

The Need To Extract And Characterize Titanium Dioxide (TiO₂) From Itakpe Iron Ore Deposits Using Hydrometallurgy And Electrospray Pyrolysis Techniques To Improve Nigeria Gross Domestic Product (Gdp)

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ABSTRACT

At present, Nigeria iron ore only accounts for 0.3% of its Gross Domestic Product (GDP) which is grossly inadequate to meet the needs of the country. The solid mineral sector contributed only about #55.82 billion to the Federal Government, accounting for a mere 4% of the country's total national export earnings. Despite iron ore reserves in Nigeria estimated at 11000 millions tones, the country still import minerals such as salt, iron ore and titanium dioxide that could produce domestically. In this research, Leaching and solvent extraction of total iron and titanium has been studied. A detailed investigation on quantitative leaching of the mineral and separation by solvent extraction were also carried out. The effect of some parameters such as acid concentrations and temperature has been investigated. Experimental results indicate that the dissolution rate is by diffusion control. With 2.0M HCl solution, about 85.4% of the ore was dissolved within 120 min. using solid: liquid ratio of 10g/L at optimal conditions. The calculated activation energy, reaction order and Arrhenius constant were 38.4 kJ/mol, 0.85 and 11.8s⁻¹, respectively. The mineralogical purity by X-ray diffraction (XRD) showed that apart from prominent Ilmenite, (FeTiO₃) peaks, the following compounds: ZnSO₄, SiO₂, CaFeO₇, Fe₂(SO₄)₃, CaTiO₄ and Mn₅O₈ were also present. The results of fundamental studies on solvent extraction of synthetic solutions of total titanium showed that extraction of metal ions increased with increasing extractant concentration. An extraction efficiency of 97% total titanium was obtained by 1.5 M TBP.

Keywords: Titanium Dioxide (TiO₂), Iron-Ore, Gross Domestic Product (GDP).

Background of the Study

Itakpe is a town in Kogi State, Nigeria. The Itakpe Hills is around the Itakpe town and contain very pure deposits of iron ore. The National Iron Ore Mining Company is located here. Its supply the steel work for Ajaokuta and Aladja, as well as producing ore for export. There are rocks and minerals from which metallic iron ore can be economically extracted. The ore are usually rich in iron oxides, and vary in color from dark grey, bright yellow, or deep purple to rusty red.

The impact of iron ore and steel in any economy is usually tremendous because its consumption and production are measures of the rate and levels of industrialization. Iron and steel are most widely used for engineering materials for production. Fabrication, construction and manufacture of most items, in such as ships, military hardware, vehicles airplanes etc. This explains why the per capital consumption of steel is an index of assessing development in the economy of any nation. The availability and development of the iron and steel sector, is essential for industrial growth, increased engineering capacity and enhancement of technical skills (Raw Materials and Development Councils, 2010).

In her bid, therefore, to promote technological growth, characterizations and reducibility tests were carried out on the Itakpe iron ore by Adedeji and Sale (1984). Analysis of Calcium, Magnesium, Iron, Aluminum, and Manganese were carried out through atomic absorption spectrometry; silica was determined by combination of gravimetric and colorimetric methods. X-rays diffraction analysis was performed using cu-ka radiation in a Siemens.

According Goswani (2007), quality of iron ore or agglomerate is judged by the assessment of several properties. Characterization of iron burden can be classified as given below: (a) Micro logical

I. INTRODUCTION

characterization (b) Chemical characterization (c) Physical characterization (d) Physico-chemical or metallurgical characterization.

II. STATEMENT OF THE PROBLEM

Nigeria iron prices slid into the bearish territory in September, 2017 by falling 12% in single week. This is due to mining of minerals such as iron ore which account for only 0.3% of its Gross Domestic Product (GDP), because of the influence of its vast oil resources. The domestic mining industry in Nigeria is underdeveloped, which lead the country to import minerals such as salt, iron ore that could produce domestically.

In 2012, Permanent Secretary of the Ministry of Mines and Steel, said Nigeria is losing N8 trillion (\$50 billion) for gold exploitation alone. According to the 2014 audit report of the Nigeria extractive industries transparent initiative (NEITI), the solid mineral sector contributed only about N55.82 billion to the Federal Government, accounting for a mere four percent of the country's total National exports earning for the period. Similarly, the sector, which is dominated by unskilled artisans, small scale miners, and largely unorganized mining activities.

There is growing need to investigate alternative iron ore of Titanium dioxide (TiO_2) to improve the GDP of Nigeria.

III. AIMS AND OBJECTIVES

The general objectives of the study is to find out how TiO_2 can be extracted from the iron ore deposits found at Itakpe and characterized it using electrospray pyrolysis techniques to improve the GDP of Nigeria.

