Korean J. Chem. Eng., **30**(1), 123-130 (2013)
DOI: 10.1007/s11814-012-0150-9

Preliminary investigations on alkali leaching kinetics of red sediment ilmenite slag

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(*Received 13 December 2011 • accepted 5 July 2012*)

Abstract−The present paper deals with soda ash roasting of red sediment ilmenite (47.03% TiO_2) and leaching of obtained titanium rich slag with hydrochloric acid for preparation of synthetic rutile. The experimental conditions used for roasting are Na_2CO_3 to ilmenite ratio of 1 : 1 at 1,223 K for 4 h. This soda ash slag product is subjected to hydrochloric acid leaching to remove the iron content. The optimum conditions for leaching achieved are 6 M hydrochloric acid at 398 K for 2.5 h (10/1 liquid/solid mass ratio) at 100 rpm. Shrinking core model is found to be fit for the experimental results. The apparent activation energy is 37.9 kJ/mol. This process of soda ash roasting is one of the best processes for preparation of high purity synthetic rutile assaying about 97.21% TiO_2.

[The badlands topography of Basanputti village Ganjam Dist., Odisha, India possesses red sediments contains ilmenite (47.03% TiO_2). The process of roasting followed by leaching is an effective process for preparation of synthetic rutile. This product is identified by wet chemical method which gives orange red colour after dissolution in solution of sulfuric acid and ammonium sulfate].

Graphical abstract of on leaching kinetics of soda ash roasted slag of red sediment ilmenite

Key words: Ilmenite, Soda Ash Roasting, Titanium Slag, Leaching, Synthetic Rutile

INTRODUCTION

Several researchers have attempted the leaching of natural ilmenite mineral and calcining ilmenite minerals for the preparation of synthetic rutile. Similarly, leaching kinetics of ilmenite slag is well published in many journals. These studies are mostly on ilmenite of beach sand source. However, so far no attempt has been made on the ilmenite which occurs from the red sediments' resources. Red sediments are formed due to chemical weathering. In arid continental climates these sediments are in direct contact with the atmosphere leading to oxidation, thus giving the sediment a red colour containing heavy minerals of economic potential. It contains industrial minerals like ilmenite, sillimanite, zircon, rutile, monazite, and garnet in various proportions. A considerable amount of literature is available on the preparation of synthetic rutile from ilmenite (of beach sands); however, till now nobody has attempted to investigate the same using ilmenite from red sediment. The objective of the present investigation is to study the leaching kinetics of soda ash roasted ilmenite of red sediments.

The decomposition of ilmenite slag is a part of an improved process to obtain high purity titanium oxide [1]. The kinetics of the decomposition reaction is investigated in terms of the slag particle size, NH_4OH concentration, NH_4OH/slag mass ratio and reaction temperature. It is found that digestion of the ground ilmenite slag in 4 M NH_4OH at a temperature of 150 °C decomposed the slag with the formation of ammonium titanate ($(NH_4)_2TiO_3$), which is readily hydrolyzed in hot water to high purity anatase (≥99.8% TiO_2). Analysis of the decomposition reaction kinetics found that the reaction is chemically controlled with apparent activation energy of 27.8± 1.6 kJ mol^{-1}. Soda ash roasting is one of the processes for upgrading titania slag product of Rosetta ilmenite to a high grade titanium dioxide (TiO_2). The optimized conditions used a Na_2CO_3 to slag ratio of 0.55 : 1 at a roasting temperature of 850 °C for 0.5 hr duration. The impurities associated with the roasted slag are subjected to leaching with water and dilute hydrochloric acid solution leaving synthetic rutile (TiO_2) as insoluble residue. To improve the quality of the synthetic rutile, an alkaline leaching step is added to remove

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the excess silica present in the treated titania slag. This is an efficient method for the preparation of high purity synthetic rutile assaying about 97% TiO_2 [2].

Leaching of the alkali roasted product is undertaken for the removal of iron and alkali to produce synthetic rutile. The process is carried out in presence of oxalic and ascorbic acids. It is observed that in the presence of ascorbic acid the dissolution of iron oxide increases as Fe^{2+} ions are generated rapidly, which then form ferrous oxalate. Alkali ions are also removed in the process as the pH of the leaching medium is maintained below 4 [3]. Many studies have been carried out for upgrading ilmenite, which can be summarized into two categories: pyrometallurgy and hydrometallurgy methods. In the pyrometallurgy process, ilmenite is partially reduced by anthracite at elevated temperature to obtain a cast and a slag with high titanium [4]. In the hydrometallurgical method, sulfuric acid or hydrochloric acid are used as the leachant for upgrading ilmenite into synthetic rutile [5-10].

Certain elements in the ilmenite concentrate (chromium, vanadium, magnesium, manganese, etc.) may affect the quality of the pigments produced [11]. Commercially, pigment grade titanium dioxide is manufactured either through a sulfate or chlorine route. When used in paints, plastics or paper, it provides maximum whiteness and opacity. It gives the paint high hiding power, meaning the ability to mask or hide a substrate. It does this more effectively than any other white pigment. Today, titanium dioxide pigment is by far the most important material used by the paints and plastics industry for whiteness and opacity.

In view of this, the ilmenite concentrates recovered from red sediment deposits are considered as one of the important raw materials for the production of synthetic rutile via sulfate process or chlorine process. In the present study, an attempt is made for preparation of synthetic rutile using the leaching process from soda ash roasted ilmenite of red sediments of Basanputti village, Ganjam Dist., Odisha, India.

