Fluorination Coupled to Sulfuric-acid Leaching for Efficient Extraction of Ta and Nb from Numbi Coltan Ore (South-Kivu, DRC)

Alidor Shikika, Fabrice Muvundja, Mac Mugumaoderha, Stoyan Gaydardzhiev

The recent developments in modern and advanced technologies, especially in the field of telecommunications, steel making, aerospace and nuclear energy have strongly increased the market demand for tantalum (Ta) and niobium (Nb) metals. However, the extractive metallurgy of Ta-Nb remains particular challenging due to the harmful environmental effects associated with the use of hydrofluoric acid (HF) as main chemical leaching agent for Ta-Nb bearing ores. Recently scientific efforts have been made for the development of a hydrometallurgical alternative to the HF-process. One of the prominent novel processes suggested consists of ore pre-treatment with ammonium bifluoride (ABF) followed by sulfuric acid leaching. Results on this process study are presented herein. The most influential process parameters on Ta and Nb recoveries during the fluorination and leaching were identified using a Taguchi L_9 orthogonal array experimental set-up. Results suggest that more than 90 % of Ta and Nb could be leached when ore granulometric fraction, ratio of ABF to ore, fluorination temperature, and sulfuric acid concentration are kept at their optimal values. Fluorination temperature was the most determinant factor. The resulting leachates are suitable for downstream separation and purification of Ta and Nb.

Keywords:

Ammonium bifluoride (ABF) – Fluorination – Tantalum – Niobium – Coltan ore

Fluorierung in Verbindung mit Schwefelsäurelaugung zur effizienten Gewinnung von Ta und Nb aus Numbi-Coltanerz (Süd-Kivu, DRC)

Die jüngsten Entwicklungen in modernen und fortschrittlichen Technologien, insbesondere in den Bereichen Telekommunikation, Stahlerzeugung, Luft- und Raumfahrt und Kernenergie, haben die Marktnachfrage nach den Metallen Tantal (Ta) und Niob (Nb) stark erhöht. Die Gewinnung von Ta-Nb-Metallen stellt jedoch nach wie vor eine besondere Herausforderung dar, da die Verwendung von Flusssäure (HF) als wichtigstes chemisches Auslaugungsmittel für Ta-Nb-haltige Erze mit schädlichen Umweltauswirkungen verbunden ist. In letzter Zeit wurden wissenschaftliche Anstrengungen unternommen, um eine hydrometallurgische Alternative zum HF-Verfahren zu entwickeln. Eines der vorgeschlagenen neuen Verfahren besteht in der Vorbehandlung des Erzes mit Ammoniumbifluorid (ABF), gefolgt von einer Schwefelsäurelaugung. Die Ergebnisse dieser Prozessstudie werden hier vorgestellt. Die einflussreichsten Prozessparameter auf die Ta- und Nb-Gewinnung während der Fluorierung und Auslaugung wurden mit Hilfe einer orthogonalen Taguchi-L_9-Anordnung ermittelt. Die Ergebnisse deuten darauf hin, dass mehr als 90 % des Ta und Nb ausgelaugt werden können, wenn die granulometrische Fraktion des Erzes, das Verhältnis von ABF zu Erz, die Fluorierungstemperatur und die Schwefelsäurekonzentration auf ihren optimalen Werten gehalten werden. Die Fluorierungstemperatur war der entscheidendste Faktor. Die resultierenden Laugungsprodukte sind für die nachgeschaltete Trennung und Reinigung von Ta und Nb geeignet.

Schlüsselwörter

Ammoniumbifluorid (ABF) – Fluorierung – Tantal – Niob – Coltanerz

Fluoration couplée à une lixiviation à l‘acide sulfurique pour une extraction efficace du Ta et du Nb du minerai de coltan de Numbi (Sud-Kivu, RDC)

Fluoración acoplada a la lixiviación con ácido sulfúrico para la extracción eficaz de Ta y Nb del mineral de coltán de Numbi (Kivu del Sur, RDC)

1 Introduction

Tantalum (Ta) and niobium (Nb) are two refractory metals known to have similar chemical and physical properties and are usually met in columbite-tantalite ores [1]. Ta is characterized by high melting point, elevated ductility and increased resistance to chemical attack below 150 °C [2]. Nb is mainly used in automobile manufacturing, as additive in low-alloy steels used in aerospace and nuclear energy sector [3, 4], while, Ta is mainly employed in miniaturized electronic capacitors and semiconductors essential for high-end technologies [5], mainly due to its ability of energy storing and release. The remaining uses of Ta include manufacturing of pipes, tanks, reactors, super alloys, coatings for jet engines and gas turbines, cutting tools and projectiles for military use [6, 7].

