Dissolution Kinetics of Ilmenite Ore in A Binary Solution

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Abstract—Leaching of iron from ilmenite ore using a binary solution (HCl-NaNO3) was investigated. The raw ilmenite ore sample was characterized using Scanning Electron Microscopy (SEM), X-ray diffraction spectroscopy (XRD) and X-ray Flourescence (XRF) techniques. The influence of acid concentration, oxidant concentration, particle size, solution temperature, stirring speed and liquid-to-solid ratios on the extent of dissolution was examined. The experimental data obtained at various process parameter conditions were tested in six kinetics models: shrinking core model’s diffusion through liquid film model(DTLF), diffusion through product layer model (DTPL), surface chemical reaction model (SCR); mixed kinetics model (MKM), Jander (three dimensional) model and Krüger and Ziegler model. The crystalline morphology of the sample was displayed by the SEM micrograph. XRF result revealed the dominance of titanium and iron in ilmenite while XRD confirmed that ilmenite exist mainly as FeTiO2. The results of the leaching studies showed that ilmenite dissolution in the binary solution increases with increasing acid concentration, oxidant concentration, reaction temperature, stirring speed and liquid-to-solid ratio; while it decreases with particle size. The study showed that 94.77% iron was dissolved by 1MHCi-0.6M NaNO3 at 75μm particle size, 75°C reaction temperature, 300rpm stirring speed and 30L/g liquid-to-solid ratio. The kinetics of the leaching process was best described by Krüger and Ziegler model with diffusion through the product layer as rate controlling step. The activation energy, Ea, was calculated to be 6.42kJ/mol. The results indicate that HCl-NaNO3 binary solution can be used as an effective lixiviant for extracting iron from ilmenite ores.

Index Terms—Activation Energy, Binary Solution, Dissolution, Kinetics, Ilmenite.

I. INTRODUCTION

Ilmenite or Titanic iron ore (FeTiO3), is widespread in igneous rocks as an accessory mineral, but is seldom concentrated or found in large crystals except in pegmatites and large bodies of plutonic rock. Iron is a major constituent in the elemental composition of Ilmenite. Many a times, the presence of iron poses a challenge in the production of TiO2 pigment from ilmenite ore. In recent times, beneficiation of ilmenite into synthetic rutile has drawn the attention of many researchers considering the increasing scarcity of natural rutile. Reference [1] reported that metal production from any metal source, like ore, concentrate, and secondary sources (various industrial wastes containing metals and scrap metals, etc.) is performed by one of the pyrometallurgical and hydrometallurgical methods, or by a combination of them.

The increased depletion of high grade ores and timely need for a shift from conventional pyrometallurgy’s high temperature processing have spurred research interest geared towards development of low temperature hydrometallurgical process for the extraction of target metals from their ores. According to [2], leaching processes in hydrometallurgy are concerned mainly with treating minerals in order to form a solution containing the metals to be recovered. The dissolution of mineral ore takes place through the following stages: (1) diffusion of reactant through the diffusion layer, (2) adsorption of the reactant on the solid, (3) chemical reaction between the reactant and the solid, (4) desorption of the product from the solid and (5) diffusion of the product through the diffusion layer. Any of these stages (1)-(5) may be the rate controlling step depending on its relative speed to the others [3].

In the leaching step in hydrometallurgy, the metal is leached using a suitable lixiviant [1]. The most efficient leaching agents are acids, due to their ability to leach both base and precious metals [4]. Researchers have published articles in recent times on the application of hydrochloric acid in many processes for upgrading ilmenite into synthetic rutile. The reactivity of ilmenite towards hydrochloric acid depends on the nature of the mineral, whether it has been altered or not. Around the globe, ilmenite deposits may contain a mixture of both unaltered and altered ilmenites. Often times, the unaltered ilmenite is more readily leached by hydrochloric acid than the altered ilmenite. Knowledge of the reactivity of a particular ilmenite towards hydrochloric acid is therefore essential for its upgrading by hydrometallurgical techniques [5]. The efficiency of the leaching stage contributes immensely to the overall success of the hydrometallurgical process. Therefore, dedicated attention is geared towards leaching reactions and kinetics of these reactions.

Leaching kinetics plays an important role in the extraction of metals and compounds [6]. An accurate understanding of the kinetics of dissolution is required in order to interpret the complex behaviour of leaching reactors, and to optimize the performance of a hydrometallurgical operation [7].

