Electrochemical Trace Analysis of Valuable Metals in Chalcopyrite ORE

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Abstract—*Processing Chalcopyrite ores, the standard way is by means of hydrometallurgical or pyrometallurgical processes, but with the odds of amplified exploitation of mixed and lower grade ores and relatively small isolated deposits, along with the conditions related to temperature and sulphatising environment and then dissolution, there has been a worldwide interest in electrochemical methods, for instance anodic and cathodic polarization. In this work we propose a technique which will eliminate of copper interferences in chalcopyrite mineral by applying an electrolytic separation and then separation of the chloride complex of iron by rapid extraction into duly isopropyl ether. The purpose of this paper is to provide a review of chalcopyrite deposits, production and consumption, mining, ore processing, and above all to explore he differential pulse polarograph method of instrumental (DPP) for determination of trace elements in ore.*

Keywords:—*Copper/Chalcopyrite, Polarography, Ore Extraction, Differential Pulse Polarography, Electrochemical Analysis.*

1. INTRODUCTION

Chalcopyrite (CuFeS2) is a copper and iron containing sulphide mineral that crystallizes in the tetragonal system.

Chalcopyrite is often confused with pyrite, although the latter belongs to a cubic crystal system. Furthermore, chalcopyrite is often massive, rarely crystalline, and less brittle. It is also a darker yellow in color, with a greenish-tinge and diagnostic greasy lustre. When exposed to air, chalcopyrite oxidizes to a variety of oxides, hydroxides and sulphates. It is often contaminated by a variety of other trace elements such as Co, Ni, Mn, Zn and Sn as substitutes for Cu and/or Fe, as well as Se, Fe and As as substitutes for S. There is a limited number of studies concerning the determination of elements in copper andiron based geological samples by various methods, such as atomic absorption spectrometry (AAS) [1–8], atomic emission spectrometry with inductively coupled plasma (ICP-AES) [9, 10] or laser ablation microprobe-inductively coupled plasma-mass spectrometry (LAM-ICP-MS) [11]. Nuclear and nuclear-related techniques (INAA, XRF, PIXE) are rarely used to determine trace elements due to matrix and inter-element interferences and background effects [12–16]. In our previous work [17] it was found that the presence of copper as a matrix element in chalcopyrite interfered on the most of the trace element determination by the k0-instrumental neutron activation analysis (k0-NAA) even after Fe removal.

Consequently, it was concluded that it is obligatory to eliminate both copper and iron matrix elements in order to increase the

number of the determined present trace elements in the samples.

2. EXPERIMENTAL - INSTRUMENTATION

Chemicals and reagents

Polarographic measurements were done on elico model CL32 Bangalore India. Three electrode system consisting of a dropping mercury electrode (DME) as the working electrode, Ag/Agcl reference electrode, a coiled platinum wire as auxiliary electrode and a saturated calomel electrode (SCE) as reference electrode.

Preparation of the sample solution

Chalcopyrite ore was obtained from Jinadal Steel Power Plant, C.G. INDIA. Sample was crushed and finally pulverized.2g of ore sample was dissolved in 20ml of hydrobromic acid and evaporated to dryness and again dry sample was dissolved in nitric acid and evaporated to dryness. Finally the dry sample is dissolved in ammonium hydroxide containing tartaric acid; the solution was makeup to 100ml by distilled water.

Preparation of analyte and Recording of voltamogram / polarogram

The procedure for preparation of analyte, in the pasture of trace analysis, should be simple convenient, should not leave any contaminating element in the analyte and should not destroy the ions of interest at any level.

The procedure for the preparation of analyte from the sample solution is described as follows.

Ore Sample (Mixture of Chalcopyrite, And Sphelerite)

The sample solution (10 ml) was mixed with 10 ml of 1 M ammonium tartrate as supporting electrolyte and 10 ml of 0.01% gelatin as maximum suppresser was taken in a polarographic cell and, the final volume was

made up to 100 ml with distilled water. The pH of the test solution was adjusted to 9.0 ± 0.02, with ammonia solution. Pure nitrogen gas was bubbled through the test solution for 15 min and the pH of the test solution was checked before recording the polarogram / voltammogram.

