

Comparative laboratory study of conventional and Electric Pulse Fragmentation (EPF) technologies on the performances of the comminution and concentration steps for the beneficiation of a scheelite skarn ore

Kathy Bru, Mickael Beaulieu, Rui Sousa, Mário Machado Leite, Ana Botleho de Sousa, Erdogan Kol, Jan Rosenkranz, Daniel B. Parvaz

▶ To cite this version:

Kathy Bru, Mickael Beaulieu, Rui Sousa, Mário Machado Leite, Ana Botleho de Sousa, et al.. Comparative laboratory study of conventional and Electric Pulse Fragmentation (EPF) technologies on the performances of the comminution and concentration steps for the beneficiation of a scheelite skarn ore. Minerals Engineering, 2020. hal-02497971

HAL Id: hal-02497971 https://brgm.hal.science/hal-02497971

Submitted on 4 Mar 2020

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Comparative laboratory study of conventional and Electric Pulse Fragmentation (EPF) technologies on the performances of the comminution and concentration steps for the beneficiation of a scheelite skarn ore

The corrections made in this section will be reviewed and approved by a journal production editor.

Kathy Bru Formal analysis Investigation Methodology Conceptualization as k.bru@brgm.fr, Mickaël Beaulieu Investigation Methodology a, Rui Sousa Resources Formal analysis b, MárioMário Machado Machado Leite Leite Resources Formal analysis b, Ana Botleho Ana de Sousa Botleho de Sousa Resources b, Erdogan Ko Investigation c, Jan Rosenkranz Investigation c, Daniel B. Parvaz Investigation Formal analysis d,e

aBRGM, F-45060 Orléans, France
bLNEG National Laboratory of Energy and Geology, S. Mamede de Infesta, Portugal
cLuleå University of Technology, Mineral and Metallurgical Engineering, SE-971 87 Luleå, Sweden
dSELFRAG AG, Biberenzelgli 18, 3210 Kerzers, Switzerland
cLightning Machines Limited, 2nd Floor, Grove House, 774-780 Wilmslow Road, Didsbury, Greater Manchester M20 2DR, UK

Abstract

Electric Pulse Fragmentation (EPF) is an innovative technology that uses High-Voltage Pulsed Power (HVPP) for the selective comminution of a material. This paper aims to compare a beneficiation flowsheet including an EPF treatment in the comminution circuit to a conventional pathway where the EPF step was replaced by a series of jaw crushers. Tests were performed on a skarn ore containing scheelite as the main mineral of interest. This ore is characterized by a fine-grained mineralogy and represents a challenge to conventional comminution processing, requiring fine grinding to liberate the valuable minerals. Fine grinding has high energy requirements and

generates large amounts of fines which can result in losses of the target mineral due to their removal before the concentration processes, especially in this case since scheelite is a brittle material. Comparison of EPF treatment to mechanical crushing with a similar product size P80 (i.e. 80% passing size) showed that the EPF treatment led to a significant increase in WO₃ content and distribution in the 0/250 µm size fraction suggesting a pre-concentration aspect to EPF treatment. Moreover, a marked improvement of the grindability of the ore treated at a discharged energy of 9.1 kWh/t was observed with values of 10.6 kWh/t compared to 14.5 kWh/t when conventional treatment was used. Subsequent grinding and concentration steps confirmed the positive impacts of the fragmentation selectivity and pre-weakening effect of the EPF treatment. In particular, a reduction in fines production was observed after ball milling and a better concentrate grade was achieved for a similar recovery rate when an EPF treatment was included in the comminution pathway compared to the conventional one. These results confirm the potential of the EPF treatment for improving the performances of the beneficiation processes of this scheelite-bearing skarn ore.

Keywords: Scheelite; High voltage pulse; Electric pulse fragmentation; Selective comminution; Concentration

1 Introduction

Tungsten (W) is a metal that is widely used in industrial, aerospace, and military applications. This is due to its unique properties including a high density, wear resistance and tensile strength, as well as a low reactivity, and the highest melting point of all metals. These properties make it irreplaceable in a wide range of applications such as in cemented carbide; super alloys; high-speed steels and mill products. Its high economic importance from its wide range of applications, a lack of viable substitutes and the EU's dependence on imports (Leal-Ayala et al., 2015) resulted in the inclusion of tungsten in the 2017 list of Critical Raw Materials for the European Union (European Commission, 2017). The primary supply of tungsten within the EU is currently less than 3%. China is the largest producer in the world, attaining 85% of total production, which represents approximately 90% of the supply of the European Union (Suárez Sánchez et al., 2015). One strategy to secure the tungsten supply is to promote and develop tungsten mining within Europe. European tungsten deposits are predominantly skarn-type, and their processing is often challenging due to a very fine grained, intergrown mineralization. Moreover, most of the high-grade ore reserves are depleting leading then to an increased need to process lower grade, more complex ores.

Scheelite (CaWO₄) is the most abundant tungsten mineral and is present in approximately two thirds of known tungsten deposit [Instruction: In the Proof version, there is a problem with the presentation of this reference: it is given in the following line leading to a large space in the dedicated sentence.](BGS, 2011). When scheelite presents a coarse liberation size, gravity concentration can be applied as concentration process. However, scheelite is a brittle mineral and tends then to produce a lot of fines during comminution (Kupka and Rudolph, 2018). Careful design of the beneficiation flowsheet is then required to avoid overgrinding and loss of tungsten to fines. Most of the recent [Instruction: The word "work" should be replaced by "works" please.] work on

scheelite processing is related to fine particle recovery by the improvement of flotation, the main challenge with scheelite flotation being that it is difficult to selectively recover scheelite in the presence of calcite because scheelite and calcite have the same cation in their crystal structure, and they have similar physical and chemical properties, such as specific gravity values, hardness, solubility and hydrophobicity (Deng et al., 2018).

An alternative approach for improving scheelite recovery could be to implement a selective comminution technology, which has the advantage of reducing fines production by generating cracks specifically at the boundaries between mineral grains rather than randomly as in a conventional mechanical comminution system. This allows the liberation of valuable components from the feed material without overgrinding. Liberation at coarser size ranges is not only beneficial for the separation techniques but can also lead to significant economic benefits for mineral processing operations, such as a reduction in the grinding energy as the material does not need to be ground as finely as in conventional comminution systems (Wang et al., 2012a). In addition, rocks not fragmented by the process are usually weakened by the dense fracture network generated, which increases the overall grindability of the material (Wang et al., 2011).

Selective comminution methods are usually based on anisotropic properties of the ore, usually related to varying properties of constituent minerals (e.g. mechanical, electrical or acoustic properties, thermal expansion). Electric Pulse Fragmentation (EPF) is one such method, and exploits differences in electrical and acoustic properties of component phases in a rock to selectively fragment them. EPF works by applying highly energetic electrical pulses (150———750 J/pulse) with a very fast pulse rise time (<500 ns) to materials immersed in a dielectric process liquid (usually water). At this pulse rise time, dielectric liquids are more resistive than solids and act as an insulator, forcing discharges through the immersed material. [Instruction:

Actually, for another manuscript recently accepted for publication in Minerals Enginnering, I have received a comment to this sentence by a reviewer and have then slightly changed it. In order to have the same description of the phenomena involved in the two papers, would it be possible to write this sentence like this. Thanks in advance.

