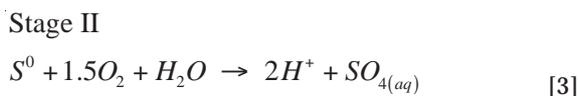
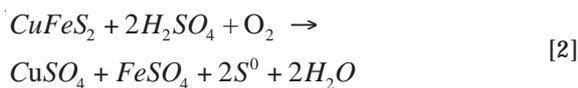
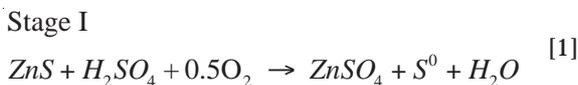


Figure 4—Leaching of Copper at a stirring speed of 160 rpm and a particle size of 75 μm

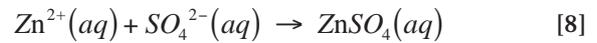
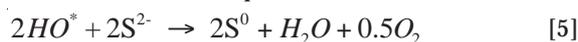
The dissolution process in the presence of H₂O₂ has been investigated by Jiang *et al.*, (2002) and Adebayo *et al.*, (2003) and it is believed to take place in two reaction stages, viz. the deposition of elemental sulphur and its conversion to sulphate (SO₄²⁻), which can be expressed as follows:



The first stage of the proposed reaction mechanism, which is the sulphuric acid leaching, is very slow and was enhanced by the addition of hydrogen peroxide. When hydrogen peroxide comes in contact with minerals, it decomposes to the hydroxyl anion (OH⁻) and hydroxyl radical (HO*) (Lin and Luong, 2004) as represented by Equation [5] (Adebayo *et al.*, 2003):



When the very reactive hydroxyl radical (HO*) comes in contact with the highly reactive sulphide mineral, elemental sulphur is formed (Equation [6]) and the elemental sulphur is oxidized to sulphate ions (Equation [7]). When copper and zinc react with sulphate (Equations [8] and [9]), copper sulphate and zinc sulphate are formed, which are very soluble and dissociated in aqueous solutions.



The significant increase in the dissolution rate of the ore resulting from an increase in the concentration of hydrogen peroxide can also be explained based on the formation of Caro's acid (Abou-Yousef *et al.*, (2005) (Equation [10]). As the percentage of hydrogen peroxide in the leach increases, the tendency for hydrogen peroxide to form Caro's acid in the presence of sulphuric acid increases. Caro's acid has an acidic property and a higher oxidizing potential on the sulphide than hydrogen peroxide (FMC Corporation).



According to FMC Corporation, the yield of Caro's acid increases as the mole ratio of H₂O₂ to H₂SO₄ increases. For 0.75 and 1M hydrogen peroxide addition, zinc and copper dissolutions increased from 72% to 80% and 68% to 74% respectively during 180 minutes of leaching of a particle size of 75 μm.

Effect of stirring speed

The effect of stirring on the dissolution rate of zinc and copper at various H₂O₂ concentrations was investigated at stirring speeds of 160, 180, 200 and 220 rpm. The results (Figures 5 to 8) showed that lower percentages of zinc and copper were leached at higher stirring speeds. The highest dissolutions were obtained at a stirring speed of 160 rpm for all the concentrations of hydrogen peroxide.

Increased stirring results in faster decomposition of H₂O₂ (Equation [11]), resulting in the evolution of oxygen that adsorbs onto the particle surface. This could hinder particle/peroxide contact (Adebayo *et al.*, 2003).

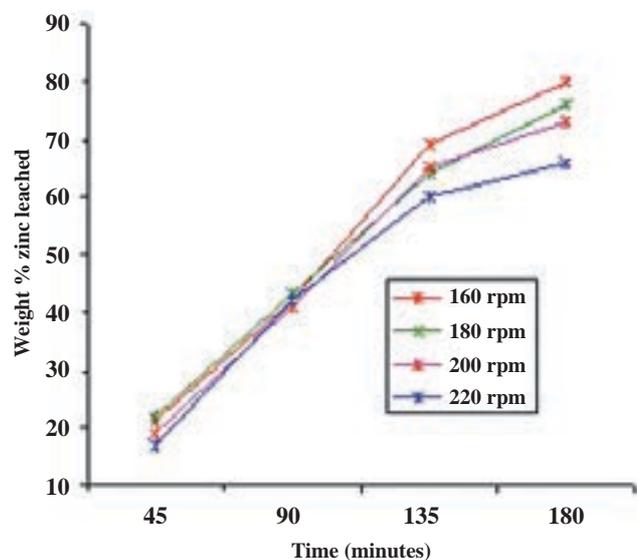


Figure 5—Leaching of zinc in 1M sulphuric acid and 1M hydrogen peroxide and a particle size of 75 μm

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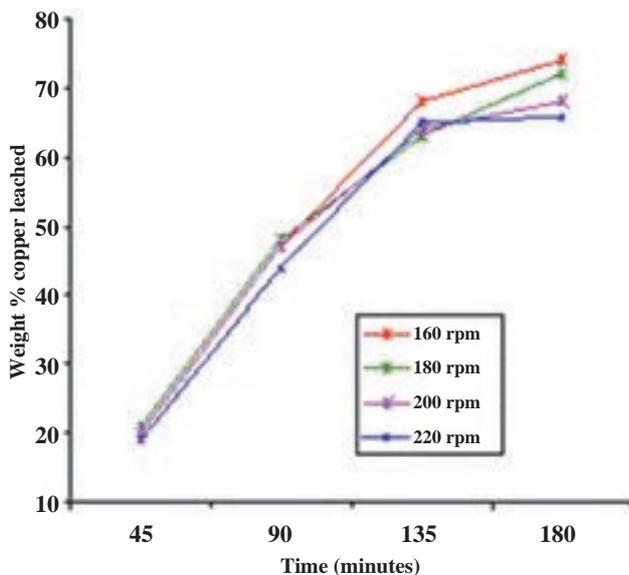


