

## **Agitation leaching recovery of lead and zinc from complex sulphide ore deposit using HF, HCL and H<sub>2</sub>SO<sub>4</sub>**

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### **ABSTRACT**

*The paper presents laboratory report on leach study of a particular ZnS ore using a range of concentrated acid. The authors carried out this work as prelude to a more systematic study on the considerable body of knowledge in processing this type of ore in terms of the Roast-Leach-Electrowin process and then considered the extent to which this method is applicable to their specific ore. The ore was roasted in air and leached in three acids HF, HCl and H<sub>2</sub>SO<sub>4</sub> using agitation leaching method with variation in leaching time and molar concentration of acids. The effect of increasing the concentration of leachants shows that both metals have higher values of recovery at lower concentration ranging from 1~3M of HF, and at 1M of HCl and H<sub>2</sub>SO<sub>4</sub>. Highest Zn dissolution was obtained in 6M H<sub>2</sub>SO<sub>4</sub> and HF; 1M HCl while Pb recovery in the trend of HF, HCl and M H<sub>2</sub>SO<sub>4</sub>.*

**Key words:** Sulphide ore, Roasting, Agitation leaching, Dissolution, Lead-zinc recovery

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### **INTRODUCTION**

The need for Africa to be developed by African through Science and Technology has been the vision driven by the combine initiative of the Regional Initiative in science Education (RISE) and the African University of Science and Technology (AUST) being supported by various stakeholders from Africa, international community and the African Diaspora. Too numerous natural, agricultural, petroleum and mineral resources have been discovered and identified in Nigeria. A large deposits of iron ore in Itakpe; galena-sphalerite complex ore at Abakaliki-Ishiagwu axis and gold deposit around Ilesa in southwest, Nigeria. Among all, a very few has been given attention to really transcribe to economic development.

Recycling of lead and zinc obtained from scrap batteries has been the commonest source of both metals in the country. The usefulness of both metals (Pb and Zn) and other associated metals such as Ag, Cu, Cd, Ni etc to engineering applications cannot be over-emphasised hence research works were done on the exploitation of these minerals [1, 2, 3, 4, 5, 6].

The local production of Zn would have been of great help to the growing metallurgical (steels and galvanising) industries in Nigeria. The extent of the Zn scarcity is reflected in the wide price differences between galvanised and non-galvanised steel products, especially iron roofing sheet and flat steel sheet for making water tanks [7].

Lead has found wide engineering applications in the recent times. It is used in the making of storage battery, alloyed with tin in solder (flux) making, manufacture of ammunition as lead-antimony alloy, used as personal protector in

radioactive materials and atomic energy installations. The metal is used in the manufacture of tetra-ethyl-lead (an anti knocking agent) used in petrochemical industries, and in the paint pigment and cable covering [8, 9, 10, 11].

The hydrometallurgical method has remained very popular in winning many metals from their ores. The method usually involves the winning of metals from their ores by leaching into suitable solution from which the metal can be easily retrieved (won back) by any other means such as precipitation, ion exchange, solvent extraction and electro-winning. The use of leaching in winning lead and zinc from their ores is becoming increasingly famous among other metals. Various works were done on lead and zinc leaching using different leaching solutions and with variations in their leaching conditions by Cole et al, 1983; Lee et al, 1990; Jolly and Neumeier, 1991 [12, 13, 14].

As part of research work necessary to find place for developing some of our largely available mineral resources in Nigeria, laboratory investigations were performed to establish some mineral processing and metallurgical extraction methods that can be used in winning lead and zinc from among the numerous minerals existing in the Abakaliki sulphide ore [15, 16, 17]. The hydrometallurgical extraction by leaching involves the use of HF, HCl and H<sub>2</sub>SO<sub>4</sub> to win the metal into solution from which they can be profitably recovered. The paper presents laboratory report on leach study of a particular ZnS ore using a range of concentrated acid. The authors carried out this work as prelude to a more systematic study on the considerable body of knowledge in processing this type of ore in terms of the Roast-Leach-Electrowin (RLW) process and then considered the extent to which this method is applicable to their specific ore.

