Minerals Engineering 94 (2016) 61-75

Increasing the grind size for effective liberation and flotation of a porphyry copper ore by microwave treatment

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Keywords

Microwave; Ore; Mineralogy; Liberation; Flotation.

Highlights

- Microwave-treatment resulted in equivalent liberation at a coarser size
- Grind size can be increased by approximately 30-50µm
- Copper recovery can be improved by approximately 1%
- Small reductions in competency can still yield improved liberation and flotation
- Intimate association of copper and iron sulphides helps liberation enhancement

Abstract

In this paper, mineralogy, grain size, dissemination, textural consistency and mineral associations were determined for a commercially exploited porphyry copper ore using a Mineral Liberation Analyser (MLA). The ore was subjected to high power density microwave treatments in a single mode cavity at 15kW and approximately 2kWh/t. The untreated and microwave-treated samples were subsequently milled to two grind sizes near the nominal plant grind size and a size-by-liberation analysis performed. The analysis revealed that equivalent liberation could be obtained at a grind size approximately 50-60µm coarser than the nominal plant grind due to the microwave treatment. Flotation testing indicated that an increase in copper recovery of approximately 1% could be achieved, or that a grind size increase of approximately 30µm may potentially yield equivalent copper recovery due to the microwave-enhanced liberation observed. However, statistical analyses demonstrated that it is difficult to attain confidence in recovery increases of approximately 1% even when conducting batch flotation tests in triplicate. The ore under investigation had previously been shown to produce only modest average reductions in strength (~8%) under similar microwave treatment conditions due to a prevalence of many unfavourable textures. However, the preferential association of copper minerals with a hard matrix mineral (quartz) and a hard microwave-absorbent mineral (pyrite) resulted in a significant change in liberation behaviour.

http://dx.doi.org/10.1016/j.mineng.2016.05.011

1 Introduction

The growing demand for many mineral commodities combined with a higher embodied energy in their production from falling grades, finer mineralisation and increased mining activity will place a strain on sustainable mining in the future (Crowson, 2012; Kesler, 2007; Mudd et al., 2013; Northey et al., 2014; Prior et al., 2012). In an effort to address these challenges, minerals industry stakeholders have identified reducing ore competency prior to energy intensive comminution and improving liberation to enable more efficient separation closer to native grain sizes as two important areas of investigation (Daniel and Lewis-Gray, 2011; Drinkwater et al., 2012; Pokrajcic et al., 2009; Powell and Bye, 2009).

High power density microwave treatment has been proposed as a technique to reduce ore competency prior to beneficiation and as a means of enhancing liberation through the generation of thermally-induced cracks along mineral grain boundaries leading to inter-granular and trans-granular fracture (Kingman et al., 2004a; Kingman et al., 2004b; Kingman et al., 2000a). The efficacy of this mechanism and the amenability of ores to microwave-induced fracture has been shown to depend on the dielectric, thermal and mechanical properties of the minerals involved, their assemblage within the ores, and the microwave energy and power density applied (Ali and Bradshaw, 2009, 2010; Jones et al., 2007; Kingman et al., 2000b; Wang et al., 2008; Wang and Djordjevic, 2014; Whittles et al., 2003). In particular, highly microwave-absorbent phases (such as nickel, copper, iron and lead sulphides, and magnetite) with a grain size d50 greater than approximately 500µm constrained by a hard matrix (such as quartz, feldspar, pyroxene and olivine) have been shown to provide the largest reductions in ore competency (Batchelor et al., 2015).

Despite the potential for microwave-induced grain boundary weakening and fracture to aid in liberation, most of the published literature on experimental microwave treatments of metalliferous ores has focused on reduced ore competency. This is likely due in part to the limited availability of automated mineralogy facilities (e.g. Mineral Liberation Analyser (MLA) and QEMSCAN) to researchers prior to the mid-2000's when the bulk of the experimental research was conducted, but also because the value obtained from reduced ore competency is more easily quantified and realised in comminution circuits and has therefore been of primary interest to research sponsors.

However, the ability to exploit microwave-induced fracture by improving flotation recovery and grade, and/or increasing the primary grind size to effect liberation has significant potential benefits in terms of increasing plant throughput and metal production while reducing metal specific energy consumption, even without significant reductions in ore competency. The scale of these benefits is evidenced by the incremental movement on earnings before interest and tax (EBIT) for a large copper producer given in Table 1 (Minera-Escondida, 2002). Mill recovery may have the largest impact on EBIT that the producer can influence and even a small increase in mill recovery can provide significant returns, potentially making the pursuit of improved recovery the largest value proposition for microwave pre-treatment. Indeed, increasing productivity to maintain competitive advantage has been identified as the second highest risk to copper mining (first for all mineral commodities) (EY, 2014). Furthermore, there is an increasingly urgent need for the historically risk-averse mining industry to innovate and establish less water and energy intensive processes, leading to it being identified as the third highest risk for copper mining (tenth for all mineral commodities) (EY, 2014).

Table 1

Key business drivers for a large copper producer

Item	Units	EBIT Movement (US$ x10^6)
Copper Price	1c/lb	21
Head Grade	0.01%	8.5
Mill Throughput	1,000t/d	7.5
Mill Recovery	1%	16
Concentrate Grade	1%	7
Power	0.1c/kWh	2
Fuel	1c/L	1

http://dx.doi.org/10.1016/j.mineng.2016.05.011

Several authors have demonstrated improved liberation on copper sulphide, iron sulphide and native gold-containing ores after microwave treatment and subsequent grinding (Amankwah et al., 2005; Andriese et al., 2012; Wang and Forssberg, 2005). Other studies have also demonstrated increased flotation recovery and concentrate grade attributed to enhanced liberation due to microwave-induced fracture on copper, lead-zinc and nickel sulphide ores (Henda et al., 2005; Kingman et al., 2000a; Kumar et al., 2006; Orumwense and Negeri, 2004; Vorster et al., 2001). However, these studies originally treated ball mill feed size material (<10mm) at low power (<3kW) in low power density multimode cavities for several minutes, resulting in very high energy inputs (>>10kWh/t) unsuitable for incorporation in a typical comminution circuit.

Kingman et al. (2004b) and Scott et al. (2008) both tested the same copper ore investigated by Kingman et al. (2000a) and demonstrated improved liberation after high power density microwave treatments on lump fragments (>10mm) in single mode cavities with up to 15kW microwave power at economically feasible energy inputs (0.1-5kWh/t). Furthermore, Sahyoun et al. (2005) conducted flotation tests on the same ore and demonstrated a 3-6% increase in copper recovery after microwave treatment (up to 12kW and 1.7kWh/t on <22mm size material) as opposed to the 1% increase reported by Kingman et al. (2000a), attributed to the higher power density sustained in the single mode cavity. Treating coarser particles as opposed to ball mill feed size material further ensured that more of the microwave-heating phases are constrained by the non-sulphide gangue matrix, thereby promoting more microwave-induced grain boundary fracture. More recently, Ali and Bradshaw (2011) have used numerical modelling techniques that suggested up to a three-fold increase in the amount of liberated lead sulphide may be achieved with high power density microwave treatments under confined bed breakage conditions. Therefore, it has been shown to be possible to treat large fragments that would likely feed an AG/SAG mill at high power density and low energy, and yet still achieve improvements in liberation and ultimately flotation.

This paper investigates the change in liberation and flotation performance on a commercially exploited porphyry copper ore at the nominal plant grind size and a coarser grind size at the coarse end of the normal flotation range after high power density microwave treatment. The relationship with mineralogical textural features is also elucidated to understand the interaction of microwave treatment with the ore and its effect on selective breakage leading to improved liberation and flotation.

2 Materials and methods

2.1 Ore sample

The ore sample used in this investigation was a porphyry copper ore from Chile. The ore was supplied as SAG mill feed and subsequently screened into a -53.0+9.5mm size class. Two representative samples of approximately 30kg were then split by rotary sample divider to provide the test samples for untreated and microwave-treated material.

A different test sample of the same ore was previously tested by Batchelor et al. (2015) for reductions in ore competency resulting from microwave treatment and was labelled as Cu-Ore 1. Ore competency was investigated using the Point Load Test (Broch and Franklin, 1972; Brook, 1985; Franklin, 1985) as it was a quick method for determining the minimum force required for first breakage of ore fragments and as such exploited any microwave-induced fracturing of the ores (a lower Point Load Index indicates a softer ore). Microwave treatments were performed in a single mode cavity (described further in section 0) at 20kW microwave power and with 1.2kWh/t microwave energy. The Point Load results, given in Figure 1, indicated that a modest average reduction in ore strength of 7.8% was achievable under these microwave treatment conditions. However, average Point Load strength reductions of approximately 5-15% were common for the other copper ores tested in that investigation at approximately 1kWh/t.

Cu-Ore 1 was chosen for further liberation and flotation analysis for several reasons. It is evident from the spread of Point Load results that only the hardest fragments (those typically containing quartz as the dominant matrix mineral) with significant copper and iron sulphide mineralisation (good microwave-heating minerals) experienced any substantial reduction in competency. Many fragments were effectively sulphide barren, had very fine sulphide mineralisation and/or contained a high proportion of soft matrix minerals (typically micas and other phyllosilicates) as illustrated in Figure 2. All of these textural features were shown by Batchelor et al. (2015) to limit microwave-induced fracture and therefore limit large average reductions in ore competency.

However, the copper sulphide minerals had a relatively coarse grain size, tended to be concentrated in a small proportion of the ore, were notably associated with hard matrix minerals and were intimately associated with pyrite (another hard and microwave-heating mineral), often tending to rim the pyrite grains. Therefore, the bulk of the copper sulphides were likely included in the group of fragments that experienced significant microwave-induced fracture. As such, despite the textural variability and modest average strength reductions, it was believed that Cu-Ore 1 was a potential candidate for improved liberation due to microwave-induced fracture.

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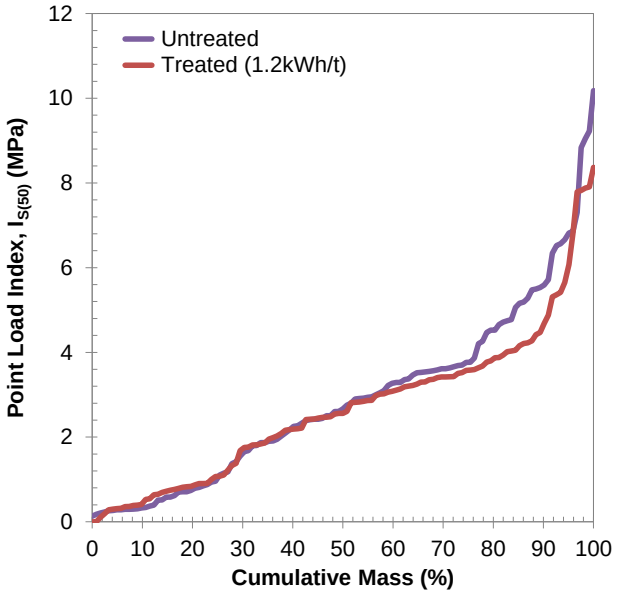

Figure 1: Point Load Test results for Cu-Ore 1

2.2 Microwave treatments

Microwaves were provided by 3-15kW variable power Sairem generator operating at 2.45GHz. The generator was connected by rectangular WR430 waveguide to an E-H plane automatic tuner with a circulator in the transmission line to dissipate reflected power in a water load. The tuner was then connected to a TE_{10n} single mode cavity and finally a short circuit tuner. A schematic illustration of the microwave system is given in Figure 3.

