minerals

Article

Effects of Different Grinding Media and Milling Conditions on the Flotation Performance of a Copper-Pyrite Ore

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Abstract: Different milling conditions, such as wet or dry, and use of different grinding media have a great impact on the flotation performance of sulphide minerals. In the present study, the effects of wet and dry grinding and the use of different grinding media, such as mild steel (MS) and stainless steel (SS), were investigated on a Cu-sulphide ore. The samples were ground as dry and wet with both grinding media, to a P_{80} value of −75 µm, and then flotation was carried out under the same conditions. The obtained data from flotation were evaluated in terms of solid/water recovery, chalcopyrite/pyrite recovery and separation efficiency. The effects of different milling conditions were discussed with the measured chemical parameters such as redox potential and dissolved oxygen level together with the flotation rate of chalcopyrite. The redox potential of the dry ground ore, irrespective of the type of milling media, was measured considerably higher than the wet grinding conditions. With SS media flotation, the rate of Cu was high for dry grinding, resulting in a higher selective concentrate in terms of grade. However, Cu recovery was lower due to the instability of the froth structure. Separation efficiency pointed out that the best flotation performance could be obtained using a wet grinding condition with MS balls.

Keywords: dry/wet grinding; stainless/mild steel ball; flotation; sulphide ore

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1. Introduction

Dissolution and electrochemical reactions, which have a great impact on flotation recovery, are influenced considerably by the grinding process. As the grinding operation is admitted as the last stage prior to concentration, the grinding environment has a significant effect on the behaviour of solids during subsequent steps, particularly in flotation [1]. The grinding process leads to the formation of highly active surface areas, some changes in the physical behaviour of solids which are attributed to permanent re-arrangement in crystal lattices and the structural alteration of particles [2,3]. Different milling procedures affect liberation, surface properties, particle morphology and surface roughness which can directly determine the hydrophobicity of the minerals and their flotation response [4,5]. The floatability of the ore, separation efficiency of minerals and selectivity are determined by surface properties which are controlled by the grinding process and its conditions [6]. Some defects such as cracks and pores may form on the surface of the particles depending on the grinding method, time and/or grindability of a mineral. These defects may have quite significant effects on the surface adsorption of collectors. Wet grinding generally produces particles of large specific surface and high surface oxidation, while dry grinding accounts for a large amount of surface defects and low surface oxidation. The results of a limited number of comparative studies performed on wet and dry grinding on flotation showed that dry ground materials are generally poorly wettable, while wet ground materials react quickly with water so that active charge centres and radicals are neutralized or disappear as a consequence of dissolving reactions. Gas molecules (especially oxygen) are physically or chemically adsorbed after dry grinding [7]. These films of gas will retard the wetting process to a greater or lesser extent. This behaviour can also be observed less prominently in many sulphides. Lepetic [8] has observed an increase in the collectorless floatability of chalcopyrite after dry autogenous grinding. This was attributed to both oxygen satisfying bonds and the formation of elemental sulphur or metal-deficient sulphur at the surface.

In industry, wet grinding is more commonly used because of the downstream processing requirements, such as target liberation of particle size, and the nature of the concentration processes, such as flotation. Feng and Aldrich [9] have compared the effects of dry and wet grinding on the flotation performance of a complex sulphide ore. They stated that flotation with dry milled ore showed more stable, highly loaded froths, and faster flotation kinetics, which were attributed to the activation of the highly formed new surface area of the particles. However, wet milled ore showed higher selectivity [10,11] and fine sulphide particles were found to have a tendency to oxidize in the air medium of dry grinding.

In addition to wet or dry grinding, the difference in the grinding media is a parameter that highly affects the flotation of sulphide minerals. The shape of the grinding media was found to have no significant effect on flotation [12]. Peng et.al [13] stated that a 30 wt.% chromium grinding media produced higher chalcopyrite selectivity against pyrite than mild steel media, while gas (oxygen and nitrogen) purging had no significant effect on chalcopyrite flotation. Other studies have been conducted on the use of balls of different types and chromium contents [14–19]. Besides grinding media, mill construction materials also have influence on the flotation performance of Cu-sulphide ores. It has been shown that ferrous ions and sulphide minerals cause galvanic interaction which reduces the redox potential of the pulp and, accordingly, results in a poor flotation response when carbon steel balls/rods are used. Utilisation of lined mills and non-ferrous or stainless steel grinding media were suggested to avoid these negative effects [1]. It has been stated that it is possible to perform pyrite flotation at alkaline pH depending on the changes in pulp potential when forged steel grinding media are used instead of chrome steel with different content [8].