## MATERIALS AND METHODS

### Concentration and roasting of ore

Samples of lead-zinc sulphide ore was collected from Abakaliki deposit. The ore was crushed, ground and sieved to obtain -150 $\mu$ m to -75 $\mu$ m particle sizes, using Denver jaw crusher, ball mill and automatic sieve shaker respectively. The compositional analysis of the sample was done using the atomic absorption spectrometry (AAS) as presented in Table 1. The ore was concentrated into PbS-ZnS as concentrate and feldspar as tailing by froth flotation using the Denver D.12 Sub-flotation machine. The concentrate was roasted at 1000 °C for 2 hrs under reducing atmosphere.

### Leaching of ore by agitation process

1000g of ore roast was weighed out and divided into (40) forty portions each containing 25g of ore roast. 250 ml acid leachants were prepared at different high molar concentrations with the concentrations of acid ranging from 1M to 9M and 11.5M for HCl, 1M to 9M and 18 M for H<sub>2</sub>SO<sub>4</sub>, and 1M to 9M and 57M for HF. 15g of the ore roast was leached in 250 ml HF and H<sub>2</sub>SO<sub>4</sub>, and 250 ml of HCl for 7 days. The Pb and Zn dissolution was determined daily for the different molar concentrations of the HF, HCl and H<sub>2</sub>SO<sub>4</sub> acids by AAS to investigate the effect of increasing concentration of acids and leaching time on the dissolution Pb and Zn. The results are presented in Figures 1 to 4. The leaching process was performed under the following conditions: The leaching temperature was raised to about 70–80 °C for 1 hr daily. There was occasional gentle agitation of the leaching process and addition of very small amount of HNO<sub>3</sub> as catalyst and oxidant.

## RESULTS AND DISCUSSION

Table 1: Chemical composition of Abakaliki lead-zinc complex ore deposit

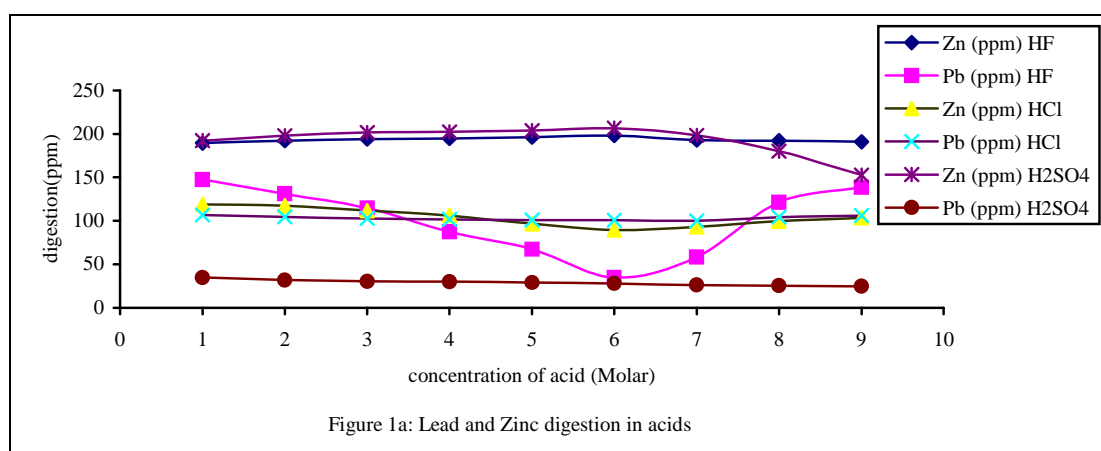
Metals	Sb		Pb		Zn		Fe		As		Cd		Cu		Au		V		Co	
Conc. (ppm)	50.25		517.43		225.32		194.8		12.4		99.26		38.37		46.61		6.85		4.9	
Metals	Ni		Ag		Sn		Na		Mg		K		Al		Ca		Si			
Conc. (ppm)	36.2		12.4		22.6		136.9		3.21		205.6		701		63.6		1587.8			

