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A Preliminary Survey of the Lithochemistry of Tantalite Mineral from Granitic Pegmatite Deposits in the Okpella Area, Northern Edo State, Nigeria

Okuo, James Majebi[1] and Agho, Timothy Imuetinyan[2*]

[1] Analytical Research Laboratory, Department of Chemistry, Faculty of Physical Sciences, University of Benin, Benin City, Nigeria
[2] Department of Science Laboratory Technology, Faculty of Life Sciences, University of Benin, Benin City, Nigeria

*Corresponding author: Timothy I. Agho (timothy.agho@uniben.edu)

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Abstract

This study investigates the lithochemistry of tantalite mineral from granitic pegmatite deposits in the Okpella area, Northern Edo State, Nigeria. Tantalite, a valuable mineral containing tantalum and niobium, was identified within these pegmatites. It has been assessed through a comprehensive evaluation of the mineral, including determination of its composition, physical characteristics and anionic behaviour employing standard analytical techniques. The X-ray fluorescence (XRF) analysis revealed a significant presence of tantalum oxide, Ta_2O_5 (41.72%) and other useful oxides of niobium, Nb_2O_5 (11.98%), titanium, TiO_2 (10.02%), iron, Fe_2O_3 (17.94%), magnesium, MgO (6.00%), silicon, (4.66%), manganese, MnO_2 (2.75%), and aluminium, Al_2O_3, (1.52%) with impurity elements such as calcium, potassium, chromium, sodium, phosphorus, tin, tungsten and thorium also observed and evaluated in the ore. The X-ray diffraction (XRD) analysis shows the compositions of the minerals ore with tantalite having 49.69%, while associated minerals like quartz (10.28%), ilmenite (16.23%), tantalite/quartz (11.73%) and the least pyrite (4.35%) in their concentrated form. Physical properties investigated showed high resistance on ignition (LOI 2.39%) and low alkalinity (6.48), grey colour, specific gravity range "7.2 – 8.0"and particle size range of "0.063 – 0.150" mm. The X-ray diffractometry spectrum also confirmed the ore to exist in a single phase with tantalite characteristics peaks at "2.56 A^O, 2.39 A^O, 1.76 A^O, 1.50 A^O and 1.43 A^O".

Keywords: Tantalite, Tantalum, Okpella, EDXRF, XRD

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1. Introduction

Tantalite is the most important mineral form of tantalum, a specialty metal used mainly in the electronic industry for the manufacture of capacitors, aerospace components and in several specialty alloy applications (Alhassan *et al*., 2010; Adetunji *et al*., 2005). Pure tantalite is rare, as the presence of elements such as Fe, Mn, Ti, W, Th and Al constitute impurities and vary considerably in their relative proportions (Cerny *et al*., 1992; Magdalena *et al*., 2022). The principal ore of tantalum occurs chiefly as accessory minerals disseminated in granitic rocks or in pegmatite associated with granites (Baba *et al*., 2018). Beneath the surface of the earth in Nigeria lies a vast array of mineral resources, which include deposits of coal, cassiterite, columbite, marble, limestone, clay, bitumen and tantalite (Adetunji *et al*., 2005; Aliyu, 1996). The mining industry is constantly exploring for new mineral deposits while some of these minerals are currently mined, others have the potential to revolutionize industries and change the country's narrative. Nigeria has renewed focus on unlocking the potential of its solid minerals wealth is expected to result in a manifold increase in development activities, including exploration, mine development, processing facilities and downstream industries (Adetunji *et al*., 2005;

Aliyu, 1996). Every unprocessed ton of Nigerian mineral ore represents lost wealth. Transforming these resources into finished products or even semi-finished goods would add significant value to the country's economy (PwC, 2023; NBS, 2015; NEITI, 2020; MSMD, 2003). Studies conducted by the Raw Materials Research and Development Council (RMRDC) have shown that the need to develop capacity for processing of these minerals into intermediate products is the most important requirement of the solid mineral sector in the country (Adetunji *et al.*, 2005; Aliyu, 1996). Developing domestic processing capacity would create jobs, boost economic growth, and reduce Nigeria's reliance on imported goods (NEITI, 2020).

