

Spatial variations in leaching of a low-grade, low-porosity chalcopyrite ore identified using X-ray μ CT

Authors

Marijke A Fagan-Endres* ^{a,b}

marijke.fagan-endres@uct.ac.za

Johannes J Cilliers ^c

Andrew J Sederman ^a

Susan TL Harrison ^b

^a Department of Chemical Engineering and Biotechnology, University of Cambridge, Pembroke
Street, Cambridge, CB2 3RA, UK

^b Centre for Bioprocess Engineering Research, Department of Chemical Engineering, University of
Cape Town, Rondebosch, 7701, South Africa, Tel: +27 21 6501806

^c Department of Earth Science and Engineering, Royal School of Mines, Imperial College London,
SW7 2AZ, UK

* Corresponding author

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ABSTRACT

This study presents an investigation, using 3D X-ray micro computed tomography (μ CT), into the effect of sulfide mineral position within an ore particle on leaching efficiency. Three sections of an unsaturated mini-leaching column that had been packed with agglomerated low-grade, low-porosity chalcopyrite ore and leached with an acidified ferric iron solution were imaged at different stages of a 102 day experiment. Image analysis was used to quantify changes in the mineral content and the influence on this of the mineral distance from the ore particle surface, local voidage and radial position within the column. The main factor affecting the mineral recovery was identified to be proximity of the mineral to the ore particle surface, with recovery decreasing with increasing distance from the ore surface. A maximum leaching penetration was observed to exist at 2 mm from the surface, beyond which no recovery was achieved. Higher recoveries at the column wall indicated that preferential flow in this higher voidage had an additional, albeit smaller, impact on leaching efficiency.

Keywords: Leaching, X-ray μ CT, surface leaching, hydrology, penetration, chalcopyrite

34 **1. INTRODUCTION**

35 Heap leaching is a low-cost mineral beneficiation method, commonly used to process low grade sulfidic
36 mineral ores. In this unsaturated process, mineral recovery is facilitated when ferric ions and protons in
37 the lixiviant come into contact and react with the mineral sulfide. This liberates the metal into solution
38 whereby it is transported out of the heap. A key advantage of the process over conventional extraction
39 techniques is that cost and energy requirements are decreased by the absence of fine milling of the ore,
40 with heaps typically constructed with coarsely crushed (≤ 19 mm (Brierley, 2008)) or run-of-mine
41 (ROM) ore. However, this means that some mineral remains located within the ore particles, distant
42 from the surface. Because contact between the lixiviant and mineral is required for leaching, unexposed
43 minerals may be slow to leach or unrecoverable. This issue consequently has the potential to limit heap
44 recoveries and so should be taken into account in the design of heap operations. Investigation into this
45 micro-scale phenomenon has previously been limited by the available traditional ‘black-box’ heap
46 monitoring techniques, but recent development of micro-scale 3D imaging procedures for leaching
47 systems now makes a comprehensive study possible.

48 X-ray micro computed tomography (μ CT) is a well-established technique for the 3D non-invasive, non-
49 destructive imaging of opaque samples. Though primarily developed and used as a medical imaging
50 technique, it can be a useful tool for the study of chemical engineering systems. Already in the field of
51 heap bioleaching it has been used in various studies. On a macro-scale Lin et al. (2005) and Yang et al.
52 (2008) used X-ray μ CT to study voidage changes that resulted from the slumping of ore beds. It has
53 also been used on the micro-scale to monitor the structure of ore particles and mineral grains. These
54 micro-scale studies are possible because the mineral grains have a higher X-ray absorbance than the
55 supporting gangue material and so appear as brighter areas in the images. Miller et al. (2003) used this
56 principle to study the effect that the crush size of the ore has on the distribution of exposed versus
57 internal mineral grains. The findings were related to the recoveries achieved when different particle size
58 samples were leached. They proposed that such studies could be used in the future to identify the
59 optimal crush size of the ore with respect to mineral recovery. More recently, Ghorbani et al. (2011a,b)
60 used X-ray μ CT to study crack and mineral dissemination in sphalerite ore particles in a fully submerged
61 system. Their porosity measurements compared well with traditional methods and they found that X-
62 ray μ CT was a more robust tool for the measurement of the spatial distribution of surface and interior
63 micro-cracks in large ore particles than physical gas adsorption and porosimetry methods. They
64 subsequently used their method to study the effect of comminution (crushing) devices, specifically high
65 pressure grind rolls (HPGR) and conventional cone crushers, on the generation of cracks in the ore.
66 Kodali et al. (2011) performed a similar X-ray μ CT study, which compared HPGR to jaw-crushing.
67 Through analysis of the images they determined that HPGR caused more particle damage, which
68 resulted in higher copper recoveries compared to ore of the same particle size that had been prepared
69 using the other technique. Like Miller et al. (2003), they found that grain exposure and consequently

