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Summary

Deliverable D1.2 concerns the concentration treatment performed on W-bearing mine waste (MW) selected under RAWMINA project: W-tailings coming from site 3 in Portugal which initial preparation is described in D1.1. This MW was considered as the only MW suitable for such physical treatment regarding available techniques: gravimetric separation and froth flotation.

Planning work on this MW and associated deliverable have been delayed since preparation priorities have been given to deliver three other MW samples to WP2 - two main sulphidic materials and one carbonated sample.

W-bearing MW has been prepared by classical grinding, classified under four main particle size fractions, chemically analysed, and then submitted to gravimetric separation to concentrate heavy minerals: W-bearing phases (ferberite and scheelite) and also sulphides (pyrite, arsenopyrite).

Two main gravimetric techniques have been used at BRGM facilities: shaking table method and centrifugal technology (MGS). Several lab tests have been performed to identify better operating conditions on the different size fractions; and a pilot operation has been performed to produce around 8 kg of concentrate named 'CC1' for further work under WP1 and WP2.

Characterization includes pXRF to orientate gravimetric tests at BRGM, chemical analyses by ICP-OES and C, S speciation at AGQ laboratories and mineralogy (XRD, SEM-EDX) at UAB.

Concentration factor of W, which content in the concentrate is measured to be 3.6 %, is around 10. As, Fe, Cu and sulphides have also been upgraded. Main minerals quantified by XRD are Al-silicates (62 %), quartz (15 %), pyrite (16 %), ferberite (5 %) and sheelite (2 %). Several flowsheets have been established in relation with W content.

In addition, froth flotation tests have been performed at AGQ on the finest size fractions (0-250 μm and 40-100 μm). W recovery is similar to MGS results; and lower compared to results on coarser fractions by shaking table.

Finally, two flowsheets have been calculated for W recovery: one related to gravimetric concentration processes (shaking table and MGS) and another one related to froth flotation and gravimetric process by shaking table.



1. Introduction

Under WP1, several metallic mine wastes (MWs) from Spain, Portugal and Chile have been selected for its evaluation in RAWMINA project. Selected bigger samples have been prepared for WP2/WP5 and characterised – chemistry, mineralogy, particle size distribution. Results are gathered in D1.1.

This deliverable D1.2. is related to the concentration treatment performed on W-tailings (site 3 in Portugal) as described in D1.1. with details on sampling. This MW was considered under RAWMINA project as the most suitable to test concentration techniques such as gravimetric techniques and flotation. The main objective is to concentrate targeted elements, tungsten (W) in this specific case, to deliver concentrates for further hydrometallurgical steps.

Tungsten mineralization in initial skarn ore was mainly scheelite (CaWO_4) and minor wolframite ($(\text{Fe}, \text{Mn}, \text{Mg})\text{WO}_4$ – mainly as ferberite, Fe-pole - associated to massive sulphides (pyrrhotite, pyrite, arsenopyrite and chalcopyrite). Ore was treated by roasting and flotation.

2. Material and method

2.1. Sample preparation

The sampling procedure undertook at BRGM facilities on the 600 kg sample is given in **Figure 1**. Some additional figures are given on deliverable D1.1.

This MW appears sand grain-sized, with some minor larger and indurated agglomerates. As the received material was still a bit wet, these samples were then dried in oven at 40° C after sample division. Once dried, a comminution path was performed including screening at 20 mm, the oversize material being crushed using a jaw crusher. Finally, a cone crusher was used to further comminute the sample to 1 mm and sent a representative subsample to AGQ laboratories to determine initial chemical composition.

Particle size distribution was determined, and W content was measured by pXRF. The particle size fractions were prepared before separation experiments: +500-1000 μm , +250-500 μm , +100-250 μm , +40-100 μm , <40 μm .

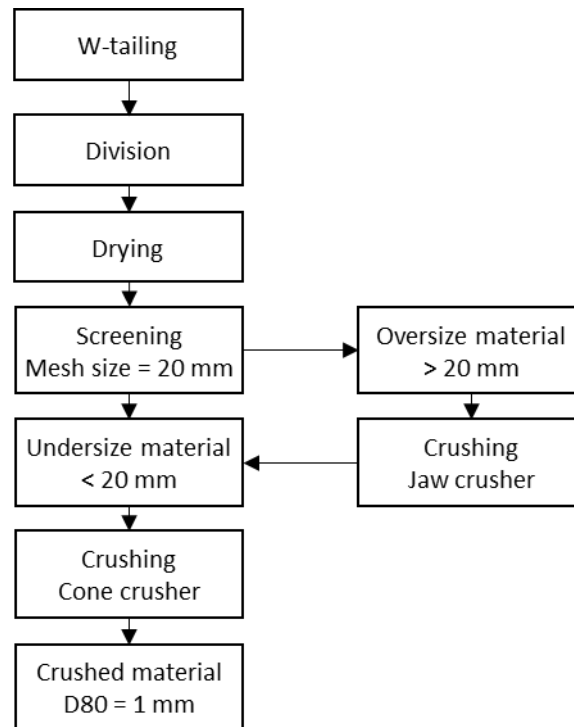


Figure 1. MW preparation procedure at BRGM.

2.2. Mineral processing material

2.2.1. Mozley shaking table

Lab-scale tests were performed with Mozley shaking table, a lab gravity separator, at BRGM.

The Mozley table (**Figure 2**) is a lab-scale shaking table from Mozley (C800 superpanner). The purpose of testing this technique over different size fractions is to evaluate the potential recovery of the elements of interest by gravity separation. Results can be used to justify the need for a grinding at lower d80, but also to choose the appropriate equipment (e.g., shaking table, MGS) required for further upscaling test work.

The table can operate with two different decks, V-shaped for 2 mm to 100 µm samples and the flat version for < 100 µm samples. Samples were around 50-100 g.



Figure 2. Mozley shaking table (@BRGM).

2.2.1. Wilfley wet shaking table and MGS

Pilot density separation was performed using a shaking table at BRGM (**Figure 3**) from Holman-Wilfley (Model 800 - 1280×640 mm = 0.8 m²). The feed was introduced by means of a vibration channel device. The table was equipped with two variable area flowmeters from Georg Fischer (type 355) for wash-water (300–3000 L/h) and feed-water (150–1500 L/h). The five discharge pipes of the shaking table were combined in such manner that three main fractions were collected: concentrates, middlings and light fraction. Tests were conducted on kilograms (5 to 70 kg).



Figure 3. Wilfley shaking table (@BRGM).

2.2.2. Multi-Gravity-Separator - MGS

Some experimental tests were carried out on the finest fraction (<100 μ m) using an MGS-Mozley C900, the smallest experimental device in the MGS-Mozley range (**Figure 4**). It consists of a very slightly conical cylinder, which rotates around a horizontal axis and is animated by sinusoidal axial stroke. The centrifugal force can reach 24G and a scraper system extracts the centrifuged fraction from the bowl. The set is slightly tilted on the horizontal. It is a continuous processing tool that requires at least 50 L of pulp to obtain

an stabilized operation. The operating parameters are: rotational speed, washwater flow rate, feed rate, cylinder axis slope and amplitude and the frequency of shaking. Other parameters are those relating to the feed pulp (solid concentration, particle size, etc.). Only few tests were performed, 10 kg/test.



Figure 4. Multi Gravity Separator – MGS (@BRGM).

2.2.3. Flotation

Two size fractions were prepared at BRGM and sent to AGQ facilities (40-100 μm and $<250 \mu\text{m}$) in order to perform froth flotation. In addition, below this size the slime are important, which increase the viscosity of the system, making the separation process even more difficult.

Flotation is a physicochemical process, whose objective is the separation of mineral species using selective adhesion of air bubbles to mineral particles. Hydrophobic particles adhere to air bubbles while hydrophilic ones remain in the water.

A series of flotation tests were designed at AGQ. Different variables were evaluated such as pH, collectors, or size of the starting material. Reagents are as followed:

- **pH modifiers:** most minerals float most effectively within a certain pH range; alkaline circuits soda ash and sulfuric acid for acid circuit flotation have been tested.
- **Collectors (promoters):** substances that give a hydrophobic behavior to the surface of the material and therefore a good adherence to air bubbles. Tested collectors are:
 - *Aero 704 promoter* is a special tall oil fatty acid with specific acid value and rosin acid content, and percent fatty acid, most widely used for alkaline flotation.
 - *Aero 726 promoter* is a formulated tall oil fatty acids that contain surfactants and other chemical coupling agents that make them much more effective than straight tall oil fatty acids.

- *Aero 801 promoter* is the traditional petroleum sulfonate-based formulation with increased petroleum sulfonate contents.
- *Aero 845 promoter* is ionic, alkyl succinamate based promoter, which was developed to provide more selectivity than can usually be obtained with fatty acids and/or petroleum sulfonates.
- *Oleic acid* is a fatty acid promoter with less specificity than Aero 704 or 726.
- **Dispersants:** many ores contain significant quantities of clay minerals and other “primary slimes”. These can have an adverse effect on flotation performances. The use of a dispersant such as sodium silicate or sodium carbonate will also help to disperse slimes, reduce pulp viscosity, thereby improving recovery and selectivity. Depressants are chemical reagents that are used to prevent the flotation of certain mineral species that would float in their absence.

The tests were carried out in a Denver-type flotation cell (**Figure 5**) in a 2.2 L bucket. Conditions are: 30 % pulp density, 900 rpm and room temperature.



Figure 5. Flotation cell at AGQ.



Figure 6. Example of concentrate recovery at AGQ.

For each experiment, MW was added to the bucket and water was added to reach half the volume. This procedure increases the contact between reagents and MW during conditioning. Then, pH was adjusted, and the reagents added at define quantities. Once the stipulated time had been reached, water was added to the float level and the test start. The foam concentrates were extracted manually using methacrylate paddles to a collection tray as shown on **Figure 6**.

Conditions of test 1 to test 8 are given below and with some details in Appendix 1.

Test 1. An initial test was performed on the W-Tailings F 40-100 μm sample with the usual conditions for the flotation of W species: flotation at alkaline pH in which fatty acids and sulfonic acids are used as collectors. Due to the nature of the sample, it was necessary to add Na_2SiO_3 as dispersant.

Test 2. The previous test is repeated with W-Tailings F 0-250 μm . Due to the high recovery in the first concentrate a second flotation stage was added.



Test 3. The previous test is repeated but, due to the large amount of material recovered in the first concentrate, it was decided to add a second dispersant as a complement: Na_2CO_3 .

Test 4. The previous test is repeated adding quebracho as a depressant to increase the selectivity of the collectors.

Test 5. Test 1 is repeated, replacing the collectors of the first stage with more specific ones.

Test 6. The previous test is repeated but using oleic acid as collector. This presents less specificity than the previous ones but usually presents better recoveries.

Final tests were performed on $F < 250 \mu\text{m}$ withdrawing finest fractions (below $30\text{-}40 \mu\text{m}$) penalizing flotation. Regarding reagents promoting ferberite flotation it has been chosen to float in an acid circuit using Aero 845.

Test 7. On $F < 250 \mu\text{m}$ a first attrition of the sample was carried out at a high pulp density to later eliminate the finer fractions as supernatant.

Test 8. On $F < 250 \mu\text{m}$ the fraction below $63 \mu\text{m}$ was removed by sieving. A first stage of flotation was carried out in an acid circuit and a second in an alkaline circuit.

The fractions obtained were filtered, dried, and subsequently weighed for further study. Chemical content was obtained by ICP-OES after acid digestion with four acids. XRF was used punctually.