EXPERIMENTAL

1. Material

The samples were collected from red sediments of badlands topography of Basanputti village, Ganjam Dist, Odisha, India (Lat. 19°21′N and Long. 85°03′E) in the form of grid pattern up to the water table level during rainy season. These red sediment samples were thoroughly mixed and prepared as a composite sample. Initially, the representative red sediment sample was scrubbed and de-slimed by using hydrocyclone. The slimes were rejected and the sand was used for further studies to recover total heavy minerals. The ilmenite mineral used in the present study is a natural placer mineral recovered after beneficiation of red sediments.

2. Methods

2-1. Recovery of Ilmenite Mineral

The deslimed bulk sample is subjected to a series of rougher, cleaner and scavenger spiral concentrators (stage spirals) to recover total heavy minerals (THM). The THM is subjected to magnetic separation using wet high intensity magnetic separator (WHIMS) to recover ilmenite in magnetic fraction. Ilmenite and garnet both are magnetic and hence report in magnetic fraction of WHIMS. This magnetic fraction (mainly ilmenite and garnet) is subjected to high tension roll separator (HTS) to separate the conducting and nonconducting minerals. The conducting fraction contains mainly ilmenite and nonconducting fraction contains garnet, monazite etc. The schematic presentation for recovery of ilmenite from red sediments of badlands topography is shown in Fig. 1.

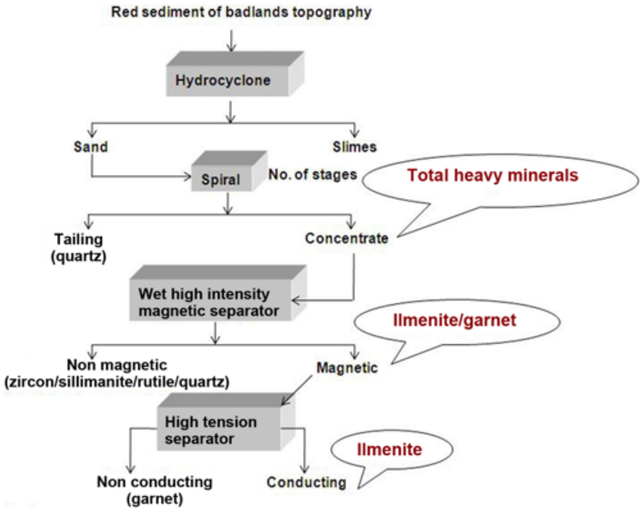

Fig. 1. Schematic presentation for recovery of ilmenite from red sediment of badlands topography.

The chemical analysis of ilmenite was done as follows. Initially, finely ground ilmenite was fused with potassium pyrosulfate, which was then dissolved in 50% H_2SO_4 and filtered. The bulk chemical composition of the ilmenite sample was determined by using ICP-OES (Model Perkin Elmer Optima 2100DV) as well as wet chemical analysis. The metallic contents were analyzed in the filtrate, and the SiO_2 content was analyzed in the insoluble residue by evaporation with HF. All reagents used for roasting, leaching and chemical analysis were of analytical grade and used without any purification.

2-2. Roasting Experiments

The ilmenite was roasted with varying stoichiometric ratio of sodium carbonate to ilmenite from 0.5 : 1 to 1.5 : 1 in a muffle furnace at 1,223 K for 4 h. All the roasting experiments were carried out batchwise by using nickel crucibles. In all the roasting experiments, a sample of 2.0 g was thoroughly roasted at 1,223 K for 4 h. The soda ash roasting of ilmenite involves two main steps [12].

(a) The decomposition of ilmenite to its constituent oxides at elevated temperatures according to Reaction (1):

$$FeO \cdot TiO_2 = FeO + TiO_2 \qquad (1)$$

(b) The decomposition of the constituent oxides, TiO_2 and FeO form complexes, namely Na_2TiO_3 (sodium titanate) and $Na_2Fe_2O_4$ (sodium ferrite), respectively as shown in Reactions (2 & 3).

$$TiO_2 + Na_2CO_3 = Na_2O \cdot TiO_2 + CO_2 \qquad (2)$$

$$2FeO + Na_2CO_3 + 0.5O_2 = Na_2Fe_2O_4 + CO_2 \qquad (3)$$

Out of the two complexes, while sodium ferrite is soluble in water, sodium titanate is insoluble. Thus, the overall roasting reaction at elevated temperature is given by Reaction (4):

$$3Na_2CO_3 + 2FeTiO_3 + 0.5O_2 = 2Na_2TiO_3 + Na_2Fe_2O_4 + 3CO_2 \qquad (4)$$

Besides, the presence of quartz in ilmenite forms sodium titanium silicate (Na_2TiSiO_5), which is shown in Reaction (5):

$$TiO_2+SiO_2+Na_2CO_3=Na_2TiSiO_5+CO_2 \qquad (5)$$

Experiments were performed by varying the concentrations of soda ash from 50% to 150% of the stoichiometric amounts in Reaction (4). The complexes formed were sufficiently porous to facilitate the subsequent leaching of the associated impurities.

