

Kinetics of acid leaching of ilmenite decomposed by KOH Part 2. Leaching by H₂SO₄ and C₂H₂O₄

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ABSTRACT

A kinetic study of the leaching of ilmenite paste (resulting from ilmenite treatment by KOH) by sulfuric and oxalic acids has been investigated. The effects of the reaction agitation speed, ilmenite paste particle size, acid concentration, acid/paste mass ratio and temperature on titanium recovery percent from the KOH decomposed ilmenite are reported. The leaching rates are significantly influenced by the reaction temperature and acid concentration. The observed effects of the relevant operating variables on the leaching rates are consistent with a kinetic model for chemical control. The apparent activation energy for the leaching of titanium has been calculated using the Arrhenius expression. The data obtained are compared and discussed with available reported results. Further a flow diagram for production of TiO₂ based on KOH decomposition of ilmenite paste–oxalic acid leaching is given.

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1. Introduction

Titanium is relatively abundant in the earth's crust, which is usually found in igneous and metamorphic rocks as ilmenite (FeO·TiO₂), rutile (TiO₂) and titanomagnetite (Fe₂TiO₄–Fe₂O₄). A survey on the use of titanium in its various forms indicate that almost 95% of its use is for the production of white colored TiO₂ pigment which has extensive application in paint, plastic and paper industries [1]. With the progress of the titanium industry a gradual depletion of known reserves of natural rutile is taking place. This has led to efforts to convert still abundant ilmenite to what is known as synthetic rutile. The conversion of ilmenite to synthetic rutile involves the reduction of iron oxides present in natural ilmenite to metallic iron which is subsequently removed by leaching in acids [2]. Jablonski and Przepiera [3] studied the kinetics of reaction of Norwegian ilmenite with sulfuric acid of concentration 14.2 M at an initial acid:ilmenite ratio of 4:1 in a non-isothermal and non-adiabatic calorimeter. Their reaction conditions were much more close to commercial operations. As a result of vigorous reaction, the temperature of the reactant mixture rose from 80 °C to about 175 °C. The kinetics of leaching Panzhihua ilmenite in 15.4 M H₂SO₄ with an initial acid:ilmenite ratio of 500 mL: 15 g was described with a shrinking core model and the activation energy was estimated to be 72.6 kJ/mol [4]. Several investigators have studied the dissolution behavior of ilmenite in sulfuric acid [4–8]. Their studies dealt

with the effect of stirring speed, particle size, temperature, acid concentration, and acid:ilmenite ratio on the kinetics of dissolution of ilmenite in hydrochloric and sulfuric acids. Studies on the leaching behavior of ilmenite with sulfuric acid showed that the chemical reaction at the particle surface was the rate-limiting step and a value of 64 kJ/mol was estimated for the activation energy [5].

Barton and McConnel [9] dissolved ilmenite with 4.7–12.5 M sulfuric acid solutions at a rather lower temperature of 65–85 °C. They suggested that the reaction was in kinetic controlling regime, and the activation energy was estimated to be 90 kJ/mol. Xu and Huang [10] tested Panzhihua ilmenite with an acid:ilmenite ratio of 400:3 at 95–118 °C. Activation energy of 44.7 kJ/mol was obtained. These reports focused on low temperature, low acid concentration and low pulp density which are far from the industrial scale operation.

In Egypt, ilmenite occurs in two main deposits; namely, a massive-type interlayer with gabbroic rock at Abu Ghalaga region in Eastern Desert and a placer type included in the black sand deposits at Mediterranean beach-especially at Rosetta and Damietta [11]. The ilmenite concentrate of both types is indeed mineralogically different. According to Hussein et al. [12] and Said [13], Abu Ghalaga is a massive ore composed of ilmenite, magnetite, hematite, rutile, goethite and anatase representing about 70% of the ore, while the balance is mainly silica. The Rosetta ilmenite concentrate is more complicated due to the presence of titanomagnetite.

In part one of this work, Nayl and Aly [14], studied the kinetics of leaching of ilmenite paste produced from KOH treatment by HCl. In this part the investigations are extended to study the leaching kinetics of the ilmenite KOH paste by sulfuric and oxalic acid. Leaching mechanisms for both systems are proposed and discussed. Further,

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Table 1
Chemical composition of the ilmenite ore and decomposed ilmenite KOH paste^a.

Compound	wt.%		Compound	wt.%	
	Ore	Paste		Ore	Paste
TiO ₂	46.38	45.1	V ₂ O ₅	0.18	0.17
FeO	26.21	25.22	Cr ₂ O ₃	0.38	0.37
Fe ₂ O ₃	21.20	20.1	MgO	1.0	0.99
SiO ₂	3.57	3.57	Al ₂ O ₃	0.8	0.8
P ₂ O ₅	0.18	0.18	Rare earths	0.1	0.1

^a Decomposed ilmenite with 70% KOH and L/S=5/1 at 100 °C for 3 h.

inter-comparison between the different leaching reagents used for recovery of titanium from the KOH treated ilmenite is given.

2. Experimental

2.1. Material

The ilmenite used in the present study is a natural placer mineral obtained from Abu Ghalaga region in the Eastern Desert in Egypt. The bulk chemical composition of the ilmenite sample and ilmenite paste used in this study is determined with an energy dispersive X-ray fluorescence (EDX) spectrometer (OXFORD) attached with SEM Model JEOL-JSM-5400, and given in Table 1. Before use the ilmenite sample was milled and sieved and different fractions of mesh size were used in all investigations. All other reagents used for decomposition, leaching and chemical analysis were of analytical grade and used without any purification.

