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## IRON REMOVING FROM TITANIUM SLAG FOR SYNTHETIC RUTILE PRODUCTION

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**Abstract.** Leaching with sulfuric and hydrochloric acids for upgrading titanium slag obtained from the Qara-aghaj ilmenite concentrate to a high grade titanium dioxide ( $\text{TiO}_2$ ) was studied. The titanium slag containing 72.7%  $\text{TiO}_2$  and 7.8%  $\text{Fe}_2\text{O}_3$  was leached at varying solid/liquid (S/L) ratio, particle size, leaching time and acid concentration. The optimum amount of S/L ratio, particle size, acid concentration and leaching time using both acids were determined as 1:4,  $-100\ \mu\text{m}$ , 8% and 2 h. Under optimum conditions, by using sulfuric acid, a titanium dioxide concentrate with 86.8%  $\text{TiO}_2$  and 1.87%  $\text{Fe}_2\text{O}_3$  was produced, while employing hydrochloric acid resulted in a concentrate containing 91%  $\text{TiO}_2$  and 0.61%  $\text{Fe}_2\text{O}_3$ . The titanium dioxide concentrate prepared with hydrochloric acid, having acicular texture and by means of elongated separate particles; has less impurities than the one produced by sulfuric acid. The results demonstrate that hydrochloric acid dissolution of titanium slag and removing some impurities such as  $\text{SiO}_2$  and  $\text{MgO}$  provide a product with good quality is prepared which is suitable as raw material for  $\text{TiO}_2$  pigment production through the chloride process.

*keywords:* titanium slag, synthetic rutile, chloride process, sulfate process, acid leaching,  $\text{TiO}_2$  pigment

### 1. Introduction

Titanium is relatively abundant in the earth's crust, which is usually found in igneous and metamorphic rocks as ilmenite ( $\text{FeTiO}_3$ ), rutile ( $\text{TiO}_2$ ) and titanomagnetite ( $\text{Fe}_2\text{TiO}_4\text{--Fe}_2\text{O}_4$ ). A survey on the use of titanium in its various forms indicates that almost 95% is for the production of white  $\text{TiO}_2$  pigment which has extensive application in paint, plastic and paper industries (Nayl et al., 2009; Nayl et al., 2009). Titanium dioxide also has high potential applications in environmental purification, gas sensors, and in photovoltaic cells due to its unique characteristics (Zang et al., 2009). Relatively minor quantities are used to produce metal and titanium chemicals (Lasheen, 2004).

Natural rutile, owing to its high titanium content and low levels of impurities, has traditionally been preferred as a feed stock for the production of titanium dioxide

pigment. Natural rutile is becoming scarcer and consequently more costly and alternative methods that use ilmenite are being favored. Ilmenite concentrates have relatively low titanium content (usually about 50% titanium dioxide compared to about 96% in the case of rutile) but have Fe as their major impurity and thus pose problems for pigment production. Nevertheless, ilmenite has been used as an alternate feed material for production of pigment through chemical routes (Samala et al., 2009).

Commercial production of  $\text{TiO}_2$  pigment uses either the sulfate process or chloride process. In the former process, relatively low grade feedstock can be used, but it gives a higher volume of waste product that requires proper treatment and disposal. It is more preferable to use the chloride process which utilizes a feedstock of high  $\text{TiO}_2$  grade as natural rutile mineral (94-98%  $\text{TiO}_2$ ), synthetic rutile (92-95%  $\text{TiO}_2$ ), anatase (90-95%  $\text{TiO}_2$ ), leucosene (>68%  $\text{TiO}_2$ ), or titanium slag (80-90%  $\text{TiO}_2$ ) as raw materials (Samala et al., 2009; El-Hazek et al., 2007; Lasheen, 2008, Zhang and Nicol, 2010; Nayl et al., 2009). The chloride process offers several advantages such as the yield of high quality products, a more eco-friendly process and the generation of a smaller amount of waste products (Zhang and Nicol, 2010).

The shortage of natural rutile and the need for higher grade titanium feedstocks has encouraged ilmenite upgrading by removing iron oxide and other impurities from the grain lattice, thus converting ilmenite into synthetic rutile for the chlorination process (El-Hazek et al., 2007; Mahmoud et al. 2004; Lasheen, 2009). There are several processes for the production of synthetic rutile from ilmenite and some of them were commercially applied. The most important of these processes are (Lasheen, 2004; El-Hazek et al., 2007; Mahmoud et al. 2004; Lasheen, 2009; Chun et al., 2008; Zhang and Nicol, 2009; Sasikumar et al. 2007; Kamala, 2006) given below.

(1) Smelting processes, where the iron part of the ilmenite is reduced and melted to separate iron from titanium. The titanium slag is then leached with sulfuric acid or with hydrochloric acid at elevated temperatures.

(2) Reduction of ilmenite to convert ferric iron partially to the ferrous form or completely to the metallic iron form followed by acid leaching.

(3) Reduction of the iron content of the ilmenite to the metallic iron followed by corrosion with oxygen and ammonium chloride.

(4) Oxidation and reduction of ilmenite followed by hydrochloric acid leaching (MURSO process).

(5) Roasting and magnetic separation followed by hydrochloric acid leaching (ERMS process).

