

Solvent Extraction of Tantalum and Niobium from Oxalic Leachates: A Novel Technique of Colombo-Tantalite Ores Processing

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ABSTRACT: Tantalum (Ta) and niobium (Nb) are two strategic metals which are extensively used in high-tech industries, such as microelectronics, defense, aerospace, manufacturing of high-strength low-alloy (HSLA) steel, etc. Current and future demand for these two metals will grow in parallel to high-tech equipment need expands. However, their extractive metallurgy remains a major challenge owing to economic and environmental constraints.

Apart the well-known hydrofluoric acid media, alkaline media has attracted reasonable attention in processing of low-grade Ta-Nb bearing ores, but it is limited in terms of Ta and Nb purification. In our previous studies, we proposed a novel method for coltan ore processing based on alkaline roasting, water leaching, precipitation and oxalic leaching of Ta and Nb with the aim to deliver a suitable leachate for downstream treatment by solvent extraction.

The current contribution reports results from the purification steps of Ta and Nb from an oxalic leachate containing about 11 g/L Nb and 2 g/L Ta. Solvent extraction was implemented using an Aliquat® 336-based extractant diluted in toluene and modified with 2% isodecanol. The used extractant is characterized by a higher flash point (132°C), compared to the conventional methyl isobutyl ketone (14°C) which is very hazardous. The studied parameters have included Aliquat® 336 concentration, contact time, pH, extraction temperature and O/A volume ratio. At their optimum found values, over 98% of Ta and Nb were extracted and purified from impurities and distribution coefficients of more than 300-Ta and 80-Nb were reached. Furthermore, selective stripping was carried out to separate the two metals, enabling to produce high-grade oxides.

Keywords: coltan ore, tantalum, niobium, solvent extraction, Aliquat®336

INTRODUCTION

Coltan ore contains two refractory metals of technological importance, namely tantalum (Ta) and niobium (Nb). Both metals are very similar in terms of their chemical and physical properties which makes their separation and purification difficult (Ayanda and Adekola, 2011). Their intrinsic properties, such as very high corrosion resistance, superconductivity, high electrical capacity, and biocompatibility make them highly strategic metals (Shikika et al., 2020). They have different applications, although being always found together in their ores. Niobium is mainly used in the production of ferroniobium for the manufacture of micro-alloyed steels which are used in the construction, ships, and refinery equipment, etc. Tantalum is the main component of miniaturized capacitors used in micro-electronics. Current industrial processes for the purification and separation of tantalum (Ta) and niobium (Nb) bearing columbite-tantalite (coltan) ores rely exclusively on hydrofluoric acid leaching and solvent extraction using MIBK (Shikika, 2023). However, due to the harmful effect of HF, more environmentally friendly processes deserve to be developed. Therefore, emerging processes such as alkaline and oxalic ones are increasingly being studied during the last decades.

Research has been conducted to extract Ta and/or Nb from leachates containing oxalic acid. Yang et al. (2015) reported on extraction of Nb from oxalic acid aqueous media using a N235-type extractant (R3N, with R= C8-10) diluted in octyl alcohol. However, the Nb stripping was not addressed in this study. Sun et al. (2021) reported on separation and purification of Ti and Nb from oxalic-sulfuric acid media using N235 and MIBK as extractants. Djordjević et al. (1966) studied the selectivity of amines including tri-n-octyl amine (TOA), tri-n-decyl amine (TDA), and tri-n-dodecyl amine (TDDA) diluted in carbon tetrachloride (CCl_4) during the extraction of Ta and Nb from a synthetic oxalic solution. Distribution coefficients of about 820 for Nb and 500, 740 and 690 for Ta were reported for the three amines TOA, TDA and TDDA respectively. Bhattacharyya and Ganguly (1986) studied the extraction of Ta and Nb from oxalic and hydrochloric acid media using a di(2-ethylhexyl) phosphoric acid (HDEHP) extractant diluted in n-heptane. The obtained results enabled approximately 85% of Ta to be extracted.

However, the conventional solvent extraction process is economically and ecologically unfavorable, as it generates toxic side products due to the use of a large amount of solvents and their losses resulting from volatility or partial blending with the aqueous phase (Turkowska et al., 2022). This work aims to investigate the feasibility of

efficient purification and separation of Ta(V) and Nb(V) from oxalic acid media using solvent extraction employing Aliquat® 336 extractant and conceptualize a process as an outcome.

