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Leaching and kinetics studies on processing of Abu-Ghalaga ilmenite ore

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Abstract - *Agitation leaching technique for upgrading Abu Ghalaga ilmenite ore for wide range of applications and major demands has been studied. Experiments of leaching and dissolution kinetics of ilmenite ore using hydrochloric acid have been carried out. The effect of variable parameters; acid concentration, particle size, solid liquid ratio, agitation speed, temperature, and time leaching were studied. The study results showed that the leaching rates were highly affected by increasing hydrogen ion concentration. Under the optimum condition, synthetic rutile assaying93% TiO_2 was obtained from an ilmenite ore assaying of about 37% TiO_2. The kinetics of reaction was found fit to the diffusion reaction model. The activation energy was calculated 17.607 KJ/mol.*

Key Words: Ilmenite, Abu-Ghalaga, Leaching, Hydrochloric acid, Synthetic rutile.

1. INTRODUCTION

The mineral ilmenite (nominally $FeTiO_3$) is huge resource of rutile (TiO_2), which can be used directly as pigment or for the manufacture of rubber, ceramics, papers, welding-rod, coatings, and in other areas of chemical industry [1]. The most important ilmenite minerals are those from the relatively rare massive deposits, which often occur in association with oxides such as hematite or magnetite, and the more widely occurring titaniferous beach sand deposits, which have resulted from natural erosion and concentration processes [2]. Ilmenite ore is another type of naturally occurring titanium ore. It is an attractive alternative to naturally occurring rutile titanium ores because it is both cheaper to mine and more common [3]. Titanium minerals are found in hard rock deposits and in beach and alluvial sands commonly know as ''beach sands.'' Other sources of titanium include titaniferous slags (70-85% TiO_2) made by electric furnace smelting of ilmenite with carbon [4]. The growing titanium metal industry also relies on high grade rutile. All these make the upgrading of ilmenite to synthetic rutile more and more important. However, the upgrading processes are generally expensive due to the involvement of multi steps of energy sensitive thermo reductive conversions and leaching to remove iron impurities [5]. Leaching procedure was carried out to remove all the Fe metal and obtain a high-grade TiO_2. After leaching and calcination of the milled and annealed mixture of Fe TiO_3/C under the optimal conditions, TiO_2 nanoparticles with a size of 10-100nm and purity more than 95% were obtained. [6]. The main source of titanium in Egypt is ilmenite. Ilmenite is present in a rock form in different localities in the Eastern Desert, and in the black sands on the Eastern part of the Mediterranean Coast [7]. Abu Ghalaga ilmenite this area lies 17 km South West of Abu Ghosoun port on the Red Sea coast and 100 km South of Mersa Alam city. The Ilmenite deposit is the largest among the ilmenite localities in Egypt. It is confined to gabbroic mass and occurs as a sheet-like body taking NW-SE and SE trend, and dips 30° to the NE direction.The main ilmenite mass forms a big lens with exposed length about 300 m, and an average width of about 150 m [8]. The beneficiation of ores to an industrial feedstock grade is always desirable before such material is chemically processed; these because it reduces the size and energy requirements of chemical processes and also it can significantly reduce the complexity of such processes while increasing their efficiency [9]. Titanium dioxide have been produced by two processes the sulfate process, and the dry chlorination process. The sulfate process which utilises ilmenite ($FeTiO_3$) as a raw material is well known and widely applied but it is lengthy, costly and the by product ferrous sulfate is less marketable [10]. The dry chlorination process which utilises rutile (TiO_2) as a raw material presently enjoys more favourable economics and generates less waste materials [11]. Shortage of natural rutile has encouraged research efforts to convert ilmenite into synthetic rutile for the chlorination process [12]. There are several processes for the production of synthetic rutile from ilmenite, most of which fall into one of five classifications:

(1) Smelting processes, where the iron part of the ilmenite is reduced and melted to separate the iron from titanium [13].
(2) Reduction of ilmenite to convert ferric iron partially to the ferrous form or completely to the metallic iron from followed by acid leaching [14].

(3) Reduction of the iron content of the ilmenite to the metallic iron followed by corrosion with oxygen and ammonium chloride [15].

(4) Oxidation and reduction of ilmenite followed by hydrochloric acid leaching, MURSO process [16].

(5) Roasting and magnetic separation followed by hydrochloric acid leaching, ERMS process [17].