"Upon discharge, a plasma channel forms between the electrodes and through the material that causes explosive expansion along its pathway through the material, and the collapse of the plasma channel produces a shock-wave that propagates through the material (electrodynamic fragmentation)."

Jupon discharge, a plasma channel forms between the electrodes and through the material that causes explosive expansion along its pathway through the material (electro dynamic fragmentation), and the collapse of the plasma channel produces a shock-wave that propagates through the material (electro hydraulic fragmentation).

The high selectivity of the process arises from the way the plasma channel and shockwaves interact with physico-chemical properties of the material: discontinuities in electrical permittivity and conductivity at phase boundaries locally enhance the electric field, forcing the discharge channels to these boundaries, while differences in acoustic impedance and elasticity of adjacent phases causes local shearing when subjected to shockwaves. Both of which serve to concentrate tensile stress at phase interfaces (Andres et al., 1999) causing breakage and allowing full liberation of components from the feed material. The potential of this technique to improve phase liberation and ore grindability (Andres et al., 1999; Wang et al., 2012a; Shi et al., 2013; Bru et

al., 2018) and to pre-concentrate an ore by selective fragmentation of metal rich phases (Zuo et al., 2015; Shi et al, 2015) was already demonstrated by several authors, but its application to a skarn ore containing scheelite has not yet been reported.

This work aims to study the influence of EPF technology on the selective fragmentation and on the concentration of a skarn ore containing scheelite. Since this technology has been shown to work better on coarse particle sizes (Wang et al., 2012b; van der Wielen et al., 2013), its integration in a traditional mechanical comminution circuit is required in order to achieve optimal technical and economic benefit. In this study, the combination of the EPF technology with subsequent crushing, grinding and concentration steps was investigated and a specific methodology was implemented in order to compare this innovative pathway to a conventional one.

2 Materials and methods

2.1 Ore samples

The ore used in this study comes from the Tabuaço deposit which is a stratabound skarn-type deposit, situated in the Central Iberian Zone, of the Iberian Variscan Belt in Portugal (Gomes, 2016). Vesuvianite and fluorite are the most common minerals, but Tabuaço skarns are also composed of a variety of banded sodic plagioclase, K-feldspar, epidote, zoisite/clinozoisite, diopside, garnet (grossular), hornblende, tremolite-actinolite, quartz, sericite, calcite, apatite and scheelite (Sousa et al., 2018). The high scheelite (CaWO₄) grades occurring in certain areas of the deposit, coupled with a low sulphide content, are good prospects to produce high grade concentrates from the Tabuaço deposit (Colt Resources Inc., 2013) but exploration works still need to be done for defining resources and estimating reserves. Part of this ore represents a big challenge for the mineral processing flowsheet with a very fine scheelite liberation size, although starting in the range of 2–5 mm. The sample used in this study was collected in this area and graded 0.3–0.4% WO₃. This sample is characterized by a fine-grained mineralogy, the scheelite minerals having a mean grain diameter of 160 μm with particles as small as 10 μm diameter. This ore has a crushability of about 13.3 kWh/t (measured with a Bond low energy impact test) and a Bond ball mill work index of about 13.3 kWh/t.

2.2 Experimental set-up

The tests were conducted using the 'Lab', a laboratory scale EPF device for the batch processing of material, manufactured by SELFRAG AG, Switzerland (Fig. 1). The Lab is designed to process samples of up to approximately 1 L volume, or single particles with a top passing size of 40–45 mm in a 4 L vessel filled with de-mineralized water. It produces high voltage (90–200 kV) electric discharges of short duration between the two electrodes: the 'working' electrode is immersed in the upper part of the vessel, while the bottom of the vessel constitutes the 'counter' electrode. The operating parameters that can be changed are the voltage, electrode gap, pulse repetition rate and number of electric pulses applied to the sample. More details about the SELFRAG Lab can be found in Bru et al. (2018) and van der Wielen (2013). In particular regarding the energy delivery of the lab system, two types of energy can be considered:

(1) the energy consumed by the machine, called generator energy (in J or kWh) or specific energy

- for a given sample mass (in kWh/t): this is the set energy delivered to the sample calculated using the set voltage, capacitance, number of pulses, and sample mass.
- (2) the energy discharged into the material, called spark energy (in J or kWh) or discharged energy for a given sample mass (in kWh/t): this is the net electrical energy delivered to the samples measured by the batch device and displayed on the control panel after each test. This is the energy considered in this work.

Fig. 1



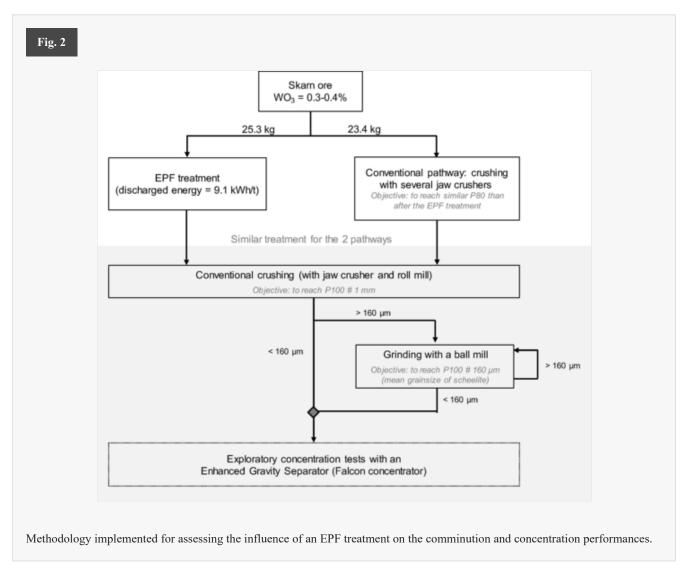
SELFRAG Lab machine.

The tests were carried out on a single-particle mode (after Shi et al., 2013) as it has been shown that when several particles are placed in the batch vessel then the energy is not evenly distributed among all particles. In single-particle testing mode, a single particle is placed each time inside the process vessel, directly under the electrode and immersed in water. The chosen number of pulses is then discharged to treat the particle. This procedure allows performing tests with a higher reproducibility and allows then comparing results obtained at different operating conditions.

Tests were performed on samples of about 40 mm diameter. Investigation of the influence of the discharged energy can be performed either by varying the number of pulses or the voltage. Van der Wielen et al. (2013) reported that overall energy input has a greater effect on particle size distribution that either voltage or number of pulses alone, it was then chosen to investigate the influence of the discharged energy by varying the number of pulses since this allows testing a larger energy range (because of voltage setup). The voltage was set at 150 kV with a pulse rate fixed at 5 Hz based on preliminary tests. Each test was replicated several times, with a minimum of 4 replicates.