More than 150 mineral ores are reported to contain Ta or Nb, with tantalite [$(Fe, Mn)(Ta,Nb)_2O_6$], columbite [$(Fe, Mn)(Nb, Ta)_2O_6$], microlite [$(Na, Ca)_2(Ta, Nb)_2(O, OH, F)O_6$] and pyrochlore [$(Na,Ca)_2(Nb,Ta)_2O_6(O,OH,F)$] being the most economical ones [1, 8]. These “twin” metals are commonly found together in mineral ores with very similar chemical properties, rendering their separation quite a challenge [9]. The earth‘s crust abundance of Ta and Nb is very low, i.e. 2 ppm for Ta and 24 ppm for Nb [10], despite their ever-increasing demand in the new technologies sector. Therefore, Ta and Nb are classified as critical metals by the EU and the USA. Brazil and Canada are the world‘s leading producers of Nb concentrates as shown in Figure 1a [11-13]. Production of Ta concentrates is abundant in Central Africa, notably around the Great Lakes region already since 2009 (Figure 1b) [14]. Based on the USGS data, from 2016 to 2020, tantalum concentrate production in the Democratic Republic of the Congo accounted between 28 and 39 % of global production, ranking it as the world‘s largest coltan supplier.

Current industrial processes for purification and separation of tantalum (Ta) and niobium (Nb) bearing columbite-tantalite (coltan) ores rely exclusively on hydrofluoric acid leaching and solvent extraction by MIBK [15, 16]. However, due to the harmful effect of HF, more environmentally friendly processes have been extensively developed on lab scale [17-19]. The alkaline route and the use of molten salts, generally based on fluorine, are among the most widely studied alternatives. In our previous studies, ammonium bifluoride ($NH_4F{\cdot}HF$) pre-treatment was used to process the Lulingu coltan ore enabling to recover over 90 % of Ta and Nb during the subsequent water leaching [20]. More recently, Ta and Nb oxides of more than 93 % purity were produced through solvent extraction of the leachates resulting from ammonium fluoride leachates [15].

Fluorination process parameters influence to a large extent the recovery of Ta and Nb during the subsequent sulfuric acid leaching. Our previous study [20] found that parameters such as ore granulometry, fluorination temperature, mass ratio of ammonium bifluoride to ore, and fluorination time governed the extent of Ta and Nb recovery from a coltan ore. Meanwhile, financial cost, energy, and environmental footprints are critical aspects to be considered for industrial upscaling of the leaching process and they depend on the given process parameters which guarantee economically acceptable extraction levels. The analysis and optimization of process parameters through established statistical methods is a tangible practice in the above context. Therefore Taguchi orthogonal array has been used during the current experimental campaign to determine the optimal conditions for bringing Ta and Nb in solution.

2 Experimental

2.1 Materials

A representative sample of a coltan ore was collected within the perimeter of the Numbi deposit (South Kivu province, Democratic Republic of the Congo). The sample was divided into two batches, one was tested in DRC (Institut Supérieur Pédagogique de Bukavu) and another one was delivered to Belgium (University of Liège) for further instrumental analyses. The results reported in this study refer to tests with the first batch. This ore sample was dried, crushed and sieved using a 2 mm sieve. The undersize material was homogenized and a representative sub-sample was collected and prepared for characterization and testing. All samples were ground using a RM200 lab grinder.

Ammonium bifluoride (ABF) with an analytical purity of 99 % was obtained from VWR (Avantor) and was used as a fluorination agent while sulfuric acid (98 %, density =

