



# Using advanced mineral characterisation techniques to estimate grinding media consumption at laboratory scale

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## ABSTRACT

Grinding media is very important in the mineral processing, mainly because its consumption involves significant costs of grinding and consequently, of the total mining operational expenditure. During grinding, chemical and physical consumptions take place. In both cases, operational variables play an important role but also mineralogy, because many pulp properties depend on it. In the present work, an experimental methodology to study grinding media consumption is proposed, following a geometallurgical approach. The methodology was applied on three Chilean copper sulfide ores to show the kind of results that can be achieved. In the laboratory case study presented, an empirical and predictive model of grinding media consumption as a function of grindability, operational variables (pulp pH and grinding time) and mineralogy, was obtained with the aid of advanced mineral characterisation techniques such as reflectance spectroscopy, X-ray diffraction and portable X-ray fluorescence. The results show that pH has an important effect on grinding media consumption, which may be due to the opposite relation between corrosion and an alkaline environment. Grindability also affects the grinding media consumption, but is related to a physical mechanism. It was found that the overall mineralogical composition and grindability of the ore may change the rate of grinding media consumption, which is greater for hard samples with highly reactive sulfide minerals. However, the highest consumption was obtained in samples with more sulfide minerals.

## 1. Introduction

Grinding is highly used in copper mineral processing to reduce particle size. Generally, grinding is the most expensive unit operation because of the high supplies consumption, representing approximately between 30% and 50% of overall operational expenditure (OPEX) of a mining project (Moema et al., 2009). The most important supplies in wet grinding are water, energy and steel, both for mill liners and grinding media. While water cost can be minimised through improvements in solid-liquid separation and water recovery, the energy cost is heavily dependent on the countries power supply system. Also, the kind of steel for lining depends on the grinding type, and it is selected to reduce the maintenance time and its cost (Wills and Napier-Munn, 2006). For this reason, operational cost associated with grinding media is greater than the cost associated with liners (Lynch, 2015; Wills and Napier-Munn, 2006).

Grinding media consumption occurs by impact, abrasion and corrosion, the latter being predominating in wet grinding, reaching between 10% and 50% of the overall steel grinding media consumption

(Radziszewski, 2002; Aldrich 2013). Impact and abrasion are considered physical consumption and aspects such as mineralogy, mineral hardness, grindability, abrasive properties, rheology, among others, are relevant in this mechanism (Aldrich, 2013). On the other hand, corrosion is defined as mass loss by oxidative electrochemical reactions. In wet grinding, wear by corrosion occurs because there is a difference of electrochemical standard potentials between steel and sulfide minerals, where the grinding media acts as the anode and the mineral acts as the cathode (Gu et al., 2004). In anode, iron from steel is oxidised to ferrous form, while in the cathode, the dissolved oxygen is reduced to hydroxyls ions (Gu et al., 2004; Aldrich, 2013), as is shown in Eq. (1). A relevant aspect to be considered is that normally all wear mechanisms occur at the same time and there is a synergy between physical and chemical wear, so the overall grinding media consumption is not the direct addition of physical and chemical wear (Peres Massola et al., 2016).



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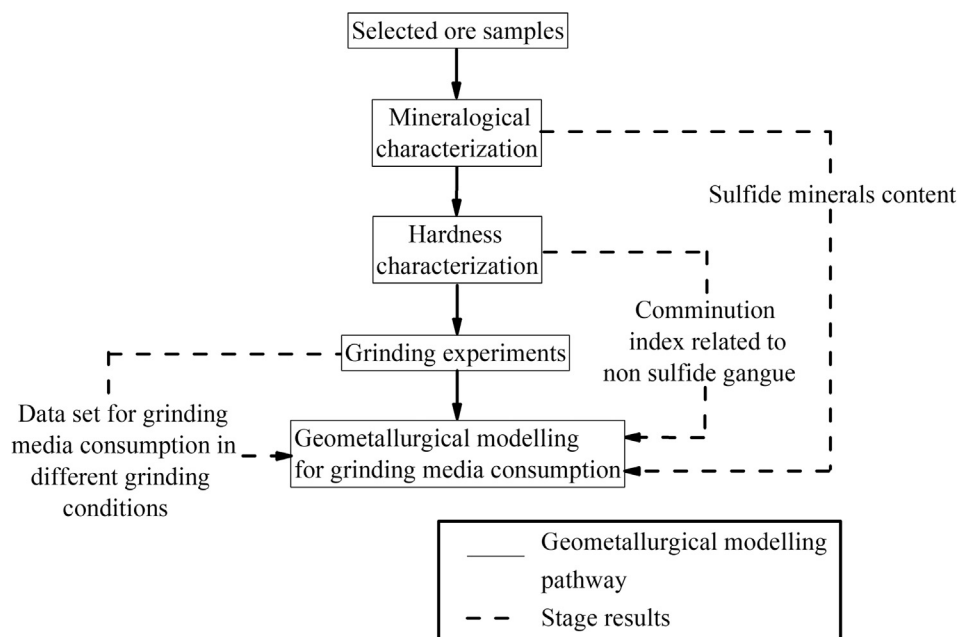
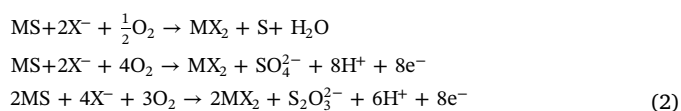


Fig. 1. Proposed pathway to develop a geometallurgical model of grinding media consumption from laboratory experimental tests and characterisation.

Corrosion depends on the kind of sulfide minerals present during grinding (Gu et al., 2004). It has been demonstrated that some sulfide minerals are more reactive than others, and when they are interacting with other sulfide minerals, they usually act as anodes. In this respect, pyrite has been recognised as one of the most reactive sulfides, so the reaction explained in Eq. (1) is more aggressive when it is promoted by pyrite (Pozzo and Iwasaki, 1989). Grinding media losses by corrosion could be the same in quantity even though they are promoted by different sulfides minerals, but their kinetics will be different and in particular, the loss rate will be slower in comparison to that generated by pyrite (Gu et al., 2004).