To study the influence of EPF treatment on comminution and concentration circuit performance, a specific methodology was implemented that allowed comparison of EPF and conventional pathways i.e. mechanical crushers, in parallel (Fig. 2). The first step consisted of producing a 25 kg EPF treated sample using the

SELFRAG Lab at a given discharged energy (obtained with 87 replicates). For conventional fragmentation, a series of jaw crushing were used to produce a 24 kg sample with a similar product size P80 (i.e. 80% passing size) to that produced by EPF at the specific chosen energy. This choice was motivated by the fact that it allows investigating the evolution of the fracture network generated during EPF in the next crushing steps. Both samples subsequently underwent similar comminution and concentration steps. They were conventionally crushed down to 1 mm before the ball milling step using a jaw crusher and a roll mill. In order to avoid overgrinding of the size fraction lower than 160 μ m (mean grain size of scheelite) during ball milling, the samples were then sieved and the undersize was removed. All fractions lower than 160 μ m (obtained before and after ball milling) were then combined before performing the concentration tests.



Fine grinding was performed in a batch wet ball mill. Milling was carried out for 7 min on a sample of about 2 kg and prepared at a solid ratio of about 66%. The ball charge was fixed at 20.1 kg. After each batch, the product was sieved at 160 µm (the mean grain size of scheelite) to remove the undersize and circulating load was calculated. The undersize was then replaced by an equal weight of fresh feed to bring the weight back to that of the original charge. This sample is returned to the ball mill and this procedure was repeated 3 times in order to produce enough material for the concentration tests.

Exploratory concentration tests were performed with a Falcon SB40 concentrator. Falcon concentrator is an enhanced gravity separator (EGS) consisting of a spinning bowl which can be operated at high rotation speed (up to 300 G), allowing to separate fine particles of different density. Tests were performed on samples of about 150 g with a fluidization counter-pressure fixed at 12 psi and a centrifugal force of 39 G. The objective of these preliminary tests was to check if the EPF treatment can have an influence on the performances of the concentration steps.

2.3 Characterization method

Textural and surface properties of the samples were studied by Scanning Electron Microscopy (SEM) on a Tescan Mira 3 XMU, equipped with a Secondary Electron (SE) Everhart-Thornley detector (Everhart and Thornley, 1960) for high vacuum SE imaging, a low vacuum (LV) SE detector (Jacka et al., 2003) for LV – SE imaging and a YAG-scintillator Backscattered Electron (BSE) detector (Autrata, 1992). Energy-dispersive X-ray Spectroscopy (EDS) microanalysis was performed on an Edax Pegasus system with an ApolloXPP silicon drift detector and TEAM software. SEM images and EDS spectra were collected using a low vacuum mode on non-coated sample at a high voltage HV = 25 kV, and nitrogen pressure P = 40 Pa.

The particle size distribution (PSD) was measured by manual sieving at twelve sizes (40 mm, 20 mm, 16 mm, 8 mm, 5 mm, 3.36 mm, 2 mm, 1 mm, 500 μ m, 250 μ m, 125 μ m and 63 μ m), which allowed to estimate the product size P80.

The WO₃ content was determined by portable XRF calibrated against in-house W standards. Representative splits of each sample were pulverized before analysis.

2.4 Small scale grindability test

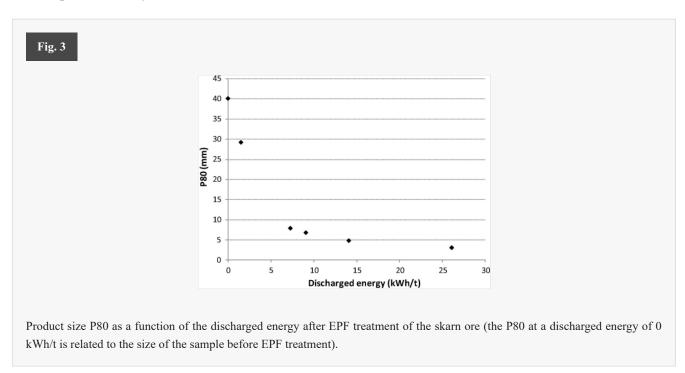
For determining the effect of EPF treatment in comparison with the conventional crushing of the samples, a small scale grindability test has been applied. The test is part of the Geometallurgical Comminution Test (GCT) and uses only 220 g of pre-crushed material and much less processing time for getting a measure for ore grindability and an estimate of the Bond ball mill work index (Mwanga et al., 2015). The procedure involves dry batch grinding in a small laboratory ball mill that has a volume of ca 1.4 L only. The mill is running on the same percentage of critical speed as the original Bond mill, thereby fulfilling the criteria of kinematic similarity as described by a constant Froude number. In addition, the fraction of mill volume occupied by the ball charge and the size range of the grinding media have been kept close to the original Bond mill test.

Particle size distributions from dry sieving are numerically evaluated for the 80% passing size for a sequence of 2, 5, 10, 17 and 25 min. Using the 80% passing size together with a recording of the gross electrical power draw of the ball mill, a GCT work index is determined from applying the Bond law of comminution. For many ores, the work index received from the GCT test is linearly correlated with the Bond work index (Mwanga et al., 2017).

3 Results and discussion

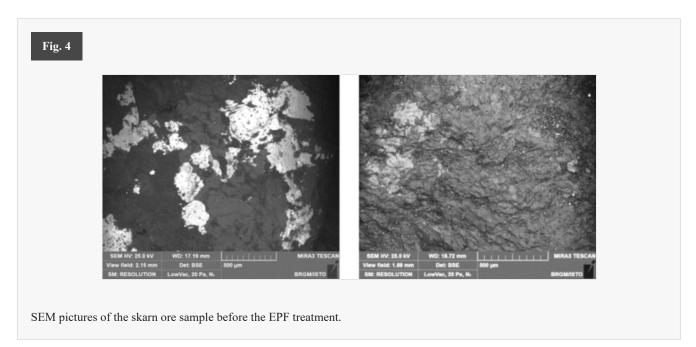
3.1 Effect of the EPF discharged energy on electrical comminution performance

