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Reductive leaching of Rosetta ilmenite in sulfuric acid solutions

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ABSTRACT

The sulfate route for digestion ilmenite ores has been applied upon Rosetta concentrate, however, under a variety of modified conditions. Instead of working in an almost solid state, it was found interesting to explore the possibility of working in a fluid state by working in a relatively low solid/liquid ratio of 1/5 to 1/4. In the meantime, the possibility of applying the sulfate route upon mixed ilmenite/Ti slag was investigated; a matter which would realize a dual benefit; namely increasing input TiO_2 assay and ensuring better reduction. In this case, the applied S/L ratio has been varied from 1/8 to 1/6.

KEYWORDS

Ilmenite;
TiO_2 leaching;
Sulfuric acid.

INTRODUCTION

Ilmenite mineral ($FeO.TiO_2$ or $TiFeO_3$) contains about 40–65% TiO_2 depending on its geological history while its natural alteration product leucoxene ($Fe_2O_3.nTiO_2$) contains more than 65% TiO_2. Nowadays and after the excessive depletion of rutile mineral (TiO_2), ilmenite has become the major ore mineral of titanium where it supplies about 91% of the world's demand for titanium minerals[15]. In 2009, the world's ilmenite production has attained 5.19 million metric tons. Besides beach sands, ilmenite can also be found in hard rock deposits (as is the case in Rosetta and Abu Ghalga respectively in Egypt) and where its world reserves are quite extensive reaching up to 1300 million tons.

The most widely used titanium product is titania or titanium dioxide (TiO_2) which is mostly used as pigment or else as filler in paper, plastics and rubber industries and as flux in glass manufacture. Only about 6% of the world production is used to produce metallic titanium. Two principal methods are commonly employed to produce TiO_2 pigment; namely the sulfate and the chloride routes. According to Hamor (1986), the two routes are quite different in both their chemistry and the required raw material. Thus, while the chloride route is advantageous regarding both cost and waste management, it requires however a high-grade titanium raw material; viz, natural or synthetic rutile or else titanium slag (> 90% TiO_2). In other words, ilmenite raw material necessitates a prior upgrading due to the shortage of natural rutile. On the contrary, the upgrading processes are generally expensive as they involve several steps of energy-sensitive thermo reductive conversion besides a post-leaching step for removal of iron impurities. In this regard, it has to be indicated that the chloride process is less expensive to operate than the sulfate process at larger scales[8]. These facts have rendered the necessity of applying the hydrometallurgical sulfate route and which was further extended to apply HCl leaching followed by solvent extraction[2].

The sulfate process was the first commercial process for the manufacture of titania from titaniferous ores such as ilmenite. It uses a lower grade, cheaper raw materials and simpler technology involving prior crystallization of $FeSO_4.7H_2O$ and directly followed by hydrolysis of titanium hydroxide results in a form of pigment called anatase. The latter is preferred over the pigment from the chloride process for use in papers, ceramics and inks. However, the major disadvantage of this route is that it produces large quantities of waste iron sulfate besides consuming large quantities of sulfuric acid i.e. higher product costs for expensive acid treatment than the chloride process. In addition, the digestion reaction proceeds very violently, emitting a large amount of SO_3 and H_2SO_4 vapors, leading to many environmental problems[7].

To avoid the drawbacks resulting from conventional sulfate digestion process, numerous investigations have been carried out and innovative techniques developed to improve the process[5,11,14]. It was concluded that dissolution of the ore material with dilute acid solutions can greatly reduce the emission of vapors during digestion and also reuse significant amount of the dilute waste acid produced during hydrolysis. However, ilmenite dissolution was found to be very slow under such conditions[10]. Recently, a process has been described for reductive dissolution of ilmenite in sulfuric acid solution media to accelerate the dissolution processes[12]. It involves digestion of the ore material in more than one leaching circuit.

The present work has studied the potential leachability of Rosetta ilmenite in sulfuric acid under less severe conditions to reduce the expensive production cost of titania pigment. Accordingly, a direct reductive leaching technique has been chosen using metallic iron. In the meantime, a trial to process mixed ilmenite/Ti slag has also been tested to realize a dual benefit; namely increasing the Ti assay of the input ore and to ensure better reduction of Fe^{3+} iron phases in the working ilmenite.