2.2. Decomposition with KOH

Concentrated potassium hydroxide solution (70%) reacts with ilmenite mainly (FeTiO₃) to form potassium titanate (K₄Ti₃O₈) and iron oxide (FeO) and this can be described as follows [15]:



Based on this equation, ilmenite of different particle sizes were decomposed by KOH. The decomposition conditions unless otherwise stated, were generally fixed at 100 g ilmenite, 70% KOH solution at 100 °C, particle size of 105–74 μm alkali:ilmenite ratio mass ratio of 5:1 with stirring speed 375 rpm and reaction period of 3 h. The hydrothermal KOH attack produced a paste contains mainly K₄Ti₃O₈ and FeO as indicated by XRD (Fig. 1).

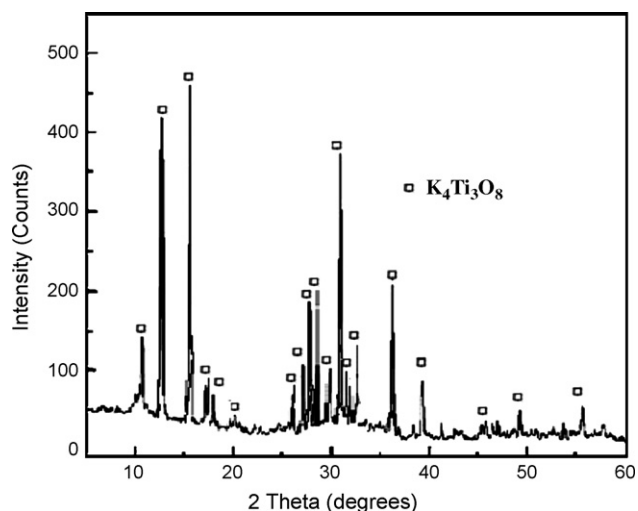


Fig. 1. XRD patterns of ilmenite paste (K₄Ti₃O₈).

From the results obtained, Table 1, the amounts of iron, titanium, vanadium and chromium released in potassium hydroxide solution are rather small and represent not more than about 1% of iron, 2.6% of chromium, 4.8% of vanadium and 1.1% of titanium. The hydroxide paste obtained contains the main bulk of iron and titanium. At the end of each decomposition experiment, the slurry was filtered, washed with distilled water and the residue paste was taken for acid leaching investigations.

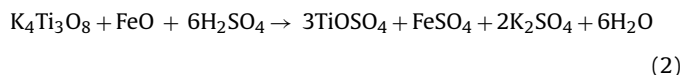
2.3. Acid leaching procedure

All the experiments were conducted in batch mode. In this respect, a known amount of the ilmenite KOH paste formed was taken with a known amount of the leaching acid (sulfuric or oxalic acid) in 500 mL conical flask to investigate the different parameters affecting the titanium leaching. The concentration of titanium in the leach solution was analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES) using the system ULTIMA-2 ICP, Jobin Yvon, France, or using chromatropic acid method [16], to calculate the leaching efficiency.

3. Results and discussion

3.1. Leaching in H₂SO₄

The reaction of ilmenite paste (potassium titanate, K₄Ti₃O₈, and iron oxide) with sulfuric acid could be described as follows [17]:



The main parameters that can influence the rate of this reaction are agitation, leaching time, acid concentration, acid:ilmenite paste mass ratio and temperature.

3.1.1. Agitation effect

To allow efficient mixing in the leach acid solution, many leaching processes required agitation to suspend solid particles. These processes were investigated using 6 M H₂SO₄ solution using particle size of 105–74 μm fraction with the L:S mass ratio of 9:1 at working temperature 150 °C and mixing rate ranging from 50 rpm to 500 rpm. The results of titanium leaching from the ilmenite paste with 6 M H₂SO₄ solution at different agitation speeds showed that the leaching rate of titanium is almost independent of stirring speeds higher than 375 rpm. Therefore, all experiments in this part were studied at constant stirring speed of 375 rpm. In this case, solid particles remain suspended in solution homogeneously.

3.1.2. Effect of ilmenite particle size

The effect of ilmenite particle size on the leaching of titanium from its ilmenite paste was investigated in 6 M H₂SO₄ solution with acid:ilmenite paste mass ratio 9:1 at 150 °C and 375 rpm using different particle sizes, namely, 420–297, 177–149, 105–74, 62–53, 44–37 and 37–20 μm. The results obtained show that the leaching rate generally increases by the decrease in the original particle size of ilmenite. For particle size less than 105–74 μm, the increase in the fraction of titanium dissolved is rather small (Fig. 2).