A single mode cavity is a metallic enclosure in which the superposition of the reflected and incident microwaves gives rise to a standing wave pattern that is very well defined in space and usually localised in a small volume. The automatic E-H tuner was employed to match the impedance of the generator and transmission line to that of the ore load in the cavity in order to maximise the absorbance of microwave energy by the test sample. The short circuit tuner was employed and adjusted to position the maximum electric field strength within the ore load, allowing maximum heating rates to be achieved during treatment.

The TE_{10n} cavity was comprised of a hollow cylindrical tube section with an internal diameter of 82mm intersecting the broadside of the waveguide. Up to approximately 1.5kg of ore load was placed in a borosilicate glass tube supported by an alumina block that could then pass through the cavity. The test sample was preceded by sacrificial ore to absorb microwave energy during the microwave power ramp up cycle in order to ensure that the test sample received microwave energy at steady state and to ensure the system was matched. Residence time within the cavity was then controlled using a pneumatic piston with an adjustable stroke, shown in Figure 4. The borosilicate glass tube, alumina supporting block and plastic piston rod were constructed from microwave-transparent materials so that only the ore load would absorb microwave energy.

In total, 22 batch treatments were performed with an average of approximately 1.25kg of material per treatment. The single mode cavity created a hot spot of high electric field on the generator side of the applicator, where microwaves were incident to the load. To ensure that all of the ore was exposed to the highest possible electric field, the sample was given a second pass treatment by rotating the tube through 180°. The average microwave treatment conditions were 14.1kW incident power and 1.5kW reflected power, giving 12.6kW absorbed power at 0.1s exposure time, resulting in an average single pass microwave energy input of 1.0kWh/t and total microwave energy input of 2.1kWh/t.

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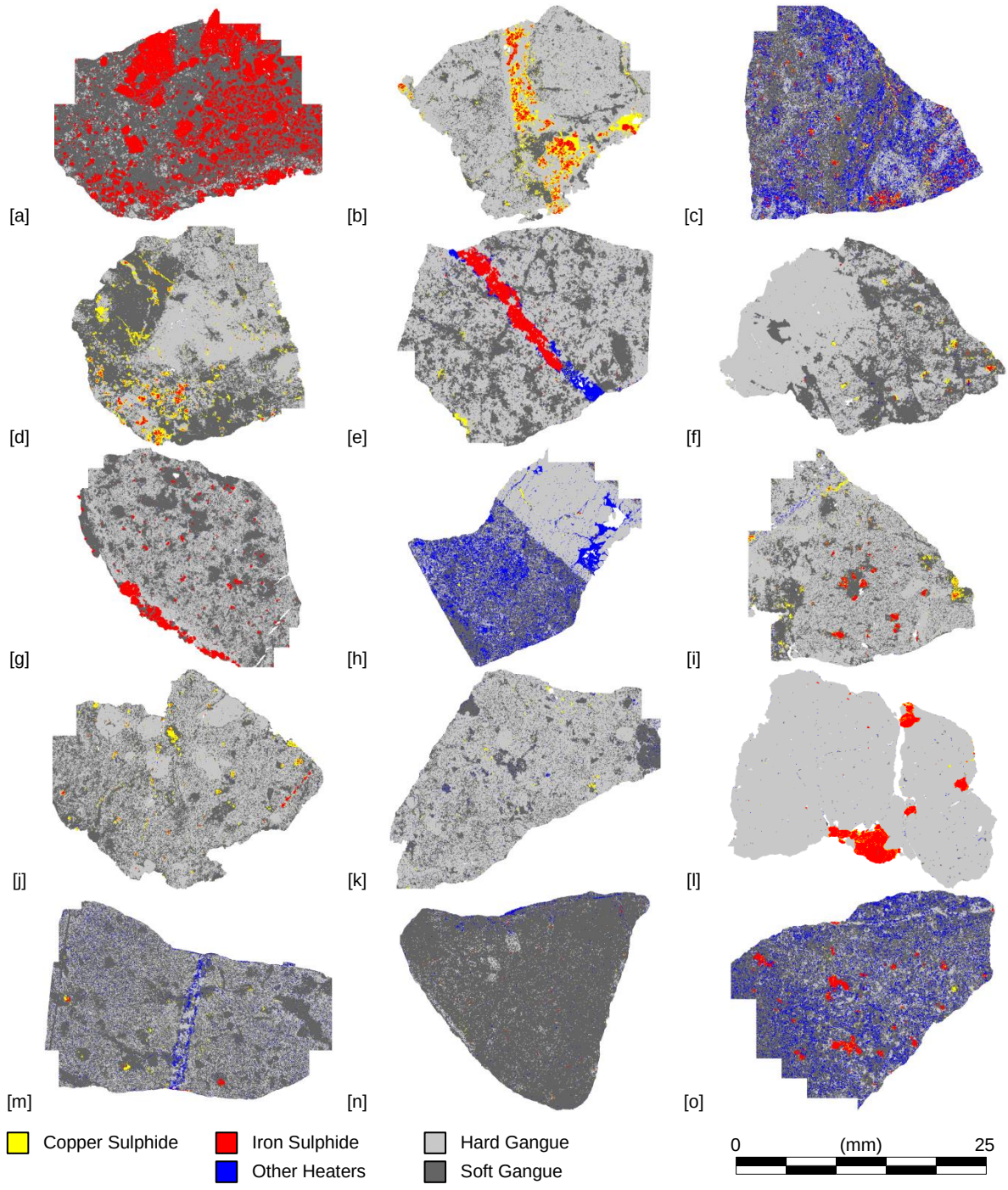

Figure 2: Example lump fragment false colour image from MLA mineralogical texture analysis

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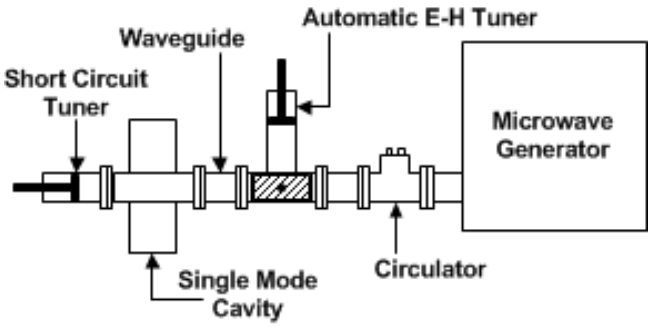

Figure 3: Microwave treatment apparatus

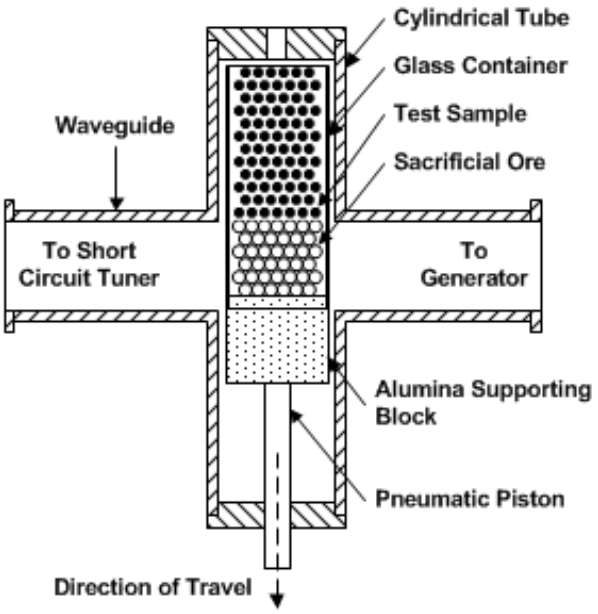

Figure 4: Single mode cavity apparatus

2.3 Sample preparation and characterisation

A Mineral Liberation Analyser (MLA) (FEI Quanta 600 platform) was used in these investigations to conduct a textural analysis of the ore as well as to determine the liberation characteristics of milled untreated and microwave-treated material.

Polished sections of 84 lump fragments up to 30mm in diameter were measured to provide mineralogical information such as modal mineralogy, mineral grains sizes and mineral associations that resulted in quantitative textural data of the natural mineral assemblage in the ore. Minerals were grouped according to their microwave-heating ability and according to their resistance to breakage, as defined by the Moh's Hardness scale, to create false colour images, selections of which were shown previously in Figure 2.

Both untreated and microwave-treated samples were stage crushed in a laboratory jaw crusher at the same closed side settings (CSS) to ensure the same conditions for breakage, and starvation fed to minimise fines production and to allow microwave-induced fractures to be preferentially exhausted at the earliest opportunity. The first stage at 9.5mm CSS was hand fed, but the second and third stages, at 4mm CSS and 1mm CSS respectively, were fed at a controlled rate via a vibrating feeder.

The crusher products were split by rotary sample divider into representative 1kg sub-samples for batch grinding. A laboratory rod mill was operated at 50%wt solids and 70% critical speed (66rpm), which was inline with the host mines flotation preparation grinding procedure.

A grinding calibration was conducted at 0, 2.5, 5, 10 and 15min on the untreated sample to find the grind time necessary to achieve the host mine plant grind P_{80} of 190µm and a P_{80} of 290µm to investigate any change in liberation 100µm coarser than the plant grind. The microwave-treated sample was then milled at the same grind times to achieve an equivalent amount of work on the ores, as opposed to targetting the same grind sizes; however, a limited grinding calibration was also performed on the microwave-treated material for comparison. Confirmation grinds were performed at the grind times selected for flotation experiments with the addition of flotation reagents to determine the variation in grind size likely to be present in replicate flotation tests. The samples milled for the chosen grind times were subsequently wet and dry screened into the root 2 series.

For liberation analysis, the sized fractions were set in resin in 30mm diameter mounts and enough mounts were prepared to ensure a minimum particle count of 10,000 particles in each size class. The mounts were then ground, polished and carbon coated to present particle sections for scanning electron microscopy. Magnification and resolution settings were selected to give a pixel size ranging from 3.5µm to 0.35µm for the >425µm to <38µm size classes respectively.

http://dx.doi.org/10.1016/j.mineng.2016.05.011

2.4 Flotation

Rougher flotation testing was performed on the untreated and microwave-treated material to determine if any changes in liberation resulted in observable changes to the copper grade-recovery curves. The procedure used in this investigation was adapted from the host mine procedure, with some alterations due to availability of reagents and equipment. Substitute reagents were sourced and approved by the project sponsors from previous work on the ore. The reagents, their function and dosage are listed in Table 2.

Table 2

Flotation reagents

Reagent	Function	Dosage
Cytec Aero Promoter AP-3758 [a]	Collector	62.4g/t
Cytec Aero Xanthate AX-317 [b]	Collector	18.0g/t
Cytec Oreprep X-133 [c]	Frother	16.2g/t
Dertol 90 (pine oil)	Frother	9.3g/t
Sodium hydrosulphide (NaHS)	Activator	200g/t
Lime (CaO)	pH Control	~1,000g/t

[a] Xanthogen formate with methyl isobutyl carbinol (MIBC).
[b] Sodium isobutyl xanthate (SIBX).
[d] Mixture of alcohols, heavy aldehydes, esters and glycols.