3. RESULT AND DISCUSSION

Ore Sample of Chalcopyrite

The DP Polarograms and DCP Voltammogram of the sample saluting (Figure 1) showed ten well defined waves / peaks with $E_{1/2}$ / E_p values = -0.06/-0.08, -0.24/-0.28, -0.40/-0.42, -0.61/-0.63, -0.91/-0.92, -01.14/-1.16, -1.34/-1.34-1.40/-1.44 V vs SCE in DCP/DPP mode and Ep values = -0.04, -020,-0.46, -0.63, -0.90, -1.14, -1.36 V vs SCE, in DPASV mode. Indicating the presence of Ag(I), Au(II), Cu (II), Pb(II), Cd(II), Ni(II), Zn(II), Fe(III), and Cr(III) respectively in the ore sample. The difference in peak Potential in DPP and DCP modes may be explained on the basis of increasing ease of reduction on carbon fiber electrode (18-21).
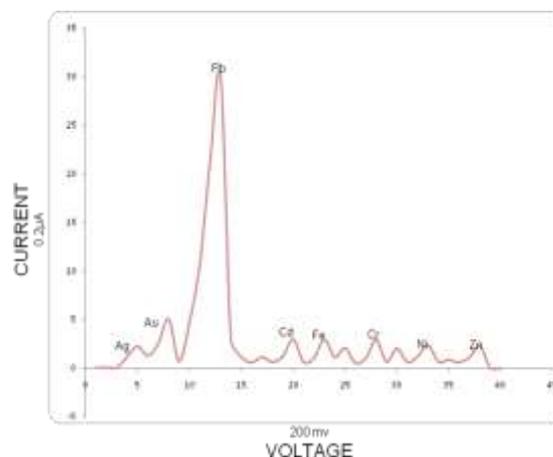
Figure 1: Direct Pulse Polarogram

To confirm the presence of the above mentioned metal ions in the sample, a known quantity of standard solution of each metal ion was added to the analyte and the resulting polarogram / voltammogram was recorded, which increased the observed wave / peak height of each metal ion signal without any change in $E_{1/2}$ / E_p values.

After confirming the presence of these metal ions in this sample, some synthetic samples of varying ion concentrations were prepared and their polarograms / voltammograms were recorded under identical experimental conditions. The linear relationship between the concentration of each metal ion and the corresponding wave / peak height was also unchanged confirming the possibility of an accurate simultaneous qualitative and quantitative determination of the metal ions in the sample. The concentration of each metal ion (taken/found) in synthetic samples using DPP has been given in the Table - 2

Table 2: Analysis of Synthetic Samples

Compositions of synthetic samples (mg/ 100 ml)

Ag	Au	Pb	Cd	Cu	Cr	Ni	Zn
0.01	1.27	10.73	0.56	0.61	0.51	0.59	0.56
(0.01)	(1.26)	(10.72)	(0.56)	(0.60)	(0.50)	(0.59)	(0.56)
0.02	2.52	21.42	1.12	1.13	1.00	1.17	1.12
(0.02)	(2.52)	(21.42)	(1.11)	(1.04)	(1.00)	(1.18)	(1.12)
0.04	5.04	42.84	2.24	3.01	2.00	2.34	2.24
(0.04)	(5.02)	(42.80)	(2.23)	(2.91)	(2.00)	(2.33)	(2.24)
1.08	0.63	2.07	1.12	0.57	0.51	0.54	0.63
1.16	0.68	2.93	1.18	0.68	0.62	0.64	0.72
(1.15)	(0.67)	(2.91)	(1.18)	(1.10)	(0.63)	(0.63)	(0.71)
2.24	4.67	1.12	2.98	1.99	2.02	0.84	0.72
(2.23)	(4.67)	(1.12)	(2.96)	(2.05)	(2.01)	(0.84)	(0.72)

* Amount Found Using DPP * Average of four determination.

Top of Form

Bottom of Form

Minimum tried detection limits of DCP, DPP

The minimum tried detection limits (smallest quantities that have been used) for measurement of the individual and combined metal ions given in Table 3. Except for Ag (I) and Au (II), all the metal ions in the sample could be determined in one run. For Ag (I) and Au (II)

Table 3. Minimum Tried Detection Limits

		DCP ($\mu g\ l^1$)	DPP ($\mu g\ l^1$)
Ag	Individual	6.3	0.63
	Combined	6.3	0.63
Au	Individual	2.6	0.26
	Combined	2.6	0.26
Pb	Individual	1.1	0.11
	Combined	1.1	0.11
Cd	Individual	1.1	0.11
	Combined	1.1	0.11
Ni	Individual	6.6	0.66
	Combined	6.6	0.66
Zn	Individual	3.6	0.36
	Combined	3.6	0.36
Fe	Individual	0.6	0.66
	Combined	5.7	0.58
Cr	Individual	5.7	0.58
	Combined	5.7	0.56

Table 3.1 : Final Analysis Results on Chalcopyrite Ore Sample and Their Comparison with aas

Metal ion	Voltammetric (mg g^{-1} of the sample)	AAS
Au	12.7	13.8
Ag	12	13
Cu	0.23	0.22
Pb	0.22	0.56
Cd	1.45	1.88
Cd	1.67	1.78
Ni	1.77	1.56

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