Electrochemical Trace Analysis of Valuable Metals in Hematite Ore Samples

Manju Rathore[1], Dr. Jyotsna Mishra[2]

[1,2]Dept of Chemistry, AISECT University, Bhopal (M.P) India.

ABSTRACT

Hematite is a main source of iron. Many other metals are also associated with it in different micro and trace level. Polarographic methods i. e. DCP and DPP have been successfully used for simultaneous determination of some metals viz. Au(I) ,Bi(II) ,Pb(II) ,U(VI),Co(II) ,Ni(II) ,Zn(II) ,Mn(II) ,Fe(III) Cr(III). Ammonium Tartrate solution was used as supporting electrolyte and gelatin was used as maximum suppressor at pH 9.0. The method of standard addition was applied for quantitative analysis. Spiked samples were also made and analyzed in same conditions for statistical approach. The obtained polarographic results were compared with those obtained using Atomic Absorption Spectrometry (AAS). The observed results clearly speaks of the superiority of Polarographic methods.

Key words: Hematite ore samples, Polarography, Atomic Absorption Spectroscopy

I INTRODUCTION

Hematite is the most important source of iron. It is an oxide mineral and its chemical formula is Fe_2O_3 . It contains high iron (about 70%) .and is abundant in nature (1). A number of investigations have been performed concerning the determination of elements, similarly geological samples have been studied by flame atomic absorption spectrometry (AAS), radiochemical neutron activation Analysis (2) and electro thermal atomic absorption spectrometry (ETAAS) with inductively coupled plasma(AES-ICP) (3-6). After the separation and concentration of the elements, its analysis have been performed (7-11). Some authors have investigated the possibility of the determination of these elements directly from the sample solution, by FAAS (11-13), and by ETAA (14). Also the influence of interfering elements was of particular interest in the flame or electro thermal methods. AAS determination and, to overcome such interferences, the addition of different matrix modifiers or the separation and concentration methods for the determination of the elements investigated were suggested (15). Neutron activation analysis (NAA), X-ray emission (XRA) (16), are rarely used to determine trace elements due to matrix and inter element interference and background effect (17).

II MATERIALS AND METHODS

Mineral samples was collected from Kanhwar Plateau, Agaria, Bijori, Ghosalpur, Lora hill, Ghoghra, Silondi, Saroli Jabalpur District (M.P.).

Conditions required for the Polarography of the Hematite ore samples

Stock Solution in Double Distilled water	:	0.01M
Supporting Electrolyte Ammonium Tartarate,	:	1M
Scan Rate /Sec.	:	5mV

III INSTRUMENTATION

Elico Pulse Polarograph model CL-90 was coupled with a Polarocard model LR-108 and a three electrode assembly (Elico Private Limited, Hydrabad, India) was used for precise qualitative as well as quantitative analysis of metal ions in samples. The electrode system consisted of a dropping mercury electrode as a working electrode, a coiled platinum wire as an auxiliary electrode and a saturated calomel electrode as a reference electrode.

IV PROCEDURES FOR SAMPLE SOLUTION

1 gm of powdered sample of hematite was dissolved in 12 ml aqua regia. A few drops of H_2O_2 were added and the solution evaporated to near dryness. The residue was then dissolved in 50 ml of 10 N HCl. After dissolution of the mineral samples, the solution was transferred into a separating funnel. Then 10 ml of iso amyl acetate was added and the mixture was shaken for 1 minute. To avoid interferences of chlorides, the inorganic layer was evaporated and the residue was dissolved in 5 ml of

2 N HNO_3 (18).

V RESULT AND DISCUSSION

The DC and DP Polarograms of the sample solution (Fig 4a & 4b) showed ten well defined waves/peaks with E½ /Ep values = -0.06/-0.08, -0.32/-0.46, -0.58/-0.56,-0.68/-0.68,-0.86/-0.84, -0.91/-0.92,-1.14/-1.16,-1.20/-1.25,-1.32/-1.34,-1.40/-1.45, V vs SCE in DCP/DPP mode indicating the presence of Au(II), Bi(II), Pb(II), U(VI) ,Co(II) ,Ni(II) , Zn(II), Mn(II), Fe(III), Cr(III) respectively in the ore sample(19).

