GEOCHEMICAL AND MINERALOGICAL CHARACTERIZATION OF ORE – MINERAL ASSEMBLAGES FROM BLACK SANDS IN AKURE SOUTH AREA, SOUTH - WESTERN NIGERIA

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ABSTRACT
The geochemical and mineralogical features of Black sand in Akure South was investigated. The head samples were sourced from four different locations and mixed properly for homogenization. 200 g crude sample was sampled out, finely ground using the Denver Ball Mill, Model: Denver D10 and prepared towards characterization. Chemical Characterization of the crude sample was carried out using Energy Dispersive X-ray Fluorescence Spectrometry (ED-XRF) - Minipal 3. Mineralogical Analysis was carried out using Scanning Electron Microscopy with Energy Dispersive X-Ray Spectrometer (SEM-EDS) and X-ray Diffraction Analysis (XRD). ED-XRF analysis revealed that the crude sample contains 17.55% Fe₂O₃, 63.7% SiO₂, 15.5% TiO₂, and other compounds in trace form. Mineralogical analysis of the black sand via XRD revealed the present of Quartz (87.07% SiO₂), Magnetite (0.59% Fe₃O₄), Ilmenite (9.89% FeTiO₃) with other associated minerals such as Rutile (0.53% TiO₂), Anatase (0.25% TiO₂), Kaolinite (0.09% Al₂O₃(SiO₂(OH)) and Albite (1.57% Na(AlSi₃O₈)). SEM images revealed interlocking of some minerals within the crystal aggregates of the crude black sand and the EDS of these microstructures revealed the presence of Al, Mn, Cu, O, Si, C, Fe and Ti; silicon, titanium and iron are the major elemental constituents of the matrix.

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1.0 INTRODUCTION

Nigeria is endowed with varieties of solid minerals which are distributed in all geo-political zones; to mention but a few of these zones are Jos, Bauchi, Kebbi, Enugu, Akwa Ibom and Oyo, Abakaliki, Isanlu Isin and Mandaka. Solid mineral endowment are fuels such as lignite, bitumen, coal, uranium, thorium, etc.; ferrous and ferro-alloys minerals which include haematite, magnetite, siderite, ilmenite, manganese, nickel, chromium etc., non-ferrous minerals such as bauxite, sphalerite, galena, chalcocytite, cassiterite, etc.; minor metallic and related non-metallic minerals – antimony, cadmium, zirconium, etc.; precious metals- gold, silver, etc.; industrial minerals – limestone, asbestos, gypsum, quartz etc.; and gem stones – emerald, amethyst, ruby, sapphire, etc. (Muduaka, 2014). The occurrence of these minerals in nature is regulated by geological conditions throughout their life span. These geological conditions such as magmatic, hydrothermal, sedimentary and weathering processes, tends to selectively concentrate desirable metallic elements in a particular mineral
deposits to enable such metals to be viable for profitable exploration and exploitation (Lucas et al., 2014).

However, prior to exploration and exploitation of these minerals, mineral characterization must have been carried out to ascertain their chemical and mineralogical characteristics (Hope et al., 2001). Characterization of a mineral provides technical data towards justifying its economic viability and the selection of process route(s) essential for optimum recovery of the valuable mineral from the gangue (Martin et al., 2011). Furthermore, mineral characterization form an integral and often crucial part in deposit investigations (Cook, 2000).

1.1 Nature of Akure South Mineral (Black Sand) in its Crude

The occurrence of Akure South mineral in its crude is of alluvial form. It occurs unconsolidated and deposited as a stratum on quartz. This deposit is mostly seen in drenches, grooves, ditches and on roads present within Akure South Local Government Area of Ondo State, Nigeria as shown in Figure 1. The occurrence of this mineral has been known for years by locals dwelling there as a natural phenomenon that do occur during rainy season, some even tagged it “blacksand or blank ants” (Olatunji et al., 2017). It occurs within the Longitude of 7.29074 and Latitude 5.160636 with total area of occurrence estimated at 175 square meters.