2-3. Leaching Experiments

Each roasted sample was first leached with warm water for half an hour, where most of the water soluble compounds (sodium ferrite) were removed. Both the leachate and the residue were analyzed to determine the leachability of iron. After filtration, the water-leached sample was subjected to leaching with (i) a mixture of oxalic acid and ascorbic acid, (ii) hydrochloric acid, and finally (iii) sulfuric acid. Acid leaching of ilmenite was carried out using a 250 cm^3 necked glass reactor provided with a reflux condenser and a mechanical agitator with teflon-coated stirring rod. During leaching, the pulp was stirred at constant rate of 100 rpm. The leached product at the optimum conditions was separated, washed, dried at 383 K and calcined at 1,173 K. The filtrate was analyzed for iron, vanadium and titanium for calculating their leachability by using ICP-OES. Leached residues were also characterized by X-ray diffraction.

2-4. Chemical Analysis and Identification of Titanium Concentrate

A chemical analysis of the titanium concentrate was performed by adding 5 ml of sulfuric acid to 0.5 g of the calcined TiO_2 concentrate sample. The dissolution of TiO_2 in the solution was enhanced by adding some amount of ammonium sulfate, which acts as a boiling point modifier, thus increasing the boiling point of sulfuric acid. Ammonium sulfate does not interfere with any and only raises the temperature of the mixture to speed up the dissolution process. The mixture was heated gently until fumes of sulfuric acid appeared and then cooled. Cautiously, the solution was diluted to about 100 ml with water and filtered. When a few drops of hydrogen peroxide were added to 5 ml clear filtrate, an orange red color appeared immediately. This indicates that the TiO_2 concentrate contains more than 95% TiO_2 [13].

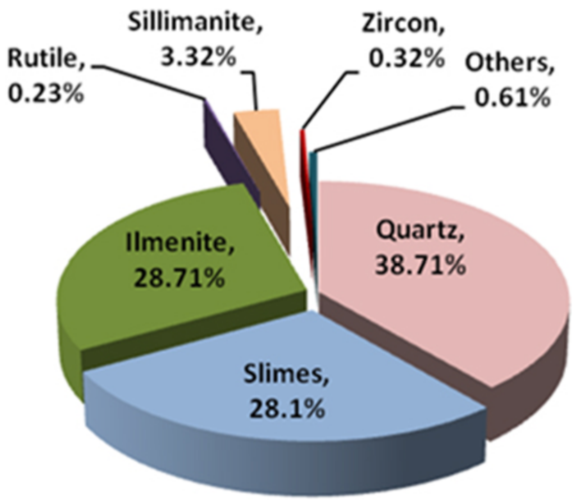

Fig. 2. Modal analysis of feed sample (red sediment of badlands topography).

RESULTS AND DISCUSSION

1. Recovery of Ilmenite

The red sediment sample of badlands topography of Basanputti village, Ganjam Dist., Odisha, India contains 33.2% of total heavy minerals, 28.1% slimes and 38.71% quartz. The mineralogical modal analysis of as-received feed sample is shown in Fig. 2, which indicates that the sample contains 28.71% of ilmenite followed by 3.32% sillimanite, 0.32% zircon, 0.23% rutile and 0.61% of other minerals.

2. Ilmenite Characterization

The detailed chemical analysis of the ilmenite sample is given in Table 1. The ilmenite contains 47.03% TiO_2, 30.13% FeO and 19.25% Fe_2O_3. This ilmenite is considered as low grade ilmenite as it contains only 47.03% TiO_2. It also contains 1.25% SiO_2 and 0.71% Al_2O_3 accompanying with other impurities such as V, Cr, Mn and Mg. The size analysis of ilmenite concentrate is given in Table 2. It indicates that the maximum percentage (40.4% to 48.4%) of ilmenite is present at size fraction of –210+100 microns. The physical properties of Ilmenite concentrate are given in Table 3. It shows that ilmenite has a bulk density of 2.9 g/cc, true density of 4.7 and porosity of 38.3. The d_{80} passing size value is found from the size analysis data, which is 188 μm.

The results of initial characterization of ilmenite involving X-ray

Table 1. Complete chemical analysis of the ilmenite

Component	Weight, %	Component	Weight, %
TiO_2	47.03	CaO	0.21
FeO	30.13	MnO	0.372
SiO_2	1.25	Cr_2O_3	0.083
Fe_2O_3	19.25	P_2O_5	0.032
Al_2O_3	0.71	V_2O_5	0.29
MgO	0.50	LOI	0.13

Table 2. Size analysis of ilmenite concentrate

Size, micron	Wt, %	Cumulative Wt, % passing
−420+300	-	100
−300+210	8.6	91.4
−210+150	40.4	51.0
−150+100	48.4	2.7
−150+75	2.0	1.70.7
−75+45	0.5	0.2
−45	0.2	-
Total	100.0	-

Table 3. Physical properties of ilmenite concentrate

Details	Basanputti
Bulk density, g/cc	2.9
True density:	4.7
Porosity, %	38.3
Angle of repose: degree	27.4
d_{80} Passing size, μm	188

diffraction (XRD) of powders and scanning electron microscopy (SEM) of transversal sections of particle are shown in Figs. 3 and 4. The XRD pattern of ilmenite revealed that it contains the maximum number of peaks of ilmenite accompanied with low amounts of quartz (Fig. 3). SEM image (Fig. 4) shows the development of a number of different micro features on the ilmenite grains. Deep cuts with smooth surface are seen in transversal section of ilmenite, which resulted from mechanical collision and later from solution activity. Hence, leaching can be possible due to the physical and chemical energy gradient, surface and sub-surface dissolution process, and post depositional digenetic modifications of ilmenite.