2.3. Chemical analyses and mineralogy

2.3.1. Portable XRF (pXRF)

Laboratory and pilot experiments performed at BRGM were monitored by portable X-Ray Fluorescence (pXRF) measurements using a Thermo NITON XL3T 900 with mining mode. This technique was validated at BRGM to measure W in similar dried samples (ore, MW). Measurement time was 30 seconds for each filter. Quantification was performed with calibration implemented in the apparatus. Dedicated preparation allows increasing precision: 3 g of ground ($< 80\mu\text{m}$) sample is manually pressed into a 26 mm cupule; free space remaining is filled with cotton ouate and the pellet cupule covered by a 6 μm Mylar film.

2.3.2. Chemical analysis

As detailed in D1.1 chemical analyses were performed at AGQ, namely by ICP-OES after four acids digestion (HF , H_3ClO_4 , HNO_3 , HCl) of the samples. Uncertainty is 10%.

2.3.1. Mineralogy

X-ray diffraction measurements were performed on ground samples by UAB using a X'Pert Powder de Panalytical apparatus. The main phases were identified using the mineralogy database implemented in the Hi-Score-Plus software by *Panalytical* and in accordance with the International Centre for Diffraction Data (ICDD).

Scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS) were performed at UAB using a Zeiss MERLIN FE-SEM; the as-received material was embedded in a resin and supported on a glass substrate and metallographic polished.

3. Mineral processing objectives

Ores mineralized with scheelite and ferberite are usually treated through gravity separation or flotation, or even a combination of both depending on the scheelite/ferberite size distribution across the ore. In the case of gravity separation, the concentration process relies on the high specific density of scheelite (5.9-6.1) and ferberite (7.1-7.5) compared to that of the gangue material (2.6-2.8).

On the other hand, flotation requires adding promoting reagents, typically carboxylic acid, in particular fatty acid, to recover scheelite when occurring fine grain-sized in the ore, typically under 100 μm . However, the relative brittleness of scheelite (Mohs hardness = 4.5-5.0) put it at its disadvantage when grinding is required, thus comminution shall be carefully employed.

3.1. Elements of interest

3.1.1. Chemical composition

Representative sub-sample of MW has been analysed by AGQ. MW is mainly rich in Si (estimated around 70 %) and contains Al (7.9 %), Fe (8.1 %), Ca (8.1 %), K (1.7 %), Na (1.7 %). It contains sulphates and sulphides: equal proportion expressed as S wt% around 2.2-2.3 wt%.

Other targeted CRMs than tungsten were quantified: 6 ppm Co, 7 ppm Sb, Ge being below detection limits (<10). Precious metals analyses give 0.2 ppm Au and 6 ppm Ag. Arsenic is also present: 1816 ppm.

W content measured by pXRF in a quantitative manner (in cup – see methods) is 4491 ppm; this same technique has been used to follow all gravimetric experiments.

3.1.2. Mineralogy

The main mineral phases were evaluated by XRD and SEM-EDS at UAB on the concentrated sample. Al-silicates, quartz, ferberite (wolframite FeWO_3), scheelite (CaWO_3), Ca-phosphates, pyrite and arsenopyrite were the main identified minerals.

Other results from a previous EU project (ENVIREE) on a raw sample from the same site identified different minerals listed hereafter (proportions evaluated at that time are indicative): quartz (18 %); silicates such as muscovite (25 %), kaolinite (12 %), clinocllore (11 %), feldspar and albite; hematite (1.6 %); ferberite (0.6 %) and scheelite (0.15 %); apatite (0.8 %); sulphides such as arsenopyrite (5.4 %) and chalcopyrite (0.5 %); and also calcite (14 %). **Table 1** gathers density data of these minerals.

Table 1. Density of selected minerals.

Mineral	Density	Mineral	Density
Quartz	2.6	Hematite	5.2
Feldspar	2.6	Ferberite	7.5
Muscovite	2.9	Scheelite	6.1
Kaolinite	2.5	Chalcopyrite	4.2
Clinocllore	2.7	Arsenopyrite	6.1
Albite	2.6	Pyrite	5.0
Calcite	2.7	Apatite	3.2

The presence of heavy minerals (hematite, chalcopyrite, ferberite, scheelite and arsenopyrite) suggests that a gravimetric separation would allow recovering both iron bearing mineral and W bearing ones.

3.1.3. UV observation

Scheelite shows a bright white-blue fluorescence when placed under a particular UV wavelength ($\lambda = 254$ nm, so called 'UV short wave'). **Figure 7** displays a picture of MW sample under such UV shortwave radiation where one can easily see white-blue bright spots across the material. These white-blue spots occur over a large spectrum of grain sizes, up to near millimetric.

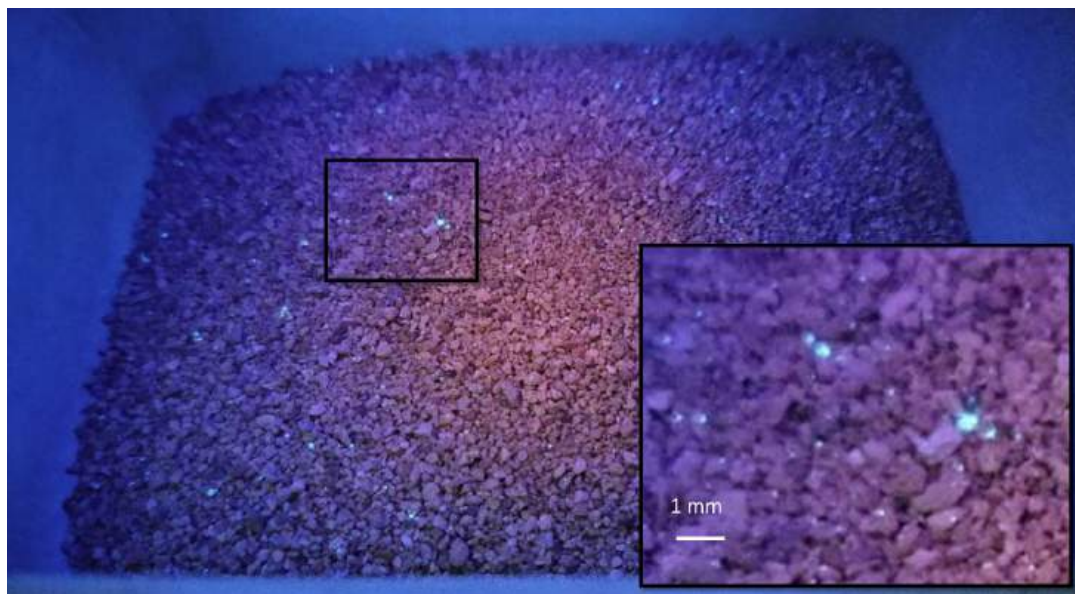


Figure 7. W-tailings after crushing under UV shortwave radiation, blue spots being scheelite mineral having bright fluorescence.

The mineralogical characterization showed the presence of scheelite and calcite. Calcite can also be visible in blue under a UV lamp (short wave). A “gold pan” was used to separate the heavy particles from the light particles (scheelite density 6.1 and calcite density 2.7), the fluorescence was visible only on the heavy particles. White-blues spots in studied sample matches the scheelite fluorescence.

3.1.4. Particle size distribution and composition

Wet sieving has been carried out on the product after crushing steps and relative proportions calculated (**Table 2.**). Chemical content has been determined in all size fractions by pXRF after sample preparation and global content calculated.

W appeared distributed in all size fractions but is higher in <40 µm which can be link to the brittle characteristic of scheelite. Regarding W distribution no particle size fraction can be really excluded from the treatment. Other elements show some variability: namely Cu and Pb being concentrated in the finest fraction (< 40 µm); Fe in coarser fractions.

Table 2. Particle size distribution of crushed MW; Fe, Al, Cu, Pb, and W contents (pXRF).

Particle size	Distribution	Fe		Al		Cu		Pb		W	
µm	% mass	% mass	Distrib. %	% mass	Distrib. %	ppm	Distrib. %	ppm	Distrib. %	ppm	Distrib. %
>1000	5.7	6.1	4.5	2.8	6.2	381	2.4	17	0.8	6612	8.4
500-1000	24.5	5.7	18.1	2.4	22.7	482	12.9	59	11.9	5361	29.3
250-500	24.9	5.9	19.1	2.7	26.0	562	15.3	52	10.6	3614	20.0
100-250	19.4	6.6	16.6	2.7	20.2	745	15.8	60	9.5	1927	8.3
40-100	13.8	7.7	13.8	2.9	15.5	1494	22.5	103	11.7	3984	12.2
< 40	11.7	18.3	27.8	2.1	9.5	2452	31.3	579	55.5	8343	21.7
Calculated	100	7.7	100	2.6	100	917	100	122	100	4490	100

3.2. Separation strategy

Three particular conclusions are to be drawn from the sample composition:

- All tungsten is associated to heavy W-minerals: scheelite and ferberite,
- Scheelite occurs in coarse (observed at 500 µm) and fine grain size fractions and,
- Tungsten is present in all size fractions.

This information fit to a beneficiation path involving gravity separation and froth flotation.

4. Results

4.1. Gravimetric separation

To test the potential of gravity separation processes, a series of tests were undertaken using:

- Mozley C800 table for the evaluation of the potential recovery of W by gravity separation.

Once having validated W concentration, a high amount of concentrate has been produced using:

- Shaking table on size fraction $>100\ \mu\text{m}$,
- MGS on size fraction $<100\ \mu\text{m}$.

4.1.1. Mozley shaking table

Gravity separation was firstly tested on a 100 g sample using a lab-scale Mozley shaking table on different size fractions: $+500\text{-}1000\ \mu\text{m}$, $+250\text{-}500\ \mu\text{m}$, $+100\text{-}250\ \mu\text{m}$ and $+40\text{-}100\ \mu\text{m}$ (**Figure 8**). Particles above 1 mm may hardly be amenable to gravity separation using a Mozley shaking table. Additionally, following the size reduction procedure, this size fraction represents only 5.7 % of the material and shall ultimately be considered directly for further regrinding below 1 mm.

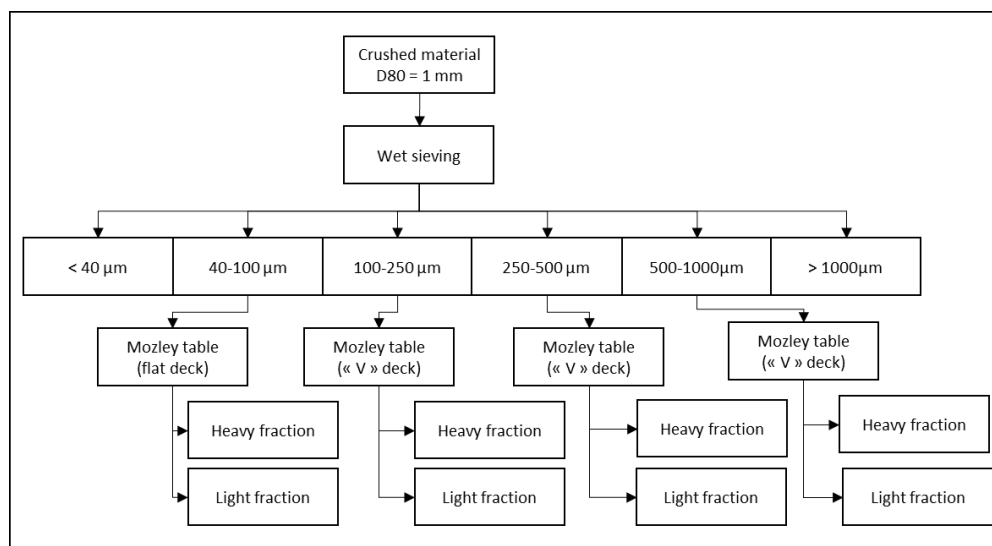


Figure 8. Test-work procedure for testing gravity separation using a lab-scale Mozley table.

