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Paramagnetic Properties and Heat Treatment Effects in the Beneficiation of Nigerian Tantalite Ores

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ABSTRACT

Nigeria is endowed with mineral resources of significant economic value, including tantalite ore. The quality of tantalite ore, determined by its elemental composition, is crucial for its utilization. This study investigates the beneficiation and purification of tantalite ores from Angwan Kwano (designated as sample A) and Farin Dutse (designated as sample B) in Nasarawa State, Nigeria. The samples were pre-treated, crushed to a particle size of 1000 µm, and subjected to rapid magnetic separation, which resulted into paramagnetic, magnetite, hematite and non-magnetic. The effect of heat treatment on the beneficiated ores was also analyzed. Elemental composition analysis using X-ray fluorescence (XRF) revealed key oxides such as Ta_2O_5, Nb_2O_5, Fe_2O_3, MnO, and SiO_2 being present in the ore. Beneficiated Sample A showed 53.08 wt% Ta_2O_5 and 17.90 wt% Nb_2O_5, while Sample B contained 46.37 wt% Ta_2O_5 and 15.90 wt% Nb_2O_5. Heat treatment further enhanced these values, with Ta_2O_5 and Nb_2O_5 concentrations in Sample A reaching 54.11 wt% and 17.80 wt%, respectively, and Sample B yielding 48.03 wt% Ta_2O_5 and 18.20 wt% Nb_2O_5.This study has successfully shown that beneficiated and heat-treated samples exhibit higher concentrations of Ta_2O_5 and Nb_2O_5, with reduced impurities such as Fe_2O_3 and SiO_2. These findings confirm the potential of the applied techniques to upgrade Nigerian tantalite ores to industrial standards. The findings confirm that magnetic separation and heat treatment significantly improve the quality of tantalite, transforming it into ferrotantalite with higher concentrations of target oxides. This research highlights the potential of these techniques for optimizing the economic value of Nigerian tantalite ores.

Keywords: *Tantalite, Ferrotantalite, Manganotantalite, Paramagnetic, Magnetite, Hematite.*

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I. INTRODUCTION

Tantalite, a mineral compound of tantalum, iron, and manganese, frequently coexists with columbite (a niobate of iron and manganese) and various accessory minerals. Tantalum, a transition metal classified as the 73rd element in the periodic table with an atomic weight of 180.95 g/mol, is renowned for its refractory properties due to its high heat resistance (Theron, 2010). Its natural oxidation state leads to the formation of a tantalum oxide (Ta_2O_5) layer, rendering it highly resistant to all acids except hydrofluoric acid (Maina, 2014; Starck, 2001).

The last two decades have witnessed a sharp rise in the global demand for tantalum and niobium, driven by their extensive applications in high-tech industries, the medical field, and the production of modern industrial materials. These elements are pivotal in manufacturing super-alloys and electronic devices such as cell phones, laptops, and capacitors (Alhassan *et al*., 2010; Nete, 2012). Historical accounts highlight the early use of tantalite as filament material in incandescent electric lamps (Singh, 2007), while its role in miniaturizing mobile phones underscores its enduring relevance.

Nigeria, rich in tantalite and columbite resources, discovered its first tantalite deposits in the 1940s as a by-product of cassiterite mining in Plateau State (Okunlola, 1999). By 2004, Nigeria was ranked as the seventh-largest producer globally, with an estimated output of 25 metric tons (Adetunji *et al*., 2005). The economic value of tantalite is intrinsically tied to its purity, necessitating efficient beneficiation techniques to remove impurities and maximize tantalum and niobium content.

Primary beneficiation techniques for tantalite ores, particularly those containing less than 0.1 wt% Ta_2O_5 and Nb_2O_5, often involve gravity separation (Nete, 2009; Nete *et al*., 2014). For ores exhibiting

paramagnetic properties, magnetic separation is a crucial preliminary step to eliminate magnetically susceptible impurities before chemical dissolution and further purification (Adetunji *et al*., 2005). Research in the Democratic Republic of Congo demonstrates the utility of gravity separation and magnetic tweezers in upgrading coltan before chemical analysis (Nete *et al*., 2014). Similarly, Berhe *et al*. (2017) showed that washing tantalite concentrates with potassium hydroxide (KOH) and sulfuric acid (H_2SO_4) effectively removed thorium and uranium oxides.