70 mineral recovery decreased with an increase in particle size. Lin and Garcia (2005) used X-ray μ CT to
71 study the evolution of specific mineral grains over the course of a 67 day acid leach. They also
72 determined the speciation of the minerals within the ore by determining the density and the effective
73 atomic number of the minerals present in the ore and comparing the results to that of standard samples.
74 Ghorbani et al. (2013) and Lin et al. (2016a, 2016b) have used 3D X-ray μ CT coupled with kinetic
75 models to interrogate the oft-used assumption of the shrinking core model applicability to describe
76 leaching kinetics from ore particles, instead finding that the spatial and size distribution of the grains
77 do impact the apparent leach kinetics.

78 The aim of this paper is to build on these studies and relate the recovery of mineral in a long term ferric
79 leach of a low-grade, low-porosity chalcopyrite ore sample under unsaturated flow conditions to both
80 the location of the mineral grains within the ore particles as well as their position within the column,
81 thereby interrogating the ability of leaching solution to access and therefore recover value from sub-
82 surface mineral grains in ore particles.

83

84 **2. EXPERIMENTAL**

85 **2.1. Ore column preparation**

86 Chilean Escondida low grade copper ore was used whose bulk sample mineralogy was 0.69% Cu,
87 2.95% Fe, 2.02% S by weight. The gangue comprised predominately of quartz, muscovite and kaolinite.
88 The sulfide minerals were mainly present as pyrite (4 wt%) and copper sulfides (1 wt%), with the copper
89 found mainly as chalcopyrite (29%), covellite (26%), chalcocite (25%) and bornite (12%). The overall
90 particle size distribution (PSD) is given in Table 1 and the average internal porosity was 4.6%.

91 The ore was acid agglomerated as described by van Hille et al. (2010) and packed into a mini-column
92 which was 220 mm high (total external) and had an internal diameter of 23.5 mm. A layer of filter paper
93 followed by mesh was laid between the irrigation point and the ore to ensure that the liquid distribution
94 was uniform across the column width. Ball bearings were attached to the outside of the column for
95 image registration (alignment). The column was drip irrigated with a ferric solution of 5 g L⁻¹ Fe³⁺ (in
96 the form of Fe₂(SO₄)₃) in 0.1 M H₂SO₄ (pH 1.5) from the top at a rate of 4 mL h⁻¹ (approximately 9 L
97 m⁻² h⁻¹) and operated at ambient temperature for a period of 102 days.

98

99 Table 1. PSD of the low grade copper ore.

Size (mm)	Weight (%)
> 16.0	6.7
8.00 – 16.0	4.6
5.60 – 8.00	47.5
2.00 – 5.60	9.4
1.18 – 2.00	13.5

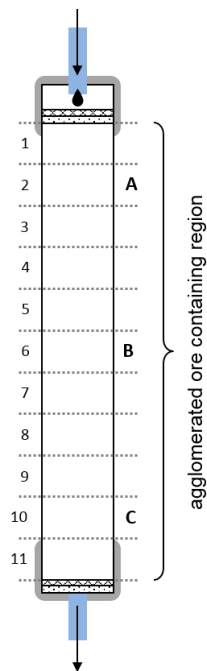
0.25 – 1.18	9.9
< 0.25	8.4

100

101 **2.2. X-ray μ CT imaging**

102 The X-ray μ CT imaging was performed on a laboratory GE Phoenix-v|tome system, with an
 103 accelerating voltage of 100kV and a current of 70 μ A. For each 3D scan 600 projections were collected
 104 at 0.6° angular increments on a 1015 \times 512 pixel detector. The targeted image resolution was 34
 105 μ m/pixel. Filtered back projection reconstruction was performed using the GE Phoenix proprietary
 106 software.