The subsequent heavy and light fractions produced from each size fraction have been dried, weighted, and analysed by pXRF to quantify W content. UV shortwave radiation was used to help visualize scheelite occurrence over the material on the tray of the shaking table – see **Figure 9**.

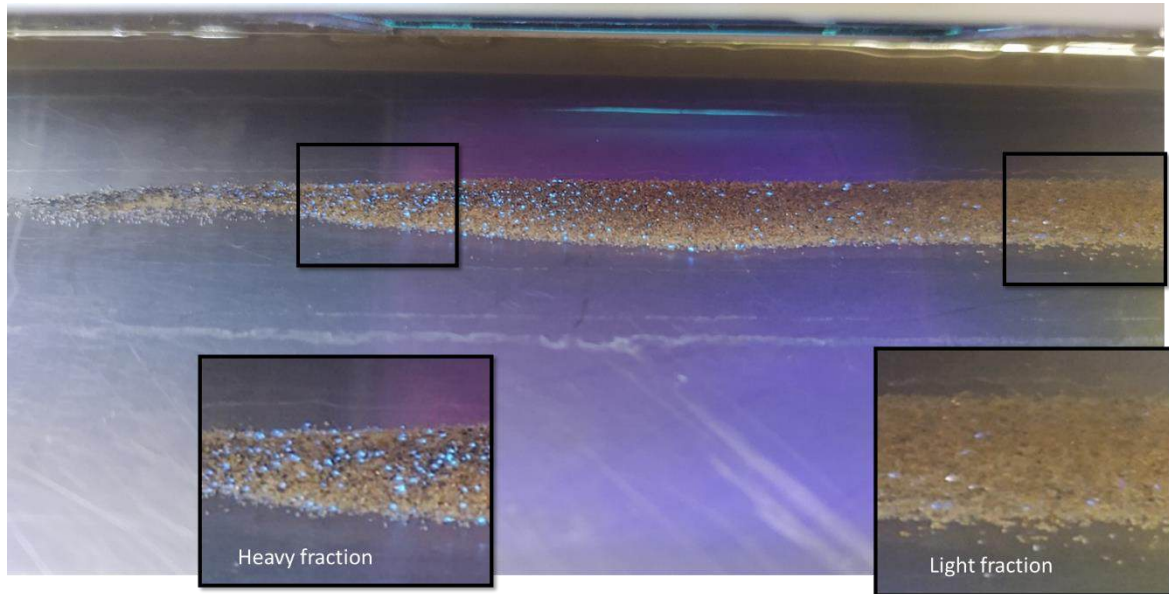


Figure 9. Observation under UV shortwave of heavy and light fractions from 250-500 µm fraction recovered by Mozley shaking table (blue bright spots correspond to scheelite minerals).

On the other hand, particles below 40 µm are not yet amenable to conventional gravity separation and would require a desliming step around 10 µm, regardless using flotation or gravity separation to recover scheelite in this size fraction.

Chemical composition of size fractions output materials provided by pXRF is shown on **Table 3**. Calculated total content of W is 4491 ppm which is the initial content value that is used to evaluate concentration work using the same analytical technique.

Table 3. Tungsten content on heavy and light fractions recovered by Mozley table.

Fractions	Raw MW		Concentration test (Mozley table)					
	Mass distribution (wt%)	Content W (%)	Distribution (wt%)		Content W (ppm)		W recovery rate (%) / fraction	
			"Heavy"	"Light"	"Heavy"	"Light"	"Heavy"	"Light"
+1 mm	5.7	6612	N/A	N/A	N/A	N/A	N/A	N/A
+500-1000 µm	24.5	5361	6.3%	93.7%	46897	3664	46.1%	53.9%
+250-500 µm	24.9	3614	11.4%	88.6%	12852	2291	41.9%	58.1%
+100-250 µm	19.4	1927	20.4%	79.6%	6118	1048	60.0%	40%
+40-100 µm	13.8	3984	20.9%	79.1%	17698	787	85.6%	14.4%
< 40 µm	11.7	8343	N/A	N/A	N/A	N/A	N/A	N/A
calculated	100	4491						

Recovery performances with the Mozley table remains acceptable (46.1 to 85.6 %) and should be improved with the shaking table and the MGS. These performances show that there is an interest in performing gravimetric separation on the material ground to 1 mm.

4.1.2. Wilfley wet shaking table

Operating tests

Tests on the Wilfley wet shaking table were performed on a continuous feeding mode with vibrating feeder SINEX on different particle size fractions: 100-250 μm , 250-500 μm and 500-1000 μm (**Figure 10**).

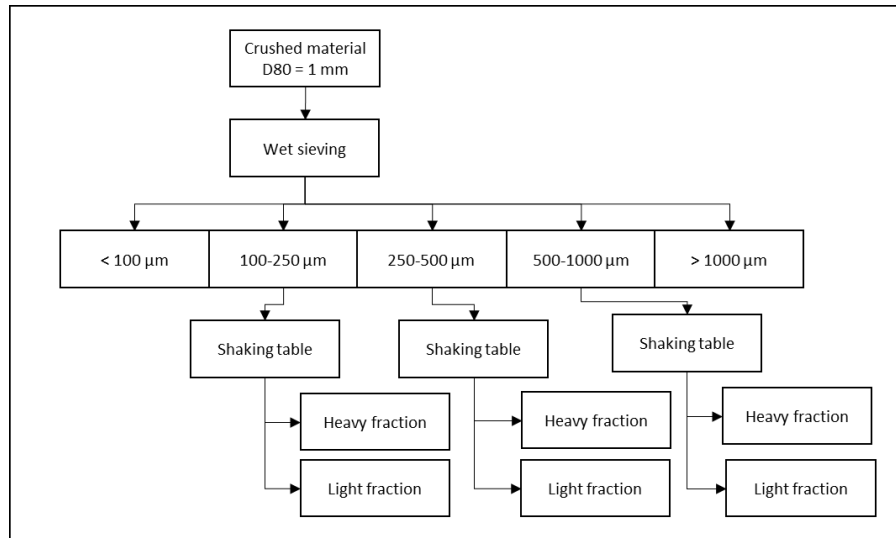


Figure 10. Test-work procedure for testing gravity separation with Wilfley shaking table.

These trials were tested under various operating conditions to assess their performance (**Table 4**).

Table 4. Shaking table operating conditions – operating tests.

	Operating condition	Solid input	Water input	Wash water	Stroke Length	Stroke Frequency	Adjustable Slope	Adjustable Tilt	Deck type
N° test	Particle size	kg/h	l/h	l/h	mm	rpm	°	°	
1			0		9	0	1	0	Sand
6	500-1000 μm	25	150	400	9	229	1	5	Sand
7		25	190	450	11	229	1	5	Sand
8		25	70	450	9	229	1	5	Sand
16		25	150	450	9	229	0.68	5	Sand
20		25	70	450	9	229	-	5	Slime
2		0	0	0	9	0	1	0	Sand
9	250-500 μm	25	75	450	9	267	1	5	Sand
10		25	75	300	9	267	1	5	Sand
11		25	70	450	9	247	1	5	Sand
17		25	70	450	9	267	0.68	5	Sand
3		0	0	0	9	0	1	0	Sand
12	100-250 μm	25	70	450	9	70	1	5	Sand
13		25	70	450	7	267	1	5	Sand
14		25	70	450	7	229	1	5	Sand
15		25	70	450	9	229	1	5	Sand
19		25	70	450	9	292	0,68	5	Sand
21		25	70	450	7	267	unknown	0,82	Slime

Table 5. Tungsten content on heavy and light fractions recovered by shaking table - operating tests

N° test	Particle size	Mass distribution (wt%)			W content (ppm)			W content (ppm)	W recovery rate (%) *
		Heavy	Middling	Light	Heavy (calculated)	Middling	Light		
1	500-1000 µm	13	44	44	28653	2596	1435	5361	67
6		7	27	66	68196	684	394	5361	92
7		14	55	31	35191	613	409	5361	91
8		5	19	76	88878	721	502	5361	90
16		7	27	66	71116	1166	323	5361	90
20		5	37	58	98741	503	587	5361	91
2	250-500 µm	14	52	34	18363	1294	1033	3614	72
9		11	35	54	27815	799	581	3614	84
10		10	34	56	31417	464	462	3614	88
11		9	32	59	36070	601	521	3614	86
17		24	70	7	13682	483	797	3614	89
3	100-250 µm	19	67	14	6770	751	815	1927	68
12		9	26	65	16838	436	440	1927	79
13		8	23	69	19466	519	404	1927	79
14		1	19	80	115925	1531	456	1927	66
15		1	33	66	105247	1177	473	1927	64
19		13	45	42	10651	676	510	1927	73
21		8	18	74	17366	654	516	1927	74

* in Heavy fraction

Regarding the 500-1000 µm fraction, the best test is n°20 with W recovery rate in the heavy fraction of 91%, contained in 5 % of total mass with W content of 9.8 %, which is a very interesting result also considering the one treatment step. Regarding the 250-500 µm fraction, the separation is a bit less efficient but still satisfactory: recovery rate reaches 86-88 % for a mass around 10 % and final W content of 3.1-3.6 %.

For the finest fraction (100-250 µm), it was difficult to achieve W content above 2 %. Only tests n°14 and n°15 showed significant increase of W content in heavy fraction (around 10 %) but the recovery rate dropped to 64-66 %, and the mass of heavy fraction represented only 1 %.

Production tests

A production test was carried at BRGM out to produce a concentrate with the highest W content as possible for further experiments under WP1 and WP2. The operating conditions (**Table 6**) were chosen according to the previous results. 8 kg of concentrate was produced corresponding to the mixture of the heavy coarser fractions (250-500 µm and 500-1000 µm). Quantitative pXRF measurements of W were performed in triplicate on heavy, middling, and light fractions (cups). Results are given in **Table 7** and **Table 8**.

Table 6. Shaking table operating conditions – production tests.

Particle size	Solid input	Water input	Washwater	Stroke Length	Stroke Frequency	Adjustable Slope	Adjustable Tilt	Deck
	kg/h	l/h	l/h	mm	rpm	°	°	



500- 1000 µm	25	70	450	11	229	0.57	5	Sand
250-500 µm	25	70	450	9	247	0.2	4.2	Sand

Table 7. Tungsten content of heavy, middling and light fractions recovered by shaking table - production tests.

Particle size	Mass distribution (wt%)			W content (ppm)			W content calculated (ppm)	W recovery rate (%)
	Heavy	Middling	Light	Heavy	Middling	Light		Heavy
500- 1000 µm	8	42	49	46091	3989	1644	6275	60
250-500 µm	10	29	61	30414	1649	1255	4193	70

Table 8. W, As, Fe calculated content (pXRF) of final concentrate - production tests.

Particle size	Masse distribution (wt%)		W content (ppm)		As content(ppm)		Fe content (%)	
	Heavy	Middling + Light	Heavy	Middling + Light	Heavy	Middling + Light	Heavy	Middling + Light
250-1000µm	9	91	35936	1913	3714	813	15	5

4.1.3. Characterisation of the concentrate (production test, sample CC1)

Chemical composition

Chemical composition of the concentrate was determined by ICP-OES (AGQ) and final W concentration validated: 3.52 wt% (3.59 % measured by pXRF) W. Some other elements are also concentrated in comparison to initial MW such as Fe (from 8.1 to 14.7 %), sulphides (2.2 to 13.9 %), Mn (0.9 to 1.6 %), As (1816 to 3746 ppm), Cu (1011 to 1570 ppm). Other targeted CRM are low (39 ppm Co, 15 ppm Sb) or below detection limit (<10 ppm).

