

Proceeding Paper

Characterization, Classification, Dry High-Intensity Magnetic Separation (DHIMS), and Re-Grinding Techniques to Improve the Mineral Performance of a Sn-Ta-Nb Mineral Concentrate ⁺

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- + Presented at the 2nd International Electronic Conference on Mineral Science, 1–15 March 2021; Available online: https://iecms2021.sciforum.net/.

Abstract: Ta and Nb are considered critical raw materials due to their properties and potential applications in a wide range of sectors. This study deals with Sn-Ta-Nb minerals from the Penouta mine (Orense, Spain), the only active mine in Europe producing tantalum minerals. These minerals are obtained from mining waste accumulated during old mining operations in tailing ponds. The industrial processing flowsheet is based on successive gravimetric stages followed by low intensity magnetic separation to reduce ferromagnetic contaminants. A Sn-Ta-Nb concentrate, with grades between 35–45% Sn and 4–7% Ta₂O₅ and Nb₂O₅, is obtained in this stage with plant recoveries around 60–70%, respectively. A chemical-mineralogical characterization by size fractions, XRF and XRD was carried out to implement a size classification stage in the processing plant. The finest fractions, containing higher grades of well-liberated Sn, Ta, and Nb minerals were utilized as the feed for dry high intensity magnetic separation (DHIMS) multifactorial tests, while coarse fractions were regrinded to maximize performance. The favorable results obtained in these tests demonstrate that two products with commercial quality can be obtained: a cassiterite concentrate with grades between 70–78% SnO₂ and a tantalite-columbite concentrate with grades ranging between 12 and 14% Ta₂O₅ and Nb₂O₅, while also increasing the overall recovery of the plant.

Keywords: high intensity magnetic separation; Sn-Ta-Nb; critical raw materials; Penouta mine

Academic Editor: Paul Sylvester 1. Introduction

Published: 25 February 2021

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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/). The modern and competitive economy relies heavily on a range of critical raw materials owing to the risk in securing their supply and their economic importance [1,2]. Among these critical raw materials are Ta and Nb, with significant applications in the technological and aeronautical fields [3]. These elements are mainly contained in minerals such as columbite and tantalite, which come mainly from the mines of Brazil and Central Africa, respectively [2]. These mines are frequently managed by armed groups in conflict areas, so that commercialization of Ta and Nb as well as their forming minerals are regulated to prevent minerals obtained from illegal mines from entering into the supply chain [4]. In order to reduce dependence on imports of Ta and Nb in Europe, it is necessary to increase the investigation of deposits that contain these elements and the processes for their concentration in Europe, within the framework of the circular economy [5].

The Penouta mine (Orense, Galicia) is the only active mine in Europe that produces concentrates of Sn, Ta, and Nb as main products from the exploitation of old tailing ponds and dumps by Strategic Minerals Spain (SMS) [6–8]. The industrial processing flowsheet is based on a grinding-classification stage, multiple stages of gravimetric concentration

Menéndez-Aguado, J. Characterization, Classification, Dry High-Intensity Magnetic Separation (DHIMS) and Re-Grinding Techniques to Improve the Mineral Performance of a Sn-Ta-Nb Mineral Concentrate. *Environ. Sci. Proc.* **2021**, *6*, 10, https://doi.org/10.3390/iecms 2021-09344

Citation: Nava, J.; Llorens, T.;

Environ. Sci. Proc. 2021, 6, 10. https://doi.org/10.3390/iecms2021-09344



such as a battery of spirals and shaking tables, followed by a low-intensity magnetic separation to reduce ferromagnetic impurities such as iron oxides. A primary concentrate of Sn-Ta-Nb is obtained as a product, with grades between 35–45% Sn and 4–7% Ta₂O₅ and Nb₂O₅, and plant recoveries around 60–70%, respectively.

