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CHEMICAL BENEFICIATION OF NIGERIAN ILMENITE ORE
Part-I. Hydrochloric Acid Leaching

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Leaching of powdered ilmenite ore was carried out in hydrochloric acid solutions in order to upgrade it. The effects of acid concentration, treatment time, particle-size, solid- liquid ratio, temperature and stirring speed were considered. The following have been found as the optimum leaching conditions for the ilmenite containing TiO_2 43.2, FeO 22.6, Fe_2O_3 16.8%, plus other impurities: HCl concentration 30%, treatment time 8 hrs, ilmenite-HCl ratio 1:5, and temperature 90°.The particle size between 100-425 µm and the stirring speed did not significantly influence leaching. The TiO_2 content of the ilmenite ore was increased to about 78%, and ferric chloride was recovered as a by-product.

Key words: Beneficiation, Leaching, Ilmenite.

Introduction

In the recent time, efforts are being made to sustain most industries in the developing nations through local sourcing of raw materials. As at today, the chief sources of titanium oxide are rutile and ilmenite. The former, beacuse of its high titanium oxide content, is usually preferred to other source ore.

Ilmenite, although not quite rich in titanium oxide, is more widely distributed than rutile in many parts of the world. Consequently, most countries have now resorted to the exploitation of ilmenite for manufacturing titanium oxide [1-5].

The Nigerian ilmenite deposit occurs in the alluvial concentrates of Plateau highlands of Nigeria. It is found in association with cassiterite, columbite, zircon, and monazite [6].

Various methods have been reported in literature for upgrading ilmenite [7-9]. However, leaching in hydrochloric acid solutions has been the mostly widely employed [10-13]. Apart from hydrochloric acid, sulphuric acid has also been used [14-16]. Results of acid leaching of ilmenite have revealed variations in the parameters of leaching [17-20]. Therefore, it is usually necessary to work out the parameters best for leaching a particular ilmenite ore. Only exploratory studies [6] have been conducted on the Nigerian ilmenite ore, there is still no report on its composition analysis. Moreover, it has never been investigated as a source of manufacturing titanium oxide. As such, titanium oxide-consuming industries in Nigeria still import titanium oxide for their uses.

In this study, however, we investigated the leaching of Nigerian ilmenite ore in hydrochloric acid solution in order to obtain some essential information on the upgrading of the ore. Data obtained will assist in pilot-plant studies on the utilization of the ilmenite ore, and will further add to the information available in literature on the acid leaching of ilmenite ores.

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Materials and Methods

Reagents. All chemicals used in this study were of analytical grade. Concentrated hydrochloric acid was diluted to the desired concentrations with distilled water.

Sample. The ilmenite ore was obtained from Consolidated Tin Mines, Nigeria. It was finely powdered in a ball mill, and screened through standard laboratory sieves. The chemical composition of the sample is given in Table 1.

Apparatus. The reaction-vessel was a 100ml three-necked flask, each neck was fitted with a water-cooled reflux, a water-sealed stirring rod, and a thermometer. The container was placed on a hot-plate with a temperature regulator.

Leaching procedure. About 5g of the ilmenite powder was dispersed in the acid solution, then the mixture was digested with stirring. At regular time intervals, 5 ml aliquots of the reaction solution was pipetted from the flask after the solids and settled down, and analyzed for titanium and iron only [21].

Analytical techniques. Standard methods of analysis were employed in all determinations. Titanium was determined colorimetrically by the peroxide method [22], silica by

TABLE 1. CHEMICAL COMPOSITION OF THE ILMENITE.

Constituent	Content (%)
TiO_2	43.2
FeO	22.6
Fe_2O_3	16.8
SiO_2	3.8
Al_2O_3	2.3
MnO	2.5
Cr_2O_3	0.6
CaO	1.4

the yellow silico-molybdate method [23] and iron (II) by the phenanthroline method [24]. Total iron and other metals were determined using Perkin-Elmer 403 atomic absorption spectrophotometer.

Results and Discussion

Table 1 shows that the ilmenite ore contained only 43.2% TiO_2, while total iron ($FeO + Fe_2O_3$) constituted more than 65% of the impurities present in the ore. The level of impurities affects the quality of the white pigment produced from the ore [25]. It, therefore, follows from the results in Table 1 that the ilmenite ore studied needs beneficiation to enhance its utilization.