All these processes depend mainly on reductive and/or oxidative thermal pretreatment of ilmenite which is an extensive energy consuming stage. Reductive pretreatment aims at converting the ferric iron in ilmenite in to ferrous state which is more soluble in hydrochloric acid. It was stated elsewhere that the dissolution of ilmenite in hydrochloric acid could be enhanced by reduction in solution using metal such as iron, zinc and tin [18]. In the present study,the hydrochloric acid agitation leaching of Abu Ghalaga ilmenite ore low grade titanium dioxide as well as the parameters affecting on the leachability which including acid concentration, particle size, solid liquid ratio, agitation speed, temperature and time of leaching. The kinetics and mechanism of dissolution process were also studied.

2. MATERIAL AND METHODS

2.1 Raw material

The ilmenite ore sample weighing about 200 kg (-25mm) was obtained from Abu Ghalaga mine in the south eastern Desert of Egypt. This sample was mixed thoroughly then crushed to (-5mm). A representative sample weighing about 200gm. This representative sample was used for performing complete physicochemical analysis using X-Ray fluorescence Spectrometry (XRF) and mineralogical analysis using X-Ray Diffraction (XRD) technique. A nest of sieves namely; 1000, 710, 500, 355, 250, 150, 90 and 63 µm. was used to fractionate the sample. A laboratory grade of hydrochloric acid (specific gravity and concentration of 1.84g/ml and 38%respectively is prepared and used as leaching agent in different stages of the experimental work.

2.2 Methods

2.2.1 Experimental setup

Acid leaching of ilmenite ore was carried out using 1000 cm^3 three necked glass reactor provided with a reflux condenser and a mechanical agitator with Teflon-coated stirring rod. A stirring speed of 300 rpm was applied to keep the slurry suspended during the leaching experiment. The desired volume of hydrochloric acid of the required concentration was filled in the reactor, then the sample was added and heated.

2.2.2 Agitation leaching

In the leaching method, each leaching experiment was performed by agitating with a mechanical agitator of a weighed amount of the ground sample 30gm with 180 ml of acid of a specific concentration at a 1:6 solid/liquid ratio for 180 min period of time and 70ºC temperature leaching. The obtained slurry was cooled, filtered, and washed with 3% HCL and introduced in a dryer at 110ºC and was calcined at 900ºC to completely dehydrate the sample. The obtained filtrates and leach liquors were analyzed for the metal values to calculate their leaching efficiency percent. The main relevant acid leaching conditions for titanium dioxide recovery include acid concentration, particle size, solid/liquid ratio, agitation speed, temperature, and leaching time.

2.2.3 Methods of analysis

For the chemical analysis of the ilmenite ore, a weighed sample of finely ground ore was fused with potassium pyrosulfate, dissolved in 1:1 H_2SO_4 and filtered. The metallic contents were analysed in the filtrate and Titanium was determined spectrophotometrically by the hydrogen peroxide method at wavelength of 410 nm. Titanium (III) was determined by titration against $FeCl_3$ using KSCN as an indicator. Total iron and ferrous iron were determined spectrophotometrically using the phenanthroline method at wavelength of 515 nm [19]. For the chemical analysis of the titanium dioxide concentrate, a weighed sample of the calcined concentrate was dissolved in ammonium sulfate and concentrated sulphuric acid. The content of titanium in the filtrate was determined spectrophotometrically.