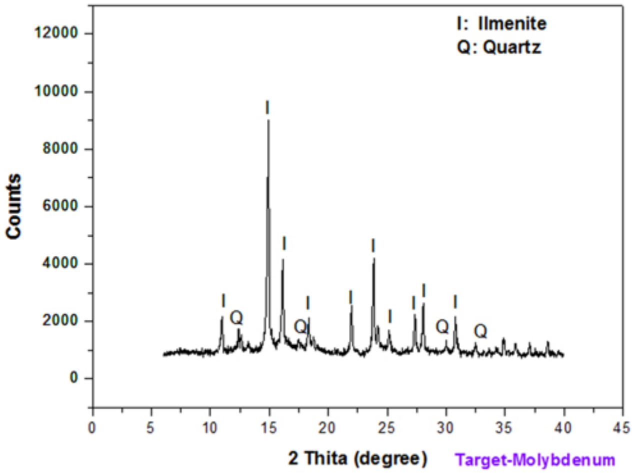

Fig. 3. XRD pattern of ilmenite concentrate.

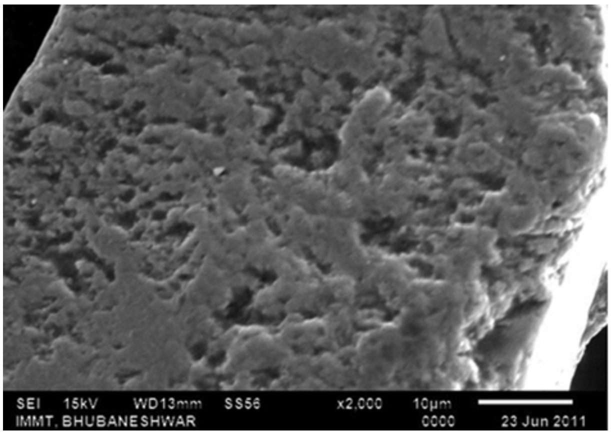

Fig. 4. SEM image of the transversal section of ilmenite.

3. Ilmenite Roasting

The total iron present in ilmenite is 36.7%. Fig. 5 shows the leachability of iron in various filtrates at 1,223 K for 4 h. Data shown in Fig. 5 reveals that the leachability of iron is increased at ratio Na_2CO_3 : ilmenite ratio from 0.5 : 1 to 1 : 1, i.e., from 5.5% to 15.7% referring to the total iron present in the sample. It is due to the conversion of different sodium titanates, namely, sodium titanate (Na_2TiO_3) and sodium titanium silicate (Na_2TiSiO_5). Hot water leaching removes considerable amounts of sodium ferrite. The leachability of iron is almost constant from 100% to 150% of the stoichiometric ratio of Na_2CO_3.

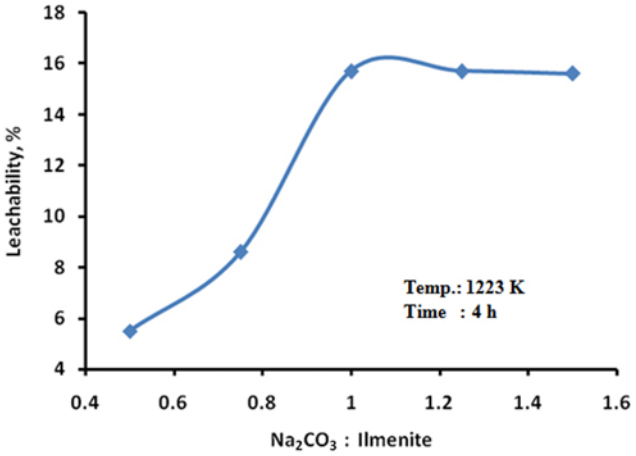

Fig. 5. Effect of Na_2CO_3/ilmenite ratio upon its decomposition efficiency with response to iron.

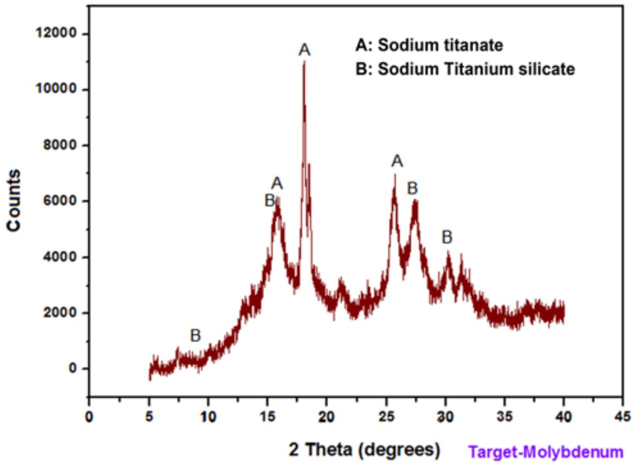

Fig. 6. X-ray diffraction pattern of soda ash roasted slag product.

