Assessment Of Beneficiation Routes Of Tantalite Ores From Key Locations In Nigeria

**[1]Adetunji, A. R[*]; [2]Siyanbola, W.O.; [3]Funtua, I. I;
[1]Olusunle, S. O. O.; [1]Afonja A.A and [1]Adewoye, O.O.**

[1]*Metallurgical and Materials Engineering Department, Obafemi Awolowo University, Ile-Ife*
[2]*Centre for Energy Research and Development, Obafemi Awolowo University, Ile-Ife*
[3]*Centre for Energy Research and Training, Ahmadu Bello University, Zaria*
*Correspondence Author; e-mail: aadetunj@oauife.edu.ng or aderade2004@yahoo.com

Abstract:

The beneficiation methods for some tantalite ores from selected deposits in Nigeria have been assessed through mineralogical and compositional analyses of the ores. Tantalite ores obtained from eight different well-known locations of the mineral in the country were analyzed with ^{109}Cd excitation source Energy Dispersive X-ray spectrometry using the emission-transmission method of quantification. The major minerals detected and quantified 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. Our analyses further show that Ta_2O_5 in the ores ranged from a minimum concentration of about 8% in Otu 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. Based on these results and the work of other authors, we deduced that clean-up operations might be adequate for Ta extraction from most of the tantalite deposits in Nigeria. The consideration of simplicity and cost of process favours the direct dissolution of the ores in HF for the clean-up operations.

Keywords: Tantalite ores, Elemental Composition, EDXRF

INTRODUCTION

Nigeria is blessed with abundant mineral resources. These include vast deposits of coal, cassiterite, columbite, marble, limestone, clay, bitumen and tantalite. While some of these minerals are currently mined, several others have been identified to have huge potentials of being exploited in commercial scale. The Federal Government of Nigeria is paying renewed attention to the solid mineral sector; therefore, development activities in the sub-sector are expected to increase many-fold. Recent 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 [1].

The recovery of tantalite in Nigeria dates back to the 1940's as a by-product of cassiterite mining [2, 3]. Later, large tonnages of granite containing about 0.26% Nb_2O_5 in urania pyrochlore (3.1% U_3O_8, 3.3% ThO_2, 37.5% Nb_2O_5 and 3.5% Ta_2O_5) were

identified. The reserves were however not confirmed. Primary deposits of huge economic value of the ore have now been reported in va rious parts of the country [4, 5, 6]. Indeed, Nigeria is currently rated to be the 7th largest producer of tantalum resources in the world [7]. The production in 2004 was estimated at 25 metric tons.

Tantalite is th e most important m ineral form of tantalum , a specialty m etal used mainly in the electronics   industry for the m   anufacture of capacitors and in several specialty alloy applications.   The beneficiation of tantalite   ores usually involves pre-concentration, prim ary concentration and con centrate clean up. The choice of any or all of these processes would depend on the nature   of the ore, p articularly the conten t of Ta_2O_5 in the o   re r elative to its as   sociated m inerals a nd im purities. Typica lly, mechanically m ined ores contain less than $0.1\%Ta_2O_5$, and would ther efore e ssentially require enrichment through the three processes stated above. The concentration processes may be carried out by wet gravity, m   agnetic or electrostatic m ethods or by flotation to produce concentrates containing up to 70% com   bined Ta_2O_5 and Nb_2O_5 to m   eet extraction requirem ents. However, th   e universally em   ployed m   ethod for the concentration of tantalite ores is g   ravity separation – the separation of two or m   ore minerals as a result of differences in speci fic gravity and their m ovement in response to the force of gravity and one or m ore other forces [8, 9]. Other m ethods, when used, are employed in the final cleanup of gravity concentrates.