Laboratory flotation tests were performed in triplicate in a four litre Denver flotation cell; however, the host mine procedure used a three litre cell. A mixture of collectors and frothers were employed, with lime to control the process water to pH10. AP-3758 was substituted for Matcol D-101 (dithiocarbamate) collector and Dertol 90 used as the pine oil frother. The host mine procedure called for an 87.5% D-101 and 12.5% AX-317 collector blend, with a 70% X-133 to 30% pine oil frother blend. Exploratory tests were performed to adjust the reagent dosage for the increased cell volume and reduced pulp density (from 26.5%w/w to 21.4%w/w), yielding the reagent dosages listed in Table 2.

The reagents were added to the mill during grinding, but with only half of the NaHS and the required lime to achieve pH10. The pulp was transferred to the flotation cell and conditioned for a further five minutes with the remaining NaHS and air flowing at 0.5L/min. The airflow was increased to 3L/min and cumulative timed concentrates were collected at 1, 2 and 5 minutes. The airflow was raised to 5L/min and a final cumulative timed concentrate taken to 12min (i.e. 1, 1, 3 and 7 minutes per concentrate). The four concentrates and tailings samples were then filtered, dried, weighed, and sub-samples pulverised and submitted for assay.

3 Results and discussion

3.1 Mineralogical characterisation

The average lump fragment and average liberation grind modal mineralogies are presented in Table 3. It is clearly seen that the textural analysis on 84 lump fragments closely approximated the modal mineralogy obtained from the more representative 1kg batch grinds. The average good microwave-heating phase content was comprised of ~2.8%wt copper sulphides, ~5.4%wt pyrite and other sulphides, and ~1.6%wt smectite. Smectite is a good microwave heater due to the presence of interlayer and bound water (i.e. $x.nH_2O$). Poorly microwave-heating phases include ~1.2%wt iron and titanium oxides. The “hard” microwave-transparent phases are dominated by ~42.1%wt quartz with ~4.5%wt feldspar. The “soft” microwave-transparent phases are dominated by ~27.8%wt illite with ~12.8%wt biotite, kaolinite, pyrophyllite and chlorite.

3.2 Crushing and grinding

The crushing, grinding, combined native copper sulphide and good microwave-heating phase grain size distributions are presented in Figure 5. It is clearly seen that under the same crushing conditions the microwave-treated sample yielded a slightly finer crushing product, with an untreated (UT) sample P_{80} of ~3.0mm compared to a microwave-treated (TD) sample P_{80} of ~2.4mm. The authors experience has shown this is a common occurrence when crushing microwave-treated samples and is indicative of the presence of microwave-induced fractures in the ore. This observation was also predicted in the numerical models run by Ali and Bradshaw (2011) as well as qualitatively described in the some of the experimental literature review previously.

http://dx.doi.org/10.1016/j.mineng.2016.05.011

Table 3

Modal mineralogy

Mineral Name	Texture (wt%)	Liberation (wt%) [a]
Good-MW Heaters		
[b] Chalcocite	1.2	1.7
[b] Chalcopyrite	0.6	0.4
[b] Covellite	0.2	0.3
[b] Bornite	0.5	0.3
[c] Iron Sulphide	5.7	5.4
[d] Smectite	1.9	1.6
Sub-Total	10.1	9.6
Poor-MW Heaters		
[d] Iron Oxides	1.2	1.0
[d] Rutile	0.1	0.2
Sub-Total	1.3	1.2
Non-MW Heating Gangue		
[e] Quartz	34.6	42.1
[e] Feldspars	4.0	4.5
[f] Illite	32.1	27.8
[f] Biotite	5.0	3.9
[f] Kaolinite	6.3	3.8
[f] Pyrophyllite	1.8	2.9
[f] Chlorite	3.7	2.3
[f] Other	1.2	2.0
Sub-Total	88.6	89.2

[a] Average of all four liberation grinds.
[b] Copper-Sulphides (also includes trace digenite, anilite and enargite).
[c] Iron-Sulphide (predominantly pyrite, includes trace molybdenite, galena and sphalerite).
[d] Other-Heaters (Iron Oxides include hematite, goethite and ilmenite; smectite classified as montmorillonite).
[e] Hard-Gangue (Feldspars include orthoclase, albite and oligoclase).
[f] Soft-Gangue.

The native grain sizes derived from the textural analysis show that the crushing product is somewhat finer than the copper sulphide grain size, which seems to indicate that these minerals would begin to liberate even at such a coarse size. However, it must be noted that the copper sulphide grain size appears artificially coarse due to the MLA software defining the grain size as the diameter of an equivalent area circle. Many of the copper sulphide grains occur as fine veins or thin rims around pyrite with touching adjacent grains. Any grains that touch are considered by the MLA software to be a single grain, thereby increasing the grain area and grain size; hence the apparent lack of grains less than ~500µm in size. Unfortunately, the MLA software offered no alternative option that would more representatively characterise the width and lengths of the adjoining grains to determine the average Feret's diameter.

The grinding calibration on the untreated material predicted grind times of 6.1 minutes and 9 minutes to achieve the target P_{80}'s of 290µm and 190µm respectively, shown in Figure 6. The resulting liberation sample grinds at these times for the untreated sample were P_{80} 288µm and 182µm with corresponding microwave-treated sample P_{80}'s of 238µm and 162µm. The microwave-treated sample grinds were finer due to the finer crushing distribution and potentially due to any remaining microwave-induced fractures in the ore after crushing.

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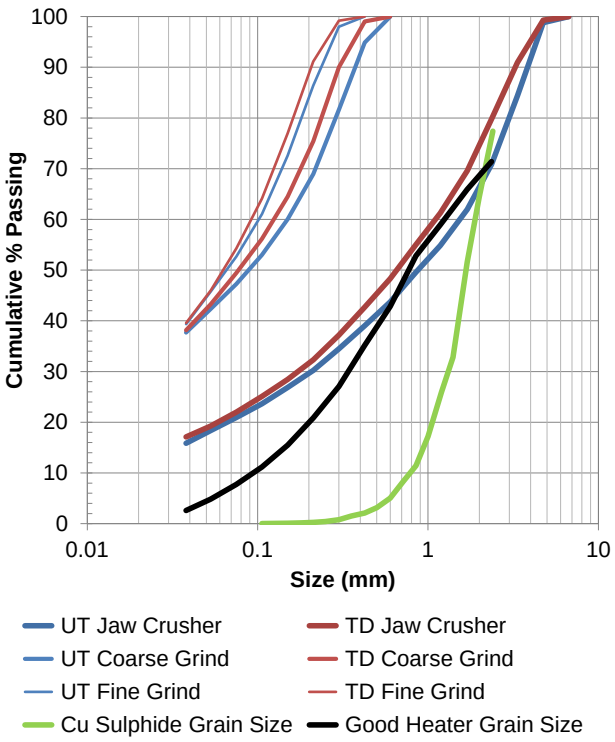

Figure 5: Crushing, grinding and native grain size distributions

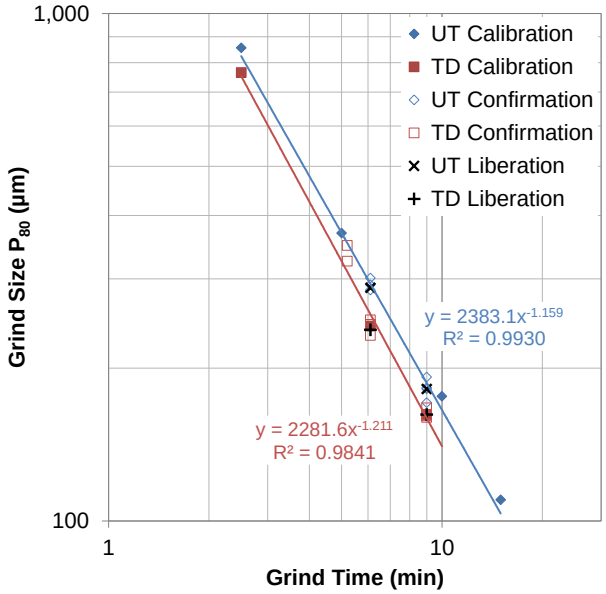

Figure 6: Grinding calibration curves

Bond Ball Mill Work Index tests were not performed as part of this study due to a limited amount of material. Ideally, to test whether there are residual fractures in the microwave-treated material a synthetic feed should be made that is equivalent in size distribution to the baseline untreated material. However, to estimate any reduction in ore competency at milling sizes, a relative Work Index was calculated from the untreated and microwave-treated batch grinds using the method proposed by Berry and Bruce (1966). This method was also applied by Kingman et al. (2000a) and Vorster et al. (2001) to determine the Work Index of microwave-treated ore from an untreated ore with a known Work Index; however, the Work Index was unknown for this sample being investigated.

Bond's third theory (Bond, 1961) is presented in Eq. (1). The same amount of work is done on both the untreated and microwave-treated ore during batch grinding since they both contain the same mass of ore, grinding media and water, and were ground for the same period of time, giving Eq. (2). The feed and product sizes from batch grinding are known, so the relative Work Index may be calculated according to Eq. (3):

$$W = WI\left(\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}}\right) \quad (1)$$

$$W = WI_{UT}\left(\frac{10}{\sqrt{P_{80,UT}}} - \frac{10}{\sqrt{F_{80,TD}}}\right) = WI_{TD}\left(\frac{10}{\sqrt{P_{80,TD}}} - \frac{10}{\sqrt{F_{80,TD}}}\right) \quad (2)$$

$$WI_R = \frac{WI_{TD}}{WI_{UT}} = \left(\frac{10}{\sqrt{P_{80,UT}}} - \frac{10}{\sqrt{F_{80,UT}}}\right) \Big/ \left(\frac{10}{\sqrt{P_{80,TD}}} - \frac{10}{\sqrt{F_{80,TD}}}\right) \quad (3)$$

Where W is the work performed on an ore (kWh/t), WI is the Work Index (kWh/t), WI_R is the relative Work Index, P_{80} is the product 80% passing size (µm), F_{80} is the feed 80% passing size (µm), and the subscripts *UT* and *TD* denote untreated and microwave-treated material respectively.

The calculated relative Work Index for the coarse grinds (6.1min) was 0.919 and the fine grinds (9min) was 0.964. This analysis suggests that there were indeed residual fractures in the microwave-treated material following crushing and that the Work Index may be reduced by approximately 4-8% across typical grind sizes for flotation following microwave treatment at approximately 2.1kWh/t. For reference, a relative Work Index of 1 (i.e. no residual fractures) would have resulted in P_{80}'s of approximately 267µm and 171µm for the microwave-treated 6.1min and 9min grinds respectively.

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3.3 Liberation analysis

The mass distributions and copper sulphide assays by size class from the liberation analysis are presented in Table 4. It can be seen that the head grades are very similar for the ~30kg untreated and microwave-treated samples, which demonstrates good sampling and comparable samples on a copper sulphide content basis. The grades across the size classes are also quite similar; therefore, any change in the liberation behaviour will be largely dependent on the liberation characteristics of the individual size classes rather than any significant redistribution of copper sulphides to other size classes for an equivalent grind size.