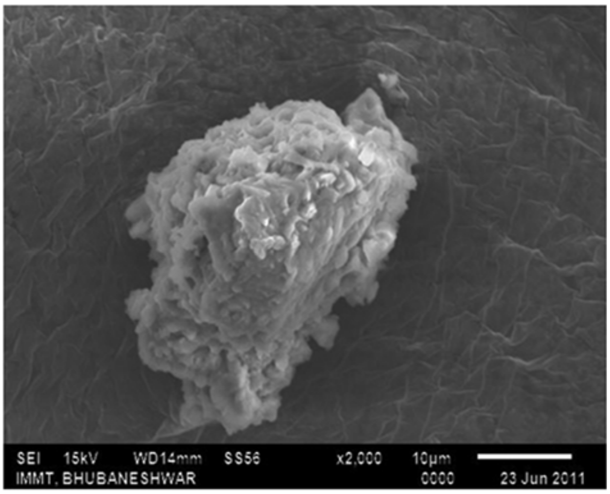

Fig. 7. Morphological features of soda ash roasted slag product of red sediment ilmenite (100% stoichiometric ratio).

The XRD pattern of soda ash roasted slag product at 100% stoichiometric ratio of Na_2CO_3 (after water leaching) is shown in Fig. 6, which clearly shows the peaks of sodium titanate and sodium tita-

Table 4. Results on the effect of mineral acids for leaching of iron
Conditions:
Material: Titanium rich slag
Pulp density: 1 : 10
Temperature: 373 K
Time: 1 h
Agitation: 100 rpm

Mineral acid	Concentration, M	Fe leached out, %
Oxalic acid+Ascorbic acid	$0.2+7.55*10^{-3}$	8.9
HCl	6	47.6
H_2SO_4	6	45.2

nium silicate. It also indicates that at 100% stoichiometric ratio of Na_2CO_3 sodium ferrite is almost removed. The residue after hot water leaching process contains 5.43% of total iron (determined by chemical analysis). Therefore, an acid leaching step is introduced to remove the remaining iron for preparation of synthetic rutile.

Fig. 7 represents the morphological features of 'soda ash roasted slag product' of red sediment ilmenite (100% stoichiometric ratio of sodium carbonate), which is showing a layer of sodium titanate on its outer surface, whereas the inner core contains untreated ilmenite.

4. Leaching Studies

Results of the leaching studies, carried out on soda ash roasted slag product by varying mineral acids, at different acid concentration, pulp density, time and temperature, are discussed in the following subsections.

4-1. Effect of Different Mineral Acids

The effect of different mineral acids on dissolution of iron from titanium-rich slag at constant pulp density, time, temperature and rpm is given in Table 4. It is clearly seen that the dissolution of iron from titanium rich slag is maximum with hydrochloric acid (47.6% of total iron removed) in comparison to other acids at 1 : 10 pulp density for 1 hour at 373 K and 100 rpm. As discussed earlier, Fig. 7 shows the morphological features of soda ash roasted slag product of red sediment, which contains some unreacted ilmenite at the inner core. Hence, during the reaction this unreacted ilmenite also reacted with hydrochloric acid. The reaction of sodium titanate (Na_2TiO_3) and unreacted ilmenite with hydrochloric acid could be described as follows:

$$Na_2TiO_3+FeO+6HCl \rightarrow FeCl_2+TiOCl_2+2NaCl+3H_2O \qquad (6)$$

The main parameters that influence the rate of this reaction are hydrochloric acid concentration, leaching time, temperature and pulp density (ilmenite to HCl ratio). At the same time, the associated vanadium and chromium are converted to alkali vanadate and chromate. By subsequent leaching procedures, it is possible to selectively leach out the alkali vanadate and chromate in water while most of the Fe, Al and Mn impurities were leached in hydrochloric acid solution.

4-2. Effect of Concentration of Hydrochloric Acid

The effect of HCl concentration on the leachability of soda ash roasted ilmenite is tested in the acid range 2 to 8 M using S/L ratio of 1/10 at 383 K and 2.5 h leaching duration. The result of this leaching study is shown in Fig. 8. It is clearly evident that, with increasing acid molarity, the total iron leachability increases steadily from 2 M to 6 M HCl concentration (13.3% to 84.1%, calculated as total iron), and beyond 6 M HCl solution leachability of iron is slightly affected, i.e., from 84.1% to 84.4%. On the other hand, with increase in molar concentration of HCl, the TiO_2 leaching efficiency is negligible as it varied from 0.2% to 1.2%. The dissolution efficiency of vanadium is increased from 5.2% to 98.6% with increase in acid concentration from 2 M to 8 M.

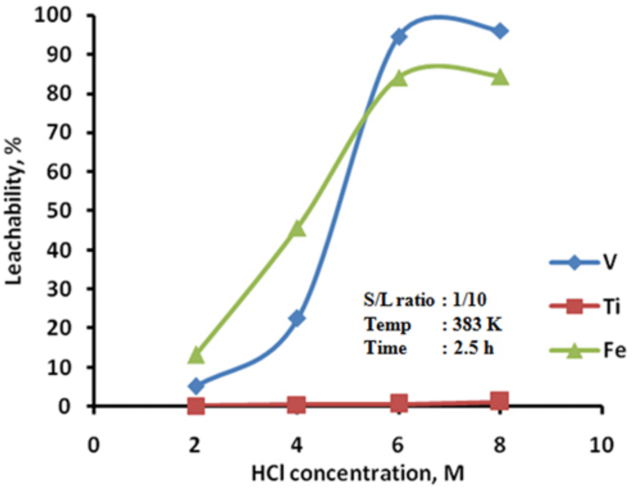

Fig. 8. Effect of HCl concentration on the leaching of soda ash roasted slag.