Furthermore, Burt [10] had noted that c oncentrates suitable for further processing to recov er Ta are generally required to ex   ceed 25% Ta_2O_5, with 50% combined Ta_2O_5 and Nb_2O_5. Primary ores with these Ta_2O_5 and Nb_2O_5 contents would only need clean-up operations for Ta extraction processes. The   clean-up operation results in the rem oval of associated m inerals and im purity elem ents to a to lerable ex tent. Typica l assoc iated minerals include zircon, rutile, m   onazite, cassiterite, ilm enite, garnet, uranium and thorium m inerals, beryl, spodum ene, tourm aline and, in som e cases, aquam arines and gold: light m inerals such as quartz and feld spar m ay also be present due to inefficient primary concentration.

A number of reports [11, 12] have give n the mineralogical composition of Ta-Nb ores obtained from a few locations in the m iddle belt region of Ni geria. However, the limitation of these previous wo rks is the failure to relate   the results to an upgrading process for a value-added Nigerian tantalite.

The main objective of this work is to assess the appropriate beneficiation schemes by identifying the as sociated elem ents and im purity elem ents in sam ples obtain ed from notable tantalite deposits across Nigeria. The associated elements are known to contribute to the physical characteristics   of the Ta m   ineral and thes e characteristics dictate the beneficiation methods. The outcom e of the wo rk reported here would therefore provide critical inform ation about the appropriate r outes for the beneficiation processes needed for value addition to the Nigerian tantalum industry.

MATERIALS AND METHODS

Eight sam ples were collected from   differe nt notable deposits in Nigeria. The sources are Egbe, Komu, Nassarawa, Agunrege, Baba Ode, Ofiki, Igbo Ijaye and Otu. Six of the locations a re in Oyo Sta te in the S outh W estern Nigeria; one location is in Kogi State while one is in Nassarawa State, both deposits located within the middle belt region of the country. The sam ples were obtained from miners in the study areas. The m iners employ manual methods of m ining, in which the samples would have been upgraded to some extent by hand sorting. Measurem ents of the major constituents and trace elem ents were carried out using a ra   dioisotope source Energy Disp ersive X-Ray Fluorescence (EDXRF) Spectrometer at the Centre for Energy Research and Training, Ahm adu Bello University, Zaria, Nigeria.

The samples were ground m anually to powder with an agate m ortar and pestle to grain size of less than 125 μm . Pellets of 19 mm diameter were prepared from $0.3 – 0.5$g powder mixed with three drops of organic liquid binder and then pressed at 10 tons with a hydraulic press. Measurem ents were perform ed using an annular 25 m Ci ^{109}Cd as the excitation source, which em its Ag-K X-rays (22.1 keV) in which case all elem ents with lower characteristic excitation en ergies were accessible for detection in th e samples. The system further consists of a Si(Li) detect or, with a resolution of 170 eV for the 5.90 keV line, coup led to a com puter controlled ADC -card. Further details about the system have been given elsewhere [11, 13,14].

Quantitative analy sis of the s amples was carr ied out us ing th e Em ission-Transmission (E-T) m ethod, for which a num ber of quantification m ethods has been developed and applied [13 – 18]. These   quantification m ethods provide different approaches to co rrect the matrix absorption as well as enhancem ent effects. In this work quantification was carried ou t using a m odified versi on of E-T m ethod [11, 19, 20], which involves the use of pure target m aterial (Mo) to m easure the absorption factors in the sam ple. The Mo target serves as a   source of m onochromatic X-rays, which are excited through the sam ple by prim ary radiat ion and then penetrate the sam ple on the way to the de tector. In this way, the abso rption factor is experim entally determined. The program then uses th is facto r in the quantification of con centration of the elem ents. In addition, the contribution to the Mo-K peak in tensity by the Zr-K is subtracted for each sample. The spectra for the sam ples were collected for 3000s with the ^{109}Cd and the spectra were then evaluated using the AXIL- QXAS program [21]. It was noted that very light mineral associations of the Ta ores could not be determined due to the absence of an ^{55}Fe excitation source.