EXPERIMENTAL

Materials

A technological sample of Rosetta beach ilmenite concentrate was kindly provided by the Black Sands Project of the Nuclear Materials Authority (NMA) after proper physical upgrading. Mineralogically, the received ilmenite concentrate is only slightly contaminated with trace amounts of magnetic zircon, rutile or leucoxene, chromite and chrome-spinel. This sample has been chemically analyzed for both the major and minor oxides before its processing in the present work.

A titania slag sample prepared from Rosetta ilmenite by carbothermal reduction in an electric arc furnace was also kindly provided from the Titanium Project of the Nuclear Materials Authority (NMA). This sample has also been chemically analyzed for both the major and minor oxides before its processing in the present work.

Experimental procedure

The dissolution experiments were performed in 250 ml conical flasks fitted with a condenser and stirrer. After adding the required amounts of the ilmenite concentrate sample and sulfuric acid solution the metallic iron powder as a reductant was added after 15 minutes and the obtained slurry was agitated at various temperatures at 350 rpm. At the end of each dissolution experiment in both working procedures of ilmenite or mixed ilmenite/Ti slag, the slurry was filtered, washed with distilled water and the obtained filtrate was analyzed for titanium and iron to calculate their dissolution efficiencies. However, in the first procedure of ilmenite, 30 g sample portions of ilmenite concentrate in a S/L ratio of 1/5 to 1/4 was used and after filtration of the obtained slurry and proper washing of the remaining solids, the leach liquor was made up to a volume of 250 ml. On the other hand, 10 g sample portions of mixed ilmenite/ Ti slag were processed in a S/L ratio of 1/8 to 1/6 and after filtration and proper washing, the leach liquor was made up to a volume of 350 ml.

Analytical procedures

(a) Material analysis

The provided ilmenite concentrate and the titania slag sample were first analyzed after preparing their solutions through flame fusion using potassium pyro-sulfate. The fused mass was then dissolved in hot concentrated H_2SO_4 until obtaining a clear solution which was then made to volume. Titanium and total iron were then analyzed using colorimetric and titrimetric analytical methods respectively. The spectrophotometric determination of titanium was performed using tiron as the complexing agent and measuring the obtained absorbance at 430 nm using Unicam UV2-100. Total iron was titrimetrically determined against EDTA using sulfosalicylic acid as indicator while the ferrous content was also titrimetrically analyzed against a standard potassium dichromate solution. Both calcium and magnesium have also been titrimetrically determined against EDTA using Eriochrome Black T and murexide indicators[13]. Finally, the molybdate reagent in the presence of tartaric acid has been used for the determination of the silica content at 640 nm while Alizarine red S was used for the Al_2O_3 content at 475 nm. Analysis of the main three trace elements present (Mn, Cr and V) was performed by means of the atomic absorption technique using Unicam 969 Model at 279.5, 357.9 and 318.4 nm respectively. TABLES 1 and 2 summarize the obtained analytical results of the working ilmenite concentrate and the Ti slag sample respectively. In addition, the X-ray diffraction pattern of Rosetta titania slag has indicated that its main component is pseudobrookite as shown in Figure 1. The stoichiometric composition of the latter is M_3O_5 which represents the solid solution of three components as will be later shown.

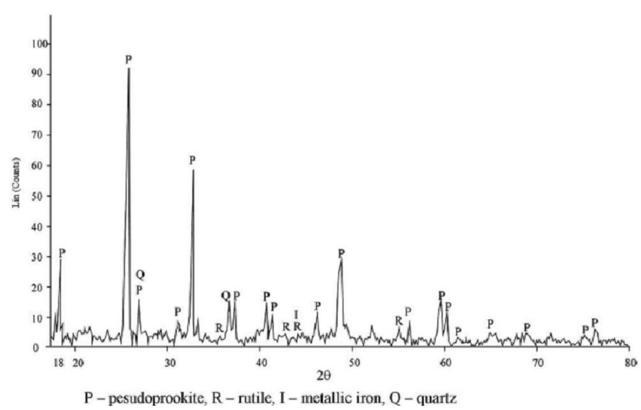

Figure 1 : X-ray diffraction pattern of titania slag of osetta ilmenite (after Lasheen, 2008)

(b) Control analysis

In order to follow the experimental work of leaching efficiencies of Ti and Fe besides their separation, all the working solutions have been subjected to colorimetric and titrimetric analysis using tiron and EDTA respectively.