107 The entire ore containing region of the column was imaged before leaching was commenced. It was
 108 imaged in 11 separate sections of approximately 17.5 mm height, from top (1) to bottom (11) as shown
 109 in Figure 1, because a smaller field of view (FOV) permitted a higher image resolution. Three sections
 110 (2, 6 and 10) were selected to be monitored over the leaching period. The sections were chosen as they
 111 were spread out along the column length and initial visual inspection identified them as containing ore
 112 particles of suitable sulphide mineral content and distribution for the study. Sections 6 and 10 in
 113 particular contained large particles of interest. The sections will be henceforth referred to as section A
 114 (2), B (6) and C (10). The column was briefly taken off line (irrigation paused) to be imaged on day 0,
 115 1, 7, 28, 40, 50, 64, 78, 88 and 102. Some scans were omitted from the final analysis due to issues with
 116 the quality (noise or poor contrast) or because differences in image resolution did not allow for accurate
 117 comparison.



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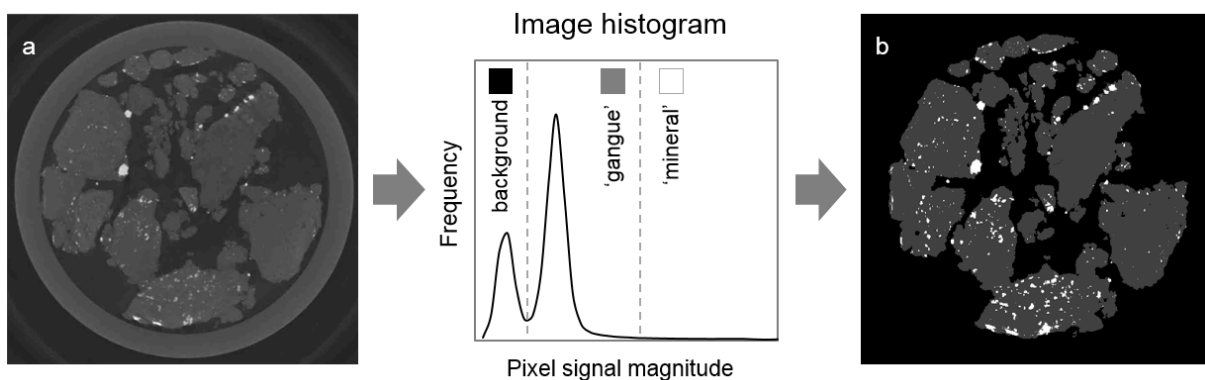
119 Figure 1. Schematic of the column, which gives the positions of the 11 imaging sections and shows the
 120 corresponding sections A, B and C which were monitored over the course of the leach experiment.

121

122 2.3. Image analysis

123 Slices 41 to 396 of the 512 slice acquisition were selected for analysis, thereby omitting boundary
124 information that contained ring artefacts. ImageJ (Schneider et al, 2012) was used for the initial slice
125 selection and to convert the images from 32 bit real to 8 bit images to allow for faster image analysis.
126 The remainder of the image analysis was performed using MATLAB® (MATLAB 7.8, 2009). The
127 images were thresholded on 2 signal magnitude levels based on a histogram analysis, as illustrated in
128 Figure 2. The lower threshold level was used to define the total solid ore region relative to the image
129 background. This lower threshold level was readily identified and easy to implement. The upper
130 threshold level was used to define the sulfide mineral position within the ore. The correct threshold
131 level to select for the sulfide minerals was more difficult to identify due to the smaller relative difference
132 in the X-ray absorption by the gangue and sulfide minerals. To account for this, pixels with a signal
133 magnitude one unit more or less than the upper threshold level were included in the analysis as an error
134 measurement. The sulfide minerals identified using the upper thresholding level were not further
135 speciated and it was assumed that variation in the mineral composition on aggregate (in each section)
136 was not significant. Note that for brevity, the sulfide minerals identified by the upper thresholding level
137 will henceforth be generally referred to as ‘mineral’, with the other ore components referred to as
138 ‘gangue’.

139 The resulting thresholded images, an example 2D slice of which is shown in Figure 2(b), were used to
140 quantify the voidage and the mineral content. A region of interest was defined in this calculation which
141 included everything within the walls of the column to avoid inclusion of any signal from the ball
142 bearings. A morphological thinning algorithm (Baldwin et al., 1996) was used to determine the position
143 of the mineral in the ore relative to the particle surface.