Figure 6—Leaching of copper in 1M sulphuric acid and 1M hydrogen peroxide and a particle size of 75 μ m

It was observed that, for all the hydrogen peroxide concentrations tested, the rates of dissolution of copper and zinc decreased with increased stirring speed. For sulphuric acid alone, a reverse trend was seen. At 1M hydrogen peroxide (Figures 7 and 8), the decrease in dissolution rate was slower than those of the lower H_2O_2 concentrations. The reason for this is probably due to the higher degree of Caro's acid that would form at 1M hydrogen peroxide/1M sulphuric acid (Equation [10]). Caro's acid does not decompose as easily as hydrogen peroxide.

Conclusions

Sulphuric acid dissolution of Nigerian bulk complex sulphide ore in the presence of hydrogen peroxide was investigated. It was found that ore mineralogy plays a major role in the dissolution process. Highest recoveries of both copper and zinc were obtained at a particle size of 75 μ m. Lower recoveries at a particle size below 75 μ m was probably due to the higher silica content in this fraction, which could reduce the ferric concentration in the solution due to the precipitation of stable iron silicates. Based on the results of this study, it was concluded that the combination of sulphuric acid and hydrogen peroxide enhances the dissolution of the bulk complex ore when compared with sulphuric acid leaching alone. The leaching efficiencies of zinc and copper were favoured by an increase in the concentration of hydrogen peroxide as well as contact time. Increased stirring rates and larger particle sizes reduced the rate of dissolution, probably as a result of an increase in the decomposition of hydrogen peroxide.

The results of this study have shown that sulphuric acid leaching in the presence of hydrogen peroxide is an effective route for copper and zinc recovery from Nigerian complex sulphide ores. Leaching at 1 M sulphuric acid in the presence of hydrogen peroxide (at increasing concentrations up to 1 M) investigated would be an economical effective process. The cost of the reagents used in this study is very low and

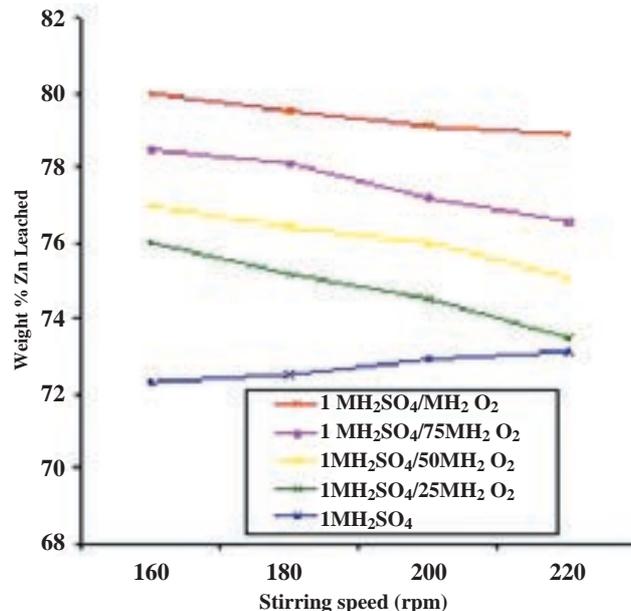


Figure 7—Leaching of Zn at varying stirring speed and a leaching time of 180 mins

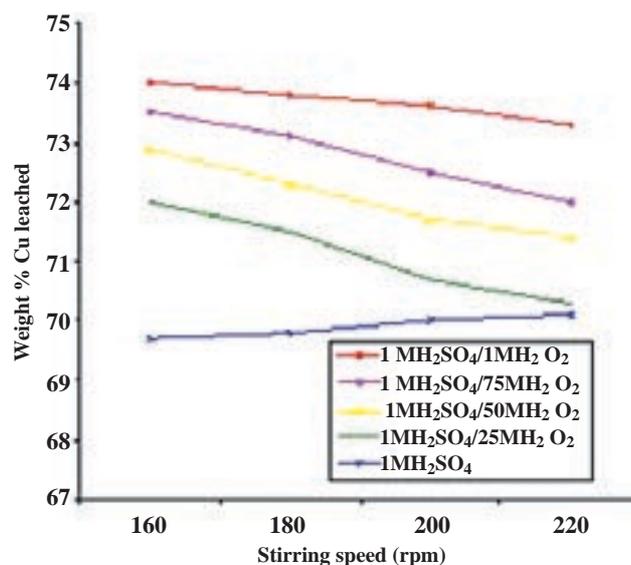


Figure 8—Leaching of Cu at varying stirring speed and a leaching time of 180 mins

there will be no cost incurred when heating up to 25°C temperature. Moreover, the process might not induce a very corrosive environment and would therefore not require very expensive corrosion resistance equipment. Hence, the maintenance of the leaching reactors might not be an industrial nightmare. The Nigerian government should therefore encourage further research in this area.

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