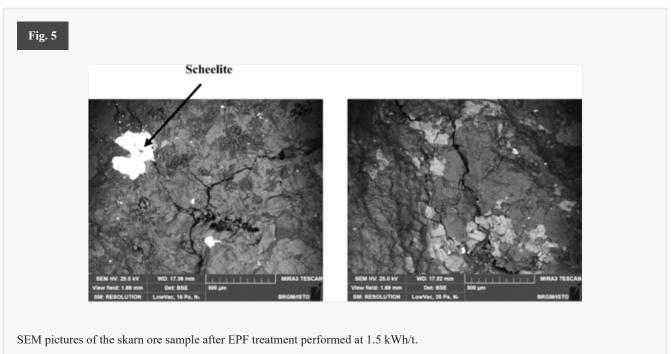
The influence of the discharged energy on the product size P80 after EPF is shown in Fig. 3. It can be observed that fragmentation occurred even at a low discharged energy (1.5 kWh/t). Increasing energy from 1.5 to 9 kWh/t correlates with a sharp decrease in P80 from 40 mm to 6.7 mm. Above 9 kWh/t, the rate of size reduction decreases before levelling off in the high energy range (>15 kWh/t). Similar trends were reported in van der Wielen et al. (2013), Zuo et al. (2015) and Bru et al. (2018), with the additional observation of an initial phase at low energy levels (<3 kWh/t) where little size reduction occurred. This initial phase was explained by the requirement of a material-specific threshold discharged energy to fully overcome particle strength and lead to the breakage of the majority of the particles (van der Wielen, 2013). This initial phase is not observed in Fig. 3, most probably because the threshold energy for breakage for this skarn ore is lower than 1.5 kWh/t. The asymptote in fragmentation above 9 kWh/t is due to the non-linear relationship between energy input and size reduction: after a certain energy input (material specific), additional input causes little further breakage (as in Bru et al., 2018). According to van der Wielen et al. (2013), this may be because a larger particle can 'bridge' the gap between the two electrodes allowing for a reduced travel distance through the processing water, which means that almost all the energy is discharged in the particle. Conversely, smaller particles are unable to fully bridge the gap between electrodes and therefore the pulses may be required to jump from particle to particle several times, involving a longer total travel distance through water and then a larger portion of energy lost to the water instead of used for fragmentation. Moreover, breakage of larger particles corresponds to a greater reduction in P80 than breakage of a smaller particle for a similar breakage ratio e.g. 50% size reduction of a 40 mm particle leads to a 20 mm reduction in size, while a 50% reduction in a 4 mm particle is only 2 mm.

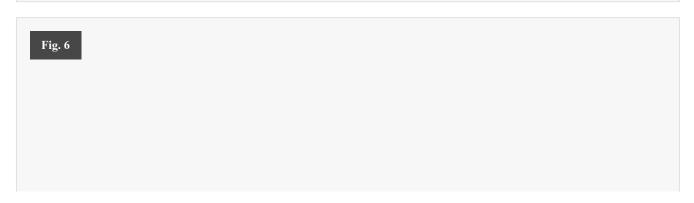


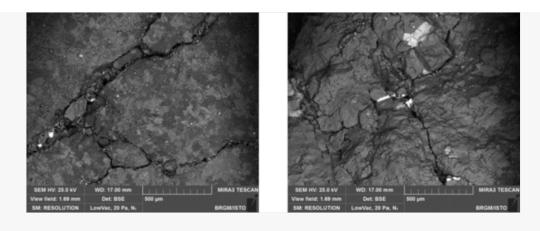
SEM investigation was performed on feed samples before the EPF treatment and on fragments obtained after the EPF treatment performed at a discharged energy of 1.5 kWh/t and of 9.1 kWh/t (Fig. 4, Fig. 5 and Fig. 6 respectively). These samples showed fractures at the surface of all the EPF treated fragments, even when

overall size reduction was relatively low (1.5 kWh/t), which were not observed in the untreated material. These 'micro-cracks' are thought to lead to a reduction in particle strength and therefore energy required to grind the ore, as observed by Wang et al. (2011) on various ores.



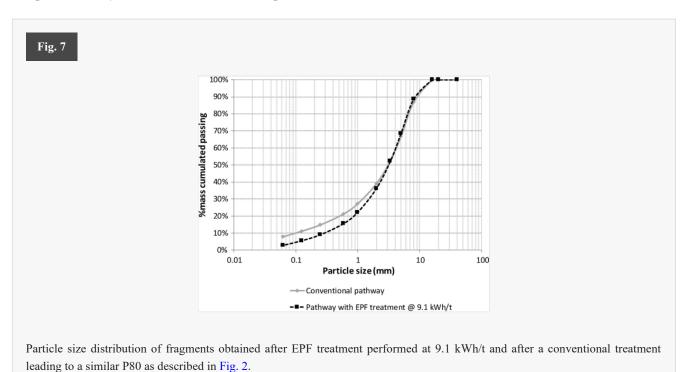






SEM pictures of the skarn ore sample after EPF treatment performed at 9.1 kWh/t.

A discharged energy of 9.1 kWh/t was chosen for performing the comparison with the conventional pathway as this represented the optimal EPF energy/breakage before the flattening of the P80 curve with higher levels of discharged energy (Fig. 3). As described in Fig. 2, the conventional pathway uses a specific combination of jaw crushers in order to produce a sample with the same P80 as the one obtained after EPF treatment performed at 9.1 kWh/t. Despite a similar P80, the conventional pathway produced a larger amount of fines than EPF (Fig. 7), with a product size P10 (i.e. 10% passing size) of 105 μm for the conventionally treated sample and 304 μm for the EPF treated sample.



The WO₃ content was analyzed in all size fractions of the fragments obtained after the various EPF treatments and after the conventional pathway. Even if a minimum of four replicates were performed for each operating condition, reaching a minimum mass of about 1 kg for each sample, it was observed that the WO₃ content is not identical between all the samples due to the low WO₃ content of the ore. The influence of the EPF technology on the selective comminution of scheelite was then evaluated by comparing the WO₃ content of

each size fraction to the initial WO₃ content in the bulk sample, allowing to calculate the increase rate of WO₃ content in each size fraction as defined by the following formula (Fig. 8):

Increase rate of WO_3 content in size fraction $i = \frac{WO_3 \text{ content in size fraction } i - \text{Average } WO_3 \text{ content of the ore}}{\text{Average } WO_3 \text{ content of the ore}}$

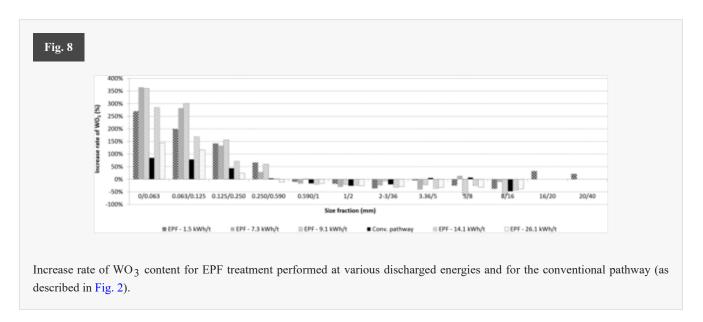


Fig. 8 shows strong increase rates of the WO₃ content in the fractions lower than 250 μm for the EPF test performed at a discharged energy lower than 9.1 kWh/t, an increase of the discharged energy leading then to a decrease of this rate. This pre-concentration effect can be explained by the selective nature of EPF treatment, wherein the electric discharge is preferentially attracted to metal rich areas of the ore rock meaning that metalrich particles (usually ore minerals) are first crushed and broken away from the host rock, while gangue remains relatively coarse (Zuo et al., 2015). This leads to an initial enrichment of metals in the fine product and depletion in the coarse fraction. If additional energy is added, increasing amounts of gangue are fragmented and become fine product, diluting the concentration of metals in the undersize. The chemistry of the fine product then tends towards that of the original rock when increasing the discharged energy until all material has been finely crushed. At a given discharged energy, the increase rate of WO₃ content rises with the fineness of the size fraction. This is because scheelite is brittle and easily breaks and deports to the fine size fractions. The brittleness of the scheelite also explains the slight increase rate of WO₃ content in the 0/250 μm size fractions observed with the conventional crushing pathway. These results indicate different breakage mechanisms between the EPF technology and the conventional crushing. While the precise selective breakage mechanism has not been identified, EPF favors the preferential deportment of WO3 in fine size fractions compared to mechanical breakage, which is due to the different properties of the minerals contained in the ore.