The variation of TiO_2 and Fe amounts with the acid concentration is illustrated in Figs. 1 and 2. From both figures, the degree of beneficiation was strongly affected by the acid concentration and the reaction time, and the behaviour of TiO_2 in the acid solutions followed almost the same pattern. Increasing the acid concentration lead to increase in the TiO_2 content of the concentrate, and shortened the reaction time of attaining optimum leaching (Fig. 1). This suggests that leaching of the impurities occured in strong solutions of HCl more intensively, and at a faster rate, than in the dilute solutions. The results shown in this Fig. 2 revealed that the amount of iron leached determined largely the extent to which the ore could be upgraded by HCl leaching. Furthermore, increasing acid concentration reduced the level of iron in the concentrate. This suggests that the iron was largely and quickly removed in strong solutions of HCl, more than in weak ones. In order to get a clearer picture of the effect of acid concentration on the degree of beneficiation, the Ti/Fe ratios were plotted against the reaction time at various HCl concentrations (Fig. 3). From these results, it is evident that the degree of beneficiation was strongly affected by the acid concentration.

Experiments with the following particle sizes: < 100μm, 100-225μm, 250-350μm, and 350μm did not affect the degree of leaching. This suggests that good results could be obtained with particles of sizes 350μm. In most leaching studies on

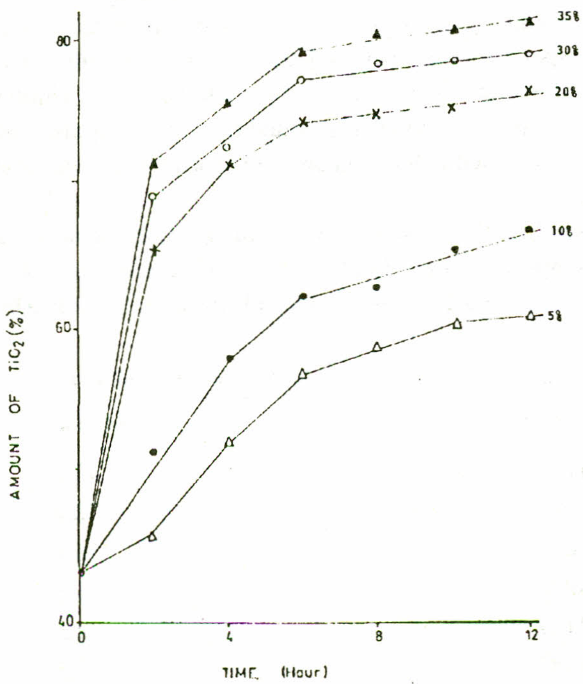

Fig. 1. Change in the amounts of TiO_2 in ilmenite during leaching (90°, 1:20, 300 rpm, 250-350 μm).

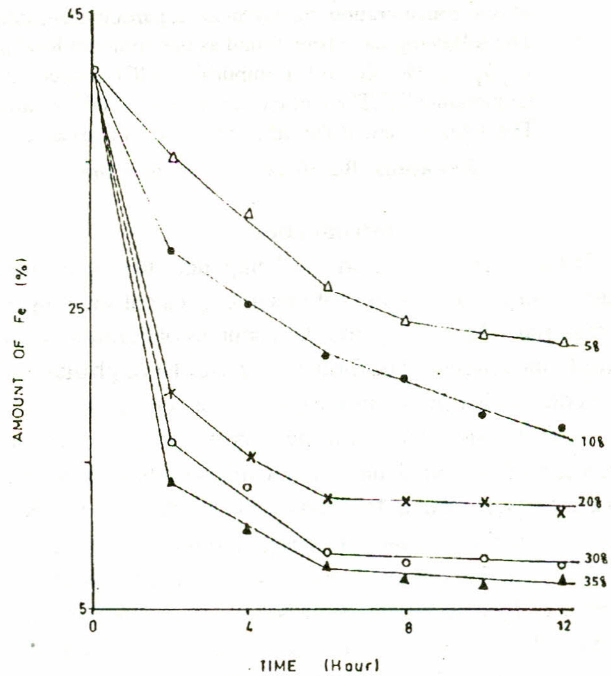

Fig. 2. Change in the amounts of Fe in ilmenite during leaching (90°, 1:20, 300 rpm, 250-350 μm).