In recent years, dry processing or water-free treatments have become more important for mineral processing applications in arid and semi-arid climates. In fact, water-free treatments or treatments using the minimum amount of fresh water should be encouraged for every part of the world due to changing climatic conditions and a shortage of consumable water resources. However, many enrichment methods in ore preparation, particularly flotation, are water-driven unit processes. In most of the cases, the mineralogy of complex ores does not allow for the use of separation processes other than flotation due to the necessity of fine grinding. Accordingly, the effects of dry grinding on flotation should be examined in order to be an alternative to the effective use of water. It has been well documented in the literature how dry or wet grinding and the use of different grinding media will affect the flotation of especially sulphide minerals. However, there are few studies in which all of these are combined and compared. This work thoroughly discusses how different grinding conditions and grinding media such as wet/dry and mild/stainless steel can affect flotation performance of a Cu-sulphide ore in terms of pulp potential, dissolved oxygen, recovery and separation efficiency.

2. Material and Methods

2.1. Ore Characterization

An ore sample taken from the rod mill feed of a Cu-sulphide ore processing plant was used in the experiments. The mineralogical composition of the ore was quite simple, consisting of 6.2% chalcopyrite, about 63% pyrite and non-sulphide gangue minerals, such as mostly quartz, gypsum, calcite, dolomite, chlorite, siderite/ankerite which accounted for the remainder. The general chemical composition of the ore is given in Table 1.

Table 1. Chemical composition of the ore.

Element	%	Element	%
Al	2.14	Mn	0.03
Ca	1.03	Na	0.81
Co	0.09	P	0.41
Cu	2.14	Pb	0.05
Fe	30.24	Si	7.83
K	0.83	Zn	0.33
Mg	0.87		

2.2. Grinding Tests

Wet and dry grinding were conducted with mild steel (MS) and stainless steel (SS) balls to determine the effects of the grinding medium on flotation. The MS balls contained 20% of chromium. The ore samples, each 1150 g, were ground as dry and wet with both grinding media, 83% of which was −75 µm, based on the plant operation information, and then flotation was carried out under the same conditions. The mill was 30.5 cm × 30.5 cm in size and was used with 25 mm (161 pcs, 49% *w*/*w*), 20 mm (192 pcs, 31% *w*/*w*) and 15 mm (284 pcs, 20% *w*/*w*) balls. Grinding in wet conditions was performed at 60% solid by weight. As a result of the grinding tests, it was determined that wet and dry grinding should be 11 and 25 min, respectively, to achieve the flotation feed target size.

2.3. Flotation Tests

The flotation tests were carried out with freshly ground samples in a volume of 3 lt Denver DR-12 conventional flotation cell with the conditions of 4 lt/min of air flow rate, 1200 rev/min of agitator speed and 30% solids by weight. The pulp pH was adjusted to 11.5 after grinding in both conditions. A TPS meter, 90FL-T model, was used to continuously record the Eh, pH and dissolved oxygen (DO) values during the flotation tests. A mixture (50:50) of a thionocarbamate (Hostaflot X231) and dithiophosphate (Aerofin 3477) was used as the collector after pH adjustment in 10 g/t, 20 g/t and 30 g/t dosages in the flotation and conditioning for 3 min for each collector. Then, Dowfroth 250 was added as a frother at 10 g/t dosage and conditioned for 1 min. Reagents were selected based on plant operating information. Four concentrates were obtained after a total of 8 min of flotation time by collecting the top-layer froth once every 15 s and analysing with a Varian Spectra AA5 Atomic Absorbance Spectrometer. The average data of the repeated flotation tests are presented in this paper. To examine the froth phase, the images captured during the flotation by a digital video camera, which was placed over the flotation cell, were analysed by the SmartFroth software. This software was used to measure the size and the speed of the bubbles.