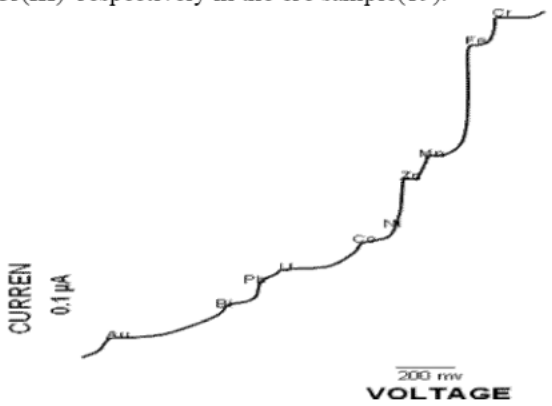

Fig 1a Direct Current Polarogram of Hematite Sample (100mg /100ml) in 1 M Ammonium Tartrate + 0.01% gelatin at pH 9.0 ±0.1.

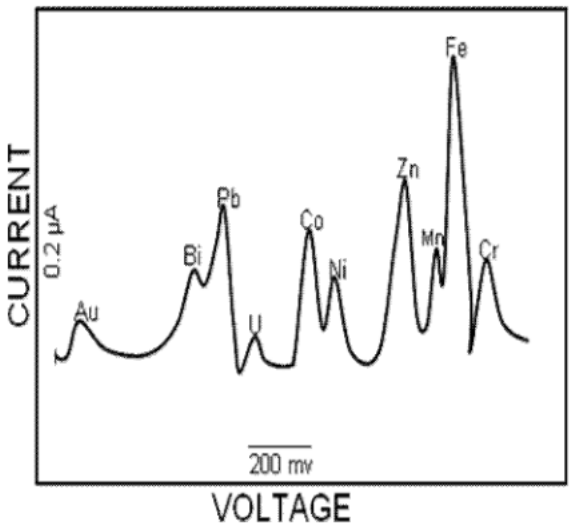

Fig 1b Differential Pulse Polarogram of Hematite Sample (100mg /100ml) in 0.1 M Ammonium Tartrate + 0.01% gelatin at pH 9.0±0.01.

To ascertain the presence of the above mentioned metal ions in the sample, a definite quantity of standard solution of each metal ion was added to the analyte and the resulting polarogram was recorded.

On the basis of above metal ions present in the sample, some synthetic samples of varying concentration of the metal ions were prepared (Table-1.1) and each synthetic samples was analyzed polarographically. The results indicated no change in $E_{½}$ / E_p values of the above mentioned metal ions and also the varying concentration of these metal ions, did not affect the DCP and DPP of each other. Quantitative analysis of the sample using DCP and DPP of Au, Bi, Pb, U, Co, Ni, Zn, Mn, Fe Cr, in the sample were carried out by using DCP and DPP methods. Spiked samples were prepared in order to evaluate the concentrations of each metal ion. The results are given in Table 1.2. The results indicated that the percentage recovery is over 99.7% for all the metal ions, with high accuracy and precision of the determination.

The final analysis of results of the sample is shows in Table 1.3. The results were compared with those obtained using AAS method and were found to be in excellent agreement.

VI CONCLUSION

The proposed polarographic procedure was found to be highly useful for the determination of species in different oxidation state also e.g. Fe may be found in +2 or +3 which is otherwise not possible using AAS method. The comparative and statistical data also proves the superiority of the polarographic method for such type of analysis over the other methods.