In the first method, ilmenite ore can be smelted in the presence of carbonaceous reducing agents in an electric furnace where good quality pig iron is obtained together with titanium-rich slag which is chemically and mineralogically different than ilmenite (Nayl et al., 2009). In spite of high power consumption, smelting of ilmenite or titanium-bearing ore has several advantages over the synthetic rutile route by chemical process. The major advantages of the slag technology are: high titanium content, low waste generation, being suitable for sulphate and chloride processes, low chemicals

cost and converting the iron oxide as part of the concentrate to value-added iron metal (Kamala, 2006). The titanium slag produced from this process represents an important feedstock for the manufacture of titanium dioxide pigment by the sulphate process. In the case of the chloride process, the titanium-rich slag is further acid-leached by a hydrometallurgical process. As a leachant, hydrochloric acid is preferred to other acids because it allows comparatively easier recovery of the useful free acid from its waste solution. In addition, the recovery of a number of metal ions by liquid–liquid extraction from hydrochloric acid solutions is considerably easier than that from sulphuric acid solutions (Olanipekun, 1999).

On an industrial scale, the metallurgical complex of QIT-Fer & Titane Inc. (formerly named Quebec Iron & Titanium and later referred to the text as QIT), located in Sorel Tracy, Province of Quebec, Canada, is one of the largest producers of synthetic rutile by this method. In the acid-leaching plant of this company hydrochloric acid is used as a leachant (Guéguina and Cardarelli, 2007).



Fig. 1. Location map of Qara-aghaj deposit

In this work, the acid leaching process was studied using titanium slag obtained from the Qara-aghaj ilmenite concentrate. Beside Kahnoj beach sands in south of Iran, Qara-aghaj hard rock deposit which is located 36 km from Euromieh in Azarbayegan province, northwest of Iran (Fig. 1) has recently been explored as another titanium resource (Irannajad, 2002; Mehdilo, 2003; Irannajad, 1990). In the Qara-aghaj ore, titanium occurs mainly in the form of ilmenite partly as separated grains, exsolved lamellae in magnetite and dissemination in silicate minerals. The maximum amount of  $TiO_2$  in ilmenite lattice is 48% (lower than the theoretical amount, 52.6%). The  $MgO$  and  $MnO$  contents of ilmenite are 0.74–1.48% and 0.38–2.4%, respectively which are relatively high. Magnetite is another valuable mineral in the ore. Pyroxene, olivine, hornblende and some plagioclase are the most important gangue minerals of the ore

(Mehdilo and Irannajad, 2010). Based on mineral processing tests an ilmenite concentrate by grading of 44.5%  $\text{TiO}_2$  has been obtained by a combination of the gravity-magnetic separators (Mehdilo, 2003; Irannajad and Mehdilo, 2007; Irannajad and Mehdilo, 2004) and then converted to titanium slag by the carbothermic reduction smelting process in a DC electric arc furnace (Mehdilo and Irannajad 2006; Mehdilo et al., 2006; Irannajad and Montajam, 2005). In the present study, the iron removing from the produced titanium slag was investigated by sulfuric and hydrochloric acid leaching tests and the obtained titanium dioxide concentrate was characterized as a final product of both acids leaching.

## 2. Materials and procedure

### 2.1. Materials

The titanium rich slag prepared from ilmenite concentrate of Qara-aghaj hard rock titanium ore (Fig. 2) was used as the raw material. The chemical composition of the ore sample and ilmenite concentrate is presented in Table 1. Merck sulfuric and hydrochloric acids were employed for leaching tests. The chemical and phase composition of both raw material and the final residue of the leaching tests were carried out by X-ray fluorescence (Philips X Unique2) and X-ray diffraction (Philips X'Pert). The same materials were examined by scanning electron microscopy (Philips, model: XL30) equipped with EDS for morphological and spot analysis studies.

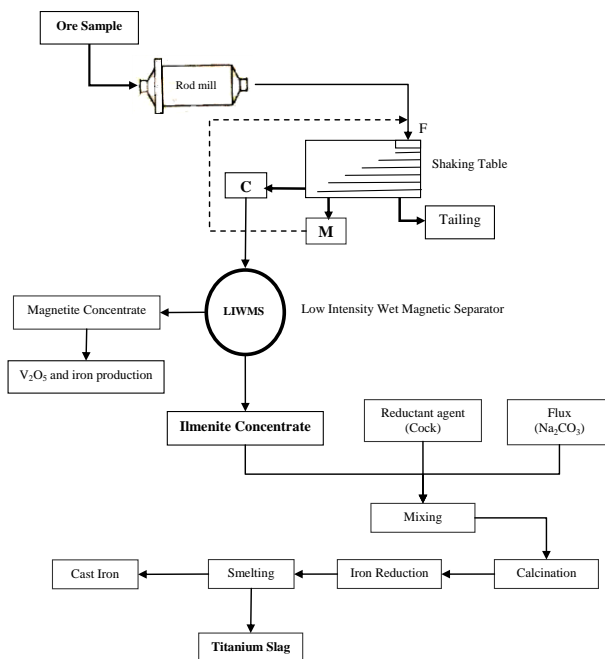


Fig. 2. Schematic diagram of ilmenite concentrate and titanium slag preparation from ore sample

Table 1. Chemical composition of ore sample and ilmenite concentrate

Composition, %	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	P <sub>2</sub> O <sub>5</sub>	V <sub>2</sub> O <sub>5</sub>	Na <sub>2</sub> O	Total
Ore sample	9.0	34.4	0.41	15.0	27.4	3.1	5.9	2.9	0.14	0.25	98.5
Concentrate	44.5	46.1	0.83	3.76	2.57	0.58	0.61	0.52	0.26	-	99.73

## 2.2. Procedure

The obtained titanium slag was crushed and ground below 100  $\mu\text{m}$  and a representative sample was prepared. The samples with 100 g weight were used in the leaching tests by sulfuric and hydrochloric acids. After leaching and filtration, the residue as titanium dioxide concentrate was analyzed by XRF and the recoveries of TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> were calculated based on feed and residue weight and grades. Finally, the product was characterized by analytical methods such as X-ray diffraction (XRD) and X-ray fluorescence (XRF), light microscopy and scanning electron microscopy.