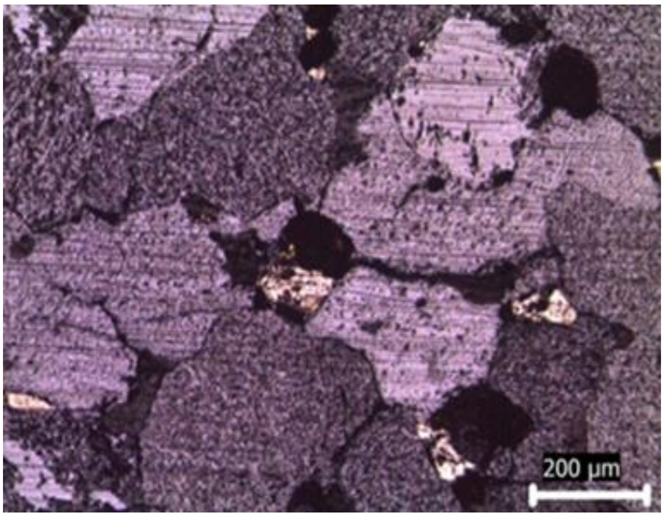
3. RESULT AND DISCUSSION

3.1Physicochemical analysis of Abu Ghalaga ilmenite ore

The magmatic Fe-Ti oxides deposits in Egypt are present in the South Eastern Desert. Among these deposits, there are considerable reserves of ilmenite ore in Abu Ghalaga region. The estimations of the reserves of ilmenite ore in Abu Ghalaga area variable, ranging from 3 million tons (Holman,1956), Through 10 million ton (Moharram,1959), to 50 million tons (Mahmoud et al., 2004). The Abu Ghalaga ilmenite ores can be classified into black (fresh) ore and red (oxidized) ore but our represents the major reserves in Abu Ghalaga ilmenite open-pit mine.

Phase identification for the ilmenite ore was carried out using the reflected-light microscope and X-ray diffraction. Under the reflected- light microscope, it can be seen that ilmenite is the predominant mineral of all minerals present. It occurs as bluish grey anhedral grains combined together in the form of granular texture Figure 1a. The characteristics of size are from 0.1 to 0.4mm. Under cross polars, the ilmenite grains exhibit polarization colors of grey shades without noticeable internal reflection. Ilmenite is not found as homogeneous grains, but instead hematite-ilmenite exsolution texture is present Figure 1b. This texture results from the breakdown of Fe_2O_3-$FeTiO_3$ solid solution series. This solid solution is homogeneous at high temperature, but it is subjected to destabilization at temperatures below 450°C. At this point, ilmenite begins to separate from hematite and forms host crystals that enclose exsolved lamellae of hematite. The XRD showed two mineral phases present; these are ilmenite and hematite Figure 2. Thus, the ore is mainly formed from ilmenite hematite together with small quantities of ilmenite albite and ilmenite hematite albite. The results of the geochemical analysis for the representative composite sample of ilmenite ore are given in Table 1. This sample contains 36.78% TiO_2 and 55.71% total iron. Consequently, the Abu Ghalaga ilmenite is considered as medium grade ore since it contains a relatively small amount of titanium. The analyzed composite ore sample also contains 4.46 %SiO_2, 0.03 P_2O_5, 0.36% MnO and 0.72% Al_2O_3. Most of the impurities in the investigated ilmenite ore sample are expected to be removed by milling and reductive leaching.

a



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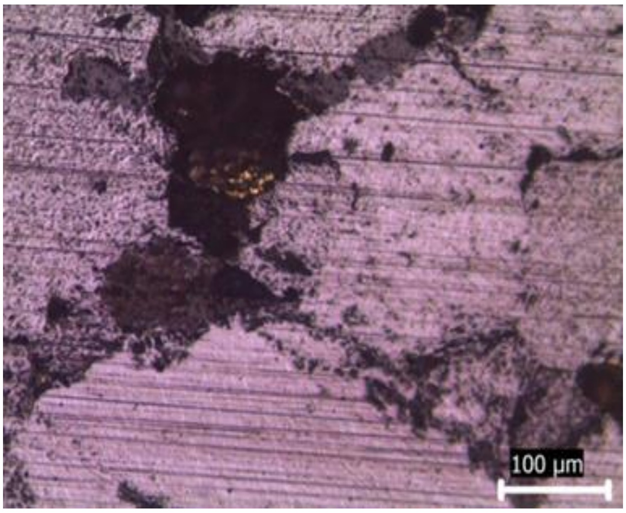

Fig -1: Reflected light Microscopic photo graphs Abu Ghalaga Ilmenite ore (a) Granular texture composed of anhedral grains of hematite-ilmenite and magnetite-hematite intergrowths, with intergranular albite and chalcopyrite. (b) Hematite-ilmenite exsolution intergrowth, with intergranular albite and chalcopyrite.