Table1.1
Analysis of Synthetic Samples
Compositions of synthetic samples (mg/ 100 ml)

Au	Bi	Pb	U	Co	Ni	Zn	Mn	Fe	Cr
0.01	0.17	1.63	0.27	0.91	0.75	3.59	0.76	5.23	0.32
(0.01)	(0.16)	(1.62)	(0.25)	(0.90)	(0.74	(3.59)	(0.76)	(5.22)	(0.31)
0.02	2.32	2.42	1.04	1.08	1.80	5.17	1.42	3.23	1.95
(0.02)	(2.32)	(2.42)	(1.04)	(1.09)	(1.80)	(5.18)	(1.42)	(3.22)	(1.95)
0.05	3.04	2.83	1.24	4.41	2.09	2.14	1.20	1.96	3.32
(0.05)	(3.02)	(2.8)	(1.23)	(4.41)	(2.07)	(2.13)	(1.2)	(1.96)	(3.32)
1.17	1.83	1.07	2.12	0.77	2.81	4.56	2.63	7.13	9.82
(1.16)	(1.82)	(1.05)	(2.12)	(0.78)	(2.92)	(4.64)	(2.62)	(7.13)	(19.82)
1.10	1.67	2.71	2.88	2.15	2.63	5.34	3.71	8.25	9.32
(1.12)	(1.67)	(2.72)	(2.88)	(2.99)	(2.62)	(5.44)	(3.72)	(8.25)	(9.32)
2.2	3.66	1.10	3.92	3.05	3.06	3.84	3.79	6.99	5.65
(2.2)	(3.67)	(1.10)	(3.95)	(3.06)	(3.09)	(3.88)	(3.8)	(6.99)	(5.65)

( ) Amount Found * Using DPP
Average of four determinations

Table 1.2:
Polarographic Analysis Results of the Hematite Sample (Mg/100 mg)*for its metal content

Metal Ion	Parameter	By DCP			By DPP		
		Added		Found	Added		Found
Au(I)	Amount	-		0.012	-		0.013
		0.014		26	0.011		0.024
	% Rec		95.5			98	
	SD **		0.002			0.001	
Bi(II)	Amount	-		0 .27	-		
		0.26		0.53			1.27
	% Rec		99.2		1.29	99.2	2.56
	SD **		0.011			0.001	
Pb (II)	Amount	-		3.36	-		
		3.46		6.82	2.96		2.99
	% Rec		99.5			99.4	5.95
	SD **		0.02			0.02	
U(VI)	Amount	-		0.145	-		
		0.134		0.279			0.125

	% Rec SD **		99 0.003		0.124	99.5 0.003	0.249
CO(II)	Amount % Rec SD **	- 0.425	96.2 0.006	0.426 0.851	- 0.825	99.1 0.007	0.826 1.651
Ni(II)	Amount % Rec SD **	- 8.03	99.8 0.004	8.025 16.05	- 8.04	99.6 0.003	8.04 16.08
Zn(II)	Amount % Rec SD **	- 3.065	97 0.004	3.015 6.08	- 1.095	97.2 0.002	1.094 2.189
Mn(III)	Amount % Rec SD **	- 10.05	100 0.005	10.02 20.07	- 10.15	100 0.006	10.17 20.32
Fe(III)	Amount % Rec SD **	- 20.34	98.5 0.003	21.34 41.68	- 16.33	98.5 0.003	16.35 32.68
Cr(III)	Amount % Rec SD **	- 9.87	97.7 0.004	9.85 19.72	- 9.87	97.7 0.004	9.89 19.76

Table –1.3
Final Analysis Results on Hematite Ore Sample and Their Comparison with AAS

Metal ion	Polarographic	AAS
	(mg g^{-1} of the sample)	
Au (I)	0.012	0.013
Bi (II)	0.27	0.28
Pb (II)	3.36	3.44
U (VI)	0.145	0.16
Co(II)	0.426	0.425
Ni (II)	8.25	8.35
Zn(II)	3.015	3.017
Mn(II)	10.02	10.06
Fe(III)	213.4	214.6
Cr(III)	9.85	9.83

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