Table 4

Coarse and fine grind copper sulphide modal abundance

Size Class	Fine Grind				Coarse Grind			
	Untreated		Treated		Untreated		Treated	
	Mass	Copper Sulphide	Mass	Copper Sulphide	Mass	Copper Sulphide	Mass	Copper Sulphide
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
+425µm	-	-	-	-	5.1	1.28	1.0	1.09
-425+300µm	2.0	1.33	0.8	0.91	13.2	1.42	9.0	1.40
-300+212µm	11.6	1.75	8.1	1.65	12.7	2.25	14.6	1.99
-212+150µm	13.8	2.12	14.2	2.29	8.9	2.79	10.9	2.56
-150+106µm	11.5	2.71	12.8	2.55	7.1	2.96	8.3	3.32
-106+75µm	8.4	3.07	9.8	3.47	5.8	2.94	6.8	3.12
-75+53µm	6.9	3.14	8.3	3.16	4.9	3.71	6.1	3.93
-53+38µm	6.4	3.50	6.4	3.92	4.7	3.66	5.1	3.51
-38µm	39.3	2.66	39.6	2.99	37.7	2.64	38.2	2.72
Total	100.00	2.58	100.00	2.83	100.00	2.51	100.00	2.65

The cumulative liberation profiles of the four grinds are given in Figure 7. It is readily seen that the finer grinds have a higher degree of liberation than their corresponding coarse grinds, as would be expected. It can also be seen that microwave-treated fine and coarse grinds have a higher degree of liberation than their corresponding untreated grinds; however, the difference in grind sizes must also be taken into account. Figure 8 gives the proportion of copper sulphide >80% liberated by grind size and shows that the microwave-treated material yields approximately 2.5% more liberated copper sulphide across a grind size P_{80} range of about 150-300µm.

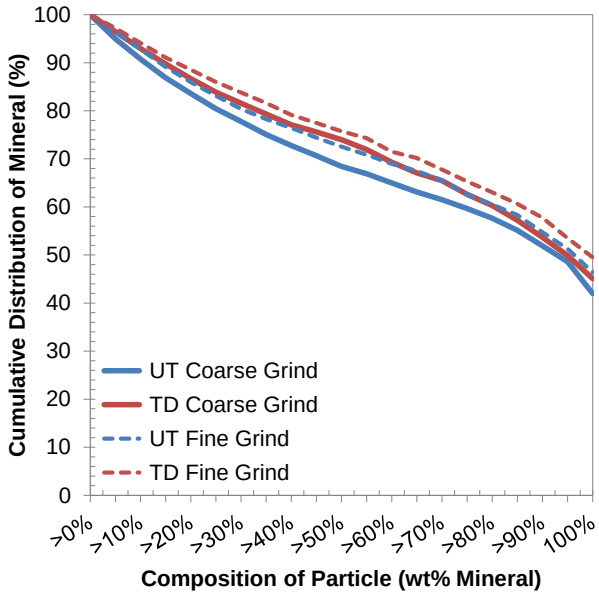

Figure 7: Cumulative copper sulphide liberation by composition profiles

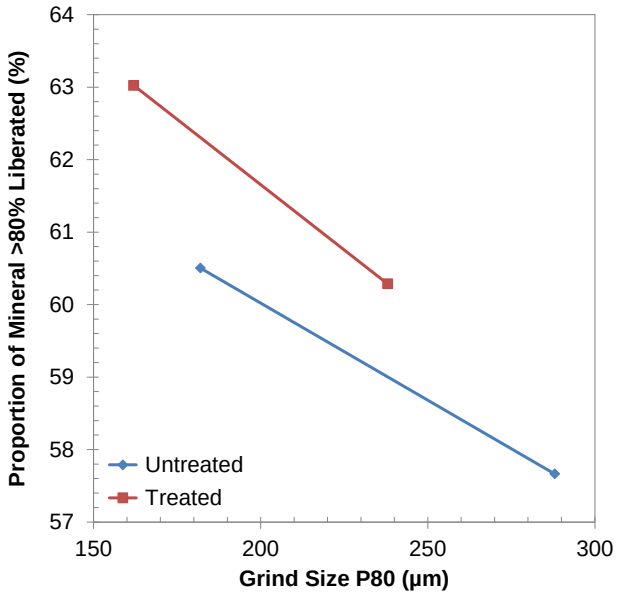

Figure 8: Copper sulphide liberation versus grind size

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Furthermore, it is evident that the microwave-treated coarse grind at P_{80} 238µm has a degree of liberation roughly equivalent to the untreated fine grind at P_{80} 182µm. Therefore, it is apparent that microwave treatment has allowed for an equivalent degree of liberation at approximately a 50-60µm increase in grind size. The apparent increase in copper sulphide liberation may be attributed to preferential breakage around grain boundaries at the location of microwave-induced fractures.

Figure 9 and Figure 10 give the copper sulphide liberation by individual size class and absolute distribution of copper sulphides by size and liberation class respectively for the untreated fine grind versus the microwave-treated coarse grind. Figure 9 shows that the microwave-treated sample has a slightly higher degree of liberation (>50% liberation class) in all but the +300µm size fractions. However, Figure 10 shows that the microwave-treated coarse grind has less copper sulphide in the -212µm fractions with more copper sulphide in the +212µm fractions due to the coarser grind size. Overall, the higher degree of liberation coupled with the redistribution of copper sulphide mineral between the two grinds yields the similar overall liberation.

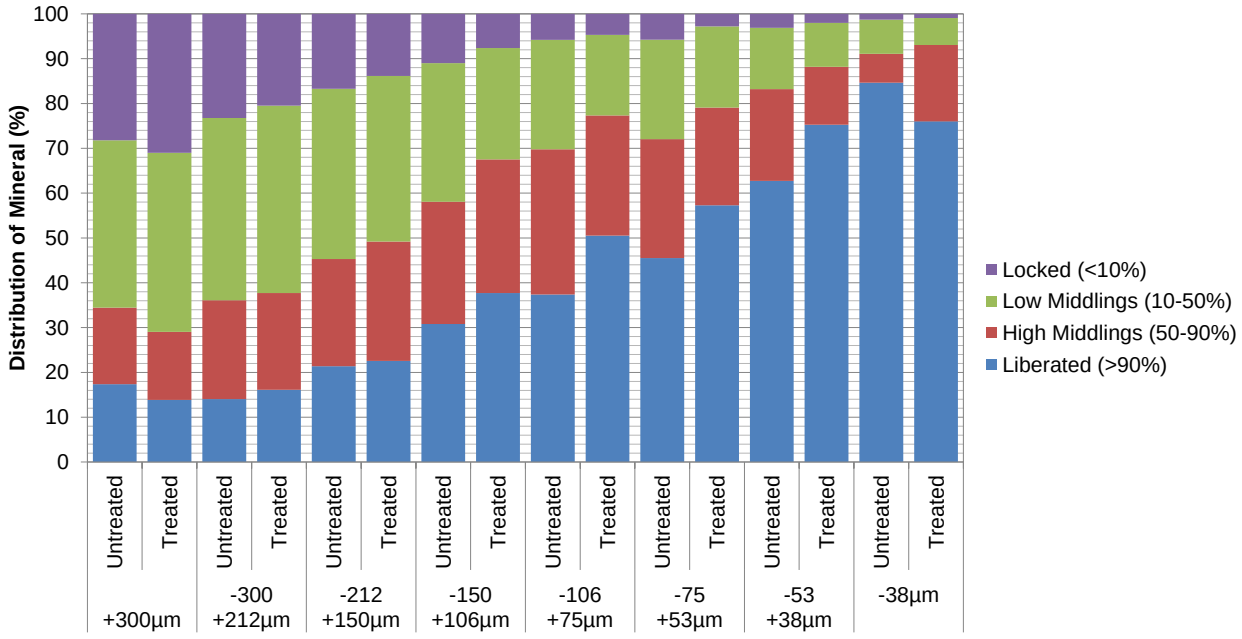

Figure 9: Distribution of copper sulphide by liberation class scaled by size fraction for the untreated fine grind vs the microwave-treated coarse grind

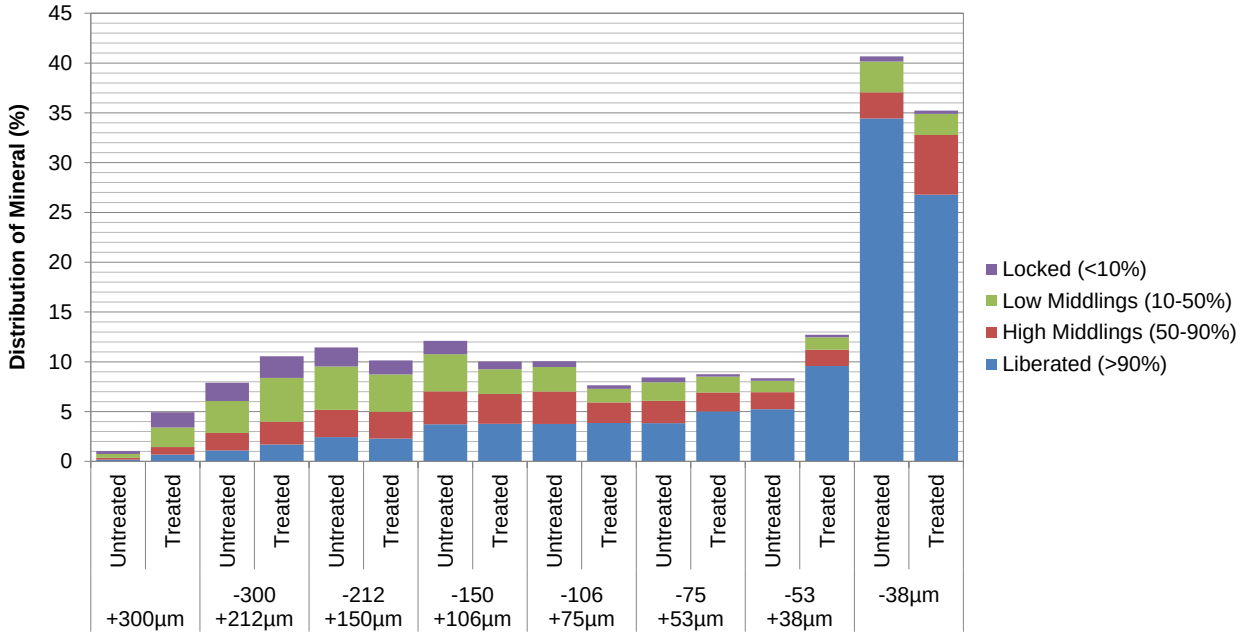

Figure 10: Absolute distribution of copper sulphide by size fraction and liberation class for the untreated fine grind vs the microwave-treated coarse grind

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Copper sulphide liberation is of critical importance to flotation and Figure 11 illustrates how microwave treatment has resulted in a redistribution of copper sulphides from the poorer floating <50 wt% liberation classes to the better floating >50 wt% liberation classes. If copper recovery could be maintained in line with liberation at a grind size approximately 50-60µm coarser than the current plant grind it would have significant implications for operation of the mill. In practice, this may only be applicable if the increased grind size is still within the normal flotation range and the flotation procedure can be modified to handle the coarser particles.