The chemical composition of the ore sample is shown in Table 1. Figures 1a and 1b present the results of comparative effects of increasing concentration of three leaching acids on the dissolution of lead and zinc (Pb and Zn) from the ore roast. It was revealed that in all these three acids used, Zn has higher dissolution (ppm) as compared with Pb dissolution. The characteristic of Pb dissolution at this increasing concentration of HF presents exactly an opposite behaviour of trend. Between 1~6M increasing concentration of HF, the Pb dissolution shows a steady decrease while further increase in the HF concentration led to a drastic increase in the trend of the Pb dissolution. The dissolution of Pb and Zn in HCl shows a similar trend for both metals. There was decrease in Zn dissolution and in Pb dissolution as the acid concentration rises from 1~7M. The trend of Pb and Zn dissolution in

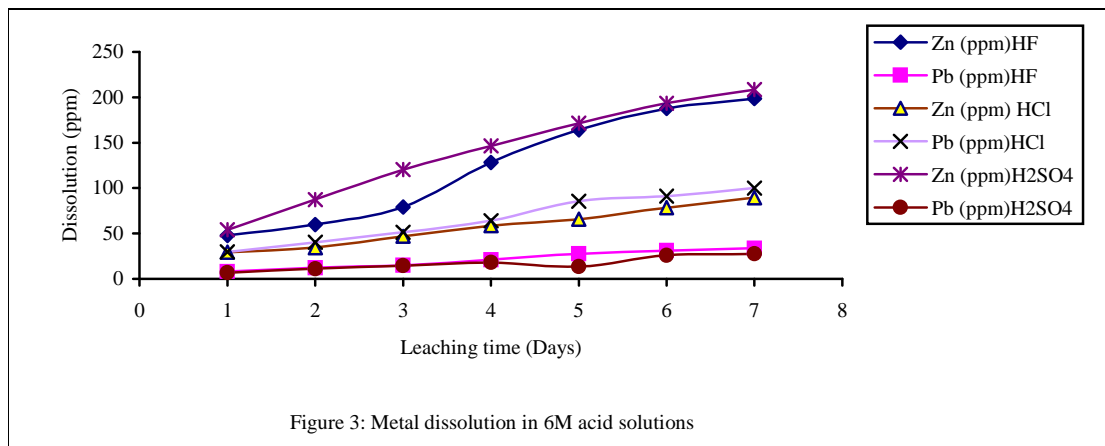
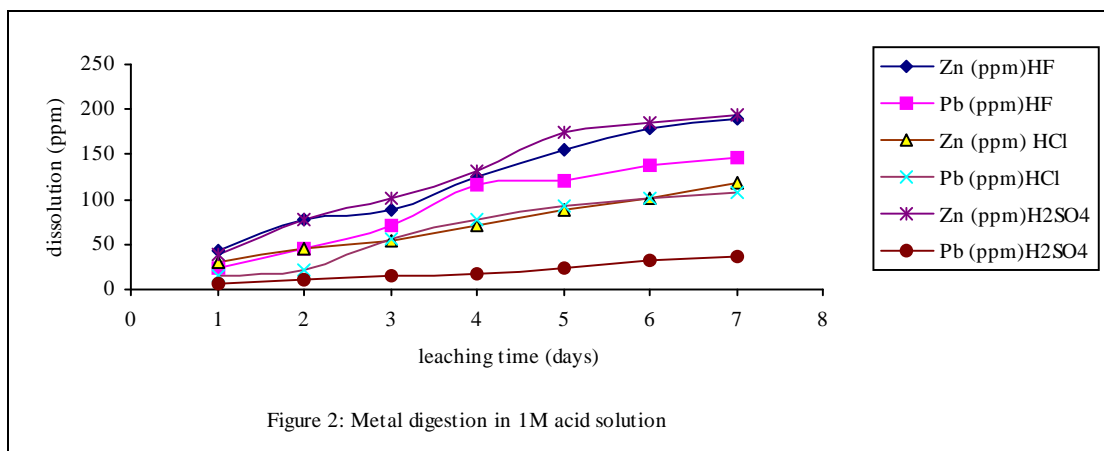
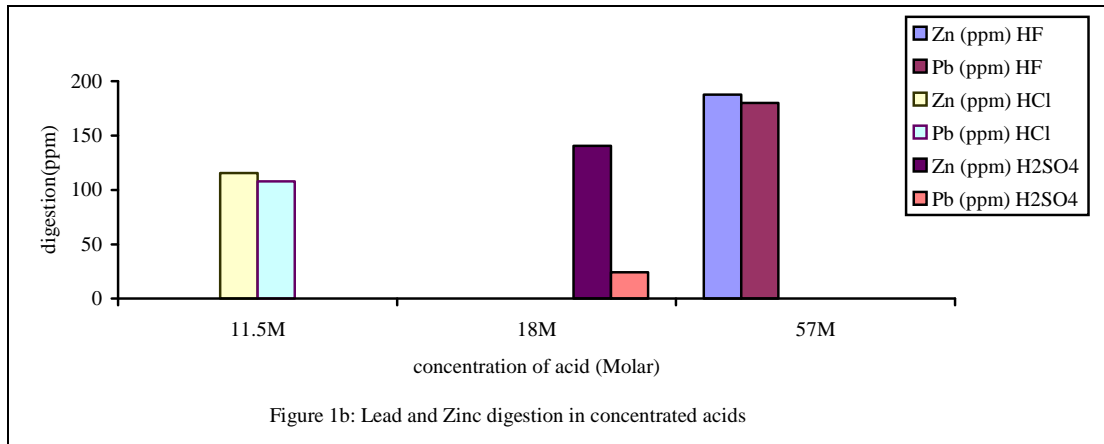
HCl is comparatively similar to what was previously obtained from the Pb dissolution in HF, though HF leaching yields higher dissolution values than in HCl leaching which produced Zn dissolution values respectively.