Baba *et al*. (2005) reported that the dissolution kinetics of tantalite in concentrated nitric acid are governed by diffusion via hydrogen ion action, with a reaction order of 0.64. Their experiments revealed that approximately 81.3% of 0.5 g tantalite ore (particle size < 0.04 mm) dissolved in 8.06 M HNO_3 within 120 minutes.

Moreover, beneficiation studies on manganotantalite and ferrotantalite from Mozambique confirm that magnetic separation removes significant iron and titanium content, while sulfuric acid dissolves thorium and uranium impurities (Nete *et al*., 2014). For example, ferrotantalite yielded 64.14 wt% and 72.04 wt% removal of Fe and Ti, respectively, while manganotantalite showed minimal removal of Ta_2O_5 and Nb_2O_5.

This study builds upon the extensive research on tantalite beneficiation by investigating the paramagnetic properties of Nigerian tantalite ores and the effects of heat treatment on their quality. Samples from Angwan Kwano and Farin Dutse in Nasarawa State were subjected to magnetic separation and heat treatment. Elemental composition analyses revealed significant improvements in the concentrations of Ta_2O_5 and Nb_2O_5, highlighting the potential of these techniques to enhance ore quality. This research aims to contribute to the optimization of beneficiation processes, ensuring the economic and industrial viability of Nigerian tantalite resources.

II. METHODOLOGY EXPERIMENTAL PROCEDURE

2.1 Physical Characterization

The tantalite ore samples were sourced from mining sites at Angwan Kwano and Farin Dutse in Nasarawa Local Government Area of Nasarawa State, Nigeria. The sample collected from Angwan Kwano was labeled Sample A, while the sample from Farin Dutse was labeled Sample B. Sample A exhibited a dark grey colour with traces of white grains. Both samples appeared similar in colour, hardness, and texture, suggesting comparable physical characteristics.

2.2 Elemental Analysis

For elemental analysis, an X-ray fluorescence (XRF) machine (Model No: 1200) was used to determine the composition of the samples. All chemicals and reagents used in this study were obtained from Quality Skill Entrepreneurship Limited, Karu, Nasarawa State. The XRF analysis was performed on both the raw paramagnetic samples and the heat-treated samples to evaluate the changes in elemental composition resulting from the magnetic separation and heat treatment processes.

2.3 Beneficiation and Heat Treatment

The samples were prepared for beneficiation through size reduction and magnetic separation. These processes were conducted at the National Metallurgical Development Centre (NMDC), Jos. Initially, 1.5 kg of tantalite ore samples were randomly collected from the mining sites and crushed to a particle size of 1 mm using a Deco raw crusher (size 1) (BB: 7141 and INSP: A779769). The crushed samples were then subjected to a rapid magnetic separator (Model No: 25459, Batch: 2071C), which segregated the materials into paramagnetic (tantalite and columbite), magnetite, hematite, and non-magnetic fractions.

The paramagnetic fraction containing tantalite and columbite underwent further treatment. The samples were heated to a temperature of 1100°C as an additional purification process. The weight loss of the samples during heating was calculated to assess the effect of the heat treatment.

This comprehensive methodological approach ensured the accurate assessment of the beneficiation and purification potential of the tantalite ores.

III. RESULTS AND DISCUSSION

3.1 Effect of Crushing and Pulverization

The tantalite ore from Angwan Kwano and Farin Dutsewere subjected to crushing and particle sizes reduction. Adetunji *et al*. (2005) had shown that crushing process increased the surface area and the pulverization process is meant to further enhanced the active sites for chemical reactions and generally improves the efficiency of the magnetic separation and leaching.