The potential of the EPF pre-concentration effect to remove barren rocks in the coarse size fractions was investigated through the calculation of the WO_3 distribution (Fig. 9). Results showed a strong increase of the WO_3 distribution with the discharged energy in the size fractions lower than 250 μ m while size fractions

coarser than 1 mm showed an inverse trend. However, the WO₃ distribution is size fractions coarser than 8 mm or 16 mm was quite significant, especially for the test performed at 1.5 kWh/t, which means that in this case the EPF technology cannot be considered as a pre-concentration step. In order to compare the performances of the comminution method, it was chosen to plot the cumulative WO₃ distribution from fine to coarse sizes, showing the effectiveness of a selective screening applied after comminution (Fig. 10). The diagonal line in Fig. 10 represents the results of a random comminution; it is called "No upgrading line" (after a selective screening) since in a random comminution each size class has the same grade, this grade being similar to the ore average grade. Results show that a selective screening can be applied after EPF to concentrate scheelite in the finer size fractions and gangue in the coarser fractions, an increase in the discharged energy leading to a higher level of discrimination compared to the random comminution. Results regarding the EPF test performed at 1.5 kWh/t seem to be quite erratic but this may be due to the fact that the discharged energy is low, being achieved with only 5 electrical discharges. The low number of discharge events increases the effect of a single discharge missing the sample, or hitting the sample with little to no effect.

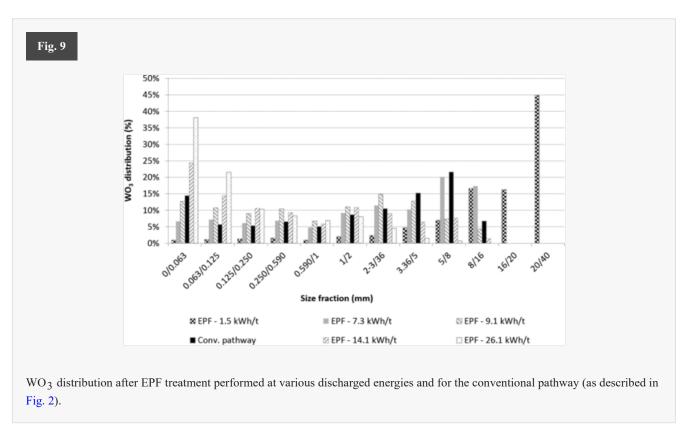
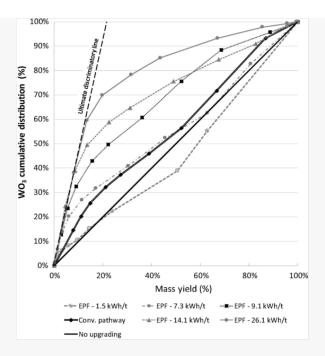


Fig. 10



WO₃ cumulative distribution, from fine to coarse size fractions, after EPF treatment performed at various discharged energies and for the conventional pathway (as described in Fig. 2).

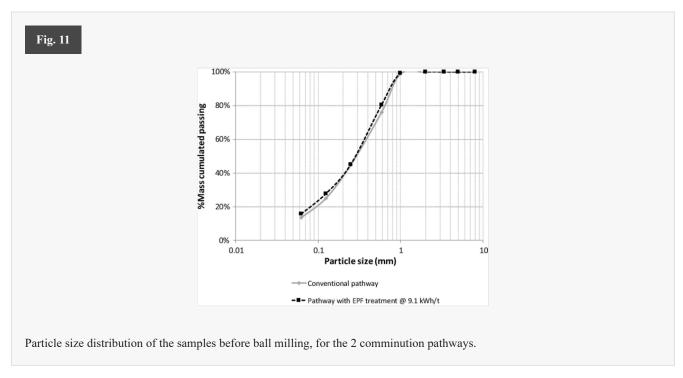
Interestingly, it can be observed in Fig. 10 that the slopes of the linear part (first ranges of mass yield) of all WO₃ cumulative distribution curves after EPF treatment are similar and are higher than the one related to the conventional pathway. This phenomenon is represented in Fig. 10 by the "Ultimate discriminatory line" which represents the highest discriminatory effect which could be possible to reach. This means that an increase of the EPF discharged energy allows improving the WO₃ recovery obtained when implementing a screening after the EPF treatment. Nonetheless, there is a threshold in the improvement of the WO₃ recovery i.e. the plots will always move away from the ultimate discriminatory line at a given mass yield whatever the discharged energy used for the EPF. Indeed, as observed in Fig. 8, an increase of the discharged energy not only lead to breakage around metal-rich particles but also in the gangue. Moreover, the improvement in WO₃ cumulative distribution is non-linear regarding the discharged energy, a strong increase being observed between 7.3 kWh/t and 9.1 kWh/t while only a small increase was obtained between 9.1 kWh/t and 14.1 kWh/t (Fig. 10). Additionally, from a process point of view, it may be requested not to use a too high discharged energy because this would generate many ultra-fine particles which would reduce the efficiency of the following upgrading stages.

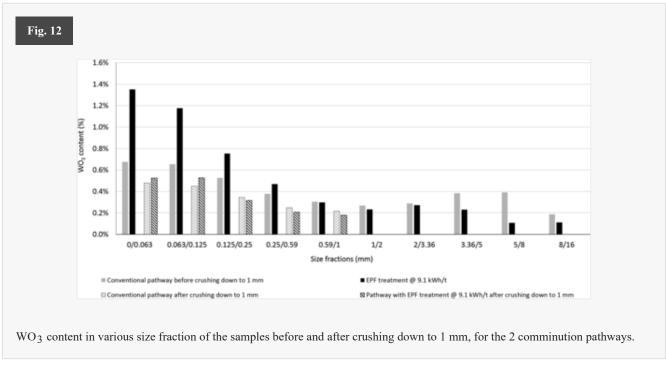
Since the EPF treatment is performed in place of some crushing steps (regarding only size reduction), additional comminution steps are required for the mineral processing of this skarn ore. It is then crucial to check that the benefits of the EPF treatment for the selective recovery of scheelite are not lost subsequently.

3.2 Comparison of the EPF treatment to the conventional pathway during the comminution circuit implemented before the concentration steps

As mentioned before, the comparison of the EPF technology to the conventional pathway[Instruction: The word "pathways" should be replaced by "pathway". Thanks.]s was done considering an EPF treatment performed at a discharged energy of 9.1 kWh/t and the conventionally treated sample was specifically prepared

for reaching a similar P80. Both samples were crushed down to 1 mm before the ball milling step using a jaw crusher and a roll mill. The PSD of the samples is given in Fig. 11 and show that the EPF-treated samples is slightly finer. Regarding the WO₃ content in the various size fractions (Fig. 12), it could be observed a dilution of the WO₃ content after crushing the samples down to 1 mm due to additional fragmentation of gangue.