Table -1: Chemical analysis of Abu Ghalaga ilmenite ore

Oxide	Wt%
TiO_2	36.78
FeO	25.85
Fe_2O_3	29.86
Al_2O_3	0.72
MgO	0.81
CaO	0.15
MnO	0.36
Cr_2O_3	0.21
V_2O_5	0.38
SO_3	0.21
P_2O_5	0.03
SiO_2	4.46
L.O.I	0.17
Total	100

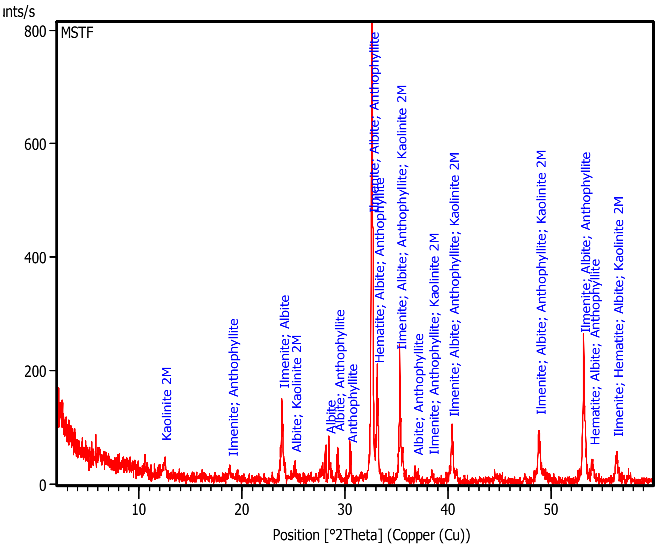

Fig -2: XRD Analysis of head sample

3.2 Factors affecting on the leaching ilmenite ore

3.2.1 Effect of HCL concentration

The effect of hydrochloric acid concentration on the leaching of ilmenite ore was investigated by using different acid concentrations 5, 10, 15, 20 and 25% v/v. The typical operating parameters were conducted as follows 180 min. agitation time, 70°C temperature, 1:6 solid/liquid ratio, -63µm Ore particle size and 300 r.p.m. Agitation speed.The results obtained are shown in Figure 3.

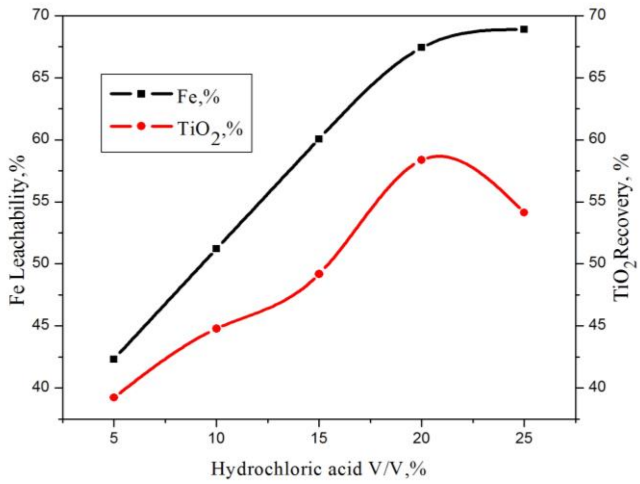

Fig -3: Effect of HCL concentration on ilmenite leaching
[Test condition comprised: particle size-150µm; S/L ratio 1:6; agitation speed 300 r.p.m; temperature 70°C; agitation time 180 min.]

These results indicated that the ore is easily amenable to hydrochloric acid leaching. Hence, by gradual increasing acid concentration from 5% to 20%, the leachability of iron increases almost linearly until it reaches to 67% at 20% acid concentration. On the other hand, increasing the HCL concentration to more than 20% sharply decreased the rutile recovery due to high titanium losses in solution reaching about 20% in solution at HCL 25%. The high acid concentration effect on the hydrolysis reaction and hence higher amounts of titanium remain soluble in HCL solution. For this reason selection of the optimum HCL concentration to be 20% is reasonable to give 67.20% of iron removal and 2.0% of TiO_2 soluble in solution.

3.2.2 Effect of particle size

The effect of particle size on the leaching process was investigated by using different size fractions, +1000, -1000+710, -710+500, -500+355, -355+250, -250+150, -150+90, -90+63 and -63µm. Fixed leaching conditions involved 20% acid concentration, 1:6 solid/liquid ratio, 180 min agitation time, 70°C temperature and 300r.p.m. agitation speed. The results obtained are shown in Figure 4. It is well known that by decreasing particle size, the dissolution increases [10]. From this figure, it is shown that, the best leachability was obtained at (-0.63µm) particle size. This may be due the larger surface area exposed to the leaching solution, when leads to increase the leachability. The titanium dioxide recovery was 68.14% for -63µm fraction, while it was 45.37% for +1000µm fraction. The size fraction of -63 µm was chosen due the higher recovery of titanium dioxide and dissolution iron.