3.1.2 Effect of rapid magnetic separation on the tantalite ore

The results obtained from rapid magnetic separation of the tantalite are presented in Table 1.

Table 1: Rapid magnetic separation of samples A and B

S/No	Component	Weight (g) of A	Weight (g) of B
1	Paramagnetic	824.93	799.39
2	Hematite	*NA	162.22
3	Magnetite	20.94	38.29
4	Non-magnetic	622.74	453.72

*NA = Not Available

From the results, magnetic separation significantly removed Fe_2O_3 (iron oxide) and MnO (manganese oxide) from the ore. In the process, Hematite content was not detectable in sample A but present in sample B. The decrease in value for the magnetite and non-magnetite in sample A and B was found to be (20.94 and 622.74g) and (38.29 and 453.72g) respectively. Paramagnetic (tantalum and niobium concentrate), (Ta_2O_5 and Nb_2O_5,) of samples A and B were 824.93 and 799.39g respectively. The increase in beneficiated Ta_2O_5 and Nb_2O_5 for both sample A and B, confirmed the efficiency of rapid magnetic separation in upgrading the ore. This is supported by study involving removal of radioactive oxides using pegmatite spodumene ores considered very rich in tantalite concentrates, it was shown that effective concentration of the paramagnetic components (Ta_2O_5 and Nb_2O_5), can be achieved by isolating the desired minerals from magnetically susceptible gangue materials like hematite (Berhe *et al*., 2017).

3.1.3 Loss on Ignition (LOI)

The results of ignition of samples A and B are shown in Table 2

Table 2: Loss on ignition for samples A and B

S/N	Initial weight (g)	Final weight (g)	Loss on ignition LOI(g)	LOI wt%
A	150	140.9	9.1	6.06
B	150	139.5	10.5	7.0

Table 2 shows the initial weight of samples A and B held at150g each., their final weights after ignition were 140.9g and 139.5g respectively. The loss of weight on ignition for samples A and B were found to be 9.1 and 10.5g respectively. Weight percentage loss was6.06 for sample A and 7.0 for sample B. Al-hassan *et al.* (2010) deduced that low LOI observed after heat treatment reflects the removal of residual impurities which can be attributed to the decomposition of volatile compounds or structural water in the mineral. The authors concluded that a lower LOI in the heat-treated samples correlates with higher purity levels of Ta_2O_5 and Nb_2O_5, confirming that heat treatment enhances the ore's chemical stability.

3.2.1 Compositional analysis of raw samples A and B

The results of chemical analysis of raw samples A and B are presented in Table 3.

From the XRF results, %wt of Ta_2O_5 and Nb_2O_5 were 41.55 and 10.70 respectively for sample A while in sample B the concentration of Ta_2O_5 and Nb_2O_5 were 31.2 and 9.16 respectively. Since wt % of Ta_2O_5 is higher than Nb_2O_5, then this shows that both samples are tantalite. For sample B, the compositional analysis of the two samples shows wt% of Fe_2O_3 to be 7.40 and 17.17.

Table 3: XRF Analysis of Raw samples A and B

S/No	Elemental Oxide	%Composition of sample A	% Composition of sample B
1	SiO_2	18	9
2	CaO	0.20	*ND
3	TiO_2	0.630	10.4
4	MnO	1.40	1.61
5	Fe_2O_3	7.40	17.17
6	ZrO_2	0.29	0.58
7	Nb_2O_5	10.70	9.16
8	SnO_2	19.1	20.0
9	Eu2O3	0.1	0.35
10	Ta_2O_5	41.55	31.2
11	WO_3	0.53	0.51

*ND = Not Detected

The concentration of MnO was found to be 1.40wt% and 1.61wt% for samples A and B respectively. The two samples contain Fe_2O_3more thanMnO. According to Cerny and Ercit (1989), tantalite ore with similar composition are called ferrotantalite. Other major impurities found in sample A and B include SiO_2 and SnO_2. Titanium oxide was also a major impurity in sample B. The Ta_2O_5 in sample A is higher compared to that reported by Al-hassan *et al.* (2010), which reported in a study of tantalite from Mai Kanji and found it to be