The specific objectives of the study are as follows to:

1. Investigate if iron ore deposit at Itakpe contain TiO_2 sample.
2. Find out if the TiO_2 sample can be extracted from the ore.
3. Characterize the TiO_2 sample using hydrometallurgy.
4. Verify the optical, structural and electrical characterization of the TiO_2 sample obtained by using the appropriate instruments.
5. Conform the commercial viability of the characterized TiO_2 sample by qualified and quantified it.
6. Map out different location within the region of the ore to investigate the extends the TiO_2 can improve the GDP of Nigeria.

Justification

The National iron ore mining company (NIOMCO) at Itakpe in Kogi State, Nigeria established in 1979 was to supply 2.1 million tons per year of 63/64% Fe grade concentrate to the Ajaokuta steel plant when fully operational. This steel plant with an annual production capacity of 1.3million tones of steel, in addition of meeting national steel requirement was aimed at addressing the problem of unemployment, diversifying the economy and helping to generate foreign exchange (Djuand Agba, 2005). But due to global poor funding and uninstalation of massive infrastructural facilities delayed the completion and full commissioning of this plant which at the time was to be Africa's largest steel making plant. Also, at present Nigeria iron ore are only account for 0.30% for its GDP which is grossly inadequate to meet the needs of the country.

The current pace of development has led to increase in the demand for minerals and their by-products. This has subsequently resulted in rapid development.

However, it become urgent right now to improve this low percentage of Nigeria iron ore that is currently has no significant effect on GDP contributes to its GDP to meet the needs of the country. This can be improved upon when TiO_2 is extracted and characterized from the iron ore of Itakpe and subjected it to vigorous commercial activities.

IV. LITERATURE REVIEW

Until recently, Nigeria has had no significant iron and steel industry. The total steel consumption in 1968 was estimated as 5.5kg per caput. Compared with 650kg per caput for industrialized nations an world average of 150kg per caput (Robinson, 1968). This steel was obtained either by very small scale internal manufacture or by large scale importation. Iron ore reserve in Nigeria have been made to use this indigenous ore, Itakpe iron ore is hematite rich. This minerals being intergrown with magnetite, silica is the majority impurity.

Production, trading and utilization of minerals have become a very important part of human lives. Existence of life without these minerals is almost unimaginable due to their vital contribution to industrial development such as construction and manufacturing industries. Few minerals fuel as Tale, abestic, and sulfur can be used just after mining without processing. However, most of the minerals, especially the more important ones such as iron ore and gold, need to be refined and reprocessed to produce utilizable materials (Klein 2002).

Mahalakshim, (2014) studied the synthesis and optical properties of TiO_2 thin film. In his study, TiO_2 films were synthesized using a simple, less expensive, low temperature and convenient for large area deposition method. A chemical bath deposition (CBD) and their structural and optical properties were examined at various calcination temperatures. Titanium oxides (TiO_2) films have several advantages for application in solar cells and very commonly used as a photo catalyst for degradation in the presence of light with energy gap.

Research Design

In this study, TiO_2 extraction was conducted by reacting iron sand with H_2SO_4 using high temperature of 450 degree. Iron sand now is physically separated into its compound and elements using X-rays Diffractometer (XRD). In this, a chemical reaction occurred between the compound of ilmenite and sulphate acid. The characterization was done using electrospray pyrolysis machine with three headers; temperature controller that control the temperature of the TiO_2 sample, atomization nozzle that dispense the substrate. A detailed investigation on quantitative leaching of the mineral and separation by solvent extraction were also carried out. The effect of some parameters such as acid concentrations and temperature has been investigated. Experimental results indicate that the dissolution rate is by diffusion control. With 2.0M HCl solution, about 85.4% of the ore was dissolved within 120 min. using solid: liquid ratio of 10g/L at optimal conditions. The calculated activation energy, reaction order and Arrhenius constant were 38.4 kJ/mol, 0.85 and 11.8s⁻¹, respectively. The mineralogical purity by X-ray diffraction (XRD) showed that apart from prominent Ilmenite, (FeTiO_3) peaks, the following compounds: ZnSO_4 , SiO_2 , CaFeO_7 , $\text{Fe}_2(\text{SO}_4)_3$, CaTiO_4 and Mn_5O_8 were also present. The results of fundamental studies on solvent extraction of synthetic solutions of total titanium showed that extraction of metal ions increased with increasing extractant concentration. An extraction efficiency of 97% total titanium was obtained by 1.5 M TBP.

Research Questions

The following research questions are formulated to guide the learners to:

1. Would Itakpe iron ore contain ilmenite sand?
2. Would the quantity of TiO_2 found be enough to commercialize?

Hypothesis

The following hypotheses are formulated to guide the research;

- (A). There are large and pure deposits of iron ore at Itakpe
- (B). Iron ore contain sample TiO_2 in large quantity.
- (C). TiO_2 sample can be extracted from iron ore and commercialized.