Iron leaching from ilmenite mineral grains was directly correlated with the molar acid concentration. An increase in acid concentration favors higher iron removal from the lattice of the mineral, and at acid molarities of 2 to 8 the process became more effective and rapid. It can be inferred that the co-dissolved titanyl chloride is hydrolyzed under these conditions of relatively high ilmenite/acid molar ratio and reprecipitates in the form of TiO_2. The characteristics of the residue products obtained from different leaching processes are shown in Fig. 9. It indicates that intensity of rutile peaks is increasing with increase in hydrochloric acid concentration from 4 M to 8 M. In this case of leaching studies, the optimum condition for leach-

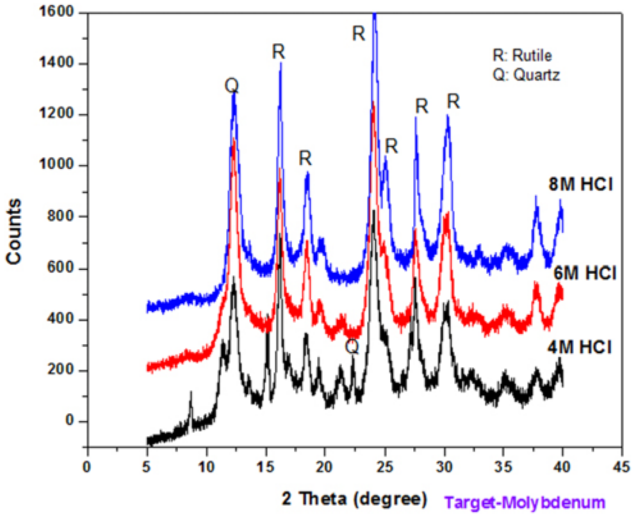

Fig. 9. XRD pattern of leached residues at 4 M, 6 M and 8 M hydrochloric acid.

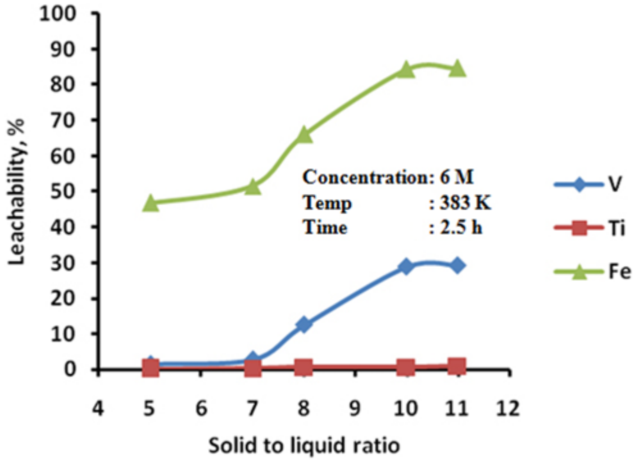

Fig. 10. Effect of solid/liquid ratio on the leaching of soda ash roasted ilmenite by using 6 M HCl.

ing of iron is selected at 6 M HCl.

4-3. Effect of Pulp Density

The effect of pulp density for the dissolution of sodium titanate is determined by using mass ratios solid/liquid varying from 1/5 to 1/11, while the other leaching conditions are fixed at 6 M hydrochloric acid in 383 K for 2.5 h. The results are shown in Fig. 10, which shows that the leachability of iron and vanadium is strongly dependent on the acid-to-residue ratio. As the mass ratio increases from 1/5 to 1/11, the recovery amount increases to about 84.7% iron and 29.2% vanadium. The TiO_2 leachability is negligible as it varied from 0.2% to 1.0%. In this case with increase in pulp density $TiOCl_2$ particles are formed and the same get hydrolyzed to $TiO_2 \cdot H_2O$ as the Ti(IV) concentration in solution increases. This would precipitate in the pores of leached particle or reported as fines in the leach solution.

4-4. Effect of Time and Temperature

The effect of time and temperature at 6 M concentration on dissolution of iron content from the soda ash roasted slag is shown in Fig. 11. It is seen that the dissolution of iron content is increasing with leaching reaction time as well as with temperature. As expected, the reaction is very fast upto 30 min, and beyond this the reaction is relatively slow. The dissolution of iron between 353-363 K is slow, whereas the rate of dissolution of iron is very fast at 373 K and is very much significant at 398 K. At higher temperature, leaching of iron content may be attributed to the swelling effect within the internal structure of the sample enabling iron ions to leach out further and/or activation of the surface of sample. The iron leaching from the sample is carried out at higher temperatures to increase the overall efficiency of a process as well as to avoid the side reaction. The dissolution of iron is maximum, i.e. 35.6%, at the optimum condition of temperature at 398 °K for 150 min.

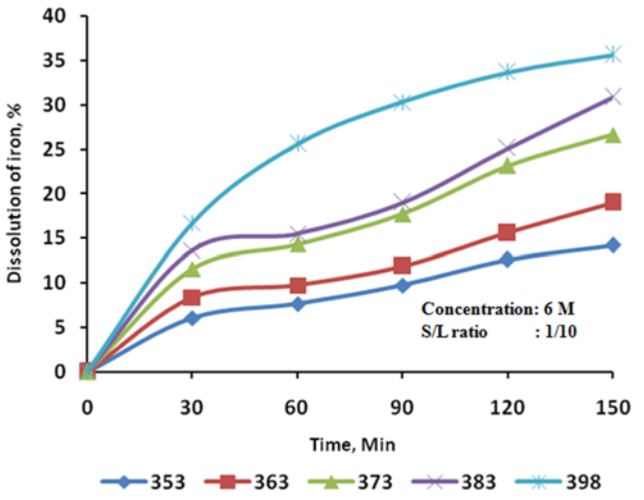

Fig. 11. Dissolution of iron from soda ash roasted slag at 6 M HCl.

