MICROWAVE ASSISTED DISSOLUTION OF SRI LANKAN ILMENITE: EXTRACTION AND LEACHING KINETICS OF TITANIUM AND IRON METALS

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ABSTRACT

The kinetics of the leaching of titanium and iron from Sri Lankan ilmenite in a sulphuric acid medium heated by microwave energy is studied. The effects of the concentration of sulphuric acid, reaction temperature and particle size on the kinetics of dissolution of ilmenite are investigated. Results show that both the acid concentration and the temperature have significant effects on the rate of dissolutions of titanium and iron from ilmenite. The dissolution of both titanium and iron from ilmenite was controlled by a chemical reaction on the surface of ilmenite particles. Maximum extractions of 74.2 (±1.1) % of titanium and 71.3 (±0.8) % of iron from the ilmenite ore were achieved using a microwave leaching system of 600 W, 2.45 GHz. Experimental results further showed that the apparent activation energies for the dissolution of titanium and iron were 137 kJ mol⁻¹ and 117.1 kJ mol⁻¹ respectively. Therefore, microwave energy has a potential for the speedy and efficient leaching of ilmenite and in a commercial context it can be used as a more economical and environmental friendly process. The results of the mineralogical and chemical analysis studies for the ilmenite ore indicated that both ilmenite (FeTiO₃) and pseudorutile (Fe₂Ti₃O₉) minerals were present with a grade of 37.9% titanium.

Keywords: Microwave energy, Ilmenite, Titanium, Leaching kinetics

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INTRODUCTION

Ilmenite (FeTiO₃) is the most abundant principal titanium bearing mineral which is used as the main feedstock for the production of titanium metal and titanium dioxide (TiO₂) pigment (Zhang and Nicol, 2010). More than 90% of the titanium ore is processed into TiO₂ pigment globally (Jayawardena, 1998). Sri Lanka has vast deposits of ilmenite. The major deposits are on the north-east coast at Pulmoddai in Sri Lanka, which have been exploited economically by Lanka Mineral Sands Ltd since 1958 (Meyer, 1983; Jayawardena, 1998). However, Sri Lanka does not produce any type of value added TiO₂ pigments. There are two main methods of manufacturing TiO₂ pigment, the traditional Sulphate process and the more modern Chloride process (Carp *et al.*, 2004). In the Sulphate process, ilmenite (with 45-65% TiO₂) is dissolved in 85% (w/w) sulphuric acid at 160 °C to form titanyl sulphate (TiOSO₄) and ferrous sulphate (FeSO₄) as shown in Eq. (1).

$$FeTiO_{3(s)} + 2H_2SO_{4(aq)} \rightarrow TiOSO_{4(aq)} + FeSO_{4(aq)} + 2H_2O_{(1)}$$
 (1)

Titania is precipitated form titanyl sulphate by hydrolysis, and is then filtered, washed, and calcined to produce TiO₂ pigment (Chernet, 1999; Han, et al., 1987). Only Sulphate process can produce both rutile and anatase crystal type TiO₂ pigments (Carp et al., 2004). The dissolution of ilmenite ore in high concentrated sulphuric acid at high temperature is one of the most energy consumption steps (Carp et al., 2004; Chernet, 1999). Since it was discovered that ilmenite can be dissolved with concentrated sulphuric acid as the first step in the Sulphate process for producing TiO₂ pigment, numerous investigations have been carried out to improve the process (Han et al., 1987). Some studies (Chernet, 1999) suggested that the solubility of ilmenite in acid solutions varied in accordance with the degree of chemical and mineralogical alteration of the ilmenite. The leaching kinetics of some other ilmenite ores in dilute and highly concentrated hydrochloric acid solutions have been investigated and it was found that dissolution rates followed the shrinking core models for spherical particles (Jacobus et al., 2002; Olanipekun, 1999). Kelly and Rowson (1995) showed that extractions of titanium and iron from Norwegian and Australian ilmenite ores in hydrochloric acid were significantly improved by the partial reduction of ores using microwave heating prior to acid leaching.