In previous works documented by [8], hydrometallurgical processing of ilmenite ores with hydrochloric acid was studied by several researchers using (i) direct leaching [5],[9],[12] (ii) leaching in the presence of oxidising agent [13],[14] (iii) leaching in the presence of reducing agent such as iron powder [15], and (iv) leaching after pre-oxidation of the concentrate at high temperature [16]. In this work leaching of iron from ilmenite using binary solution as lixiviant will be investigated.

To the best of our knowledge, there are no or very scanty publications on the kinetics describing leaching of ilmenite in hydrochloric acid/sodium nitrate binary solution. Furthermore, the influence of process parameters such as HCl acid concentration and sodium nitrate concentration, reaction temperature, stirring speed and liquid-to-solid ratio on ilmenite dissolution rate was examined.

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II. MATERIALS AND METHODS

Ilmenite was collected from Egon mine in Egon Local Government Area, Nasarawa state, Nigeria. The ore sample was crushed and size reduced with standard test sieve to different particle sizes for the leaching studies. X-ray Fluorescence Spectroscopy and X-ray Diffractometry analyses were conducted to evaluate the elemental and mineralogical compositions respectively of the ilmenite ore. SEM micrograph displayed the morphology of the ore. All chemicals for this research were all of analytical grade. The leaching experiments were conducted in a 500ml borosilicate glass reactor fitted with a condenser to maintain constant volume of the solution in the reactor through the set experimental time. The reactor set-up was placed on a magnetic stirrer with hot plate for temperature and stirring speed control. A known mass of ilmenite was added to the lixiviant at pre-determined experimental conditions. At defined time intervals, aliquots of 5ml of the leaching solution were accurately withdrawn from the reactor and filtered. The concentration of iron in filtrate was measured using atomic absorbance spectroscopy (AAS, model FS 240 AA). The ilmenite dissolution fraction was calculated using equation 1:

\[
x = \frac{\text{Conc. of Fe in filtrate}}{\text{Conc. of Fe in ore}}
\]

III. RESULTS AND DISCUSSION

A. XRF Characterization

The result of the elemental composition of ilmenite was investigated using the X-ray fluorescence technique and presented in Table I. The result reveal that the dominant elements in ilmenite ore sourced from Egon mine in Egon Local Government Area, Nassarawa state, Nigeria are titanium and iron. Other elements occurred in traces as it is evident in Table I.

| TABLE I: XRF RESULT FOR ILMENITE |
|-----------------------------------|
| Element | Al | Si | P | S | Ca | Ti | Mn | Co | Fe | Ni | Cu | Zn | Nb | Mo | Sn |
| Content | 1.68 | 3.04 | 1.04 | 1.79 | 1.19 | 22.53 | 0.74 | 0.54 | 17.61 | 0.03 | 0.08 | 0.10 | 0.57 | 0.15 | 9.06 |

B. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) displayed the morphology of the ilmenite ore (Plate 1). The micrograph for the raw sample seems highly crystalline with irregular shapes and rough edges.

C. X-Ray Diffraction Analysis

Table II records the XRD result for the mineralogical compositions of the raw ilmenite sample. The result show that ilmenite exist mainly as FeTiO₂. It is seen in Table II that ilmenite ore gave three principal peaks 2.7310, 2.7563 and 2.2407 Å. Ilmenite diffractogram displayed in Fig. 1 shows other associated minerals such as Euxenite and Titanium oxide anatase.

![Plate 1. SEM micrograph for raw ilmenite sample](image)

![Fig. 1. X-ray diffractogram for raw ilmenite](image)

D. Effect of leaching experimental conditions on ilmenite dissolution process

The result of the investigation to ascertain the effect of hydrochloric acid concentration on the leaching rate of iron is presented in Fig. 2a. The synergetic effect recorded may possibly show that the rate of mineral dissolution is affected directly by the hydrogen ion [H⁺] concentration.

Similar findings were reported by [17]. Fig. 2b reveals the effect of NaNO₃ concentration on the leaching rate of iron from ilmenite ore. The results show an increase from 60.59% to 78.27% when NaNO₃ concentration was increased from 0.15M to 0.60M after a reaction time of 180 minutes. The result 78.27% presented for 1M HCl - 0.6M NaNO₃ binary solution as lixiviant compared with 68.51% iron leached in HCl₉ₗ lixiviant suggests that the binary solution better enhanced the leaching rate. This may be due to high electrode potential of nitrate (NO₃⁻ + 2H⁺ + e⁻ ↔ NO₂⁻ + H₂O; E⁰V = 0.800) which considerably contributes to increasing in the dissolution of the ilmenite ore.