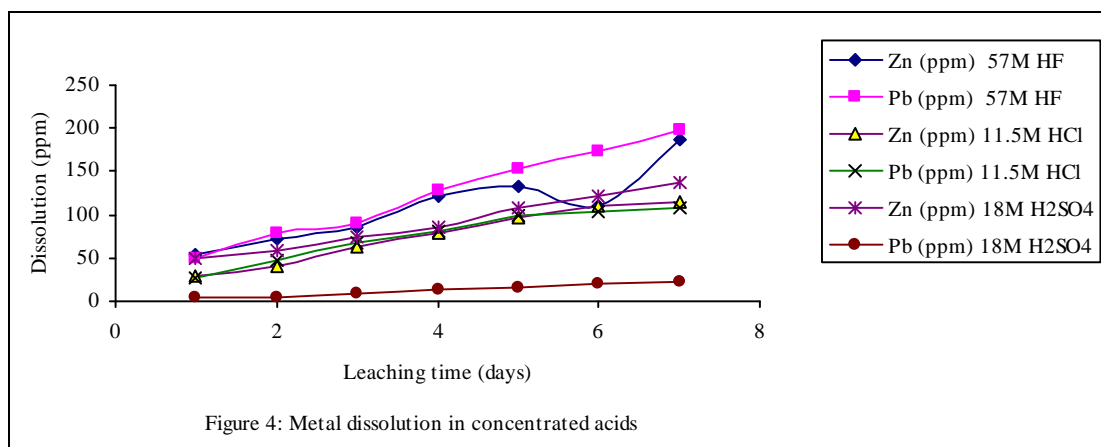
The manner of Zn dissolution in  $H_2SO_4$  is comparatively similar to that in the HF leaching. The results showing an increase in Zn dissolution as the  $H_2SO_4$  concentration increase from 1 to 6M, above which value there was decrease in the amount of Zn dissolved in the acid. Thus the Zn dissolution dropped down to a lower dissolution value as the  $H_2SO_4$  concentration increased.  $H_2SO_4$  leaching presents the least Pb dissolution values of all the three acids used. It is clearly understood that at very high concentration such as 18M, PbO does not have leach in  $H_2SO_4$  but for the application of heat, because metal is generally inactive in cold concentrated  $H_2SO_4$  in the complete absence of water. On the other hand, hot concentrated  $H_2SO_4$  acts as any other oxidising agent, accepting electrons in a chemical reaction. Hence at lower concentration of leachants (dilute  $H_2SO_4$  and HCl) lead II chloride and lead II sulphate are not formed quantitatively. Both products are almost insoluble in water and since the reaction can proceed only on the outside of the oxide (roast particle), each tends to form a layer of coat that is not permeable to these acids and hence the reaction is aborted before a substantial amount of salts ( $PbSO_4$  and  $ZnSO_4$ ) are formed. The risks involved in the use of the highly dangerous concentrated acids in this context appear not justified by the corresponding quantities of metals extracted.