3.1.3. Effect of H₂SO₄ concentration

The effect of H₂SO₄ concentration on the leaching of titanium from its paste was investigated at 150 °C at 375 rpm stirring speed and 9:1 acid:ilmenite paste mass ratio. The H₂SO₄ concentration used was varied from 2 M to 8 M. The fraction of titanium leached vs. time plots for the different H₂SO₄ concentrations was illustrated in Fig. 3. The concentration of the acid has a significant effect on

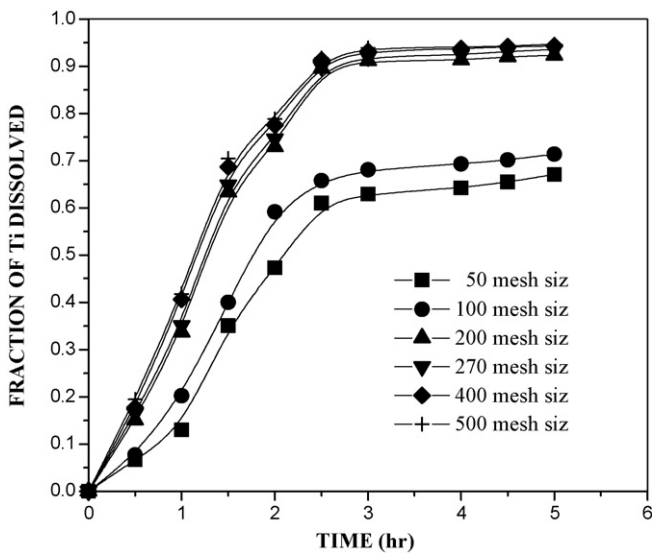


Fig. 2. Effect of particle size on the leaching of Ti in 6 M H₂SO₄ solution at 150 °C.

the leaching of the ilmenite paste but in no case was the fraction of titanium extracted more than 89%.

3.1.4. Effect of H₂SO₄:ilmenite paste mass ratio

To study the effect of H₂SO₄:ilmenite paste mass ratio on the leaching process, several experiments were carried out using ratios varying from 3:1 to 11:1 mass ratio as in Fig. 4. The other leaching parameters were fixed at acid concentration 6 M, 375 rpm stirring speed and at temperature 150 °C. From the results obtained, it is clear that the leaching efficiency of titanium is strongly dependent on the acid:ilmenite paste mass ratio which indicates that the leaching of titanium increases with the increase of acid:ilmenite paste mass ratio till 9:1. At low liquid:solid mass ratio there is insufficient amount of acid to react with the paste and with the increase in this ratio there is enough amount of acid to react with the paste and give maximum recovery.

3.1.5. Effect of temperature

Several leaching experiments were performed at temperatures ranging from 75 °C to 175 °C under leaching conditions of L:S mass ratio 9:1, 6M H₂SO₄ and at a stirring speed of 375 rpm. The results in

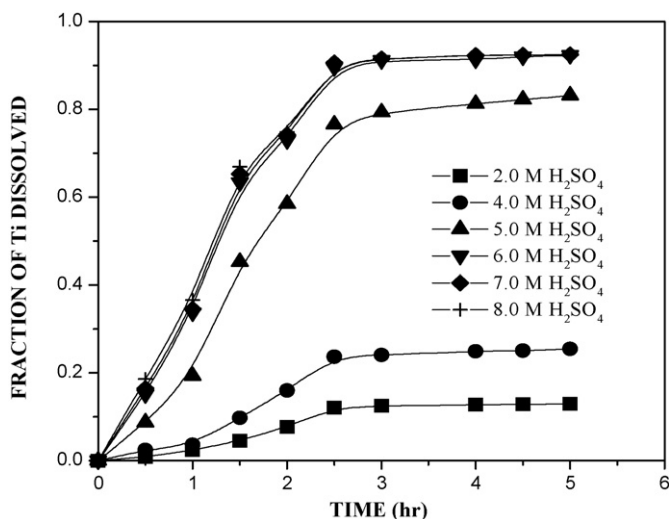


Fig. 3. Effect of H₂SO₄ concentration on the leaching of Ti at 150 °C.

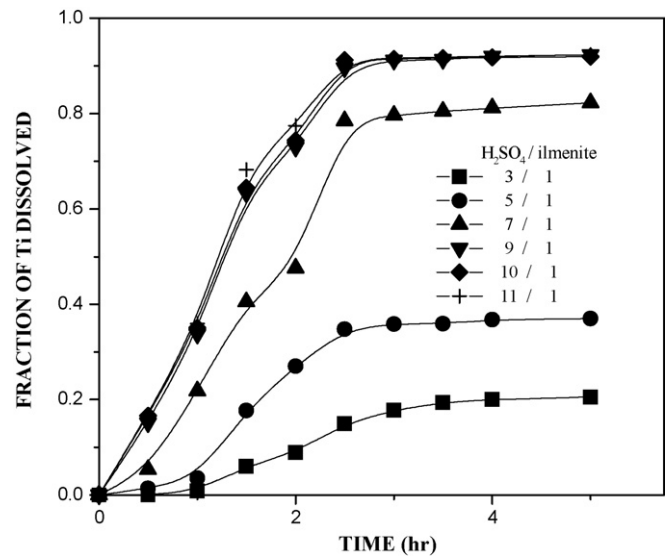


Fig. 4. Effect of H₂SO₄/ilmenite paste mass ratio on the leaching of Ti in 6 M acid solution at 150 °C.

Fig. 5 show that the extent of ilmenite paste leaching increases significantly with temperature. At 75 °C, only 21% titanium is leached then increases to about 94% titanium at 150 °C. The low leaching amounts at low temperature is due to the low reactivity of ilmenite paste, and the decrease in titanium recovery with the increase in temperature may be due to a partial hydrolysis of the ilmenite paste.