Grinding media consumption by corrosion plays an important role in mineral processing because there is evidence that this wear mechanism generates hydrophilic products on the mineral surfaces, inhibiting the flotation of some of them (Huang et al., 2006; Bruckard et al., 2011). Besides this, corrosion has a negative impact on the collector adsorption in minerals (electrochemical adsorption), because it promotes the precipitation of iron oxy-hydroxide species and reduces the dissolved oxygen and the pulp chemical potential (Huang and Grano, 2006; Hu et al., 2009). Eq. (2) shows the most important overall reactions between thio-collectors and sulfide minerals, where  $X^-$  represents a thio ion and MS is a sulfide mineral. Electrochemical reactions with grinding media are not the only reactions occurring in the process. Galvanic interactions between mineral species also occur (Gu et al., 2004). Therefore, all different types of electrochemical reactions must be considered simultaneously since the resulting pulp chemical potential depends on the overall reaction (Aldrich, 2013).



Some authors (Aldrich, 2013; Chen et al., 2006; Azizi et al., 2013) have established that there are many factors affecting the grinding media consumption, mainly associated to operational variables, such as rotation speed, grinding media load, solid content, pH, grinding time, pulp rheology, among others. Chen et al. (2006) presented a model based on aforementioned variables for total wear rate in phosphates grinding. Azizi et al. (2013) obtained models based on the same variables for corrosive wear in a copper minerals grinding process, using

alloy steel media and high carbon chromium steel media. In both cases, the authors agree that the pH is one of the most relevant variables affecting grinding media consumption.

Steel consumption is important regarding costs as well as flotation performance. It has been demonstrated that the greater grinding media consumption, the lower floatability of different sulfide minerals, due to changes in physicochemical conditions in the grinding environment. This affects the collector-sulfide mineral interactions by a decrease in the pulp chemical potential and dissolved oxygen (Huang et al., 2006; Greet et al., 2004; Gonçalves et al., 2003; Erdemoğlu and Sarikaya, 2002). Therefore, there is a need to either monitor or model steel wear through predictive equations, as proposed by Chen et al. (2006) and Azizi et al. (2013). However, in previous works mineral and mineralogical properties were not considered, although it is known that they can affect grinding media consumption through modifications in the intensity of wear mechanisms (Aldrich, 2013). In this work, an empirical methodology with a geometallurgical approach to study the overall grinding media consumption is proposed and it is applied in a case study based on Chilean rocks samples associated to copper sulfide deposits, using a rods laboratory mill and advanced characterisation techniques to describe and quantify mineralogy phases in the samples. The advantage of performing a study following a geometallurgical approach is that an exhaustive mineralogical and geological description allows reducing the risk in the operation related to geological variation in the ore body, being the first stage to develop an accurate mathematical descriptor of a certain phenomenon in order to interpolate to the complete orebody (Coward et al., 2009; Lamberg et al., 2013; Lund et al., 2015).

## 2. Experimental procedure

### 2.1. Methodology for grinding media consumption study

Fig. 1 shows the proposed pathway to develop a geometallurgical study for grinding media consumption from an experimental basis. The first stage in the methodology considers an exhaustive mineralogical characterisation both for sulfide minerals, due to their great influence in electrochemical reactions, and for non-sulfide gangue minerals (mainly silicates), because of their high hardness and abrasive power. In this step, mineralogical characterisation may be performed with

different techniques (e.g., QEMSCAN, XRD, XRF, among others) but it is suggested that combinations of them are used to compare and validate results.

The second step in the methodology is obtaining some grindability or hardness indicator for the studied samples because they could be related to physical wear and can contribute or modify this kind of consumption since changes in abrasive conditions or resistance in energy transfer processes (e.g., impact). Also, as proposed by Hunt et al. (2013), some comminution indices can be well estimated using mineralogical and geological parameters (BWi, Axb, SPI, etc.), especially non-sulfide gangue. Then, it is possible that a comminution index may summarise the non-sulfide gangue content through mathematical relations. In this regard, it is important to have information about the grindability and the correlation with mineralogy, so that the grindability may be used with modelling purposes instead of the content of different mineral families or single mineral quantities.

The following stage in the methodology is the experimental one. A series of experimental grinding tests must be performed under specific conditions (grinding time, grinding pH, pulp chemical potential, grinding media materials, solids content, among others). These experiments allow obtaining a dataset for the dependent variable (grinding media consumption) as a function of the independent variables (mineralogy, grindability and operational variables). Then, an analysis can be performed where the grinding media consumption can be explained by the selected variables. Also, a mathematical and empirical predictive model can be obtained by applying modelling or machine learning tools. In this case, it is essential to clarify that a mathematical model is a consequence of the overall methodology application and it is not an objective on its own.

Below, an example of the application of the proposed methodology is shown. In this particular case, the pathway was applied to three ore samples from Chilean copper deposits, and a mathematical and predictive model was obtained as a result of the overall study.

## 2.2. Case study

### 2.2.1. Samples, reagents and mechanical preparation

Three samples of ores (called Mineral A, Mineral B, and Mineral C) from different deposits and with varying mineralogy were obtained from Chilean copper deposits. Each ore sample was individually crushed 100%–3350  $\mu\text{m}$  according to ASTM E 11-01 standard (ASTM International, 2001) and homogeneously split with a rotatory sample splitter, to obtain subsamples of 1 kg.

Bond Ball Mill Work Index tests and standard grinding tests were performed individually for each ore type. Grinding experiments were performed at four times (2, 4, 8 and 16 min) and two different pH (7.5 and 12). Sodium hydroxide was used to modify the pH.