3. Results and Discussion

3.1. Variations in Pulp Redox Potential

Variations in redox potential, dissolved oxygen and pH of the pulp were measured continuously during the flotation tests. Figure 1 illustrates the variations in redox potential of the pulp with the time for different grinding conditions. This graph represents the change in pulp potential in the system during flotation from the moment air is introduced to initiate flotation following conditioning. It is obvious that the wet-MS grinding process produced the lowest redox potential, as low as −400 mV. MS media decreased the pulp potential drastically due to the release of ferrous ions which in turn consumed all of the dissolved oxygen in the pulp. It is known that the optimum Eh value for collector adsorption ranges is between 0 to 200 mV for this sulphide type of ores. Increasing oxygen content in the pulp consumes the excess electrons and therefore increases the pulp potential. As a result of that, adsorption of thiol collector on sulphide minerals takes place at suitable pulp potential values. Therefore, it is not likely for the collectors to adsorb on the mineral surfaces until the potential reaches the suitable value. It is known that particularly in wet grinding conditions, oxygenation during conditioning and flotation is a significant parameter in the selective flotation of chalcopyrite from pyrite [20,21]. Introduction of air to the pulp at the beginning gradually increased the potential after 2–3 min of flotation, which affected the rate of flotation of copper minerals significantly. In wet grinding, the pulp potential increased from −400 mV to −200 mV by changing the medium from MS to SS, but it was observed that the potential still remained in the reducing condition, which was attributed to the galvanic interaction between chalcopyrite and pyrite. The dissolved oxygen was almost completely consumed in wet grinding by both grinding media. However, the presence of dissolved ferrous ions due to the galvanic interaction between the MS media and the sulphide minerals resulted in lower Eh. The redox potential of the dry ground ore, irrespective of the type of milling media, was measured considerably higher than the wet grinding conditions. In parallel to that, the DO concentrations were about 3–4 ppm in the beginning of the flotation. The absence of galvanic interaction in dry grinding affected the pulp chemistry considerably with both grinding media, and resulted in oxidizing pulp conditions. The higher redox potential and DO concentration enhanced the adsorption of collectors and flotation kinetics accordingly, as expected.

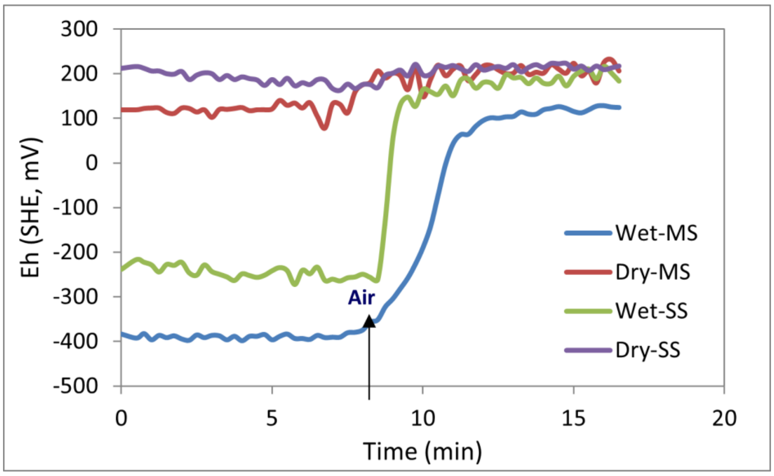

Figure 1. Redox potential of the pulp during at different milling conditions.

Figure 2 shows the relationship between the flotation rate constant of the chalcopyrite and redox potential of the pulp as measured in the conditioning stage. As the pulp potential was low in wet grinding conditions, the flotation rate of Cu was reduced. However, the dry grinding condition, particularly with SS media, resulted in the highest flotation rate.

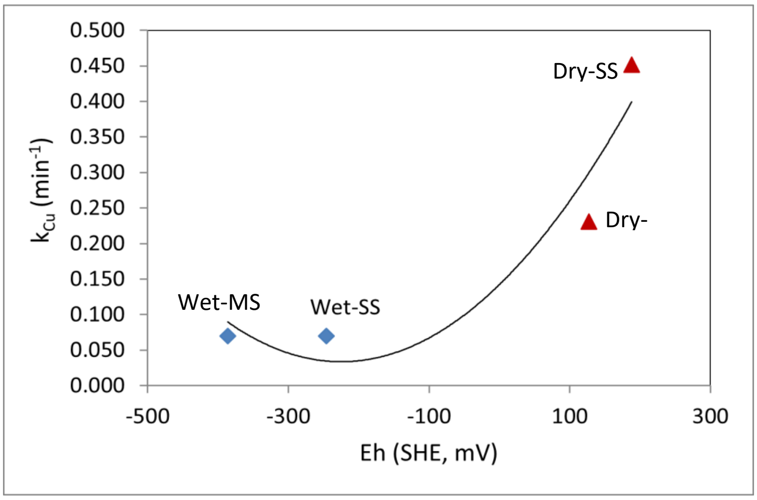

Figure 2. The relationship between pulp redox potential and flotation rate of Cu after different milling conditions.