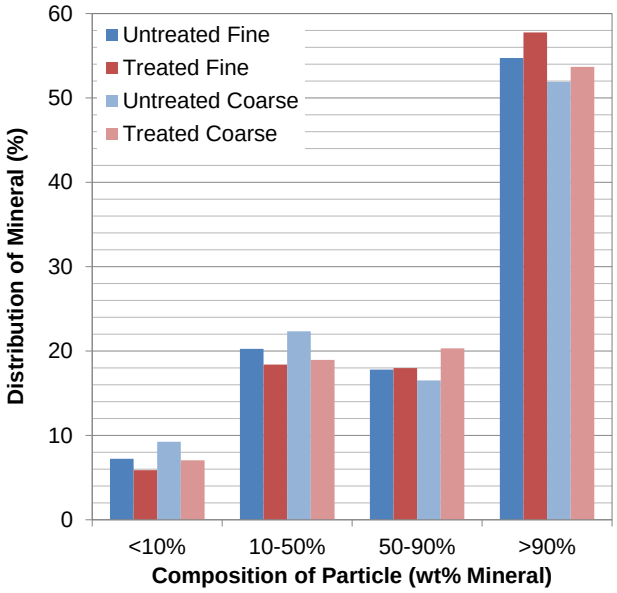

Figure 11: Absolute distribution of copper sulphide by liberation class

Figure 12a-e shows the copper sulphide associations (percentage shared boundary) with the mineral groupings of interest considered in the textural analysis. Free surface appears to be driven by grind size with little difference between untreated and microwave-treated samples. The association with hard and soft gangue also appears to be very similar between untreated and microwave-treated material. Free surface increases with decreasing grind size and association with hard and soft gangue decreases with decreasing grind size as is expected.

The apparent reduction in association of the copper sulphides with iron and other sulphides between the untreated and microwave-treated samples (~4%) may be attributed to their association in lump fragments. As mentioned previously, a large proportion of the copper sulphides appear to rim the pyrite grains or else be intimately associated together. Therefore, the copper sulphides are not only constrained by a hard or soft gangue matrix mineral, but also by a hard microwave-heating mineral providing even greater differential thermal expansion along the grain boundaries. The preferential breakage under load observed between harder and softer minerals (Djordjevic, 2013, 2014; Wang, 2015), such as pyrite and chalcopyrite, would also serve to exploit any microwave-induced weakness at the grain boundaries, resulting in enhanced liberation between the two sulphide phases.

The association of copper sulphides and other microwave-heating minerals (Fe/Ti oxides and smectite clays) was also very similar between untreated and microwave-treated material. There was little remaining association for both untreated and microwave-treated material and it appeared to be independent of grind size over the range tested. There was little association of other microwave-heating minerals with copper sulphides in the lump fragments, but it also appears that these minerals liberate well from copper sulphides in the untreated ore.

Figure 13a-e gives the iron and other sulphides associations with the mineral groupings of interest. The microwave-treated coarse grind appears to have a slightly lower free surface compared to the untreated material, which corresponds to a slightly higher association with copper sulphide minerals. This is believed to be due to their intimate association in lump fragments and preferential co-liberation during crushing and grinding from non-heating non-sulphide gangue minerals. There appears to be no other significant changes in association due to the coarse native iron sulphide grain size and high association with soft non-sulphide gangue in the lump fragments.

The ore under investigation demonstrated only modest reductions in ore competency after microwave treatment that would contribute directly to reductions in specific crushing and grinding energy requirements. However, by increasing the primary grind size, less grinding would be performed, thereby indirectly reducing the specific grinding energy requirement. Alternatively, if copper recovery can be increased at the same grind size due to a higher degree of liberation, then value would be obtained by increasing copper production.

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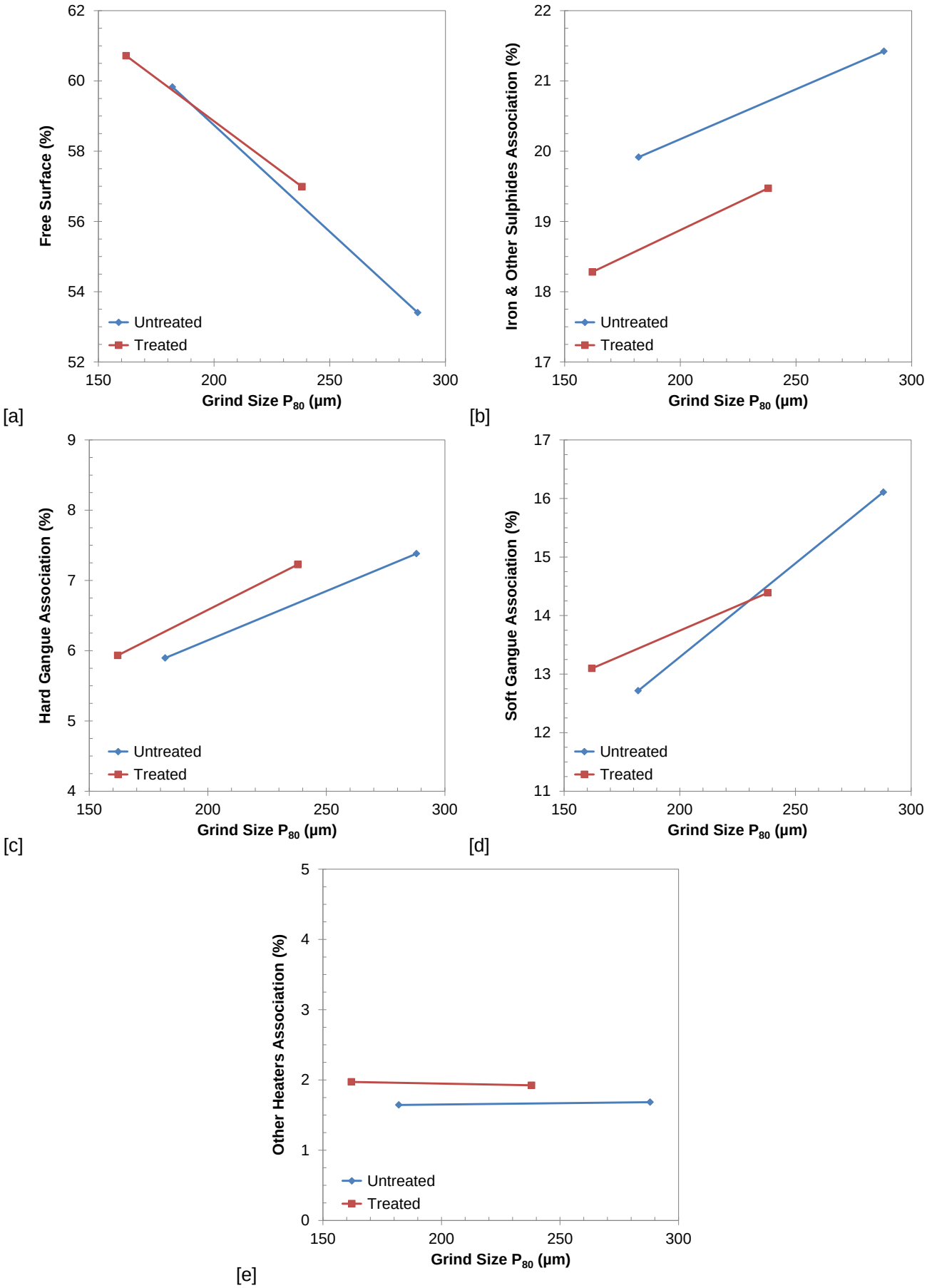

Figure 12: Change in copper sulphide associations with grind size

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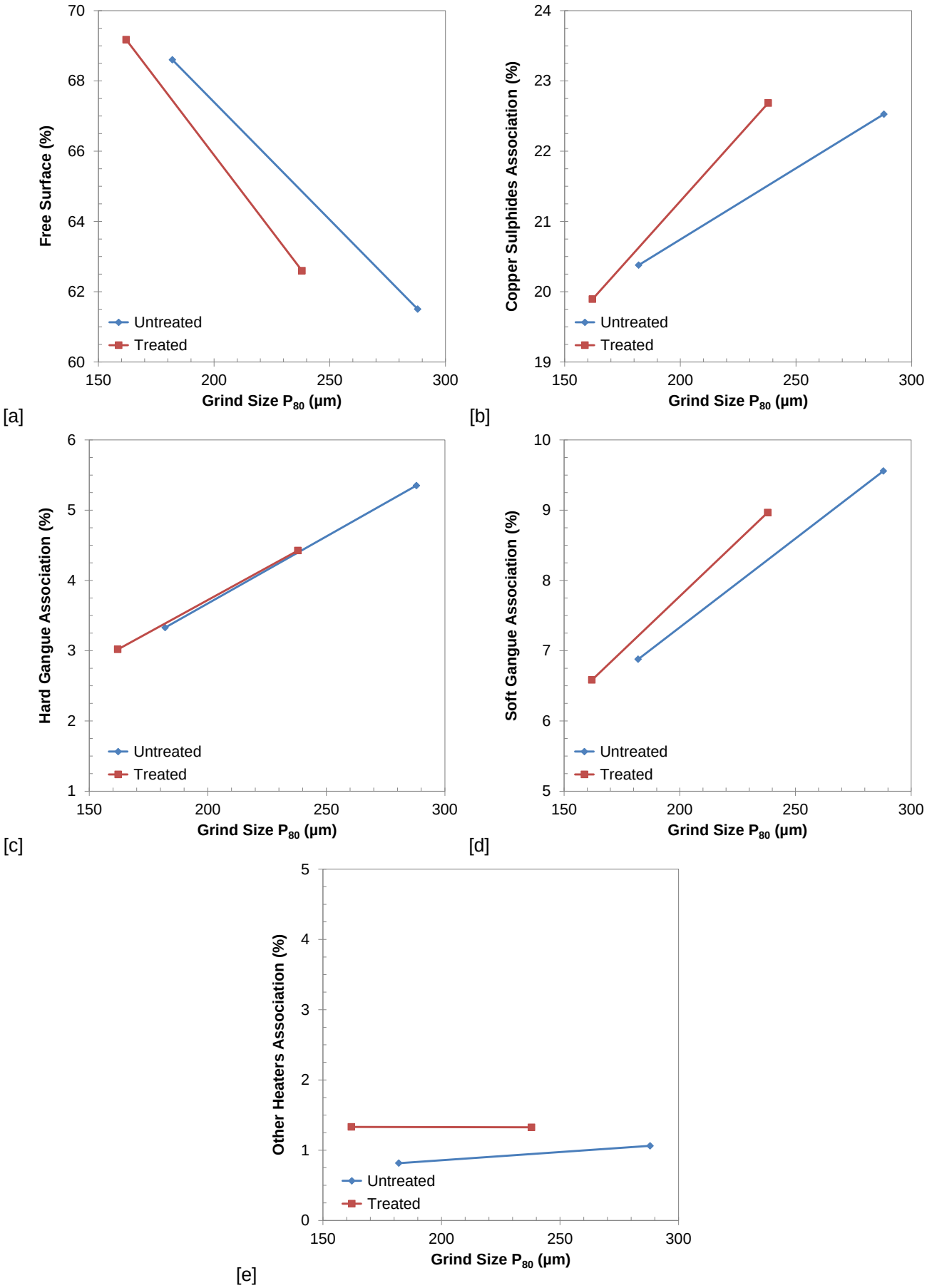

Figure 13: Change in iron and other sulphide associations with grind size

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3.4 Flotation analysis

In order to compare a microwave-treated coarse grind with a similar degree of liberation to the untreated coarse grind sample, a target P_{80} of 340µm (50µm coarser) was selected, which required a reduction in the grind time from 6.1 minutes to 5.2 minutes. Confirmation and liberation grinds suggested the untreated and microwave-treated fine grind P_{80}'s ranged from 172-183µm and 160-166µm respectively, and the untreated and microwave-treated coarse grind P_{80}'s ranged from 285-301µm and 325-349µm respectively.