3.1.6. Proposed reaction mechanism

The reaction between the KOH paste and sulfuric acid is rather complex and needs a simplified model. The solid paste reactant was assumed to exist as round granules which react from the outer surface to the center of the paste particles without movement of the unreacted mass. During reaction the liquid acid is transported through the boundary layer and the solid product to the reaction interface. At the same time the liquid product diffuses back from the reaction interface to the bulk solution. Stirring eliminate any possible rate limit from diffusion boundary layer at the beginning of the reaction. This reaction is regarded as being probably controlled by interfacial chemical reaction. This can be related to

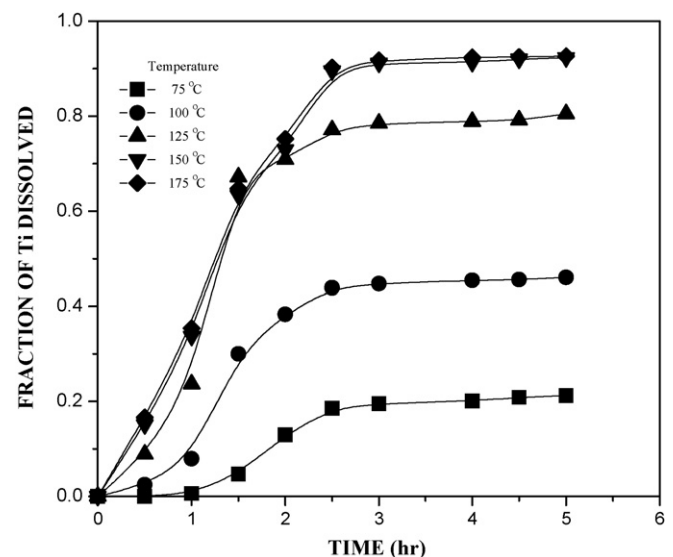


Fig. 5. Effect of temperature on the leaching of Ti in 6 M H₂SO₄ solution.

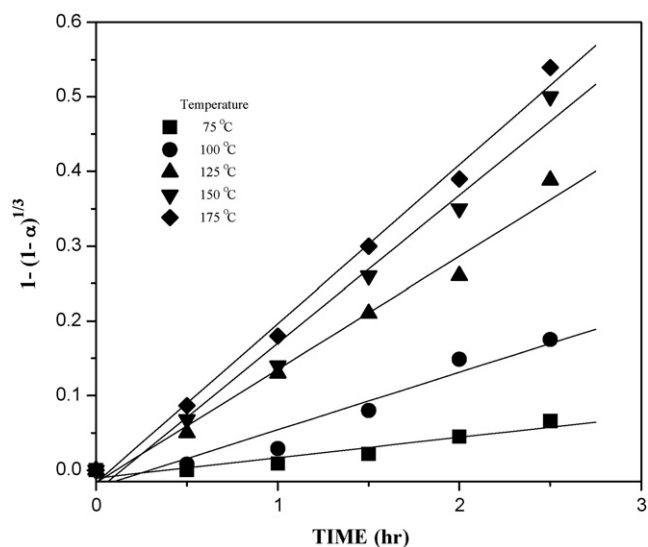


Fig. 6. Plots of $1 - (1 - \alpha)^{1/3}$ vs. time for Ti leaching at various temperature in 6 M H_2SO_4 solution.

the shrinking unreacted core model which is represented by the following relation [18]:

$$1 - (1 - \alpha)^{1/3} = \frac{Mk_c C_A t}{dr} = k_1 t \quad (3)$$

where k_c is the first-order rate constant (m min^{-1}), M is the molecular weight of the solid reactant (kg mol^{-1}), C_A is the acid concentration (mol m^{-3}), d is the density of the particle (kg m^{-3}), r is the initial radius of the particle in (m), α is the fraction reacted at time t (min) and k_1 (m min^{-1}) is the overall rate constants. This relation is also applicable to chemically controlled process.

To verify this model, the data presented in Fig. 5 are plotted according to Eq. (3). The plot of the above kinetic equation as function of time show that straight lines are obtained for the relation between $1 - (1 - \alpha)^{1/3}$ and reaction time at different temperatures (Fig. 6). This means that the leaching reaction of titanium paste granules is controlled by the chemical reaction taking place on the surface of the ilmenite paste, and verifies the present model.

3.1.7. Estimation of activation energy

The relation between the overall rate constant, k , obtained from Fig. 6 at different temperatures may be expressed by the Arrhenius equation:

$$k = A \exp\left(\frac{-E_a}{RT}\right) \quad (4)$$

where k is the overall rate constant (m min^{-1}), A is the frequency factor (min^{-1}), E_a is the activation energy (J mol^{-1}), R is the universal gas constant ($8.314 \text{ J K}^{-1} \text{ mol}^{-1}$) and T is the reaction temperature (K).

Plotting the values of $\ln k$ obtained at different temperatures against $1/T$ for the system gave a straight line, with a correlation coefficient of 0.94 (Fig. 7). This supports the Arrhenius equation and the slope of this straight line is equal to $-E_a/R$. From the slope of this plot, the activation energy of the leaching reaction of titanium from the ilmenite KOH paste by H_2SO_4 is found to equal $30.6 \pm 1.9 \text{ kJ mol}^{-1}$. This value of activation energy supports the proposed chemically controlled mechanism. Further, the value obtained is within the range of activation energy reported by several investigators. Sasikumar et al. [19], reported that in the leaching of titanium from mechanically activated ilmenite by H_2SO_4 , the activation energy varied from 14 kJ mol^{-1} to 30 kJ mol^{-1} . The activation energies reported for direct leaching of titanium from dif-