Central Nigeria boasts pegmatite deposits where cassiterite, the heart of tin, and tantalite frequently find themselves intertwined, however, separating these valuable minerals has traditionally relied on rudimentary methods like hand sorting and gravity concentration. These methods were labour intensive and limited in efficiency (Ebikemefa, 2020). The recovery of tantalite in Nigeria as a by-product of cassiterite mining began in the early 1940s (Miller, 1959; Kock and Paschen, 1989; Kolawole *et al.*, 2017). The recovered tantalite concentrates were typically exported, especially to Europe for further processing and refining (TMF, 2022). By the mid-1940s, technological advancements led to the introduction of more efficient recovery techniques. Jigging, tabling and magnetic separation methods became more prevalent, leading to improved yields and purity of the tantalite concentrate (NEITI, 2020; Ebikemefa, 2020). The 1950s and 1960s saw a significant increase in tantalite production in Nigeria, fueled by rising global demands for the metal used in electronics and capacitors (Lindagato *et al.*, 2022). However, challenges emerged due to its demand, which include smuggling of tantalite because of the disparities in local prices compared to international markets. The lack of domestic processing facilities also limited profitability for local miners (Lindagato *et al.*, 2022).

Tantalite ore has a higher concentration of Ta_2O_5 than Nb_2O_5, while columbite ore has a higher concentration of Nb_2O_5 than Ta_2O_5. Numerous studies have been carried out on tantalite ore. Burt (1996) conducted a study on the beneficiation of tantalite ore and noted that concentrates suitable for further processing to recover Ta are generally required to exceed 25% Ta_2O_5, with 50% combined Ta_2O_5 and Nb_2O_5. Ores with these Ta_2O_5 and Nb_2O_5 levels solely require cleaning operations for Ta extraction processes. The cleaning operation will result in the removal of associated minerals and impurity elements to a bearable measure. Co-occurring minerals include zircon, rutile, monazite, cassiterite, ilmenite, garnet, uranium and thorium minerals, beryl, spodumene, tourmaline and in some cases, aquamarines and gold: light minerals such as quartz and feldspar may also be present due to inefficient primary concentration. Adetunji *et al.* (2005) researched the features of tantalite ores from key locations in Nigeria. In their study, eight well-documented tantalite location deposits in the country were analyzed with 109Cd excitation source Energy Dispersive X-ray spectrometry using the emission-transmission method of quantification. The geochemical signature minerals include TiO_2, MnO, Fe_2O_3, Ta_2O_5, Nb_2O_5, WO_3, Th and U. Impurity elements such as Hf, Zn, Zr, Co, Pb, Rb and Y were also observed and evaluated. Their investigation further revealed that Ta_2O_5 in the ores ranged from a minimum concentration of about 8% in Out to about 60% in the Egbe deposits, while the Nb_2O_5 concentrations in the ores ranged from about 20% to 37.5% with the highest coming from the Ofiki deposit. Analysis from their results suggests simple cleaning could suffice for Ta extraction from most tantalite deposits in Nigeria. Baba *et al.* (2005) studied the kinetics of the dissolution of a Nigerian tantalite ore in hydrochloric acid. They investigated the effects of temperature, acid concentration, stirring rate and particle size on the dissolution of tantalite ore; and properties like moisture content, loss of mass on ignition, pH and the elemental analysis by inductive coupled plasma-mass spectrometry (ICP-MS). The results show about 67.2% of tantalite ore was dissolved within 120 mins, using 0.1 mm particle size and stirring rate of 270 rpm at 800 ^oC which indicated that the dissolution reaction is lattice-controlled and is greatly influenced by hydrogen ion H^+ concentration. Alhassan *et al.* (2010) researched on EDXRF analysis of tantalite deposits of Mai-Kabanji, Northwestern Nigeria. They studied the elemental compositions by EDXRF, physical properties and anionic composition by standard methods. The results confirmed high concentrations of tantalum oxide, Ta_2O_5 "31.99%±0.83" and other commercially valuable oxides of niobium, Nb_2O_5 "0,029%", titanium, TiO_2 "1.702% ±0.42" and iron, Fe_2O_3 "1.702% ±0.30" were also high. Physical properties tested showed high resistance on ignition (LOI 3.00%) and low alkalinity (8.51), grey colour, specific gravity range (7.2-8.0) and an average size of 0.12 mm. the sample was generally richer in tantalum oxide and other valuable mineral oxides of niobium, titanium, iron and manganese compared with other investigations reported, hence, it is economically valuable for exploration.