Mineralogy (XRD and SEM-EDS)

Main crystalline phases in the concentrate were determined by XRD at UAB. Al-silicates accounting for around 62 %; there was not a unique selection of aluminium silicates providing a reliable matching with the experimental diffraction pattern, however, albite ($\text{NaAlSi}_3\text{O}_8$) and different biotites ($\text{K}(\text{Mg,Fe})_3\text{AlSi}_3\text{O}_{10}(\text{OH})_2$) were the most recurrent matches among aluminium silicates.

The sample also contains quartz (15 %) and heavy minerals that have been concentrated: W-bearing minerals ferberite (5 %) and scheelite (2 %), and also pyrite (16 %). Considering the two W-bearing minerals, the amount of W in sample agrees well with the 3.5 wt% of W determined by pXRF measurements at BRGM and by benchtop XRF at AGQ.

Back-scattering electrons SEM images allow evaluating mineral liberation and distinguishing different mineral phases by chemical contrast namely those already identified by XRD.

Particle size distribution is confirmed to be below 100 μm and SEM-images showed that most of the particles were composed by single mineral (**Figure 11**). Ferberite is confirmed to be the main W mineral, their particles being smaller than pyrite particles. Scheelite was also identified. The main Fe-bearing mineral present is pyrite, although there are several smaller grains of arsenopyrite. Quartz appears as two types: large grains and very fine powder whereas the grain size of the aluminium silicate phases is more homogeneous. Biotite is confirmed to be the main aluminium silicate; its composition is variable, namely Mn (enriched content in the concentrate) being associated to Fe-rich biotite. Also, large grains of calcium phosphate were detected.

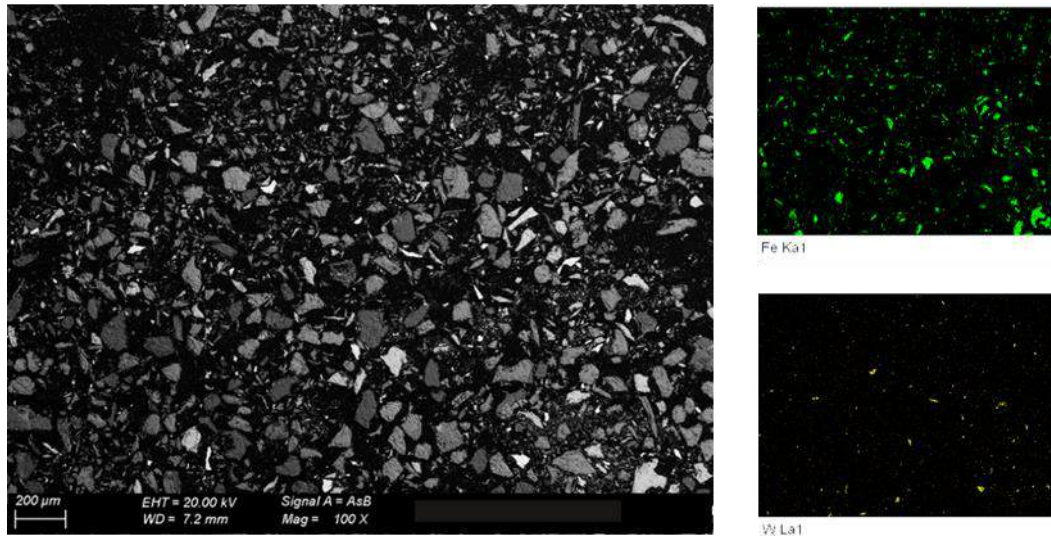


Figure 11. SEM image of MW concentrate (CC1) - @UAB

Some details and SEM elemental mapping images are given in **Appendix 2 Mineralogy study of mining wastes: XRD and SEM-EDS (UAB report)**.

4.1.4. MGS (fine fraction)

In addition, two tests were carried out at BRGM with MGS equipment on <100 µm fraction, without desliming since fines are considered to be eliminated within light fraction. Only one test showed reliable results. Conditions are gathered in **Table 9**.

Table 9. MGS operating conditions – operating test.

Particle size	Pulp input	% solid	Washwater	Shake amplitude	Shake Frequency	Adjustable Slope	Rotational speed
	L/h	%	L/h	mm	cps	°	rpm
< 100 µm	90	17	150	19	5.7	3.6	150

The best result even if not optimised is detailed in **Table 10**: 21 wt% of total mass concentrate W at 1.4% in heavy fraction. This result shows some potential to recover W in fine fractions which gives perspectives to further test MGS technology.

Table 10. Tungsten content recovered by MGS (three fractions) - operating test.

Particle size	Masse distribution (%mass)			W content (ppm)			W content calculated	W recovery (%)
	Heavy	Middling	Light	Heavy	Middling	Light		Heavy
< 100 µm	21	13	66	13870	7564	2503	5526	53

4.2. Froth flotation

4.2.1. Particle size distribution (0-250 μm)

Particle size distribution of the samples received from BRGM was performed by AGQ and all size fractions obtained were analyzed for determine the W content. **Table 11** gathers main results.

Table 11. W content and mass distribution (0-250 μm fraction).

Fraction (μm)	Mass (g)	Mass distribution (%)	W Content (%)	W Content (g)	W distribution (%)
250	17.85	5.04	0.634	11.32	6.89
125	87.05	24.58	0.139	12.12	7.38
75	95.6	26.99	0.187	17.85	10.87
63	33.2	9.37	0.438	14.53	8.85
<63	120.45	34.01	0.900	108.37	66.00
Σ	354.15	100	0.464	164.19	100

The results shows that the largest proportion of W remains in the finest fraction (66 % in the <63 μm fraction), which is also the one with the bigger mass (34 %). This could be a problem to obtain high recovery rate because the fines are hardly floatable due to the high consumption of flotation reagents, the rheological behavior of the foams and the specificity that decreases with the size of the particle.

4.2.2. List of experiments

Test 1 to Test 6 allow testing flotation with usual reagents for the flotation of W species at alkaline pH. These tests were carried out with the two size fractions provided: 40-100 μm and 0-250 μm .

Tests 7 and 8 were performed on 0-250 μm sample after a first step of desliming - particle size is estimated around 30-40 μm - to eliminate smallest particles that could interfere with flotation process by reducing the selectivity of the collectors. In addition, work was carried out at acid pH with the aim of increasing the recovery of ferberite.

Table 12 – Flotation reagents and concentration range (8 tests)

Test	W-Tailings raw 40-100 µm	W-Tailings raw 0-250 µm	pH	Aero 726 (g/t)	Aero 801 (g/t)	Aero 704 (g/t)	Aero 845 (g/t)	Oleic Acid (g/t)	Na ₂ SiO ₃ (g/t)	Na ₂ CO ₃ (g/t)	NaF (g/t)	Quebracho (g/t)
1	•		10	400	100				1000			
2		•	10	400+400	100				1000			
3		•	10	400+400	100				1000	1000		
4		•	10	400+400	100				1000	1000		50
5	•		10	400		400	100		1000			
6	•		10	400				200	1000			
7		•	2,5				200		1000		500	
8		•	2,5 + 10				250		1000		500	

Test	Na ₂ SiO ₃ (g/t)	Na ₂ CO ₃ (g/t)	NaF (g/t)	Quebracho (g/t)
1	1000			
2	1000			
3	1000	1000		
4	1000	1000		50
5	1000			
6	1000			
7	1000		500	
8	1000		500	

4.2.3. Tungsten recovery

The results of the tests are summarized in **Table 13**. W selectivity is low which can be related to the high level of slime; since tests 5 and 6 show some better recovery. Fine particles are hardly depressed and increase the doses of collector to be added which increases the dilution of the minerals. In addition, ferberite is known to be more difficult to float than scheelite

Table 13. W recovery by froth flotation results.

Test	Mass recovery (%)	W recovery (%)	W in concentrate (%)
1	3.0	3.0	0.51
2	51.9	53.4	0.59
3	73.8	78.1	0.59
4	67.7	73.9	0.61
5	9.5	26.8	1.37
6	16.9	29.7	0.92
7	7.6	4.6	0.62
8	2.1	1.7	0.21

5. Flowsheets of W-tailings treatment

The flowsheets were extrapolated from experimental results, as follow:

For > 100 μm fraction:

- Optimised shaking table tests (test 20 for 500-1000 μm fraction, test 9 for 250-500 μm fraction and test 14 for 100-250 μm fraction – see section 4.1.2)

For < 100 μm fraction:

- MGS test (lower W content than expected but optimisation is needed – see section 4.1.4)
- Flotation tests (test 5 – see section 4.2).

For gravimetric separation (shaking table and MGS), the middling and light fraction have been gathered. The metal contents of heavy fraction were calculated on the basis of mass balance presented in **Figure 12** and **Figure 13**. W can be concentrated by gravimetric techniques from global sample up to 3.4 % and by flotation and gravimetric techniques process up to 4.8 %.

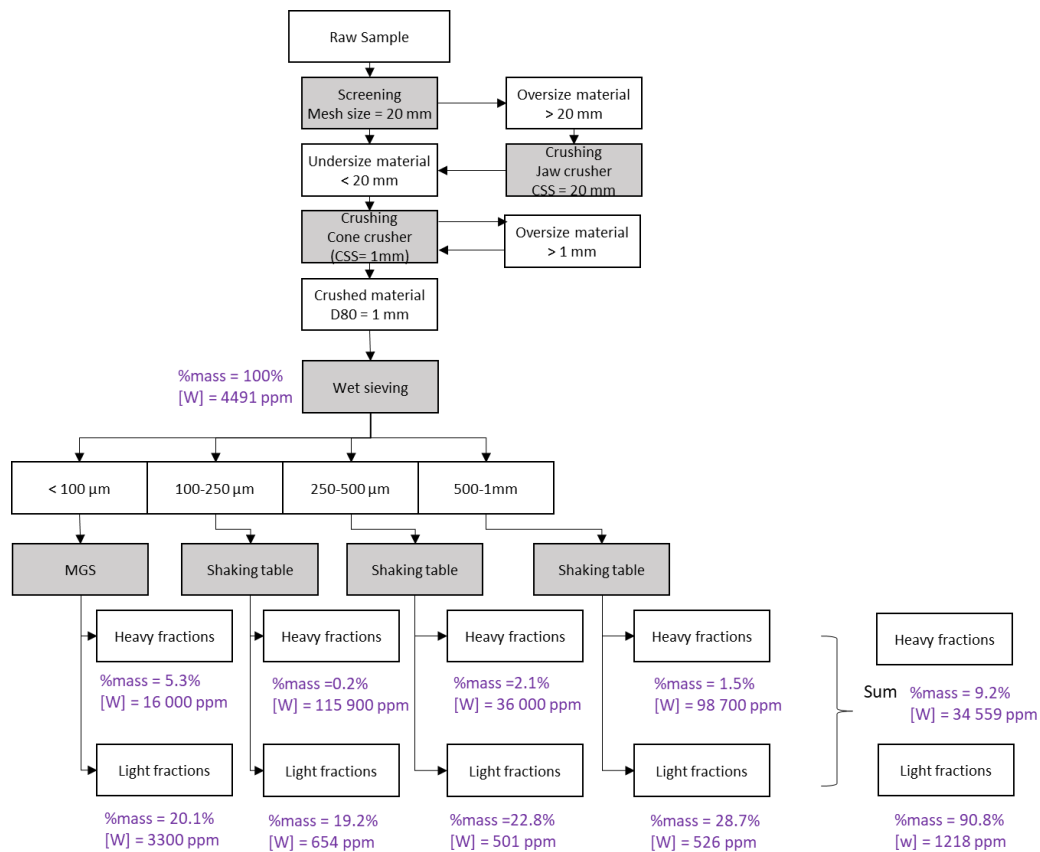


Figure 12. Flowsheet and mass balance of gravimetric concentration process.