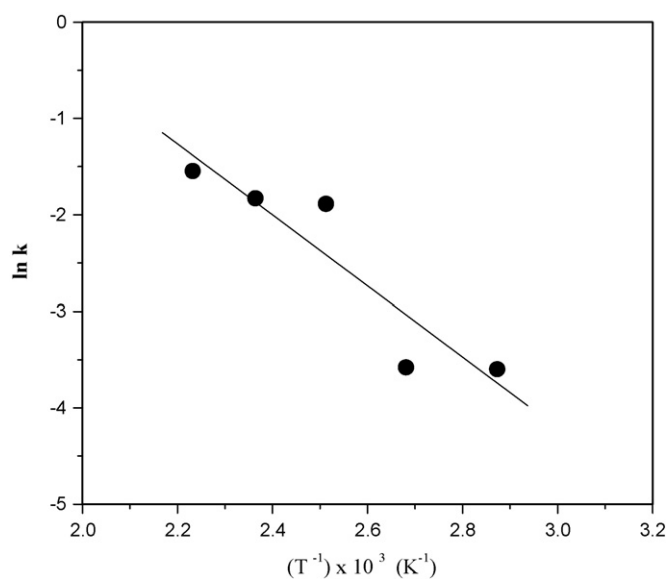
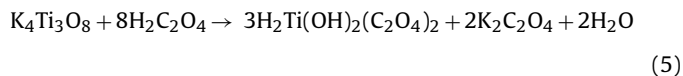


Fig. 7. Arrhenius plot for Ti leaching in 6 M H_2SO_4 .

ferent ilmenite by sulfuric acid are in the range of 41.4–49.5 kJ/mol (44.7 kJ/mol reported by Xu and Huang [10] and 41.4 kJ/mol, 49.5 kJ/mol reported by Jablonski and Przepiera [3]). These values are slightly higher than the value of activation energy obtained in this work. This is mainly related to the potassium hydroxide treatment and the formation of potassium titanate as a result of KOH treatment which requires less activation energy rather than iron titanate originally present in the ilmenite ore. Further, the results of recent studies indicated that some chemical controlled reactions could have unusually low activation energy. Olanipekun [20] stated that, it appears that the rate-controlling mechanism of heterogeneous dissolution reactions is sometimes better predicted from plots of the kinetic equations than from the activation energy values.

3.2. Leaching in $H_2C_2O_4$

At low titanium concentration, less than 0.02 M, tetravalent titanium forms with oxalate ions in solution a main mononuclear species of the type $Ti(OH)_2(C_2O_4)_2^{2-}$ rather than $TiO(C_2O_4)_2^{2-}$ [21]. Therefore, we can propose the following reaction for potassium titanate formed in the KOH treated ilmenite paste and leached in oxalic acid;



3.2.1. Agitation effect

The effect of agitation on the leaching of titanium from ilmenite paste was investigated in 80% $H_2C_2O_4$ solution with the 105–74 μm fraction of the ilmenite paste at 150 °C, using stirring speeds of in the range 125–625 rpm and acid:ilmenite paste mass ratio of 5:1. The results indicate that the extraction rate of titanium firstly increases with the increase in stirring speed till 500 rpm then becomes almost independent of the stirring speed with further increase. Therefore, subsequent experiments were performed at a stirring speed of 500 rpm to ensure that the influence of the external mass transfer is negligible; the data is not given for brevity.

3.2.2. Effect of ilmenite paste particle size

The effect of particle size on the dissolution of iron and titanium was investigated in 80% $H_2C_2O_4$ solution at 150 °C, using six

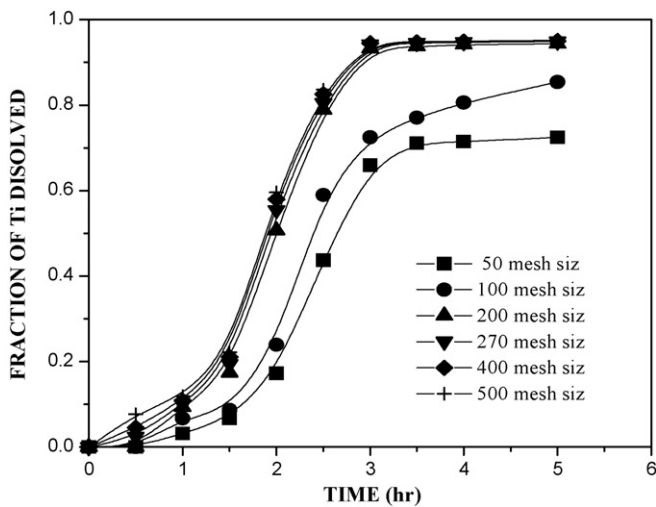


Fig. 8. Effect of particle size on the leaching of Ti in 80% oxalic acid solution at 150 °C.

particle sizes, namely, 420–297, 177–149, 105–74, 62–53, 44–37 and 37–20 μm . As expected, the results show that the leaching rates are inversely proportional to the average initial diameter of the particles less than 105–74 μm and the rate of leaching by $\text{H}_2\text{C}_2\text{O}_4$ increases with time, Fig. 8.

3.2.3. Effect of $\text{H}_2\text{C}_2\text{O}_4$ concentration

The dependence of $\text{H}_2\text{C}_2\text{O}_4$ concentration on the leaching of titanium was investigated at 150 °C using the 105–74 μm fraction of the ilmenite paste with the acid:ilmenite paste mass ratio of 5:1. The acid concentration used are 30, 40, 50, 60, 70 and 80%. The recovery of titanium vs. time plots for the different oxalic acid concentrations given in Fig. 9 show that the recovery of titanium increases with the increase of acid concentration. Such results indicate that the rate-controlling step of this system is not controlled by external diffusion as if the reaction was controlled by external diffusion, the titanium recovery would decrease with the increase of oxalic acid concentration.