144

145 Figure 2. Example of an (a) original X-ray μ CT slice from section A on day 0 whose histogram is used
146 to create (b) its thresholded equivalent, thereby identifying the position of the sulfide ‘mineral’ (white)
147 and the gangue (grey) while excluding information from outside the ore containing region. Note that

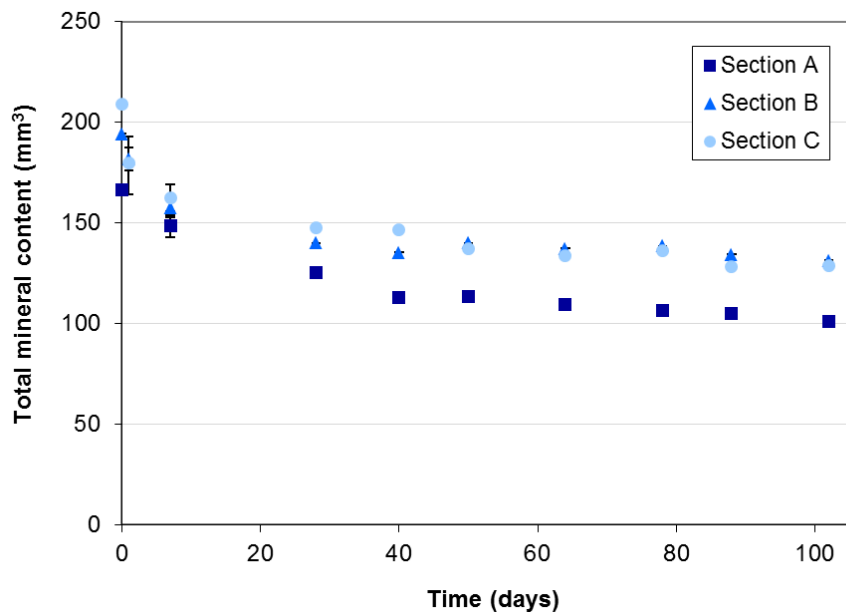
148 the frequency of the sulfide mineral pixels is much lower than for the other regions, making their
149 selection comparatively more difficult.

150

151 3. RESULTS AND DISCUSSION

152 3.1. Overall sulfide mineral leaching

153 The volumetric mineral content changes over time in the three sections of the column as calculated from
154 the thresholded 3D acquisitions are shown in Figure 3. The lowest initial total mineral content was
155 recorded in section A (166 mm^3) while sections B and C started with similar higher mineral volumes
156 (194 and 209 mm^3 respectively). The majority of the leaching in all of the sections occurred within the
157 first 28 days of operation, with the leaching rate decreasing with time as the more readily recoverable
158 mineral was removed. The overall mineral recovery (amount leached on a volumetric basis) after 102
159 days was $39 \pm 2\%$, $32 \pm 3\%$ and $38 \pm 4\%$ in sections A, B and C respectively. Hence slight differences in
160 the mineral leaching existed despite the three sections having been exposed to the same leaching
161 conditions. Two key factors were identified as possible causes of these differences: (1) variations in the
162 packed bed structure that would affect the liquid flow path and (2) the location of the mineral grains
163 within the ore particles.



164

165 Figure 3. Changes in total volumetric sulfide mineral content in section A, B and C over time.

166

167 3.2. Voidage

168 The voidages of the three sections were calculated from the thresholded images to be $35.6\pm 0.5\%$,
169 $34.0\pm 0.6\%$ and $31.3\pm 0.6\%$ respectively. Insignificant variation over time (considering the error margin)
170 was observed. This is slightly lower than has typically been quoted for heap leaching beds of
171 comparable PSD where the voidage is circa 40% (Bouffard and Dixon, 2001; Lin et al., 2005). This
172 deviation from the norm was because the bed was carefully packed to include as many ore particles in
173 the sample as possible. The column is also much smaller than typical beds (to allow for a higher X-ray
174 CT imaging resolution) in which a range of voidages would be expected to be present, including regions
175 of low voidage comparable to this sample. No correlation between these values and the recoveries in
176 the different sections was evident.

177

178 **3.3. Mineral position in the ore**

179 The distributions of the mineral filled voxels within the ore expressed as the distance from the ore
180 particle surface on day 0, 7, 28, 64 and 102 are shown in Figure 4. The position of the mineral voxels
181 and the final recovery as a function of their position are summarised in Table 2.