31.99 wt%. Similarly, Ta_2O_5 in sample A was found to be higher than that reported by Maina (2014) in the study of tantalite ores from Niger, Nasarawa and Plateau States. In his study, the concentrations of Ta_2O_5 were found to be 14.1, 25.4 and 29.06 wt% for Niger, Nasarawa, and Plateau State respectively. Sample B was found to contain higher percentage weight of Ta_2O_5 than that reported by Maina (2014), but slightly lower than that reported by Al-hassan *et al.*(2010). Considering health safety concerns, results obtained in samples A and B were found to be better in quality than that reported by Al-hassan *et al.* (2010) and Maina (2014) because they are free from radioactive compounds such as Uranium and thorium as well as heavy metals like zinc, copper and lead. The values presented in Table 3 has successfully shown that beneficiated and heat-treated samples exhibit higher concentrations of Ta_2O_5 and Nb_2O_5, with reduced impurities such as Fe_2O_3 and SiO_2. These findings confirm the potential of the applied techniques to upgrade Nigerian tantalite ores to industrial standards.

3.2.2 Effects of beneficiation on the samples

The chemical analysis of beneficiated samples A and B are shown in Table 4

Table 4: Effect of beneficiation on samples A and B

S/No	Elemental Oxide	% Composition of sample A (before beneficiation)	% Composition of sample A (after beneficiation)	% Composition Sample B(before beneficiation)	% Composition Of sample B(after beneficiation)
1	SiO_2	18	15	9	17
2	CaO	0.20	0.46	0.0	0.0
3	TiO_2	0.630	0.58	10.4	3.35
4	MnO	1.40	1.95	1.61	2.04
5	Fe_2O_3	7.40	9.31	17.17	11.9
6	ZrO_2	0.29	0.28	0.58	0.32
7	Nb_2O_5	10.70	17.9	9.16	15.9
8	SnO_2	19.1	0.0	20.0	2.32
9	Eu_2O_3	0.1	0.1	0.35	0.1
10	Ta_2O_5	41.55	53.08	31.2	46.37
11	WO_3	0.53	0.59	0.51	0.88
12	V_2O_5	*ND	0.009	*ND	*ND
13	Sb_2O_3	*ND	0.54	*ND	*ND
14	BaO	*ND	ND	*ND	0.08

*ND = Not Detected

The effect of beneficiation showed that the percentage weight of some compounds increased, while that of others decreased. Tantalum oxide and niobium oxide were found to have increased by 11.53 and 7.20 wt % respectively in sample A and 15.17 and 6.74 wt% respectively in sample B. The beneficiation completely removed SnO_2 in sample A and reduced it to 2.32 %wt in sample B. For sample A, the result showed a small increase in percentage weight of CaO, MnO, WO_3, V_2O_5 and Sb_2O_3 while the concentration of TiO_2 and ZrO_2 decreased slightly. Major Oxides such as SiO_2 decreased by 3 wt% while Fe_2O_3 increased by 1.91 wt%. In sample B, minor impurities, such as MnO, WO_3 and BaO showed slight increase in concentration while the concentration of ZrO_2 and Eu_2O_3 decreased slightly. The major impurities, Fe_2O_3, TiO_2 and SnO_2 decreased by 5.27, 17.68 and 7.05 wt% respectively while SiO_2 increased by 8 wt%. The beneficiation result obtained for sample B showed a remarkable increase in Ta_2O_5 compared to that reported by Maina (2014) which showed an increment of 13.1 wt% when direct dissolution of tantalite ore occurred using 15M HF. However, Maina's result was better than that obtained for sample A. In general, considering the negative impact of HF to the ecosystem, beneficiation using magnetic separation is preferred based on the inherent advantages.

3.2.3 Effect of heat on beneficiated samples

The chemical analysis results of the heat treatment on the beneficiated samples are shown in Table 5.