Figure 14 and Figure 15 give the average cumulative copper grade-recovery curves for the fine and coarse grinds respectively, with range bars showing the maximum and minimum values obtained from the triplicate tests. Figure 14 shows that the untreated and microwave-treated fine grinds have similar curves largely within the range of experimental repeatability; however, final average copper recovery was approximately 1% higher for the microwave-treated sample. Figure 15 shows that despite the microwave-treated sample being approximately 25-65µm coarser than the untreated sample, a higher grade concentrate was obtained with a final average copper recovery approximately 1% higher than the untreated sample, just within the range of experimental repeatability.

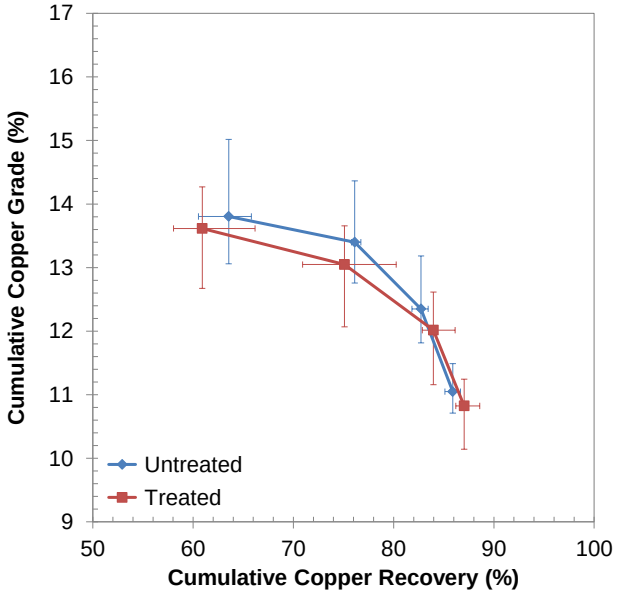

Figure 14: Fine grind grade-recovery curves

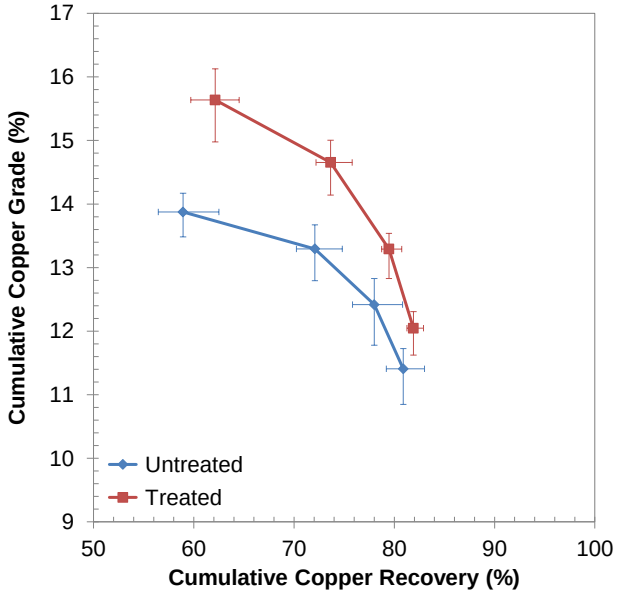

Figure 15: Coarse grind grade-recovery curves

In order to test whether the observed average increase in recovery and cumulative copper grades were statistically significant, the method proposed by Napier-Munn (2012) was employed. Cumulative grade was converted to cumulative enrichment ratio (defined as cumulative grade / head grade) to account for the differences in head grade between each sample.

T-Tests were conducted on the difference between microwave-treated and untreated sample enrichment ratio and copper recovery. The tests indicated that there was no significant difference (>90% confidence) in recovery between microwave-treated and untreated samples at any of the timed concentrates, with only an 86% and 76% confidence that the observed 1% increases in final recovery were real for the fine and coarse grinds respectively. However, given that the coarse grinds were different in grind size, this analysis suggested that equivalent recovery was possible with the microwave-treated sample at a 50µm coarser grind size. Furthermore, the first two concentrates for the coarse grinds demonstrated a significant improvement in enrichment ratio (with 98% and-94% confidence respectively), which suggested that the improved liberation allowed for recovery of higher grade particles early in the flotation process with the microwave-treated sample. The mean, standard deviation and 90% confidence intervals for enrichment ratio and recovery covering the first and last concentrates are given in Table S.1 and Table S.2 respectively in the Supplementary Information. Details of the T-Tests are given in Table S.3 in the Supplementary Information.

The flotation kinetics were modelled after Klimpel (1980) in Eq. (4) rather than the simple first order rate equation used by Napier-Munn as it provided a better fit to the experimental data in these investigations:

$$R = R_{max}\left(1 - \frac{1}{kt}(1 - e^{-kt})\right) \qquad 4$$

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Where R is the cumulative recovery (%) at a given time, t (min), and R_{max} and k, the first order rate constant (min^{-1}), are parameters to be determined from regression.

The fitted kinetics model parameters and regression performance are given in Table 5, with the fine and coarse grind kinetics curves given in Figure 16 and Figure 17 respectively. A bootstrap analysis was conducted with 1,000 replicates and the difference between the untreated and microwave-treated parameters compared. Only the difference in the rate constant for the fine grinds demonstrated a significant difference with 95% confidence, suggesting that the kinetics of the microwave-treated material was similar to the untreated material. Full details of the kinetics modelling difference tests are given in Table S.4 in the Supplementary Information.

Table 5

Flotation kinetics modelling statistics

Quantity	Fine Grind		Coarse Grind	
	Untreated	Treated	Untreated	Treated
R_{max} Fitted Value	88.01	89.86	83.20	83.96
k Fitted Value	3.534	2.968	3.388	3.813
Standard Error of Fit	1.40	3.13	2.41	1.58
Coefficient of Determination (R^2)	0.978	0.926	0.936	0.965

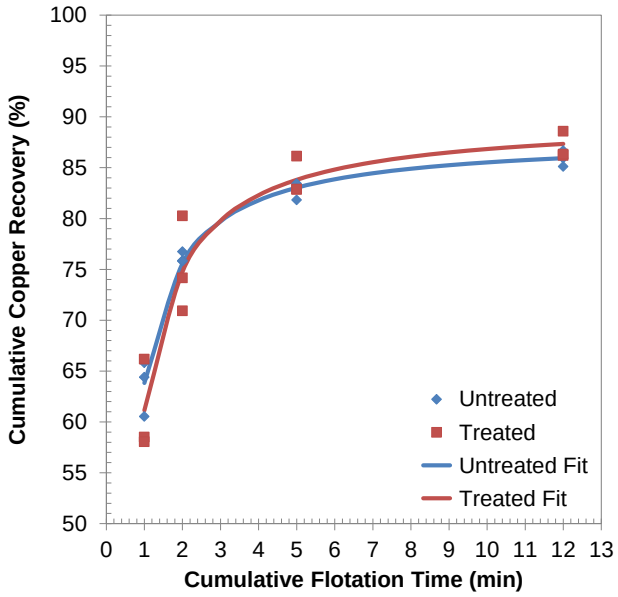

Figure 16: Fine grind copper recovery kinetics curves

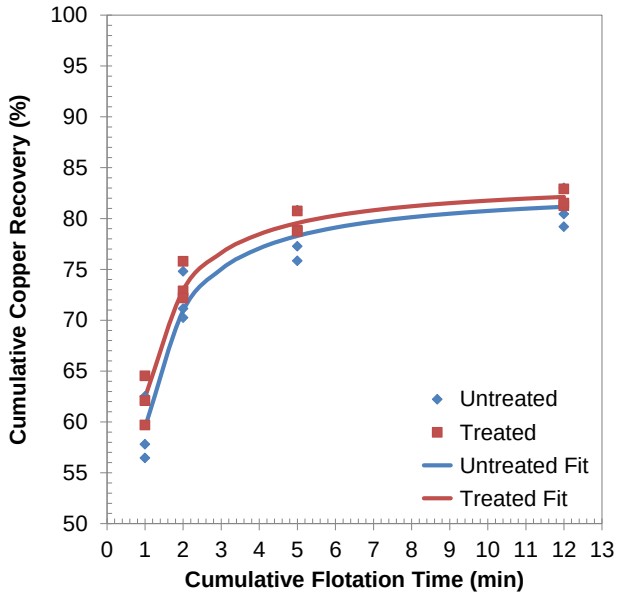

Figure 17: Coarse grind copper recovery kinetics curves

The grade-recovery curves, in the form of enrichment ratio-recovery curves, were modelled after Bruey (Napier-Munn, 2012) in Eq. (5), although the model proposed by Vera et al. (2000) in Eq. (6) also provided virtually identical curves:

$$R = R_{max} - e^{(10-c)} \sinh\left(\left(\frac{ER}{ER50}\right)\sinh^{-1}\left(\frac{R_{max}-50}{e^{(10-c)}}\right)\right) \quad 5$$

$$R = R_{max} - a \cdot \sinh\big(b(ER-1)\big) \quad 6$$

Where R is the cumulative recovery (%), ER is the cumulative enrichment ratio, and R_{max} (theoretical maximum recovery, ≤100%), $ER50$ (the value of cumulative ER for which the cumulative recovery is 50%), a, b and c are parameters to be determined from regression.

The fitted model parameters and regression performance are given in Table 6. The cumulative enrichment ratio versus copper recovery curves for the fine grinds and coarse grinds are given in Figure 18 and Figure 19

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respectively. The fine grind curves are very similar, whereas the coarse grind curves show a clear difference, as was demonstrated in the cumulative grade-recovery curves.

It appeared that one of the microwave-treated fine grind flotation tests yielded lower than expected enrichment ratios for the copper recovery values obtained (the four left-most squares in Figure 18). The model fitting analysis was therefore also run with this test removed (i.e. the microwave-treated fine grind test model fitting analysis was performed in duplicate as well as in triplicate) to determine if considering the test to be an outlier would change the confidence in whether the two curves were different.