![TABLE II: X-RAY DIFFRACTION DATA FOR RAW ILMENITE SAMPLE](image)

The results for leaching experiments carried out with Ilmenite in 1M HCl - 0.6M NaNO₃ are shown in Fig. 2c. Particle size was varied between 75µm and 600 µm at 300rpm stirring speed, 20L/g liquid-to-solid ratio and 60°C solution temperature. Iron dissolution reached 78.27% and 63.05% after 180mins leaching for 75µm and 600 µm particle sizes respectively. The higher iron dissolution recorded at smaller particle size may be as a result of higher
specific surface area available for interaction with the lixiviant. Similar result was reported by [18].

The effect of reaction temperature on the leaching rate of iron from ilmenite ore in HCl-NaNO3 binary solution was investigated at reaction temperatures of 30°C, 45°C, 60°C, 75°C and 90°C. Other process variables were kept constant at 1M HCl - 0.6M NaNO3, 75µm, 20L/g and 300rpm stirring speed respectively. Fig. 2d shows that increase in reaction temperature enhances the leaching rate. At the end of reaction time of 180 minutes, it was obvious that extent of iron leached from ilmenite ore increased from 47.23% to 89.18% when the reaction temperature increased from 30°C to 90°C. Arrhenius equation established an exponential dependence between the rate constant of a chemical reaction and the reaction temperature therefore, it is in tandem with the submission that the reaction rate increases with increasing temperature.

Fig. 2e displays the effect of stirring speeds of 100, 200, 300, 400 and 500rpm on the dissolution rate of ilmenite ore. The concentration of the binary solution, particle size, liquid-to-solid ratio and reaction temperature were kept constant at 1M HCl - 0.6M NaNO3, 75µm, 20L/g and 75°C respectively. The trend followed by the plots show that increase in particle size generally had a positive effect on the dependent variable (% iron dissolved). However above 300rpm the effect of stirring speed became marginal on the dependent variable. Further, experiments therefore were done at a stirring speed of 300rpm.

The relationship between the dissolution rate and dissolution time of ilmenite ore particles at different liquid-to-solid ratios in 1M HCl - 0.6M NaNO3 was evaluated at 75µm particle size, 300rpm stirring speed and 75°C reaction temperature and presented in Fig. 2f. A direct proportional relationship was observed between the dependent and independent variables. This relationship may be as a result of availability of more leachable particles of the ilmenite ore.

Fig. 2. Effect of (a) HCl acid concentration (b) NaNO3 concentration (c) particle size (d) reaction temperature (e) stirring speed and (f) liquid-to-solid ratio on iron dissolution

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E. Kinetics study

Leaching kinetics is controlled either by the diffusional mass transfer of the reactant through a liquid boundary layer or ash layer or chemical reaction at the ore surface [19]. Dissolution of ilmenite in binary solution is a typical example of a heterogeneous reaction. For better understanding of the dissolution of ilmenite in the investigated binary solution, shrinking core model’s diffusion through liquid film model (DTLF), diffusion through product layer model (DTPL), surface chemical reaction model (SCR); mixed kinetics model (MKM) – a combination of surface reaction and diffusion, Jander (three dimensional) model and Kröger and Ziegler model were tested. The selected models are tabulated in Table III:

The experimental data were fitted into the six models at various acid concentrations, oxidant concentrations, particle sizes, reaction temperatures, stirring speeds and liquid-to-solid ratios. Based on the coefficient of determination ($R^2$) data presented in Table IV, Kröger and Ziegler model (equation 7) best described the kinetics behaviour of ilmenite dissolution in HCl-NaNO$_3$ binary solution.

The plots of $(1 - (1 - x)^{1/3})^2$ vs ln t as a function of acid concentration, oxidant concentration, particle size, reaction temperature, stirring speed and liquid-to-solid ratio are shown in Fig. 3 – 8.