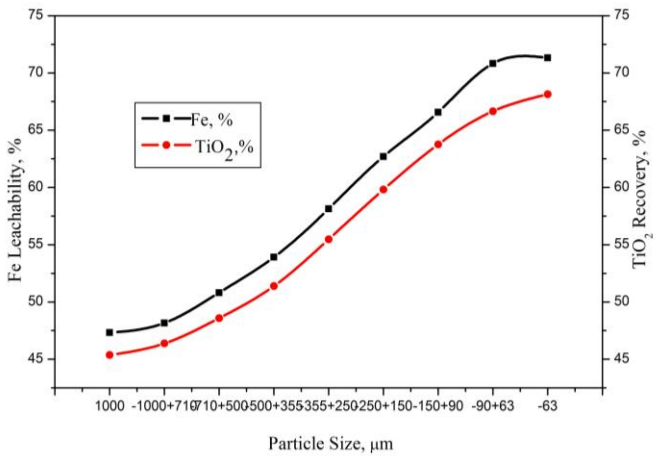

Fig -4: Effect of particle size on ilmenite leaching
[Test condition comprised: acid concentration 20%; S/L ratio 1:6; agitation speed 300 r.p.m; temperature 70°C; agitation time 180 min.]

3.2.3 Effect of Solid/liquid ratio

The effect of solid/liquid ratio on the leaching of ilmenite was studied at 1:2, 1:4, 1:6, 1:8 and 1:10 solid/liquid ratios, while the other testing parameters comprised at 20% acid concentration, 180 min agitation time, 70ºC temperature, with –63 µm Ore particle size and 300 r.p.m. agitation speed. The results are shown in Figure 5. In the all experiment's liquid volume was kept constant, and the amount of solid was changed to obtain the desired solid/liquid ratios. From these data, it was found that beyond 1:6 S/L ratio only slight steady in all leaching efficiencies of ilmenite has been achieved. Accordingly, a solid ratio of 1/6 would be considered as optimal ratio at which the leaching of iron 67.72% and recovery of titanium dioxide 64.78%. Improving the latter could be realized by extending the leaching time or temperature.

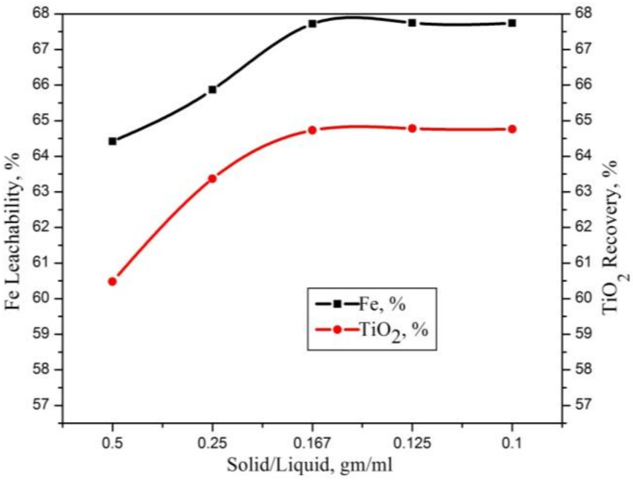

Fig -5: Effect of solid-liquid ratio on ilmenite leaching
[Test condition comprised: acid concentration 20%; Particle size -63µm; agitation speed 300 r.p.m; temperature 70ºC; Agitation time 180 min.]

3.2.4 Effect of Agitation speed

The effect of agitation speed was studied. The agitation speed was varied from 100 to 500 r.p.m. keeping the other testing parameters fixed at 20% hydrochloric acid concentration, 1:6 solid/liquid ratio, 70ºC temperature and 180 min agitation time. The results are shown in Figure 6. The results indicated that by increasing the agitation rate the diffusion of liquor to the surface of the particles increases. And this leads to enhances leachability of iron and higher titanium dioxide recovery at 400 r.p.m. It was found that there is no significant effect on iron leachability and titanium dioxide recovery after 400 r.p.m accordingly an optimum agitation speed of 400r.p.m. was obtained. The obtained results were found to be agreed with these obtained by [12].