Research Instruments

The under listed instruments were used to locate, collect, extract and characterized TiO_2 from the iron ore. These instruments includes: magnetic separator, grid type ball mill, pinch valve, wear resistant rubber, hydraulic cone crusher, energy saving ball mill, autogenous mill, hammer crusher, jaw crusher, iron sand ore, H_2SO_4 (p.a), distilled water, HNO_3 (p.a), HCl (p.a).

Research Analysis/Interpretation.

- Five (5) samples of iron ores are obtained from different location within the iron deposits. The samples were subjected to test with the help of the aforementioned instruments. Pure TiO_2 thin films were obtained from the ores and characterized using the electrospray pyrolysis techniques. High titanium slag. Ilmenite is smelted in an electric arc furnace to produce pig iron and high titanium slag which typically contains about 85–90% TiO_2 . Nano-particles of TiO_2 components of visible light were reflected and refracted differentially, leading to the phenomenon of iridescence. Nano-particles of TiO_2

Titanium occurrence and resources

Titanium is the ninth most abundant element in the earth's crust and the fourth most abundant element (Knittel 1983; Minkler and Baroch 1981). Its elemental abundance is about five times less than iron and 100 times greater than copper, yet for structural applications titanium's annual use is 200 times less than copper and 2000 times less than iron. Its commercial production commenced in 1948 driven by the demand from the aircraft industry. World titanium sponge metal production reached 166,000 metric tons in 2008 (Gambogi, 2009a, Gambogi, 2010). The main titanium-containing minerals are rutile, ilmenite and leucocoxene (Table 1). Ilmenite supplies about 91% of the world's demand for titanium minerals and the world's ilmenite production reached 5.19 million metric tons in 2009 (Gambogi, 2009b, Gambogi, 2010).

Table 1 Titanium minerals and their chemical compositions.

Mineral	Composition	TiO ₂ content (%)
Rutile	TiO ₂ (tetragonal, twinned)	~ 95%
Anatase	TiO ₂ (tetragonal, octahedral)	near ~ 95%
Brookite	TiO ₂ (orthorhombic)	~ 95%
Ilmenite	FeO·TiO ₂	40–65%
Leucoxene	Fe ₂ O ₃ ·nTiO ₂	> 65%
Arizonite	Fe ₂ O ₃ ·nTiO ₂ ·mH ₂ O	---
Perovskite	CaTiO ₃	---
Geikielite	MgTiO ₃	---
Titanite or sphene	CaTiSiO ₅	---
Titaniferous magnetite	(Fe·Ti) ₂ O ₃	---

In the 1950s, titanium deposits containing 30% rutile were common. However, by 1981, the content had decreased to less than 1% rutile (Minkler and Baroch 1981). Ilmenite, not only found in beach sands, but also in hard rock deposits, has quite extensive reserves: about 1300 million tons. There is no doubt that ilmenite as the most important titanium resource will continue to dominate the titanium industry in the future.

Ilmenite (FeO·TiO₂ or TiFeO₃) contains 40–65% TiO₂, depending on its geological history. Leucoxene (Fe₂O₃·nTiO₂) is a natural alteration product of ilmenite, typically containing more than 65% TiO₂. Table 1 lists the most common titanium minerals and their chemical compositions (Barksdale 1966; Rhee and Sohn 1990; Whitehead 1983). Rutile contains about 95% TiO₂ and is the most titanium-rich mineral. Its deposits are often found in coastal areas such as beach sands, and are the simplest to mine and concentrate in a form adaptable for metal production.

Titanium products, usages and process overview

The most widely used titanium product is titanium dioxide (TiO₂), being as pigment, as filler in paper, plastics and rubber industries and as flux in glass manufacture. Only about 6% is used to produce metallic titanium (Rosebaum 1982). The major sources of TiO₂ are from:

- Natural rutile. After a series of concentration of beach sands through gravity concentration, electrostatic separation to remove non-conducting zircon materials and magnetic

separation to separate magnetic ilmenite, the rutile concentrate containing about 95% TiO₂ is used as TiO₂ raw material directly (Kahn, 1984).

- Synthetic rutile. There are a few processes to remove the iron from ilmenite to produce synthetic rutile which typically contains about 92% TiO₂.
- High titanium slag. Ilmenite is smelted in an electric arc furnace to produce pig iron and high titanium slag which typically contains about 85–90% TiO₂.

The biggest consumer of TiO₂ is the pigment industry. Unusual optical properties appear when the average particle size of TiO₂ is reduced to < 100 nm, including high transparency to visible light and high UV absorption (Ellsworth et al. 2000). Nano-particles of TiO₂ also cause some components of visible light to be reflected and refracted differentially, leading to the phenomenon of iridescence. Nano-particles of TiO₂ have found applications in cosmetics, porcelains and ceramics industries as coating material and additives. Nano-particles of titanium oxide have received great attention recently for their potential applications in catalysis and as photo-electrochemical material. The high photo-catalytic activity of titanium oxide by irradiation of visible light has been rapidly developed in recent years (Sakatani et al., 2002, Sakatani and Koike, 2003, Sakatani and Koike, 2004).