MATERIALS AND METHODS

Leachates and Reagents

The aqueous phase (pH=1.1) used in this study presents a combined solution generated during oxalic leaching tests of Ta-Nb precipitates as described in our previous study (Shikika et al., 2022). The chemical composition of this stock solution is given in Table 1.

Table 1. Chemical composition of the aqueous stock solutions (mg/L)

Elements	Ta	Nb	Fe	Mn	Ca
Concentration, mg/L	1977	10513	291	67	610

All chemical reagents used, namely sulfuric acid (95%), nitric acid (65%), were of analytical grade, purchased from VWR (Avantor). Deionized water was used for aqueous solutions preparation. Isodecanol (98%) and Aliquat® 336 (98%) of analytical grade were purchased from Merck KGaA (Germany) and used without further purification, while kerosene was used as diluent. For the solvent extraction using Aliquat® 336, the organic phase consisted of Aliquat® 336 (extractant), a modifier (isodecanol, 3% v/v) and kerosene as diluent. Due to the elevated viscosity of the Aliquat®336, the mixture was stirred for at least 15 minutes to ensure complete dissolution prior to tests begin.

Extraction and Stripping Procedures

The extraction procedure consisted in contacting inside 250 mL beaker equal volumes of aqueous and organic phases under the selected experimental conditions. The both phases were agitated at 400 rpm using a magnetic stirrer. Once equilibrium time reached, the phases were separated through a separating funnel and the resulting aqueous phase was assayed as described in the following section.

Stripping experiments were conducted to back-extract and separate Ta and Nb from the Aliquat® loaded organic phases. The latter were generated using the optimal conditions being found during the solvent extraction of Ta and Nb. Stripping was performed by contacting equal volumes of the loaded organic and aqueous solutions. Agitation speed and contact time for phases mixing were 400 rpm and 10 minutes respectively. Following phases separation through a separating funnel, the resulting aqueous phase was also assayed.

Analysis

Ta, Nb, Ca, Fe and Mn concentrations in the aqueous phase were assayed by ICP-AES (Varian Liberty 100), whereas those in the organic phase were calculated based on the difference in metal concentration inside the aqueous phase before and after extraction. The distribution coefficient and the extraction efficiency were then determined as follows:

$$D = \frac{[M]_{\text{org}}^{\text{eq}}}{[M]_{\text{aq}}^{\text{eq}}} \quad (1)$$

$$E (\%) = \frac{(100 \times D)}{\left(D + \frac{V_A}{V_O}\right)} \quad (2)$$

where D represent the coefficient distribution, $[M]_{\text{org}}^{\text{eq}}$ and $[M]_{\text{aq}}^{\text{eq}}$ represent the metal concentration in organic and aqueous phase respectively, V_O and V_A are the volumes of organic and aqueous phases.

FT- IR spectroscopy (Nicolet iS5 FTIR spectrometer) was used in the wavenumber range of $400\text{--}4000\text{ cm}^{-1}$ to observe the structure of the loaded organic phase. To obtain a Ta/Nb-loaded organic phase of high concentration (more than 5g/L) for FT-IR analysis, 5 mL of organic phase was contacted with 40 mL of aqueous phase.

RESULTS AND DISCUSSION

Effect of the Aliquat® 336 Concentration

The effect of the concentration of Aliquat® 336 on the extraction of Ta and Nb was studied at room temperature using an O/A ratio of 1, a contact time of 15 minutes and the agitation speed of 400 rpm. Aliquat® 336 concentrations ranging from 3–14% were investigated. The results are shown in Figure 1.

The relationships seen in Figure suggest that the concentration of Aliquat® 336 has a pronounced effect on the distribution coefficient and the extraction efficiencies of Ta and Nb. The increase of the Aliquat® 336 concentration leads to raise in the distribution coefficient, as well as in the extraction efficiencies of both metals. High extraction efficiency and distribution coefficients of Nb (78% and 3.6) and Ta (42% and 0.73) were reached using Aliquat® 336 concentration of 14%.

However, given the chemical composition of the initial aqueous phase, the Ta and Nb distribution coefficients using O/A ratio of 1 came significantly lower than expected and hence need to be improved. Therefore, the O/A ratio was increased to 2, with the respective results being displayed in Figure 2 where the coefficients distribution and extraction

efficiencies of about 39.63 (98.75%)—Nb and 109.33 (99.54%) – Ta were reached. These results indicate that the solvent extraction mechanism of Ta and Nb by amines occurs via anion exchange (Deblonde et al., 2019a). An increased concentration of Aliquat® 336 logically leads to a large amount of anions likely to be released and complex further with the Ta and Nb oxalate complexes.