3.2. Variations in DO Concentrations

There is a close relationship between the rate of DO consumption, pulp redox potential and therefore flotation selectivity [7,10]. Figure 3 shows the DO measurements taken after introducing air at a 4 lt/min rate at every 15 s interval. The curves indicate a clear difference between the rate of increase in DO concentration and the grinding conditions. The lowest rate was obtained with wet-MS grinding and the highest rate with dry-SS milling. Under wet milling conditions, the DO concentration appeared to increase gradually in the pulp and stabilized at around 7–8 ppm. However, in dry grinding conditions, DO concentration increased with an instant increase and remained stable around 9 ppm, especially in the use of SS media. It can be seen that the shapes of the curves in Figure 3 are quite similar to the metal recovery curves. Therefore, the rate of increase in DO concentration was calculated using the following equation:

$$DO = DO_{max}\left(1 - e^{-k_{DO}*t}\right) \quad (1)$$

where:

DO: Dissolved Oxygen

DO_{max}: Maximum dissolved oxygen

k_{DO}: Rate of dissolved oxygen concentration

t: time

DO_{max} was approximately 9 ppm for this condition, and it was the maximum attainable DO concentration for fully aerated pulp. In this case, the rate of increase of DO concentration was determined by the rate of galvanic interaction between the grinding media and sulphide minerals, and between the sulphide minerals.

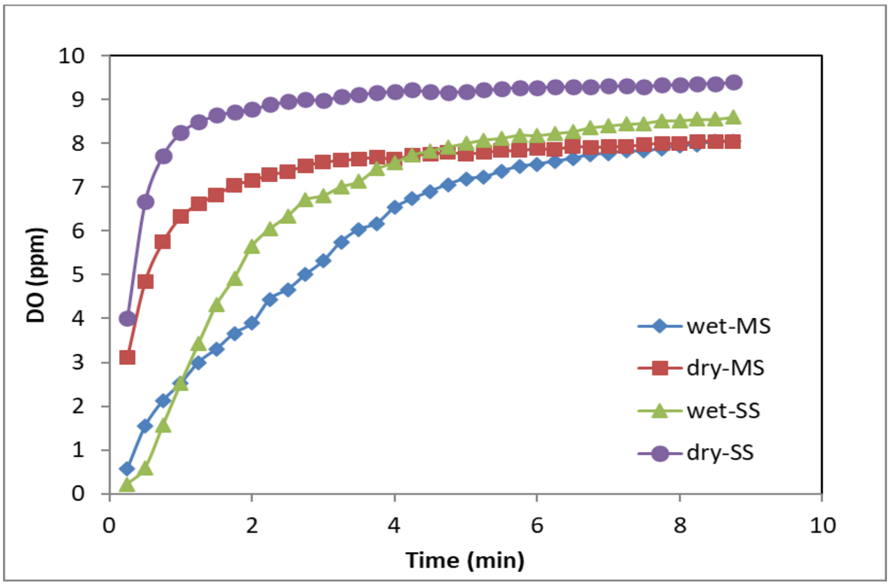

Figure 3. DO concentrations in the pulp during flotation for different milling conditions.

Figure 4 shows the relationship between the flotation rate constant of copper (k_{Cu}) and DO rate constant. There is a linear relationship between the two parameters, showing the dependence of flotation on the DO consumption of the ore.

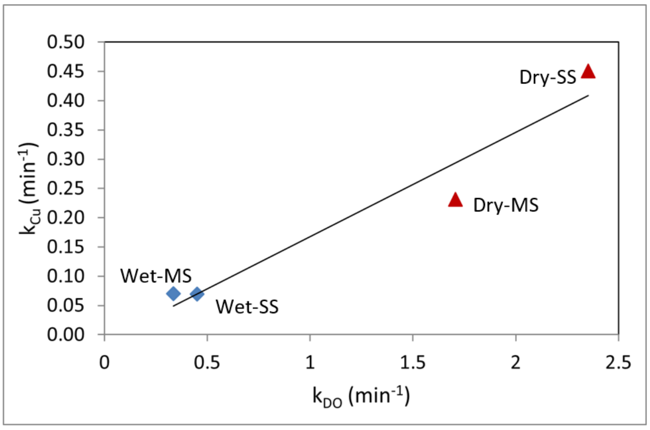

Figure 4. The relationship between DO concentration and flotation rate of Cu after different milling conditions.