### 2.2.2. Characterisation techniques

The mineralogical characterisation of each sample was performed using hyperspectral technology (Hylogger3™ system) and X-ray powder diffraction (XRD; Rigaku Miniflex 600). Hyperspectral characterisation was applied through Hylogger3™. This system has 531 channels for VNIR – SWIR range (380–2500 nm), 341 channels for TIR range (6000 – 14500 nm) and a high-resolution camera. The analysis was performed on hand rock samples because reflectance spectroscopy in Thermal Infrared range has a better performance when the porosity of samples is almost non-existent. In this sense, many hand rock samples of each ore were selected before comminution processes and mechanical preparation. To select these samples, each ore was homogenised and divided into subsamples. Finally, one hand rock sample was selected randomly from each subsample. All faces in each sample were analysed to obtain representative results. The obtained spectra were analysed in The Spectral Geologist software (TSG) (Schodlok et al., 2016).

The XRD analysis was performed with an emphasis on determining the main ore and gangue minerals detected by hyperspectral

characterisation. The Rigaku Miniflex equipment used for XRD analysis has a copper and a cobalt anode, the latter specially designed for clays detection. The analysis was performed in duplicate, selecting a sample from each ore type following a sampling protocol to obtain a representative sample, both in particle size and mineralogical terms. This sample was pulverised to obtain 100%–75  $\mu\text{m}$  according to ASTM E 11-01 standard (ASTM International, 2001). The results of the XRD were analysed using the PDXL software (Rigaku).

Finally, to validate mineralogical results, X-ray fluorescence was applied using an Olympus Delta Premium portable equipment (pXRF) in representative samples from each ore, following the same sampling protocol and criterions used for XRD. The elemental quantification obtained was corrected using rocks standards previously characterised by chemical analysis and provided by ORE: Ore Research and Exploration Company (ORE, 2017).

The results precision and validation method differ for each technique. In hyperspectral characterisation, the equipment performance is validated by a QA/QC process, where specific features in the spectra of certain minerals of interest in each range are analysed (Schodlok et al., 2016). In VNIR range, the iron transitions are common, so iron oxides and hydroxides are analysed by studying the presence of goethite and hematite. In SWIR range, hydroxides are well recognised, so aluminium hydroxides are analysed by studying the presence of kaolinite. Finally, tectosilicates are well recognised in TIR range, so the same analysis is performed using quartz as a representative mineral. In all cases, the absorption peak depth in the minimum reflectance wavelength is compared to the wavelength, and the analysis is validated if the points show a peak at a specific wavelength.

In XRD the validation of the Rietveld Refinement is given by the statistical parameters  $R_{wp}$  (standard residual of least squares) and  $S$  (quotient between  $R_{wp}$  and simple standard residual,  $R_p$ ).  $R_{wp}$  has to be lower than 10%, and  $S$  has to be close to 1. Finally, each element given by pXRF has a limit of quantitation (LOQ), which is calculated statistically from analysis of the results when the assay is performed at least in duplicate (Kadachi and Al-Eshaikh, 2012). If the quantity obtained by pXRF is greater than LOQ, then the results are considered adequate if it was properly corrected using known standards. It is relevant to mention that LOQ is different to limit of detection (LOD), which is related to the lowest analyte concentration that can be recognised in comparison to a blank sample that contains the analyte, while LOQ is the lowest concentration that the analyte can be detected meeting some predefined goals for bias and imprecision. For this reason, LOQ is higher or equal to LOD.

### 2.2.3. Grindability characterisation

To obtain the grindability, Bond Ball Tests were performed on each sample. In this case, the energy consumption expressed by Bond Work index (BWi) was used as a grindability indicator (Bond, 1968).

### 2.2.4. Grinding experiments

Grinding experiments were performed in a laboratory forged steel rod mill, using 1 kg of mineral sample, a solids content of 70 wt% and a grinding media charge of 16.18 kg, in a specific distribution (5 rods of 1404.23 g each, 4 rods of 627.78 g each, 4 rods of 438.94 g each and 18 rods of 271.91 g each). The liquid solution to achieve such solids concentration was Santiago tap water (natural pH 7.5) and a solution of tap water and sodium hydroxide (pH 12). After each grinding test, the ground material was dried, and its particle size distribution was obtained through screening. Finally, the grinding media consumption was obtained by measuring the rods mass loss. For this, before and after each experiment, the rods were washed, dried and their mass was registered to a precision of 0.01 g. The overall consumption was normalised by the ground mass.

Finally, the dependent variable (grinding media consumption) is linked to the following variables, which will be considered as explicative variables in an empirical context: grinding time, pulp pH,

**Table 1**  
Results of bond work index of samples A, B and C.

BWi Mineral A [kWh/t]	BWi Mineral B [kWh/t]	BWi Mineral C [kWh/t]
16.46	8.36	16.23

**Table 2**  
Minerals detected by hyperspectral characterisation for Samples A, B and C.

Minerals on Sample A	Minerals on Sample B	Minerals on Sample C
Gypsum (Anhydrite)	Dolomite	Gypsum
Siderite	Calcite	Phlogopite
Chlorite	Siderite	Chlorite
Phengite	Pyrophyllite	Muscovite
Muscovite	Illite	Illite
Montmorillonite	Augite	Oligoclase
Oligoclase	Labradorite	Albite
Albite	Anorthite	Orthoclase
Quartz	Albite	Microcline
Biotite	Orthoclase	Quartz

overall sulfide content and BWi.

### 3. Results

#### 3.1. Characterisation

##### 3.1.1. Grindability characterisation

Table 1 shows the grindability of each sample expressed as Bond Work index, obtained by the application of Bond Ball Test.

##### 3.1.2. Mineral characterisation

3.1.2.1. *Hyperspectral characterisation.* Table 2 shows the mineral species detected by hyperspectral characterisation for Minerals A, B and C. The experimental spectra obtained were compared with reference spectra for known minerals, registered in the TSG software to recognise the main non-sulfide gangue minerals in the samples. Mineral A and C showed a high presence of high hardness silicates and phyllosilicates, while Mineral B showed the presence of carbonates, although tectosilicates and phyllosilicates were also recognised. It should be noted that Hylogger3™ only gives qualitative information about recognised minerals.