This study aims to identify the chemical-mineralogical characterization by size fractions of the Sn, Ta, and Nb primary concentrate to study the feasibility of continuing their mineral processing by carrying out operations such as classification by circular vibrating screen and dry high-intensity magnetic separation (DHIMS) multifactorial tests. The objective is to explore the possibility of obtaining independent concentrates of Sn and Ta-Nb that could be commercially more competitive, thus contributing to increased grades and the overall mineral recovery at the industrial plant of the Penouta mine.

2. Material and Methods

A representative sample of the Sn-Ta-Nb primary concentrate from the Penouta mine was taken to carry out these tests, characterizing chemically and mineralogically by size fraction through sieving and X-ray fluorescence (XRF) using a Bruker 4 kW set up in the ALS laboratory of the Penouta mine. Three size fractions were obtained, >150, 150/90, and <90 μ m. Fractions 150/90 and <90 μ m were used for the DHIMS multifactorial assays. The fraction >150 μ m was regrinded in order to achieve maximum yields of Sn, Ta, and Nb.

DHIMS multifactorial tests were developed using a Felemamg laboratory-scale induced rotor high intensity magnetic separator (Figure 1) at Felemamg facilities in Gijón, Spain.



Figure 1. Induced rotor high intensity magnetic separator. Felemamg facilities in Gijón, Asturias, Spain.

Table 1 shows the operational variables studied by size fractions for the DHIMS tests. The intensity of the magnetic field was varied, changing the intensity (A) of the electric field, where the maximum intensity is 21,000 G. Subsequently, the non-magnetic products resulting from all the tests were reprocessed again with the DHIMS in order to simulate industrial equipment with two induced rotors. A first test with a wide granulometry (150/0 μ m, test 1 showed in Table 1) was carried out; five tests were conducted with the 150/90 μ m fraction (tests 2–6) and another five tests with the <90 μ m fraction (tests 7–11). Each product obtained, magnetic and non-magnetic, was analyzed by XRF to determine

the grades and yields for the mineral species of interest, thus evaluating the feasibility of implementing a high-intensity magnetic separator on an industrial scale in the Penouta mine. The phase compositions were characterized by X-ray diffraction analysis (XRD) using a Siemens D5000 (Siemens, Berlin, Germany) apparatus and quantified by the Rietveld method on the tantalite-columbite concentrate <90 μ m obtained, in order to identify mineral phases that could be responsible for decreasing grades of Ta and Nb, and then to study the possibility of continuing its processing.

Test N⁰	Size Fraction (µm)	Magnetic Field Intensity (A)	Roll Speed (rpm)
1	150/0	Low	High
2	150/90	Low	High
3	150/90	High	High
4	150/90	High	Low
5	150/90	Low	Low
6	150/90	Changing split inc	lination
7	90/0	Low	High
8	90/0	High	High
9	90/0	High	Low
10	90/0	Low	Low
11	90/0	Changing split inc	lination

Table 1. DHIMS assays configuration. Due to SMS data protection, they are considered qualitatively.

Afterwards, representative samples of the fraction >150 μ m of the primary concentrate were prepared to carry out the regrinding tests in a 0.18 m diameter mill, 4.5 L capacity, charged with 7 kg of balls, resulting in 20.74% filling of balls (156 balls of 19.06 mm and 42 balls of 29.72 mm); 1020 g of mineral for 9.3% mineral filling and 75% critical speed (80.23 rpm). Subsequently, the grinded products were chemically and mineralogically analyzed by size fraction after five different milling times: 0.5, 2.5, 5, 10, and 15 min, throughout sieving and XRF analysis, respectively. Finally, the evolution of the percentage passing through the 100 μ m mesh was studied with respect to time, as this is the liberation size of tantalite [9–11], as well as the distribution of the minerals of interest.

3. Results and Discussion

3.1. Characterization and Classification of the Sn, Ta, and Nb Primary Concentrate

The results obtained from the assay by size study, as well as the metallic distribution of the elements of interest for the Sn, Ta, and Nb primary concentrate, are reported in Table 2.