The slope method can be applied to process the data generated using the O/A ratio of 1 in order to investigate the extraction mechanism of Ta and Nb oxalate complexes

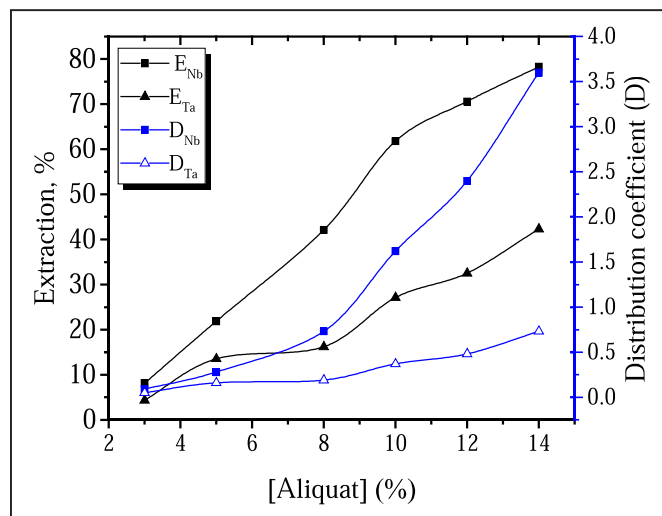


Figure 1. Effect of the Aliquat® 336 concentration on the extraction of Ta and Nb using O/A ratio of 1

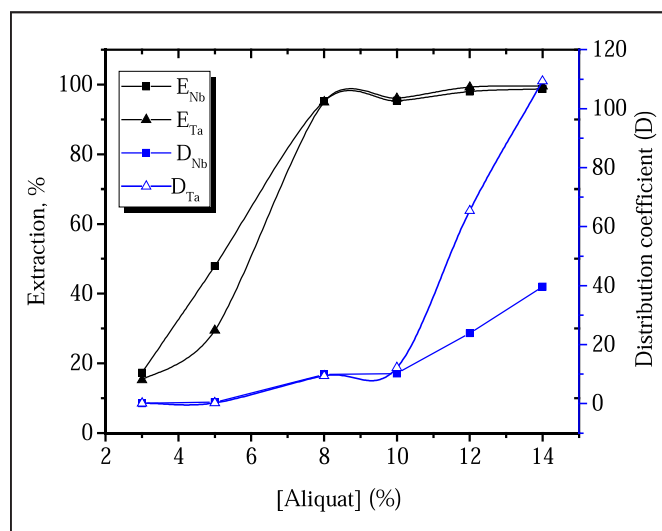


Figure 2. Effect of the Aliquat® 336 concentration on the extraction of Ta and Nb using O/A ratio of 2

by Aliquat®336. This method consists in plotting $\log D_{(Ta,Nb)} = f(\log [\text{Aliquat}^\circ 336, M])$. The slope of the resulting straight line should be close to 3, considering that Ta and Nb oxalate complexes are in the forms $TaO(C_2O_4)_3^{3-}$ and $NbO(C_2O_4)_3^{3-}$ respectively (Jehng and Wachs, 1991). Figure 3 illustrates the relationship between the logarithm of $D_{(Ta,Nb)}$ and that of Aliquat®336 concentration.

Figure 3 indicates that the trend lines for both metals have slopes closer to 2 rather than to 3. This suggests that not all of the Ta and Nb oxalate complexes involved in the extraction mechanism with Aliquat® 336 are met in the form of $TaO(C_2O_4)_3^{3-}$ and $NbO(C_2O_4)_3^{3-}$. Literature data (Deblonde et al., 2019b; Jehng and Wachs, 1991), affirm that Ta and Nb can form different complexes in oxalic media. Depending on the aqueous phase pH, Nb can present different oxalate complexes such as: $NbO(C_2O_4)_2(H_2O)_2^-$, $NbO(C_2O_4)_3^{3-}$, $NbO(C_2O_4)_2(OH)_2^-$, $NbO(C_2O_4)_2(OH_2)(H_2O)^-$, $Nb_2O_4(C_2O_4)_2(H_2O)_2^{2-}$. A similar chemistry is also valid for Ta, given its similarities with Nb. Based on these observations, we are inclined to assume that it is the bivalent complex that is extracted by Aliquat® 336.