3.3. Variations in Flotation Performance

Flotation tests performed after grinding processes with both MS and SS balls in wet and dry conditions were evaluated briefly below for three different dosages of collector mixture in terms of solid-water recovery (Rsolid and Rwater), copper-pyrite recovery and pyrite recovery mechanism. The water recovery can be used as an indicator of changes in the froth phase. High water recovery indicates a fast and watery froth structure, while low water recovery indicates a dry froth. Figure 5 shows that there is a linear relationship between the solid and the water recovery values. It is seen that after grinding with MS balls, both solid and water recoveries were much higher than the grinding tests with SS balls. This pointed out that froth fluidity and stability were higher in MS-ball milling conditions. Compared to the tests with MS balls, it was observed that the froth obtained in flotation after wet grinding with SS balls was very dry and a much smaller amount of solid recovered to the concentrate (Figure 5a). This situation can be attributed to the presence of metallic iron mixing with the pulp via MS balls which results in the formation of iron oxidation species. When the metallic iron does not exist, the froth is very brittle and the air bubbles may burst immediately. Higher water recovery can be obtained because of the more stable froth structure [22].

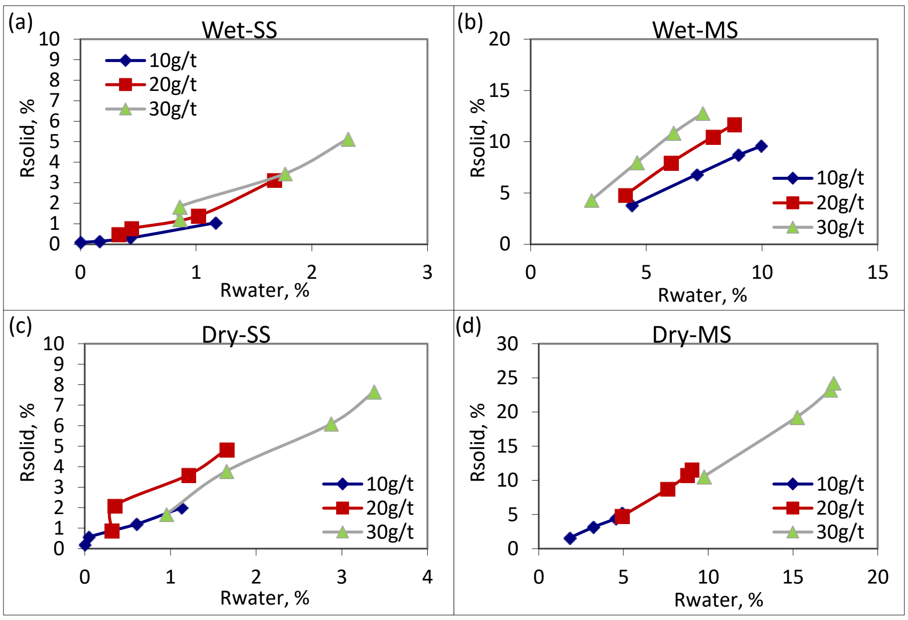

Figure 5. Solid vs. water recovery in flotation after milling conditions of (**a**) Wet-SS (**b**) Wet-MS (**c**) Dry-SS (**d**) Dry-MS.

Figure 6 shows the relationship between cumulative pyrite and copper recovery values after flotation for different milling conditions. Obtained data indicated that cumulative copper and pyrite recoveries increased with the collector dosages. Metallic iron mixed from the MS balls to the pulp during grinding caused an increase in the pyrite and copper recoveries by making the froth more stable in the flotation experiments. However, it is seen that the water recovery was very low in grinding conditions with SS balls (Figure 5a,c). The excessive viscous nature of the froth caused bubble coalescence and then easily burst (Figure 7). As a result, a significant decrease in copper recovery was noticed in the case of using SS balls compared to grinding conditions with MS balls. A similar observation was found by [23]. Chalcopyrite selectivity against pyrite was found to be reduced with SS media, while it was restored when grinding was performed using MS media. Ekmekçi [24] stated that the flotation behaviour of chalcopyrite and pyrite in mixtures were different from experiments with single minerals, and due to the galvanic interaction between the two minerals, the surface properties of the minerals were affected by each other. For example, it has been determined that copper ions dissolved from chalcopyrite were replaced by Fe^{2+} ions on the pyrite surface and caused hydrophobic sulphur rich CuS layer formation [25]. It is also reported by Peng et al. [13] that MS ball media generate reducing grinding conditions which favoured the formation of a copper(I) sulphide phase on pyrite surfaces, which results in high pyrite activation. However, in this case, besides pyrite activation, it is thought that the significant increase in pyrite recovery, particularly in the milling condition with MS balls, was most likely due to the increase in water recovery. The highest cumulative copper recovery for dry MS condition was 60.25% and cumulative pyrite recovery was 25.21% at a 30 g/t collector dosage. In addition, it was determined that the pyrite recovery, corresponding to one unit of copper recovery, increased with the increase in the collector dosage. This shows that the increase in the collector dosage significantly increased the amount of floating pyrite.