Table 5: Effect of heat on beneficiated samples

S/No	Elemental Oxide	% Composition Sample A (after beneficiation)	% Composition Sample A (after heating)	% Composition Sample B (after beneficiation)	% Composition Sample B (after heating)
1	SiO_2	15	11	17	11
2	CaO	0.46	0.02	ND	0.39
3	TiO_2	0.58	0.70	3.35	4.04
4	MnO	1.95	2.02	2.04	2.30
5	Fe_2O_3	9.31	10.0	11.9	13.3

6	ZrO_2	0.28	0.44	0.32	0.63
7	Nb_2O_5	17.9	17.8	15.9	18.2
8	SnO_2	0.0	2.7	2.32	*ND
9	Eu_2O_3	0.1	0.2	0.1	0.31
10	Ta_2O_5	53.08	54.11	46.37	48.03
11	WO_3	0.59	0.68	0.88	0.91
12	V_2O_5	0.009	0.006	*ND	*ND
13	Sb_2O_3	0.54	*ND	*ND	0.36
14	BaO	*ND	0.08	0.08	0.31
15	Cr_2O_3	ND	0.036	ND	0.02

*ND = Not Detected

The result shows increase in weight percentage of some compounds, a decrease in some and the complete incineration of others. After heating sample A, Ta_2O_5 increased by 1.03 wt% while Nb_2O_5 decreased slightly by 0.1 wt %. The concentration of minor impurities such as CaO, TiO_2, MnO, ZrO_2, Eu_2O_3, WO_3, BaO and Cr_2O_3slightly increased while that of V_2O_5 decreased. The heat treatment completely removed Sb_2O_3. Impurities such as Fe_2O_3 and SnO_2 increased by 0.29 and 2.7 wt% respectively while SiO_2 decreased by 4 wt%. For sample B the heat treatment increased Ta_2O_5 and $Nb_2O_5$by 1.66 and 2.30 wt% respectively. The minor impurities such as MnO, ZrO_2, Eu_2O_3, WO_3, and BaO increased slightly while CaO and Cr_2O_3which were absent in the magnetically beneficiated samples were present after heating. This is probably because prior to heating, these oxides existed only in trace amounts. In the heat treated sample, SnO_2 was conspicuously absent. The major impurities, Fe_2O_3 and TiO_2 increased by 1.4 and 0.69 wt% respectively while SiO_2 decreased by 6 wt%.

IV. Conclusion

This study has demonstrated the effectiveness of magnetic separation and heat treatment in enhancing the quality of tantalite ores sourced from Angwan Kwano (Sample A) and Farin Dutse (Sample B) in Nasarawa State, Nigeria. XRF analysis was performed on both the raw paramagnetic samples and the heat-treated samples to evaluate the changes in elemental composition resulting from the magnetic separation and heat treatment processes. Elemental analysis using X-ray fluorescence (XRF) revealed significant improvements in the concentrations of tantalum pentoxide (Ta_2O_5) and niobium pentoxide (Nb_2O_5) after beneficiation and heat treatment. Beneficiated sample A contained 53.08 wt% Ta_2O_5 and 17.90 wt% Nb_2O_5, while sample B showed 46.37 wt% Ta_2O_5 and 15.90 wt% Nb_2O_5. Further heat treatment elevated these values to 54.11 wt% Ta_2O_5 and 17.80 wt% Nb_2O_5 for sample A, and 48.03 wt% Ta_2O_5 and 18.20 wt% Nb_2O_5 for sample B.

The use of size reduction and magnetic separation proved to be highly effective in removing impurities such as iron and manganese oxides, thereby improving the ore's overall quality. Additionally, the transformation of the ores into ferrotantalite with higher concentrations of target oxides underscores the industrial viability of these processes. These findings highlight the potential of integrating magnetic separation and heat treatment to optimize the beneficiation of Nigerian tantalite ores, contributing to their economic value and aligning with sustainable mineral resource utilization. This research establishes a foundation for further exploration of advanced beneficiation techniques to support Nigeria's growing role in the global supply chain of tantalum and niobium.

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