



# The effect of using different comminution procedures on the flotation of sphalerite

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## ABSTRACT

High Pressure Grinding Rollers (HPGR) are known to reduce energy consumption and wear costs and improve the throughput in the circuit. It has been suggested that they can also modify the liberation characteristics of the ore. In the present study the effect of using conventional crushing as opposed to HPGR in combination with either dry or wet milling was investigated using a base metal sulphide, viz. sphalerite, in order to determine whether there may be an improvement in flotation performance following the use of different comminution procedures. It was found that, irrespective of the crushing procedure (HPGR or conventional), dry milling resulted in the highest grades and recoveries of zinc. These were typically 94% recovery at 40% grade. In order to gain an insight into the effect which these comminution procedures had on the ore, samples of feed and product were investigated using surface analytical techniques. Mineralogical analyses showed no differences in the liberation characteristics of sphalerite irrespective of the comminution procedures used. However, dry milling produced a lower d<sub>50</sub>. The paper proposes possible reasons to explain these observations.

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## 1. Introduction

In the preparation of ore for flotation, various grinding procedures can be applied which have the possibility of influencing the flotation performance of the valuable minerals by changing the degree of liberation, the particle size distribution or the surface characteristics of the ore sample. With respect to liberation it has been shown by Wightman et al. (2008), in a study of copper/lead/zinc ore, using MLA to analyze the mineralogy of the samples, that particle composition distribution of a given size fraction from a comminution circuit is constant for that mineral in that ore regardless of whether the particle has been crushed, ground or subjected to impact breakage or compressive breakage and also regardless of the overall size distribution of the comminution product. In that study a rod mill, hammer mill and piston and die were used. However it has been proposed that HPGR as a comminution device not only improves energy efficiency but may also lead to preferential liberation of valuables due to micro-cracking along grain boundaries (Bearman, 2006; Dunne et al., 1996; Morley, 2006; Daniel, 2007). With respect to particle size distribution Klymowsky et al. (2006) proposed that the effect of inter-particle crushing in the HPGR produced more fines and resulted in higher reduction ratios. With respect to the surface properties of the ore particles it is well known that water has a major influence on the galvanic interactions which may occur during the comminution

process. These galvanic interactions that take place within the pulp between different mineral surfaces and between grinding media and mineral surface can affect the flotation of the valuable minerals. In the grinding environment the sulphide minerals behave like cathodes and the steel grinding media like anodes which release iron ions into the pulp. Therefore the grinding environment and media may play a significant role in the flotation recovery and selectivity of sulphide ores. Wei and Sandenbergh (2007), for example, showed that a ceramic mill produces an oxidizing environment, allowing sphalerite activation by copper ions, whilst steel media produces a reducing environment, preventing this action. Seke (2005) observed that the grinding environment (wet or dry milling) can influence pulp potential and subsequently the surface properties of the mineral to be floated, dry grinding resulting in more positive pulp potentials, and the recovery of sphalerite being higher when milled in a dry environment. This paper presents results obtained in a study of the flotation of sphalerite which had been subjected to the different comminution methods as described above, viz. HPGR and conventional crushing, respectively, followed by either wet or dry milling.

## 2. Experimental procedure

### 2.1. Bulk sample preparation

A bulk sample of a base metal sulphide ore was used in this study. It was prepared for the experimental test work by jaw-crushing the bulk sample to obtain a representative sample of size

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<8 mm. This bulk sample was then blended and split into two samples of 100 kg and 50 kg respectively. The 50 kg sample was further crushed to obtain a sample of size <3.3 mm using a cone crusher. This sample was then split into 2 kg and 10 kg aliquots to be used as the feed to the wet and dry rod mills, respectively. The 100 kg sample was fed through the HPGR at 60 bar and then the entire sample again at 90 bar to obtain a sample of size <3.3 mm. This sample was also split into 2 kg and 10 kg aliquots to be used as the feed to the wet and dry rod mills respectively.

Before the ore was fed to the rod mill the sample was screened at 90  $\mu\text{m}$  and the +90  $\mu\text{m}$  size fraction was fed to the mill. The milling time was determined to ensure that the reconstituted feed to float of the milled product and the –90  $\mu\text{m}$  size fraction had a  $P_{80}$  of 90  $\mu\text{m}$ . The wet milling was carried out in batches of 2 kg and the reconstituted feed was fed to the flotation cell. The dry milling was carried out in 10 kg samples where the reconstituted product was blended and split into 2 kg batches for flotation.