## 3. Results and discussion

### 3.1. Slag characterization

The chemical and phase composition of the titanium slag are shown in Table 2 and Fig. 3, respectively. However, after slagging, the Fe<sub>2</sub>O<sub>3</sub> content of the slag has been reduced to 7.8% but it is the most important of all impurities which should be removed. Fe<sub>2</sub>O<sub>3</sub> is mainly in the form of iron metal which could not be separated from the slag. The other impurities are mainly light elements such as Si, Mg, Al, Ca, Mn and K which are concentrated in the slag product. SiO<sub>2</sub> and some of MgO, CaO and Al<sub>2</sub>O<sub>3</sub> are sourced from silicate minerals. MnO and some of MgO, CaO and Al<sub>2</sub>O<sub>3</sub> are related to impurities in ilmenite lattice. The high content of K<sub>2</sub>O in the slag is due to potassium carbonate consumption as flux in the slagging process. The XRD pattern of the slag shows that rutile is the most important phase in the sample. The other titanium containing phases are Ti<sub>2</sub>O<sub>3</sub>, Ti<sub>2</sub>O<sub>5</sub>, anatase and negligible amount of pseudorutile (Fe<sub>2</sub>Ti<sub>3</sub>O<sub>9</sub>). The impure phase of the slag was detected as FeO. The BSE images of the produced slag are shown in Fig. 4 and different parts or phases were chemically analyzed by EDX (Table 3). These results indicate that some parts of the slag have been transformed to titanium rich phases with spherical morphology (Fig. 4–b) containing up to 85% TiO<sub>2</sub> content (Table 3). The other titanium containing phase is probably pseudorutile which has been found by the EDX spot analysis (Table 3). The analysis of light particles shows that they are pig iron. These iron particles can be removed by the acid leaching process.

Table 2. The chemical composition of titanium slag

Composition	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	Total
%	72.7	7.8	0.97	3.21	5.24	2.90	0.30	1.08	0.20	4.22	99.28

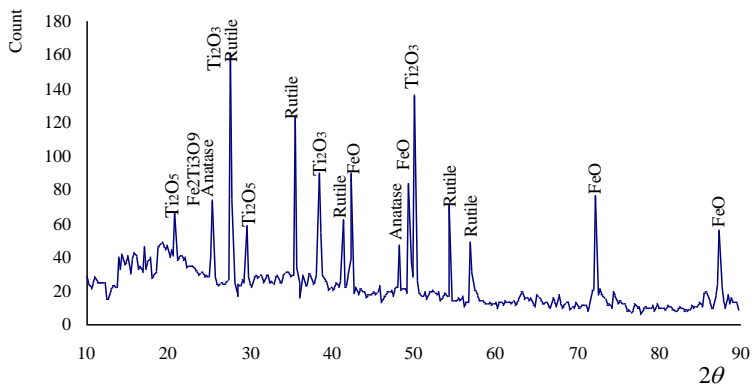
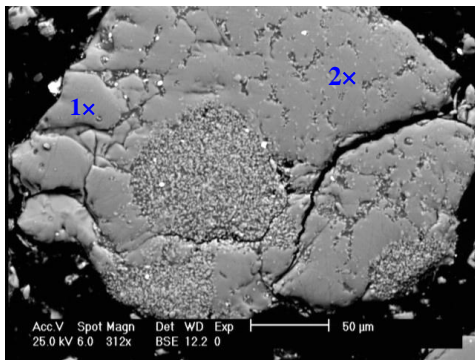
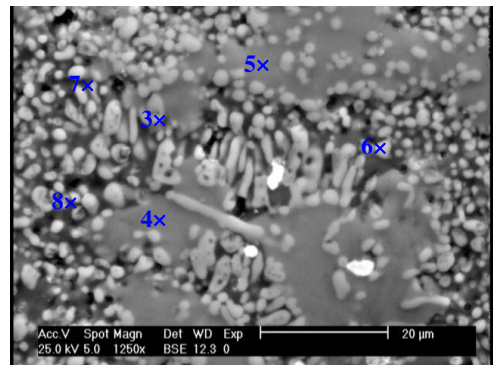


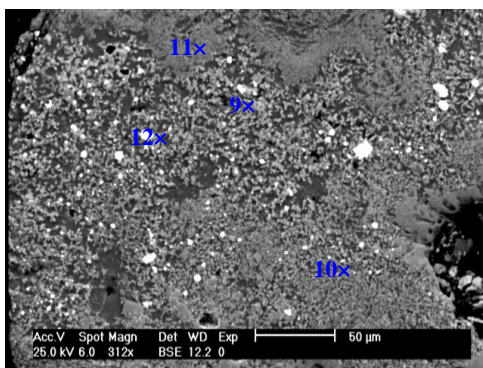
Fig. 3. XRD pattern of titanium slag used as raw material