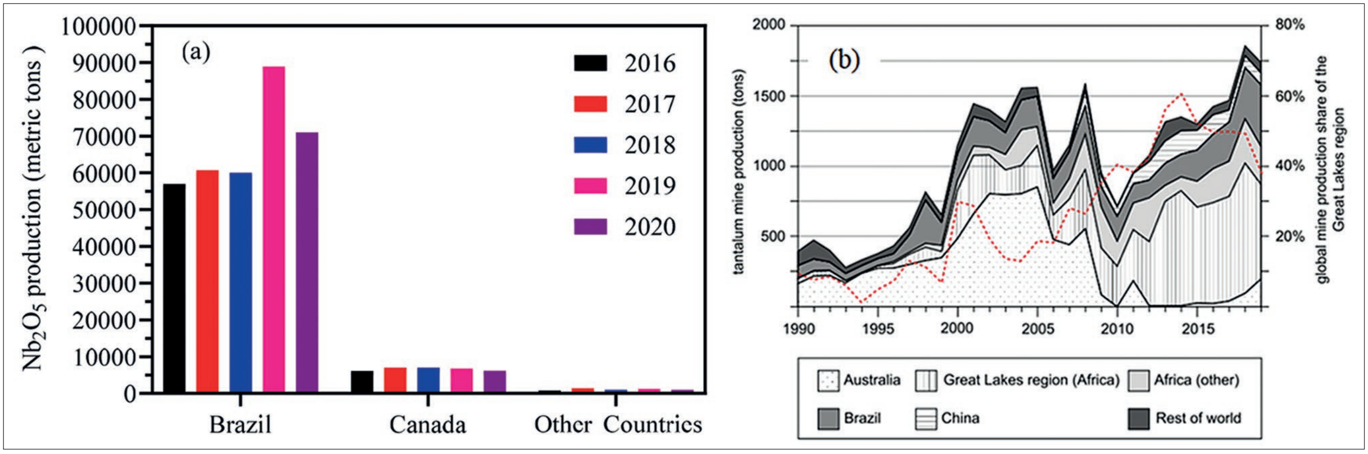


Fig. 1: Global production of: a) niobium concentrates from 2016 to 2020 [11-13], and b) tantalum concentrates 1990 to 2016 [14]

1.84), supplied by Central Drug House, was used to prepare the leaching media for the fluorinated ore. Distilled water was employed for preparation of solutions and washing of leached residues.

2.2 Ore characterization

The ore characterization details based on X-ray diffraction (XRD), Scanning Electron Microscope Energy Dispersive Spectroscopy (SEM-EDS), and Particle Inducted X-ray Emission (PIXE) are reported in our previous works [20, 21]. During XRD analysis, the pulverized sample was scanned between 3 and 90° 2θ at a rate of 0.02° 2θ per second. Mineralogical identification and semi-quantitative analyses were carried out through the Panalytical X'Pert

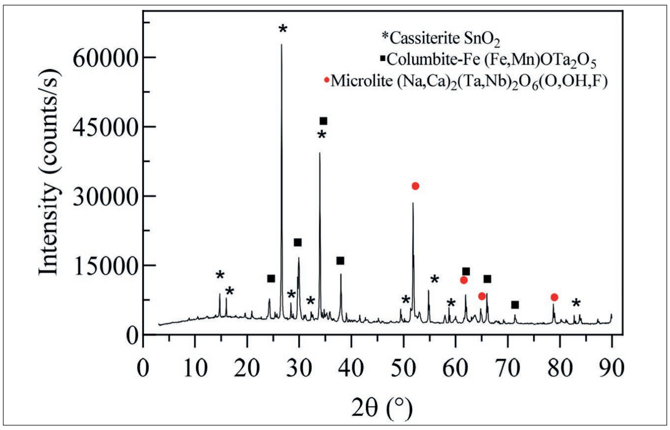


Fig. 2: XRD pattern of the Numbi raw coltan ore (from [21])