The occurrence of tantalite ore is scattered across several parts of Nigeria and the major ones include Kaduna State (Abia Hill, Tare Naudu, Gerti station); Nassarawa State (Wambe Denga, Akwanga, Kokona-ndegi, Agwa-Doka); Federal Capital Territory (Takwashera, Bize, Kusaki); Kogi State (Okene, Akuta, Ejuku, Takete-ishanlu Isa and Egbe); Kwara State (Oke-Onigbin) and Edo State (Okpella). Okpella, located within the Benin-Nigeria-Cameroon (BNC) shield, is renowned for its intricate geological formations and abundant mineral diversity (Obaje, 2009; Ogunyele *et al*., 2020). The region features granitic pegmatite deposits known to host minerals such as tantalite, columbite, tin, and others (Ogunyele *et al*., 2018; Ogunyele *et al*., 2020). Mining operations in Okpella have a longstanding history, predominantly comprising artisanal and small-scale activities that have been a cornerstone of the local economy, supporting livelihoods in the area (Oramah *et al*., 2015). The mining sector in Okpella confronts various challenges including environmental degradation, insufficient infrastructure, and informal mining practices (Vanguard News, 2015). Nonetheless, there are prospects for fostering sustainable mining practices, promoting economic growth, and empowering communities through responsible mining initiatives (Adetunji *et al*., 2005). This paper delves into the elemental and mineral makeup of tantalite deposits in granitic pegmatite rock in Okpella area of Edo State, Nigeria. By utilizing Energy Dispersive X-Ray Fluorescence (EDXRF) spectrophotometry and X-Ray diffraction (XRD) techniques, physical properties and anionic composition of the mineral ore were obtained.

2. Materials and Methods

2.1 Sample collection and Pre-treatment

The sample- material used in this research work was obtained from Okpella in Etsako East Local Government Area of Edo State; Situated along the Benin-Abuja federal highway, Okpella is a dynamic clan in Etsako East tracing its lineage back to Benin. The ores were received from local miners in the study area, packed in plastic container and brought to the Analytical Research Laboratory, University of Benin, Benin City, Nigeria where the sample was manually crushed before gravity concentration of the ore-minerals. The chemical reagents used were of analytical grade from Avantor Sciences and were used without any further purification process.

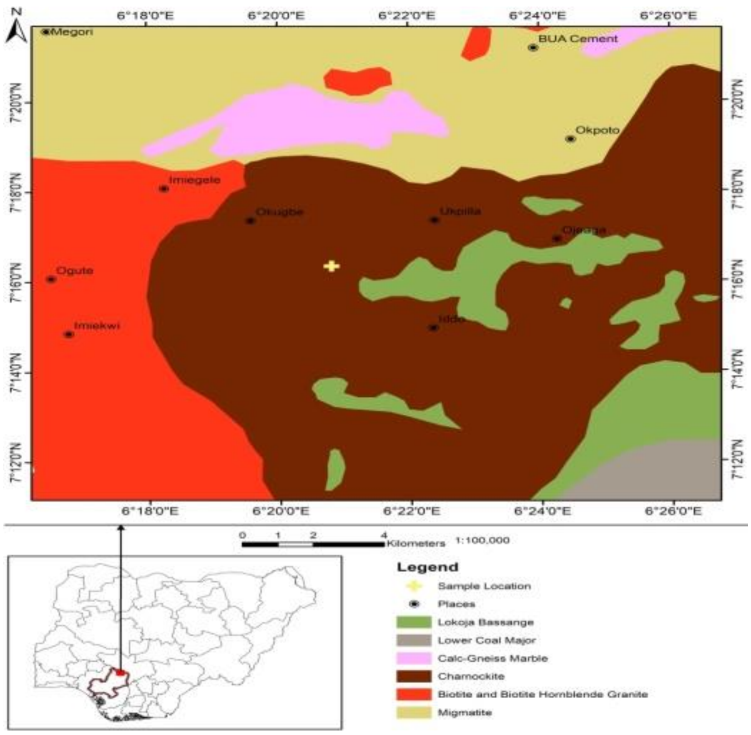

Figure 1. The Geological map of the tantalite ore deposit site (Zurmotai, 2020).