3.2.4. Effect of $\text{H}_2\text{C}_2\text{O}_4$:ilmenite paste mass ratio

To study the effect of $\text{H}_2\text{C}_2\text{O}_4$:ilmenite paste mass ratio on the leaching process, several experiments were performed using ratios

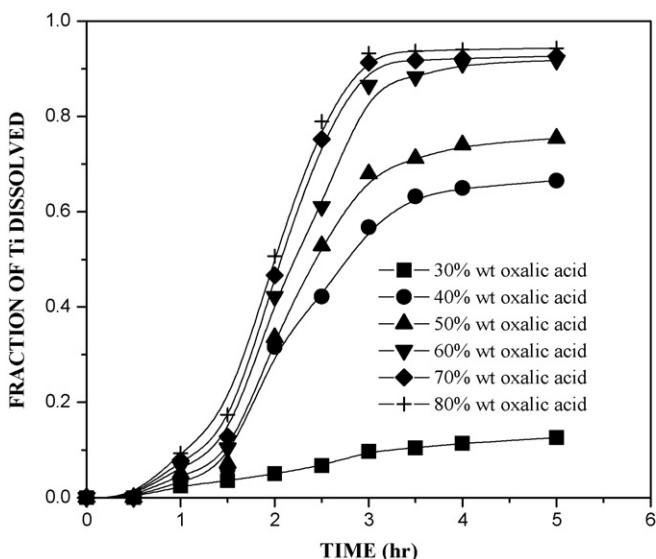


Fig. 9. Effect of oxalic acid concentration on the leaching of Ti at 150 °C.

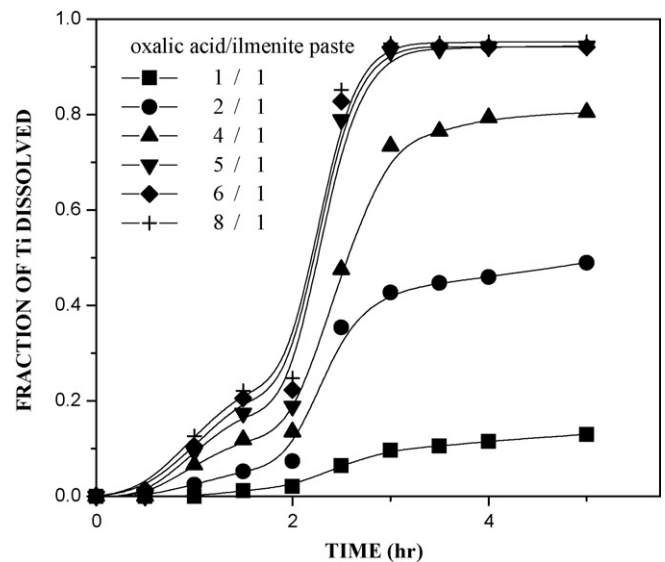


Fig. 10. Effect of oxalic acid:ilmenite paste mass ratio on the leaching of Ti in 80% oxalic acid solution at 150 °C.

varying from 1:1 to 8:1 mass ratio as in Fig. 10. The other dissolution parameters were fixed at 80% acid concentration, 500 rpm stirring speed, 105–74 μm and 150 °C.

From the results obtained, it is clear that leaching efficiency of titanium is strongly dependent on the acid:ilmenite paste mass ratio. However, the fraction of titanium dissolved was found more or less similar for oxalic acid:ilmenite paste ratios 5:1, 6:1 and 8:1, where the increase in the amount of oxalic acid did not change the amount of titanium dissolved.

3.2.5. Effect of temperature

The effect of temperature on the leaching of titanium from ilmenite paste was investigated in 80% $\text{H}_2\text{C}_2\text{O}_4$ solution using the 105–74 μm fraction with the L/S mass ratio of 5/1 at temperatures of 100, 125, 150 and 175 °C. From the results shown in Fig. 11, it can be observed that titanium was largely dissolved at 150 °C, after three hours reaction period. At higher temperatures, the fraction of titanium dissolved was found to decrease, a behavior which can

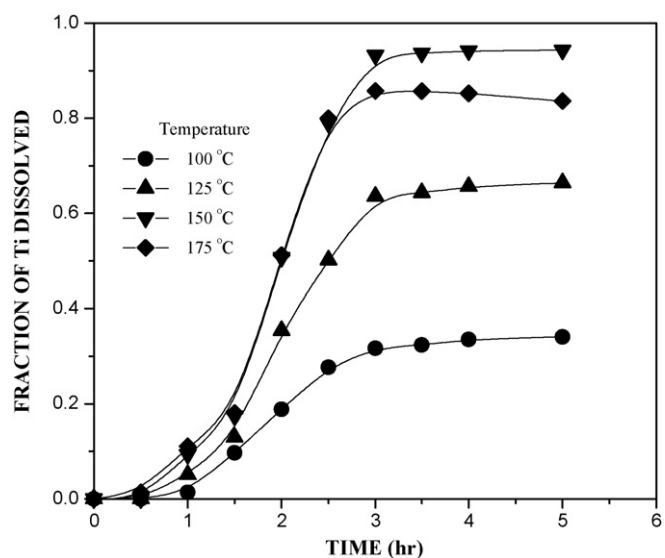


Fig. 11. Effect of temperature on the leaching of Ti in 80% oxalic acid solution.