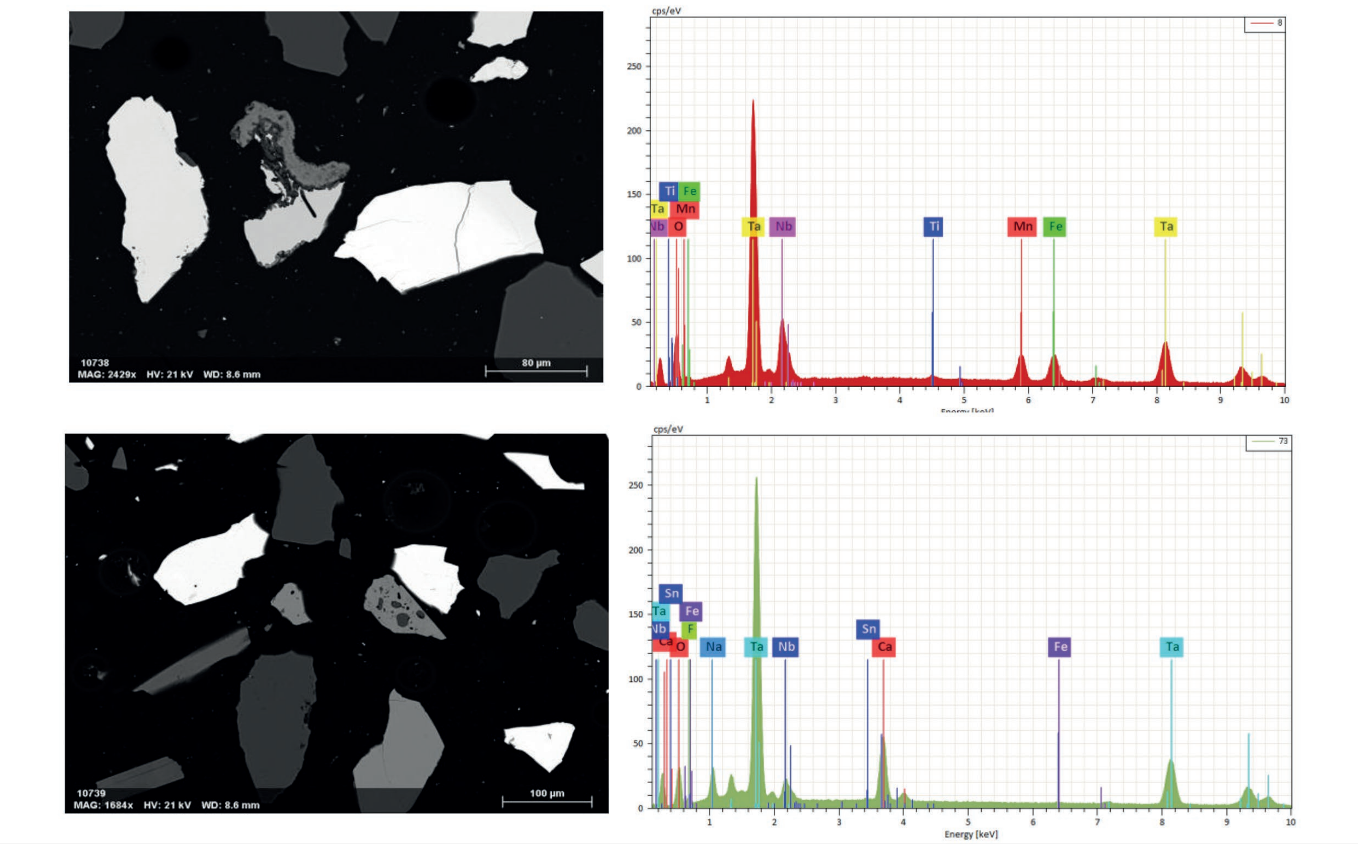
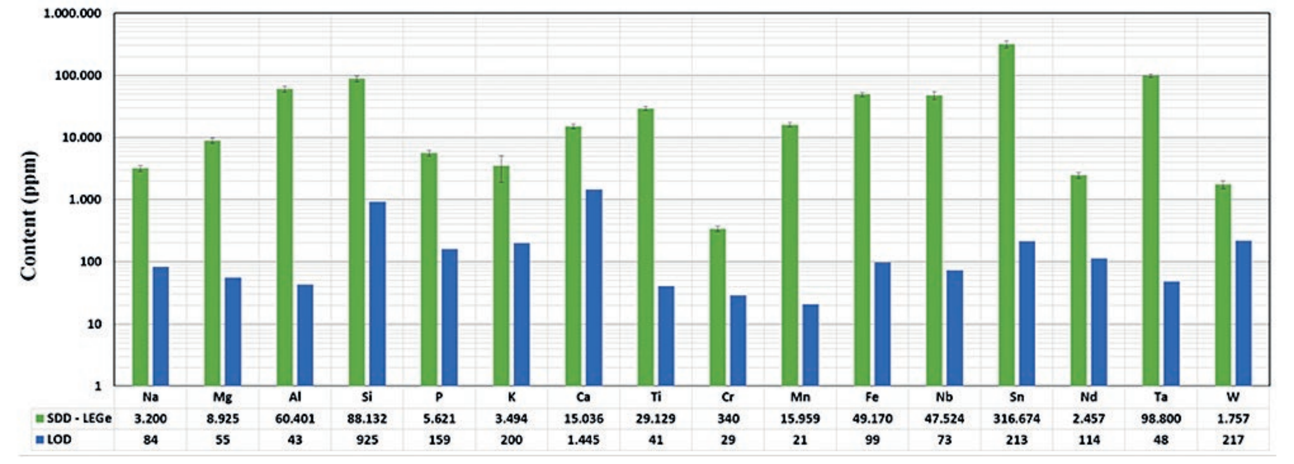