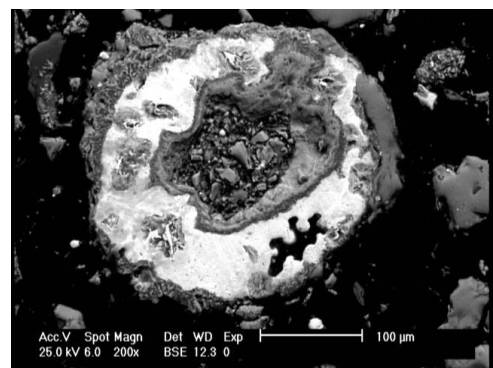
(a) BSE image of crushed titanium slag. Partial transformation of slag to titanium reached phases



(b) titanium rich phases of image a) with higher magnification



(c) presence of pig iron particles inside titanium slag



(d) larger grain of pig iron surrounding a titanium slag grain

Fig. 4. The BSE images of titanium slag

Table 3. Selected EDX analyses of titanium slag

Spot No.	Analyzed Points	Composition (%)										
		TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	V <sub>2</sub> O <sub>5</sub>	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	Total
1	compact parts of slag (spot 1- Fig 3a)	69.42	0.61	0.57	-	0.69	0.16	17.91	0.62	9.84	0.16	99.98
2	compact parts of slag (spot 2- Fig 3a)	70.45	0.44	1.18	0.20	0.75	2.40	4.55	13.40	6.32	0.27	99.96
3	pseudorutile (Fig 3b)	75.46	0.49	0.44	-	0.55	1.95	10.50	4.02	6.22	0.33	99.96
4	pseudorutile (Fig 3b)	77.72	0.55	1.17	-	0.44	2.24	0.97	12.66	3.92	0.32	99.98
5	pseudorutile (Fig 3b)	77.61	0.31	0.94	-	2.03	1.05	8.92	5.09	3.78	0.22	99.95
6		89.24	0.51	0.28	-	0.77	0.72	2.63	0.61	4.95	0.26	99.96
7	spherical particles (Rutile or Anatase)	90.43	0.78	1.0	0.25	1.05	-	3.96	0.74	1.15	0.62	99.97
8	(spots 6, 7, 8- Fig 3b)	88.32	0.71	0.58	0.07	0.51	0.14	5.95	0.87	2.43	0.38	99.96
9	(spots 9, 10- Fig 3c)	88.67	0.81	0.44	0	0.60	1.51	0.85	4.64	2.22	0.22	99.96
10		85.71	0.8	0.67	-	0.73	0.37	7.27	0.28	3.70	0.43	99.96
11	compact parts of slag (Fig 3c)	65.69	4.13	0.53	-	-	3.94	8.24	8.94	8.14	0.34	99.95
12	light grains- iron (Fig 3c)	4.60	90.53	0.33	0.17	0.85	0.13	0.60	1.11	0.95	0.71	99.98

### 3.2. Leaching tests

The leaching tests to remove iron were carried out on a titanium slag (Fig. 2) assaying 72.7% TiO<sub>2</sub> and 7.8% Fe<sub>2</sub>O<sub>3</sub> using sulfuric and hydrochloric acid using different conditions. The parameters including solid/liquid (S/L) ratio, particle size, leaching time and acid concentration were investigated. Based on literature all leaching tests were carried out at a constant temperature of 95°C.

#### 3.2.1. Effect of solid/liquid (S/L) ratio

Several leaching experiments were performed using sulfuric and hydrochloric acid, with S/L ratios of 1:1, 1:2, 1:4, 1:6 and 1:8. In these experiments, leaching was performed on the -100 µm feed material applying 60 minutes of agitation and using an acid concentration of 4%. From the results plotted in Fig. 5a and 5b, it is clearly evident that with increasing S/L ratio, the total iron leaching efficiency increased but its increasing after S/L=1:4 occurs slowly. By decreasing the S/L ratio or increasing acid consumption the titanium dissolution also increases. Therefore for decreasing acid consumption and titanium dissolution the S/L=1:4 is selected as optimum amount for the next experiments.

#### 3.2.2. Effect of particle size

For investigation of the effect of particle size on leaching efficiency different samples were ground below 200, 150, 100 and 75 µm and were examined using both acids with the S/L ratio 1:4 applying 60 minutes of agitation and acid concentration of 4%. By increasing particle size the leaching efficiency is decreased as illustrated in Fig. 4. With -100 µm particle size, 81.7% of Fe<sub>2</sub>O<sub>3</sub> and 9% of the TiO<sub>2</sub> was extracted

using sulfuric acid while in hydrochloric acid solution the leaching efficiency of  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$  were 88.3 and 3.9%, respectively. However, the iron recovery is increased for sample with  $-75 \mu\text{m}$  particle size but the titanium dissolution is also increased in this size fraction. So, in order to decrease titanium losses in solution and reduce grinding energy consumption, the  $-100 \mu\text{m}$  size fraction was used as the optimum particle size in the next experiments.

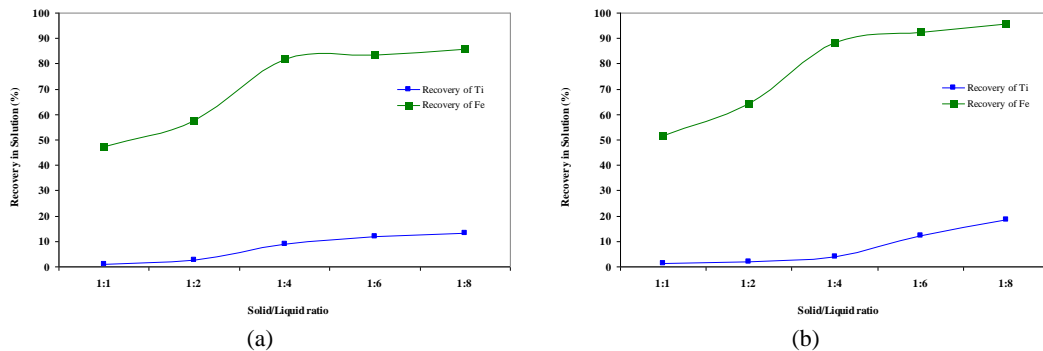


Fig. 5. Effect of S/L ratio on leaching (60 minutes, 4% acid concentration,  $95^\circ\text{C}$ ) efficiency; (a) recovery of  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  in solution using sulfuric acid, and (b) recovery of  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  in solution using hydrochloric acid