The processes for production of pigment grade TiO₂ and titanium metal are schematically

presented in Fig. 1. There are two processes to manufacture titanium pigment: the sulphate process and the chloride process. The two processes differ in both their chemistry and raw material requirements (Hamor 1986). Because the chloride process has some advantages over the traditional sulphate process in cost and waste management, it has dominated the pigment industry in recent times. However, unlike the sulphate process, in which low-grade titanium raw material is acceptable, the

chloride process needs a high grade of rutile (Rosebaum, 1982). 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.

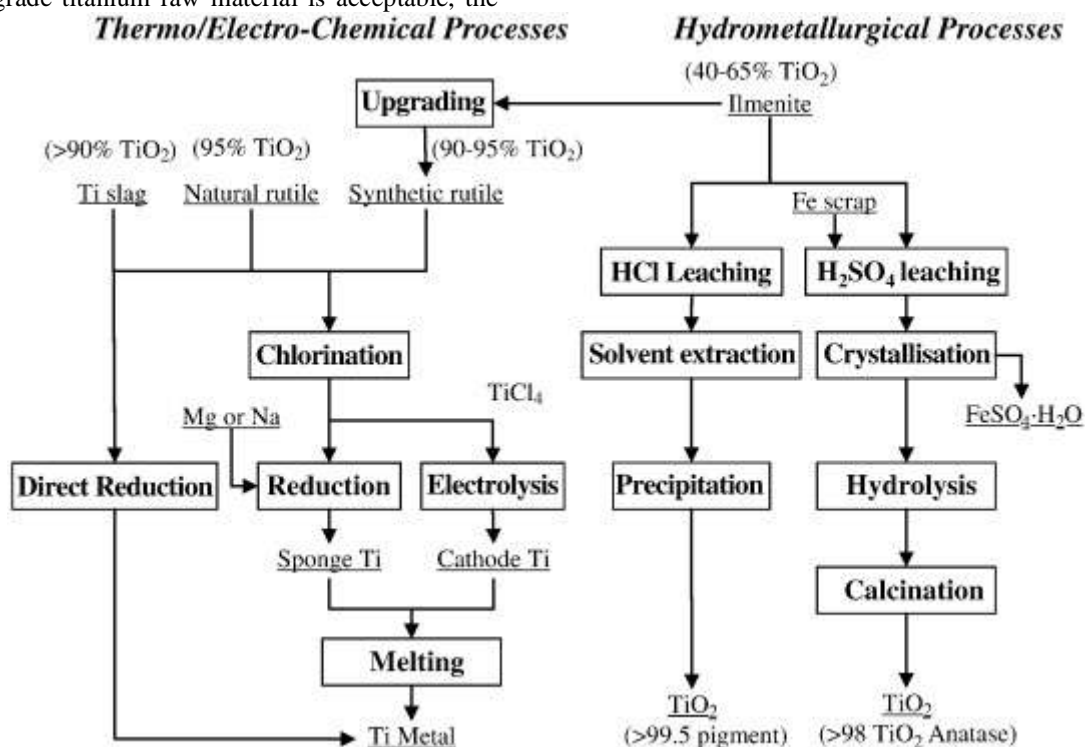


Fig. 1. A schematic of processes for the production of Ti metal and pigment TiO₂.

V. CONCLUSION

Various titanium metallurgical processes have been reviewed and used for titanium dioxide and titanium metal, mainly focusing on the future development of hydrometallurgical processes. Prepared and fabricated TiO₂ nanopowders thin films by pyrolysis method in the temperature range of 400 – 600°C. The nano particles have a solid spherical after a series of concentration of beach sands through gravity concentration, electrostatic separation to remove non-conducting zircon materials and magnetic separation to separate magnetic ilmenite, the rutile concentrate containing about 95% TiO₂ is used as TiO₂ raw material directly (Kahn, 1984).

VI. RECOMMENDATIONS

- i. Direct hydrometallurgical leach processes are advantageous in processing abundant ilmenite ores, low energy consumption and produce

sufficiently high quality of pigment grade TiO₂ products for a wide range of applications and major demand.

- ii. Since ilmenite is becoming increasingly important due to the rapid depletion of natural rutile. Many processes are commercially used or proposed to upgrade ilmenite to synthetic rutile. Most of these processes involve a combination.
- iii. Caustic leach with high selectivity and titanium dioxide nano-technology has also been developed. Further development of direct leaching ilmenite coupled with solvent extraction for titanium pigment and metal production, is recommended.

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