This primary concentrate shows a D₈₀ of 99 μ m. The calculated feed grades were 33.75% Sn, 5.24% Ta and 4.64% Nb, with significant MnO, Fe₂O₃, and SiO₂ contents. It can be noted that Sn, Ta, and Nb contents increase with decreasing particle size, so that the highest concentration of these metals are found below 106 μ m; on the contrary, MnO, Fe₂O₃, SiO₂, and MgO are commonly concentrated in coarser fractions, according to the findings of [10,11]. In order to try to remove these impurities as much as possible from the coarser fractions prior to carrying out the magnetic separation tests, a vibrating circular sieve was used, throughout 150 and 90 μ m meshes (Figure 2), thus obtaining three size fractions: >150, 150/90, and <90 μ m. The two finest factions (150/90 and <90 μ m) would be used as the feed for the DHIMS multifactorial tests, while the fraction >150 μ m would be regrinded.

Size	ze Retained Weight Ac		Accumulated Weight		Grades (%)						
(µm)	(g)	(%)	(%)	Sn	Ta ₂ O ₅	Nb_2O_5	Fe ₂ O ₃	Al ₂ O ₃	MnO	SiO ₂	
300	0.00	0.00	100.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
212	3.30	0.28	99.2	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
150	25.30	2.14	97.58	11.80	2.77	2.58	13.30	14.20	23.50	20.4	
106	136.10	11.51	86.07	14.85	3.30	3.23	12.60	12.75	20.80	17.5	
63	434.30	36.74	49.32	26.50	5.05	4.99	8.59	7.32	12.75	13.4	
45	330.10	27.93	21.40	41.50	6.06	5.13	3.15	1.80	4.64	7.19	
38	118.90	10.06	11.34	45.10	5.94	4.69	1.73	0.58	2.32	6.30	
0	134.00	11.34	0.00	52.30	5.93	4.19	1.22	0.11	1.44	4.99	
Total:	1182.00	100.00	Calculated feed grade:	33.75	5.25	4.64	6.08	5.04	9.28	10.5	

Table 2. Assay by size of Sn, Ta, and Nb primary concentrate from the Penouta mine

Size (µm)			Distributior	n of Metal Co	ntent (%)		
	Sn	Ta ₂ O ₅	Nb ₂ O ₅	Fe ₂ O ₃	Al ₂ O ₃	MnO	SiO ₂
300	0.00	0.00	0.00	0.00	0.00	0.00	0.00
212	0.00	0.00	0.00	0.00	0.00	0.00	0.00
150	0.75	1.13	1.19	4.68	6.04	5.42	4.12
106	5.07	7.24	8.02	23.85	29.16	25.82	19.08
63	28.85	35.05	39.51	51.88	53.42	50.51	46.50
45	34.34	32.05	30.88	14.46	9.98	13.97	18.96
38	13.44	11.39	10.17	2.86	1.16	2.52	5.99
0	17.57	12.82	10.24	2.27	0.25	1.76	5.34
Total:	100.00	100.00	100.00	100.00	100.00	100.00	100.00

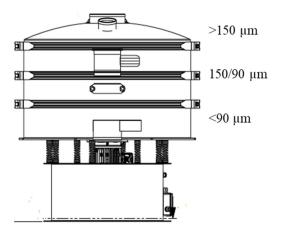


Figure 2. Scheme of the vibrating circular sieve used to obtain three size fractions: >150, 150/90, and <90 μ m.

3.2. Results of Laboratory Scale DHIMS Multifactorial Tests

3.2.1. Grades and Recoveries of the New Concentrates Obtained

The fractions 150/90 and <90 μ m obtained in the previous classification stage were selected for the DHIMS multifactorial tests. Two products were obtained after DHIMS performance: a magnetic concentrate of columbite–tantalite and a non-magnetic concentrate of cassiterite. Grades and recoveries are reported in Tables 3 and 4, respectively, for each of the fractions and variables considered in Table 1.