FT-IR spectroscopy was used to identify the interaction pattern between Aliquat® 336 and Ta and Nb oxalate complexes within the 4000–400 cm^{-1} spectra region. The IR spectra of the organic phases of Aliquat® 336 and the organic phase loaded with Aliquat® 336-Nb (Ta) are depicted in Figure 4.

The presence of Aliquat® 336 functional groups in both IR spectra witnessed in Figure 4, confirms that the loaded organic phase comprises both oxalates-Nb(Ta) complex and Aliquat® 336. Both spectra show characteristic quaternary amine peaks at 1462 and 1383 cm^{-1} . These peaks occur at 1378 cm^{-1} and in the region from 1460 to 1467 cm^{-1} have been attributed to the vibrations of the N^+-CH_3 bonds of the quaternary amine (Emam and El-Hefny, 2023; Kunene et al., 2020; Lee et al., 2020). Peaks 2967 cm^{-1} and 2860 cm^{-1} are attributed to C–H bond vibrations. Peaks 1060 cm^{-1} (1051 in our case) and 724 cm^{-1} (727 in our case) are assigned to the C–N bond and Cl^- ion vibrational bands, respectively (Emam and El-Hefny, 2023). In the spectrum of the loaded organic phase, peaks 1051 and 727 cm^{-1} are shifted to 1021 and 715 cm^{-1} respectively, indicating that extraction of the Ta and Nb oxalate complexes has taken place. However, further investigations are required to identify the nature of extracted Ta and Nb complexes. The presence of a broad peak (3384 cm^{-1}) in the loaded organic phase spectrum is due to vibrations of O–H bonds,

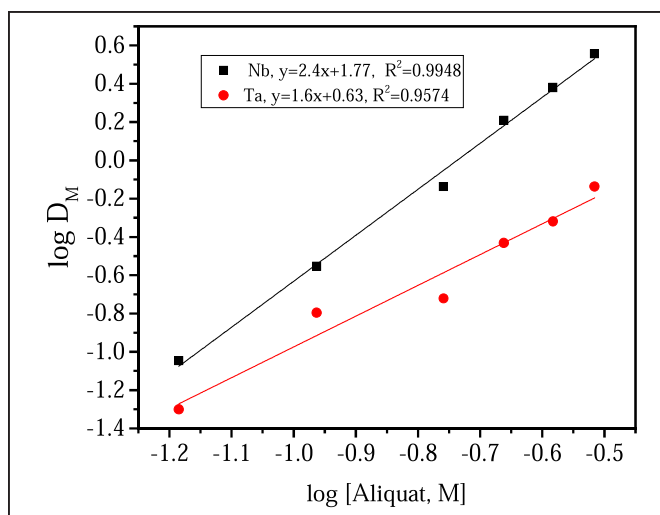


Figure 3. Linear relationship $\log D_{(Ta,Nb)} = f(\log [\text{Aliquat}^\circ 336, M])$ for the extraction of Ta and Nb from the aqueous oxalic phase

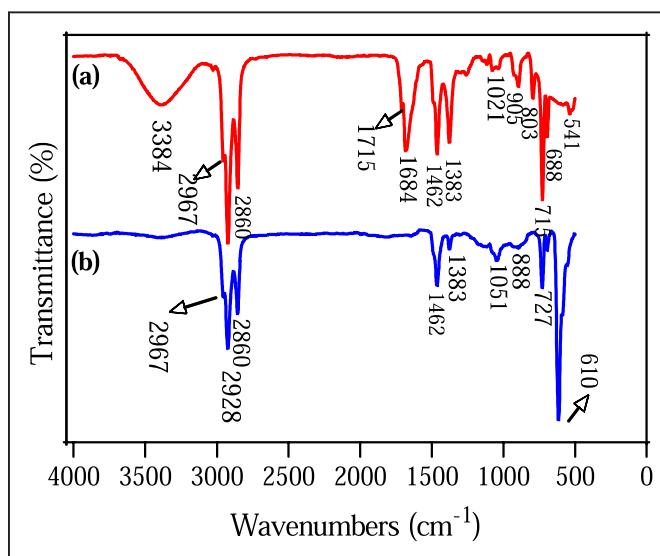


Figure 4. IR spectra of an Aliquat® 336 loaded with Ta and Nb oxalate complexes (a), and an organic phase of Aliquat® 336 (14% diluted in kerosene) - (b)

indicating the presence of water. This presence is probably attributed to the water molecule in the oxalic complexes of Ta and Nb.

