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Leaching Studies of Braunite With Sulphuric Acid and Phenol-Reducer

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In this paper, sampling was carried out on the manganese deposit at Benesbord, 65 kilometers northwest Sabzevar in Khorasan, as the representative of the province deposits (regarding mineralogy and genesis) and its mineral dressing operations were conducted. The main minerals constituting this ore include quartz, braunite (manganese silicate) and hematite in which the weight percentages of SiO_2, Mn and Fe_2O_3 are 64.5, 14.0 and 8.5 %, respectively. Owing to its genesis, this ore has two structures. First, filling into vein lets which may be concentrated through (shaking table and jig) physical methods. The other one, being dispersed throughout the whole ore, has dimensions of about some microns. It may not be recovered and concentrated with the above-motioned physical methods, having a gangue grade of at least *ca.* 6 %. Thus, for the complete recovery of manganese in the ore, the leaching operations were performed using sulphuric acid and the phenol-reducer on the sample with dimensions of about 1 mm. However, the final findings suggested that the recovery of the leaching operations using sulphuric acid and the phenol-reducer, in different weight percentages and amounts, were not successful, reaching a maximum of *ca.* 13.5 %.

Key Words: Braunite, Manganese, Benesbord, Leaching, Sulphuric acid, Phenol-reducer.

INTRODUCTION

Nowadays, almost everyone knows the importance of manganese. Its increasing applications have caused the craftsmen to attach more value on such a metal. Because of economic reasons and its having special chemico-physical characteristics, manganese is considered as an industrial strategic metal. In steel industries, it is mainly used as an alloy factor for producing resistant steel[1-3].

Manganese, moreover, has other applications such as producing batteries, dyeing bricks, clothes, ceramics, glass and earthenware: producing chemical fertilizers, providing livestock and poultry feed, making welding electrodes, producing colour-drying materials, water and waste-water treatment, making fuel additives and providing oxidizers required in chemical processes. After steel industries, other ones like battery-producing, agricultural and chemical industries are among major consumers of this metal's compounds, most of which being imported from abroad at present.

Therefore, providing manganese or the required concentrate from national resources not only meets the needs of domestic industries, but also prevents a considerable amount of foreign exchange from going out abroad. It is for this reason that the leaching of the ore, as a potential for manganese, has been studied.

EXPERIMENTAL

The mineral identification has been carried out by different methods such as XRD (for qualitative analysis), chemical analysis (for qualitative analysis) and screen analysis test. The polished sections studies were used for recognizing the ore mineral types, its oxides' grades, manganese dispersion layout in different grading, as well as the ore genesis type of the sample, respectively.

Mineralogical studies: The major minerals of the ore, regarding their order of frequency, were recognized by the XRD method as quartz, braunite, hematite and calcite, respectively[3].

Chemical analysis: According to standard methods of providing samples[4], an indicter was sampled and chemically analyzed. Table-1 briefly shows the percentages of the ore elements.

TABLE-1
CHEMICAL ANALYSIS RESULTS OF THE ORE PRIMARY SAMPLE AT BENESBORD

Chemical composition	Oxide grades (%)	
	Low-grade sample	High-grade sample
SiO_2	64.50	45.11
MnO_2	18.36	32.41
Fe_2O_3	8.56	6.67
CaO	4.45	8.84
MgO	0.19	0.60
P_2O_5	0.03	n.d
Al_2O_3	n.d	n.d
Na_2O	0.60	0.53
K_2O	0.05	0.04
L.O.I	3.00	4.12

Practical size analysis: After the grinding stage, a pack of the primary sample was analyzed by means of screens with the meshes of 10, 14, 18, 25, 30, 40, 50, 70, 100, 140, 200, 270 and 400. The results obtained from screen analysis are summarized in Table-2.

According to the results of the screen analysis test, it is concluded that: (1) Manganese dispersion in all sections is almost the same and no significant difference, regarding the grades, is observed among various sections of grading. In other words, The grades' changes do not follow a definite procedure so that one cannot carry out a concentrated grading. (2) Manganese distribution in coarser grindings is more due to the high weight percentages of these sections.

TABLE-2
RESULTS OF THE MANGANESE PRIMARY SAMPLE
SCREEN ANALYSIS AT BENESBORD

Dimensions		Wt (g)	Weight %	Cumulative weight fraction		Manganese (%)	
+2	-7+10	420	21.07	78.93	21.07	18.74	13.18
+1.4	-10+14	210	10.54	68.39	31.61	9.96	14.00
+1	-14+18	317	15.91	52.48	47.52	16.66	15.52
+0.71	-18+25	203	10.19	42.29	57.71	10.22	14.84
+0.6	-25+30	250	12.54	29.75	70.25	12.22	14.99
+0.425	-30+40	110	5.53	24.23	75.77	5.75	15.42
+0.3	-40+50	127	6.37	17.86	82.14	7.66	17.83
+0.212	-50+70	80	4.01	13.85	56.15	4.37	16.15
+0.15	-70+100	51	2.56	11.29	88.71	3.03	17.52
+0.106	-100+140	18	0.90	10.39	89.61	1.54	17.14
+0.075	-140+200	48	2.41	7.98	92.02	2.75	16.89
+0.053	-200+270	18	0.90	7.08	92.92	0.98	16.06
+0.038	-270+400	45	2.26	4.82	95.18	2.32	15.25
-0.038	-400	96	4.82	–	100	3.84	11.80
Sum	–	1993	100	–	–	100	14.82

Microscopic studies: In the microscopic studies done on the sample. The four minerals of braunite, pyrolusite, cryptomelane and psilomalane have been respectively mentioned the most important one of which is braunite. Braunite is constituted in the form of automorphic crystals with compact texture. The dimensions of its crystals ranging from 10 to 150 µ. About 50 % of the braunite has changed into psilomalane due to the alteration process. These alterations occurred inwards the braunite crystals so that the crystal residuals are surrounded by an aurobe of psilomalane. At last, through the microscopic studies conducted on the primary sample, two structural geneses have been mentioned, *i.e.*, manganese filling inside the veinlets and the almost uniform dispersion into the ore with the dimensions of about some microns. The lack of manganese quite separability through physical methods proves the presence of the second type genesis in the ore, wich has caused the grade of the manganese in mine gangues not to decrease less than 6 % *via* gravity methods.