182 In the fresh ore (day 0) the majority of the mineral was positioned within 0.5 mm of the ore particle
183 surface in all three sections. A peak in the mineral population was observed at a distance of
184 approximately 0.07 mm (or 2 pixels) from the ore particle edge in all three cases, which may be
185 indicative of partial volume effects at the ore surface. In section A the mineral was located a maximum
186 of 4 mm from the ore particle surface, consistent with being centrally located in the most abundant ore
187 particle size (5.6 – 8 mm). There was a longer tail in the mineral position distribution for section B,
188 with some mineral located as far as 7.1 mm from the surface. Similarly, section C contained mineral a
189 maximum of 6.1 mm from the ore surface. The larger maximum distances in sections B and C were due
190 to large particles being contained in these regions of the column. These sections also had a larger
191 percentage of the mineral positioned further than 2 mm from the ore surface. This is in agreement with
192 Miller et al. (2003) who found that the amount of mineral exposed on the ore surface increases with
193 decreasing particle size.

194 The highest recovery was of the mineral particles immediately on the ore surface, where 70%, 73% and
195 67% of the mineral is leached over the 102 day period in sections A, B and C respectively. The mineral
196 recovery then decreased as the distance from the surface increased until there was negligible change in
197 the mineral content located further than 2 mm from the ore surface. The position of the mineral within
198 the ore therefore had a measureable impact on the mineral recovery, with the overall recovery increasing
199 as the percentage of the mineral within 2 mm of the ore surface increases.

200 The preferential leaching of surface mineral is expected since the leaching rate of mineral from ore is
201 dependent on the mineral surface area that comes into contact with the leaching solution (Petersen,
202 2010). Recent studies have reported that leaching, especially from large particles, occurs only at the

203 surface and subsurface regions in the ore particle, where subsurface refers to those mineral nodes that
 204 are accessible through cracks and pores (Ghorbani et al., 2011a). Mineral grains on or close to the ore
 205 particle surface are more likely to come into contact with the leaching solution, thereby increasing their
 206 chance of recovery whereas mineral not connected with the ore surface is more difficult to recover as
 207 they are less likely to come into contact with the leaching solution. The decreasing overall rate of
 208 mineral recovery with time may therefore be attributed to the fast depletion of readily contactable
 209 mineral at or near the ore surface. This corresponds with the findings of Lin et al. (2016), who
 210 demonstrated the prevalence of diffusion limited leaching characteristics further from an ore particle
 211 surface.

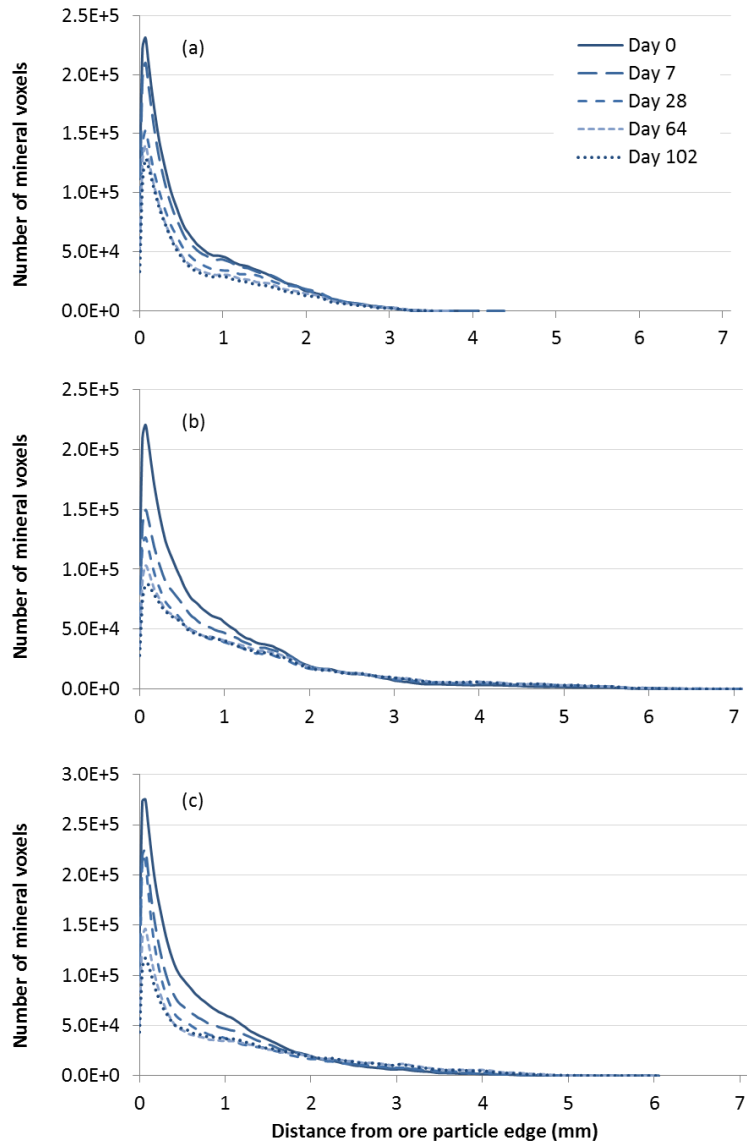
212 A greater variation in recovery with increasing distance from the ore surface was also observed. This is
 213 because the leaching of mineral further from the ore surface is more dependent on its environment, such
 214 as whether it is in contact with leachable mineral closer to the surface or is in contact with a crack.