Fig. 3: SEM-EDS data of the Numbi coltan ore – similar SEM images are provided in [21]



	Na	Mg	Al	Si	P	K	Ca	Ti	Cr	Mn	Fe	Nb	Sn	Nd	Ta	W
🟩 SDD - LEGe	3.200	8.925	60.401	88.132	5.621	3.494	15.036	29.129	340	15.959	49.170	47.524	316.674	2.457	98.800	1.757
🟦 LOD	84	55	43	925	159	200	1.445	41	29	21	99	73	213	114	48	217

Fig. 4: PIXE data of the Numbi raw coltan ore (based on major elements)

PRO suite and the WebPDF4 + ICDD relational database. The XRD pattern of the raw coltan ore (Figure 2) indicates that columbite and microlite were the main Ta-Nb mineralogical types therein. This is also confirmed by the SEM-EDS observations (Figure 3) where coltan appears as a Fe-columbite phase – an association of Ta, Nb, Fe, and Nb, while microlite is an association of Na, Ca, F, Nb and Ta.

The PIXE results from the Numbi ore analysis are provided in Figure 4, indicating Sn, Ta, Nb and Ti as main metals of economic grades. The mineralogical and chemical compositions of the Numbi coltan ore are very different from those of the previously studied Lulingu coltan ore [20]. The Numbi ore is a mixture of cassiterite and coltan, the latter being closer to tantalite (Ta content > Nb content), whereas the Lulingu ore consisted essentially of columbite (Nb content > Ta content).

2.3 Experimental procedure

The fluorination and leaching procedures were adapted from the protocol detailed in [20]. All fluorination and leaching tests were performed inside a fluoroplastic (Teflon) vessel coupled with a polypropylene (PP) condenser following the Taguchi design experimental conditions (Table 1). The fluorination was carried out under agitation mode (300 rpm) and the vessel was coupled to a condensation unit to capture ($HF + NH_3$) gases. The fluorination was always performed on a 5 g coltan sample. A predetermined amount of ABF was added with respect to the required ABF-ore mass ratio and the mixture was introduced into a vessel that had been heated in advance to the desired temperature. At the same time, the reactor Teflon cover was placed on the reactor coupled to a PP condenser having a circulating cooling water. Magnetic stirring was then started and the reaction was carried out for a given time duration. After 60 min., the fluorinated product was cooled down to room temperature and then leached with 200 mL of sulfuric acid solution of a given concentration. The leaching step was performed at room temperature while stirring at 600 rpm for a fixed duration of 30 min. Upon leaching completion, the unreacted residue and the pregnant leach solution (PLS) were separated by filtration. The resulting phases (liquid and solid) formed the filtrate (PLS) and the leached cake, respectively. The latter was washed thoroughly with distilled water to remove traces of fluorine, dried in an oven at 60 °C for 24 h and finally weighed. The dried residue was then analysed by PIXE (or PXRF) to calculate the recovery as:

$$\text{Metal recovery} = \left(1 - \frac{M_r \cdot C_r}{M_i \cdot C_i}\right) \cdot 100,$$

where C_i is the initial content (wt.-%) of the metal in the raw coltan, and Cr is the concentration [wt.-%] of the metal in the insoluble residue. M_i is the weight of the input ore before fluorination, M_r is the weight of insoluble residue.

3 Results and discussion

3.1 Results based on the Taguchi design

The L_9 results are shown in Table 2. This table shows that more than 90 % of Ta and Nb could be leached in sulfuric acid solution (1 N) after being fluorinated using the ABF to ore mass ratio of about 5/1 and a fluorination temperature of 200 °C. It can be also seen that Fe, Mn, Si and Sn were leached to more than 90 % which is due to the fact that their fluoride complexes are highly soluble in acidic media. However, the observed high dissolution degree of iron during the experiments do not agree with results obtained by Kabangu & Crouse [23], which was less than 20 % when fluorination was carried out at 250 °C. This difference could be explained by the fact that the fluorinated products were water leached in [23], while in this study sulfuric acid solutions were used. Likewise, coltan leaching with a fluorine-based leaching reagent is not selective since it is impossible to bring Ta and Nb into solution without dissolving the remaining impurities.