3.1.2.2. *X-ray fluorescence.* Table 3 shows the quantitative elemental composition of samples A, B and C obtained by pXRF. The raw data were corrected using known standards of rocks (OREAS 151b–153b and OREAS 501b–504b) (ORE, 2017) and the analyses were performed in

**Table 3**  
Quantitative elemental composition of Minerals A, B and C by X-ray fluorescence.

Element	Mineral A [wt%]	LOQ A [wt%]	Mineral B [wt%]	LOQ B [wt%]	Mineral C [wt%]	LOQ C [wt%]
Mg	1.60	0.43	1.48	0.22	1.57	0.19
Al	5.61	0.27	6.65	0.22	7.29	0.24
Si	26.62	0.26	7.18	0.09	25.03	0.27
P	0.08	0.01	0.08	0.02	0.08	0.01
S	4.14	0.05	7.21	0.07	2.74	0.06
K	2.81	0.04	2.63	0.07	5.31	0.05
Ca	3.48	0.10	2.90	0.06	3.34	0.11
Mn	0.03	0.01	0.03	0.00	0.01	0.00
Fe	3.48	0.14	3.84	0.02	0.90	0.06
Cu	0.51	0.02	19.65	0.01	3.95	0.01
Zn	< 0.1	0.00	< 0.1	1.2.E–03	< 0.1	0.00
As	< 0.1	7.9.E–04	< 0.1	1.05.E–03	< 0.1	6.08.E–04
Mo	< 0.1	6.9.E–04	< 0.1	2.62.E–04	< 0.1	3.15.E–04
Pb	< 0.1	0.00	< 0.1	0.00	< 0.1	0.00
Others	51.64	–	48.33	–	49.75	–

**Table 4**  
Statistical errors for Rietveld refinement.

Parameter	Mineral A	Mineral B	Mineral C
R <sub>wp</sub> [%]	8.79	4.77	8.15
R <sub>p</sub> [%]	5.22	3.58	5.96
S	1.68	1.33	1.37

triplicate, so the LOQ could be calculated as is explained by Kadachi and Al-Eshaikh (2012). From the results, it can be noticed that all elements except arsenic values can be used because its LOQ is lower than the obtained content in samples B and C.

3.1.2.3. *X-ray diffraction.* Quantitative mineralogy was obtained by applying Rietveld Refinement to diffractograms for each rock type, where hyperspectral results (qualitative mineralogical results) were used as reference information to select mineralogical phases for the refinement process. Also, the sulfide mineral phases were selected by visual inspection of hand rock samples, using diagnostic properties of sulfide mineral (crystalline shape, brightness, colour, stripe, hardness, exfoliation, specific gravity) (Tarbuck and Lutgens, 2006). Finally, they were added to the dataset of mineralogical phases to apply the refinement.

The statistical parameters to validate the Rietveld Refinement are presented in Table 4, and quantitative results for mineralogy are shown in Table 5. As it is shown in Table 4, good statistical parameters were obtained using hyperspectral results, demonstrating the good correlation between both mineralogical characterisation techniques.

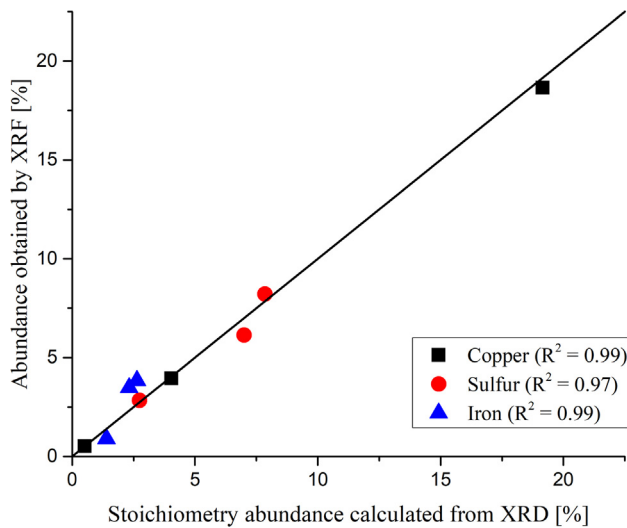
3.1.2.4. *Mineralogical results validation.* The XRF results were used to validate the mineralogical quantification, focused on the sulfide content to use these values as an input in the mathematical model. Fig. 2 shows the comparison between elemental quantity for Cu, Fe and S (contained in the sulfide minerals) measured by pXRF and that obtained by stoichiometry using XRD results. The good correlation in results for copper and sulfur (see R<sup>2</sup> in Fig. 2) validates the quantitative mineralogy. In the case of iron, it must be noticed that a high quantity can be contributed by phyllosilicates (e.g., biotite). Therefore, pXRF results for iron are underestimating the real content in ore samples.

##### 3.1.3. Grinding kinetics

The results of grinding experiments are presented in Fig. 3 (grinding media consumption). It should be noted that there is not a uniform behaviour in all cases, but each sample presented a single behaviour which was modified with pH. Also, for Mineral A the media consumption increased up to 8 min and then it was almost constant with

**Table 5**  
Quantitative mineralogy of minerals A, B and C by X-ray diffraction.

Mineral	Mineral A [wt%]	Mineral B [wt%]	Mineral C [wt %]
Quartz	22.4	0.7	20.0
Albite	11.4	5.4	12.9
Biotite	20.6	–	0.2
Chalcocite	–	9.8	2.7
Phlogopite	21.2	3.1	2.5
Phengite	2.0	–	2.3
Bornite	–	16.3	0.1
Chalcopyrite	1.5	2.7	2.0
Pyrite	4.0	–	–
Molybdenite	–	–	0.5
Muscovite	4.1	3.6	2.4
Galena	–	–	0.4
Illite	–	0.9	5.9
Sphalerite	–	–	0.7
Covellite	–	0.1	1.7
Calcite	–	12.1	–
Montmorillonite	4.5	–	1.6
Orthoclase	–	21.2	15.0
Plagioclase	–	2.1	16.3
Gypsum	3.4	0.6	2.3
Kaolinite	–	–	1.9
Chlorite	1.6	3.7	2.5
Nitranite	–	6.8	–
Antigorite	–	4.5	1.1
Barite	–	4.3	–
Others (phyllosilicates and zeolites of low content)	3.3	2.1	5.0