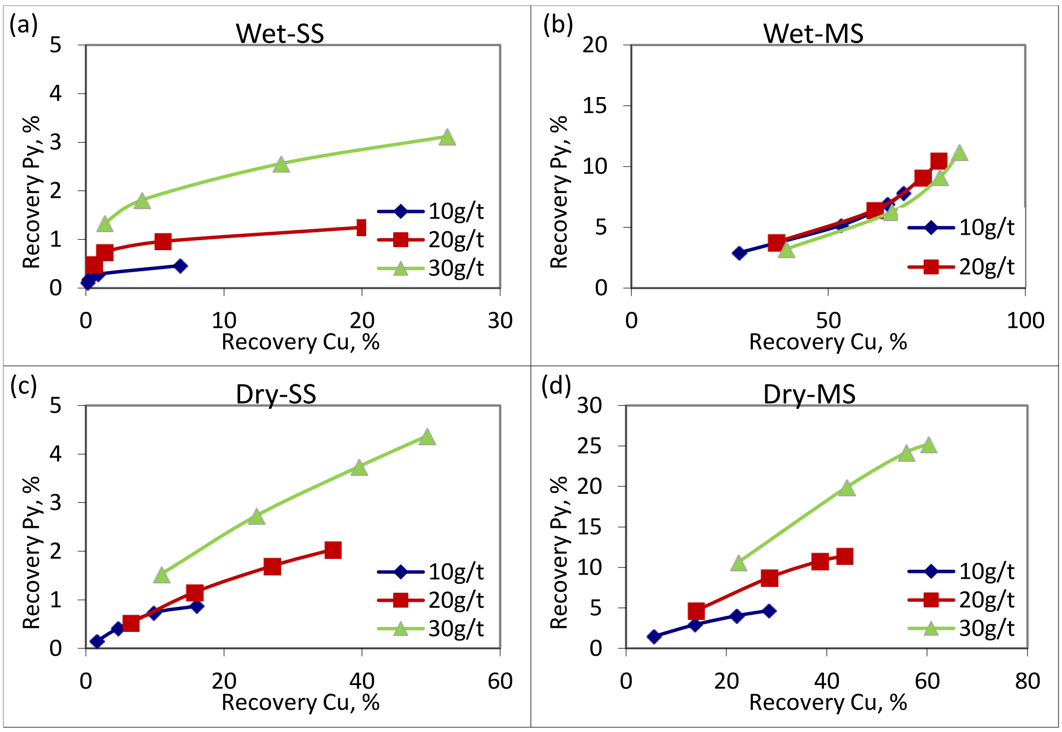

Figure 6. Pyrite vs. copper recovery in flotation after milling conditions of (**a**) Wet-SS (**b**) Wet-MS (**c**) Dry-SS (**d**) Dry-MS.

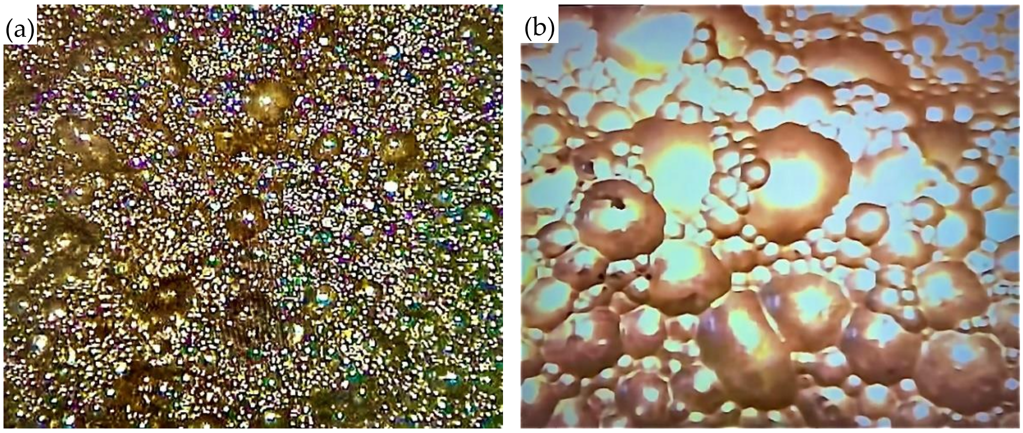

Figure 7. Froth images captured by Smart Froth for 30 g/t collector dosage. (**a**) Wet grinding with MS (**b**) Wet grinding with SS.