RESULTS AND DISCUSSION

Sulfate route of Rosetta ilmenite digestion

(a) Effect of acid concentration

To study the effect of H_2SO_4 concentration for titanium dissolution under the working S/L ratio of 1/4, several dissolution experiments were performed using acid concentrations varying from 7.14 to 10.70 M. The other dissolution parameters were fixed at a temperature of 120°C for 3 h reaction time and using ilmenite concentrate sample portions ground to -325 mesh size besides using metallic iron in a weight ratio of 10% of the input ilmenite as a reductant. The expected reactions can be represented as follows:

$$FeTiO_3 + 2H_2SO_4 \rightarrow FeSO_4 + TiOSO_4 + 2H_2O \quad (1)$$

TABLE 1 : Chemical compositions of East Rosetta ilmenite concentrate

SiO_2	CaO	Al_2O_3	MgO	Cr_2O_3	V_2O_5	MnO	FeO	Fe_2O_3	TiO_2	Oxide
0.75	0.44	0.90	0.80	0.29	0.18	1.15	28.50	21.41	44.01	Wt.%

TABLE 2 : Chemical composition of the titania slag

SiO_2	CaO	Al_2O_3	MgO	Cr_2O_3	V_2O_5	MnO	$Fe_{(total)}$	TiO_2	Oxide
9.00	0.78	1.80	0.60	0.32	0.55	1.63	12.65	72.00	Wt.%

$$Fe_2O_3 + 3H_2SO_4 \rightarrow Fe_2(SO_4)_3 + 3H_2O \quad (2)$$

$$Fe_2(SO_4)_3 + Fe \rightarrow 3FeSO_4 \quad (3)$$

From the obtained results shown in TABLE 3, it is

TABLE 3 : Effect of acid concentration upon TiO_2 dissolution efficiency (3 h, 120°C, S/L ratio 1/4 and10% metallic iron)

Acid Concentration,M	7.14	8.03	8.92	9.81	10.70
TiO_2 dissolution efficiency, %	66.3	81.4	90.0	88.2	86.8

clearly evident that the dissolution efficiency of TiO_2 is directly proportional to the acid concentration where at 8.92 M H_2SO_4, a dissolution efficiency of up to 90% was realized. The low dissolution of TiO_2 at 7.14 M might be due to the relatively low acidity, while the observed low dissolution efficiency of TiO_2 beyond 8.92 M is most probably due to possible precipitation of $TiOSO_4$ (and $FeSO_4$) at higher acidity[9] according to the following reaction:

$TiOSO_4 + 2H_2O \rightarrow TiOSO_4.2H_2O$

It covers the surface layer of ilmenite grains in a manner to inhibit both H^+ and product diffusion and in turn its further dissolution and early solidification occurs. In addition, it might also be possible that this decrease in TiO_2 dissolution might be ascribed to the formation of Ti_2O_3 which is less soluble in H_2SO_4 than the tetravalent titanium species besides precipitation of titanyl sulfate[3]. It is interesting to mention here that the acid amount required for dissolving ilmenite according to reaction (1) attains 1.29 g for 1 g ilmenite mineral. In the present work, the remaining acid has been analyzed in the leach liquor that has been obtained under the optimum conditions that will be later mentioned. From the latter, the acid consumption has been found to attain 0.88 g for 1 g of the working ilmenite sample. The same obtained by Roche et al[8] who have found a consumption of 0.89 - 0.88 g acid for 1 g ilmenite.

(b) Effect of time

Four dissolution experiments for a reaction time periods of 1 to 3.25 h were carried out at 120°C in presence of a metallic iron weight ratio of 10% to input ilmenite and using 8.92 M H_2SO_4 acid in a S/L ratio of 1/4.

From the obtained results of titanium dissolution summarized in TABLE 4, it is clear that for 1 to 3 h

TABLE 4 : Effect of time upon TiO_2 dissolution efficiency (8.92 M H_2SO_4, 120°C, S/L ratio 1/4 and10% metallic iron)

Time, h	1	2	3	3.25
TiO_2 dissolution efficiency, %	79.1	86.1	90.0	90.2

reaction time, the dissolution efficiency of titanium increased from about 79 to 90%. Extending the leaching time to 3.25 h, only a slight increase in the dissolution efficiency was obtained and therefore, only 3 h would represent an optimum time for obtaining 90% leaching efficiency of titanium.

(c) Effect of S/L ratio

Working with 8.92 M H_2SO_4, the effect of S/L ratio was studied in the range of solid to liquid ratio of 1/3 to 1/6 while the other dissolution conditions were fixed at a temperature of 120°C and a reaction time of 3 h besides using 10% metallic iron relative to ilmenite samples.