RESULTS AND DISCUSSION

The results of the m ineralogical analys is includ ing a num ber of im purities/trace elements are shown in Tab le 1. The results show tha t the Egb e sam ple contain s the highest Ta$_2$O$_5$ content at 59.58%. The Ta $_2$O$_5$ contents of Kom u, Nassarawa, Agunrege,

Baba Ode, Ofiki, Igbo Ijaye and Otu   ore sam ples are respectively 49.57%, 46.15%, 45.42%, 42.00%, 36.63%, 23.64% and 8.00%. The resu   lts further showed that the samples also contain appreciable am ounts of Nb_2O_5 and TiO_2. Other m ineral ores/phases identified and quantified include MnO, Fe_2O_3, WO_3, Th and U. Im purities/trace elements such as Hf, Zn, Zr, Co, Pb, Rb, and Y were also determined.

Table 1: Mineralogical Analysis of Tantalum Ore Samples From 8 Different Locations in Nigeria.

Oxide	Egbe	Komu	Nassarawa	Agunrege	Baba Ode	Ofiki	Igbo Ijaye	Otu
TiO_2%	-	-	3.81 ±0.42	1.64 ±0.37	2.34 ±0.42	1.05 ±0.33	20.36 ±0.58	33.38 ±0.67
MnO%	4.15 ±0.10	9.03 ±0.17	5.80 ±0.13	6.69 ±0.14	8.62 ±0.17	10.10 ±0.18	3.46 ±0.12	0.74 ±0.09
Fe_2O_3%	7.76 ±0.18	3.51 ±0.14	10.69 ±0.23	7.37 ±0.18	4.69 ±0.16	2.86 ±0.13	9.66 ±0.21	9.70 ±0.20
Ta_2O_5%	59.58 ±0.62	49.57 ±0.54	46.15 ±0.52	45.42 ±0.49	42.00 ±0.46	36.63 ±0.42	23.64 ±0.40	8.00 ±0.17
Nb_2O_5%	19.74 ±0.17	29.18 ±0.23	24.86 ±0.20	31.18 ±0.26	32.90 ±0.26	37.48 ±0.29	28.90 ±0.29	22.43 ±0.17
WO_3%	-	0.38 ±0.13	1.80 ±0.14	-	-	-	0.32 ±0.09	0.17 ±0.05
SnO_2%	-	-	8.43 ±1.97	-	-	-	-	-
Hf%	0.22 ±0.05	0.26 ±0.05	0.31 ±0.05	0.18 ±0.05	0.17 ±0.05	0.11 ±0.04	0.13 ±0.04	-
Zn%	-	0.22 ±0.03	0.09 ±0.03	-	0.09 ±0.03	0.07 ±0.02	-	-
Zr%	0.31 ±0.01	0.12 ±0.01	0.26 ±0.01	0.15 ±0.01	0.22 ±0.01	0.17 ±0.01	0.06 ±0.01	0.06 ±0.01
Pb (ppm)	489 ±146	570 ±146	1720 ±195	-	1010 ±168	720 ±146	-	-
Rb ppm)	-	120 ±47	-	-	-	-	-	-
Y ppm)	(-	-	333 ±51	-	-	-	-	-
ThO_2 %	0.34	0.03	0.15	0.02	0.02	0.05	0.03	0.01
U_3O_8%	0.17	0.37	0.65	0.42	0.94	1.57	0.28	0.27

The elem ental Ta content in the   respective ore sam ples are 48.79%, 40.59%, 37.79%, 37.19%, 34.39%, 30%, 19.36% and 6.55%   respectively for Egbe, Kom   u, Nassarawa, Agunrege Baba Ode, Ofiki, Igbo   Ijaye and Otu. An earlier work by Funtua [11] put the Ta content in the central p egmatite region around Jos, Nassarawa and Jama'a areas of central Nigeria at   between 21 and about 30%.   We note that this figure is somehow less than the Nassarawa ore found to be about 38% in the current investigation. The difference could probably be explained in term s of the diversity in the ore contents from site to site and from ore vein to ore vein.