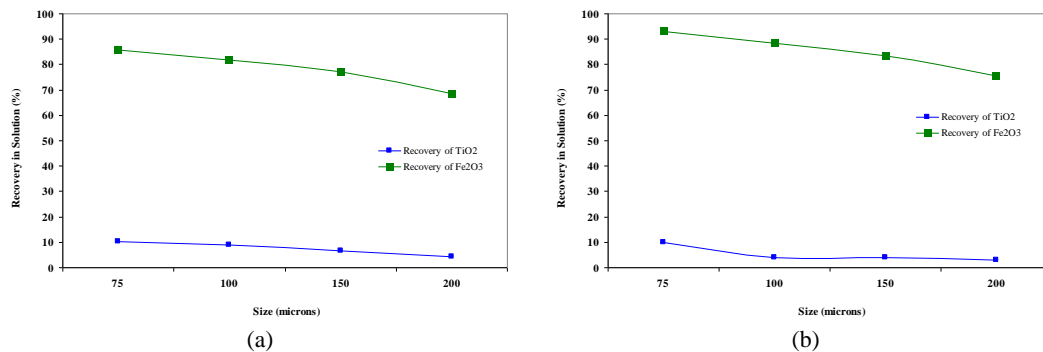


Fig. 6. Effect of particle size on leaching (60 minutes, 4% acid concentration,  $95^\circ\text{C}$ ) efficiency; (a) recovery of  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  in solution using sulfuric acid, and (b) recovery of  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  in solution using hydrochloric acid

### 3.2.3. Effect of leaching time

The effect of time on the leaching efficiency of titanium slag was examined between 0 and 240 minutes using the S/L ratio of 1:4 at  $95^\circ\text{C}$ . The results shown in Fig. 7a and 7b indicate that using sulfuric acid the residue obtained within 120 minutes has a maximum  $\text{TiO}_2$  content (86.7%) and recovery (92%). At this time about 82% of  $\text{Fe}_2\text{O}_3$  is brought into solution. With time, by increasing the iron dissolution



efficiency, the recovery of  $\text{TiO}_2$  in the residue as titanium dioxide concentrate decreases from 92% to 88.5% by increasing the leaching time from 0 to 240 minutes. In the case of hydrochloric acid, the results presented in Fig. 8a and 8b indicate that the  $\text{TiO}_2$  content in the residue (synthetic rutile) is greater than that produced by sulfuric acid leaching. This is due to higher solubility of iron in hydrochloric acid. With time, with increasing  $\text{TiO}_2$  content in the residue from 88.7 to 91.3% the  $\text{Fe}_2\text{O}_3$  content is decreased from 1.51% to 0.42%. The increase in leaching time results in the increase of titanium dissolution and decrease of titanium recovery in produced synthetic rutile. At 120 minutes leaching time, the  $\text{Fe}_2\text{O}_3$  dissolution efficiency is about 94% and 95% of  $\text{TiO}_2$  is recovered in the leaching residue as titanium dioxide concentrate.

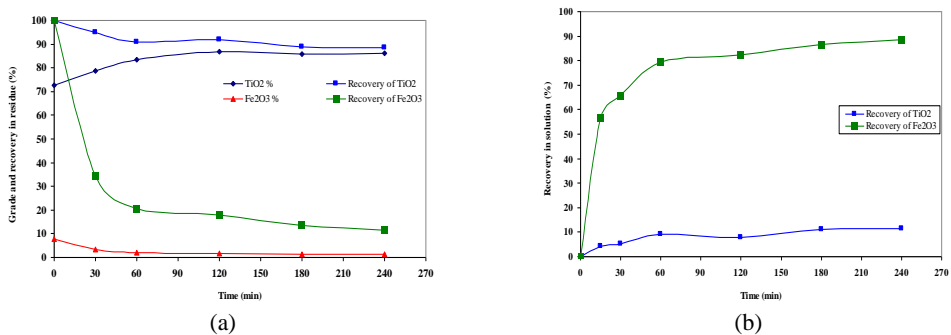


Fig. 7. (a) Effect of time on  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  contents and on their recoveries in residue, (b) Effect of time on recovery of  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  in solution during sulfuric acid leaching (S/L ratio 1:4, 95°C)

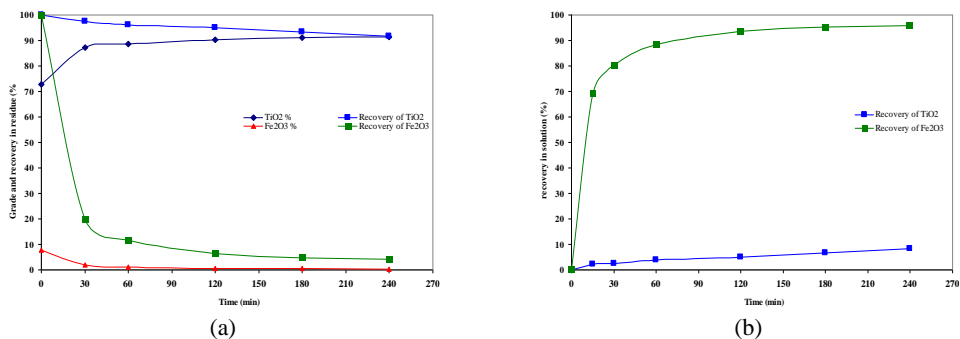


Fig. 8. (a) Effect of time on  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  contents and on their recoveries in residue, (b) Effect of time on recovery of  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  in solution during hydrochloric acid leaching (S/L ratio 1:4, 95°C)