The obtained results (TABLE 5) indicate that the

TABLE 5 : Effect of S/L ratio upon TiO_2 dissolution efficiency (8.92 M H_2SO_4, 3 h, 120°C and10% metallic iron)

S/L Ratio	1/3	1/4	1/5	1/6
TiO_2 dissolution efficiency, %	65.8	90.0	91.4	93.3

dissolution efficiency of TiO_2 is directly proportional to the decrease in the pulp density and in turn effective availability of acid solution to the ilmenite grain surface. So, at the S/L ratio of 1/3, the dissolved titanium is only about 66% while decreasing the S/L to 1/4 and 1/5, increased the available acid and in turn an increase in TiO_2 dissolution up to 90 and about 91% respectively. Further decreasing the S/L ratio to 1/6 has only increased the Ti dissolution efficiency by about 2% i.e. 93% and therefore a S/L ratio of 1/4 to 1/5 would be considered as the optimum value.

(d) Effect of reductant amount

Using a solid/liquid ratio of 1/5 and fixing the working conditions at an acid concentration of 8.92 M at a temperature of 120°C, another series of leaching experiments have been made in which the added metallic iron ratio to the input ilmenite was varied between 7 and 12%.

The obtained results summarized in TABLE 6 re-

TABLE 6 : Effect of reductant amount upon TiO_2 dissolution efficiency (8.92 M H_2SO_4, 3 h, 120°C and S/L ratio 1/5)

Reductant,%	7	8	9	10	12
TiO_2 dissolution efficiency,%	83.8	86.5	89.0	91.4	87.0

veal the importance of incorporating the metallic iron in the leaching medium as a reductant to increase the dissolution rate of ilmenite via reduction of the Fe^{3+} iron. This is due to the fact that ferrous iron of the processed ilmenite is partially oxidized to its trivalent state which is much less soluble besides the presence of mixed or inter-grown phases containing Fe^{3+} like the following mineral phases; namely:

$FeO.Fe_2O_3$–Fe_2TiO_4: magnetite–ulvo–spinel series

Fe_2O_3. $FeTiO_3$: hematite–ilmenite series

Fe_2TiO_5–$FeTi_2O_5$: pseudobrookite series.

Incorporation of a suitable reductant like metallic iron would reduce the relatively refractory oxidized Fe^{3+}/Ti phases. Accordingly, it was found that the dissolution efficiency of titanium has steadily increased from about

84 to about 91% by increasing the metallic iron ratio from 7 to 10%. However, further increasing the reductant beyond 10% has led to a decrease in TiO_2 dissolution by about 4% due to increased acid consumption (as a result of iron dissolution).

To demonstrate the importance of iron as a reductant in improving the leaching efficiency of titanium from Rosetta ilmenite, some experiments have been performed to determine the optimum iron value. However, the performed experimental conditions involved a sulfuric acid concentration of 7.14 M in a S/L ratio of 1/8 besides working at 90°C for 4 h. TiO_2 dissolution efficiencies of these experiments have been as follows:

Metallic iron / ilmenite, %	TiO_2 dissolution efficiency, %
0	46.6
5	50.9
7.5	69.1
10	81.2

Thereafter, at 15 and 20% iron ratio to the input ilmenite, the dissolution efficiency was decreased to 74.0 and 70.8% respectively; a matter which might be due to excessive acid consumption by iron dissolution.

(e) Effect of temperature

To study the effect of temperature, different reaction temperatures ranging from 100 up to 140°C have been applied while fixing the other experimental conditions at an acid concentration of 8.92 M for 3 h reaction time in a S/L ratio of 1/5 and using 10% metallic iron.

From the obtained results (TABLE 7), it was found

TABLE 7 : Effect of temperature upon TiO_2 dissolution efficiency (8.92 M H_2SO_4, 3 h, S/L ratio 1/5 and 10% metallic iron)

Temperature, °C	100	110	120	130	140
TiO_2 dissolution efficiency, %	72.6	82.4	91.4	85.3	78.5

that the extent of TiO_2 dissolution increases as the temperature rises from 100 to 120°C. Thus while the obtained dissolution efficiency was only about 73% at 100°C due to the lower breakdown of ilmenite, it was increased up to about 91% at 120°C. On the contrary, increasing the temperature beyond 120°C, the dissolution efficiency of titanium has adversely been affected; a matter which might be due to premature hydrolysis[8] according to the following reaction:

$$TiOSO_4 + 2H_2O \rightarrow TiO(OH)_2 + H_2SO_4$$

The latter would thus result in the increased viscosity and to the varying degrees of solidification. Accordingly, 120°C would be considered as the optimum temperature for dissolution of about 91% of titanium at the mentioned conditions.