In terms of beneficiation, the density of Ta-Nb minerals allows concentration with other heavy minerals by gravity methods; sluices, jigs, spirals and shaking tables are used in conjunction with screen sizing; other proc esses, when used, are em ployed in the final cleanup of gravity concentrates. Ta-Nb is   separated from other heavy m inerals by a combination of high-tension electrostatic   and high-intensity electrom agnetic m eans.

Concentrates from Nigerian placer deposits , which ar e worked pr imarily for cass iterite, are dried and treated by m agnetic separators to r emove the m agnetic cons tituents: magnetite, ilmenite, columbite, monazite and magnetic cassiterite. The magnetic fraction is then re- treated to sepa rate the d ifferent m inerals. Colum bite, m onazite and m agnetic cassiterite are weak ly magnetic. Columbite and magnetic cass iterite are separated on air flotation tables, whereas colum bite and m onazite are s eparated e lectrostatically [2]. Classification, screening, and deslim ing ar e em ployed as adjuncts, depending on the composition of the ore. Treatment schemes vary from the most primitive hand methods to extremely com plex m odern plants [22]. The liberation of values from waste m ust be carried out with care, since all tantalum minerals are friable, and, ac cording to Burt [10], concentration efficiency decreases with decreasing particle size.

Gravity p lant con centrate, grad ing approx imately 25% Ta $_2O_5$ is g enerally upgraded to 50% in a clean-up plant. The Nb content in the ore is very im portant. Concentrates suitable for further processi ng to recover Ta are generally required to exceed 25% Ta_2O_5, with 50% combined Ta_2O_5 and Nb_2O_5 [10]. The ores below this level of $Ta_2O_5 – Nb_2O_5$ content need to be enriched by methods discussed above. The tantalum ores from Egbe, Komu, Nassarawa, Agunr ege, Baba Ode and Ofiki all have Ta $_2O_5$ - Nb_2O_5 combined content of about 70%, w ith the Ta_2O_5 contents of over 25%. Thus these ores can be subjected directly to clean- up operations, without pre-concentration or primary concentration, for the rem oval of a ssociated m inerals. Many of the ores m ay even be subjected to direct hydrom etallurgical extraction processes without going through clean-up operations. This is because their Ta $_2O_5$ contents fall into c ategories for which technologies have specifically been developed as raw materials of different origins and grades [23], as shown in Table 2. The ore sample from Igbo Ijaye, which contains 23.64%Ta_2O_5, is rich enough for prim ary concentration, while the Otu ore sam ple, with 8%Ta_2O_5, would be enriched right from the pre-concentration stage (Table 3). These two ore sam ples were however found to have a high T iO$_2$ content of 20.36% and 33.38% respectively. Although the Otu figure is lower than that of a recently studied rutile sample [24], it would be appropriate to treat the ore pr imarily f or rutile r ecovery and then secondary recovery for tantalite and niobite.

Table 2: Correlation between technology and raw material input [23]

Technology	Raw Material Input	%Ta/Ta_2O_5
Pyrometallurgy	Low – Medium Grade Tin Slags	$2 – 10\%Ta_2O_5$
Chlorination	Alloys, Scraps	$40 – 100\%Ta$
Hydrometallurgy	Natural Ores	$20 – 40\%Ta_2O_5$
	Synthetic Concentrates	$20 – 40\%Ta_2O_5$
	High Grade Tin Slags	$>15\%Ta_2O_5$

The main associations of Ta $_2O_5$ in th e ores und er investigation apart from Nb_2O_5 and rutile are hematite, wolframite, uranium, cassiterite minerals as well as hafnium, zinc and zirconium elements with other trac e elements. We note that because T iO$_2$ and MnO are param agnetic and Fe $_2O_3$ is ferrom agnetic, they can be separated from the ore by magnetic separation, whereas electrostati c separation can be em ployed to rem ove cassiterite, hem atite, ilm enite, tan talite, n iobite and wolf ramite, which a re e lectrically

conducting, from the ore, leaving the non-   conducting zirconium com pounds in the gangue. An earlier work by our group [25] had not been able to present the data shown in this work on th e im purity/trace e lement conten ts of the o res. Mo st of thes e elem ents would go into the gangue during the elimination of the major constituents by the physical and subsequent chemical methods.