The data shown in Table 2 were analysed by means of the Minitab 17 software to evaluate the effect of each parameter on the optimization criteria (maximization). The results are displayed in Figure 5. In order to identify the effect of the investigated fluorination parameters, the marginal values for Ta and Nb recovery were the focus.

The results from Figure 5c show that the most influential fluorination parameters are temperature and the Ore/ABF mass ratio. By contrast, the effect of granulometric fraction (Figure 5a) is less pronounced than the one of other parameters. The optimal level parameters which secure optimal leaching recoveries for Ta and Nb are the following: temperature of 200 °C, ABF/Ore mass ratio of 5/1 and granulometric fraction –75+45 µm. The XRD pattern of the residue resulting from the acid leaching of the fluorination

Test No.	Granulometric fraction [µm]	T [°C]	Ore/ABF mass ratio
1	–106+75	150	1/2
2	–106+75	180	1/5
3	–106+75	200	1/6
4	–75+45	150	1/5
5	–75+45	180	1/6
6	–75+45	200	1/2
7	–45	150	1/6
8	–45	180	1/2
9	–45	200	1/5

Table 1: Taguchi experimental design

Table 2: Ta and Nb recoveries based on the Taguchi design provided in Table 1

No.	Recovery [%]					
	Fe	Mn	Ta	Nb	Si	Sn
1	51.6	45.3	27.9	39.1	39.5	39.5
2	93.6	93.6	86.9	93.0	90.3	80.1
3	93.2	93.2	92.0	92.6	92.3	83.4
4	66.4	61.5	68.7	51.7	2.6	36.0
5	86.1	87.7	78.0	85.9	74.8	59.0
6	71.1	70.3	59.2	68.7	66.2	47.1
7	67.7	64.8	53.3	60.6	69.7	40.9
8	69.8	65.9	60.0	62.7	72.5	38.5
9	**96.3**	**96.3**	**96.0**	**96.0**	**94.6**	**85.7**

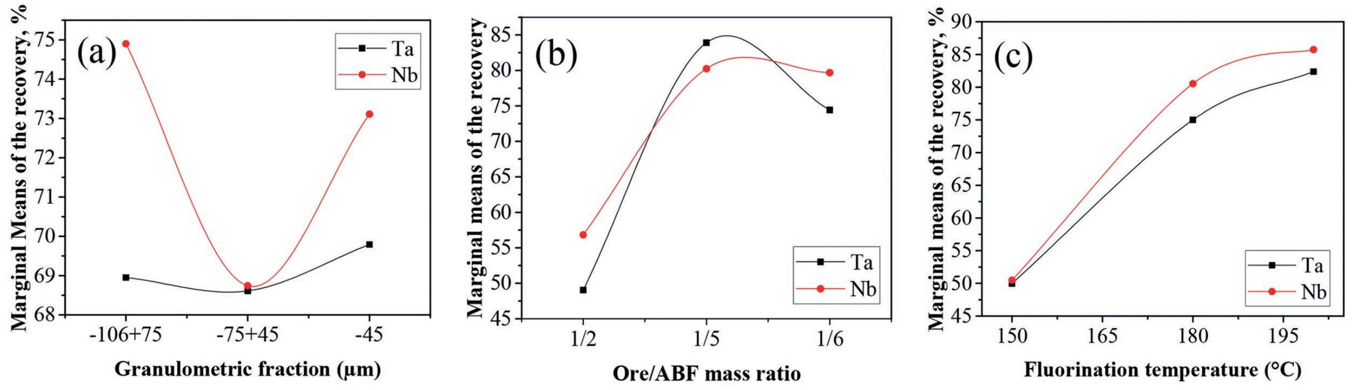


Fig. 5: Effect of fluorination parameters on the marginal means of the recovery of Ta and Nb