The potential application of microwave energy in mineral processing and in extractive metallurgy has been investigated (Haque, 1999). Microwave heating is unique and offers a number of advantages over conventional heating. These include low

processing time, direct heating, selective and volumetric heating. Besides, it is a more controllable heating process (Al-Harahsheh *et al.*, 2006). Microwave assisted leaching has been investigated in an attempt to improve the yield of extracted copper metal from chalcopyrite and to reduce process time as a more economical and environmental friendly process (Ai-Harahsheh and Kingman, 2004). The heating characteristics and dissolution behaviour of minerals with acids in a microwave field are primarily determined by their dielectric properties and mineralogy (Haque, 1999). The usage of microwave radiation to augment the leaching of valuable metals from ores is still at an experimental stage.

The application of microwave heating in the Sulphate process for the production of TiO₂ pigment from ilmenite has not been reported. Moreover, it is apparent from previous work that there are no detailed investigations of the dissolution behavior and mechanism of Sri Lankan ilmenite in sulphuric acid for the production of TiO₂ pigment. Therefore, this research work presents a study of the leaching kinetics and the dissolution behaviour of titanium and iron from Sri Lankan ilmenite in sulphuric acid medium that was heated by microwave volumetric heating. This investigation is also intended to study the mineralogical and chemical characteristics of Sri Lankan ilmenite.

MATERIALS AND METHODS

Mineralogical and chemical analysis

The ilmenite mineral sample, which was supplied by Lanka Mineral Sand Ltd, was used for this investigation. This ilmenite concentrate was separated from Pulmoddai beach sand deposit in Sri Lanka. Particle size analysis was carried out on the ilmenite concentrate by the dry sieving method. X-ray diffraction (XRD) analysis, employing a Philips Analytical X-ray machine, was used to ascertain mineral phases of the ilmenite sample. Particle shapes of ilmenite were examined by a Jeol, JSM-5200 scanning electron microscope using an accelerating voltage of 20 kV. For chemical analysis, ilmenite samples were decomposed by fusion with KHSO₄ at 800 °C and the melt was dissolved in 20% H₂SO₄ acid (Jeffery and Hutchison, 1981). Titanium analysis was carried out in a UV/Visible spectrophotometer based on the yellow complex formed by titanium and H₂O₂ (Mendham *et al.*, 2000). Total iron was analyzed using an atomic absorption spectrophotometer. The relative errors of analysis of titanium and iron were ±3.5% and ±2.0% respectively.

Microwave assisted dissolution experiments

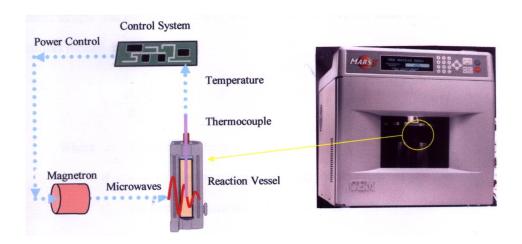


Figure 1: Microwave accelerated reaction system

In each microwave assisted leaching test run, approximately 0.2 g of ilmenite (FeTiO₃) was used. The ilmenite sample was added into 25 mL of sulphuric acid (H₂SO₄) solution of various concentrations in a chemically resistant Teflon vessel which is transparent to microwave so that the suspension can absorb the maximum amount of incident microwave energy. Leaching experiments were carried out in a 600 W, 2.45 GHz microwave field utilizing the MARS 5 microwave accelerated reaction system illustrated in Figure 1.

In the microwave field, the solution sample was heated to a designated temperature rapidly and the temperature inside the reaction vessel was automatically monitored regulating the microwave power output according to a feedback control signal. The sample was leached with microwave energy for a set time. The sample solution was magnetically stirred during the microwave leaching process. After leaching, the sample was filtered and titanium and iron in the leach solutions were analyzed. Each experiment was carried in duplicates. When ilmenite was leached with sulphuric acid by microwave radiation, the reacted fractions of titanium and iron were calculated by Eq. (2).

$$X = \frac{C.V}{W} \tag{2}$$

Where, X is fraction reacted, W is total analyte in the completely decomposed sample (µg), C is the analyte concentration in leaching solution (µg/mL) and V is the volume of leaching solution (mL).

RESULTS AND DISCUSSION

Mineralogical and chemical analysis

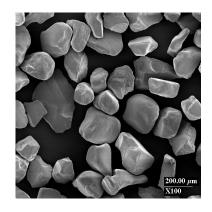
The results of titanium and iron content in different size fractions of the ilmenite concentrate are presented in Table 1. The titanium and iron content of the ilmenite concentrate were analyzed and it was found that the titanium and iron assays were 37.9% (63.3% TiO₂) and 24.7% respectively. According to the particle size analysis data, a maximum of 51.0% by weight of the ilmenite concentrate was found in the particle size range of 90-125 microns. In this particle size fraction, titanium and iron assays were found to be 38.4% and 23.9% respectively. The highest titanium assay was detected as 39.3% in the particles size fraction of 125-180 µm.