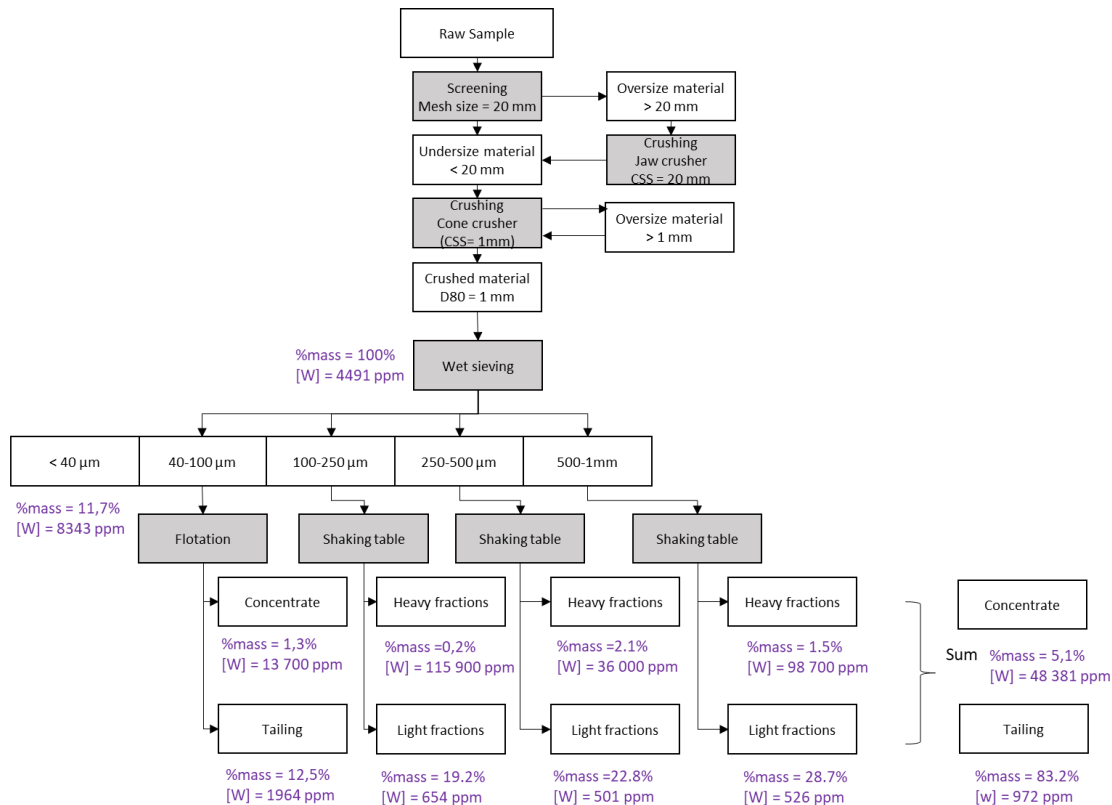


Figure 13. Flowsheet and mass balance of flotation and gravimetric concentration process.

6. Conclusions

Gravimetric techniques (shaking table, MGS) and froth flotation have been evaluated on W-tailings after dedicated preparation of representative subsamples and classification in several size fractions. Lab test results show higher W content with gravimetric concentration on the coarser fractions which lead to consider this latter to produce enough concentrate for WP2. 8 kg of MW concentrate 'CC1' have been produced by gravimetric separation techniques to be further used on the RAWMINA project. The concentrate is rich in heavy minerals, namely ferberite, scheelite and also sulphides. W content is 3.6 %.

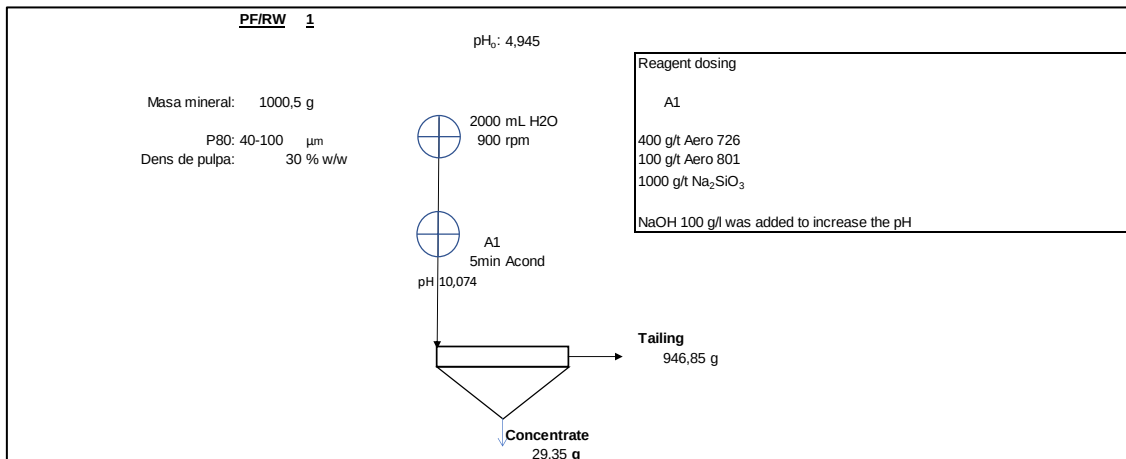
Flotation tests with and without deslamming have been also performed on the finest fractions.

Finally, two flowsheets have been calculated for W recovery: one related to gravimetric concentration processes (shaking table and MGS) and another one related to froth flotation and gravimetric process by shaking table.

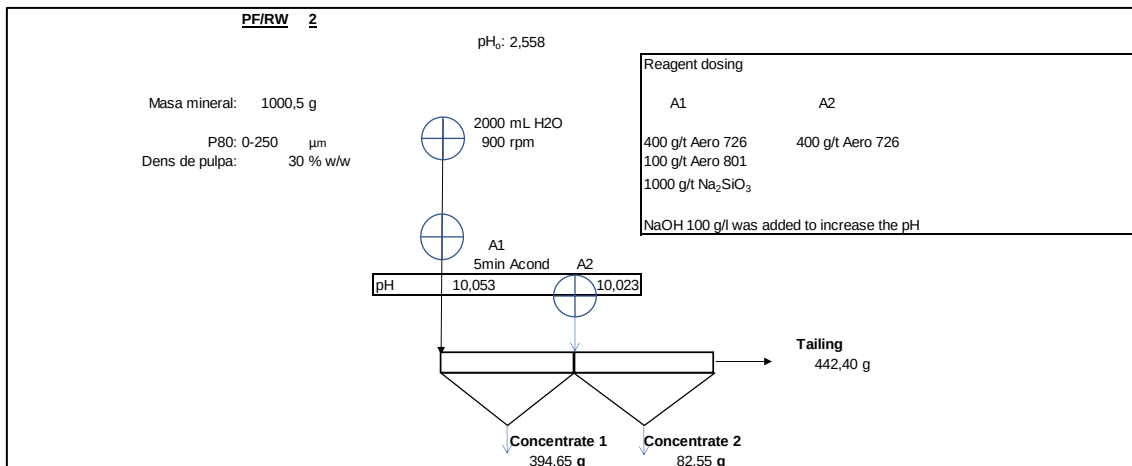
Appendix 1 – Froth flotation tests (AGQ)

Details of experimental conditions are given below.

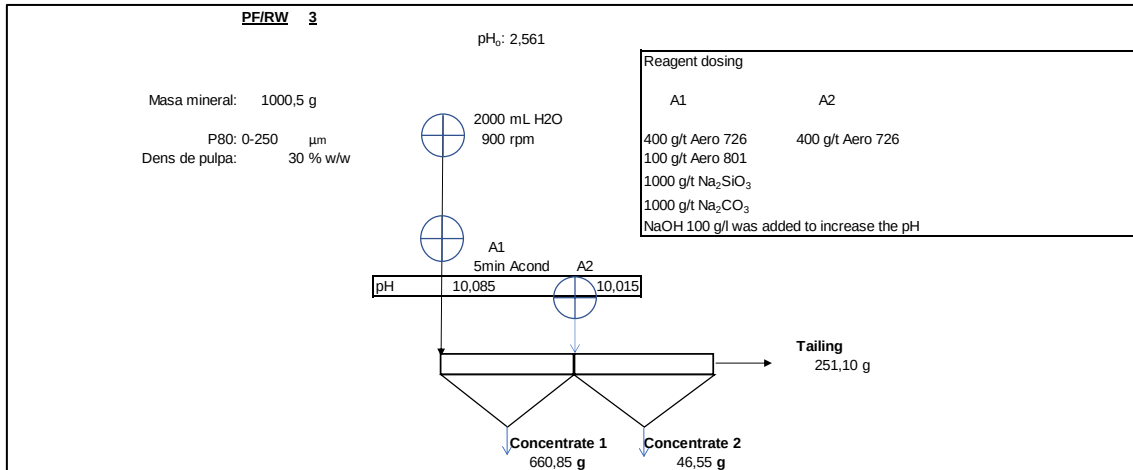
Test 1. An initial test is performed on the W-Tailings F 40-100 μm sample with the usual conditions for the flotation of W species. In this case, it is a flotation test at alkaline pH in which fatty acids and sulfonic acids are used as collectors. Due to the nature of the sample, it was necessary to add Na_2SiO_3 as dispersant.



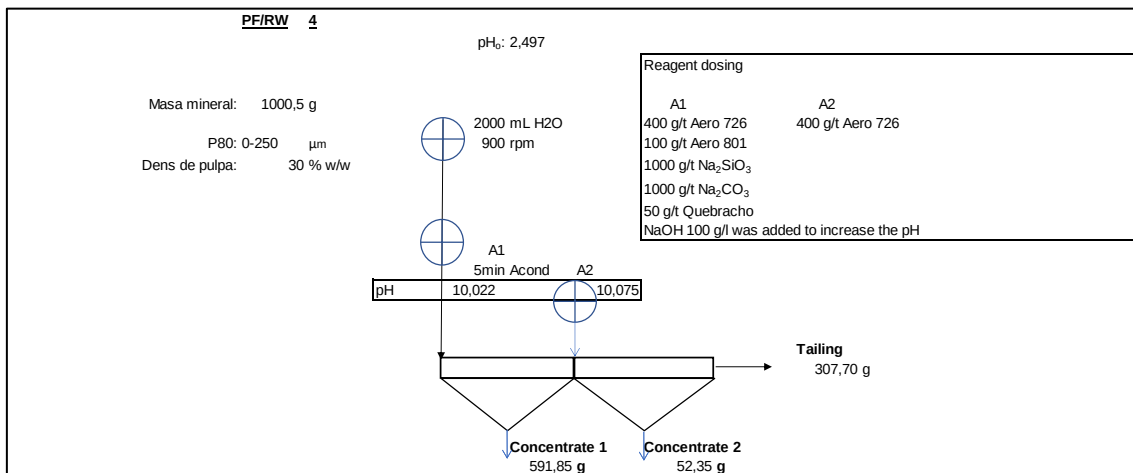
Test 2. The previous test is repeated but changing the sample for the W-Tailings F 0-250 μm . Due to the high recovery in the first concentrate, we decided to add a second flotation stage.