Table 3: Recommended beneficiation routes for different grades of Ta_2O_5 ores (Adapted from table provided by Burt, [10])

Ta_2O_5 Content (%)	Sample Sources Content (%Ta_2O_5)	Beneficiation Route
<10	Out (8.00)	Preconcentration (sizing, gravity separation
10 – 25	Igbo Ijaye (23.64)	Primary concentration (jigs and sluices) Hydraulic classification Stage treatment (spirals, shaking tables)
>25	Egbe (59.58), Komu (49.57, Nassarawa (46.15), Agunrege (45.42), Baba Ode (42.00), Ofiki (36.63)	Concentrate clean-up (floatation, leaching, magnetic or electrostatic separation)

We note further that based on the characteristics of the ores analysed in this study, a prelim inary tre atment f or the samples would be fusion with K $_2CO_3$, which would separate Sn, Fe, Mn and TiO $_2$ from the earth acid s [26]. The approach in this ca se, is to fuse at red heat with threefold excess of K $_2CO_3$, dissolve th e fused m ass in water, filter, then precipitate the tantalum and niobium as the sodium salts. However, the num ber of steps and am ount of reagents required could make this process expensive. Gustion and Pilloton [26] observed that direct dissolution of ores in HF as a c lean-up operation would not always produce adequate solution for separation by crystallization or liquid extraction. Thus an alternative route to eff ect a rough purification is to fuse them with caustic. The fused m ass would then be leached with wa ter to rem ove excess c austic, Sn and W. The Ta and Nb, in the form of complex tantalates and niobates, are insoluble. An acid leach would then dissolve Fe, Mn, Alk alies, and alkaline earths, leaving as a res idue a m ixture of the acids, T i, and traces of other contam inants. However, Gustion and Pilloton [26] showed that six add itional op erations m ust be added to those required by direct HF dissolution in order to accomplish this. These are caustic fusion, crushing of the fusion mass, water leach, filtration of water-leach liquor, acid leach and filtration of acid-leach liquor. Th e caustic fusion step is a hi gh-labour operation. Furth ermore, the labou r requirements for each filtration step are about the same as for direct dis solution, so that filtration co sts rise 100 -200% ov er th e dir ect d issolution op eration. Leaching in water requires abou t 50% as m uch labour as direct d issolution, bu t since leach ing in acid is comparable to direct d issolution the wet processing of the upgra ded ores altogether requires about 100-150% m ore labour than a direct dissolu tion operation. The consideration of sim plicity and cost of proc ess therefore favours the direct dissolution of the ores in HF to dissolve Ta, Nb, Fe, Ti, Mn and W . In practice, the adv antages and disadvantages of caus tic fusion p rior to th e HF leach m ust be carefully evaluated. The decision to use caustic fusion depends m ainly on the type of ore that is treated and on the method of separation used. If chem ical a nd equilibria data were available for the

dissolution process, it m ight be possible   to m aximize Ta and Nb dissolution with minimum dissolution of the gangue material.

Finally, it is appropriate to m ention here that depending on the country or region of exportation, som e Ta/Nb m inerals cannot be shipped out of thei r country of origin because of their levels of radioactiv ity. The limits of allowed uranium and thorium oxide contents are 0.1 %U_3O_8 and 0.1%ThO $_2$ [27]. All the ore sam   ples under investigation would not pass through such trans portation regulation for their U $_3O_8$ content while Egbe and Nassarawa sam ples would not pa ss through as a result of their ThO $_2$ conte nt. This measure in principle could create a m arket problem for the tantalite ores originating from these Nigerian deposits. Where there are ready buyers because of the strategic importance of the end product, i.e. the tantalum   metal/compounds, the crude ore is substantially reduced in value because of their radioactive contents. However, Krismer and Hoppe [28] have developed a process for the recovery of the non-radioactive m etal compounds from the ores containing com plexes of U and Th.   The gangue can now be treated to recover uranium as an additional economic by-product [29].