Table 3 shows that the best grades of Ta₂O₅ and Nb₂O₅ in the columbite–tantalite concentrate were obtained with test #6 for the 150/90 μ m fraction, reaching grades of 11.71% Ta₂O₅ and 12.21% Nb₂O₅, while test #7 was the best configuration for the <90 μ m fraction,

overtaking grades of 13.66% Ta₂O₅ and 13.21% Nb₂O₅. Specifically, test #7 showed a better Ta₂O₅ grade than test #1, which was carried out with a wider size 150/0 μ m.

Mineral	Ta	2 O 5	Sn	ıO2	Nb	2 O 5	Si	O 2	Fe	2 O 3	M	nO	
Test No.	G	R	G	R	G	R	G	R	G	R	G	R	
Test Ino.	(°	%)	(%)		()	(%)		(%)		(%)		(%)	
1	12.20	57.62	7.10	2.95	12.65	76.76	8.97	30.87	8.74	65.87	12.71	79.18	
2	10.33	68.18	3.34	2.09	11.10	89.39	13.44	52.81	12.01	83.44	17.36	96.14	
3	10.15	68.09	3.33	2.11	11.02	88.59	13.83	55.02	12.13	83.38	17.51	95.22	
4	8.80	69.82	26.99	19.18	8.79	85.61	9.41	49.87	7.62	78.34	10.84	89.55	
5	8.79	68.96	28.09	19.82	8.78	85.97	9.31	50.78	7.50	78.38	10.66	90.97	
6	11.71	70.11	7.29	3.87	12.21	91.88	10.13	42.86	9.47	80.89	13.86	97.86	
7	13.66	67.78	10.09	4.23	13.21	89.42	6.30	25.49	6.83	71.40	10.04	92.31	
8	13.47	67.80	10.77	4.61	13.61	90.95	6.15	24.76	6.65	70.24	9.75	91.11	
9	8.89	76.07	39.01	28.59	8.23	91.63	5.47	36.89	4.37	76.75	6.01	94.01	
10	9.03	76.34	37.93	26.69	8.42	94.08	5.38	35.81	4.38	77.93	6.03	95.78	
11	13.13	68.24	13.65	5.93	13.24	91.15	5.36	22.51	5.89	69.52	8.69	90.58	

Table 3. Columbite-tantalite concentrate yields resulting from DHIMS tests. G: grades and R: recovery.

Table 4. Cassiterite concentrate yields resulting from DHIMS tests. G: grades and R: recovery.

Mineral	Τa	a2 O 5	Sn	O ₂	Nb	2 O 5	Si	iO2	Fe	2 03	Μ	nO	
Test No.	G	R	G	R	G	R	G	R	G	R	G	R	
Test No.	(%)		(%)		(%	(%)		(%)		(%)		(%)	
1	1.84	27.12	75.06	97.51	0.41	7.78	6.56	70.89	0.62	14.61	0.13	2.53	
2	2.01	26.37	75.57	93.73	0.58	9.25	6.36	49.62	0.84	11.59	0.29	3.19	
3	1.95	25.00	76.96	93.40	0.49	7.50	6.34	48.28	0.72	9.47	0.21	2.19	
4	2.13	24.93	73.91	77.48	0.78	11.18	6.56	51.28	0.88	13.35	0.51	6.21	
5	2.15	25.13	73.91	77.61	0.80	11.67	6.58	53.42	0.86	13.37	0.52	6.61	
6	1.89	25.76	78.23	94.39	0.41	7.07	6.27	60.37	0.61	11.86	0.12	1.93	
7	1.85	26.81	76.58	93.96	0.37	7.13	6.67	78.87	0.57	17.43	0.11	2.96	
8	1.85	26.93	77.34	95.57	0.38	7.26	6.70	77.81	0.58	17.67	0.11	2.97	
9	1.65	18.67	74.17	71.30	0.35	5.12	7.52	67.25	0.53	12.35	0.11	2.28	
10	1.65	19.17	74.68	72.33	0.34	5.21	7.63	69.90	0.54	13.23	0.10	2.19	
11	1.79	26.26	75.57	92.72	0.36	6.96	6.73	79.90	0.55	18.36	0.10	2.95	

