International Journal of Mineral Processing 134 (2015) 36–40

Contents lists available at ScienceDirect

International Journal of Mineral Processing

journal homepage: www.elsevier.com/locate/ijminpro

Catalytic leaching of ilmenite using hydrochloric acid: A kinetic approach

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ARTICLE INFO

Article history:
Received 27 November 2013
Received in revised form 26 September 2014
Accepted 11 November 2014
Available online 18 November 2014

Keywords:
Ilmenite
Catalytic leaching
Synthetic rutile
Titanium dioxide
Gypsum

ABSTRACT

The iron content present in the ilmenite is removed by leaching the ore with hydrochloric acid. The enriched TiO_2 mineral thus obtained is called synthetic rutile (SR) or beneficiated ilmenite (BI). The quality of the synthetic rutile (in terms of TiO_2 content) mainly depends on the condition of leaching process. The present study envisages the effect of acid concentration, temperature, and agitation speed on the leaching of the ore. The kinetics of the leaching reaction follows the first order at normal and stimulated leaching conditions. The highest rate constant value of 5.18×10^{-3} min^{-1} was observed for leaching the ilmenite with 30% hydrochloric acid under 700 rpm and at 70 °C. The enthalpy of the reaction (ΔH) was calculated as 24.157 kJ/mol and the entropy of the reaction (ΔS) was calculated as 26.25 J/mol/K. The positive value of ΔH indicates the endothermic nature of reaction. The value of ΔG at different temperature was found to be negative indicating the feasibility of the process and the spontaneous nature of the reaction. The addition of gypsum enhances the leaching process. The rate constant increases with the increase in the gypsum concentration in leaching process. The rate constant increased to 6.15×10^{-3} min^{-1} by introducing 100 mg of gypsum in the leaching process carried out with 30% hydrochloric acid at 70 °C and 500 rpm.

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1. Introduction

Titanium dioxide pigment has extensive applications in wide variety of fields including sensors, semiconductors, catalyst etc., but the main application is in the field of paint and plastics (Diebold, 2003; Croll and Taylor, 2007; Saeed, 2009; Narayan and Raju, 2000; Guez and Lorang, 1997). Commercial production of TiO_2 pigment is either through sulphate route or chloride route. The chloride process has many advantages over sulphate process due to its high quality products, eco-friendly process and generation of small amount of waste (Mehdilo and Irannajad, 2012; Zhang and Nicolo, 2010). Nowadays, about 60% of the world production of TiO_2 pigment is through the chloride route, in which a natural or synthetic rutile or titanium rich slag is used (Li et al., 2007). The shortage of natural rutile encouraged ilmenite upgradation by removing iron oxide and other impurities from the grain lattice of ilmenite and thereby converting ilmenite into synthetic rutile for chlorination process (Mehdilo and Irannajad, 2012). There are several processes for the production of synthetic rutile (Hazek et al., 2007; Mahmoud et al., 2004; Lasheen, 2009; Baba et al., 2009; Olanipekum, 1999), and the most suitable method in commercial production is the benalite process (Chen and Huntoon, 1977). Benalite process involves the reduction of ferric content of the ilmenite to leachable ferrous state by carbo-thermic reduction method. The reduced ilmenite was leached with hydrochloric acid to remove the iron content in the ilmenite and thereby enhancing the titanium dioxide content. The enriched titanium bearing material produced by the leaching of reduced ilmenite is called synthetic rutile (SR) or beneficiated ilmenite (BI). The quality of beneficiated ilmenite mainly depends on the effectiveness of the leaching process. Different techniques were suggested by many researchers for improving of the leaching process of ilmenite. The improvement in leaching process is achieved by the chemical and physical modification of reduced ilmenite, modification in leaching process parameters, varying the quality and quantity of acids, and addition of other soluble and insoluble ions. Improvement of leaching process by mechanical activation of ilmenite was already reported (Welham and Llewellyn, 1997; Li et al., 2006). The effects of oxidation–reduction treatment and mechanical activation on the hydrochloric acid leaching performance of Panxi ilmenite concentration was reported by Zhang et al., 2011. The presence of metallic iron during the leaching of ilmenite with hydrochloric acid was reported in the literature by Mahmoud et al. (2004). The effect of addition of soluble chlorides of ferrous, manganese, magnesium, nickel, calcium and ammonium on the leaching process of ilmenite with hydrochloric acid and the kinetics of the leaching process with these ions were reported by Reid and Sinha (1975). Dissolution of mechanically activated Panzhihua ilmenite in hydrochloric acid for the preparation of synthetic rutile was investigated by Li et al. (2008). A patent was reported in literature for improving the leaching process of ilmenite by the addition of gypsum ($CaSO_4 \cdot 2H_2O$) and sulphuric acid (Chen and Huntoon, 1977). The present study deals with a detailed investigation including the kinetic and thermodynamic approach on the leaching process of ilmenite using hydrochloric acid with and without

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http://dx.doi.org/10.1016/j.minpro.2014.11.004
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Table 1
Composition of raw ilmenite.

	Compounds	Percentage
1	TiO_2	59.7
2	FeO	9.6
3	Fe_2O_3	24.6
4	SiO_2	1.0
5	Al_2O_3	1.0
6	Cr_2O_3	0.2
7	V_2O_5	0.3

the presence of small amount of gypsum. The leaching parameters such as temperature, concentration of acid, and ilmenite to hydrochloric acid ratio, are varied to find out the optimum leaching process.

