Recovery of Acid and Ferrous Chloride from Leach Liquor of Reduced Ilmenite by Hydrothermal Process

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Abstract

Ilmenite is the most abundant mineral for the extraction of titanium. It contains more than 50% TiO_2 along with iron, silica and alumina. The spinel structure of ilmenite ($FeO.TiO_2$) is so that iron is bounded in the lattice of TiO_2 matrix. To remove iron from the ilmenite is a major task for process metallurgists to increase the content of TiO_2. Various processes have been applied to remove this iron. One of them is reduction and leaching. The carbon containing pellets and environment of jhama coal reduces the iron oxide to metallic state. This metallic iron in then leached in dilute hydrochloric acid (20 vol%) and a greenish color leach liquor is obtained. The pH of the leach liquor solution is found 0.31. This leach liquor contains hydrochloric acid and iron as ferrous chloride. These acid and precipitated ferrous chloride was separated by hydrothermal process in the present investigation. The residue containing ferrous chloride was characterized with the help of XRD and EPMA. During experiment it was found that complete separation of HCl and precipitated ferrous chloride is feasible. The final pH of separated HCl is found 1.66 and it is suitable for reuse in the leaching of reduced ilmenite and the process-loop. The purity of ferrous chloride is in line with commercial grade which is a sellable product.
Keywords: Ilmenite, Reduction, Ferrous chloride, Hydrochloric acid.

1. Introduction

Ilmenite is the main source of titanium found in the earth crust [1]. It is found in India near sea beach of Kerala and under mines of Orissa [2]. The recovery of titanium is difficult from ilmenite as it is bounded with iron. Therefore, to recover titanium, iron has to be removed from the ore [3]. For this a thermal treatment prior to recovering iron is required. CSIR-NML has developed a process to recover titanium as titanium dioxide [4] by removing iron from the ilmenite ore. In this process ilmenite is reduced with carbon where iron is reduced to metallic iron. This iron is then leached with moderate hydrochloric acid where iron is recovered as chloride leaving behind titanium dioxide in the residue. This titanium dioxide is for sell and have good market potential. During leaching with hydrochloric acid the ferrous chloride containing leach liquor is obtained. This also contains free acid. The aim of the present invention is to recover these byproducts at laboratory scale and the results are depicted here.

2. Materials and method

The raw material for the use of this study was leach liquor obtained during the upgradation of ilmenite reduced product. The leach liquor used in this investigation contains Fe: 21.94%, HCl: 80 gpl and Ti: 1.28%. The pH of initial sample was pH: 0.31. The leach liquor was processed in the Rotary Evaporator in which the evaporated hydrochloric acid was condensed in a separated vessel attached to the reflux condenser unit, having glass spiral cooling system along under the vacuum system connected with the vacuum pump. The round bottom flask containing leach liquor was fitted in hot water bath to increase

the temperature to evaporate the hydrochloric acid. During heating under the vacuum pressure of 400-500 mill bar water along with acid vaporized and go through spiral condenser and is finally collected as cooled mass in the attached vessel.

3. Results and discussion

The metallized iron in reduced product was 15.38%. The XRD of reduced ilmenite also shows the presence of metallic iron as shown in Fig 1. This metallic iron has to be removed to get pure titanium dioxide. It is leached with hot hydrochloric acid (20% v/v) in a condenser. After leaching iron comes into the solution and a greenish solution obtained.

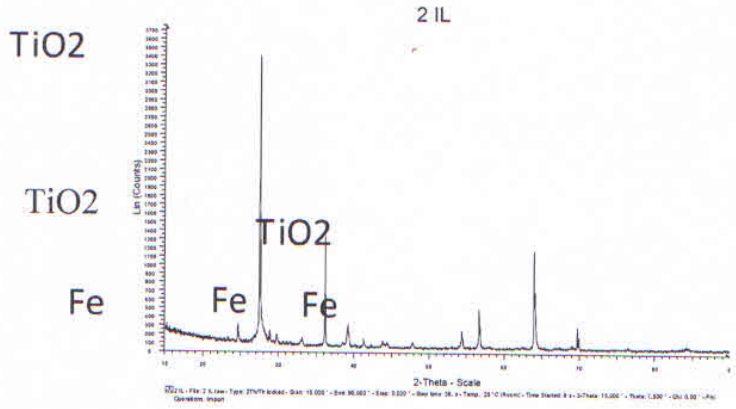

Fig.1. XRD pattern of reduced ilmenite

From this leach liquor the separation of acid and dissolved ferrous chloride is done. At 95-98°C the evaporation done and the cooled mass is collected as acid in the bottom of flask. During evaporation the pH of solution changes which signifies the rate of acid evaporation with time as depicted in Fig 2.