Table 6

Flotation grade-recovery modelling statistics

Quantity	Fine Grind			Coarse Grind	
	Untreated	Treated (Triplicate)	Treated (Duplicate)	Untreated	Treated
R_{max} Fitted Value	100.00	100.00	94.58	86.72	87.68
c Fitted Value	9.77	9.49	13.37	14.59	12.64
ER50 Fitted Value	8.78	8.74	8.43	8.46	9.34
Standard Error of Fit	5.43	8.22	4.71	3.62	2.34
Coefficient of Determination (R^2)	0.667	0.487	0.991	0.856	0.924

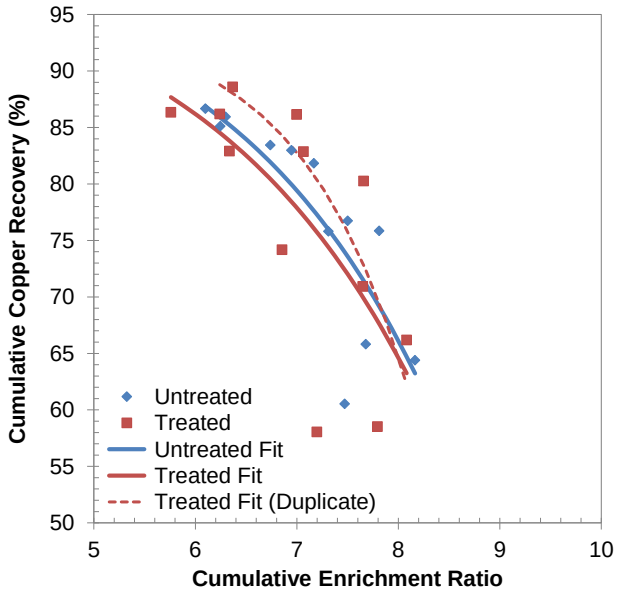

Figure 18: Fine grind enrichment ratio-recovery curves with fitted Bruey model

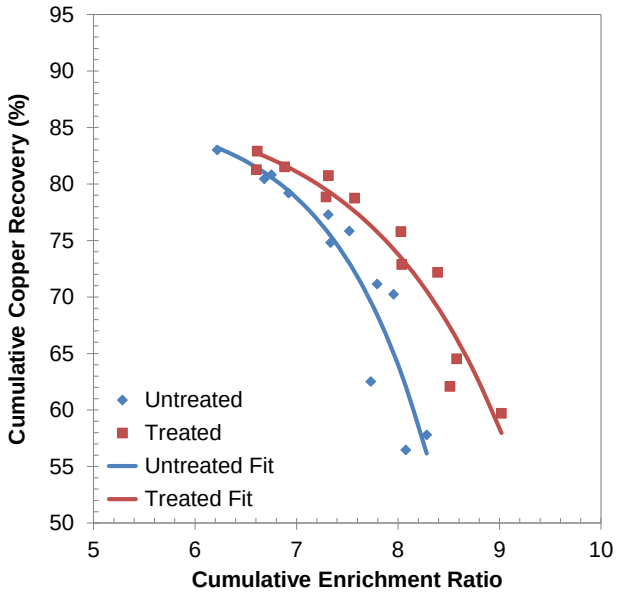

Figure 19: Coarse grind enrichment ratio-recovery curves with fitted Bruey model

A bootstrap analysis was conducted with 1,000 replicates and the difference between the untreated and microwave-treated parameters compared. Only the difference in the ER50 value for the coarse grinds demonstrated a significant difference (with >99% confidence), which supports the higher grade achieved in the early concentrates following microwave treatment. The other model fitted parameters were within the range of standard error from replicate testing. The full details of the enrichment ratio-recovery modelling difference test are given in Table S.5 in the Supplementary Information.

The fitted curves with the 95% confidence limits determined from the bootstrap analysis are given in Figure 20 and Figure 21 for the fine and coarse grinds respectively. It can be seen that the difference between untreated and microwave-treated fine grinds are within these limits. However, for the coarse grinds, the curves are statistically different over the early concentrates. A statistical analysis was performed on predicted recovery values for given enrichment ratio values from the fitted model parameters. The approximate 10% higher recovery in the coarse grind early concentrates at ER=8 after microwave treatment is highly significant with >99% confidence, where the faster floating more highly liberated material would be recovered first. However, the approximate 1.5% higher recovery at ER=6.7 achieves only 79% confidence. Full details of the predicted recovery values are given in Table S.6 in the Supplementary Information.

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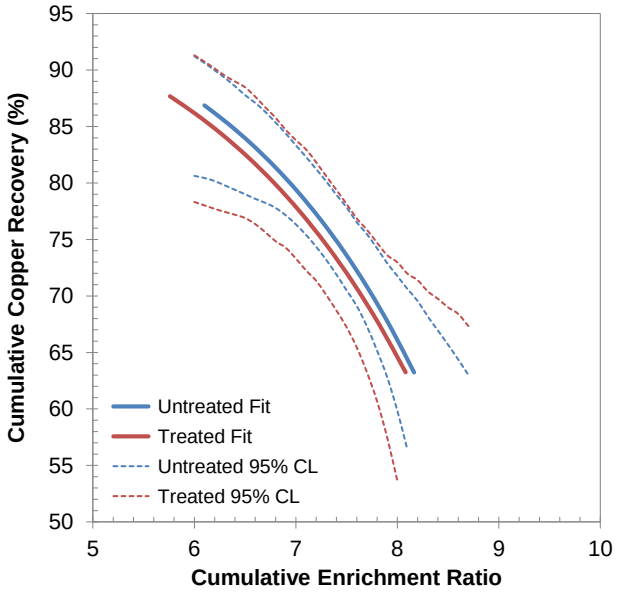

Figure 20: Fitted fine grind enrichment ratio-recovery curves with 95% confidence limits

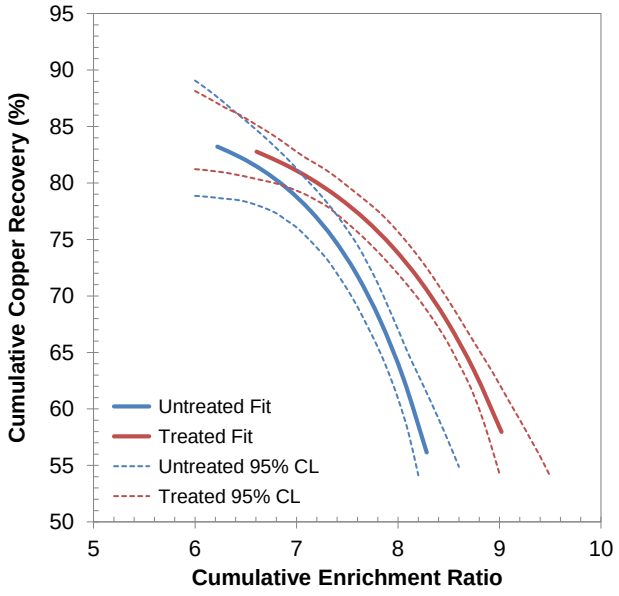

Figure 21: Fitted coarse grind enrichment ratio-recovery curves with 95% confidence limits

The previous statistical analysis has focused on determining the confidence in differences over particular ranges of the untreated and microwave-treated curves. To determine if the two curves are different an F-Test may be performed, which compares the residual sum of squares for the separate fits of the two data sets to that obtained by fitting the same model to the combined data set (i.e. the global fit). The F-Test statistic, *F*, may be determined by Eq. (7):

$$F = \frac{(SS_1 - SS_2)/(DF_1 - DF_2)}{(SS_2/DF_2)} \qquad 7$$

Where the subscripts *1* and *2* define the global fit and 2-fit models respectively, *SS* is the residual sum of squares for the model fits and *DF* are the degrees of freedom (defined as the number of data points (*n*) minus the number of parameters (*p*)).

The results of the F-Test showed that the curves for the fine grinds are statistically the same, achieving only 3% to 19% confidence, for the triplicate and duplicate analysis respectively, that the two curves are different. However, there is great confidence (>99%) that the two curves for the coarse grinds are indeed different. Full details of the F-Test are given in Table S.7 in the Supplementary Information.

The statistical analysis was heavily influenced by the repeatability of the batch flotation tests in these investigations, particularly for the fine grinds. The analysis also suggested that, given the standard deviation of recoveries obtained, at least six repeats would be required to improve the robustness of the averaged flotation results when attempting to prove a recovery difference of 1% at 90% confidence. In addition, the difference in grind sizes between the untreated and microwave-treated samples may have influenced the result as the grind sizes were not directly comparable.

Figure 22 shows the copper recovery versus grind size P_{80} for both untreated and microwave-treated samples. The average results appear to suggest that improved liberation due to microwave treatment has given rise to higher copper recovery during flotation. However, given the degree of repeatability and statistical uncertainty with the limited number of tests, and the lack of optimisation of the flotation procedure for tests approximately 100µm coarser than the plant grind at the very limits of the normal flotation range, the results must be interpreted with caution and may not be considered conclusive. An intermediate grind size would also be required to better characterise the recovery versus grind size relationship.

Nevertheless, if the average results are considered to be indicative of actual performance then it can be calculated that at the plant grind of P_{80} 190µm the microwave-treated sample may achieve an approximate 0.8% increased recovery, from 85.4% to 86.2%. Alternatively at a fixed copper recovery value of 85.4%, the microwave-treated sample may achieve the same recovery at a grind size P_{80} of approximately 219µm, 29µm greater than the nominal plant grind. The increase in copper recovery due to microwave treatment at the coarser grind size of 219µm equates to approximately 1.3%, from 84.1% to 85.4%.

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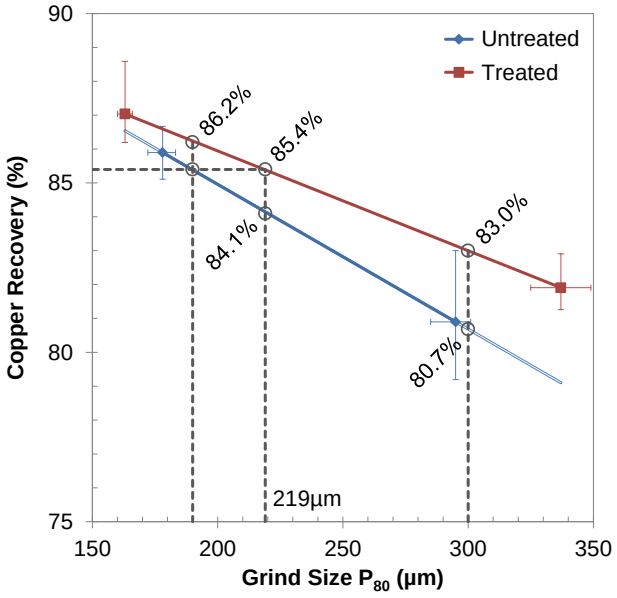

Figure 22: Copper recovery versus grind size

Plant grind sizes constantly change with differing feeds and operating conditions, typically leading to reduced recovery when the grind size becomes coarser as a result of poorer liberation and/or flotation performance. By adopting microwave treatment prior to milling, it has been demonstrated that higher recoveries may be obtained when the grind size is subject to inherent fluctuations or targeted at a coarser size.