2. Materials and methods

2.1. Raw material

The Quilon grade ilmenite (Q Grade) is used as the ore (raw material) for the study. The composition of the ilmenite is given in Table 1. The elemental analysis of the ore is carried out by an atomic absorption spectrometer (AA 100) of Perkin Elmer and X-ray fluorescence analyser (XRF) of Bruker. The Q grade ilmenite contains 59.7% of TiO_2, 24.6% of ferric oxide (Fe_2O_3) and 9.0% of ferrous oxide (FeO) and smaller percentage (less than 1%) of alumina, silica and other metal oxides. All reagents and chemical used in this study are of AR grade quality.

2.2. Conversion of raw ilmenite to reduced ilmenite

The ilmenite is subjected to carbothermic reduction in air tight cylindrical stainless steel Tube of 50 CC capacity at 900 °C for 15 min using petroleum coke as reductant. The ilmenite to petroleum coke was in the ratio of 10:1.

2.3. Leaching process

The leaching of reduced ilmenite was performed in a 500 mL round bottom flask with ground joint at the top. 10 g of ilmenite is taken in the RB flask. 100 mL of hydrochloric acid of different concentrations was added to the system. The RB flask is attached with a water condenser to condense the hydrochloric acid vapour generated in the RB flask at elevated temperatures. A magnet with Teflon coating was introduced into the RB flask for agitation. The RB flask is placed in a rotamantle at different temperatures and different speeds to find out the effective leaching process parameters. Varying amount of catalyst (gypsum) is introduced in to the RB flask and the leaching efficiency is calculated based on the percentage of iron removed from the reduced ilmenite. The iron present in the ilmenite were analysed by using AAS technique.

Table 2
Composition of reduced ilmenite.

	Compounds	Percentage
1	TiO_2	60.6
2	FeO	30.0
3	Fe_2O_3	1.7
4	SiO_2	1.1
5	Al_2O_3	1.1
6	Cr_2O_3	0.2
7	V_2O_5	0.3

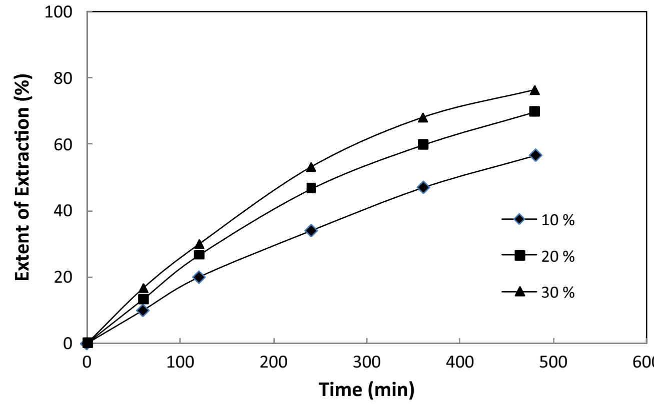

Fig. 1. Extent of extraction of iron from ilmenite by leaching with different concentrations of hydrochloric acid at different durations of leaching.

3. Results and discussion

3.1. Reduction of raw ilmenite

The raw ilmenite is subjected to carbothermic reduction at elevated temperature (900 °C) to convert the ferric state iron to ferrous (Section 2.2.) The composition of reduced ilmenite is given in Table 2. The composition of raw ilmenite is given in Table 1. On reduction of ilmenite, the ferrous content is enhanced to 30% from its initial value of 9.4% and the ferric content is reduced to 1.7% from its initial value of 24.6%. There is no considerable variation in other constituents present in raw ilmenite and reduced ilmenite. The reduced ilmenite is more leachable than raw ilmenite due to the presence of more ferrous content, since the ferrous content is more soluble in hydrochloric acid than ferric content. Moreover, the ferrous state is generated by the reduction of ferric by removing the oxygen from the crystal lattice of ilmenite, generating porous surface enhancing the hydrochloric acid attack.

3.2. Leaching performance

3.2.1. Varying the of acid concentration

Fig. 1 shows the percentage iron oxide removed from the ilmenite (leaching efficiency) by leaching with HCl having varying concentration (10–30%, *v*/*v*) at 50 °C under different leaching duration. As the acid concentration increases, the leaching performance also increases. This is due to increased quantity of acid content for removal of iron in ilmenite. As the time for leaching increases, the amount of iron removed form the ilmenite also increases. The iron leaching efficiency was found to be 77% by using 30% HCl for a leaching duration of 8 h.

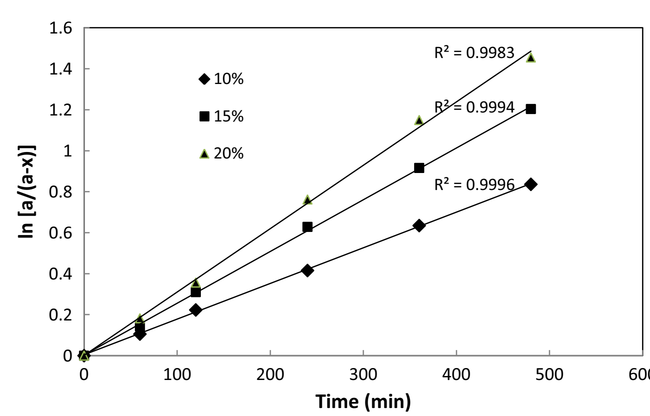

Fig. 2. A plot of ln(a / a − x) against time for the leaching of ilmenite with different concentrations of hydrochloric acid.

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