Nevertheless, the distinct appearance and yearly occurrence of this mineral initiated the drive carry out mineral characterization towards ascertaining its composition and mineralogical characteristics. This will provide technical data tailored towards ascertaining the mineral geological nature, estimated reserves and consequently determination of possible process route(s) to be adopted towards exploration and exploitation of this rock sample.

2.0 Research Methodology

2.1 Material Sourcing

The mineral samples were sourced from three different locations, namely Obanla, Orita - obele and Road block area, which are all regions within the Akure South Local Government Area of Ondo State, Nigeria. The samples were collected using random sampling method. The collected samples were properly mixed for homogenization and weighed as 50 kg.

2.2 Sample Preparation

200 g of the crude sample was sampled out via cone and quartering sampling method and properly homogenized. The homogenized sample was finely ground using Denver Ball Mill (Model: Denver D 10) to 100% passing -300+250 μm, homogenized and prepared towards chemical and mineralogical characterization.

2.3 Chemical Characterization
Chemical characterization of the crude was carried out using Energy Dispersive X-ray Fluorescence Spectrometer (PANanalytical Minipal 7). 20 g of the sample was finely ground to pass through a 200-250μm mesh sieve. Thereafter, the sample was intimately mixed with a binder in the ratio of 5.0 g sample(s) to 1.0 g cellulose flakes binder and pelletized at a pressure 5 of 10-15 tons/inch² in a pelleting machine. At this stage, the pelletized sample(s) are stored in a desiccator for analysis. The ED-XRF machine was switched on and allowed to warm up for 2 hours. Finally, appropriate programs for the various elements of interest were employed to analyze the sample material(s) for their presence or absence. The result of the analysis was either reported in parts per million (ppm) or percentage (%) for minor and major concentrations of elements.

2.4 Mineralogical Characterization via XRD

Qualitative and Quantitative determination of the nature of the phases and the amount of the phases present in the sample were determined by a PANalytical Empyrean diffractometer with PIXcel detector and fixed slits with Fe filtered Co-Kα radiation. The material was prepared for XRD analysis using a backloading preparation method. The phases were identified using X’Pert Highscore plus software. The relative phase amounts (% weight) were evaluated using the Rietveld method.

2.5 Mineralogical Characterization via SEM-EDS

Morphological and qualitative analyses of the bulk ore were performed using SEM-EDS. The SEM provides information on spatial distribution of mineral phases present in the crude, while EDS provides information on their elemental composition (Olubambi et al., 2008). Mineralogical analysis via SEM-EDS was conducted on representative samples in two stages using SEM (Model: JEOL 840). All the samples were carbon coated in order to make the mineral’s surface conductive. Samples for analysis were cut, polished mounts in embedded epoxy resin, and finally polished to obtain a mirror-like surface. The polished surfaces were finally carbon coated before analysis. Qualitative chemical analysis of the samples was carried out using EDS detector attached to the SEM.

3.0 Results and Discussion

3.1 Results

Results obtained along the course of this research work were presented in Table 1 and Figure 1 respectively.

<table>
<thead>
<tr>
<th>Sample/Assay %</th>
<th>Al₂O₃</th>
<th>SiO₂</th>
<th>SO₃</th>
<th>K₂O</th>
<th>CaO</th>
<th>TiO₂</th>
<th>V₂O₅</th>
<th>MnO</th>
<th>Fe₂O₃</th>
<th>ZnO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crude</td>
<td>BDL</td>
<td>63.7</td>
<td>0.2</td>
<td>0.691</td>
<td>0.23</td>
<td>15.5</td>
<td>0.38</td>
<td>0.29</td>
<td>17.55</td>
<td>0.009</td>
</tr>
</tbody>
</table>

BDL – Below Detectable Limit
Figure 1: XRD pattern of Akure South Earth sipped Black Sand (mineral) showing the diffraction peaks of the major minerals present with their respective chemical formulae and phase amounts (wt. %)

Figure 2: Scanning Electron Microscopy (SEM) Micrograph of the head sample, showing (a) Holistic analysis, (b,c) Point Analysis
Figure 3: Energy Dispersive X-ray Spectroscopy (EDS) peaks for the various elements present in the head sample SEM Micrographs for (a) Holistic analysis, (b,c) Point analyses
3.2 Discussions