Separation Factors of Ta and Nb Against Fe, Mn and Ca

The separation factors of Ta and Nb against Fe, Ca and Mn are shown in Figure 5. It can be seen that Ta and Nb could be successfully separated to a high degree from these impurities.

Effect of Contact Time on the Extraction Efficiency of Ta and Nb

The effect of contact time on the extraction efficiency of Ta and Nb was studied at room temperature using Aliquat® 336 concentration of 14%, an O/A ratio of 2, with pH of the aqueous phase being maintained at 1.1. The results are presented in Figure 6.

The results shown in Figure 6 indicate that complete coextraction of Ta and Nb could be achieved within 4 minutes of contact time. Exceeding the time over this period did not improve Ta and Nb extraction degree. The distribution coefficients for Nb and Ta decrease from 81 and 494 respectively to 39 and 329 when contact time increases from 4 to 15 minutes. A contact time of 4 minutes has been accordingly selected as an optimal one and used in further tests.

Effect of Aqueous Phase pH

The pH of the aqueous phase is an important parameter to evaluate during solvent extraction of Ta and Nb. It generally allows the chemical properties of Ta and Nb complexes to be modified, so that they could be separated with high selectivity factors. In the current work, sulfuric acid and NaOH (5M) solutions were used to adjust pH. Depending on the volume used to achieve a given pH value, Ta and Nb grades inside the aqueous phase were recalculated. The effect of the aqueous phase pH was studied at room temperature using an Aliquat® 336 concentration of 14%, O/A ratio of 2 and contact time of 4 minutes. These results are illustrated in Figure 7.

Figure 7 suggests that selective extraction of Ta and Nb is not possible through varying the pH of the aqueous phase. When pH is increased from 1.1 to 1.2, the