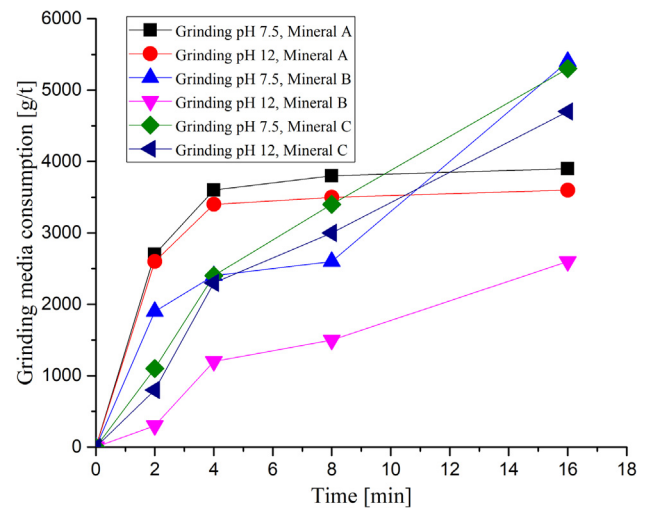


**Fig. 2.** Validation of quantitative XRD using pXRF. The elemental quantities of some elements obtained by different mechanisms are compared.

time, while for Mineral B and C the consumption increased constantly with time.

**3.2. Mathematical modelling for grinding media consumption**

A mathematical and empirical model is proposed considering the studied variables in this work. To obtain a statistically appropriate mathematical model, a series of machine learning predictive algorithms (simple linear regression, polynomial regression and multiple linear regression) were performed in Stata software (StataCorp, 2007) considering directly the grinding media consumption and also its value in logarithmic terms (experimental training dataset). To discard variables for modelling, the backward methodology (Hosmer et al., 2013) was applied on *t*-Student statistical obtained for each parameter to be included in the equation in order to have only significant and explanatory



**Fig. 3.** Grinding media consumption for Minerals A, B and C as a function of the grinding time and the grinding pH.

variables in the model. The backward methodology is an iterative process that considers a first mathematical model including all parameters and the less explanatory is discarded in each iteration until to have only significant variables in terms of some appropriate statistical. It has to be noted that the parameters obtained through statistical analysis would not necessarily be related to the observed effects (phenomenology), so it is expected that some parameters in the model cannot be explained from a phenomenological point of view. In fact, machine learning algorithms point to obtaining the best combination of independent variables to describe dependent variables, regardless of physical causality.

Both for direct values of grinding media consumption and for logarithmic values, a linear and a quadratic model were obtained. All models were evaluated with Corrected Akaike’s Information Criterion (AICc) and Bayesian Information Criterion (BIC) (Schwarz, 1978; Kass and Raftery, 1995), which rate the model better if it presents a good fit and punish the model if it has too many variables. The best model will have the minimum AICc and BIC, which means that the model can explain the phenomenon satisfactorily using the least possible number of variables.

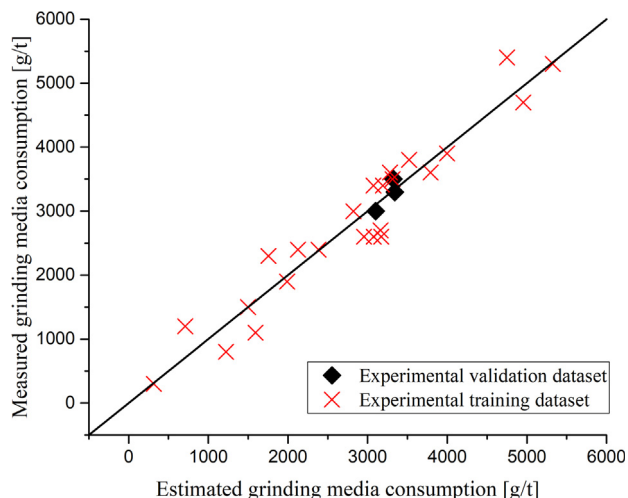
The variables considered for the model were grinding time, BWi, sulfide content (wt%), grinding pH and their combinations to obtain quadratic explanatory variables. Table 6 shows the results of AICc, BIC, cross-validation error (leave one out cross validation) and adjustment R<sup>2</sup>. The results showed that the best model considering all criteria was the quadratic model. Finally, the regression model was validated using experimental results (experimental validation dataset), applying different grinding conditions to Minerals A, B and C. Table 7 shows the experimental conditions and the obtained error after model application. The adjustment of this model and its experimental validation is presented in Fig. 4. The experimental model for grinding media consumption (G.M.C.), in g/t, is presented in Eq. (3).

**Table 6**  
Criteria of model selections.

Model	BCI	AICc	R <sup>2</sup>	Cross Validation Error
Linear	395.20	395.82	0.71	–0.19
Quadratic	374.09	379.53	0.93	–0.64
Log – linear	412.85	413.47	0.50	–0.19
Log – quadratic	385.71	394.03	0.88	–0.77

**Table 7**  
Experimental conditions to validate the mathematical model.

Mineral	pH	Grinding time [min]	Error [%]
A	10	12	5.02
B	10	14	−3.44
C	9	10	−1.28



**Fig. 4.** Grinding media consumption model. Predictive and real values for grinding media consumption are shown using the experimental training and validation datasets.

$$\begin{aligned}
 \text{G.M. C.} = & -5220.55 \times t + 1.56 \times \text{pH}^2 + 28.45 \times \text{BWi}^2 + 18.14 \times \text{wt\%Sulf}^2 \\
 & + 285.78 \times \text{BWi} \times t - 52.23 \times \text{BWi} \times \text{wt\%Sulf} \\
 & + 104.80 \times t \times \text{wt\%Sulf} - 13.92 \times \text{pH} \times \text{wt\%Sulf} \quad (3)
 \end{aligned}$$

## 4. Discussion

### 4.1. Characterisation

#### 4.1.1. Mineral characterisation

The purpose of this study was to propose a methodology to evaluate the grinding media consumption using experimental information following a geometallurgical approach. In the case study, a new technology for mineral characterisation (Hylogger3™) was applied as a complement of traditional techniques (XRD and pXRF). X-ray diffraction is a technique which needs a high level of knowledge of the sample to have a good adjustment and a correct Rietveld refinement. This could be more complicated in mineral samples because there are many different mineral phases contained in the sample, and therefore, there are many peaks to identify and assign. Also, even if the focus is the sulfide content, non-sulfide minerals have to be clearly identified and incorporated to the Rietveld algorithm, so that an accurate diffractogram modelling can be performed and mineralogical quantification is more reliable.