3.5 Discussion

A summary of the pertinent statistical confidence levels from the previous analysis are given in Table 7. It is evident from the flotation data presented that the potential benefits of enhanced liberation due to microwave-induced fracture were less pronounced and less statistically significant at finer grinds that approached the nominal plant grind. This is believed to be due to the increasing state of liberation of the minerals of interest, in this case the copper sulphides, as the grind size reduces past the native grain size of the minerals of interest and more mineral becomes recoverable during flotation. In other words, the more highly liberated the material the less room there is for improvement, as any preferential breakage around grain boundaries has been exhausted and further grinding diminishes observable differences between untreated and microwave-treated material. This phenomenon appears to manifest as a convergence of the grind size versus copper recovery curves at finer grinds, shown in Figure 22.

Table 7

Flotation statistics confidence levels (%) summary

Description	Quantity	Fine Grind	Coarse Grind
Difference in enrichment ratio and recovery for the final concentrate (Treated – Untreated)	Enrichment Ratio	66.2	64.5
	Recovery	85.5	76.2
Difference in kinetics model parameters (Treated – Untreated)	R_{max}	86.1	70.1
	k	94.9	88.4
Difference in enrichment ratio and recovery model parameters (Treated – Untreated)	R_{max}	53.5	51.1
	c	53.1	67.1
	ER50	50.4	>99.99
Difference in predicted recoveries at given enrichment ratios (Treated – Untreated)	ER = 6.3/6.7	61.9	79.1
	ER = 8.0	64.4	>99.999
F-Test summary		3.0	>99.9

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The importance of mineralogy and ore texture is further illustrated by comparing the work of Sahyoun et al. (2005) to this investigation, which were both performed under similar microwave treatment conditions. The copper carbonatite ore tested by the authors contained predominantly magnetite (~20-50%) and copper sulphides (~0.4%) as the microwave-heating phases constrained within a predominantly non-heating calcite, dolomite, apatite and olivine matrix. The microwave-heating phases were also quite coarse grained, particularly the magnetite with grains likely present in the order of millimetres given the modal abundance, resulting in a flotation target grind size P_{80} of 300µm for recovery of copper. The authors demonstrated that significantly higher grades were achieved in the early concentrates following microwave treatment attributed to a higher degree of liberation, which is in agreement with the findings of this investigation. However, the authors quoted final recovery increases of 3-6%, which are substantially higher than the approximate 1% increase determined during this investigation at a P_{80} of 190µm and approximate 2.3% increase predicted at an equivalent P_{80} of 300µm.

The authors omitted the -45µm fraction from their flotation experiments, which may have artificially inflated the stated recovery increase on a full size distribution basis due to removing a likely sizable mass fraction with the highest degree of liberation. However, the higher recovery in the coarser size fractions may also be attributable to the copper sulphide associations within the ore. Although not specified in the literature, the copper sulphides may have been highly associated with magnetite or at least subject to microwave-induced fracturing caused by the magnetite. Magnetite is a hard mineral and an excellent microwave heater with a high coefficient of thermal expansion, which would have aided the differential thermal expansion at the copper sulphide to magnetite grain boundaries promoting a high degree of inter-granular fracture between the calcite, magnetite and copper sulphides (Ali and Bradshaw, 2009; Djordjevic, 2014).

In contrast, the porphyry copper ore tested in this investigation appeared to retain a fairly high degree of copper sulphide association with softer non-heating gangue minerals following grinding (shown in Figure 12), which may suggest that the textural analysis overestimated the association with hard minerals in lump fragments (illustrated by Figure 2a compared to Figure 2d). Therefore, a relatively high association with soft non-heating gangue minerals may have limited the extent of grain boundary fracture, despite an appreciable association with pyrite.

In summary, when considering the potential for microwave-enhanced liberation and flotation recovery, the influence of factors such as grain size, grind size and valuable mineral associations should be well understood, as is the case for conventional processing (Tungpalan et al., 2015).

4 Conclusions

It has been demonstrated that ores which do not exhibit large reductions in average ore competency (<10%) following microwave treatment at economically feasible energy inputs (due to the presence of many barren, fine grained or soft fragments) may still exhibit significant changes in liberation due to favourable valuable-mineral mineralogical characteristics.

For the porphyry copper ore tested in these investigations, a coarse copper sulphide grain size coupled with a high association with hard microwave-transparent (i.e. quartz) and hard microwave-heating (i.e. pyrite) minerals resulted in a grind size increase of 50-60µm for equivalent liberation or an approximate 2.5% increase in liberation at an equivalent grind size.

Subsequent laboratory batch flotation testing suggested that copper recovery could be increased by up to approximately 1% at nominal plant grind sizes, or that a grind size increase of approximately 30µm may potentially yield equivalent copper recovery. Increases in grind size may allow for an indirect reduction in specific comminution energy by grinding the ore for a shorter period of time. Alternatively, an increase in copper recovery would add value to a mining operation by increasing copper production. However, statistical analyses demonstrated that it is difficult to attain confidence in recovery increases of approximately 1% by triplicate flotation testing, even when the same analyses can give confidence that the grade-recovery curves are different between untreated and microwave-treated samples.

Acknowledgements

The authors would like to thank the sponsors of the AMIRA P879a project for funding, supply of the ore materials and engagement throughout the project: Anglo American, Rio Tinto, BHP Billiton and Outotec.

http://dx.doi.org/10.1016/j.mineng.2016.05.011

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http://dx.doi.org/10.1016/j.mineng.2016.05.011

Supplementary Information

Table S.1

Flotation enrichment ratio statistics

Statistic	Fine Grind				Coarse Grind			
	Untreated		Treated		Untreated		Treated	
	Conc.1	Conc.4	Conc.1	Conc.4	Conc.1	Conc.4	Conc.1	Conc.4
Mean	7.76	6.21	7.69	6.11	8.02	6.59	8.70	6.70
Standard Deviation	0.36	0.10	0.45	0.32	0.28	0.36	0.28	0.16
90% Confidence Interval	0.60	0.17	0.76	0.54	0.47	0.60	0.47	0.27

Table S.2

Flotation recovery statistics

Statistic	Fine Grind				Coarse Grind			
	Untreated		Treated		Untreated		Treated	
	Conc.1	Conc.4	Conc.1	Conc.4	Conc.1	Conc.4	Conc.1	Conc.4
Mean	63.55	85.90	60.91	87.04	58.93	80.90	62.13	81.91
Standard Deviation	2.73	0.78	4.57	1.34	3.18	1.94	2.42	0.89
90% Confidence Interval	4.61	1.31	7.70	2.27	5.36	3.27	4.07	1.50

Table S.3

Flotation T-Test summary

Value (Treated – Untreated)	Fine Grind				Coarse Grind			
	Enrichment Ratio		Recovery		Enrichment Ratio		Recovery	
	Conc.1	Conc.4	Conc.1	Conc.4	Conc.1	Conc.4	Conc.1	Conc.4
Difference in Mean	-0.07	-0.10	-2.64	1.14	0.68	0.11	3.20	1.01
t-Value	0.22	0.51	0.86	1.27	3.00	0.48	1.39	0.82
1-sided P(t)	0.411	0.338	0.222	0.145	0.021	0.355	0.122	0.238
Confidence Level (%)	58.9	66.2	77.8	85.5	97.9	64.5	87.8	76.2

Table S.4

Flotation kinetics modelling difference test

Quantity (Treated – Untreated)	Fine Grind		Coarse Grind	
	R_{max}	k	R_{max}	k
Difference in Mean	1.84	-0.543	0.72	0.433
Standard Deviation	1.69	0.331	1.37	0.362
95% Lower Confidence Limit	-1.37	1.125	-1.95	-0.271
95% Upper Confidence Limit	5.07	0.132	3.36	1.131
z-test Statistic	1.085	1.639	0.528	1.196
1-sided P(z)	0.139	0.051	0.299	0.116
2-sided P(z)	0.278	0.101	0.597	0.232
Confidence Level (%)	86.1	94.9	70.1	88.4

http://dx.doi.org/10.1016/j.mineng.2016.05.011

Table S.5

Flotation enrichment ratio-recovery modelling difference test

Quantity (Treated – Untreated)	Fine Grind (Triplicate)			Fine Grind (Duplicate)			Coarse Grind		
	R_{max}	c	ER50	R_{max}	c	ER50	R_{max}	c	ER50
Difference in Mean	-0.83	0.41	0.01	0.02	2.16	-0.27	0.23	-2.03	0.87
Standard Deviation	9.40	5.23	0.83	8.35	4.95	0.44	8.12	4.58	0.22
95% Lower Confidence Limit	-18.58	-9.81	-1.17	-14.26	-8.35	-1.21	-15.82	-11.80	0.43
95% Upper Confidence Limit	16.94	10.58	2.04	16.96	10.69	0.51	15.87	5.69	1.32
z-test Statistic	0.088	0.079	0.010	0.002	0.437	0.604	0.028	0.442	3.929
1-sided P(z)	0.465	0.469	0.496	0.499	0.331	0.273	0.489	0.329	4.26×10^{-5}
2-sided P(z)	0.930	0.937	0.992	0.999	0.662	0.546	0.977	0.658	8.51×10^{-5}
Confidence Level (%)	53.5	53.1	50.4	50.1	66.9	72.7	51.1	67.1	99.996

Table S.6

Flotation recovery predictions at selected enrichment ratios

Quantity (Treated – Untreated)	Fine Grind (Triplicate)		Fine Grind (Duplicate)		Coarse Grind	
	Recovery at ER = 6.3	Recovery at ER = 8.0	Recovery at ER = 6.3	Recovery at ER = 8.0	Recovery at ER = 6.7	Recovery at ER = 8.0
Difference in Mean	-1.21	-2.15	3.19	-1.33	1.56	9.77
Standard Deviation	4.00	5.84	3.50	4.35	1.92	1.85
95% Lower Confidence Limit	-8.86	-13.99	-4.02	-9.97	-2.13	6.31
95% Upper Confidence Limit	6.21	9.50	9.96	7.40	5.33	13.33
z-test Statistic	0.302	0.369	0.914	0.306	0.809	5.289
1-sided P(z)	0.381	0.356	0.180	0.380	0.209	6.15×10^{-8}
2-sided P(z)	0.763	0.712	0.361	0.760	0.419	1.23×10^{-7}
Confidence Level (%)	61.9	64.4	82.0	62.0	79.1	>99.999

Table S.7

Flotation F-Test summary

Quantity	Fine Grind (Triplicate)			Fine Grind (Duplicate)			Coarse Grind		
	Global (1)	2-Fit (2)	Diff. (1-2)	Global (1)	2-Fit (2)	Diff. (1-2)	Global (1)	2-Fit (2)	Diff. (1-2)
Residual Sum of Squares, *SS*	983.1	969.9	13.2	551.5	516.5	35.0	506.8	185.6	321.2
Number of Data Points, *n*	24	24	-	20	20	-	24	24	-
Number of Parameters, *p*	3	6	-	3	6	-	3	6	-
Degrees of Freedom, *DF*	21	18	3	17	14	3	21	18	3
F-Test Statistic, *F*	-	-	0.082	-	-	0.316	-	-	10.386
1-sided P(F)	-	-	0.969	-	-	0.813	-	-	3.41×10^{-4}
Confidence Level (%)	-	-	3.0	-	-	18.7	-	-	99.97

http://dx.doi.org/10.1016/j.mineng.2016.05.011