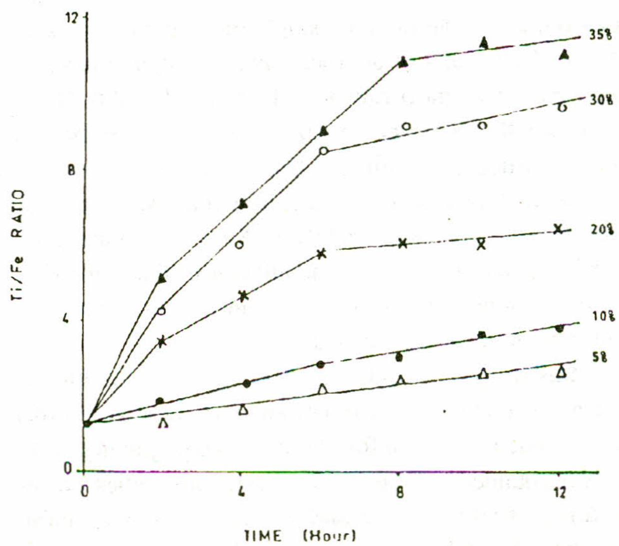

Fig. 3. Change in the Ti/Fe ratios of ilmenite during leaching (90°, 1:20, 300 rpm, 250-350 μm).

ilmenite [10, 13, 19, 20], little attention has been given to particle size, probably due to its insignificant effect on leaching.

By keeping other parameters constant, experiments were carried out at different solid-liquid ratios, result of which is given in Table 2. It is obvious from this table that leaching was influenced by varying the solid-liquid ratio. The degree of leaching was fairly constant between ratios of 1:20 and 1:5, after which it dropped sharply. This observation was so because large liquid volumes cause better dissolution than small ones.

By varying the stirring speed from 200–1000 rpm, while the other parameters remain unchanged, it was observed that agitation plays no serious role in the upgrading of ilmenite. The reason is probably because of the precipitation of some TiO_2 on the ore particles by hydrolysis of Ti^{4+} ions present in the leach solution [21]. Under this condition, the rate of leaching becomes independent of the stirring speed because it is controlled by transport within the solid [26].

TABLE 2. EFFECT OF SOLID-LIQUID RATIO ON LEACHING.

Ratio	Content (%)	
	TiO_2	Fe
1:2	67.3	18.9
1:2.5	68.1	18.2
1:3	70.3	15.9
1:5	78.4	9.3
1:10	78.8	9.0
1:20	79.4	8.3

TABLE 3. EFFECT OF TEMPERATURE ON LEACHING.

Temp. (°C)	Content (%)	
	TiO_2	Fe
50 ± 2	57.1	26.5
60 ± 2	65.4	19.2
70 ± 2	71.3	15.6
80 ± 2	76.2	12.1
90 ± 2	79.4	8.3
100±2	80.1	7.8

TABLE 4. ANALYSIS OF ILMENITE CONCENTRATE.

Constituent	Content%
TiO_2	78.4
Fe	9.3
MnO	0.4
Cr_2O_3	0.2
SiO_2	3.8
Al_2O_3	2.2
CaO	0.4

Finally, the effect of temperature on the degree of upgrading was considered, and it was found that leaching was very rapid at high temperatures, > 70°. However, there was no significant difference in the results obtained between the temperatures of 90 and 100° (Table 3). This suggests that 90°C might be an ideal temperature for leaching the ore in HCl solution.

Conclusion

This work has shown that leaching of ilmenite ore in HCl solution was significantly influenced by the acid concentration, reaction time, solid-liquid ratio and temperature, and the following were obtained as the optimum parameters, respectively; 30%, 8 hrs, 1:5, and 90°. These gave a concentrate containing about 78% TiO_2, 9.3% Fe, and other impurities (Table 4), which will be a suitable starting material for the manufacture of highly pure titanium oxide for paint industries. Iron (III) chloride was recovered from the leach liquor as a by-product.

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