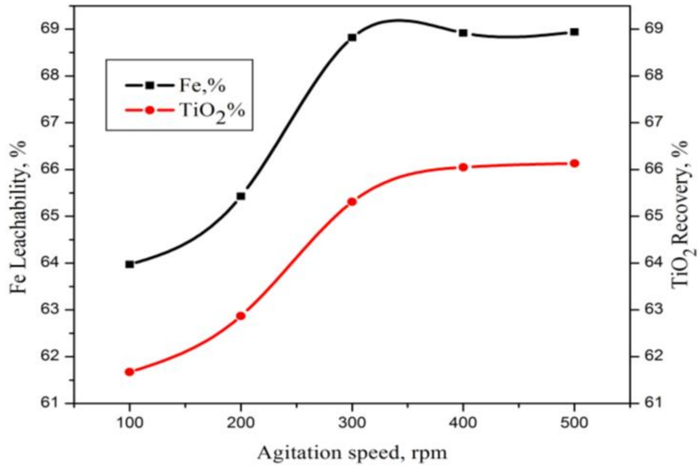

Fig -6: Effect of agitation speed on ilmenite leaching
[Test condition comprised: acid concentrati20%;particle size -63µm; S/L ratio 1:6; temperature 70ºC; agitation time 180 min.]

3.2.5 Effect of leaching temperature

The effect of temperature on the leaching has been studied in the temperature range from ambient (about 40ºC) up to110ºC. The other leaching conditions were fixed at 20% hydrochloric acid concentration, -63 µm Ore particle size, 1:6 solid/liquid ratio, 180 min agitation time and 400r.p.m. agitation speed. The results are shown in Figure 7. The results revealed that actually the importance of temperature to obtain reasonable leaching efficiency of ilmenite. Studying at ambient temperature under the above condition did not leach more than 64.93% only. Increasing the leaching temperature to 70ºC increases the leachability of iron and recovery of titanium dioxide to 67.43% and 65.18% respectively. Further increasing of temperature to 110ºC increased the leachability of iron to 78.13% and titanium dioxide recovery increased to76.92%, the leaching temperature is quite important for ilmenite leaching. The 110ºC was found to be optimal temperature for saving energy consumption. These results were found to be agreed with these obtained by [12]. Who indicated that rise of temperature to 110ºC gives a significant increase in ilmenite leachability.

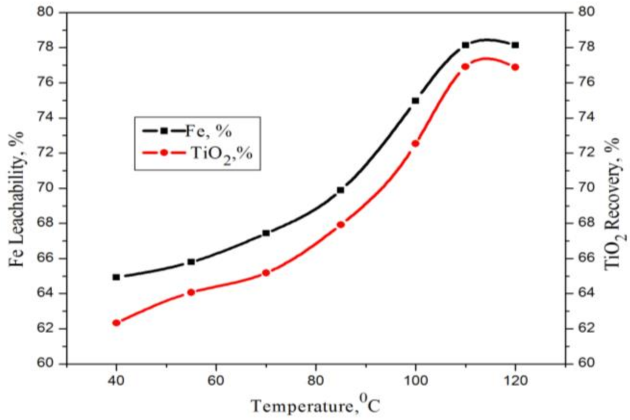

Fig -7:Effect of temperature on ilmenite leaching
[Test condition comprised: acid concentration 20%; Particle size -63µm; agitation speed 400 r.p.m; S/L ratio 1:6; Agitation time 180 min.]

3.2.6 Effect of agitation time

The effect of agitation time on ilmenite leaching was investigated to determine the necessary time required for ilmenite to achieve maximum dissolution and titanium dioxide recovery as possible. So, different intervals of time (60, 120,180, 240, 300, 360min). Were investigated under testing parameters as follows; 20% hydrochloric acid concentration, -63µm Ore particle size, 1:6 solid/liquid ratio, agitation speed 400 r.p.m and 110ºC reaction temperature. The results are shown in Figure 8. From these data it is clear that a substantial percent of iron was leached within 60 min 73.08% and recovery titanium dioxide is 71.58% and increased dissolution iron to 96.37%, and recovery of titanium dioxide to 92.87% in 300min., above this period, there was only a slight increasing in iron dissolution and titanium dioxide recovery until 360min. for this reason was chosen 300min optimum agitation time.