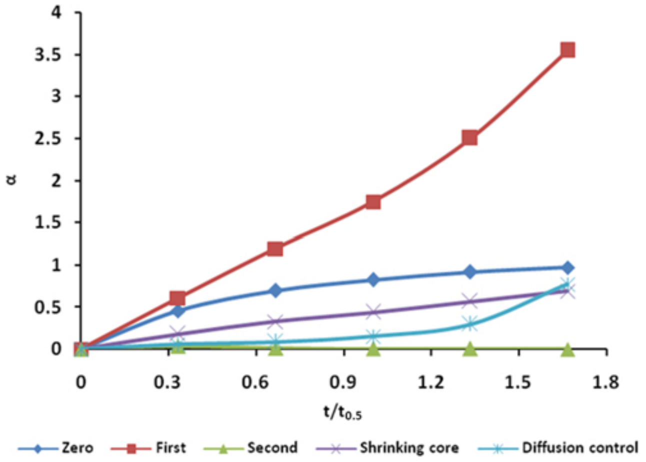

Fig. 12. Reduced time plot: Fraction of reduced time $t/t_{0.5}$ for leaching at 6 M HCl.

4-5. Kinetic Studies

Identification of the kinetic model for the dissolution of iron from the ore is carried out by plotting the experimentally obtained values of α (fraction reacted) against $t/t_{0.5}$ ($t_{0.5}$=time to reach α=0.5) and comparing with calculated standard reduced time plot for different kinetic model equations [25]. The results shown in Fig. 12 for 6 M concentration indicate that the mechanism of iron dissolution appears to follow closely the shrinking core reaction model, i.e., $[1-(1-\alpha)^{1/3}]$. The integral form of the shrinking core reaction model is a function of time at different temperatures as presented in Fig. 13 for leaching at 6 M concentration. This kinetic study plot follows the shrinking core model, and the rate constants are found from this. The leaching rate constant values are in Table 5 for 6 M acid concentration.

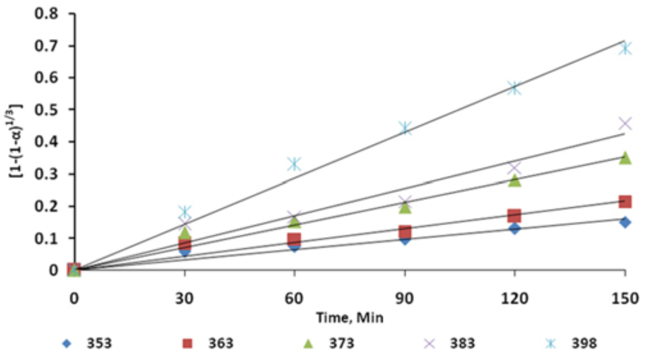

Fig. 13. Kinetic plot for first order reaction model from soda ash roasted slag at 6 M concentration.

Table 5. Rate constants "k" at different temperatures for overflow sample leached at 6 M HCl

k, Min^{-1}	0.0011	0.0015	0.0024	0.0028	0.0048
T, K	353	363	373	383	398

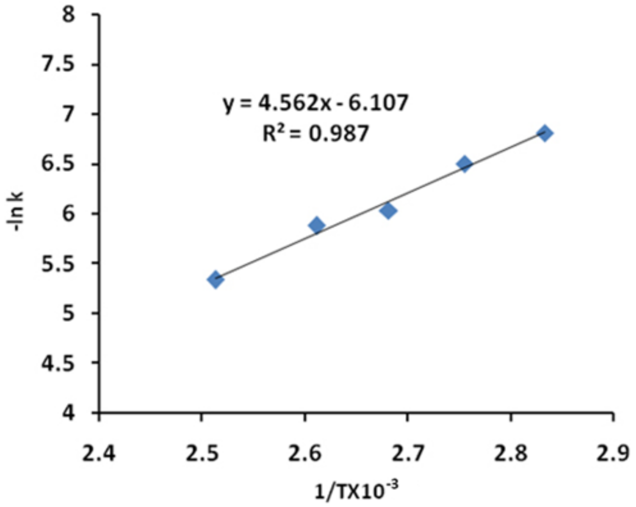

Fig. 14. Arrhenius plot of constant "k" for soda ash roasted slag sample at 6 M concentration of HCl.

The relationship between the rate constant k and temperature is given by the Arrhenius equation,

$$k = Ae^{-\frac{E}{RT}} \tag{7}$$

$$\text{or, } \ln k = \ln A - \frac{E}{RT} \tag{8}$$

Where A is the frequency factor and E is the apparent activation energy.

The logarithmic base - e of rate constants is plotted in Fig. 14 for the leaching process at 6 M acid concentration against the temperature inverse, by which activation energy is determined. The rate of iron dissolution followed first-order reaction mechanism $[1-(1-\alpha)^{1/3}]$ with an apparent activation energy is 37.9 kJ/mol for 6 M concentration leaching reaction.