Table 1: Titanium and iron content in different size fractions of the ilmenite sample

Particle	Weight	Ti	Ti	Fe	Fe
size	(%)	grade	distribution	grade	distribution
fraction		(%)	(%)	(%)	(%)
(µm)					
-355+250	1.3	24.87	0.85	24.72	1.30
-250+180	6.1	34.22	5.50	23.65	5.85
-180+125	20.4	39.28	21.13	23.60	19.52
-125+90	51.0	38.42	51.66	23.88	49.38
-90+63	19.1	37.46	18.86	27.87	21.58
-63+45	2.0	36.33	1.92	27.79	2.25
-45	0.1	29.35	0.08	25.27	0.10
Total	100.0	37.93	100.00	24.66	100.00

Figure 2 shows the scanning electron microscopy (SEM) micrograph of a representative sample of the Sri Lankan ilmenite concentrate. The ilmenite concentrate, having been derived from a beach sand deposit, comprises of some slightly rounded particles. This could be due to the combination of wave action and current to separate heavy minerals of beach sand deposits. An X-ray diffraction analysis of ilmenite concentrate (Figure 3) indicated that it contained only ilmenite (FeTiO₃) and pseudorutile (Fe₂Ti₃O₉) minerals. Therefore, the Sri Lankan ilmenite concentrate comprised particles,

which had been naturally eroded from a primary deposit and subjected to weathering processes. The extent of weathering was indicated by the presence of pseudorutile alteration phase.



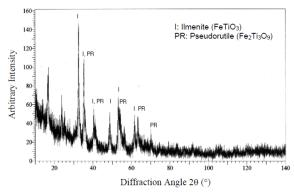


Figure 2: SEM image of ilmenite

Figure 3: XRD pattern of ilmenite

Microwave assisted dissolution of ilmenite

The effect of H₂SO₄ concentration on dissolution rate

The effect of sulphuric acid (H_2SO_4) concentration on the rate of extraction of titanium and iron was investigated at 383 K (110 °C) using the 63-90 µm size fraction of ilmenite. The H_2SO_4 concentrations used were 6.2, 8.2, 10.2, 12.2 and 14.0 M. Figures 4(a) and 4(b) present the effect of H_2SO_4 concentration on the dissolution of titanium and iron in a 600 W, 2.45 GHz microwave field respectively.

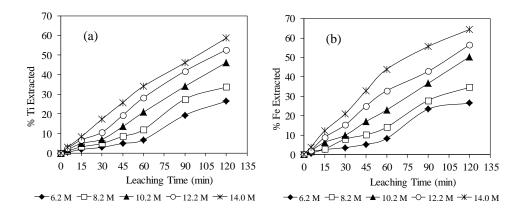


Figure 4: The effect of H_2SO_4 concentration on the leaching of (a) titanium and (b) iron from ilmenite as a function of time (Temperature: 383 K, Particle Size: 63-90 μ m)

The experimental results showed that the dissolution of titanium and iron from ilmenite increased with the leaching time. The concentration of H_2SO_4 had a substantial influence on the dissolution of both titanium and iron from ilmenite. At a low H_2SO_4 concentration, increasing the leaching time did not have a significant impact on the dissolution of titanium and iron.

However, the rate of dissolution of titanium and iron increased dramatically with increasing acid concentration from 6.2 to 14.0 M. For example, only $6.7(\pm0.1)\%$ of titanium and $8.2(\pm0.1)\%$ of iron were leached in 6.2 M H_2SO_4 acid medium while $34.1(\pm0.2)\%$ of titanium and $43.9(\pm0.1)\%$ of iron recoveries were observed in 14 M H_2SO_4 acid medium after 60 minutes of leaching in both cases. The maximum extractions of $58.7(\pm0.6)\%$ of titanium and $64.4(\pm1.7)\%$ of iron from ilmenite were achieved in 14 M H_2SO_4 acid medium after 120 minutes.