The effect of different grinding media on the flotation behaviour of pyrite is shown in Figure 8. It was observed that the least pyrite recovery to the concentrate was obtained in the experiments with SS balls grinding. However, it should be noted that the water and solid recoveries obtained under this condition were also very low. It is seen that pyrite was reported to concentrate depending on the water recovery, or, in other words, with entrainment, in the dry grinding condition with MS balls (Figure 8d). However, in the wet grinding condition with MS balls, when the collector concentration was increased to 30 g/t, it is seen that the amount of pyrite corresponding to the unit water recovery was recovered by true flotation (Figure 8b). It can be said that the similar situation is valid for the grinding condition made with SS balls, although the pyrite recovery is very low (Figure 8a,c). The increase in the collector dosage activated the pyrite, and hence increased its recovery.

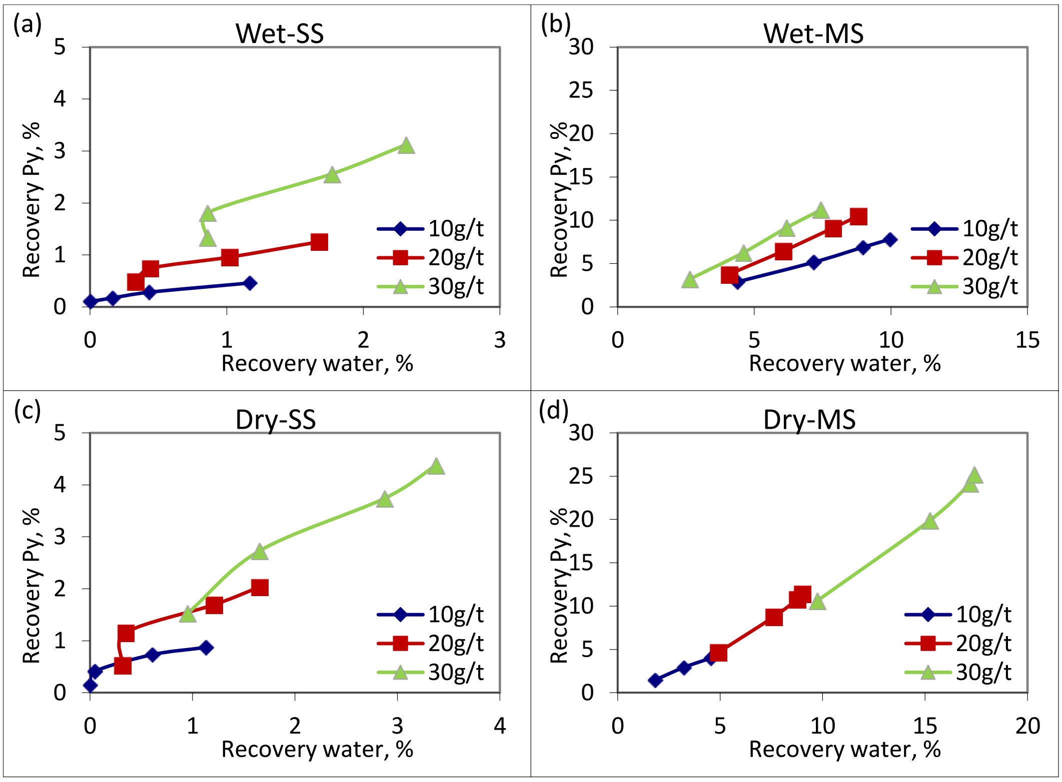

Figure 8. Pyrite vs. water recovery in flotation after milling conditions of (**a**) Wet-SS (**b**) Wet-MS (**c**) Dry-SS (**d**) Dry-MS.

The wet grinding condition with MS balls provided higher Cu recoveries in copper flotation (Figure 6). Although the copper flotation efficiency was very low in the experiments with SS balls, it was observed that the highest Cu concentrate grade was obtained under these conditions (Figure 9). After grinding with SS balls, the stability of the froth was very low and the chemical conditions were suitable for chalcopyrite flotation. However, the flotation efficiency was very low due to the very high hydrophobic particles making the froth extremely brittle and unstable. Therefore, in copper flotation after grinding with SS balls, a more selective concentrate in terms of Cu grade could be obtained.

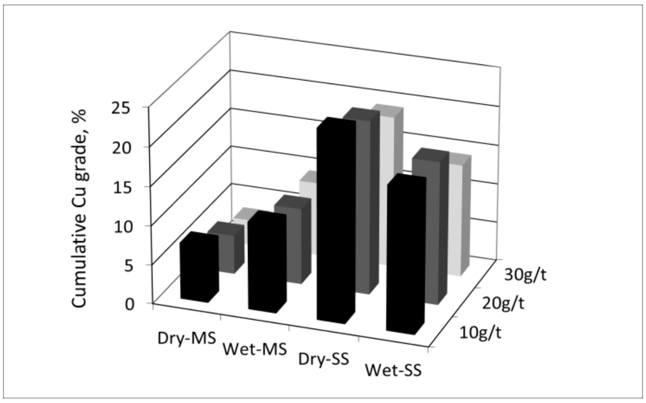

Figure 9. Cumulative Cu grade of the concentrates in flotation after different milling conditions.

