

## Separation of Iron from Manganese Ore Roast-leach Liquor

Pravat K. Sahoo, Sarat C. Das, Subir K. Bose<sup>a</sup> and Sisir C. Sircar<sup>a</sup>

*Regional Research Laboratory, Bhubaneswar-751013, India and*

*<sup>a</sup>Department of Metallurgical Engineering, Indian Institute of Technology, Kharagpur-721302, India*

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The leach liquor obtained after the extraction of manganese from a low grade manganese ore contained both manganese and iron. In this paper an attempt has been made to remove iron by aerial oxidation. Parameters such as pH, temperature and time were varied in order to arrive at an optimum condition. It was observed that at a temperature of 333 K, pH 5.5 and aeration time of 2.25 h the iron could be completely removed at an air-flow rate of 1000 cm<sup>3</sup> min<sup>-1</sup> without any loss of manganese.

### 1. Introduction

The removal of iron from leach liquor is a common problem in many hydrometallurgical processes. The separation of iron from leach liquor is usually accomplished by oxidising all the iron present to ferric state and then precipitating Fe(III) by hydrolysis.

During the extraction of manganese from the ore in a hydrometallurgical method, some iron along with other impurities usually pass into the leach liquor and these need to be separated before further treatment of the liquor. Such studies have been carried out by different authors.<sup>1-5</sup> Kishnannaiv has separated iron from manganese rich liquor by aerial oxidation at a pH of 5 using a mixture of ammonium chloride and ammonium hydroxide. The iron from the manganese leach solution can be separated by precipitation of iron by lime addition.<sup>2</sup> Nikolov *et al.*<sup>3</sup> precipitated iron in the form of iron hydroxide by oxidation with manganese dioxide and then by neutralisation with ammonia solution whereas Ichiro and Okajimo<sup>4</sup> used sodium hydroxide to precipitate ferric iron from manganese sulphate solution. The separation of Fe(III) from manganese sulphate solution was best carried out at 333 K and pH 4.6–5.6. However, when the solution contained both ferrous and ferric iron, precipitation had to be carried out at pH 7.5–8.0. Shengeliya and Agladze<sup>5</sup> separated iron from manganese sulphate solution by extraction with fatty acid.

In the present study an attempt has been made to separate iron completely from the leach liquor obtained after the extraction of manganese from a low grade manganese ore. The initial pH of the leach liquor was 2.0 and pH was further raised by the addition of lime slurry (20%). The pH could be raised by the addition of ammonium hydroxide, sodium hydroxide and mixture of ammonium hydroxide and ammonium chloride.<sup>1-4</sup> In the present study lime slurry was used because of its availability and low price.

### 2. Experimental

#### 2.1. Ore and its treatment

The manganese ore used in the present study was obtained from the Keonjhar district, Orissa, India and contained 28% manganese and 26% iron along with other impurities like silica and alumina. The low manganese content makes it unsuitable for use in metallurgical industries and hence it was proposed to leach out selectively the manganese value from the ore in order to prepare high pure metal or its salts. The process consisted of grinding the ore (–250 B.S.S.), blending with

ammonium sulphate, roasting at 923–973 K for 1 h and finally leaching in water at 313–323 K. Under optimum conditions, the experiments were carried out taking manganese ore (1 kg) and the required amount of fertiliser grade ammonium sulphate. The resultant solution leached out 80% manganese and 10% iron present in the ore.

## 2.2. Procedure

The calcium hydroxide used was of laboratory reagent grade. Distilled water was used for all the experiments. The apparatus used consisted of a three-necked, round-bottom flask (250 cm<sup>3</sup> capacity), a voltage regulator a heating mantle, an air compressor for bubbling air, an air flow meter to measure the volume of air, a thermometer and a pH meter.

A measured amount of leach liquor was placed in a glass beaker. Initially, the solution was analysed for manganese and iron and its pH was measured. The pH was raised to the required value by adding lime slurry (20% solution) stepwise with constant stirring. The solution was then transferred into the three-necked flask which was subsequently kept on a heating mantle. When the required temperature was attained, air bubbling commenced. The air was bubbled through the suspension with the help of a glass bubble head. The bubbler was fitted to the central neck of the three-necked flask and it was always immersed in the suspension to the same depth (~20 mm). The bubble head had about ten holes of approximately 1 mm diameter symmetrically distributed over its surface and it almost touched the bottom of the flask. While the air was bubbled through the bubbler the other two necks of the flask were closed. There were arrangements in the bubble head for exit of air after bubbling. The pH of the solution was checked intermittently by the pH meter and the pH was adjusted to the required level by adding lime slurry. The time was noted as soon as the bubbling commenced. After the aerial oxidation the solution was filtered at once into a flask containing dilute hydrochloric acid, so that further oxidation of iron did not take place. The filtrate was subsequently analysed for iron (using the standard method of analysis with potassium dichromate) and manganese by colorimetry.<sup>6</sup>

## 3. Results and discussion

Experiments were carried out in order to obtain an optimum condition for the removal of iron. Each time 100 cm<sup>3</sup> of solution was taken for study. The original pH of the solution was 2.0 and the manganese and iron concentration were 18.75 and 0.745 g dm<sup>-3</sup> respectively. Parameters like pH, temperature and time were studied.