The effect of temperature on dissolution rate

The effect of temperature on the dissolution of titanium and iron was studied in 12.2 M H₂SO₄ acid solution using the 63-90 µm size fraction of ilmenite concentrate. Reaction temperatures used were 353, 363, 373, 383 and 393 K. Experimental results for the extraction of titanium and iron at various temperatures using microwave radiation are presented in Figures 5(a) and 5(b) respectively.

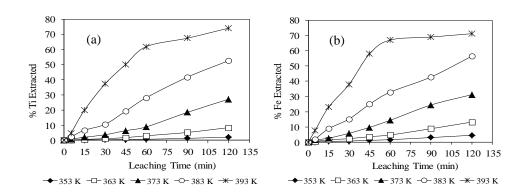


Figure 5: The effect of temperature on the leaching of (a) titanium and (b) iron from ilmenite as a function of time ([H₂SO₄]: 12.2 M, Particle Size: 63-90 μm)

As it can be observed, dissolution rates of titanium and iron from ilmenite in a sulphuric acid medium are extremely sensitive to reaction temperatures. At relatively low

temperatures such as 353 K (80 °C) and 363 K (90 °C), increasing the leaching time had a slight impact on the dissolution of both titanium and iron from ilmenite.

For example $0.7(\pm0.1)\%$ of titanium and $1.6(\pm0.1)\%$ of iron were extracted after 60 minutes of dissolution at 353 K (80 °C) temperature, while $62.0(\pm0.8)\%$ of titanium and $67.1(\pm0.3)\%$ of iron were leached at 393 K (120 °C) after the same time period. However, no significant increases in titanium and iron extractions were found with increasing the leaching time after 60 minutes at 393 K temperature. It was observed that both titanium and iron were largely dissolved at 393 K and the maximum fraction of titanium and iron extracted at this temperature were $74.2(\pm1.1)\%$ and $71.3(\pm0.8)\%$ respectively after 120 minutes.

The effect of particle size on dissolution rate

The effect of particle size on the dissolution of titanium and iron from ilmenite ore was also investigated in 12.2 M H_2SO_4 acid solution at 383 K (110 °C) using five different particle size fractions in a 600 W, 2.45 GHz microwave field. Particle size fractions used were 45-63, 63-90, 90-125, 125-180 and 180-250 μ m. The experimental results showed that the dissolution rates of titanium and iron were inversely proportional to the average initial diameter of the particles. For example, only 27.1(±0.4)% of titanium and 33.3(±0.8)% of iron were leached with 180-250 μ m size particles while 54.4(±0.7)% of titanium and 71.2(±1.6)% of iron recoveries were observed with 45-63 μ m size particles after 120 minutes of microwave leaching in both cases.

Study of reaction kinetics

Leaching reactions are characterised in two ways: firstly by thermodynamics, which is concerned with the direction and extent of chemical change, and secondly by kinetics, which considers the rate at which a chemical reaction takes place. It is considered that kinetics provide a more detailed description of the system involved (Burkin, 2001). The dissolution of ilmenite minerals in a sulphuric acid system is a heterogeneous reaction, which has an interface between the solution and solid particle. The dissolution reaction of ilmenite in sulphuric acid is expressed in Eq. (1) chemical reaction. Eq. (1) assumes that there is no solid product formed during the leaching reaction.

Normally, the kinetic models of the dissolution reaction of ilmenite in an acid medium can be classified into well-known shrinking core models for surface reaction control, diffusion control through product layers and mixed control by surface reaction and diffusion control. In the case of homogeneous spherical particles, these models can be expressed as follows (Burkin, 2001; Huang and Rowson, 2002).

For surface reaction control:

$$1 - (1 - X)^{1/3} = K_s t \tag{3}$$

For product layer diffusion control:

$$1 - 2X/3 - (1 - X)^{2/3} = K_D t \tag{4}$$

For mixed control:

$$1 - (1 - X)^{1/3} + y/6[(1 - X)^{1/3} + 1 - 2(1 - X)^{2/3}] = K_M t$$
 (5)

Where, X is the fraction reacted at time t, and K_S , K_D and K_M are apparent rate constants. These constants are given in the following equations:

$$K_S = bkM[H_2SO_4]^n / \rho r_0 \tag{6}$$

$$K_D = 2bD_e M[H_2 SO_4] / \rho r_0^2 \tag{7}$$

$$K_{M} = bkM[H_{2}SO]/\rho r_{0}$$
(8)

$$y = kr_0 / D_a \tag{9}$$

Where, b is the stoichiometric coefficient, M is the molecular weight of reacted particle (kg mol⁻¹), ρ is the density of reacted particle (kg m⁻³), r_0 is the initial particle radius (m), k is intrinsic rate constant, D_e is the effective diffusivity (m² min⁻¹), $[H_2SO_4]$ is the bulk concentration of H_2SO_4 (mol m⁻³), n is the reaction order in the term of H_2SO_4 concentration.