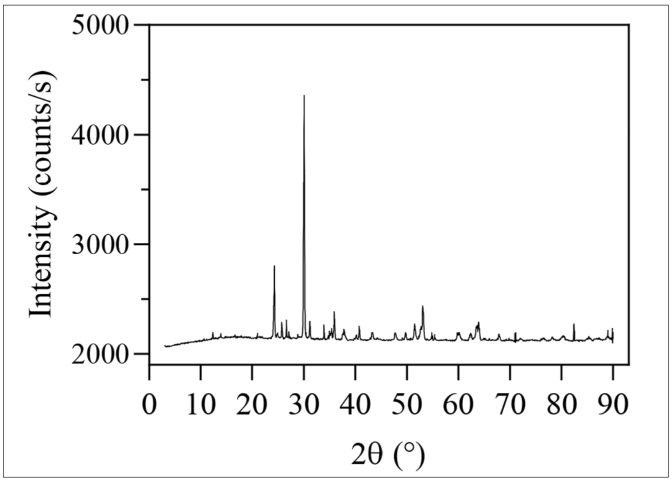


Fig. 6: XRD pattern of the leaching residue from ABF/ore mass ratio of 5/1

product at these optimal conditions is shown in Figure 6. The perusal of Figure 6 trends indicate that the coltan phase peak lengths decrease compared to the ones of the non-treated coltan ore (see Figure 2).

3.2 Effect of sulfuric acid solution

The effect of sulfuric acid concentration on the leaching of the fluorination product was studied under the following fluorination conditions: temperature 200 °C, ABF/Ore mass ratio of 5/1 and a particle size of –75+45 µm. The effect of acid concentration was studied in the range 0 to 2 N for a duration of 60 min. The results are shown in Figure 7.

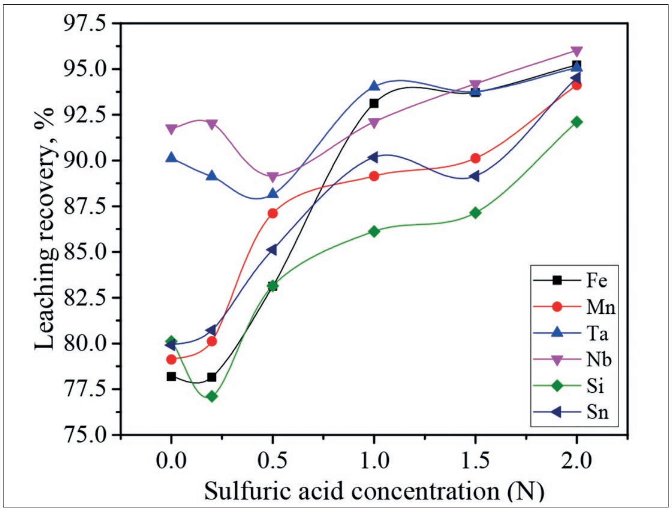


Fig. 7: Effect of sulfuric acid concentration on the leaching of Ta and Nb (coltan ore fluorinated using an ABF/ore of 5/1 at 200 °C)

The results in Figure 7 clearly show the effect of sulfuric acid concentration on metal dissolution. When the acid concentration is 0 N (aqueous leaching), the extent of Ta and Nb dissolution reach 90.1 % and 91.8 %, respectively. At the same concentration, the dissolution degrees for Fe, Mn, Si and Sn were 78.2 %, 79.1 %, 80.1 % and 79.9 %, respectively. A slight increase in the dissolution of these metals was observed when acid concentration was increased to 0.5 N. With a sulfuric acid concentration of 1 N, the relative increases in dissolution degree of Ta and Nb are quite negligible, being less than 3 %. From 0 to 2 N, there is a clear increase in the dissolution of metals. We can therefore conclude that sulfuric acid concentration provokes two effects inside the fluorinated product. The first is that if absent (concentration of 0 N), the dissolution of metal impurities is limited to less than 80 %, and the recoveries of Ta and Nb are 90.1 and 91.8 % respectively. The second is that when present, the dissolution of the target metals (Ta, Nb) reach values of 95.1 % Ta and 96 % Nb, the optimal concentration being of 1.5 N. This phenomenon reflects the fact that the presence of sulfuric acid enables hydrofluoric acid to be generated in situ during the reaction with fluorine ions present in the reaction medium. Two processes therefore take place during leaching of the fluorination product: dissolution of the fluoride complexes formed during fluorination, and leaching of the unconverted part of the ore by the HF generated in situ.