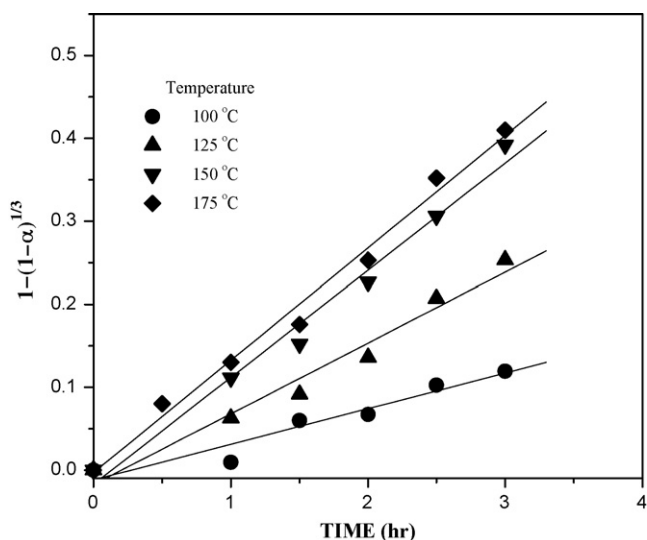


Fig. 12. Plots of $1 - (1 - \alpha)^{1/3}$ vs. time for Ti leaching at various temperature in 80% oxalic acid solution.

be related to hydrolysis of the formed soluble titanium oxalate in solution.

3.2.6. Proposed reaction mechanism

To determine the kinetic equation applicable to the results obtained, the data presented in Fig. 11, are plotted according to Eq. (3). Plots of the function $1 - (1 - \alpha)^{1/3}$ vs. time at the leaching temperatures are shown in Fig. 12. The plot of this relation gives straight lines from 0 min to 180 min. This indicates that the extraction rate gives a good correlation with the chemically controlled kinetic equation (Eq. (3)). This suggests that the leaching reaction of titanium is controlled by the chemical reaction taking place on the surface of the ilmenite paste and oxalic acid.

3.2.7. Estimation of activation energy

The rate constants for different temperatures were obtained from Fig. 12, and plotted against temperature according to Arrhenius type equations. The Arrhenius plot of the rate constants gives a very good linear straight line with a correlation coefficient of

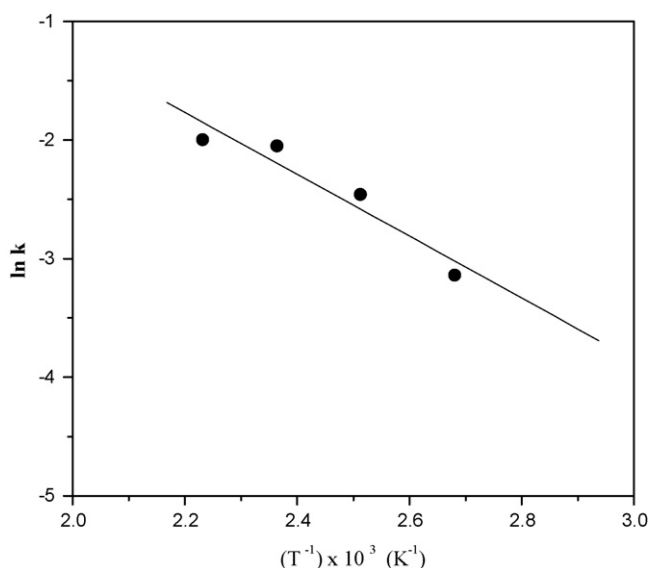


Fig. 13. Arrhenius plot for Ti leaching in 80% oxalic acid.

Table 2

Main parameters for the best working leaching conditions of ilmenite paste by HCl^a, H₂SO₄ and H₂C₂O₄.

Parameter	Acid		
	HCl	H ₂ SO ₄	H ₂ C ₂ O ₄
Stirring speed (rpm)	375	375	500
Time (h)	2	2.5	3
L/S (mass ratio)	4/1	9/1	5/1
Acid concentration	9.0 M	6.0 M	70%
T (°C)	125 °C	150 °C	150 °C
%R (Ti)	92%	89%	93%
%R (Fe)	76%	91%	73%

^a Reference: A.A. Nayl, H.F. Aly, 2009.

0.96 for titanium, Fig. 13. The slope of this straight line is equal to $-E_a/R$. The Arrhenius activation energy of the leaching titanium from ilmenite paste by oxalic acid was calculated and found to equal $22.6 \pm 1.5 \text{ kJ mol}^{-1}$. This value of activation energy supports the proposed chemically controlled mechanism for this reaction.

4. Comparison between different leaching reagents

To assess the efficiency of the different reagents used for leaching the KOH treated ilmenite in terms of the amount of titanium leached and the amount of iron impurities in the leached solution, the main parameters for the best working leaching conditions are given in Table 2. From this table, it is clear that to obtain high yield

