In. 23rd Int. Conf. Nonferrous Minerals and Metals,12-13th July 2019; Eds. B.D.Pandey, Abhilash, M.Ghosh, M.Najar

Utilization of roast reduced ilmenite leach liquor for ferrous chloride production 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. 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 colour 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 solid mass 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 $FeCl_2$ has done. The final pH of separated HCl is found 1.66 and it is suitable for reuse in the leaching of reduced ilmenite and process makes a loop. The purity of ferrous chloride is in line with commercial grade which is a sellable product

Keywords: Ilmenite; reduction; ferrous chloride; acid

1.Introduction

Ilmenite is the main source of titanium found in the earth crust, Chen et al., 2013. It is found in India near sea beach of Kerala and under mines of Orissa, Ray et al., 1985. 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, Deventer, et al., 1987. For this a thermal treatment prior to recovering iron is required. CSIR-NML has developed a process to recover titanium as titanium dioxide, Randhawa and Prasad, 2019, 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 has good market potential. During leaching with hydrochloric acid ferrous chloride containing leach liquor is obtained. This also contains acid. The present invention is with aims to recover these by-products 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 of ilmenite reduced product. Its composition is Fe: 21.94% HCl: 20 vol% and Ti: 1.28%. The pH of initial sample was pH: 0.31. The method

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used was a vaporization condenser method. It was fitted with spiral cooling system along with vacuum system. The round bottom flask containing leach liquor was fitted on hot water bath. During heating water along with acid vaporized and go through spiral condenser and finally collected as cooled mass in the attached flask.

3. Results and discussion

The metallised iron in reduced product was found 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.

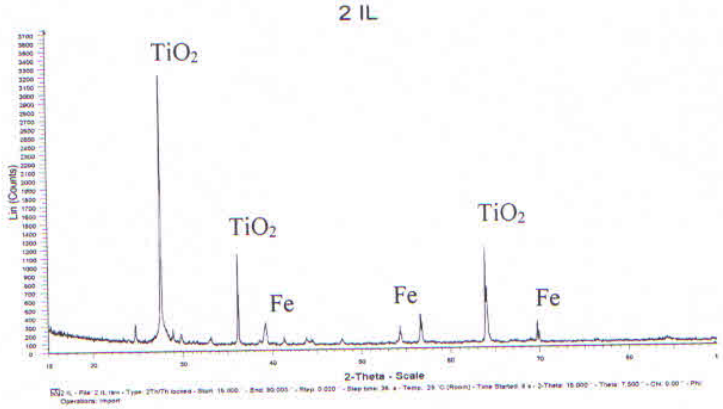

Fig.1. XRD pattern of reduced ilmenite

From this leach liquor the separation of acid and dissolved ferrous chloride is done. At 95-98^{0}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 signify the rate of acid evaporation with time as depicted in Fig 2.

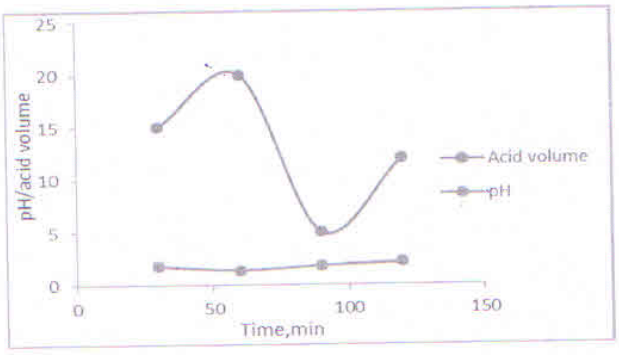

Fig.2. Acid and variation of pH with time

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 shows only acid is recovered.

After evaporation of acid the solid mass remains in the flask. It was scratched and dried. Some portion of sample was subjected to XRD and SEM analyses. It is inferring with the peaks of XRD the deposited mass was ferrous chloride as depicted in Fig.3. The peaks found as hydrated ferrous chloride confirms the presence of these phase. The purity of the product was also analyzed 99.5% pure which is sellable product.

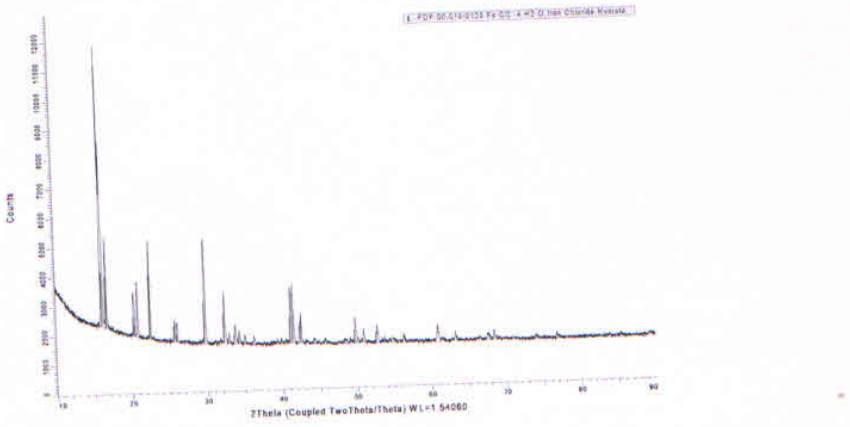

Fig.3. XRD pattern of hydrated ferrous chloride

A portion of sample product was subjected to SEM 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 Fig 4.

Fig.4. SEM analysis of hydrated ferrous chloride

4. Conclusions

Therefore, it is possible to extract iron from waste leach liquor of reduced ilmenite and following conclusions may be drawn.

1. The recovery of acid and iron as ferrous chloride is possible by hydrothermal process.
2. Initially acid recovery is slow which increases with time and almost complete recovery of acid is possible.
3. The solid product hydrated ferrous chloride is recovered and is of commercial grade.

Acknowledgments

Authors are thankful to CSIR-NML iPSG group for giving permission to work in the project and part finance of the project.

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