3.3 Leachates treatment perspectives

As mentioned earlier, Ta and Nb are not used together and therefore they need to be separated and recovered in advance as individual oxides. The reported studies on Ta and Nb separation from ammonium bifluoride leachates are predominantly focused on solvent extraction using a 2-OCL extractant [22, 23]. The usual composition of the leaching solution subjected to this step is about 2.5 g/L Ta and 1.2 g/L Nb. Apart from these two metals, other impurities such as Si, Sn, Fe and Mn are also commonly present. A solution with similar characteristics was studied in our earlier work [15] with the aim to compare the separation performance of three extractants, namely methyl isobu-

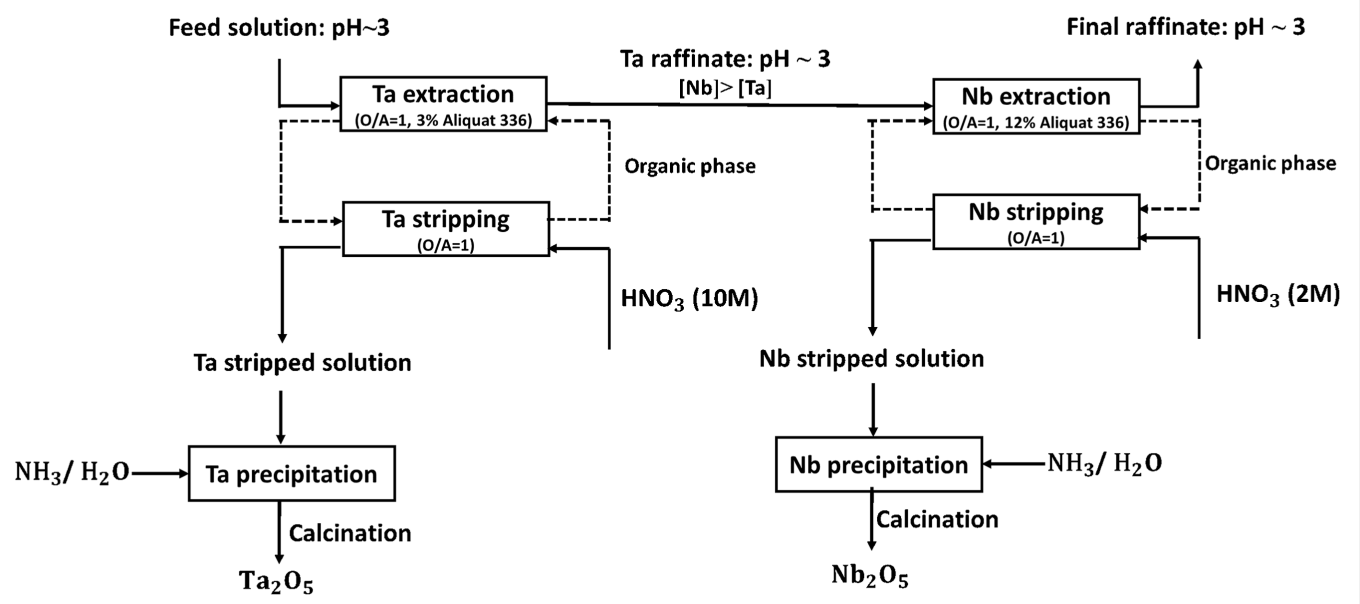


Fig. 8: Simplified flowsheet proposed for the extraction and stripping of Ta and Nb from ammonium bifluoride solution using Aliquat® 336

thyl ketone (MIBK), 2-Octanol (OCL) and Aliquat® 336. It was found out that the separation performance could be ranked in the following order: MIBK > Aliquat®336 > OCL. The results suggested that Aliquat® 336 could be ultimately selected as a viable extractant to replace MIBK at industrial scale due to its known process relevant advantages (higher flash point (132 °C) and lower solubility <0.5 %). The MIBK, scores as not suitable due to its very low flash point rendering it inflammable and hence not sufficiently safe for industrial applications. The idealized flowsheet involving two-stage extraction and one-stage striping stages targeting both metals using Aliquat® 336 is displayed in Figure 8.

4 Conclusion

This study confirms that ammonium bifluoride can be used as an alternative to the hydrofluoric acid in the extractive metallurgy of niobium and tantalum. The fluorination process parameters were optimized using Taguchi method in order to maximize Ta and Nb recoveries from a coltan ore originating from the DRC. The results show that more than 90 % of Ta and Nb can be recovered when the ore is fluorinated with ABF/ore mass ratio of 5/1 for 60 minutes, temperature of 200 °C and granulometric fraction of –75+45 µm.

The extraction degree of both niobium and tantalum could be enhanced considerably (close to 100 %) by introducing a weakly concentrated sulfuric acid as leaching agent for the fluorinated material. The resulting leachates could be processed downstream by involving solvent extraction towards yielding individual Ta and Nb products within commercial grades.

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