5. Product (Synthetic Rutile) Characteristics

The calcined synthetic rutile product is a fine white powder. The complete chemical analysis of the obtained product is given in Table 6. It indicates that the TiO_2 content of the product is 97.21%, whereas Fe_2O_3 is 1.68%. It contains 0.27% of SiO_2 accompanied with 0.05% of V_2O_5 and 0.02% of Cr_2O_3. The XRD pattern of synthetic rutile obtained after leaching shows (Fig. 15) contains maximum number peaks of rutile (TiO_2) followed by quartz.

Table 6. Complete chemical analysis of synthetic rutile (leached residue)

Constituents	Wt, %	Constituents	Wt, %
TiO_2	97.21	V_2O_5	0.05
Fe_2O_3	1.68	MgO	0.21
SiO_2	0.27	MnO	0.12
Al_2O_3	0.31	P_2O_5	0.13
Cr_2O_3	0.02	-	-

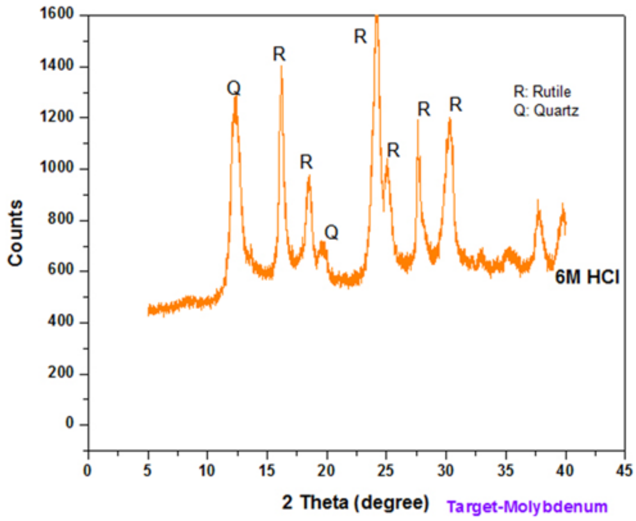

Fig. 15. XRD pattern of product obtained after leaching.

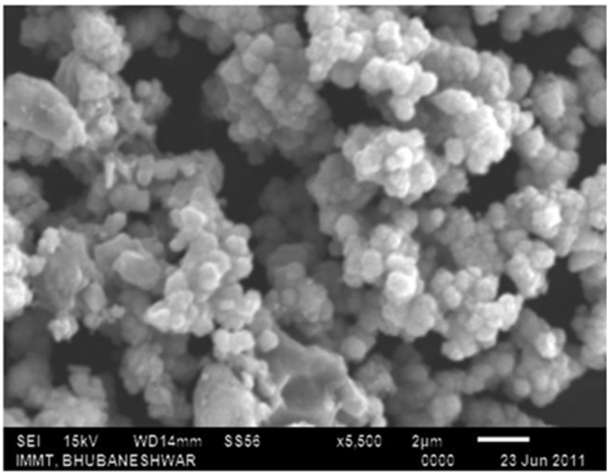

Fig. 16. Morphological textures of leached residue.

The product is observed under scanning electron microscopy (SEM). The morphological features of product obtained at 6 M are shown in Fig. 16. In this case, dissolution takes place massively on each particle because of random distribution of iron oxide followed with sodium titanate on them. The SEM image clearly shows that negligible amount of iron is reported in the product. In addition, there is a strong evidence of the effect of porosities on the microstructure, which allows solution penetration through the particle assisting in iron removal, otherwise dissolution could be more difficult.

CONCLUSIONS

The red sediment sample of badlands topography of Basanputti village, Ganjam Dist., Odisha, India contains 33.2% of total heavy minerals. The mineralogical modal analysis reveals that the sample contains ilmenite (28.71%) followed by sillimanite (3.32%), zircon (0.32%), rutile (0.23%) and other heavy and gangue minerals (0.61%).

A successful method for preparation of synthetic rutile from red sediment ilmenite of Orissa by soda ash roasting and hydrochloric acid leaching has been developed. The process of roasting followed by leaching is an effective process for preparation of synthetic rutile which can be used in the paint and plastic industries. Results of soda ash roasting reveal that at 100% stoichiometric ratio of Na_2CO_3, the leachability of iron is 15.7%. Then, to remove the remaining iron, hydrochloric acid leaching gives a better result in comparison to oxalic acid leaching and sulfuric acid leaching. The optimum condition for leaching of remaining iron is at 6 M hydrochloric acid at 398 K for 2.5 h (1/10 solid/liquid mass ratio). The dissolution of iron is maximum, i.e. 35.6%, at the optimum condition of temperature at 398 K for 150 min. Finally, calcined product contains 97.21% of titanium dioxide (TiO_2) and 1.68% of Fe_2O_3.

The results of leaching studies on removal of iron from soda ash roasted slag product of ilmenite reveal that leaching of iron present in the sample follows the shrinking core kinetic model. The rate of iron oxide dissolution follows the equation $[1-(1-\alpha)^{1/3}]$ with an apparent activation energy of 37.9 kJ/mol for 6M concentration leaching reaction.

ACKNOWLEDGEMENTS

The authors are thankful to Prof. B.K. Mishra, Director of the Institute of Minerals and Materials Technology (CSIR), Bhubaneswar for giving permission to publish this paper. Thanks also to Mr. P.S.R Reddy, Head of Mineral Processing Department for his constant encouragement to carry out this work for publication. One of the authors, Ms. T. Laxmi, is thankful to BRNS for granting an SRF position to her.

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