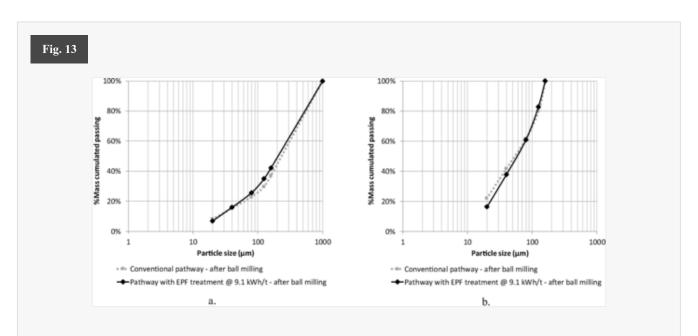




In order to avoid overgrinding of the size fraction lower than 160 μ m (mean grain size of scheelite), the samples were sieved before ball milling and the undersize was removed. Similar ball milling steps were then performed on the EPF and on the conventionally treated samples. All fractions <160 μ m (obtained before and after ball milling) were then combined before performing the concentration tests. For this combination, the ratio of the sieving at 160 μ m before ball milling was taken into account to make representative samples.

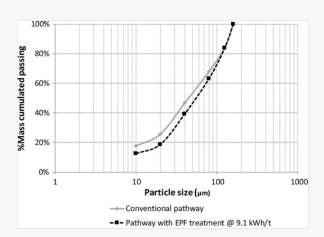
Different circulating loads were obtained during ball milling, with the circulating load of the EPF treated sample being 145% while a circulating load of 170% was measured for the conventionally treated sample. These results confirm the weakening effect of the EPF treatment as described in other publications (Wang et al., 2011; Shi et al., 2013). While this can be advantageous regarding the energy required for the comminution circuit, it can also be detrimental to the comminution process if this leads to overgrinding: an increased amount of fine particles can impede the concentration processes and are often necessarily removed before these steps (Sivamohan, 1990; Roy, 2009). Without flowsheet redesign, this may lead to a loss of the target metal, especially here since scheelite is a brittle material.

During ball milling, the PSD of the sample coming from the conventional comminution circuit was slightly coarser than the one coming from the comminution circuit with an EPF treatment (Fig. 13a), however this is no longer the case when considering only the size fraction 0/160 μm (Fig. 13b). This effect is retained after recombining the size fractions 0/160 µm before performing the concentration tests, as showed in Fig. 14. In particular, the amount of particles lower than 10 µm was 12% for the EPF treated sample while it was 18% for the conventional comminution circuit. When looking at the WO₃ distribution in various size fractions (Fig. 15), it is observed that the EPF-treated product had more WO₃ in the size fractions above 20 μ m. In the 0/20 μ m size fraction, the trend changes with firstly a similar WO₃ distribution in the 10/20 µm size fraction and then a WO₃ distribution being 1.6 times larger in the conventional pathway in the 0/10 μm size fraction. This is probably due to EPF's tendency for preferentially fracturing the rock along grain boundaries of metal-bearing particles rather than through grains, resulting in less overall breakage of pure scheelite particles - once liberated, the discharge and resulting shockwave preferentially fractures grain boundaries in polymineralic particles over monomineralic. These results highlight the positive impact of EPF on the selectivity of the comminution. These results are crucial for optimizing the performances of the beneficiation process since particles finer than 10 µm are often removed before the concentration steps; in this case, the WO₃ loss will be 19.8% for the conventional pathway compared to a WO₃ loss of 12.4% for the pathway with the EPF treatment.



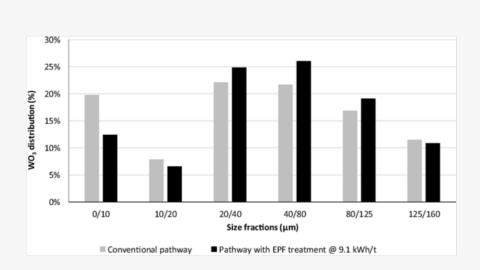
Particle size distribution of the samples produced during ball milling, for the 2 comminution pathways. (a. considering the whole samples, b. considering the size fraction $0/160 \mu m$ of the ground samples).





Particle size distribution after ball milling and before the concentration tests for the samples from the two comminution pathways.





WO₃ distribution in various size fractions after ball milling and before the concentration tests for the samples from the two comminution pathways.

3.3 Influence on the energy consumption of the comminution circuit

The influence of the EPF treatment performed at a discharged energy of 9.1 kWh/t on the grindability of the ore was analyzed using the small scale grindability test that allows determining the energy needed to grind the material down to $100 \, \mu m$, as described above. Estimates for the work index of the skarn ore are given in Table 1. In comparison with the conventionally crushed samples, the specific grinding energy required for the EPF treatment on this sample.

Table 1

(i)

The table layout displayed in this section is not how it will appear in the final version. The representation below is solely purposed for providing corrections to the table. To preview the actual presentation of the table, please view the Proof.

Grindability of the skarn ore fragments after the EPF treatment or the related conventional treatment (with similar P80).

	GCT work index from small scale grindability test (kWh/t)	Variation from conventionally crushed sample	
EPF treatment (discharged energy = 9.1 kWh/t)	10.6	-27.2%	
Conventional treatment	14.5		

Considering the energy consumption of the whole comminution circuit studied here, i.e. taking into account the energy used for the first fragmentation step (either conventional and with the electric pulse fragmentation), Table 2 shows that using an EPF treatment in the comminution circuit leads to an increase of about 23% of the grinding energy. The crushability index was considered here for the energy consumption of the jaw crushing steps implemented in the conventional pathway instead of the EPF treatment. However, it should be mentioned that this comparison is an estimation only and should then be considered carefully. Indeed, the energy consumption of the crushing steps replacing EPF treatment was estimated based on a robust and validated method (using the Bond formula law) while the energy consumption of the EPF treatment was measured during tests performed at lab-scale. Bru et al. (2018) showed that the energy consumption of a batch lab-scale EPF equipment largely exceeds the one of a continuous pilot-scale E[Instruction: EDF should be replaced here by "EPF" (typo mistake). Thanks.]DF system for similar performances. This is due to a lower electrical energy efficiency of the generator of the lab-scale equipment, and also to the single-particle mode used for the lab-scale tests in which the particle decreases in size with each discharge, reducing breakage efficiency (feed size effect, see above), while a continuous feed of particles can be used at pilot scale

Table 2



The table layout displayed in this section is not how it will appear in the final version. The representation below is solely purposed for providing corrections to the table. To preview the actual presentation of the table, please view the Proof.