CONCLUSION

The analysis of the tantalum ores from ei ght different notable deposits in Nigeria using EDXRF spectrom etry has shown that the   Nigerian tantalum ores are of relatively high quality and can therefore   be subjected to direct pr ocessing for tantalum products without the need for com plex and costly pr econcentration procedures. The consideration of si mplicity and cost of process favours the   direct dissolution of the ores in HF. The simultaneous evaluation of rutile and niobium in the ores allows for the assessment of the suitability of the ores for prim ary/secondary recovery processes   of rutile and niobium products. In addition, the XRF technique offers the possibility of determining other major constituents such as Fe, Mn, W   , Th, U,   Hf, Zn and Zr. The tantalite m   ineral can be separated from the ore leavi ng the radioactive elem ents in the gangue, the secondary recovery of which is an added econom ic a dvantage. The knowledge of the presence of Co, Pb, Rb, and Y, as well as radioactive   elements in the s amples would assis t in evaluating co-dissolved species as well as   the development of appropriate beneficiation routes for value-addition to the mineral in Nigeria.

ACKNOWLEDGEMENTS

REFERENCES

1. Aliyu, A.; 1996; Potentials of the Soli   d Minerals Industry   in Nigeria; Raw Materials Research and Development Council, Abuja, Nigeria.

2. Miller, G. L.; 1959; *Tantalum and Niobium*; Butterworths Scientific Publications, London; p12.

3. Kock, W . and Paschen, P.; 1989; "Tantalum   – Processing, Properties and Applications"; *JOM* 10, pp33-39.

4. Okunlola, O. A.; 1999; "Specialty   Metal Potentials of Nigeria";   *Proceedings of the First Mining in Nigeria Conference and Workshop*; pp67-90.

5. Saint Simon de, P.; 1999; "Specialty Metals in Nigeria: Potential for W orld Class Deposit; *Second "Mining in Nigeria" Conference*, Abuja, Nigeria; p15.

6. Ministry of Solid Minerals Developm   ent; 2003; "Tantalite and the Rare Earth Metals".

7. Cunningham, L. D.; 2005; "Tantalum   "; *USGS Mineral commodity Summaries;* pp166-167.

8. Burt, R. O.; 1984; "Gravity Concentrat   ion Technology"; Elsevier Science BV, Amsterdam, p607.

9. Wills, B. A.: 1992; "Mineral Processing Technology", 5   th Ed.; Pergam on Press, p408.

10. Burt, R.; 1996; "Beneficiation of Tantalum Ore – How it is Achieved and Could it be Better?"; *Proceedings of 125th TMS Annual Meeting and Exhibition*; Anaheim, CA. 17-22

11. Funtua, I. I.; 1999a; "Analysis of Nb-Ta ores by Energy Dispersive X-ray Fluorescence Spectrometry". *J. Trace Microprobe Techniques*, 17, 2 (1999a), pp89-195.

12. Obiajunwa, E. I.; 2001; "Analysis of some Nigerian solid mineral ores by energy-dispersive X-ray fluorescence spectroscopy". *Nuclear Instruments and Methods in Physics Research B,* 184, pp437-430.

13. Markowicz, A. A; and Van Grieken, R. E.; 1993; "Quantification in XRF analysis of interm ediate th ickness sam ples". In: Van Grieken and Markowicz (eds) *Handbook of X-Ray Spectroscopy*, Marcel Dekler Inc., New York, 339-358.