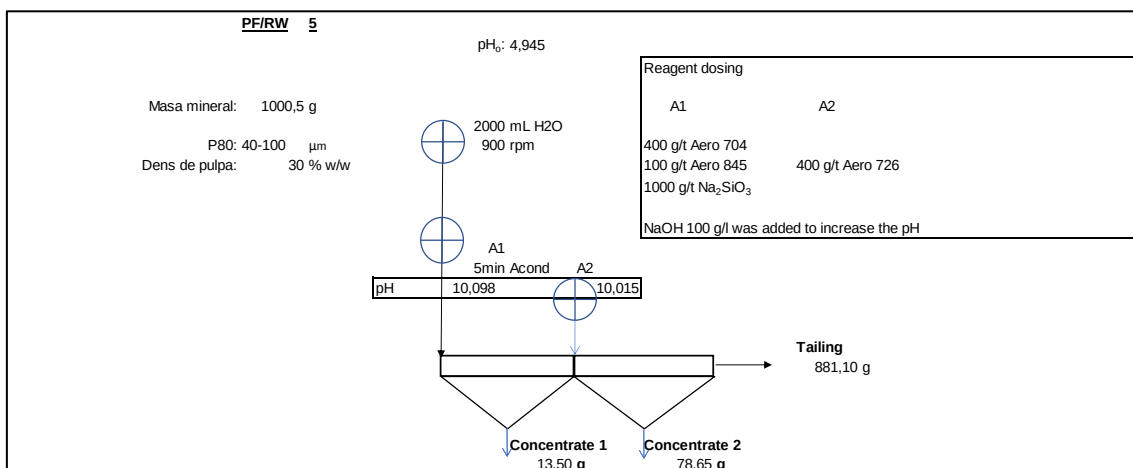
Test 3. The previous test is repeated but, due to the large amount of material recovered in the first concentrate, it was decided to add a second dispersant as a complement, in this case we add Na_2CO_3 .



Test 4. The previous test is repeated adding quebracho as a depressant to increase the selectivity of the collectors.



Test 5. Test 1 is repeated, replacing the collectors of the first stage with more specific ones.



Test 6. The previous test is repeated but using oleic acid as collector. This presents less specificity than the previous ones but usually presents better recoveries.

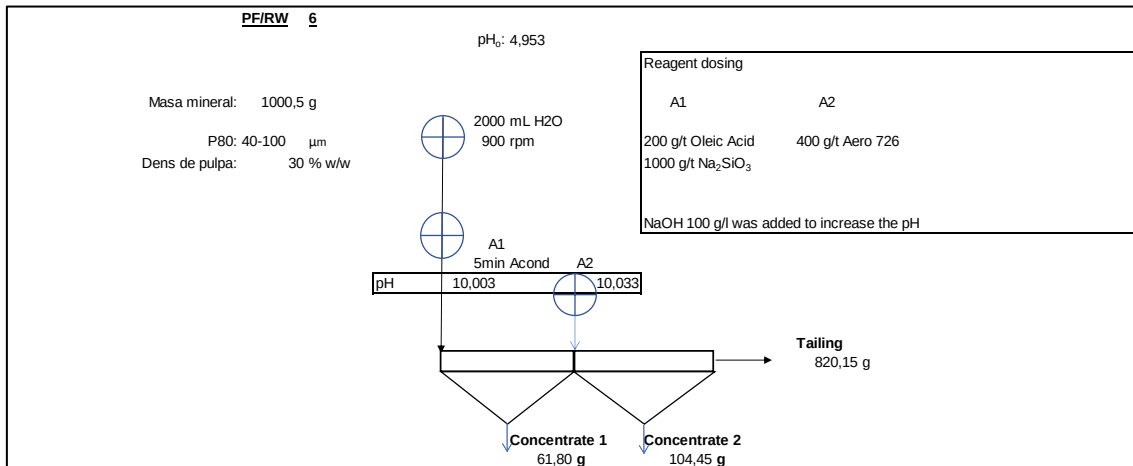
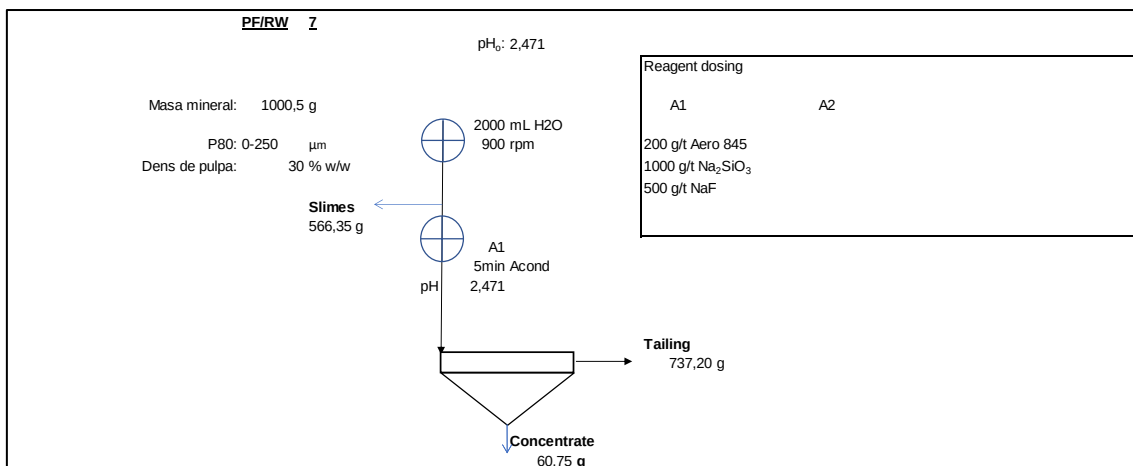


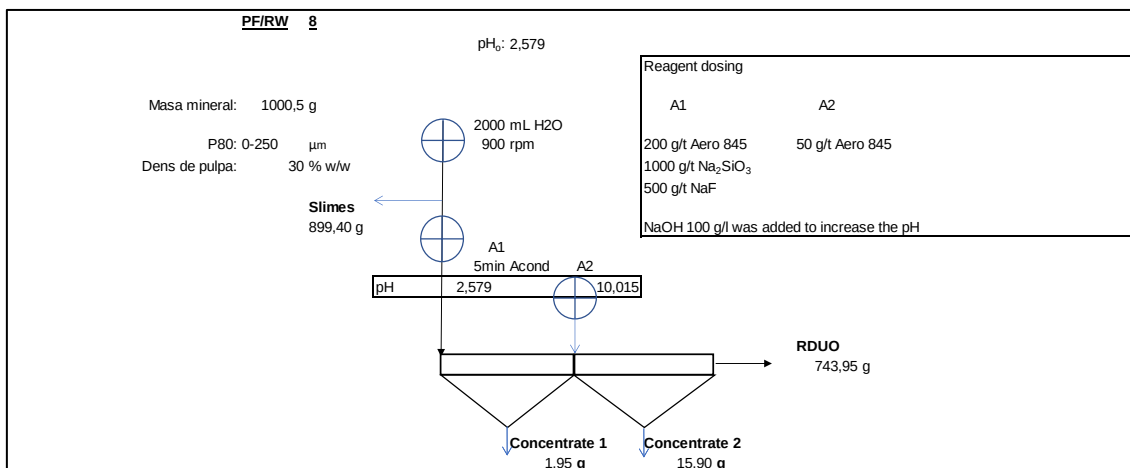
Figure 9. Froth flotation: test 6

From this point, no more sample F 40-100 μm was available, so the rest of the tests were carried out with the sample F <250 μm. The tests carried out with said sample presented problems due to the finer fractions, which in these cases were withdrawn. Regarding the reagents, to favor the flotation of ferberite, it has been chosen to float in an acid circuit using Aero 845.

Test 7. In this case, a previous attrition of the sample was carried out at a high pulp density to later eliminate the finer fractions as supernatant.



Test 8. In this test, the fraction below 63 was removed by sieving. A first stage of flotation was carried out in an acid circuit and a second in an alkaline circuit.





Appendix 2 – Mineralogy study of mining wastes: XRD and SEM-EDS (UAB report)



RAWMINA Project

Deliverable 1.2 - Appendix 2

Mineralogy study of mining wastes: XRD and SEM-EDS

Date: 19/05/2022

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7. Introduction

The characterization of the main mineral phases comprising the mine wastes of W-tailings2 (Portugal) concentrate produced at BRGM on the coarser fractions was performed via X-ray powder diffraction (XRD) and scanning electron microscope energy dispersive spectroscopy (SEM-EDS) analysis at the Universitat Autònoma de Barcelona.

Three concentrates with different grain size were received from BRGM:

- small grain size (250-500 μm) concentrate: “W-tailings2_S”
- large grain size (500-1000 μm) concentrate: “W-tailings2_L”
- ground mixed concentrate: (<100 μm): “W-tailings2_CC1”

Hereafter, for the shake of simplicity, the samples will be denoted as “S”, “L” and “CC1”, respectively.

The former two samples, “S” and “L” are the heavy fractions obtained from the shaking table after having processed the sample as described in previous sections of this deliverable. The latter sample, “CC1”, is the mixture of the two other samples at proportions around 61/39 (pilot operation) that was ground to have a more appropriate size for the bioleaching and alkaline stages.

For the X-ray diffraction measurements (X'Pert Powder de Panalytical), samples “S” and “L” were ground prior the measurements using a manual agate mortar. The main phases were identified using the mineralogy database implemented in the Hi-Score-Plus software by Panalytical. The codes referred in the tables are according to the International Centre for Diffraction Data (ICDD)⁴.

For the SEM-EDS measurements (Zeiss MERLIN FE-SEM), the as-received material was embedded in a resin and supported on a glass substrate. A thin section of the mixture (ca. 100 μm thick) supported on the glass substrate was obtained and metallographic polished. To avoid the building up of the charge on the surface of the sample, the surface of the sample was carbon-coated, and carbon tape was used at the base and on the sides of the sample to connect the top of the sample with the sample holder.

⁴ International Centre for Diffraction Data (ICDD) <https://www.icdd.com/>



8. Results

- **W-tailings2 concentrate**

→X-ray diffraction measurements

The results from the XRD analysis are shown in **Table 1**.

Table 14. Mineral phases of the W-tailings2 concentrates mine residues determined by database matching.

	Pyrite	Quartz	Ferberite	Scheelite	Aluminium silicates
250-500 µm ("S")	18%	18%	8%	1%	55%
500-1000 µm ("L")	21%	10%	5%	2%	62%
<100 µm (ground mixture, "CC1")	16%	15%	5%	2%	62%

Pyrite (FeS_2) [Ref. ICDD 01-071-2219]

Quartz (SiO_2) [Ref. ICDD 01-078-1252]

Ferberite (FeWO_4) [Ref. ICDD 01-071-2391]

Scheelite (CaWO_4) [Ref. ICDD 01-072-1624]

The mineral samples are mainly composed by aluminium silicates (>50 %). Due to the complexity of the diffraction pattern (see **Figure 1**), there was not a unique selection of aluminium silicates providing a reliable matching with the experimental diffraction pattern, however, Albite ($\text{NaAlSi}_3\text{O}_8$) and different biotites ($\text{K}(\text{Mg},\text{Fe})_3\text{AlSi}_3\text{O}_{10}(\text{OH})_2$) were the most recurrent matches among aluminum silicates. Regarding the mineral of interest, a relatively high amount of Pyrite (>15 %) was found in the samples. Regarding the CRM of interest, the main mineral phase containing W is Ferberite (~5 %), but there is a small amount of Scheelite (~2 %). Considering these two W-bearing minerals, the amount of W in sample agrees well with the 3.8 wt.% of W determined by pXRF measurements at BRGM and by benchtop pXRF at AGQ.