## 2.2. Synthetic plant water

The wet milling and all flotation was carried out in synthetic plant water which was prepared using reverse osmosis water containing the following ions:  $\text{Ca}^{2+}$  80 ppm;  $\text{Mg}^{2+}$  80 ppm;  $\text{Na}^+$  135 ppm;  $\text{Cl}^-$  270 ppm;  $\text{SO}_4^{2-}$  250 ppm;  $\text{NO}_3^-$  135 ppm;  $\text{NO}_2^-$  40 ppm;  $\text{CO}_3^{2-}$  40 ppm. The TDS value was 1030 and the ionic strength was  $3.5\text{E}-02$ .

## 2.3. Batch flotation tests

Batch flotation tests were carried out on samples prepared using various combinations of crushing techniques and milling conditions. These are shown in Table 1. Mild steel grinding media was used during dry milling and stainless steel was used during wet milling. The base metal test work was carried out as a base case for further studies on a platinum bearing ore which was extended to wet milling with stainless steel and mild steel.

Batch flotation tests were carried out using synthetic plant water and in three stages. The first stage is carbon flotation; the

second stage is lead (Galena) flotation and lastly the zinc (Sphalerite) flotation stage. The carbon present in the gangue is detrimental to the concentration of lead and zinc by flotation. Therefore, preflotation, which is flotation in the absence of collector and prior to galena flotation, is employed to remove some of the organic carbon, some of which is naturally hydrophobic, from the feed (Gredelj et al., 2009). The pH was maintained at 9.5 for the carbon and lead flotation stages and then increased to 10.5 during the recovery of the zinc concentrates. The reagents, dosages, conditioning times and flotation times are illustrated in Table 2. Calcium hydroxide was added to regulate the pH. Sodium cyanide and zinc sulphate deactivate the sphalerite that has been inadvertently activated. After the carbon and lead has been removed through flotation copper sulphate is then added as an activator and sodium ethyl xanthate as a collector. Dow 200 was used as the frother. Before the final concentrate was collected Aerofloat 211 (AF211) was added as a collector.

The temperature, conductivity and  $E_h$  were measured after reagent addition.

## 2.4. Surface analysis techniques

The surface analysis of the minerals was carried out using a PHI TRIFT IV ToF-SIMS (Time of Flight Secondary Ion Mass Spectrometry) instrument operating in the static SIMS regime. Throughout the study a 30 kV, 100  $\mu\text{m}$ -Au<sub>1</sub> bunched cluster beam with charge compensation was used. The grains were analysed and imaged for Ca, Mg, Al, Si, Na, Mn, Fe, Cu, Zn and Pb during positive ion analysis and O, OH, S and xanthate during negative ion analysis. The data obtained was evaluated using Statistica. The intensities obtained are normalized for the elements of interest and presented as a relative percent normalized yield.

The XPS (X-ray Photoelectron Spectroscopy) analyses were carried out with an Escalab 250 using a monochromatic MgK alpha source (15 kV, 150 W). Binding energy accuracy is  $\pm 0.025$  eV. The Escalab charge neutralizer system was used on all specimens. Survey spectra were collected with pass energy of 150 eV and an analysis area of 500  $\mu\text{m}$ . High-resolution spectra (region scans) were obtained using a 40 eV pass energy and an analysis area of 500  $\mu\text{m}$ . Spectra were analysed using Thermo Advantage Software (version 3.91). Charge compensation was used.

The flotation feed, the first zinc concentrate and the tails for each condition were analysed using ToF-SIMS and XPS. The samples collected for analysis were screened to obtain a 38–106  $\mu\text{m}$  size fraction. The sample was washed with water (conductivity  $0.7 \mu\text{S cm}^{-1}$ ) that was adjusted to the desired pH to remove any physically attached ions. They were stored under argon and frozen.

**Table 1**  
Feed to float ore preparation.

Crushing	Milling	Media	Nomenclature
Cone	Wet	Stainless steel	CC-WM
Cone	Dry	Mild steel	CC-DM
HPGR	Wet	Stainless steel	HPGR-WM
HPGR	Dry	Mild steel	HPGR-DM

**Table 2**  
Flotation procedure.

Stage	Float (min)	Cond (min)	pH	CaOH	NaCN (g/t)	ZnSO <sub>4</sub> (g/t)	CuSO <sub>4</sub> (g/t)	Dow 200 (g/t)	SEX (g/t)	AF211 (g/t)
Grind			9.5	As req						
Carbon		1	9.5	As req						
	2	1			100	300				
Lead		1						10	25	
Zinc	1	1	10.5	As req			600			
		4								
		1	10.5	As req						
		1						15	25	
	2									
	3									
	5									
	10	1								15

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