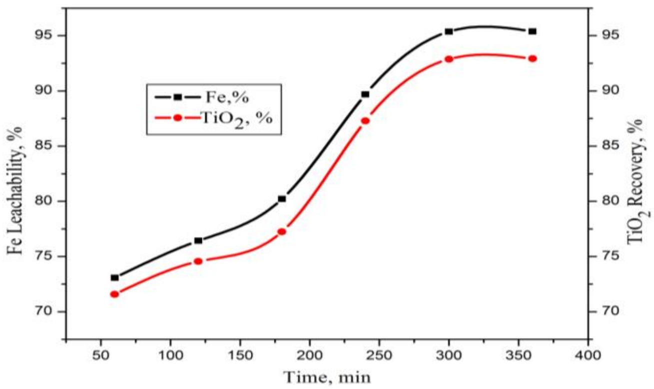

Fig -8: Effect of agitation time on ilmenite leaching
[Test condition comprised: acid concentrati20%; particle size -63µm;S/L ratio 1:6; agitation speed400r.p.m; temperature 110ºC.]

3.3 Kinetics of leaching

The leaching of ilmenite ore was carried out at optimum conditions in the presence of 20% hydrochloric acid concentration, -63µm particle size, 1:6 solid/ liquid ratio and 400 r.p.m. agitation speed at temperature ranges between 40ºC and 110ºC. Figure .9 shows that the leachability of ilmenite increases gradually by increasing of time and temperature. The maximum leachability was found to be 96% at 110ºC and after leaching time of 300 min. The un-reacted shrinking-core model is the most commonly used mathematical model to describe the heterogeneous reactions like mineral leaching from ores. The rate of reaction was controlled by the following steps: solid or product layer diffusion and chemical reaction. One or more of these factors might control the rate of the reaction [21]. In order to understand the leaching mechanism prevalent for ilmenite ore, models. (1) and (2) were used in this study.

$[1-3(1-X)^{2/3}+2(1-X)] = k_d\, t$ ………………………………. (1)

$[1-(1-X)^{1/3}]= k_c\, t$ ……………………………………………. (2)

Where [X] is the conversion fraction of solid particle, [k_d] is the rate constant (min^{-1}) for diffusion through the product layer,[kc] is the apparent rate constant (min^{-1}) for the surface chemical reaction and [t] is the reaction time. The relationship between reaction model and leaching time, at different temperatures is given in figure 10. The mean values of the reaction rate constants [K] were determined from the slopes of the straight line of the relation between kinetic model and time. The best fit has R^2 of 1.0. The K_d values given in table 2. Vary in the range of 0.0007-0.0023 min^{-1} while K_c was between 0.0011and 0.0023 min^{-1}. The R^2 values for K_d was 0.6565 to 0.9753 while for K_c it was in the range of 0.8684-0.3656. Based on the R^2 values it can be inferred that the predominant dissolution mechanism of ilmenite from Abu Ghalaga ore sample is diffusion controlled only. From the obtained data, logarithmic values of these reaction rate constant [K_d] were plotted against the reciprocal of the absolute reduction temperature according to the Arrhenius equation as shown in figure 11. The apparent activation energy (Ea) was calculated from the slope of straight line obtained to be 17.603KJ/mol for diffusion controlled reaction model. Based on the (Ea) values it can be inferred that the predominant dissolution mechanism ilmenite ore is diffusion controlled only. This value is less than the amount mentioned by (Madakkaruppan, 2014). Who pointed out that the activation energy for diffusion controlled reactions is below 20 KJ/ mol and is above 40 KJ/mol for chemical controlled reactions.

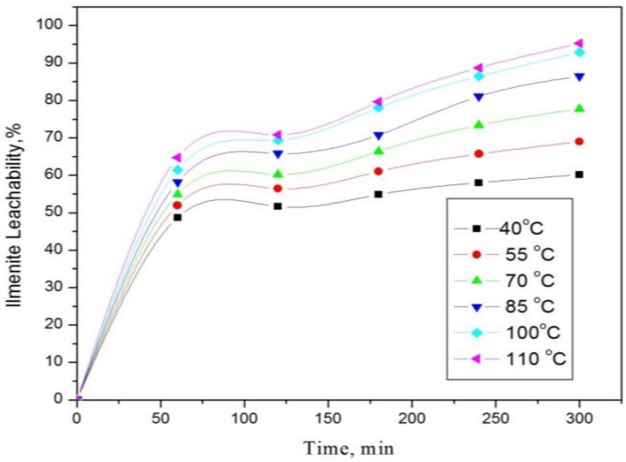

Fig -9: Plot of the ilmenite leachability versus time of leaching at different temperatures