Leaching process is characterised by the combination of oxidation and reduction reactions, hence, in multi-component oxide-acid reaction as applicable to this study, the effect of preferential discharge of ions in a redox reaction is obvious. From the characterised trends of results for Zn dissolution, Zn is more electropositive than Pb, and that  $ZnSO_4$  being more water solute than  $PbSO_4$ , make an advantage for Zn over Pb. At lower concentration the ionic activity is higher than what features at higher concentration. With the increasing concentration, the kinetic theory holds. At higher concentration of HF and  $H_2SO_4$  above 6M, the increase in concentration definitely affects the ionic activity (collision rate) [18]. The kinetic theory explains why there was decrease in the Zn dissolution at increasing 1 to 6M HCl concentration resulting from influenced effect of the reduction in the ionic activity, while the increased dissolution which occurred at higher increasing 7 to 11.5M HCl concentration [19].



Figures 2, 3 and 4 show the results of digestion of the lead - zinc oxide roast in 1M, 6M and concentrated solutions of HF, HCl and  $H_2SO_4$  respectively at increasing leaching time from 1 to 7 days. The result shows that for all cases of concentrations of leachants (HF, HCl and  $H_2SO_4$ ) examined for 7 days, there was increase in Zn and Pb dissolution as the leaching time (days) increases. Generally, the rate of leaching decreases as the metal dissolution comes closer to completion between 6 and 7 days leaching time. The slow down in the reactions is attributed to decrease in the concentration of the metals yet to go into solution, a decrease in strength of reagent (concentration of acid) and the low diffusion rates of leach liquors as the metallic dissolve the solid being leached.





Therefore, at the expiration of 7 days, the result shows that for 1M solutions, the Zn dissolution (ppm) increased in HCl and H<sub>2</sub>SO<sub>4</sub> just as Pb dissolution increased.

### CONCLUSION

The study has shown that hydro-metallurgical extraction by leaching method proved that HF, HCl and H<sub>2</sub>SO<sub>4</sub> has the economic potential to dissolve large quantity of both metals (Pb and Zn) present in the Abakaliki sulphide concentrate. In all cases of increasing concentration, there was corresponding reduction in the dissolution of Pb in H<sub>2</sub>SO<sub>4</sub> giving a decreasing Pb dissolution as the acid concentration increase. The result of the chemical analysis of the ore revealed the presence of large quantity of impurities of both higher and lower electropositive characteristics than Pb and Zn in the electrochemical series. The leaching became more effective with the application of heat, which raised the leaching temperature while the leaching rate increased with time.

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