To obtain diffractograms with a low level of noise, a step of 1 degree per minute was used and the Hylogger3™ results were used as initial information about mineral phases in each sample. An aspect to remember in hyperspectral characterisation is the problem to recognise sulfide minerals because their characteristics spectral features are recognised in the far infrared range (Soong and Farmer, 1978). However, by simple inspection of the samples it was possible to identify the most important sulfides in each sample. Therefore, other added phases were selected by diagnostic properties of hand rock samples (sulfide minerals) and from the same family than those recognised by hyperspectral analysis (non-sulfide mineral).

Hyperspectral characterisation demonstrated to be an appropriate

technique to acquire qualitative mineralogical information in a fast and non-destructive way, which could be corroborated by the statistical parameters of Rietveld refinement (Table 4). Statistical results accounted for a profitable process of adjustment with the dataset of mineralogical phases selected. Results in hyperspectral techniques are directly influenced by the type of analysed sample. In this case, the non-sulfide gangue minerals were well identified in each rock sample because all faces in the samples were analysed, giving the possibility to less abundant minerals to be characterised. Hyperspectral analysis through Hylogger3™ has many advantages. It does not need an exhaustive sample preparation (samples have to be free of moisture and dust), the analysis is fast in comparison to other techniques (1 min per meter of samples linearly disposed) (Schodlok et al., 2016), the analysis is non-destructive and the TSG can give information on site. Therefore, according to results, it was completely beneficial the joint application of hyperspectral characterisation and XRD, since the speculation and uncertainty on the present crystalline phases were reduced, and there was an adequate starting point for the refinement without spending large quantities of time and resources.

From the application of mineralogical techniques, it can be noticed that there is a good correlation between the XRD mineralogical quantification results and the minerals identified by hyperspectral characterisation using Hylogger3™. For Minerals A and C, the main minerals were quartz, albite and other different silicates (phyllosilicates and tectosilicates), while for Mineral B, there is a high presence of carbonates, and some tectosilicates and phyllosilicates were recognised. Using the overall information obtained by inspection and hyperspectral characterisation, the main peaks in the diffractogram were easily recognised knowing in advance some minerals with a high probability to be present in the sample. Finally, the elemental quantification obtained with pXRF validates the mineralogical quantification, because it was observed a clear correlation between quantity of Cu, Fe and S detected for the three samples using pXRF and the values obtained by stoichiometry based in the quantitative XRD (see Fig. 2).

#### 4.1.2. Grindability characterisation

Regarding the grindability, a relation between mineralogy and grindability can be proposed. The high BWi values for Minerals A and C might be related to the high abundance of quartz and other tectosilicates, which are minerals of high hardness (6–7 in Mohs scale), while for Mineral B, the lower value of BWi might be related to the lower hardness of carbonates and sulfides (3–4 in Mohs scale). However, more samples must be analysed to check the correlation. In the case of Mineral A, it should be noted that the presence of phyllosilicates, like biotite and phlogopite, also can have an important effect in grindability due to these minerals present a high resistance to reduce their size through breakage mechanisms (impact, abrasion, attrition and compression) because of their laminar structure.

However, although a comminution index can summarise the non-sulfide gangue composition of a rock sample, not only the mineralogy is important to explain the grindability because it is possible to find the same minerals in similar quantities in two samples, but they could have different grindability. In the analysed case, Mineral B contains a large quantity of orthoclase, but its grindability is much lower than Minerals A and C (which also have important quantities of tectosilicates). This could be related to differences in grain space arrangements, grain size or shape and the presence of structures that favour the breakage. All mentioned aspects are incorporated in the geological texture concept. Hunt et al. (2013) reported that mineralogy could explain BWi and other comminution indices, as a first approach, but textural geology must be considered to have a better description of grindability. However, for the current geometallurgical modelling, considering BWi as a grindability indicator is a good way to summarise the content of non-sulfide minerals.

#### 4.1.3. Grinding kinetics

Results for grinding media consumption showed a clear dependence with time (Fig. 3). However, each sample had a particular behaviour. Mineral A reaches a plateau for steel consumption, whereas Minerals B and C showed a lower rate of grinding media consumption, without reaching a plateau for the times tested in this study. The high grindability of Mineral A and the sulfide composition strongly influenced by the presence of pyrite, generate a fast increase of grinding media consumption at short grinding times, represented by the major slope of the curve in Fig. 3 (high increase rate), while the low grindability and the presence of less reactive sulfides in Mineral B generate a lower rate of consumption. A similar explanation could be given for the behaviour observed for Mineral C, which also had a slower increase for grinding media consumption than Mineral A. In this particular case, although grindability and gangue mineral composition for Mineral C were very similar to Mineral A, the higher content of sulfide minerals could be related to the greater values for grinding media consumption at longer grinding times. This leads to suppose that the dominant consumption mechanism (chemical/physical) can vary on time. However, this cannot be proved from the results herein presented and the effect of each mechanism should be isolated to verify its variation in time.