Table 3 also shows that the best recoveries for the 150/90 μ m fraction resulted from #6, with 70.11% and 91.88% of Ta₂O₅ and Nb₂O₅, respectively. In contrast, #10, with recoveries of 76.34% and 94.08% of Ta₂O₅ and Nb₂O₅, respectively, was the best configuration for the <90 μ m fraction. Such results are in accordance with the liberation size below 100 μ m reported for the columbite–tantalite [9–11], thus demonstrating the influence of the size particle on the increase of the grades and the recoveries of the species. Furthermore, the fact of a higher content of SiO₂, Fe₂O₃, and MnO in the columbite–tantalite concentrate in coarser granulometry stands out [12].

Regarding the non-magnetic cassiterite concentrate, the best grades and recoveries of SnO₂ were obtained in #6 and #8 tests, for the 150/90 and <90 μ m fractions, respectively, reaching grades of 78.23% and 77.34% SnO₂ and recoveries of 94.39% and 95.57% SnO₂, respectively (Table 4). Therefore, test #6 is considered the optimal configuration for the 150/90 μ m fraction, since it shows the best results for both grades and recoveries of SnO₂ and Ta₂O₅. Whereas, the fraction <90 μ m through the configuration of #7 shows the best results to favor the increase of Ta₂O₅ and Nb₂O₅ grades and one of the best for SnO₂.

The XRD pattern of the columbite–tantalite concentrate <90 μ m (Figure 3) shows the presence of minerals such as xenotime, cassiterite, aluminum silicates of Mn, goethite, and zircon as main impurities associated with the predominant columbite–tantalite (Mn) [12]. These results complement the information obtained by XRF (Table 3), confirming that elements such as Mn and Fe are intrinsically associated with the mineralogy of columbite–tantalite, indeed. Consequently, their physical separation is impossible.

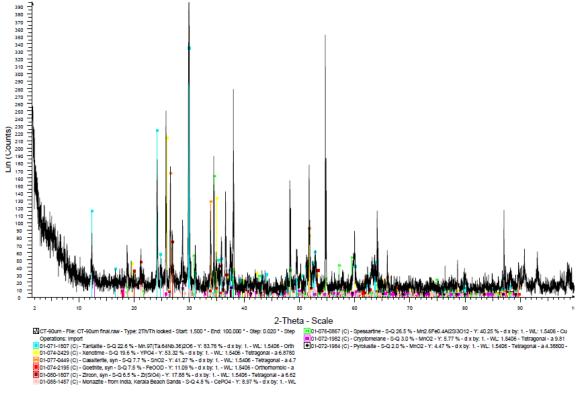


Figure 3. XRD pattern of the columbite-tantalite concentrate sample <90 µm.

3.2.2. Influence of Operational Parameters in DHIMS Tests

According to the results shown in Tables 3 and 4, a higher roller speed seems to favor greater recoveries, not only for the cassiterite in the non-magnetic product, but also for columbite–tantalite in the paramagnetic product obtaining good grades, as a better separation of the streams occurs, which allows the adjustment of the split in order to increase the cassiterite grades in the non-magnetic product and recovery of tantalite in the magnetic product.

Regarding the intensity of the magnetic field, as the particle size is smaller, a greater intensity is necessary than with a coarser granulometry for the same roller speed. Likewise, the results suggest that a finer particle size favors both grades and the recovery of Ta₂O₅ and SnO₂.

In summary, when the feed is classified in two fractions, the selectivity of the operation increases, obtaining better results than for larger size fractions such as $150/0 \,\mu$ m.