The following separation efficiency equation [26] was used in order to evaluate the overall flotation performance in terms of both recovery and grade depending on the difference in the grinding medium and the concentration of the collector dosage:

$$E = R(1 - (f(c_m - c)/c(c_m - f))) \quad (2)$$

where:

E: Separation efficiency

R: Recovery of valuable mineral (%)

f: Grade of feed (%)

c: Grade of concentrate (%)

c_m: Maximum grade of concentrate (%) (Maximum Cu grade can be obtained from chalcopyrite in theory)

The calculated separation efficiency values for different milling conditions and the collector dosages are plotted in Figure 10. It is seen that the highest separation efficiency in terms of recovery and grade value was obtained in the wet grinding condition made with MS balls. In any case, the separation efficiency increased with the increase in the collector dosage.

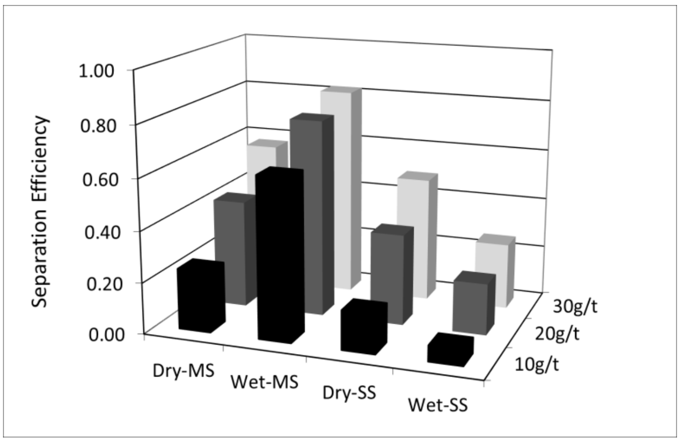

Figure 10. Separation efficiencies in flotation after different milling conditions.

4. Conclusions

In view of investigating different grinding conditions with wet/dry operation and different grinding media, in terms of grade and recovery in sulphide mineral flotation, the following conclusions can be deduced:

1. Dry grinding eliminates the galvanic interaction between sulphide minerals and the grinding media. Therefore, the redox potential of the dry ground ore, irrespective of type of milling media, was measured considerably higher than the wet grinding conditions. This situation resulted in a high flotation rate of Cu, particularly in the dry grinding condition with SS media.
2. In the dry grinding condition in which SS media were used, the dissolved oxygen amount was also measured as the highest value, around 9 ppm, and showed a linear relationship with the Cu flotation rate.
3. The dry grinding condition, particularly with SS balls, resulted in a higher selective concentrate in terms of Cu grade due to it serving a better electrochemical condition for sulphide minerals. This could be explained with the high amount of dissolved oxygen which enhanced the adsorption of collectors and flotation kinetics, accordingly. However, the Cu recoveries of the concentrates were lower due to the instability of the froth structure and the coalescence of air bubbles in flotation after grinding with SS balls. The most stable froth structure was observed in the tests in which there was milling with MS balls, and it was concluded that it was due to the metallic iron mixed into the pulp from the MS balls.
4. It was determined that the pyrite dilution was higher in Cu concentrate after milling with MS balls, and the pyrite recovery mechanism was by entrainment, especially in the dry milling condition. The pyrite dilution in the concentrate was lower in the case of milling with SS balls, but another possible reason was that the solid and water recoveries were already low under this condition.
5. Separation efficiency pointed out that the best flotation performance could be obtained using the wet grinding condition with MS balls.
6. Since dry grinding eliminates galvanic interaction, it has potential for the future in reducing the contamination of recycling plant water, using less fresh water and providing higher flotation kinetics. It is known that the flotation of sulphide minerals is strongly dependent on the redox potential and dissolved oxygen content of the pulp. After dry grinding, the surface of sulphide minerals will not be oxidized when they are introduced into the flotation pulp. Therefore, very high flotation rates and even collectorless flotation in some cases can be obtained after the dry grinding of sulphide ores. However, after dry grinding, the instability of the froth structure needs to be improved, especially in case SS balls are used.

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