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Extraction and Characterization of Rutile from Mining Gauge

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Abstract
The progressive exhaustion of principal sources to acquire metals has led to the quest for other sources for their recovery. In the precise case of titanium, titaniferous sands are a practicable option for obtaining this metal. Titanium oxide, also known as titanium (IV) oxide, or titanium, is the natural form of oxidized titanium. Titanium dioxide is commonly detected in three forms of anatasis, rutile, and brookite. The methodology for the concentration of titanium oxide was based in industrial extraction of titanium ores and consists of solubilizing the impurities present in the sample by means of chemical attack. It has been determined experimentally that at temperatures around 70°C, hydrochloric acid provides a better solubilization of impurities, without extracting titanium, and it is possible to obtain a residual solid phase with about 58.5% of titanium on it. The results were determined by using the Energy Dispersive X-Ray Fluorescence (EDXRF) technique.

Keywords: *titanium; titaniferous sands; solubilizing; concentration*

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I. Introduction

Demand for minerals and metals are increasing most especially from waste. There are several minerals in some of these wastes that are very valuable and important for economy and for technological development. An example is a residue derived from a step of physical handling of the phosphate rock, containing about 30% of titanium in its composition [1].

Titanium is the ninth most abundant chemical element in the earth's crust, mainly found in the oxide form, and which ilmenite and rutile are the major mineral raw materials for its extraction [2].

Titanium dioxide (TiO2) is non-toxic, recognized as safe for human food, drugs, cosmetics, and food contact materials. The natural source of oxidized titanium is titanium oxide, also known as titanium (IV) oxide, or titanium [3, 4].

Titanium can be used in various applications, such as a component in high-tech metallic alloys, or, in the form of titanium dioxide, which is the form that presents the greatest value in industry. The most remarkable use of titanium is in the dioxide form, covering most of industrial applications such as the manufacture of paints, paper production, rubber, textiles, plastics, and other products worldwide manufacturing.

This study focus at the concentration of titanium oxide present in a residue from the mine waste, by attacking the material chemically and doing its characterization by analytical methods. This would contribute and increase to the national titanium concentrated production, by transforming an unused waste in a high value product.

Experimental Procedure

A total of 15 tailing samples were collected from different locations in fifteen townships in Osun State, Nigeria. The samples were kept in air tight polyethylene bags. In the laboratory, all the samples were air-dried at room temperature, pulverized and passed through a 0.8 mm sieve.

The residue from gauge, which is in the solid form, was subjected to a sequence of solubility experiments with temperature around 70 °C and with constant stirring, using sulfuric acid, hydrochloric acid, nitric acid and sodium hydroxide in order to concentrate the titanium dioxide present in the sample [5,6]. The metal contents of the gauge samples and the liquid and solid phases obtained were analyzed using Proton Induce X-Ray Emission (PIXE) with 30 MeV proton beam obtained from Centre for Energy Research and Development (CERD) ion beam analysis (IBA) facility, with a beam spot of 4mm in diameter and a low beam current of 3-6nA . The irradiation was for about 10-20 minutes. A Canberra Si (Li) detector model ESTX 30-150, beryllium thickness of 25mm, with full width half maximum (FWHM) OF 150eV at 5.9KeV, with the associated pulse processing electronics, and a Canberra Genie 2000 (3.1) MCA card interfaced to a PC were used for the x-ray data acquisition with respect to the beam director, the sample's normal was located at OO and the Si (Li) detector at 45O. The PIXE set-up was calibrated using some pure element standards and NIST geological standard NBS278.

SAMPLE	Ti	Mn	Fe	Cu	Zn
1	13034±25.7	889±19.70	83656±111.20	315±4.35	74±2.34
2	9560±22.30	311±6.50	81543±20.10	53±4.20	74±2.30
3	12766±21.08	479±5.50	101087±18.0	387±5.60	63±1.20
4	16451±18.46	563±7.60	62487±14.8	486±5.60	229±19.95
5	10071±11.45	546±5.50	144010±40.00	580±10.10	195±4.00
6	9797±5.70	970±5.40	43340±16.9	173±6.57	72±3.20
7	14900±18.99	589±6.00	44842±12.20	96±4.20	100±5.80
8	6105±17.10	220±6.20	54142±15.00	177±5.60	66±3.60
9	280±12.60	175863±77.29	293±12.70	77±6.20	0.00
10	9157±17.10	214±3.00	15795±13.30	21±4.60	28±2.60
11	11097±12.20	1725±47.28	105063±10.20	508±11.30	171±4.50
12	3715±13.20	139±2.50	43217±31.87	48±2.20	0.00
13	13298±18.80	1406±32.63	74279±20.49	257±11.20	179±3.20
14	10428±12.84	659±7.12	45589±12.20	162±12.20	52±2.30
15	5797±11.20	267±10.10	30715±13.40	142±6.20	39±2.34

Table 1: Result of Elemental Profile (mg/kg) of Mine Gauge Samples

II. Results and Discussions

The results from Table 1 above shows the concentration of analyzed elements from the raw samples. Concentration of heavy metals in gauge samples collected from mining gauge showed significant variation. The variation in heavy metal concentrations in vegetables collected from the same farm may be ascribed to their morphological and physiological differences in uptake, exclusion, and accumulation of heavy metals [7].

Sample with highest concentration of titanium were then subjected to solubilisation using H_2SO_4 and HCl of varying concentration of 2M and 4M respectively.

After chemical treatment with constant heating and stirring, the residual solid phase of each trial was analyzed using the proton induce X-ray emission system. Table 2 shows the results of this analysis, where one can observe that the 4 M hydrochloric acid showed the best results, because the solid phase of this trial showed a lower proportion of iron and a higher proportion of titanium when compared with the original sample. The proton induce X-ray emission results compares the liquid and solid phases after the attack with sulphuric and hydrochloric acid. It is possible to verify that titanium was concentrated in the solid phase, as well as manganese, ferrous, cuprous and zinc.

Table 2: Solid Proton Induce X-Ray Emission (PIXE) Results

%	Original	H_2SO_4 2M	H_2SO_4 4M	HCl 2M	HCl 4M
Ti	20.51	26.20	28.70	43.50	49.60
Mn	0.70	0.20	0.24	0.35	0.48
Fe	77.90	64.50	62.10	46.50	45.10
Cu	0.60	0.50	0.68	0.45	0.50
Zn	0.29	0.10	0.12	0.03	0.02

III. Conclusion

In this work, Energy Dispersive X-Ray Fluorescence and diffraction techniques was used to study the concentration of Ti from mine gauge after several chemical process stages. According to the composition and the physic-chemical characteristics of the magnetic gauge sample, it was possible to perform treatment using hydrochloric acid in order to concentrate the titanium present in the sample. The analysis of Xray fluorescence and EDS showed that it was obtained a phase rich in titanium and ferrous.

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