



Leaching studies of alkali roasted bomarilmenite and anatase during the processing of synthetic rutile



Jeya Kumari Ephraim^{a,*}, Animesh Jha^b

^a School of Engineering and Informatics, University of Bradford, Bradford BD7 1DP, United Kingdom

^b The Institute for Materials Research, Houldsworth Building, Clarendon Road, University of Leeds, Leeds LS2 9JT, UK

ARTICLE INFO

Article history:

Received 6 March 2014

Received in revised form 2 December 2014

Accepted 5 December 2014

Available online 8 December 2014

Keywords:

Sodium oxide

Ilmenite

Anatase

Synthetic rutile

ABSTRACT

The leaching of sodium (Na^+) and Fe^{2+3+} ions from the soda-ash roasted ilmenite (FeO-TiO_2) or anatase minerals has been examined as a chemical method for beneficiating titani-ferous minerals for the production of pigment grade TiO_2 . In the leaching step, the roles of catalytic aeration, dilute concentrations of organic liquids (methanol) in the range of 0.5% (v/v) in 1.5% (w/v) ammonium chloride are explained. The results of leaching experiments are also compared in the presence of inorganic acid with that of ammonium salt and acetic acid for retaining the particle size. The role of pH in the removal of sodium and iron ion is also explained based on E_h -pH diagram. The paper also explains the selective separation of rare earth oxides during the processing of synthetic rutile. The data obtained at each step of processing is explained and interpreted on the basis of results obtained through XRD, SEM and EDX. Rare earth oxides are separated unattacked by the processing steps.

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1. Introduction

Titanium dioxide (TiO_2) is widely used in pigment, paper, and welding industry. It is also the main source of raw material for making titanium metal via the production of titanium tetrachloride and its reduction via liquid magnesium, the process known as the Kroll process (Kroll, 1940). More recently high-purity TiO_2 is also deoxidised by direct reduction to titanium metal (>98% pure) using calcium metal as reductant (Ephraim and Patel, 2013). In all the applications of TiO_2 , the control of impurities is essential. For example, the rare-earth and transition metal impurities in white pigment grade TiO_2 reduce the whiteness by creating colour centres, whilst their presence in titanium metal result in the embrittlement and reduced shape forming.

Ilmenite ($\text{FeO} \cdot \text{TiO}_2$) contains 40–65% TiO_2 depending on its geological environment (Barksdale, 1966). Most of the commercial processes involve a combination of thermal oxidation and reduction by roasting, leaching and physical separation steps. Iron oxide in the ore is dissolved in acid or reduced at high temperature followed by acid leaching to produce synthetic rutile (Zhang et al., 2011). The commercial technologies for the manufacture of pigment grade titanium dioxide are the sulphate process and chloride process (Langmesser et al., 1973; Chernet, 1999; Sasikumar et al., 2004; Jabłoński and Przepiera, 2001). In sulphate process ilmenite is treated with concentrated sulphuric acid to form titanylesulphate which is then hydrolysed and precipitated to form titanium dioxide (TiO_2) pigment. In chloride process rutile or synthetic rutile is chlorinated in a fluidized bed reactor (FBR) to form titanium tetra

chloride which is then converted to TiO_2 pigment. Both these processes develop huge amounts of hazardous waste leading to pollution problems (Zhang et al., 2009).

In spite of the above problems there is always an increasing demand for oxide and metal products of titanium which has forced the industry to explore lesser graded ores which have much higher concentrations of lanthanides and actinides (500–30,000 ppm total), calcium and aluminium (at few weight %). Consequently the lower-grade ores are difficult to beneficiate via the conventional acid leaching (Gambogi, 2009, 2010) and electric-arc smelting processes (Mukherjee et al., 2002). Soda-ash roasting of titani-ferous (e.g. ilmenite) minerals (Jeya Kumari et al., 2006) provides a much better route for selective separation of not only iron as a major element, but also the minor and trace elements.