[Instruction: In the proof, the first column appears very small. It should be enlarged for a better writting of the information inside please.] Energy consumption of the whole comminution circuit (from about 40 mm down to 100 μm) for the two comminution pathways.

	consumption (kWh/t)	conventionally crushed sample
EPF treatment (discharged energy = 9.1 kWh/t)	19.7	
Conventional treatment (energy consumption of the crushing steps replacing EPF treatment = 1.5 kWh/t estimated using the Bond formula law and a crushability index of 13.3 kWh/t for the studied ore)	16.0	+23.1%

3.4 Influence on the separation step with a Falcon concentrator

The exploratory separation tests were performed with a Falcon concentrator on samples ball milled to 160 μm. Results are given in Table 3. Inclusion of EPF treatment in the comminution pathway led to an increase by more than 44% of the WO_3 content in the heavy fraction, with a WO_3 content going from 3.27% to 4.72%. The WO₃ recovery rate was slightly improved with the EPF pathway but optimization still need to be performed in order to reach a higher WO3 recovery rate and a lower WO3 content in the light fraction which could then be considered as tailings. Potassium was also analyzed in the samples since it was shown by Foucaud et al. (2019) that it could be used as a proxy to represent the light silicates fractions. Results show that the light silicates fractions were mainly found in the light fraction as it could be expected, the recovery rates being around 97%, with no effect of the EPF treatment. More works could however be performed in order to still improve the separation of all the gangue minerals from the WO_3 concentrate.

Table 3

(i) The table layout displayed in this section is not how it will appear in the final version. The representation below is solely purposed for providing corrections to the table. To preview the actual presentation of the table, please view the Proof.

Concentration test with a Falcon apparatus set at 39 g and 12 psi for the samples from the two comminution pathways.

	Pathway with EPF treatment @ 9.1 kWh/t				
	0/ mass	WO ₃		К	
	%mass	Content (%)	Recovery rate (%)	Content (%)	Recovery rate (%)
Heavy fraction	4.8	4.72	57.4%	4443	2.8%
Light fraction	95.2	0.18	42.6%	7664	97.2%

	Conventional pathway					
	%mass	WO ₃		K		
		Content (%)	Recovery rate (%)	Content (ppm)	Recovery rate (%)	
Heavy fraction	5.9	3.27	49.3%	4359	3.3%	

Light fraction	94.1	0.21	50.7%	8074	96.7%
----------------	------	------	-------	------	-------

These results suggest that including an EPF treatment in the comminution pathway allows improving the quality of the concentrate after gravity concentration for a given set of operating conditions. However, additional tests need to be performed to optimize the scheelite recovery, in the perspective of a flowsheet design. This positive effect of EPF treatment on the recovery of the target metal in a concentration process was already reported by several authors (Andres et al., 2001; Parker et al., 2015). Mineral Liberation Analysis (MLA) conducted by Parker et al. (2015) on products obtained after both EPF treatment and a specific jaw crushing combination (to produce the same overall particle size distributions) showed that chalcopyrite was more liberated in the EPF treated products than in the mechanically treated ones. The significant improvement in the rod mill product's flotation performances was attributed to this improvement in mineral liberation.

4 Conclusions

A detailed study was undertaken to investigate the influence of Electric Pulse Fragmentation (EPF) technology on the selectivity of fragmentation and on the performances of subsequent comminution and concentration processes of a scheelite bearing skarn ore. A specific methodology was implemented to compare the pathway including an EPF treatment to the conventional pathway.

SEM observation of EPF treated fragments showed fractures at the surface of all fragments, even when size reduction was relatively low as observed after EPF treatment performed at a discharged energy of 1.5 kWh/t. These fractures were not observed in the initial material. These cracks can lead to a reduction in particle strength and then in the energy required to grind the ore. In particular, an improvement in the grindability of the ore was observed after EPF treatment performed at a discharged energy of 9.1 kWh/t, with values going from 14.5 kWh/t when conventional treatment was applied to 10.6 kWh/t when the EPF treatment was used, underlining the weakening effect of the EPF treatment. While a slight increase of the WO₃ distribution was observed in the size fractions lower than 250 µm for conventionally crushed material, most probably due to the brittle nature of the scheelite mineral, EPF treated material showed a strong increase of the WO₃ distribution with the discharged energy for these size fractions. These results underlined the selectivity of the EPF machine contrary to the mechanical breakage, and the potential for EPF to 'pre-concentrate' an ore mineral in the fine fraction.

The selectivity and the 'pre-weakening' effect of this technology were also highlighted during ball milling where a decrease in the circulating load and a reduction of the WO₃ loss in the 0/10 µm size fraction was observed. The exploratory gravity separation tests showed that a higher concentrate grade was obtained for a similar recovery rate when an EPF treatment was included in the comminution pathway compared to the conventional one. This may be due to a better mineral liberation obtained from EPF treatment. Further characterization works (including in particular mineral liberation analysis) and concentration tests are required to confirm these preliminary results. Moreover, since flotation is often required after the gravity separation step, it would be important to study the effect of an EPF treatment on the surface chemistry of the minerals and then on the resulting flotation performances.

CRediT authorship contribution statement

Kathy Bru: Conceptualization, Methodology, Investigation, Formal analysis. Mickaël Beaulieu: Methodology, Investigation. Rui Sousa: Formal analysis, Resources. Mário Machado Leite: Formal analysis, Resources. Ana Botleho de Sousa: Resources. Erdogan Kol: Investigation. Jan Rosenkranz: Investigation. Daniel B. Parvaz: Formal analysis, Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This study was performed under the project FAME ('Flexible And Mobile Economic processing technologies) which has received funding from the Instruction: In the proof, the typeface is smaller. This should be corrected please. European Union's Horizon 2020 research and innovation programme under grant agreement no 641650. The authors acknowledge the contributions of the FAME team for sample collection and scientific discussions that led to the development of this paper.

References



(i) The corrections made in this section will be reviewed and approved by a journal production editor. The newly added/removed references and its citations will be reordered and rearranged by the production team.

Andres, U., Jirestig, J., Timoshkin, I., 1999. Liberation of minerals by high-voltage electrical pulses. Powder Technol. 104, 37–49. doi:10.1016/S0032-5910(99)00024-8.

Andres, U., Timoshkin, I., Jirestig, J., Stallknecht, H., 2001. Liberation of valuable inclusions in ores and slags by electrical pulses. Powder Technol. 114, 40–50. doi:10.1016/S0032-5910(00)00260-6.

Autrata, R., 1992. Single crystal detector suitable for high resolution scanning electron microscopy. EMSA Bull. 22, 54-58.

BGS British Geological Survey, 2011. Mineral profile - Tungsten. MineralsUK.

Bru, K., Touzé, S., Auger, P., Dobrusky, S., Tierrie, J., Parvaz, D.B., 2018. Investigation of lab and pilot scale electric-pulse fragmentation systems for the recycling of ultra-high performance fibre-reinforced concrete. Miner. Eng. 128, 187–194. doi:10.1016/j.mineng.2018.08.040.

Colt Resources Inc., 2013. Tungsten mineralization in Portugal - the case of the São Pedro das Águias Deposit. In: Mineral resource assessment workshop: Assessment of the tungsten potential in Greenland, Copenhagen.