### 3.2.4. Effect of acid concentration

Several experiments were carried out using sulfuric and hydrochloric acid concentrations varying from 4 to 18% at 2 h leaching time and 95°C. From the results plotted in Figs. 9a and 9b it is clearly evident that, with increasing sulfuric acid

concentration, the total iron leaching efficiency increases steadily from 82.2% to about 90.4%. The  $Fe_2O_3$  content of residue is reduced from 3.8% in 4% acid concentration to 1.85% in 18% acid concentration. The variation of the  $TiO_2$  content in the residue at different acid concentrations is very negligible but its recovery is reduced from 92% to 88.2% due to the increase in titanium dissolution by increasing acid concentration. The results in Fig 10a and 10b revealed that by increasing hydrochloric acid concentration, more impurities are dissolved and the  $TiO_2$  content in the residue is increased but the recovery is decreased due to high titanium losses in the solution. At 4% HCl, the recovery of  $Fe_2O_3$  in the synthetic residue is 10.3% and this value dropped to 4.8% by increasing the HCl concentration to 8%. When the HCl concentration reaches 18%, the recovery of  $Fe_2O_3$  is 3.5% and a higher increase in HCl concentration does not have a greater effect on the iron removal. Thus, 8% is the optimum HCl concentration where up to 95% of  $Fe_2O_3$  is removed from the titanium dioxide concentrate and the  $TiO_2$  and  $Fe_2O_3$  contents are 91% and 0.61%, respectively.

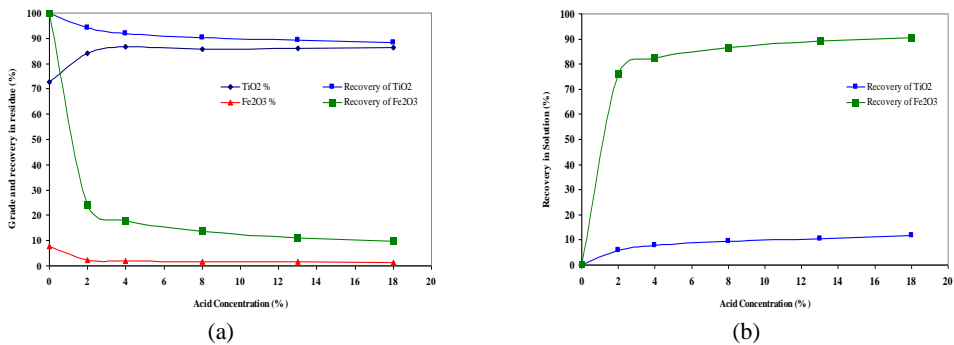


Fig. 9. (a) Effect of acid concentration on  $TiO_2$  and  $Fe_2O_3$  contents and their recoveries in residue, (b) effect of acid concentration on recovery of  $TiO_2$  and  $Fe_2O_3$  in solution during sulfuric acid leaching (S/L ratio 1:4, 95°C, 120 minutes)

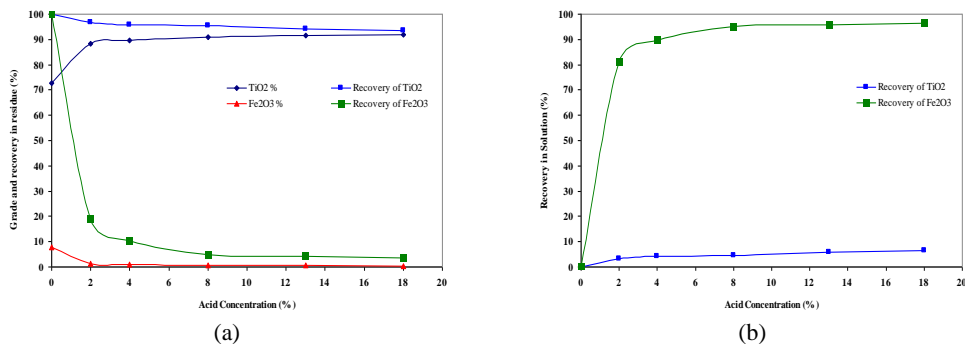


Fig. 10. (a) Effect of acid concentration on  $TiO_2$  and  $Fe_2O_3$  contents and their recoveries in residue, (b) effect of acid concentration on recovery of  $TiO_2$  and  $Fe_2O_3$  in solution during hydrochloric acid leaching (S/L ratio 1:4, 95°C, 120 minutes)