14. Bernasconi, G. B; Bam ford, S. A; Dosa n, B; Haselberger, N; Markowicz, A; Mahmoud, A; and Valcovic, V.; 1996; "Appl   icability of annular source excited systems in quantitative XRF analysis"; *X-ray Spectrom.* 23, pp65-70.

15. Leroux, J; and Mahm oud, M.; 1996; "X-ray qua ntitative analysis by an em ission-transmission method"; *Anal. Chem.*, 38, pp76-82.

16. Giauque, R. D; Garrett, R.   B; and Goda, L. Y.; 1979; "Determ   ination of trace elements in light e   lement m atrices by X-ray fluorescen   ce with incohe rent scattered radiation as an internal standard"; *Anal. Chem.* 51, pp512-516

17. Markowicz, A. A.; 1979; "A m ethod for co rrection for absorption m atrix effects in sam ples of interm ediate thickne ss in EDXRF analysis"; *X-Ray Spectrom.*, 8, pp14-18.

18. Tang, S. M; Kump, P; Yap, C. T; and Bila l, M. G.; 1986; "Cal culation of relative fluorescence intensity for annular-sour ce geom etry by Monte Carlo m ethod"; *X-Ray Spectrom.*, 15, pp289-293.

19. Angeyo, K. H; Patel, J. P; Mangala,   J. M; and Naraya, D. G. S.; 1998; "Optimization of X-ray Fluorescence analys is: an exam ple from Kenya" *Appl. Radiat. Isot.*, 49, pp885-891.

20. Funtua I. I.; 1999b; "Applic ation of the transmission-emission method in EDXRF for the dete rmination of trace elements in geological and biological m aterials"; *J Trace Microprobe Techniques*; 17, 3, pp293-297.

21. Bernasconi, G. B.; 1996; "AXIL-QXAS", *Instruction Manual*, IAEA, Vienna.

22. Warde, J. M., Mee, V. A. and Spedde n, H. R.; 1963; "Colum bium and Tantalum Minerals, Ores, and Ore Preparation"; In: *Columbium and Tantalum*; Scisco, F. T. and Epremian, E. (eds.); John Wiley & Sons, p17

23. Eckert, J; 1996; "The Industrial Applicat ion of Pyrom etallurgical, Chlorination and Hydrometallurgy for Producing Tantalum Compounds"; *Proceedings of 125th TMS Annual Meeting and Exhibition*; Anaheim, CA. pp55-61.

24. Siyanbola, W. O, Fasasi, A. Y, Funtua, I. I, Afolabi, O. M, Adesiyan, T. A and Adetunji, A. R.; 2004; "Elem ental Com position of Rutile From South-W estern Nigeria Using X-Ray Techniques"; *Nuclear Instruments and Methods in Physics Research B* 215, pp240-245.

25. Adetunji, A. R, Siyanbola, W. O, Funtua, I. I, Afonja, A. A and Adewoye, O. O: 2004; "Energy Dispersive X- Ray Fluorescence Analysis of Tantalite Ores From Various Deposits in Nigeria"; In: *Proceedings of the Nigerian Materials Congress (NIMACON) 2003*; Eds: B. Babatope and W . O. Siyanbola; Materials Society of Nigeria; pp38-42.

26. Gustion, R. A. and Pillo ton, R. L. ; 1963; "Extraction and Sep aration of Columbium and Tantalum "; In: *Columbium and Tantalum*; Sc isco, F. T. and Epremian, E. (eds.); John Wiley & Sons, pp17.

27. Simandl, G. J.; 2001; "Tantalum Mark et and Resources: An Overview"; *Geological Fieldwork*; British Colum bia Geol ogical Survey; paper 2002-1, pp313-318.

28. Krismer, B. and Hoppe, A.; 1984; US Patent No 4446116

29. Funtua, I. I, Ogunleye, P. O, Um ar, I. M. and Elegba, S. B.; 1998; "Prelim inary Studies on the Recovery of Uranium From Mika, Northeastern Nigeria"; *Journal of Mining and Geology*, vol 34, No 2, pp219-224