### Table III: Leaching Kinetics Equations

| Model               | Equation                                      | Plot made                   | Eqn no |
|---------------------|-----------------------------------------------|------------------------------|--------|
| DTLF                | $k_t = x$                                      | $x$ vs $t$                   | 2      |
| DTPL                | $k_{dt} = 1 - 3(1 - x)^{2/3} + 2(1 - x)$      | $1 - 3(1 - x)^{2/3} + 2(1 - x)$ vs $t$ | 3      |
| SCR                 | $k_t = 1 - (1 - x)^{1/3}$                      | $1 - (1 - x)^{1/3}$ vs $t$  | 4      |
| MKM                 | $(1 - \frac{2x}{3}) - (1 - x)^{2/3} + \frac{1}{b} \frac{1}{b} [1 - (1 - x)^2] = Kt$ | $(1 - \frac{2x}{3}) - (1 - x)^{2/3} + \frac{1}{b} [1 - (1 - x)^2]$ vs $t$ | 5      |
| Jander (three dimensional) | $(1 - (1 - x)^{1/3})^2 = k t$                  | $(1 - (1 - x)^{1/3})^2$ vs $t$ | 6      |
| Kröger and Ziegler  | $(1 - (1 - x)^{1/3})^2 = k_{dnt}$              | $(1 - (1 - x)^{1/3})^2$ vs ln $t$ | 7      |

### Table IV: Coefficient of Determination Values for Investigated Kinetic Models at Various Process Variables

| Acid Conc | DTLF | DTPL | SCR | MKM | JANDER | KROGER |
|-----------|------|------|-----|-----|--------|--------|
| 1         | 0.625 | 0.880 | 0.713 | 0.872 | 0.910 | 0.971 |
| 2         | 0.645 | 0.895 | 0.748 | 0.889 | 0.922 | 0.976 |
| 3         | 0.631 | 0.893 | 0.750 | 0.887 | 0.925 | 0.972 |
| 4         | 0.626 | 0.894 | 0.754 | 0.888 | 0.929 | 0.972 |
| Ox. Conc  |      |      |     |     |        |        |
| 0.15      | 0.691 | 0.902 | 0.759 | 0.895 | 0.919 | 0.990 |
| 0.3       | 0.571 | 0.705 | 0.619 | 0.701 | 0.713 | 0.963 |
| 0.45      | 0.596 | 0.828 | 0.684 | 0.821 | 0.859 | 0.967 |
| 0.6       | 0.568 | 0.815 | 0.670 | 0.807 | 0.858 | 0.959 |
| Particle Size |      |      |     |     |        |        |
| 75        | 0.568 | 0.815 | 0.670 | 0.807 | 0.858 | 0.959 |
| 150       | 0.518 | 0.716 | 0.686 | 0.709 | 0.754 | 0.943 |
| 300       | 0.573 | 0.805 | 0.656 | 0.797 | 0.840 | 0.960 |
| 600       | 0.574 | 0.798 | 0.641 | 0.789 | 0.828 | 0.959 |
| Temperature |      |      |     |     |        |        |
| 70        | 0.711 | 0.905 | 0.758 | 0.896 | 0.916 | 0.992 |
| 45        | 0.615 | 0.859 | 0.694 | 0.850 | 0.887 | 0.970 |
| 60        | 0.568 | 0.815 | 0.670 | 0.807 | 0.858 | 0.959 |
| 75        | 0.502 | 0.727 | 0.611 | 0.721 | 0.788 | 0.938 |
| 90        | 0.483 | 0.700 | 0.598 | 0.695 | 0.711 | 0.931 |
| Stirring Speed |      |      |     |     |        |        |
| 100       | 0.521 | 0.750 | 0.625 | 0.744 | 0.804 | 0.944 |
| 200       | 0.509 | 0.731 | 0.612 | 0.725 | 0.786 | 0.940 |
| 300       | 0.502 | 0.727 | 0.611 | 0.721 | 0.788 | 0.938 |
| 400       | 0.496 | 0.716 | 0.607 | 0.711 | 0.799 | 0.935 |
| 500       | 0.492 | 0.711 | 0.600 | 0.706 | 0.776 | 0.934 |
| Solid – Liquid |      |      |     |     |        |        |
| 10        | 0.470 | 0.638 | 0.540 | 0.632 | 0.684 | 0.926 |
| 15        | 0.489 | 0.686 | 0.578 | 0.680 | 0.739 | 0.934 |
| 20        | 0.502 | 0.727 | 0.611 | 0.721 | 0.788 | 0.938 |
| 25        | 0.504 | 0.756 | 0.641 | 0.751 | 0.831 | 0.936 |
| 30        | 0.540 | 0.840 | 0.732 | 0.835 | 0.918 | 0.951 |

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The apparent rate constants, $k$, derived from the slope of the plots in Figs. 3 – 8 are presented in Table V.