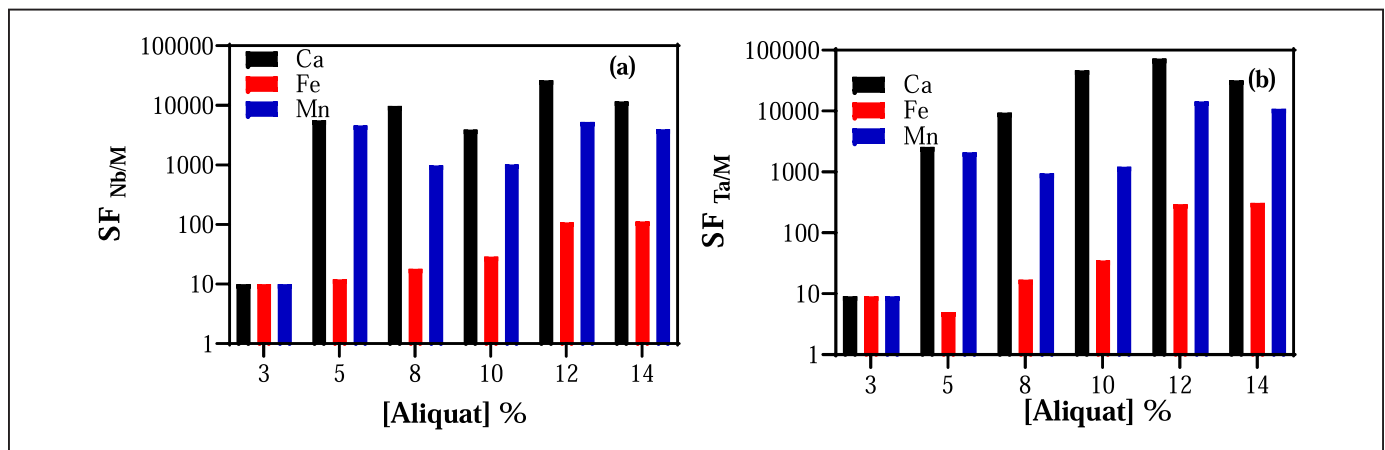


Figure 5. Separation factors of Nb (a) and Ta (b) with respect to Ca, Fe and Mn

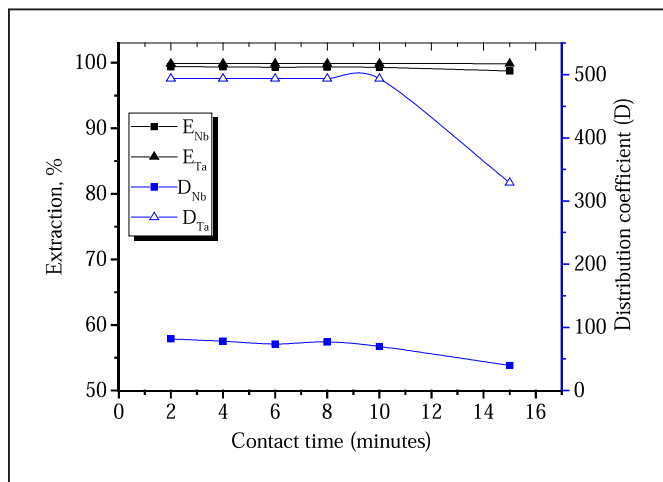


Figure 6. Effect of contact time on the extraction efficiency of Ta and Nb by Aliquat® 336

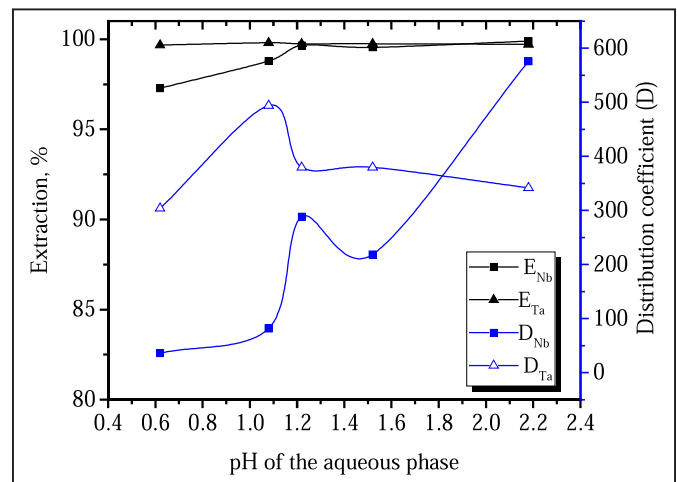


Figure 7. Effect from aqueous phase pH on the extraction of Ta and Nb

distribution coefficients of Nb and Ta change from 81 and 493 to 288 and 380 respectively. The variation in extraction efficiency for both metals above pH 1.2 is less pronounced and is less than 2%. The highest distribution coefficient of Nb ($D=576$) was observed when the pH of the aqueous phase was maintained at 2.2, while the one for Ta (493) was observed at pH of 1.1. On the other hand, decreasing the pH below 1.1 seems less significant since it leads to decrease in both the extraction efficiencies and the distribution coefficients of both metals. To avoid pH adjustments during solvent extraction, a pH range of 1.1–2.2 is recommended. However, to prevent the formation of emulsions that would result from the hydrolysis and polymerization of the Ta and Nb complexes in a slightly acidic medium, we maintain the starting pH of 1.1.

Effect of Extraction Temperature

The temperature of the aqueous phases may change significantly during solvent extraction. It is therefore important to study the extraction of Ta and Nb under different temperature conditions. Its effect was studied in the range of 293–318 K using Aliquat® concentration of 14%, an O/A ratio of 2, and a contact time of 4 min. The results from the trials in this direction are illustrated in Figure 8.

The enthalpy change (ΔH°) of Nb and Ta extraction was determined based on the slope of the plot of $\log D_M$ ($M=Ta, Nb$) versus $1000/T$ (K^{-1}) using the derived Van't Hoff equation (Sun et al., 2021):

$$\log D_M = -\frac{H^\circ}{2,303 RT} + C \quad (3)$$

where R is the universal gas constant and C is the integration constant. The integration constant was assumed to be constant at a given temperature and under the same experimental conditions.

Figure 9 evidenced a linear relationship between $\log D_M$ and $1000/T$, from which the ΔH° of the extraction reaction was estimated to be -6.7 kJ/mol (Nb) and -4.4 kJ/mol (Ta), suggesting a slightly exothermic nature of the extraction reaction in the range 293.15–318.15 K. Thus, a low temperature seems preferable during solvent extraction

of niobium and tantalum, without significant decreasing the distribution coefficients.

Effect of O/A Ratio

Figure 10 shows the results from the effect of the O/A ratio (0.2:1 to 2.5:1) on extraction efficiency under the most appropriate conditions: temperature of 20°C, concentration of Aliquat® 336 of 14%, pH of the aqueous phase 1.1, and a contact time of 4 minutes.