In this paper we examine the physical chemistry of leaching reaction of bomar ilmenite and anatase in particular the removal of iron and sodium from the iron-sodium titanate and sodium titanate phases, which is formed as a result of the roasting reaction between soda and ilmenite mineral at high-temperatures. These two phases appear to be insoluble in water; by contrast sodium ferrite is soluble in water. The room-temperature aeration leaching reaction was investigated under the controlled pH and oxygen partial pressure, and the results are compared with the leaching condition when only a mineral acid was used in air. The control of pH was achieved via the addition of organic solvent methanol, acetic acid and a small fraction of ammonium chloride (Becher et al., 1965). Their roles in the generation of protons during leaching are discussed in the context of a computed E_h -pH diagram.

The ion diffusion pathways in the controlled pH condition play an important role in determining the final particle size range of original

* Corresponding author.

E-mail address: E.J.Kumari@bradford.ac.uk (J.K. Ephraim).

Table 1
Chemical composition of roasted bomarilmenite with soda-ash at 900 °C for 4 h.

Constituents	Roasted ilmenite (%)	Roasted anatase (%)
TiO ₂	48.3	45.2
Fe ₂ O ₃	18.69	12.32
Na ₂ O	41.8	43.1
Al ₂ O ₃	1.36	3.25
Cr ₂ O ₃	0.13	0.08
Mn ₃ O ₄	0.17	0.33
CaO	0.01	1.35
SiO ₂	1.52	1.38
P ₂ O ₅	0.2	3.62
ThO ₂	0.24	0.07
CeO ₂	0.97	0.82
Nb ₂ O ₅	0.63	0.76
ZrO ₂	2.41	1.13

ilmenite, which is essential for subsequent selective separation of transition and rare-earth elements. The new physical chemistry of leaching offers an excellent route for the production of high-grades of synthetic rutile for chlorination.

2. Experimental materials and methods

Bomar ilmenite or anatase was roasted with soda-ash and alumina (Jha et al., 2008) at around 900 °C, washed and dried prior to leaching studies. The chemical composition of the raw material used for leaching is given in Table 1. Laboratory grade and GR grade chemical reagents were used for the leaching experiments and chemical analysis, respectively. X ray diffraction studies were carried out for the bomar ilmenite and anatase that both show the presence of phases like TiO₂, Fe₂O₃, pseudorutile, P₂O₅, and Al₂O₃. After roasting the phases present were

sodium titanate as Na₂TiO₃, Na₄TiO₄ and sodium aluminate NaAlO₂ shown in Fig. 1. During roasting the sodium oxide attacks the surface of the grain and creates pores on the surface of ore for the migration of sodium oxide ion into the core of the grain to react with iron and titanium to form sodium ferrite and sodium titanate respectively. When once the reaction has taken place the ilmenite crystal lattice is altered for further movement of Na⁺ ions into the lattice. EDX confirms the attack of sodium ions in to the core of the ilmenite lattice which is shown in Fig. 2.

The ilmenite roasted with soda-ash and alumina at 900 °C was suspended in 2 M HCl acid and a magnetic stirrer was used to stir the reaction mixture. The magnetic stirrer whilst stirring, powdered the ilmenite grain. Therefore after the reaction it was found very difficult to separate the iron oxide and the beneficiated powder. Hence a new leaching vessel was designed to carry out leaching.