Experimental methods

Acidic leaching tests with sulphuric acid: According to the microscopic studies conducted on the ore, the presence of the second type genesis (with an almost uniform dispersion throughout the ore) has caused the grade of manganese gangues to be at least 6 % by the (shaking table and jig) gravity methods. For this reason, acidic leaching tests have been performed on the primary sample using the four parameters of time, acid concentration, pulp concentration and the phenol-reducer to increase the general recovery and eliminate the grading physical processes. Finally, providing optimum test conditions, in case of the leaching operations with sulphuric acid being positive, is one of the main objectives of the project.

Sample preparation: About 4.5 kgs of the primary sample underwent grinding by means of a rod mill, in periods of 15 min, with a pulp concentration of 50 %, using a screen with a mesh of 14 in a closed cycle. The grinding products were passed through the screen having the meshes of 18, 40, 60, 120, 130 and 400, respectively. The results of the screen analysis are summarized in Table-3.

TABLE-3
SCREEN ANALYSIS RESULTS OF THE MILLING PRODUCTS FOR THE LEACHING OPERATIONS WITH SULPHURIC ACID

Screen No.		Weight (g)	Weight percentage	Cumulative weight fraction	
Mesh	Micron			Passed	Retained
-14+18	+1000	462	10.41	10.41	89.59
-18+40	+420	837	18.87	29.28	70.72
-40+60	+250	554	12.49	41.77	58.23
-60+120	+125	1090	24.57	66.34	33.66
-120+230	+62	448	10.10	76.44	23.56
-230+400	+37	238	5.36	81.80	18.20
-400	1-36	807	18.19	100.00	0.00
Sum	–	4636	100.00	–	–

According to Table-3, it may be concluded that the grading of the sample indicator for acidic leaching tests was about 1 mm. Also, two of the prepared samples were subjected for manganese analysis, the median concentration of the primary sample being 14 %.

Test designing based on the quality K-4 software: To save time and expenses, attempts were made to obtain the best attainable results with the Quality K-4 software. Therefore, the lower and upper limits of the leaching parameters are guessed, in the primary stage, based on experience and also available information of the resources. The results obtained are summarized in Table-4. The conditions and number of the tests are summarized in Table-5.

TABLE-4
LOWER AND UPPER LIMITS OF DESIGNING PARAMETERS IN LEACHING OPERATIONS

Parameters	Lower limit	Upper limit
Time (min)	10	60
Sulphuric acid concentration (%)	5	30
Phenol-reducer (mg)	2	6
Pulp concentration	20	5

After recognizing the conditions required for carrying out the tests, they were conducted according to the conditions presented in Table-5. Their solutions were, at the end, sampled and analyzed with the atomic absorption method. In fact, the test with the highest recovery may have the best test results.

TABLE-5
CONDITIONS OF CONDUCTING MANGANESE LEACHING TESTS WITH SUCH PARAMETERS AS TIME, ACID CONCENTRATION, PULP CONCENTRATION AND THE REDUCER

Parameters	Test No.							
	1	2	3	4	5	6	7	8
Time (min)	10	10	10	10	60	60	60	60
Sulphuric acid concentration (%)	5	5	30	30	5	5	30	30
Phenol (mg)	2	6	2	6	2	6	2	6
Pulp concentration	20	50	50	20	50	20	20	50

Therefore, after calculating the recovery for each of the tests above and entering them into the computer, the best conditions and attainable recovery ranges may be guessed for the sample with a confidence level of more than 90 %.

For the final controlling, the determined test is carried out and its recovery in the relevant interval is studied. If the obtained recovery lies in the arithmetic recovery, the optimum conditions may be accessible through test designs (Table-6).

TABLE-6
RESULTS OF ATOMIC ABSORPTION ANALYSIS AND RECOVERY CALCULATION

Conditions	Test No.							
	1	2	3	4	5	6	7	8
Charged Solution volume (L)	0.37	0.23	1.21	0.46	0.24	0.48	0.49	0.39
Grade (g/L)	1.19	1.88	3.04	3.49	2.15	1.34	3.78	2.69
Recovery (%)	3.16	3.00	4.60	11.48	3.69	4.60	13.20	7.60

In optimum laboratory conditions, the parameters of time, acid concentration, pulp concentration and phenol were 1 h, 30 %, 20 % and 3 mg, respectively. Its accessible recovery, with a confidence level of more than 90 %, was R = 10.91 2.41. At last, the recovery of the final test mentioned was about 9.5 % that lies in the recovery the lower and upper limits of which were about 8.5 and 13 %, respectively. The above test confirms all the stages of the experiments.

Conclusion

(1) The maximum recovery of leaching operations with sulphuric acid is about 13 %. (2) The improper solubility and the low of the recovery may be attributed to rather ineffective role of the phenol-reducers on this mineral. (3) Due to the importance of the mineral (braunite) before leaching and the rather ineffective role of the phenol-reducer, it is suggested the effects of other reducer such as $KmNO_4$, O_3 and $NaClO_3$ as strong reducers be studied[5]. (4) Because of its being silicated (900 ºC), owing to its being inexpensive and frequent, the leaching methods with sulphuric acid is proposed.

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