### 3.3. Characterization of titanium dioxide concentrate

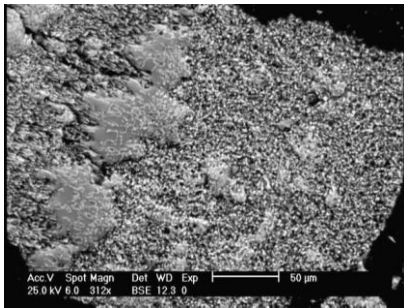
The produced titanium dioxide concentrate at the optimum conditions was washed and dried. In this concentrate rutile is the dominant phase but  $Ti_2O_3$ , anatase and iron oxide phases were also found. The chemical analysis of the dried product obtained under optimum conditions is presented in Table 4. The product of HCl leaching contains about 91%  $TiO_2$  and only 0.6% iron as  $Fe_2O_3$  while their content in the titanium dioxide concentrate obtained by  $H_2SO_4$  leaching are 86.8 and 1.87, respectively. The contents of most impurities in the product of  $H_2SO_4$  leaching are a little greater than the HCl leaching product. The BSE images of titanium dioxide concentrate produced by sulfuric and hydrochloric acid are compared in Fig. 11. These images indicate that the texture and morphology of the product obtained by HCl and  $H_2SO_4$  are very different. The morphology of titanium dioxide produced by HCl leaching is more granular than that prepared by  $H_2SO_4$  leaching (Fig. 11a and 11b). In the product of HCl leaching, the grains are mostly elongated with a clear and sharp boundary while the product of the  $H_2SO_4$  process has grains with less elongation and unclear boundaries (Fig. 11c and 11d). Most of the particles produced by HCl leaching are needle-like crystals but the acicular texture is observed scarcely in the product of  $H_2SO_4$  leaching (Fig. 11e and 11f). The particle size of the titanium dioxide concentrates produced by  $H_2SO_4$  and HCl leaching was also determined by the image analysis method using a Clemex software. The cumulative percent of particles as a function of circular and spherical diameter is shown in Fig. 12 and 13. The information about size distribution of both products is presented in Table 6. The results indicate that the mean size of particles obtained from HCl leaching is a little smaller than that produced with  $H_2SO_4$  leaching. Some of the particles shown in Fig. 11 were analyzed by EDX (Table 5). The chemical compositions of titanium dioxide particles produced by different acids leaching are closely similar. The maximum content of  $TiO_2$  is 93% and the minimum content of iron as  $Fe_2O_3$  is 0.48%. Among the coloring metals, the content of  $V_2O_5$  and  $Cr_2O_3$  in the produced material does not exceed 0.12% and 0.3%, respectively but the quantity of  $MnO_2$  sourced from ilmenite lattice varies from 0.88 to 1.31% and could negatively affect the chlorination process. The content of other impurities ( $MgO$ ,  $SiO_2$ ,  $Al_2O_3$ ,  $CaO$  and  $P_2O_5$ ) is relatively high. Silica is not a major problem in the chloride process and could be removed with alkaline leaching (Nayl et al., 2009) but other impurities ( $MgO$ ,  $Al_2O_3$ ,  $CaO$  and  $P_2O_5$ ) are the chlorine consuming components and should be removed before using this process.

Table 4. The chemical composition of produced titanium dioxide

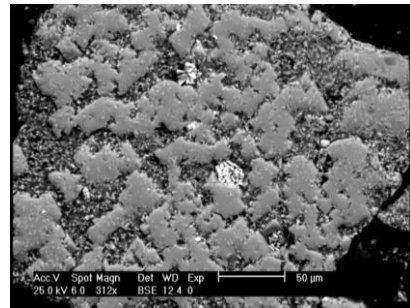
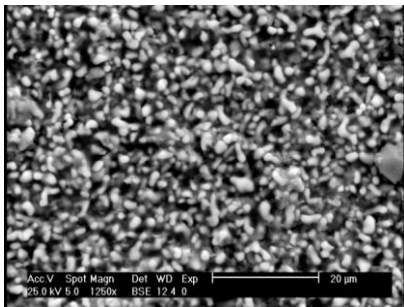
%	$TiO_2$	$Fe_2O_3$	MnO	MgO	$SiO_2$	$Al_2O_3$	$V_2O_5$	CaO	$K_2O$	$SO_3$	Cl	Total
$H_2SO_4$	86.8	1.87	0.57	2.79	4.6	1.02	0.22	1.04	0.21	0.51	-	99.63
HCl	91.0	0.61	0.62	2.11	3.28	0.86	0.18	0.58	0.09	-	0.38	99.51

Table 5. Selected EDX spot analysis of synthetic rutile prepared by sulfuric and hydrochloric acids leaching

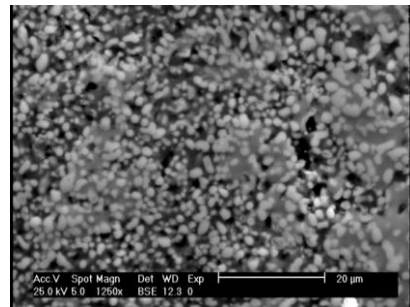
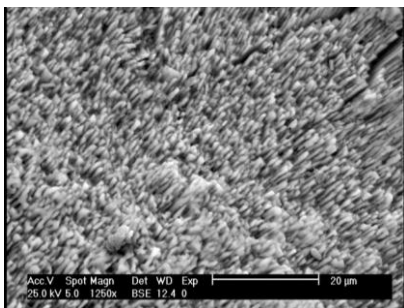
Product	Composition (%)										
	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	V <sub>2</sub> O <sub>5</sub>	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	Total
Synthetic rutile produced by H <sub>2</sub> SO <sub>4</sub> leaching	88.60	0.78	1.31	-	0.84	0.41	4.07	2.55	1.14	0.22	99.92
	91.42	0.68	1.14	-	0.39	0.11	4.23	0.95	0.90	0.14	99.96
Synthetic rutile produced by HCl leaching	92.23	0.58	0.88	-	0.47	0.61	2.08	1.89	0.77	0.27	99.85
	89.94	0.61	1.16	-	0.69	0.49	4.86	0.92	1.0	0.30	99.94
	93.01	0.48	0.96	0.12	0.13	0.45	3.05	0.58	0.90	0.22	99.99