The data given in Figures 5(a) and 5(b), which present the effect of temperature on the dissolution of titanium and iron form ilmenite ore in a $12.2 \text{ M H}_2\text{SO}_4$ acid medium, were analyzed with the above shrinking core models. It was found that all of the results fitted with the surface reaction control model giving perfectly straight lines (from 0 to 60 minutes) and failed to agree with the product layer diffusion control and the mixed control models. The analyzed data related to surface chemical reaction control process are presented in Figures 6(a) and 6(b).

Figures 6(a) and 6(b) show that the dissolution rates of titanium and iron at 353 to 393 K (80 to 120 °C) give a good correlation with the surface reaction kinetic according to

Eq. (3). This suggests that microwave assisted dissolution of titanium and iron from ilmenite in a sulphuric acid system is controlled by the chemical reaction process on the particle surface.

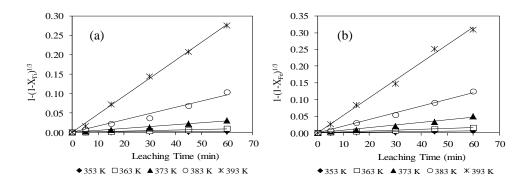


Figure 6: Plots of 1- $(1-X)^{1/3}$ vs. time for (a) Ti leaching and (b) Fe leaching from ilmenite at various temperature

Estimation of activation energy

Activation energy is calculated by the Arrhenius equation given in Eq. (10). It is characteristic of a reaction and determines the influence of temperature on the reaction (Burkin, 2001).

$$K = A.\exp(-E_a/RT) \tag{10}$$

Where, K is the reaction rate constant, A is the frequency factor (min⁻¹), E_a is the activation energy (J mol⁻¹), R is the universal gas constant (8.314 J K⁻¹ mol⁻¹), T is the reaction temperature (K).

For example $0.7(\pm0.1)\%$ of titanium and $1.6(\pm0.1)\%$ of iron were extracted after 60 minutes of dissolution at 353 K (80 °C) temperature, while $62.0(\pm0.8)\%$ of titanium and $67.1(\pm0.3)\%$ of iron were leached at 393 K (120 °C) after the same time period. However, no significant increases in titanium and iron extractions were found with increasing the leaching time after 60 minutes at 393 K temperature. It was observed that both titanium and iron were largely dissolved at 393 K and the maximum fraction of titanium and iron extracted at this temperature were $74.2(\pm1.1)\%$ and $71.3(\pm0.8)\%$ respectively after 120 minutes.

Rate constants for different temperatures were calculated from plots in Figures 6(a) and 6(b) taking the slope of the plots for titanium and iron respectively. Arrhenius plots of

 lnK_S versus 1/T were created for rate constant (K_S) values obtained for titanium and iron dissolution respectively and they are shown in Figure 7. The activation energies for the dissolution of titanium and iron from ilmenite ore were determined as 137 kJ mol⁻¹ and 117.1 kJ mol⁻¹ with a correlation coefficient of 0.9997, for the temperature range 353-393 K (80-120 °C).

These magnitudes of activation energy for titanium and iron strongly support the theory that the leaching of titanium and iron from ilmenite ore is controlled by the chemical reaction on the particle surfaces because leaching reaction controlled by chemical reaction process usually has a high activation energy (Huang and Rowson, 2002; Olanipekun, 1999).