3.2.1 Chemical Analysis of the Crude
Table 1 shows the result of chemical analysis carried out on the collected head sample of Akure black sand. Chemical analysis of the crude via ED-XRF revealed that the chemical composition of the crude Akure earth sipped mineral (black sand) contains 17.55 % Fe₂O₃, 63.7 % SiO₂, 15.5 % TiO₂, and other compounds in trace form, thus confirming the presence of iron in the crude and the mineral being ferromagnetic in nature. This also certified the standard of 1-5% iron requirement in an ore to be tagged an iron bearing mineral (Weiss, 1985; Yaro and Thomas, 2009).

3.2.2 Mineralogical Characterization of the Crude via XRD
Figure 1 shows the XRD pattern of the crude sample of Akure South mineral, the peaks of the various mineral compounds present and their respective phase amounts. The minerals present in the ore and their respective weight percent are as follows; Quartz (87.07 % SiO₂), Magnetite (0.59 % Fe₃O₄), Ilmenite (9.89 % FeTiO₃), Rutile (0.53 % TiO₂), Anatase (0.25 % TiO₂), Kaolinite (0.09% Al₂(Si₂O₅)(OH)₄) and Albite (1.57% Na(AlSi₃O₈)). The diffractogram revealed that ilmenite and quartz are predominant when compared with other minerals in the ore matrix. The predominance of ilmenite is attributed to the mineralization of the mineral of iron and titanium during formation of the ore (black sand) (Sewertmann and Cornell, 2006).

3.2.3 Mineralogical Characterization of the Crude via SEM-EDS
Figure 2 shows the Scanning Electron Microscopy (SEM) micrograph of the crude sample at 10 microns while Figure 3 shows the detailed EDS analysis data of the crude sample. The SEM micrograph revealed the interlocking nature of minerals within the crystal aggregates in the ore matrix. From this, it can be observed that the minerals are coarsely packed as such easy liberation via comminution is facilitated; because of the more coarsely packed the minerals the easier their liberation (Weiss, 1985; Wills, 2006). Holistic and point analyses of the mineral phases present within the ore matrix were done using EDS technique. The locations of the analyzed zones and EDS peaks of the determined elemental compositions with their relative amounts are as shown in Figure 1 and 2 respectively. Holistic analysis of the crude revealed the presence of C, Al, Cu, O, Fe, Ti, and Si. The identified carbon element is probably due to the carbon coating done on the sample during prior to analysis in order to make the mineral surface conductive. However, further point analyses carried out revealed that iron, titanium, and silicon are the predominant elemental constituents of the ore matrix. The result obtained further compliment the analyses done on the crude sample using ED-XRF and XRD and also reveals that Akure South black sand (mineral) contains iron as the mineral of interest in association with other minerals that can hinder the utilization of the ore unless the impurities are either reduced or removed by processing. That is, associated impurities are separated or reduced to a minimal level by separation techniques such that the proportion of the mineral of interest in the concentrate increases while it decreases in the tailing (Yaro and Thomas, 2009).

4.0 CONCLUSIONS
On premise of the experimental results obtained along the course of the research and their discussion, the following conclusions were drawn;

i. The Geochemical characterization of the crude Akure South mineral via ED-XRF revealed that it is an iron-bearing mineral assaying 17.55% Fe₂O₃; and

ii. The mineralogical characterization of the crude Akure South mineral XRD revealed that it contains Quartz, Magnetite, Ilmenite, and other trace minerals. SEM image revealed that these minerals are interlocked within the crystal aggregates of the crude and
the EDS analysis at different spectrum revealed the presence of C, Ti, Al, Mn, Cu, and O within the mineral matrix; such that iron, titanium, and silicon are the major elemental constituents of the matrix.

iii. Finally, Akure South black sand can be concluded on to be another source of Iron ore which when process to a metallurgical grade can be used in Iron and Steel production.

RECOMMENDATION
On account of these findings, keen interests should be developed towards investigating the geological nature and the estimated quantity of Akure South mineral for the purpose of ascertaining its viability.

REFERENCES