(a) Synthetic rutile produced by HCl leaching

(a) Synthetic rutile produced by H<sub>2</sub>SO<sub>4</sub> leaching

(c) Synthetic rutile produced by HCl leaching

(d) Synthetic rutile produced by H<sub>2</sub>SO<sub>4</sub> leaching

(e) Synthetic rutile produced by HCl leaching

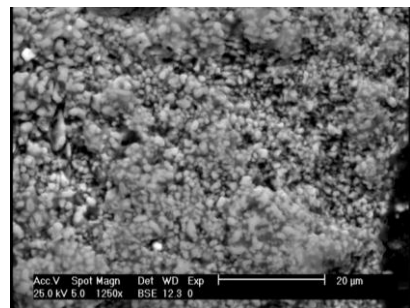
(f) Synthetic rutile produced by H<sub>2</sub>SO<sub>4</sub> leaching

Fig. 11. Textural and morphological comparison of synthetic rutile particles prepared by sulfuric and hydrochloric acids leaching at the optimum conditions

Table 6. Information on size distribution of particles produced by H<sub>2</sub>SO<sub>4</sub> and HCl leaching

Leaching solution	Diameter	Minimum size (μm)	Maximum size (μm)	Mean size (μm)	D80 (μm)	Standard Deviation (μm)
H <sub>2</sub> SO <sub>4</sub>	Circular	0.2	3.6	1.47	1.95	0.577
	Spherical	0.2	4.4	1.8	2.4	0.707
HCl	Circular	0.1	5.2	1.2	1.95	0.849
	Spherical	0.2	6.3	1.47	2.31	1.04

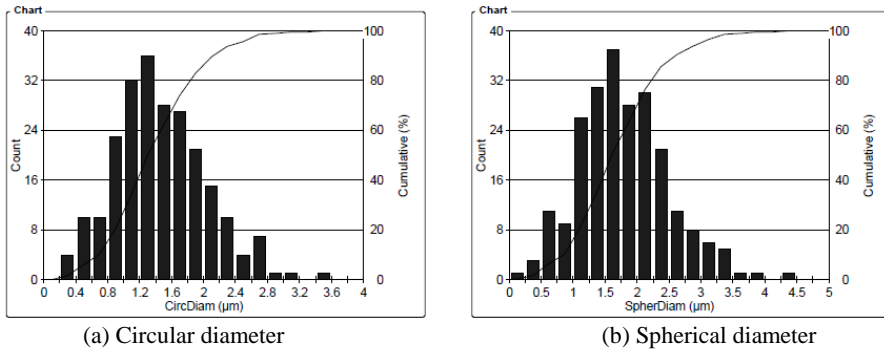
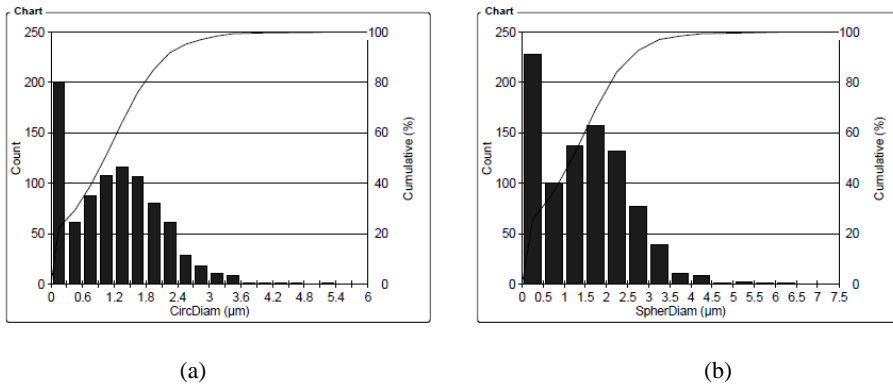
Fig. 12. Size distribution of titanium dioxide particles produced by H<sub>2</sub>SO<sub>4</sub> leaching

Fig. 13. Size distribution of titanium dioxide particles produced by HCl leaching; (a) circular diameter, (b) spherical diameter

#### 4. Conclusion

The leaching of titanium slag by both sulfuric and hydrochloric acid solutions at 95°C using an S/L ratio of 1:4 revealed that with a 2-hour leaching and 8% acid concentration, the Fe<sub>2</sub>O<sub>3</sub> content in the residue is reduced to 1.87% and 0.61%, respectively. The increase in the leaching time and acid concentration does not have a great effect on iron removal efficiency. Under mentioned conditions, by using sulfuric

acid, the production of titanium dioxide concentrate containing more than 87%  $\text{TiO}_2$  is not possible, while with hydrochloric acid as leachant, a product can be prepared containing up to 91%  $\text{TiO}_2$ . In addition to higher titanium purity and lower content of iron, the specific texture and morphology of titanium dioxide concentrate results in the hydrochloric acid being preferred. An easy recovery of hydrochloric acid from waste solutions is another profit of this reagent for upgrading titanium slag in order to produce suitable raw material for the chloride process. However, a high content of some impurities such as  $\text{MgO}$  and  $\text{CaO}$  may result in difficulties in chloride process and should be eliminated. More detailed investigation to remove these impurities is being performed by the authors.

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