During grinding media consumption, physical wear can be modified by changes in pulp flow and abrasive properties, which may be related to the presence of fine particles and clay minerals (Tangsathikulchai, 2003; Aldrich, 2013). In the case of corrosion, a change in grinding environment could promote less corrosion, for example, surface passivation after a certain grinding time or the dissolved oxygen consumption by accelerated initial galvanic interactions with pyrite. On the one hand, although sulfide minerals promote the chemical consumption of steel, without the presence of high chemically reactive minerals, a lower increase rate for consumption at short grinding times would be expected (Gu et al., 2004). In difference to Mineral A, sample B is composed of 7 different sulfide minerals less reactive than pyrite and of low hardness non-sulfide gangue minerals, being a less abrasive sample. According to results, in Sample B the aforementioned properties combination promotes a low grinding media consumption at short grinding times, while at longer grinding times the effects are more evident (greater values for consumption were observed). Therefore, the combination of different properties (hardness, grindability, types of minerals, presence of sulfide minerals, among other) determines the rate and magnitude of grinding media consumption. For this, consumption promoted by Mineral A is faster than Mineral B (Sample A reaches quickly high values and the plateau), but the final effect is greater in Mineral B (properties in Sample B promotes a higher magnitude in grinding media consumption at longer grinding times).

Mineral C is an intermediate case between Samples A and B, because of its grindability and sulfide content. It was observed that the grinding media consumption was not the same in different grinding intervals: at short grinding times the increase rate for steel consumption is lower than in the other mineral samples, but at longer grindings, the rate changes and the overall grinding media consumption increases faster than at the beginning. However, longer times are required to reach a plateau for grinding media consumption. As an intermediate case, it would be expected that the grinding media consumption increase rates and values were in the middle of samples A and B. However, this was not observed for all evaluated times. Main difference between Mineral A and C is the sulfide mineralogy: the consumption by corrosion in sample C is not as fast as in the case of Mineral A. Then, as with Mineral B, the longer the grinding time, the greater the effects of grinding media consumption, but in this case also the high grindability must be considered as an important contributor to consumption, so a greater increase rate is obtained for Mineral C at longer grinding times.

Grinding media consumption is a phenomenon with two main mechanisms (physical and chemical wear), but they are acting at the same time, being possible a synergism between them. During grinding, at any moment there will be a contribution from physical and chemical effects

to steel consumption. Also, as it was previously reviewed, mineralogical properties may have an important role in both mechanisms. According to results, minerals with less reactive sulfides than pyrite (only identified in Mineral A) and a low grindability will have low increase rates for steel consumption and the effect of a higher overall presence of sulfides will promote a higher rate at longer grinding times, due to the wear mechanism (chemical plus physical wear) will be slower but greater.

According to results, pH has an important role in grinding media consumption. It was observed that the higher the pH, the lower the steel consumption, as presented in Fig. 3. The results of this work agree with those reported by Chen et al. (2006) and Azizi et al. (2013), where the chemical consumption was decreased in the alkaline grinding environment. On the one hand, the behaviour of the grinding media consumption with pH is related to  $H^+$  ions availability. At low pH values, there is a larger availability of  $H^+$  ions, which reduces the probability of forming a passivating layer on the surface of the grinding media. Therefore, promoting the active dissolution of the steel together with the reduction of hydrogen ions to hydrogen gas (Davis, 2000). The previous mechanism is added to the grinding media – sulfide interaction, having higher values for grinding media consumption with a low pH, as could be experimentally demonstrated (Fig. 3). On the other hand, with an alkaline environment, due to the larger presence of  $OH^-$  ions, it is probable that the iron from grinding media will form iron hydroxides ( $Fe(OH)_3$  and  $Fe(OH)_2$ ) in the surface of the grinding media (Davis, 2000), consuming a part of the dissolved oxygen through the reduction reaction. Then, as explained by Davis (2000), with high values of pH (higher than 9) a passivating layer made of iron hydroxides is formed in the surface of the forged steel, and this layer would be able to protect the media from corrosion, both by the compounds precipitation and by the decrease in dissolved oxygen availability.

An additional aspect related to iron hydroxides formation is the consequent effect in sulfide surface and the later effect in flotation. Iron hydroxides can be adsorbed and/or precipitated in sulfide minerals surface, creating a hydroxide layer and changing the sign of zeta potential (from negative to positive). In general, the hydrophobicity of sulfide minerals can be related to the surface oxidation and the formation of a sulfur-rich surface. This process is reversed by the hydrophilic metal hydroxides precipitation, resulting in the depression of sulfide mineral (Fairthorne et al., 1997; Peng et al., 2003). The level of flotation depression will depend on the reactivity of sulfide surface. The nobler the sulfide, the greater the flotation depression (Peng and Grano, 2010).

#### 4.2. Mathematical modelling for grinding media consumption

As shown by the experimental results of this work, it is possible to explain the grinding media consumption using the following studied variables (identified as independent variables): grinding time, pulp pH, BWi and sulfide content. Then, a predictive model based on a geometallurgical approach is useful to quantify the consumption under specific conditions, considering in all moment that the training set to obtain a mathematical model is specific to the evaluated samples. Indeed, according to the application of geometallurgy, every geometallurgical model will be specific for a well delimited geological or geometallurgical context. Therefore, the application of the proposed methodology will always be framed in the analysed samples, being site specific (Vann et al., 2011).

The proposed model considers operational variables (pulp pH and grinding time) and mineral properties (mineralogy and grindability). It is necessary to have appropriate measurements for each of them to generate the best estimator. While pulp pH, grinding time and grindability can be measured using relatively simple techniques, technologies or standard protocols, mineralogy has many options to be defined. The most typical techniques in mining industry to evaluate mineralogy are electron microscope techniques (SEM, TEM, QEMSCAN, among others), which gives relative abundances between identified minerals,

sometimes needing some calibration parameters to it (reference information obtained by other techniques); and X-ray techniques, mainly X-ray diffraction, which allows quantifying mineralogical phases by standards addition or by diffractogram modelling, that is more appropriated when the sample has many phases (Mukherjee, 2012). Both techniques need previous knowledge of the sample to obtain accurate results, even if a specific kind of mineral is looked for (Kolka et al., 1994; Mukherjee, 2012). In the current work appropriate results were obtained for mineralogy in the three samples since the high utility of reflectance spectroscopy through Hylogger3™. However, this technology does not allow obtaining quantitative mineralogy, becoming a good support technique to give reference information for other methods.