### 3.1. Effect of pH on co-precipitation of iron and manganese

As the pH is raised iron precipitates as ferric hydroxide but at higher pH values some manganese also precipitates along with the iron. This is not desirable. This set of experiments was carried out to find a suitable pH value where maximum iron separated without any loss of manganese. The results are shown in Table 1.

**Table 1.** Effect of pH on co-precipitation of iron and manganese. Temperature=330 K. Rate of air bubbling, none

Expt no.	pH of soln	Fe removed (%)	Mn loss (%)
1	3.0	15.66	No loss
2	4.0	38.79	No loss
3	4.5	48.09	No loss
4	5.0	50.44	No loss
5	5.5	59.45	No loss
6	6.0	64.87	0.001

From Table 1, it can be seen that even at pH 3, 15.66% of iron is removed and with increase of pH from 3 to 6, the percentage of iron removal increases from 15.66 to 64.87. Up to pH 5.5 the percentage of iron removed increases without any loss of manganese. But at pH 6, although more iron is removed, a small amount of manganese is co-precipitated. Therefore, for all other studies a pH of 5.5 was taken as the optimum.

### 3.2. Effect of rate of air flow

The air flow was varied from  $250 \text{ cm}^3 \text{ min}^{-1}$  to  $1250 \text{ cm}^3 \text{ min}^{-1}$  with an interval of  $100 \text{ cm}^3$ . The aeration was carried out for 2.5 h at pH 5 and 333 K. It was observed that with air flow rate of  $1000 \text{ cm}^3 \text{ min}^{-1}$  or above the solution became free of iron. For all further studies an air flow rate of  $1000 \text{ cm}^3 \text{ min}^{-1}$  was taken as the optimum.

### 3.3 Effect of temperature on complete iron removal

To find out a suitable temperature, the aerial oxidation of iron was studied at different temperatures at pH 5.5 and an air flow rate of  $1000 \text{ cm}^3 \text{ min}^{-1}$ . The oxidation was carried out at each temperature until there was a complete removal of iron. Thus at each interval of 15 min  $5 \text{ cm}^3$  of the solution was pipetted, filtered and the filtrate was tested for both ferrous and ferric iron. The results are presented in Table 2.

Table 2. Effect of temperature

Expt no.	Temperature (K)	Time taken for complete removal of iron (h)
1	300 (Room temp.)	4.5
2	313	3.5
3	323	3.25
4	333	2.25
5	343	2.25
6	353	2.25

The results (Table 2) indicate that at room temperature it takes the maximum time for complete removal of iron whereas with rise in temperature the required time decreases and remains constant beyond 333 K. For further experiments a temperature of 333 K was fixed as the optimum.

### 3.4. Effect of pH on complete removal of iron

The aerial oxidation of iron was studied at 333 K and air flow rate of  $1000 \text{ cm}^3 \text{ min}^{-1}$ . Here the oxidation process was continued until there was complete removal of iron at each pH. From the results (Table 3) it can be seen that at lower pH values it is difficult to remove the iron completely and it takes a long period of time. Beyond pH 4.5 only, is complete removal of iron possible. The rate of oxidation of  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  increases with increase in pH. Thus, it may be assumed that the oxidation of  $\text{Fe}^{2+}$  is pH dependent.

Table 3. Effect of pH on total iron removal

Expt no.	pH of the soln	Time taken for complete removal of total iron (h)
1	3.5	24 <sup>a</sup>
2	4.0	12 <sup>a</sup>
3	4.5	4.5
4	5.0	2.5
5	5.5	2.25

<sup>a</sup> Traces of iron still present.

### 3.5. Effect of time on iron removal

The rate of iron removal with time was studied at the optimum conditions i.e. pH 5.5; temperature, 333 K and air-flow rate; 1000 cm<sup>3</sup> min<sup>-1</sup>. The results are presented in Table 4.

Table 4. Rate of iron removal with time

Expt no.	Time of oxidation (min)	Iron removal (%)
1	20	91.01
2	40	93.33
3	60	96.03
4	90	98.35
5	120	99.56
6	135	100.00

It can be seen that even after 20 min of oxidation more than 90% of the iron is removed. Further, with increase in aeration time, the rate of iron removal seems to be slow. This may be explained as follows: as the pH of the solution is increased above 3, all the ferric ions are hydrolysed at a faster rate and thus precipitate under experimental conditions. The ferric part being the major portion, most of the iron is removed during the first 20 min. Further aeration assists in the oxidation of ferrous to ferric iron which subsequently hydrolyses and finally precipitates as the hydroxide. From this study it may be assumed that the oxidation process of ferrous iron is a slow process. Though the major portion of iron is separated within the first 20 min, it takes about 2 h for the complete precipitation of the iron.

### 4. Conclusion

The present study demonstrates that the iron can be completely separated from the leach solution without co-precipitation of manganese. However, as it is a complex solution it was not possible to study the kinetics of the process. It is proposed that such studies will be carried out with a synthetic solution.

### References

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