Deng, R., Huang, Y., Hu, Y., Ku, J., Zuo, W., Yin, W., 2018. Study of reverse flotation of calcite from scheelite in acidic media. Appl. Surf. Sci. 439, 139–147. doi:10.1016/j.apsusc.2017.12.231.

European Commission, 2017. Communication from the Commission to the European Parliament, the Council, the European Economic and Social Committee and the Committee of the Regions on the 2017 list of Critical Raw materials for the EU.

Everhart, T.E., Thornley, R.F.M., 1960. Wide-band detector for micro-microampere low-energy electron currents. J. Sci. Instrum. 37, 246–248.

Foucaud, Y., Dehaine, Q., Filippov, L.O., Filippova, I.V., 2019. Gravity processing of a complex tungsten skarn with a Falcon concentrator. In: MEI conference: Physical Separation, June 13–14, 2019, Falmouth, UK.

Gomes, G.L., 2016. Spatial simulation of the W-Sn ore grades of São Pedro das Águias skarn mineral deposit (Tabuaço, northern Portugal). Geological Engineering. Universidade Nova de Lisboa, Lisboa.

Jacka, M., Zadrazil, M., Lopour, F., 2003. A differentially pumped secondary electron detector for low-vacuum scanning microscopy. Scanning 25, 243–246. doi:10.1002/sca.4950250505.

Kupka, N., Rudolph, M., 2018. Froth flotation of scheelite – A review. Int. J. Min. Sci. Technol. 28, 373–384. doi:10.1016/j.ijmst.2017.12.001.

Leal-Ayala, D.R., Allwood, J.M., Petavratzi, E., Brown, T.J., Gunn, G., 2015. Mapping the global flow of tungsten to identify key material efficiency and supply security opportunities. Resour. Conserv. Recycl. 103, 19–28. doi:10.1016/j.resconrec.2015.07.003.

Mwanga, A., Lamberg, P., Rosenkranz, J., 2015. Comminution test method using small drill core samples. Miner. Eng. 72, 129–139. doi:10.1016/j.mineng.2014.12.009.

Mwanga, A., Rosenkranz, J., Lamberg, P., 2017. Development and experimental validation of the Geometallurgical Comminution Test (GCT). Miner. Eng. 108, 109–114. doi:10.1016/j.mineng.2017.04.001.

Parker, T., Shi, F., Evans, C., Powell, M., 2015. The effects of electrical comminution on the mineral liberation and surface chemistry of a porphyry copper ore. Miner. Eng. 82, 101–106. doi:10.1016/j.mineng.2015.03.019.

Roy, S., 2009. Recovery improvement of fine iron ore particles by Multi Gravity Separation. Open Miner. Process. J. 2, 17–30. doi:10.2174/1874841400902010017.

Shi, F., Zuo, W., Manlapig, E., 2013. Characterisation of pre-weakening effect on ores by high voltage electrical pulses based on single-particle tests. Miner. Eng. 50–51, 69–76. doi:10.1016/j.mineng.2013.06.017.

Shi, F., Zuo, W., Manlapig, E., 2015. Pre-concentration of copper ores by high voltage pulses. Part 2: Opportunities and challenges. Miner. Eng. 79, 315–323. doi:10.1016/j.mineng.2015.01.014.

Sivamohan, R., 1990. The problem of recovering very fine particles in mineral processing - A review. Int. J. Miner. Process. 28, 247–288. doi:10.1016/0301-7516(90)90046-2.

Sousa, R., Simons, B., Bru, K., de Sousa, A.B., Rollinson, G., Andersen, J., Martin, M., Machado Leite, M., 2018. Use of mineral liberation quantitative data to assess separation efficiency in mineral processing – some case studies. Miner. Eng. 127, 134–142. doi:10.1016/j.mineng.2018.08.004.

Suárez Sánchez, A., Krzemień, A., Riesgo Fernández, P., Iglesias Rodríguez, F.J., Sánchez Lasheras, F., de Cos Juez, F.J., 2015. Investment in new tungsten mining projects. Resour. Policy 46, 177–190. doi:10.1016/j.resourpol.2015.10.003.

van der Wielen, K.P., 2013. Application of High Voltage Breakage to a Range of Rock Types of Varying Physical Properties. Thesis Camborne School of Mine, University of Exeter.

van der Wielen, K.P., Pascoe, R., Weh, A., Wall, F., Rollinson, G., 2013. The influence of equipment settings and rock properties on high voltage breakage. Miner. Eng. 46, 100–111. doi:10.1016/j.mineng.2013.02.008.

Wang, E., Shi, F., Manlapig, E., 2011. Pre-weakening of mineral ores by high voltage pulses. Miner. Eng. 24, 455–462. doi:10.1016/j.mineng.2010.12.011.

Wang, E., Shi, F., Manlapig, E., 2012. Mineral liberation by high voltage pulses and conventional comminution with same specific energy levels. Miner. Eng. 27, 28–36. doi:10.1016/j.mineng.2011.12.005.

Wang, E., Shi, F., Manlapig, E., 2012. Factors affecting electrical comminution performance. Miner. Eng. 34, 48–54. doi:10.1016/j.mineng.2012.04.011.

Zuo, W., Shi, F., van der Wielen, K.P., Weh, A., 2015. Ore particle breakage behaviour in a pilot scale high voltage pulse machine. Miner. Eng. 84, 64–73. doi:10.1016/j.mineng.2015.09.025.

Highlights

- Study of the Electric Pulse Fragmentation (EPF) of a scheelite bearing skarn ore.
- Specific methodology for investigating the effect on comminution and concentration.
- Comparison with a conventional pathway where the EPF was replaced by jaw crushers.

- Improvement of the grindability of the ore with an EPF treatment.
- Increase of the concentrate grade after the gravity separation tests.

Queries and Answers

Query: Your article is registered as a regular item and is being processed for inclusion in a regular issue of the journal. If this is NOT correct and your article belongs to a Special Issue/Collection please contact a.chinnappan@elsevier.com immediately prior to returning your corrections.

Answer: Yes

Query: The author names have been tagged as given names and surnames (surnames are highlighted in teal color).

Please confirm if they have been identified correctly.

Answer: I have updated the surnames of 2 authors.

Query: We find that the role provided for the authors 'K. Bru, R. Sousa, M. Machado Leite, A.B. de Sousa, J. Rosenkranz, D.B. Parvaz' do not match the list of acceptable roles. Please choose a role from the below list for this author: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing - original draft, Writing - review & editing.

Answer: The role of each author is described below:

Kathy Bru: Formal analysis, Investigation, Methodology, Conceptualization.

Mickaël Beaulieu: Investigation, Methodology.

Rui Sousa: Resources, Formal analysis.

Mário Machado Leite: Resources, Formal analysis.

Ana Botleho de Sousa: Resources.

Erdogan Kol: Investigation.

Jan Rosenkranz: Investigation.

Daniel B.Parvaz: Investigation, Formal analysis.

Query: Please check the hierarchy of the section headings.

Answer: I have check it and this is fine for me.