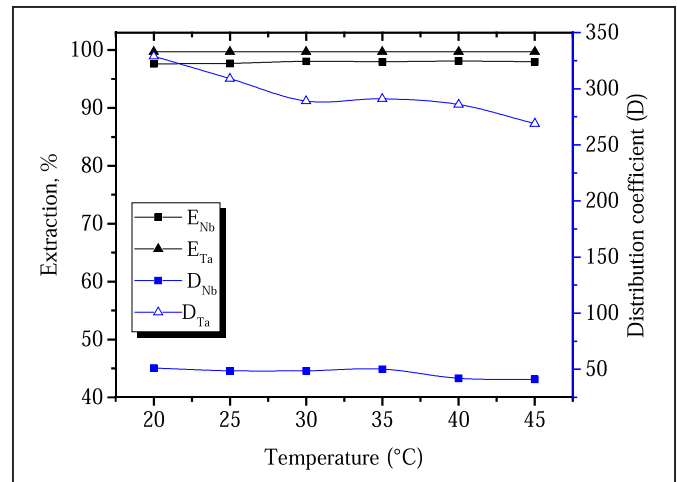


Figure 8. Effect of temperature on the extraction efficiency of Ta and Nb

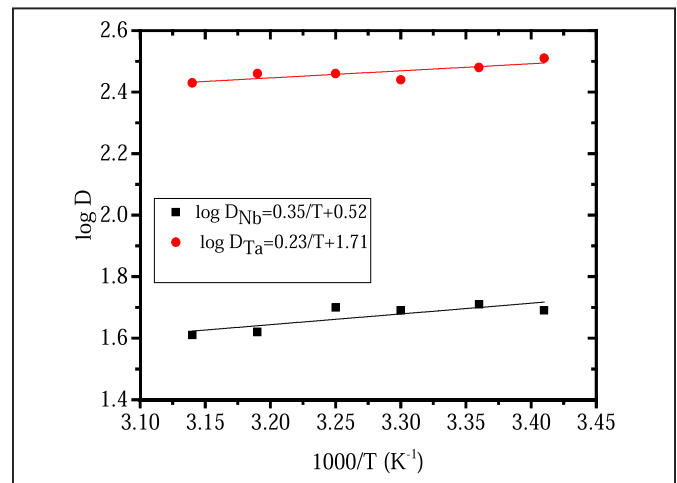


Figure 9. Relationship between $\log D_M$ vs $1/T$ for the extraction of Ta and Nb