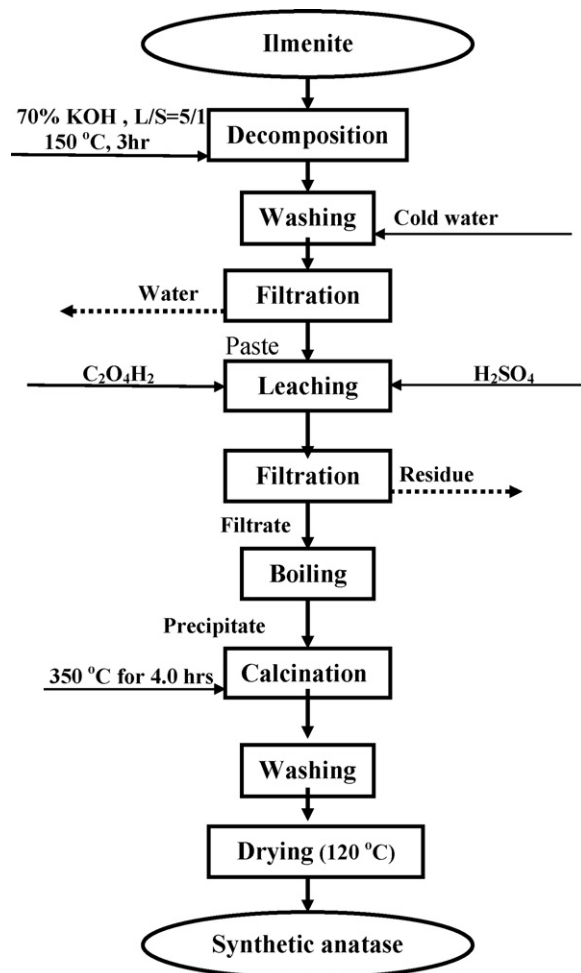


Fig. 14. Schematic flow diagram of the decomposition and leaching of titanium from ilmenite paste by H₂SO₄ or C₂O₄H₂.

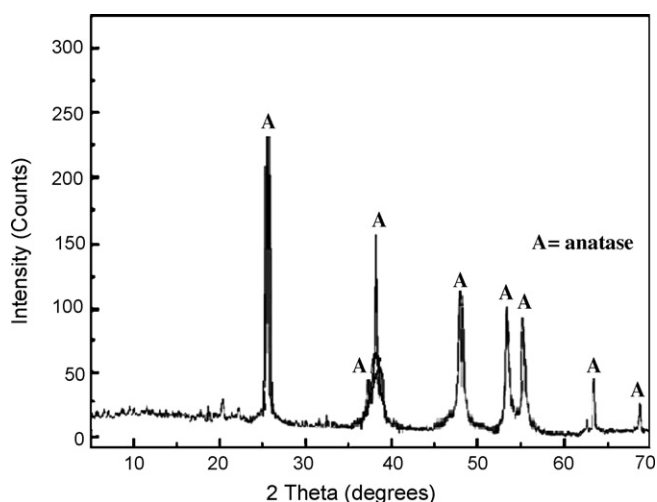


Fig. 15. XRD patterns of anatase TiO_2 .

for titanium with lower iron concentration, oxalic acid is the preferred leaching reagent. Other advantages are the reasonable L:S ratio and the less corrosive action of this weak acid. A drawback of this reagent is the relatively high energy required and the high cost of oxalic acid than the two other reagents. While HCl or H_2SO_4 need less energy yet these leaching reagents produce less titanium and more iron in the product. Further their corrosive action should be taken into consideration. Therefore, a proposed flow diagram based on leaching using oxalic acid is developed.

5. Proposed flow diagram

Based on the aforementioned investigations, a diagram based on oxalic acid leaching is proposed and tested, Fig. 14. In this case, one portion of 100 g ilmenite paste with particle size of 105–74 μm was taken and digested with 500 g of 70% KOH solution for three hours at 150 °C. The obtained paste was washed with water till neutrality. To this treated ilmenite, 500 g of 70% oxalic acid solution was added and agitated with a stirrer at 500 rpm for 3 h and a temperature of 150 °C. The obtained solution was filtered and analyzed by ICP for Ti, Fe, Cr, and V. The results obtained indicated the recovery 23.0 g of titanium, 13.3 g of iron, 0.24 g of chromium and 0.11 g of vanadium. The obtained solution was further concentrated by boiling to 1/5 of its volume, where titanium was hydrolysed and a gelatinous precipitate is formed and separated by filtration. This product was taken and calcinated at 350 °C for 4 h, washed with 0.5 M HCl solution then with distilled water and dried at 120 °C. The X-ray diffraction pattern of the product shown in Fig. 15 indicates a well crystalline anatase type TiO_2 . Chemical analysis of this product indicates that it contains more than 98% TiO_2 , 1.3% Fe_2O_3 , 0.04% SiO_2 and 0.3% MnO.

6. Conclusions

In this study, the leaching kinetics of ilmenite paste obtained from KOH treated ilmenite by both sulfuric and oxalic acid solutions were investigated in the batch reactor. The results revealed that the

leaching rate increases with increasing the reaction temperature, acid concentration, stirring speed, liquid:solid ratio, and particle size. From the results the most significant parameters affecting the leaching rate were found to be the L:S mass ratio, acid concentration and temperature.

Ilmenite paste leaching kinetics was better described by the chemical control model than the others models for the both acids. The activation energy of titanium leaching from ilmenite paste by H_2SO_4 solution was found to equal $30.6 \pm 1.9 \text{ kJ mol}^{-1}$, the activation energy value was decreased to $22.6 \pm 1.5 \text{ kJ mol}^{-1}$ when titanium was leached with oxalic acid.

Comparative studied between the different investigated leaching reagents, showed that leaching of the KOH-treated ilmenite by oxalic acid has some advantages over HCl or H_2SO_4 . A flow diagram for KOH decomposition of ilmenite and leaching with oxalic acid is proposed and tested. The developed process has an efficiency of 93% and produced anatase TiO_2 with a purity of more than 98%.

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