As the leaching in HCl medium was found to be highly unsatisfactory a new fabricated leaching set-up was designed in which about 30 g of roasted ilmenite was suspended in a plastic beaker containing, 120 ml of 1.5% NH₄Cl weight/weight (w/w) ratio and 0.5% volume/volume (v/v) methanol. The pH of the aqueous medium was adjusted to 4 with the addition of acetic acid. During leaching, a continuous flow of air was maintained at the rate of 4–5 l min⁻¹, whilst the mixture was stirred at 1500 rotation per minute (rpm). The leaching was carried out for 5 h, after which the leached grains of roasted minerals were separated from iron oxide by repeated washing with de-ionised water. We define this leaching as the “aeration leaching” throughout the text. For detailed phase, chemical and microstructure analyses, the partially leached minerals were sampled every hour, and were washed thoroughly with water and analysed for total iron, sodium oxide and TiO₂ after drying in an air oven at 100 °C. The chemical analysis for iron, sodium, minor and trace impurities and TiO₂ in bomar ilmenite and

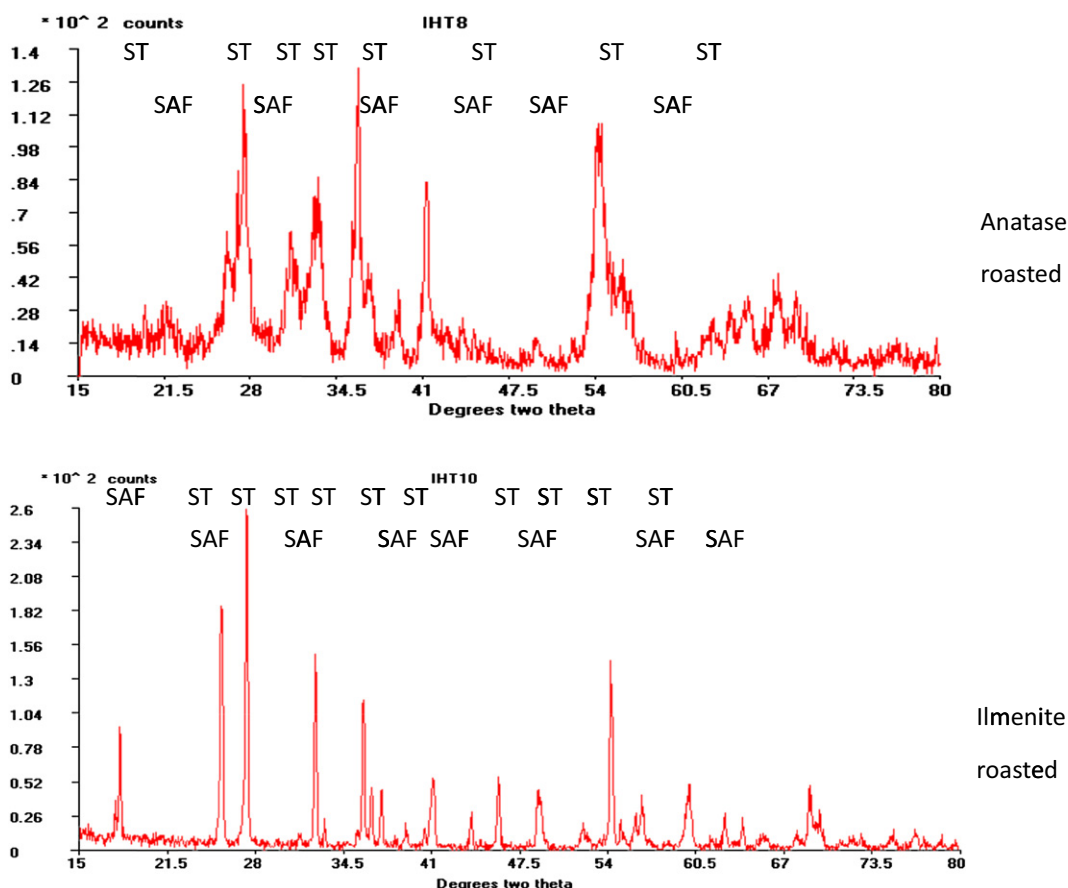


Fig. 1. XRD of ilmenite and anatase roasted with soda-ash + alumina at 900 °C for 4 h. ST – sodium titanate, SAF – sodium aluminium ferrite.

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