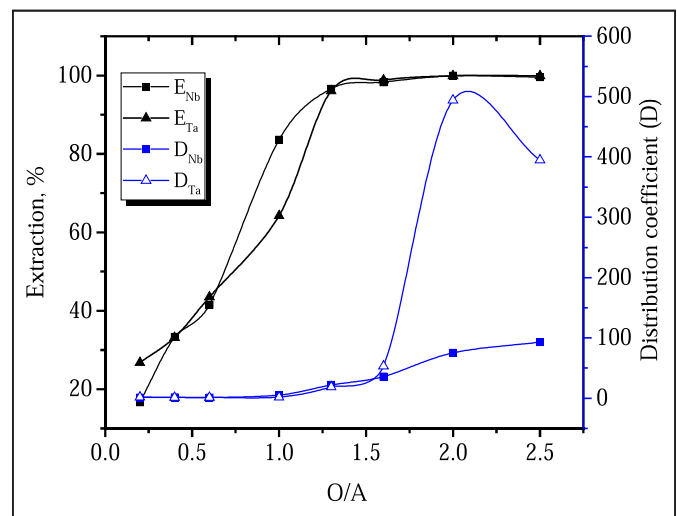


Figure 10. Effect of O/A ratio on Ta and Nb extraction

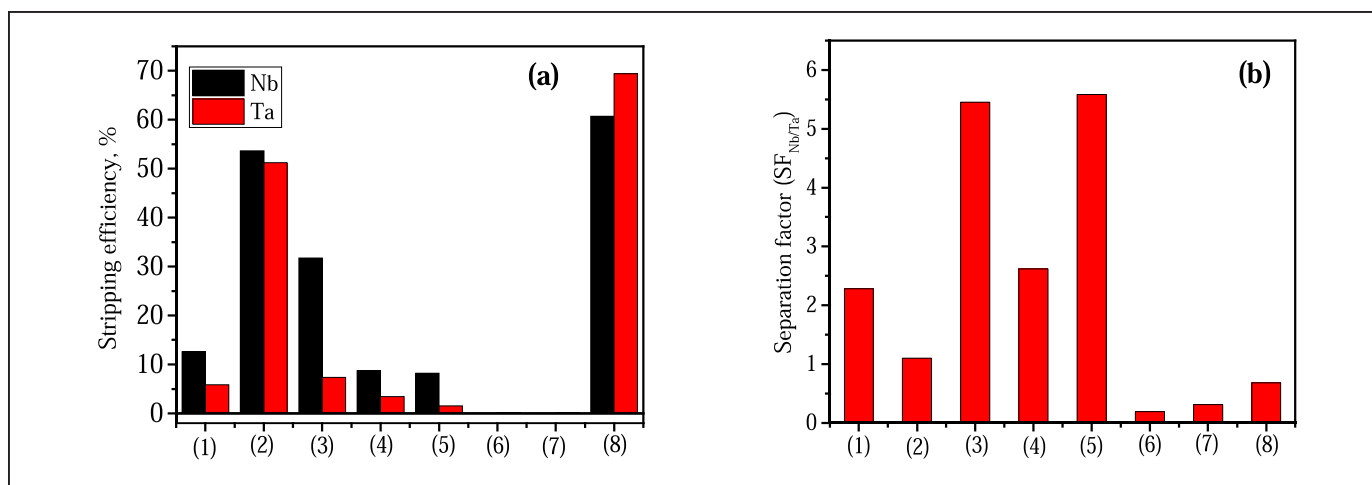


Figure 11. Stripping efficiency (a) with respective separation factor of Nb/Ta (b) from the loaded organic phase for different acidic solutions: (1) H₂SO₄ (2M), (2) HNO₃ (0.7M) +H₂C₂O₄ (0.5M), (3) HNO₃ (0.5 M), (4) NH₄F (1 M), (5) HCl (1 M), (6) H₂O, (7) H₂C₂O₄ (1 M), (8) H₂C₂O₄ (0.5 M) +HNO₃ (0.3 M) +NaNO₃ (0.15 M)

The relationships seen in Figure 10 indicate that the O/A ratio has a marked effect on the extraction and distribution coefficients of Ta and Nb. It is apparent that increasing the O/A ratio leads to an increase in the extraction efficiencies and distribution coefficient of both metals.

Based on the data shown in Figure 10, the corresponding equilibrium isotherms indicate for the case of Ta, a maximum Ta loading achieved in the organic phase (14% Aliquat 336) of about 2645 mg/L. For Nb, the respective loading was 8760 mg/L. These results showcase the ability of Aliquat 336 to serve as a promising extractant for separation of Ta and Nb from oxalic-based solutions. However, defining the theoretical number of stripping/extraction stages in view process upscaling would require further study on the effect of accompanying metals presence and the role of Ta and Nb repartitioning in the aqueous phase.

Ta and Nb Stripping

The co-extraction of Ta and Nb with Aliquat® 336 requires a selective back extraction for their separation and recovery. Stripping experiments were carried out in parallel with extraction experiments, where the loaded organic phase was obtained by equilibrating the organic and aqueous phase under the optimal found conditions, namely: 14% Aliquat® 336, 4 minutes of contact time, O/A phase ratio of 2, temperature of 20°C. Stripping experiments were conducted using an O/A phase ratio of 1:1 and employing different acid solutions, the results being summarized in Figure 11.

From Figure 11, it can be deduced that 0.5 M HNO₃ media (3) secures the highest separation factor of Nb against Ta (SF_{Nb/Ta}=6). However, the obtained Nb stripping efficiency is relatively low, being close to 32% and around 7%

for Ta. This low stripping efficiency shows that the stripping and separation steps for Nb from the Ta-loaded organic phase requires more stages to enable complete stripping of Nb.

CONCLUSIONS

A fluoride-free extraction and separation process of Ta and Nb has been investigated. The operational process conditions of solvent extraction were optimized to purify Nb and Ta from oxalic-based productive solutions. Almost complete extraction of Ta and Nb can be achieved using an O/A ratio of 2, aqueous phase pH of 1.1, Aliquat®336 concentration of 14% and an extraction temperature of 20°C. Stripping and separation of Nb using 0.5 M nitric acid solution, yield a separation factor of about 6 for Nb against Ta.

Ultimately, the obtained results are suggesting that an oxalic acid based process could be an economically feasible and environmentally responsible extractive metallurgical approach for sourcing Ta and Nb from primary resources.

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