Sulfate digestion route of mixed Rosetta ilmenite/ Ti Slag

The Ti slag product of ilmenite ore is essentially required to increase its Ti content in a manner to be convenient for the chloride route. However, in the present work it was thought to increase the Ti assay in Rosetta ilmenite via its partial smelting followed by application of the sulfate route upon a mixed raw ilmenite/Ti slag product. Besides increased Ti assay, there would be no need to use an additive reductant during dissolution due to presence of both remnant metallic iron and trivalent titanium species in the Ti slag. From the assay of the working ilmenite and Ti slag (TABLES 1 and 2 respectively), the Ti and metallic iron assay in the mixed raw ilmenite/Ti slag in different ratios would be as follows:

Ti slag in ilmenite,%	TiO_2,%	Metallic Fe*,%
0	44.0	37.0
5	45.4	35.8
10	46.8	34.6
15	48.2	33.4
20	49.6	32.2
25	51.0	31.0
30	52.4	29.8

*Fe_2O_3 and FeO of ilmenite are calculated as total metallic iron

(a) Effect of acid concentration

To study the effect of H_2SO_4 concentration upon the dissolution efficiency of the working mixed 80% ilmenite / 20% Ti slag ground to -325 mesh size, several experiments were performed using acid concentrations varying from 5.35 to 12.49 M. The other dissolution parameters were fixed at a S/L ratio of 1/8 at a temperature of 120°C and for 4 h reaction time. Under these conditions, the metallic iron wt. ratio in the working sample would attain 2.5% (equivalent to 2 g slag of 12.65% iron in 10 g mixed ilmenite/Ti slag).

From the obtained results shown in TABLE 8, it is

TABLE 8 : Effect of acid concentration upon TiO_2 dissolution efficiency from mixed Rosetta ilmenite / Ti slag in 20% wt. ratio (4 h, 120°C and S/L ratio 1/8)

Acid concentration		Dissolution efficiency, %	
Vol. %	M	Titanium dioxide	Total iron
30	5.35	46.4	68.4
40	7.14	64.9	87.6
50	8.92	80.3	95.9
60	10.70	83.7	97.9
70	12.49	79.5	92.8

obvious that the dissolution efficiency of either TiO_2 or total iron is directly proportional to the acid concentration up to 10.70 M H_2SO_4 where at this acidity the dissolution efficiency of TiO_2 and total iron attained about 84 and 98% respectively. Thereafter, the dissolution efficiency of both TiO_2 and iron has decreased to about 80 and 93%; a matter which can be interpreted as due to possible precipitation of titanyl and iron sulfates besides possible formation of Ti_2O_3 which is less soluble as previously mentioned. Accordingly at the higher acidity input of 12.49 M the diffusion would thus be hindered and solidification of the reacting mass would occur in varying degrees. On the other hand, the almost complete dissolution of iron at 10.7 M acid and in the presence of only 2.5% metallic iron could be due to effective reduction of Fe_2O_3 content of input ilmenite by presence of both metallic iron as well as some Ti in the added Ti slag in its trivalent state. The latter is due to the fact that the main component of the titania slag is pseudobrookite (M_3O_5) and which is a solid solution of $Fe^{2+}Ti_2^{4+}O_5$, $Ti^{4+}Ti_2^{3+}O_5$ and $Ti^{4+}Fe_2^{3+}O_5$ end terms implying the occurrence of multiple valencies of Fe and Ti (Dondi et al, 2008).

(b) Effect of titania slag / ilmenite ratio

Using a solid/liquid ratio of 1/8 and fixing the working conditions at an acid concentration of 10.70 M at a temperature of 120°C, another series of leaching experiments have been made in which the ratio of the input mixed ilmenite/Ti slag content was varied from 0% to 30%.

From the obtained results (TABLE 9), it is clearly evident that the dissolution efficiency of either TiO_2 or total iron is directly proportional to titania slag/ilmenite ratio up to 10% where the obtained efficiencies attained about 89 and 100% respectively. It is clear that titania slag plays an important role as a reductant in spite of the presence of only 1.25% metallic iron in the working sample. Although increasing the titanium dioxide content in the input working material, presence of titania slag beyond 10% in the input ore would however lead to a decrease in the dissolution efficiency of both TiO_2 or total iron. It might be due to presence of greater amount of inert composition titania slag and which would require working in more severe conditions.