3.3. Regrinding of >150 µm Fraction

The assay by size of the resulting products passing the 100 μ m mesh, after regrinding of the >150 μ m fraction for each grinding time (0.5, 2.5, 5, 10, and 15 min) are shown in Table 5.

Time (min)	Pass Weight through	rough P ₈₀ Distribution of Metal Content (%)							
	100 µm (%)	(µm)	Sn	Ta ₂ O ₅	Nb_2O_5	Al ₂ O ₃	Fe ₂ O ₃	MnO	SiO ₂
0.5	11.20	218	26.26	21.07	20.44	5.59	7.45	5.45	7.13
2.5	17.60	197	38.25	30.81	29.66	8.77	11.67	8.91	11.39
5.00	22.40	192	43.74	36.57	34.43	12.93	16.53	13.13	15.60
10.0	30.90	185	51.09	46.81	43.13	20.75	26.47	22.14	23.05
15.00	32.90	184	50.39	47.47	43.93	24.44	29.89	25.54	26.49

Table 5. Evolution of the P₈₀ with respect to time and assay by size results on the regrinded >150 μ m fraction.

Table 5 shows that as grinding time increases, the amount of milled material passing through the 100 μ m mesh increases. The *P*₈₀ with respect to time highlights the little difference between 10 to 15 min, suggesting the optimum time as 10 min for grinding to avoid regrinding of fines and loss of energy efficiency. Furthermore, the distribution of the metal content in the species of interest of the milled product passing through the 100 μ m mesh after the milling time shows an increase, reaching a higher liberation of at least 50% in the species of interest such as Sn, Ta, and Nb after 10 min.

4. Conclusions

From the present research it is concluded that by implementing a size classification, better grades and recoveries of Sn, Ta, and Nb are obtained when performing DHIMS instead of using a wide granulometric interval (150/0 μ m), since selectivity increases during operation. Consequently, a circular vibrating screen was implemented to generate three size fractions. The finest fractions (150/90 and <90 μ m) were used as the feed of DHIMS multifactorial tests; the coarsest fraction (>150 μ m) was regrinded in a closed-circuit ball mill installed at the Penouta mine plant.

The multifactorial DHIMS tests on the Sn, Ta and Nb primary concentrate generated two new products: a magnetic columbite–tantalite concentrate and a non-magnetic cassiterite concentrate with better grades, being therefore rather commercially competitive, thus leading to better profitability and feasibility for the Penouta mine project. The results for these tests suggest that a finer particle size favors both the grade and the recovery of Ta₂O₅, Nb₂O₅, and SnO₂; hence, it is key to control the grinding-classification circuit; maintain the *D*₈₀ of the primary concentrate and a better liberation of the mineral species of interest, in order to reproduce the results obtained in this investigation.

This work proposes conducting electrostatic separation multifactorial tests to cut down on minerals such as zircon, monazite, and xenotime and to purify the paramagnetic and non-magnetic concentrates obtained, as well as passing the columbite–tantalite concentrate thorough the DHIMS to increase the grades of Ta₂O₅ and Nb₂O₅. Regarding the regrinding tests on the fraction >150 μ m, the optimum time was 10 min to obtain a well-liberated matter for the mineral species of interest: Sn, Ta, and Nb.

Author Contributions: Conceptualization and experiments, J.N.; methodology, J.N.; formal analysis, J.N. and T.L.; investigation, J.N.; data curation, J.N.; writing—original draft preparation, J.N.; writing—review and editing, J.N., T.L., and J.M.-A.; supervision, T.L. and J.M.-A. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Oviedo Siembra Talento Industrial Ph.D. grant from the Council of Oviedo, in coordination with the University of Oviedo.

Institutional Review Board Statement: Not applicable

Informed Consent Statement: Not applicable

Acknowledgments: The authors thank Strategic Minerals Spain, S.L., for access to concentrates and Felemamg for the use of its facilities and equipment to conduct the tests.

Conflicts of Interest: The authors declare no conflict of interest.

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