2.2 Sample Preparation for EDXRF and XRD

The EDXRF and XRD analyses were carried out at the Centre for Energy Research and Training (CERT) "Ahmadu Bello University, Zaria", Nigeria using a 702HS Shimadzu EDXRF spectrometer and a 2400H Shimadzu XRD diffractometer. The procedure reported by Alhassan *et al*. (2010) and Adetunji *et al*. (2005) were followed. The pulverized sample (0.5 g) was mixed with 3 mg of an organic binder (polystyrene dissolved in toluene) and homogenized. This was pressed at 10 tons with a hydraulic press (SPECAC) to form a pellet of 19 mm diameter die. The pellets were introduced into the energy dispersive spectrophotometer consisting of an annular 25 m ci Cd 109 isotope as the excitation source emitting Ag-K x-ray (221 KeV) and a Mo X-ray tube (50kV, 5mA).

2.3 Loss on Ignition

The pulverized sample, (1.0 g) was placed in platinum crucibles at a temperature of 25 ^{o}C and fired in a laboratory muffle furnace operated at 1000 ^{o}C for 2 h, then removed and cooled in a desiccator for 10 min before the final weights were taken (Alhassan *et al*., 2010). The loss on ignition was calculated as:

$$\text{LOI} = \text{Weight before firing} - \text{Weight after firing}$$

2.4 pH determination

Each grounded sample was weighed (5 .0 g) to an accuracy of 0.001 grams and dissolved in 10 cm^3 of distilled water and stirred vigorously to ensure homogeneity. The pH was measured after calibration (Alhassan *et al*., 2010; AOAC, 2000; Agho & Archibong, 2023).

2.5 Determination of anions

The pulverized sample, after digestion was analysed for the following anions, chloride, phosphate, nitrate and sulphate. Standard method was adopted for the analyses (AOAC, 2000).

3. Results and Discussion

The results of the elemental, oxide composition and mineral analysis revealed valuable insights into the composition of the material from the Okpella sample are shown in **Tables 1** and **2**. The result revealed high percentage of tantalum (34.17%), iron (12.55%), niobium (8.38%), and titanium (6.01%). Percentage of other elements are less than 5. Their oxides were also observed which exhibited a similar trend. Elements like thorium and tungsten exist as impurities in the ore sample and their presence could be used to design a separation method for their extraction and applications for radioactive purposes based on their economic values. **Table 2** shows the crystalline structure of the ore sample whose chemical composition is dominated by Ta, Fe, Ti, Nb, Mg, and Si indicates ore mineral constituents of a mixture of tantalite [$(Ta, Nb)O_2$], ilmenite ($FeTiO_3$) , Pyrite (Fe,Mn)S and Quartz (SiO_2). The niobium and Manganese, by their respective affinity for tantalum and iron are probably incorporated into the tantalite and pyrite phases respectively based on their chemistry. The ilmenite, pyrite and quartz give information on the reducing conditions of formation of the ore while the thorium value points to its lithosphere origin according to Baba *et al*. (2008). Okunlola (2006), stated that tantalite ore could be radioactive by virtue of its very low thorium and tungsten content, suggesting why some Ta/Nb minerals cannot be shipped out of the country, because of their levels of radioactivity based on the limits of allowed thorium and tungsten contents 0.08% and 0.15% respectively. The X – ray diffraction spectrum with tantalite characteristics diffraction peaks at "2.56 A^{O}, 2.39 A^{O}, 1.76 A^{O}, 1.50 A^{O} and 1.43 A^{O}".

Physical properties, such as colour, are key to identifying minerals. Tantalite has similar physical properties with columbite. **Table 3** shows the physical properties of the tantalite. The loss on ignition (LOI) (**Table 3**) entails low volatile matter, which indicates that greater percentage of the material was retained after excessive heating at 1000 ^{o}C, while the pH showed low alkalinity and close to neutrality.

Table 1. Elemental composition and their oxides from XRF analysis

S/N	Element	% Element	% Oxide	% Oxide
1	Si	2.180	SiO_2	4.660
2	Al	0.800	Al_2O_3	1.520
3	Ta	34.170	Ta_2O_5	41.720
4	Ca	0.310	CaO	0.430
5	Fe	12.550	Fe_2O_3	17.940
6	Mg	3.620	MgO	6.000
7	K	0.007	K_2O	0.090
8	Cr	0.027	Cr_2O_3	0.040
9	Na	0.015	Na_2O	0.020
10	P	0.013	P_2O_5	0.030
11	Sn	0.134	SnO_2	0.170
12	Mn	1.738	MnO_2	2.750
13	Ti	6.006	TiO_2	10.020
14	Nb	8.375	Nb_2O_5	11.980
15	W	0.119	WO_3	0.150
16	Th	0.066	ThO_3	0.080