The kinetic equation describing the extraction of iron from ilmenite ore can be expressed as:

$$\left(1 - \left(1 - x\right)^{1/3}\right)^2 = k_0 (A/C)^a (P/S)^b (S/L)^c \exp\left(-E_a/RT\right) \ln t$$ \hspace{1cm} (8)

From Fig. 9 to 13, the values of the constants, $\alpha$, $\beta$, $\gamma$, $\theta$ and $\varphi$, were estimated from the slope of the plots of the natural logarithm of apparent rate constants versus natural logarithm of the process parameters.

| TABLE V: Apparent Rate Constants $k_0$ and Coefficient of Determination ($R^2$) Values for Kröger and Ziegler Kinetic Model |
|---------------------------------------------------------------|
| Process Parameters       | $k_0$ | $R^2$ |
|--------------------------|-------|-------|
| Acid Concentration (M)    |       |       |
| 1                        | 0.151 | 0.971 |
| 2                        | 0.159 | 0.976 |
| 3                        | 0.166 | 0.972 |
| 4                        | 0.170 | 0.972 |
| Oxide Concentration (M)   |       |       |
| 0.15                     | 0.141 | 0.990 |
| 0.30                     | 0.148 | 0.963 |
| 0.45                     | 0.158 | 0.967 |
| 0.60                     | 0.166 | 0.959 |
| Particle Size (µm)        |       |       |
| 75                       | 0.166 | 0.959 |
| 150                      | 0.157 | 0.943 |
| 300                      | 0.156 | 0.953 |
| 600                      | 0.144 | 0.959 |
| Solution Temperature (°C) |       |       |
| 30                       | 0.119 | 0.992 |
| 45                       | 0.147 | 0.970 |
| 60                       | 0.166 | 0.959 |
| 75                       | 0.177 | 0.938 |
| 90                       | 0.182 | 0.931 |
| Stirring Speed            |       |       |
| 100                      | 0.173 | 0.944 |
| 200                      | 0.175 | 0.940 |
| 300                      | 0.177 | 0.938 |

(continued)
The reaction orders recorded for acid concentration (AC), oxidant concentration (OC), particle size (PS), stirring speed (SS), liquid-to-solid ratio (LS) from Fig. 9 to 13 are 0.086, 0.116, -0.062, 0.020, 0.090 respectively. The activation energy was calculated from the slope of the Arrhenius plot on Fig. 14. The activation energy of the leaching process and the value of pre-exponential constant, A, were calculated to be equal to 6.42 kJ/mol and 0.03s⁻¹ respectively. The value of activation energy in the dissolution process may be characterized to predict the controlling step. The activation energy of a diffusion controlled process is usually 21kJ/mol or less, when chemical reaction is the rate controlling step, the activation is estimated to be between 40-100kJ/mol [20]. Activation energy of 6.42kJ/mol calculated for dissolution of ilmenite in HCl-NaNO₃ confirms that the dissolution process within the scope of investigation is diffusion controlled. The equation describing the dissolution kinetics of ilmenite in HCl-NaNO₃ medium can be written as:

\[(1 - x)^{1/3} - 1 = 0.03(AC)^{0.008}(OC)^{0.116}(PS)^{-0.062}(SS)^{0.320}(LS)^{0.090}\exp(–6.42/RT)\]t\]  

IV. CONCLUSIONS

From the results presented in this study, the following conclusions can be drawn:

1. The XRF characterization result show the dominance of titanium and iron for Ilmenite from Egon mine in Egon Local Government Area, Nigeria. Also, the XRD results confirmed the originality of the ore.

2. The dissolution rate of ilmenite in HCl-NaNO₃ binary solution is dependent on the process.
variables (acid concentration, oxidant concentration, particle size, reaction temperature, stirring speed and liquid-to-solid ratio).

3. The overall results of the dissolution studies reveal that the kinetics data fitted the Kröger and Ziegler model with diffusion through the product layer as rate controlling step.

4. The activation energy, Ea, of 6.42kJ/mol calculated for the leaching process suggests that the system is diffusion controlled.

5. The potential capability of HCl-NaNO₃ binary solution to serve as a lixiviant for the recovery of iron from ilmenite ore has been established.

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