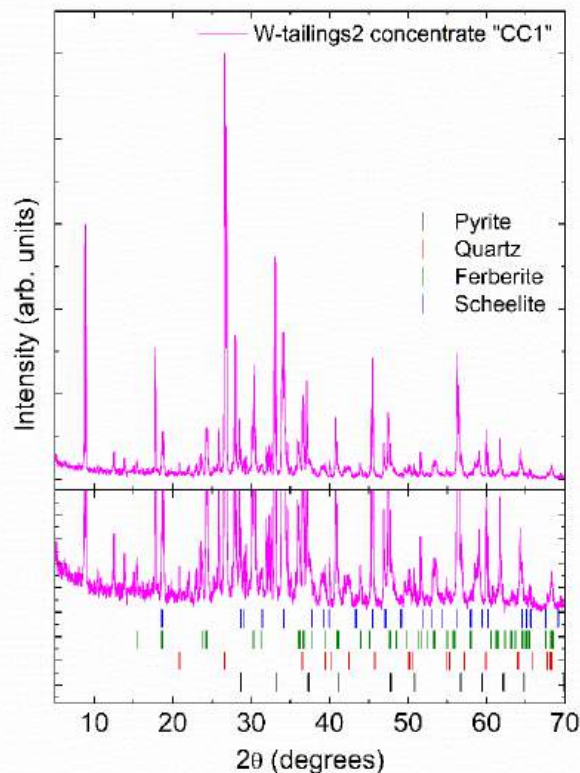


Figure 14. XRD patten of CC1 sample. Vertical lines denote the theoretical reflections of the phases of interest.

→Scanning electron microscopy – energy dispersive X-ray spectroscopy (SEM-EDS)

The polished sections were observed by back-scattering electrons SEM distinguish different mineral phases by chemical contrast and then EDS analyses were performed to identify the phases determined by XRD.

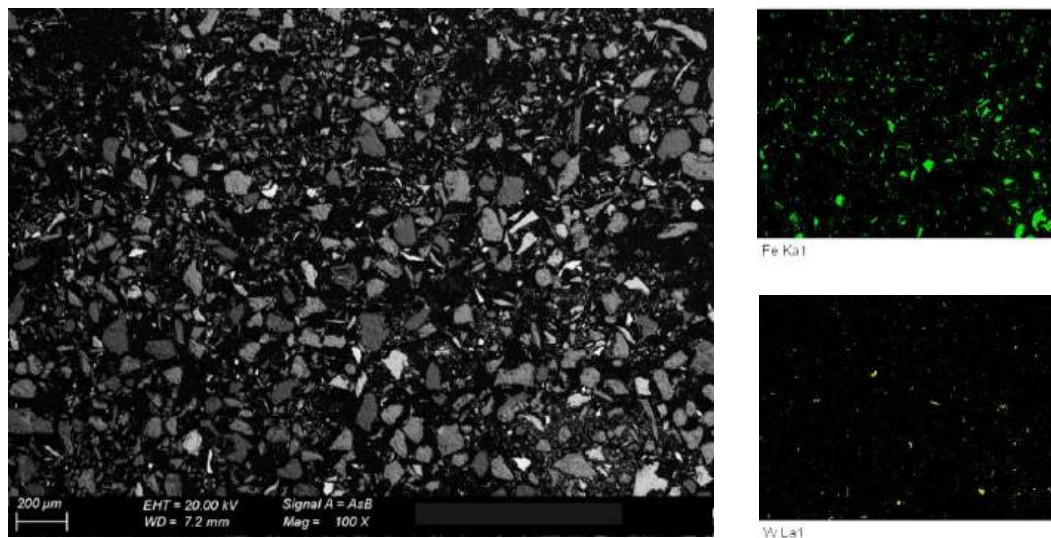


Figure 15. SEM image (x100) of W-tailings concentrate CC1. Right panels display SEM-EDS images for different elements: Fe (top) and W (bottom)

From the overview image of “CC1” (**Figure 2**), it can be clearly corroborated that the particle size is below 100 µm. The SEM-EDS images for Fe and W revealed that, overall, the particles of W minerals are smaller than the pyrite particles.

The detailed SEM-EDS images collected with enlarged magnification (x500), **Figure 3 and Figure 4**, showed that most of the particles were composed by single minerals. Only few larger grains were showing mineral inclusions. The presence of Ferberite as the main W-bearing mineral was confirmed. The comparison of the elemental W, Mn and Al maps confirmed that Mn is not associated to Wolframite (see **Figure 5**).

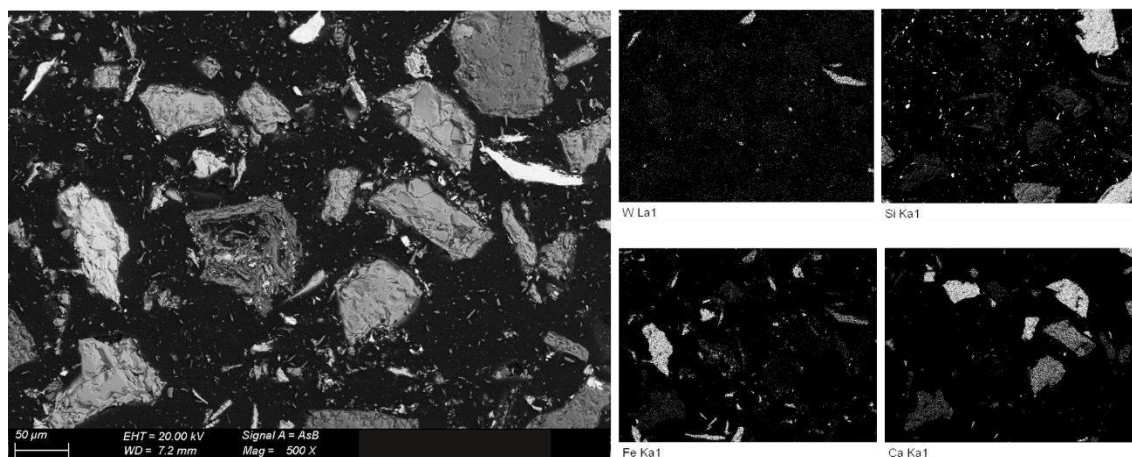


Figure 16. SEM image (x500) of W-tailings concentrate CC1. Right 4-quadrants represent the SEM-EDS images for different elements of interest: W, Si, Fe and Ca.

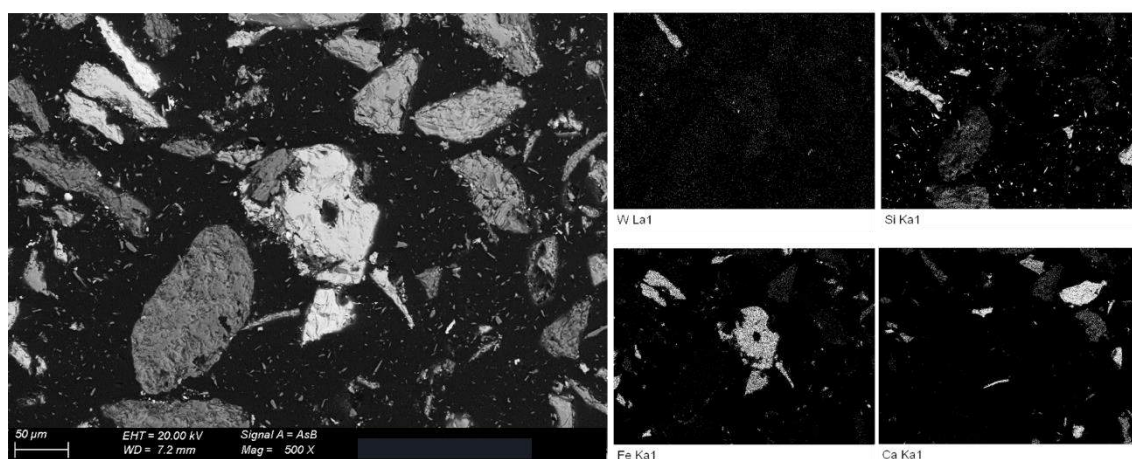


Figure 17. SEM image (x500) of W-tailings concentrate CC1. Right 4-quadrants represent the SEM-EDS images for different elements of interest: W, Si, Fe and Ca.

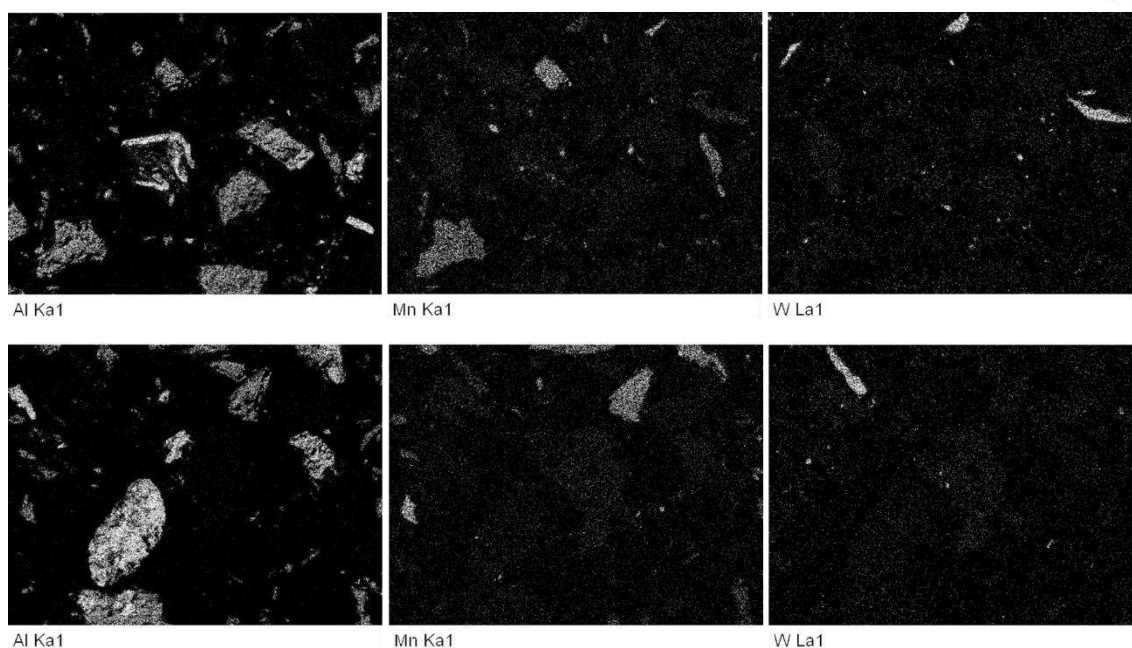


Figure 18. SEM-EDS of Al, Mn and W for two different regions of the W-tailings concentrate CC1.

Regarding Fe-bearing minerals, most of the Fe is associate with S as it can be seen in the elemental mapping comparison for both elements (**Figure 6**). This confirmed that the main Fe-bearing mineral present is pyrite, although there are several smaller grains of arsenopyrite (see **Figure 7**).