It is important to mention that the terms composing the model equation, for example, content sulfide multiplied with pH, might not make physical sense, but they make an effort to have a better fit and a low adjustment error. From the theory of grinding media consumption, it is known that consumption is a contribution from physical and chemical mechanisms (and their combination) (Aldrich, 2013). Therefore, doing a comparison with the model, it is possible to observe that not all consumption expressed in the singular terms  $BW_i^2$  and  $wt\%Sulf^2$  come from the respective variable, but also from the synergy between them. This gives a clear example that the machine learning algorithms application is useful under a mathematical viewpoint, but it does not give a correct phenomenological explanation. Finally, the results showed that the highest error from the experimental values was 5% for Mineral A, so it can be considered as a robust predictive model and also the error in cross-validation showed a minimal underestimation.

A relevant aspect which must be considered as a complementary contribution of this work is the industrial applicability of the results. An appropriate understanding of grinding media consumption would allow knowing which will be the most important aspects to be considered during the processing of specific mineral samples to avoid high levels of steel wear and contribute to reducing operational costs. First of all, the proposed methodology can be applied to different grinding types (e.g., balls and rods) and it gives the versatility of relating the phenomena with different operational variables. In the specific case study, a rod mill was used to grind three different samples, which delimited the geometallurgical context. The nature of this system does not allow the general applicability in all grinding cases. Therefore, specific tests and measurements must be performed for each specific case (mill type and grinding media type). However, the presented laboratory application clarified the fact that grinding media consumption can be mathematically related through appropriate models to operational and mineralogical variables. Then, it is possible to estimate the wear in the case of eventual changes both in mineralogical and operational aspects.

## 5. Conclusions

An experimental methodology to study the grinding media consumption following a geometallurgical approach is proposed. The methodology considers an appropriate characterisation of samples (mineralogy, grindability and their relation) and a well-defined set of operational variables to evaluate the phenomena. The procedure can be applied to different mill types, and different variables (mineralogical and operational) can be evaluated to describe and estimate grinding media consumption. Also, diverse technologies and characterisation techniques can be applied to assure a dataset of relevant information to explain the dependent variable. Through a case study at laboratory scale, it was found that machine learning tools allow obtaining an appropriate estimator for steel wear considering operational and mineralogical variables, so operational costs and steel consumption can be estimated under different conditions and eventual changes in a mining operation.

Experiments at laboratory scale showed that different combinations of grindability and mineralogy could modify the grinding media

consumption, under fixed operational variables. Specifically, rocks with less reactive sulfide minerals and with a lower grindability showed a lower increase rate for grinding media consumption. In the opposite case (high grindability and the major presence of high reactive sulfide minerals) a greater increase of consumption was obtained at short grinding times. Also, under these conditions, a plateau for consumption can be quickly reached by accelerated surface passivation or protective layer formation. The most detrimental combination is given by the high presence of sulfide minerals and a large grindability, since wear mechanisms depends on mineral properties and not only on operational variables. To know the concrete effect of this combination, longer grinding times must be evaluated because there was not a plateau during the evaluated times. It was found that grinding pH has a major influence on the overall grinding media wear, which is believed to be due to a reduction of corrosion in an alkaline environment. Therefore, pH can be adjusted as an operational practice to reduce steel consumption, but its relationship with hydroxides formation and its effect on flotation should be considered.

The aim of the proposed methodology is giving enough information to understand which are the most important variables for grinding consumption, but at the same time, clarify that not only operational variables are relevant in this process and that mineral properties must be considered. In the current work a mathematical and empirical model was obtained by applying machine learning tools, using mineralogical composition (sulfide content) and grindability (as an indicator of non-sulfide gangue mineral) as variables for the mathematical model. The proposed model was validated with mathematical tools and experimental results, showing a high accuracy (less than 5% error in experimental validation and a cross-validation error of  $-0.64$ ). However, due to the geometallurgical nature of the methodology, the model is valid only in similar cases than the one evaluated. Each new application of the methodology will have the same particular character for the case studied. The current work must be complemented using different mill types and grinding media, as well as evaluating other relevant variables in wear processes (e.g. rheological or pulp chemical properties).

## 6. Recommendations

To have an accurate approach to the industrial operation, experiments at laboratory scale have to be performed using the same mineral samples that will be treated in the mineral processing plant with the purpose of delimiting and assuring the geometallurgical context. Then, the operational variables to be evaluated must be measurable and controllable at industrial scale (e.g., pulp pH). Finally, scaling parameters must be developed to obtain the industrial response of the studied phenomena. In first place, physical wear is divided into two components: impact and abrasion. While abrasion depends mainly on mineral and pulp properties and can be approximated at laboratory scale through simulating pulp chemical conditions of the industrial mill, impact depends on the energy transferred, which will be highly influenced by the potential energy of grinding media during its fall. Based on this, the first scaling parameter must be related to mill size, which in turn will be related to potential energy.

In second place, the following scaling parameter is proposed to correct the differences between exposed area of grinding media in different scales. It is important to consider the exposed area because corrosion is based on chemical reactions, which are heavily dependent on the available surface area. According to Peres Massola et al. (2016), an accelerated consumption by corrosion depends on a synergism between both consumption mechanisms: first, it is important the amount of area on the grinding media that depassivates at each erosion event and second, it is relevant the quantity of metals from sulfide minerals that have to oxidate the exposed depassivated area in grinding media to generate the consumption by a redox reaction according to Eq. (1). It is suggested that the laboratory equipment is the same type than industrial scale equipment (e.g., ball or rod mill and material of grinding



media) because there are considerable differences in the exposed area between them (Lynch, 2015). Then, the superficial exposed area of grinding media can be calculated from its charge distribution in the mill, so it is possible to correct the model from a comparison between this parameter at different scales.

Finally, the third parameter which may be also important at different scales is the feed particle size distribution because it is also related to the exposed area for chemical reactions. Therefore, it is suggested to do a particle size characterisation at both in industrial and laboratory scales. Then, using the results, it is possible to compare the mineral surface area available at the beginning of the grinding process in different conditions to correct the model.

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