TABLE 9 : Effect of titania slag / ilmenite ratio upon TiO_2 dissolution efficiency (10.70 M H_2SO_4, 4 h, 120°C and S/L ratio 1/8)

Titania slag / ilmenite ratio wt. %	Dissolution efficiency, %	
	Titanium dioxide	Total iron
nil	61.7	75.0
5	84.9	96.4
10	88.7	99.9
15	86.5	99.6
20	83.7	97.9
25	79.5	95.3
30	74.6	92.5

(c) Effect of S/L ratio

Working with 10.70 M H_2SO_4, the effect of S/L ratio was studied in the range of 1/4 to 1/8 while the other dissolution conditions were fixed at a temperature of 120°C and a reaction time of 4 h besides using ilmenite samples mixed with 10% titania slag.

TABLE 10 : Effect of S/L ratio upon TiO_2 dissolution efficiency (10.70 M H_2SO_4, 4 h, 120°C and 10% titania slag)

S/L ratio	Dissolution efficiency, %	
	Titanium dioxide	Total iron
1/4	65.1	84.4
1/5	77.1	93.4
1/6	87.8	99.3
1/7	88.0	99.8
1/8	88.7	99.9

The obtained results are summarized in TABLE 10 and indicate that the dissolution efficiency of both TiO_2 and total iron is directly proportional to the decrease in the pulp density. At the S/L ratio of 1/4, the dissolved TiO_2 and total Fe attained about 65 and 84% respectively and which was increased to about 88 and 99% at the S/L ratio 1/6. Thereafter only slight increase in the dissolution efficiency of both was obtained.

(d) Effect of temperature

Using 10% wt. ratio Ti slag in ilmenite and fixing the S/L ratio at 1/6, several dissolution experiments were performed at reaction temperatures ranging from 90 up to 160°C while the other working experimental conditions involved an acid concentration of 10.70 M for 4 h reaction time.

From the obtained data (TABLE 11), it was found that TiO_2 dissolution increases as the temperature rises

TABLE 11 : Effect of temperature upon TiO_2 dissolution efficiency (10.70 M H_2SO_4, 4 h, S/L ratio 1/6 and 10% titania slag)

Temperature, °C	Dissolution efficiency, %	
	Titanium dioxide	Total iron
90	66.5	78.3
100	75.4	88.5
120	87.8	99.3
130	92.2	99.4
140	97.1	99.7
150	89.7	95.1
160	84.0	90.7

from 90 to 140°C. Only about 67% of TiO_2 together with about 78% of total iron have been obtained at 90°C. It increased up to about 92 and 97% titanium dissolution with almost complete total iron leaching at 130 and 140°C respectively. However, increasing the temperature to 150 and 160°C, the dissolution efficiency of both TiO_2 and total iron has adversely been affected to about 90 and 84% with respect to titanium and to about 95 and 91 with respect to iron respectively. This decrease in dissolution efficiencies is related to possible premature hydrolysis of titanyl sulfate as previously mentioned in a manner to obtain an increased degree of solidification that would inhibit diffusion.

CONCLUSION

Two modified procedures of the sulfate digestion route have been applied upon Rosetta ilmenite. Applying a solid/liquid ratio of 1/5, it was found that the optimum conditions required to realize 91.4% TiO_2 dissolution efficiency involve using a H_2SO_4 acid concentration of 8.92 M at 120°C for 3 h and using 10% metallic iron addition and grinding the ilmenite sample to -325 mesh size. Under these conditions, the obtained Ti and acid concentrations would attain 28.95 g/l and 419.0 g/l respectively. Applying the sulfate route to a mixed Rosetta ilmenite/Ti slag, it was possible to realize up to 97.1% TiO_2 dissolution efficiency using H_2SO_4 acid concentration of 10.7M at 140°C for 4 h. Under these conditions, the obtained Ti concentration assays 7.79 g/l and 156.8 g/l acid concentration. The process of the present work has a significant advantage in terms of reducing the disposal problems of waste acid solutions whereas recycled spent acid can be used and accelerating the rate of leaching ore material. In addition, the use of a reducing in the digestion step eliminates the prior arts for a separate and independent reduction step following digestion step.

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