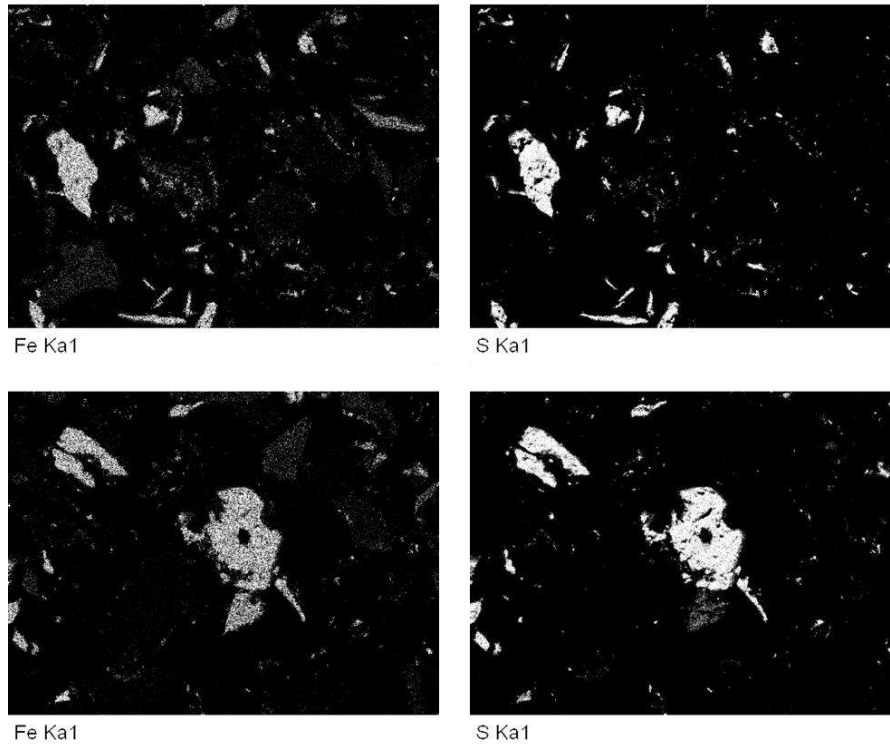


Figure 19. SEM-EDS of Fe and S for two different regions of the W-tailings concentrate CC1.

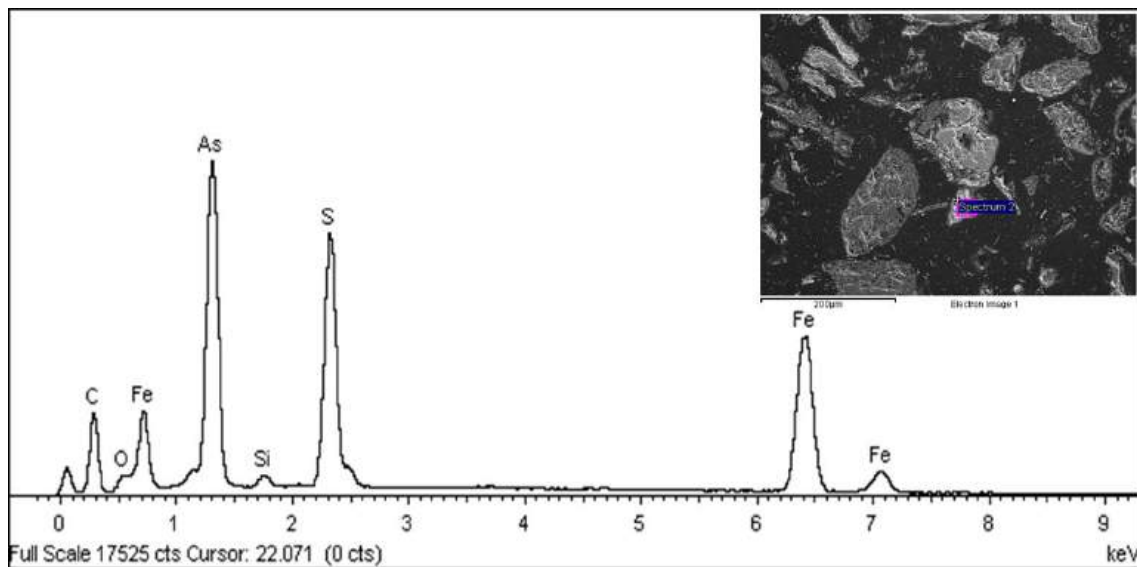


Figure 20. SEM-EDS of the typical arsenopyrite grain found.

The SEM-EDS analysis of Al and Si allowed the identification of quartz and aluminium silicates grains (**Figure 8**). The quartz appears as large grains and very fine powder whereas the grain size of the aluminium silicate phases is more similar. Regarding the aluminium silicate phases, the SEM-EDS analysis of different grains confirmed biotite as one of the main aluminium silicate phases **Figure 9**.

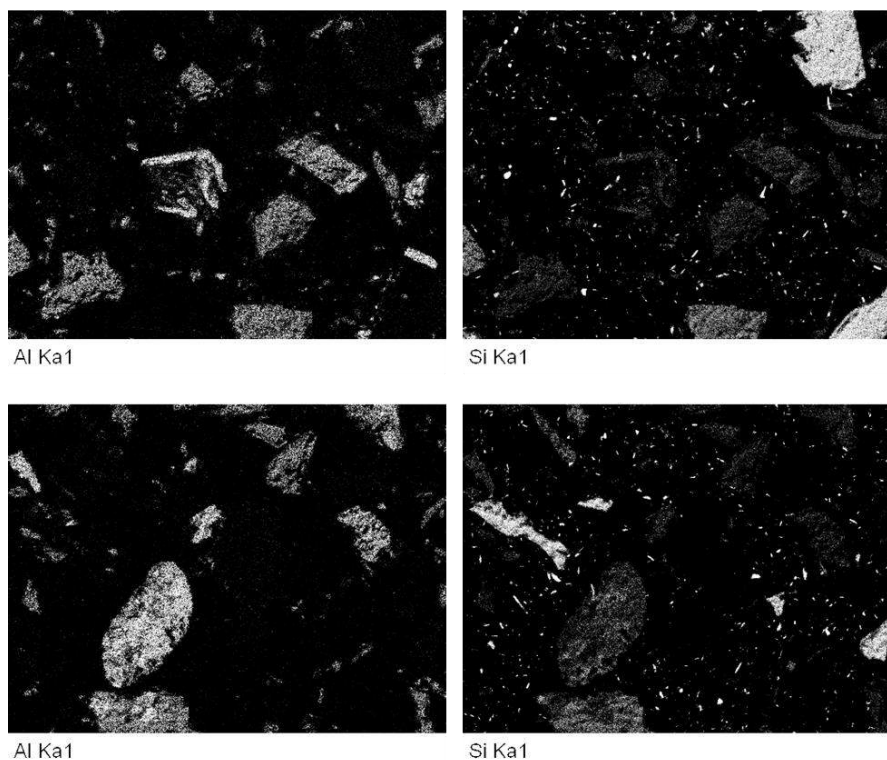


Figure 21. SEM-EDS of Al and Si for two different regions of the W-tailings concentrate CC1.

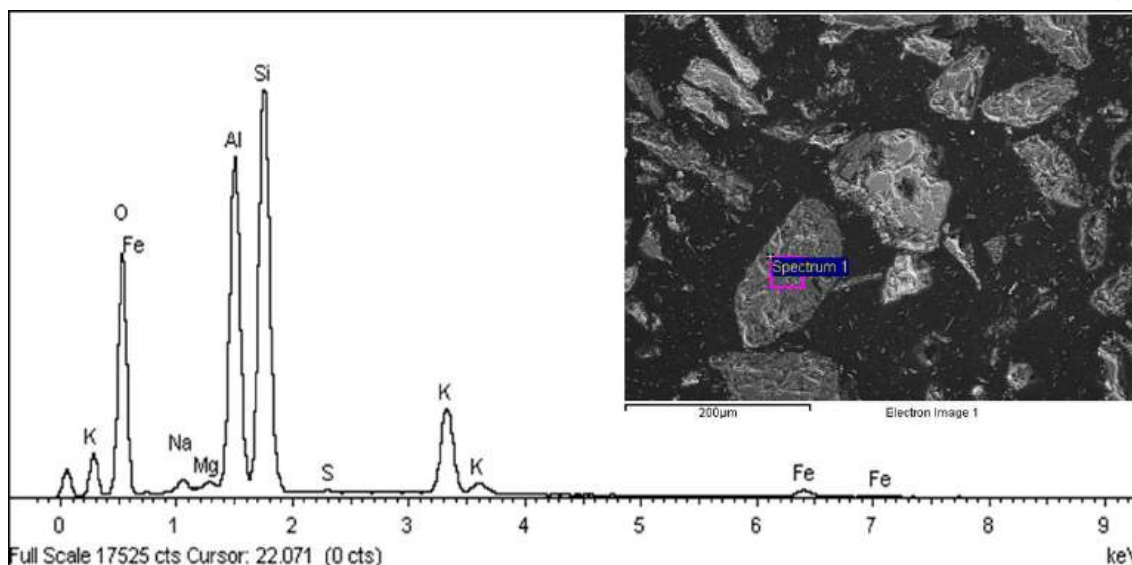


Figure 22. SEM-EDS obtained from an aluminium silicate grain of a biotite mineral.

According to the estimation of the elemental composition of different grains of the biotite minerals, Mn is present in the Fe-rich phases (see **Table 2**).

Table 15. Elemental composition of biotite mineral grains found in CC1.

Element	Weight (%)	Element	Weight (%)
O	51.3	O	42.1
Na	0.9	Mg	1.1
Mg	0.3	Al	10.7
Al	15.6	Si	17.3
Si	23.6	K	0.5
K	6.8	Ca	7.9
Fe	1.3	Mn	8.6
		Fe	11.8

In addition, although no calcium phosphate was detected by XRD, there elemental analysis performed showed several larger grains of this phase. **Figure 10** displays the elemental maps of Ca, P and W which allows distinguishing between mineral grains of the calcium phosphate and the minority scheelite phase.

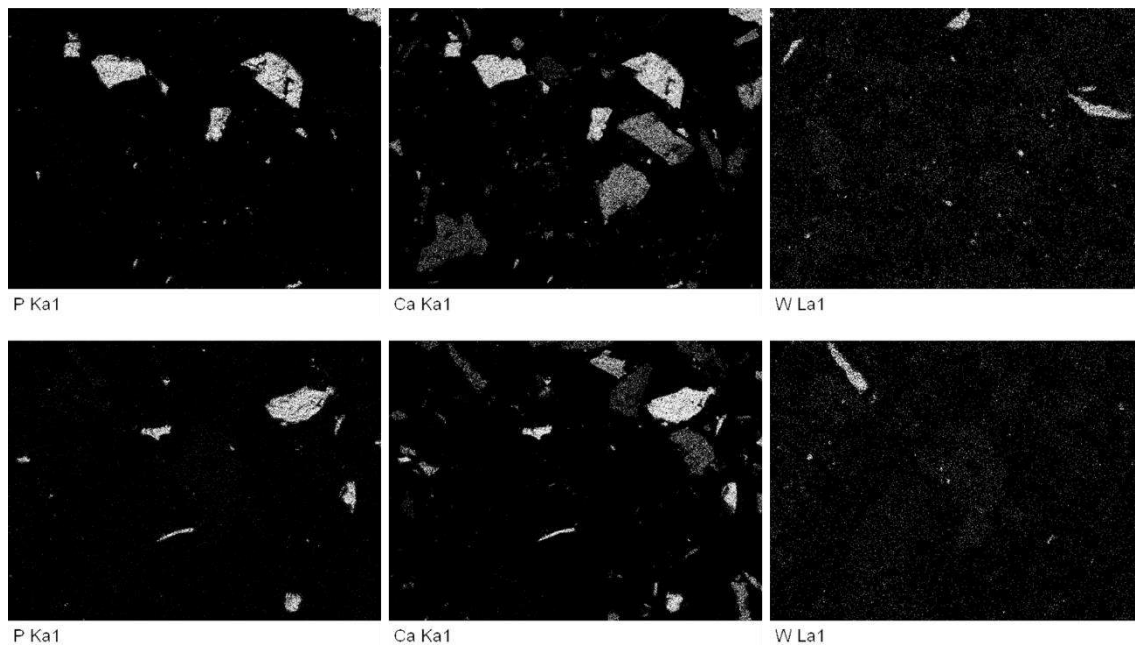


Figure 23. SEM-EDS of Al, Mn, and W for two different regions of the W-tailings concentrate CC1.