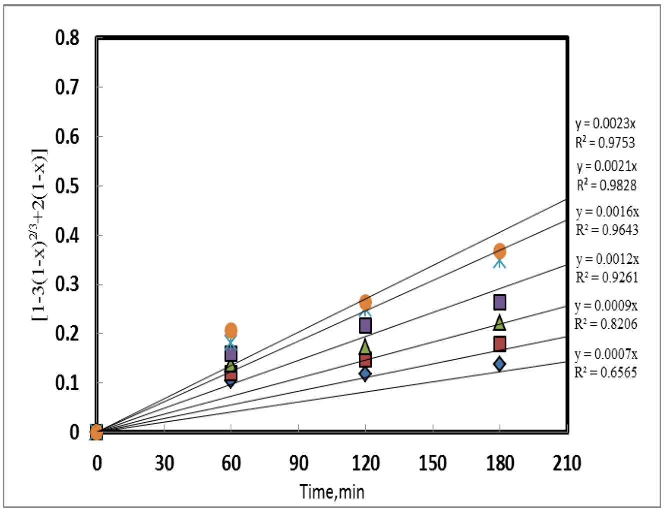

Fig -10: Plot of 1-3(1-X)$^{2/3}$+2(1-X) Versus time for different temperature.

Table -2: The apparent rate constant with their coefficient of determination at different temperature

Temperature (ºC)	Apparent rate constant (min $^{-1}$)		coefficient of determination(R^2)	
	Chemical control (K_c)	Diffusion control (K_d)	Reaction control	Diffusion control
45	0.0011	0.0007	0.3656	0.6565
55	0.0013	0.0009	0.5231	0.8206
70	0.0015	0.0012	0.6738	0.9261
85	0.0018	0.0016	0.7851	0.9643
100	0.0021	0.0021	0.8565	0.9828
110	0.0023	0.0023	0.8684	0.9753

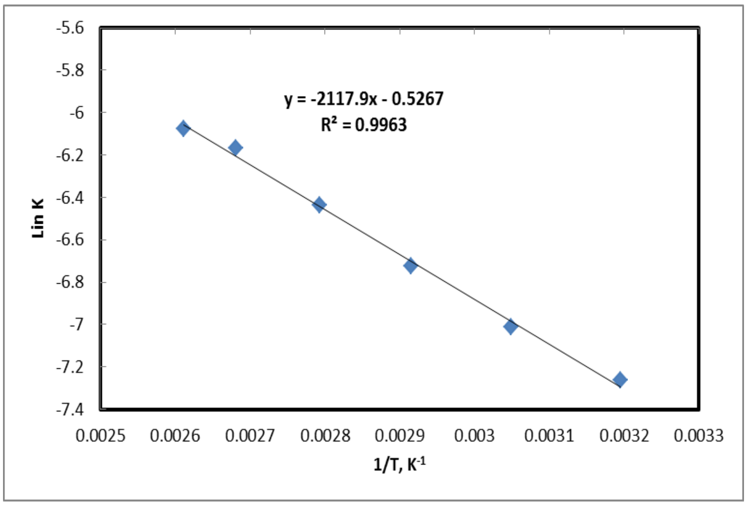

Fig -11: Plot of Ln K(min^{-1})against reciprocal of absolute temperature (K^{-1}).

4. CONCLUSIONS

From this study, it can be concluded that the optimum leaching conditions are:20% acid concentration, -63 μm particle size, 1:6 solid/liquid ratio, 400 r.p.m. agitation speed, 110°C reaction temperature, and 300 min. agitation time, a calcined synthetic rutile product contains about 93.21% TiO_2 assaying and iron dissolution 96.62% assaying are obtained at these conditions. The leaching kinetics of ilmenite ore showed that the rate of dissolution using HCL acid is a diffusion controlled and follows the shrinking core model; [1-2(1-X)$^{2/3}$+2(1-X)]= K_dt with an apparent activation energy of 17.607KJ/mol.

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