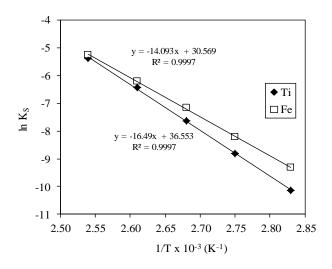


Figure 7: Arrhenius plots for Ti and Fe disxsolution in 12.2 M H₂SO₄ solution

The most common problem associated with hydrometallurgical operations is the low recovery of extracted metals from their ores (Al-Harahsheh *et al.*, 2006). The extent of weathering of Sri Lankan ilmenite was indicated by the presence of pseudorutile (Fe₂Ti₃O₉) with a grade of 63.3% TiO₂. The dissolution of weathered ilmenite always shows poor solubility in acids compared to pure ilmenite which contains theoretically 52.7% TiO₂ (Chernet, 1999). However, the experimental results showed that the high dissolution rates of both titanium and iron from ilmenite ore were achieved using the microwave energy. An extraction of 74.2% of titanium was achieved after 2 hours leaching of Sri Lankan ilmenite (63.3% TiO₂) in 12.2 M H₂SO₄ solution at 393 K (120 °C) using microwave radiation. This leachate which has more than 74% of titanium as titanyl sulphate (TiOSO₄) is a promising precursor for the production of TiO₂ pigment according

to the Sulphate process (Carp *et al.*, 2004). Therefore, the present investigation showed that the application of microwave heating in the Sulphate process for the production of value added TiO₂ pigment from Sri Lankan ilmenite can be developed as a promising method.

The dissolution behaviour of ilmenite with sulphric acid to microwave heating totally depends on its dielectric properties and mineralogy. Microwaves obey the law of optics and can be transmitted, absorbed or reflected. Their behaviour depends on the types of materials with which they are interacting. Ilmenite (FeTiO₃) which is an excellent absorber of microwave energy is easily heated and is classed as a dielectric material (Haque, 1999). In a microwave field, microwaves will penetrate into the solution to lead to efficient volumetric heating. Therefore, microwaves should have an effect on the localized reactions between solid mineral, which may preferentially absorb microwave energy, and solvent. Local overheating taking place on individual particles may improve reaction kinetics and result in the rapture of the particles to produce refresh surface (Al-Harahsheh et al., 2006). The differential dielectric constants between the liquid and the solid may also result in a localized temperature difference on the surface of solid, which creates strong convection currents on the surfaces of the microwaved solid. The local convection can promote the diffusion of the reaction products into solution and also sweep away the stagnant product layers, which provide fresh surfaces to contact with solution (Huang and Rowson, 2002). With regard to the dissolution of titanium and iron from ilmenite in an acid solution, the difference in the local temperature and the local convection on the particle surface would be of advantage to increase the diffusion of the reaction products formed into the solution. This also causes a better contact between the particles and the acid solution giving higher dissolution rates.

CONCLUSIONS

Microwave assisted dissolution kinetics of Sri Lankan ilmenite in sulphuric acid medium have been investigated. Experimental results showed that the high dissolution rates of both titanium and iron from ilmenite in sulphuric acid were achieved using a microwave leaching system. The reaction temperature and the concentration of the sulphuric acid had a significant influence on the dissolution of titanium and iron. The dissolution rate increased with an increase in reaction temperature and the concentration of sulphuric acid and with a decrease in particle size.

Experimental data characterising the rates of dissolution of titanium and iron have been analysed with the shrinking core model for spherical particles for chemical reaction control process. The kinetics study showed that the dissolution of both titanium and iron from ilmenite was controlled by a chemical reaction process on the surface of particles. Experimental results indicated that the apparent activation energies for the dissolution of titanium and iron were 137 kJ mol⁻¹ and 117.1 kJ mol⁻¹ with a correlation coefficient of 0.9997. Maximum extractions of 74.2(±1.1)% of titanium and 71.3(±0.8)% of iron from Sri Lankan ilmenite were achieved after 2 hours using 12.2 M H₂SO₄ solution at 120 °C temperature. This leachate which has more than 74% of titanium as titanyl sulphate is a promising precursor for the production of TiO₂ pigment according to the Sulphate process. The results of mineralogical and chemical analysis studies for the ilmenite ore revealed that both ilmenite (FeTiO₃) and pseudorutile (Fe₂Ti₃O₉) minerals were present with a grade of 63.3% TiO₂. The extent of weathering of ilmenite was indicated by the presence of pseudorutile that always shows poor solubility in acids compared to pure ilmenite. The present studies showed that the application of microwave heating in the Sulphate process for the production of value added TiO₂ pigment from Sri Lankan ilmenite can be developed as a promising method. Furthermore, the application of microwaves coupled with existing technology could offer a new method for the hydrometallurgical processing of ilmenite ore.

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