Table 2. XRD characterization of tantalite ore

Peak	2Θ/degree	Plane	Minerals	Compositions (wt.%)
1	23.00	110	Quartz	3.92
2	24.89	101	Quartz	6.36
3	32.93	111	Ilmenite	12.68
4	35.02	020	Tantalite/Quartz	11.73
5	37.60	121	Tantalite	38.47
6	42.50	211	Pyrite	7.70
7	48.53	202	Ilmenite	3.55
8	52.00	123	Tantalite	3.20
9	55.24	232	Pyrite	4.35
10	62.00	331	Tantalite	3.28
11	65.08	152	Tantalite	4.74

Table 3. Physical properties of the tantalite ore

Physical properties	Quality
Loss on ignition (%)	2.39
pH	8.43
Colour	Ash/grey
Average size (mm)	0.10
Specific gravity	7.2-8.0
Particle size (mm)	0.063 - 0.150

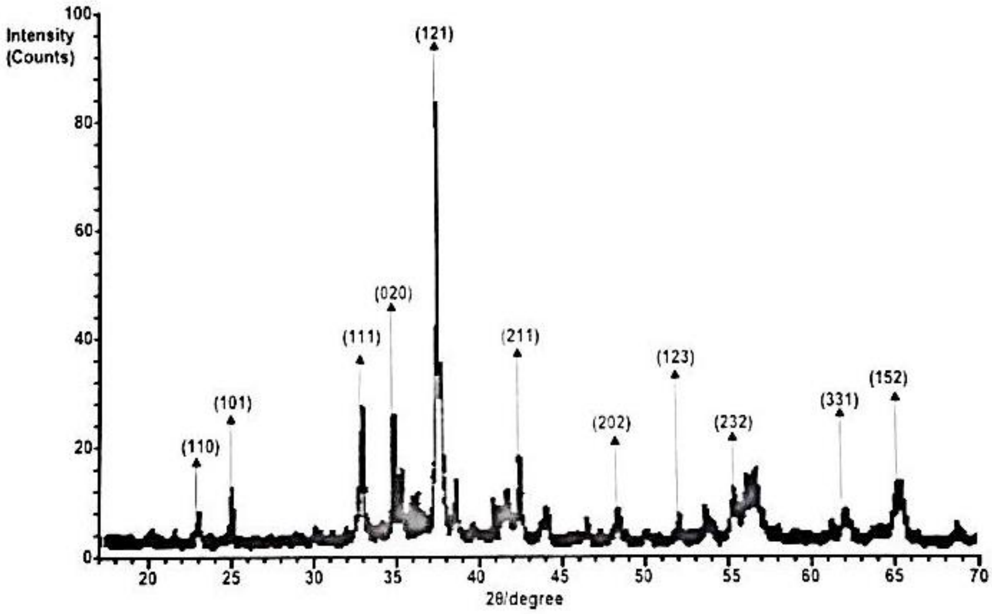

Figure 2. XRD Characteristic peaks of tantalite

Table 4. Anions in Tantalite Ore

Sample	**Anions**			
	Cl^-	PO_4^{2-}	NO_3^-	SO_4^-
(mg/l)	180	210	630	112.8

4. Conclusion

The mineral composition in terms of the oxides, elemental and crystalline mineral concentrations of the tantalite ore of Okpella was determined by EDXRF and XRD analysis and was found to contain high concentration of %Ta_2O_5 "41.72" within the acceptable limit of the rich tantalite deposits across the world. Other associated mineral oxides include Fe_2O_3, Nb_2O_5, TiO_2 and MgO. The physical properties tested showed low composition of volatile matter and anionic composition. This preliminary survey has provided a foundational understanding of tantalite deposits in Okpella, setting the stage for comprehensive studies and strategic planning across geological, metallurgical, environmental, economic, and social dimensions aiming to maximize the sustainable utilization of this mineral resource.

Authors' Contributions

Conceptualization and design: O.J.M., A.T.I.; Supervision and resources: O.J.M., A.T.I.; Methodology and data curation: O.J.M, A.T.I.; Investigation, data collection, analysis and interpretation: O.J.M., A.T.I.; Writing - original draft: O.J.M., A.T.I.; Writing -editing and review: O.J.M., A.T.I. Authors read and approved the submitted version.

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Availability of data and materials

Data sharing is not applicable to this article as no new data were created or analyzed in this study

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