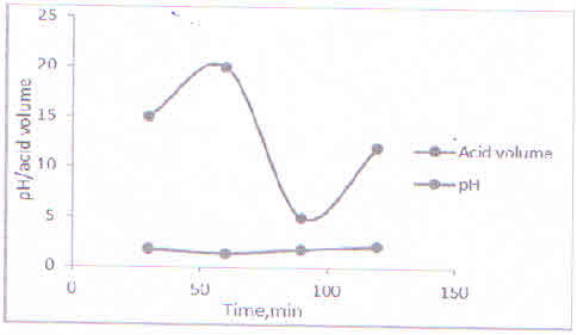

Fig.2. Acid and variation of pH with time

It can be seen from the Fig 2 that the pH of solution increases with time. At 30 min of evaporation the pH of condensed acid is 2 which becomes down at 60 min and thereafter it increases to 2.5. The observations indicated that at initial course of reaction acid and water evaporated simultaneously thereafter only acid recovered. The volume of solution is also initially very high which indicates the water evaporation along with acid. After that volume is low which is only acid recovery.

After the complete evaporation of acid, the solid mass remains in the flask. It was taken out by scratching with spatula and dried. Some portion of the sample was subjected to XRD and EPMA analyses. It is inferring with the peaks of XRD the deposited mass was hydrated ferrous chloride as depicted in Fig.3. The peaks found as hydrated ferrous chloride confirms the presence of this phase, only. The purity of the product was also analyzed to be 99.5% pure which is a sellable product. It is used in dyeing, in medicine and sewage treatment.

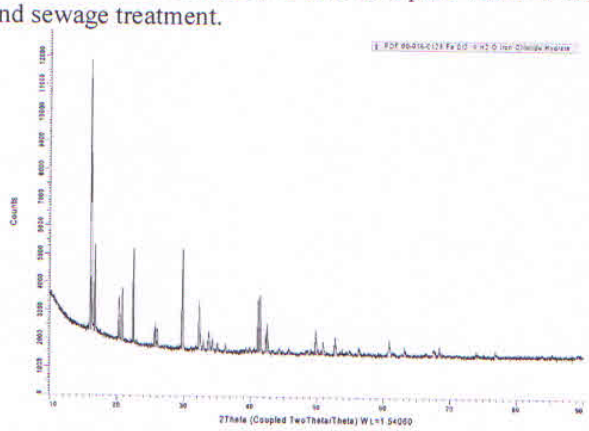

Fig.3. XRD pattern of hydrated ferrous chloride

A portion of sample product was subjected to EPMA analysis. It was found from the images that it is dendritic like structure. All along the image similar type of phases are present as shown in Fig4.

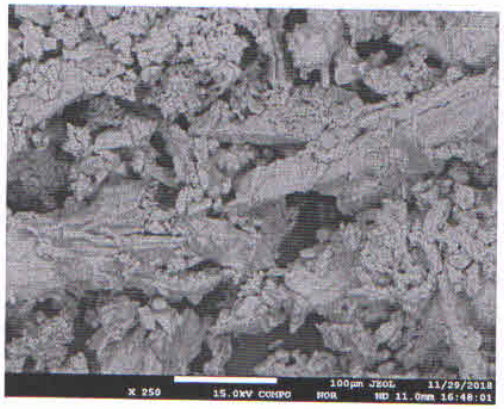

Fig.4. EPMA analysis of hydrated ferrous chloride

4. Conclusions

1. It is feasible to extract iron values from the waste leach liquor of reduced ilmenite as a ferrous chloride.
2. The recovery of acid along with the iron values as ferrous chloride is possible by hydrothermal process.
3. Initially acid recovery is slow which increases with time and almost complete recovery of acid is possible.
4. The high purity solid crystalline hydrated ferrous chloride is recovered and is of commercial grade.

5. Acknowledgment

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6. References

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