215 This information may also be conceived as changes in the average distance of the mineral from the ore
 216 surface, as presented in Figure 5. Sections A and C had similar initial average distances, 0.72 mm and
 217 0.76 mm, while section B had an initial average value of approximately 0.94 mm. The average distances
 218 increased with time in sections B and C due to the effect of preferential leaching of the surface mineral.
 219 However, only a small change in the average distance of the mineral from the ore surface was observed
 220 for section A. This was because mineral located further than 2 mm from the surface accounted for only
 221 a small percentage in this section and the maximum distance mineral was positioned from the ore
 222 surface was significantly less than in sections B and C.

223 Table 2. Sulfide mineral distribution and recovery as a function of the distance of the mineral from the
 224 ore particle surface in the three sections.

	Section A	Section B	Section C
Mineral position on day 0			
At & < 1 mm from surface	72%	66%	70%
1 - 2 mm from surface	22%	21%	20%
> 2 mm from surface	6%	13%	10%
Mineral recovery			
At surface	70 ± 1%	73 ± 2%	67 ± 3%
< 1 mm from surface	42 ± 1%	47 ± 2%	53 ± 3%
1 - 2 mm from surface	33 ± 4%	19 ± 2%	24 ± 1%
> 2 mm from surface	< 1%	< 1%	< 1%

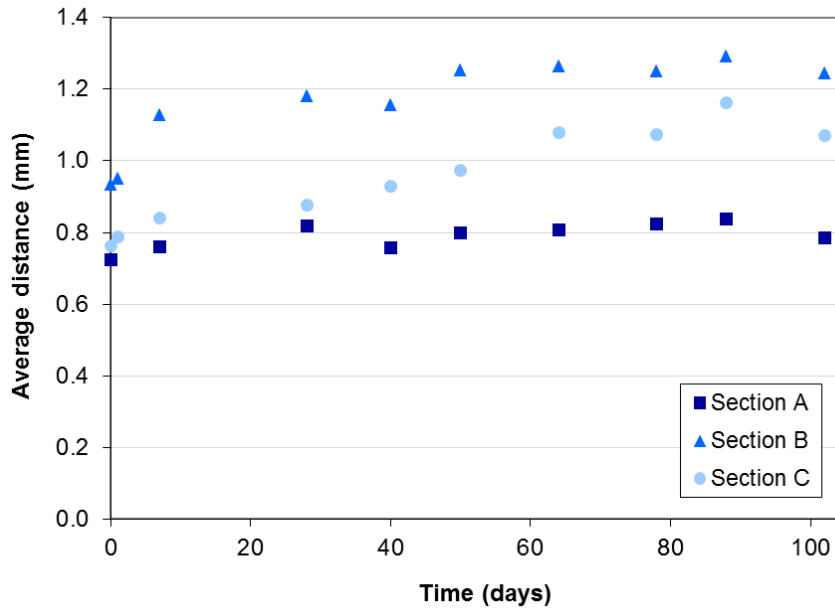
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226

227 Figure 4. Change in the distribution of the sulfide mineral filled voxels as a function of time in section

228 (a) A, (b) B and (c) C.



229

230 Figure 5. Average distance of the sulfide mineral from the edge of the ore particles as a function of time
 231 in section A, B and C.

232

233 **3.4. Radial variations**

234 The mineral leaching varied as a function of distance from the centre of the column as is shown in
 235 Figure 6. The highest recovery in all three sections occurred in the outermost 1.5 mm, next to the column
 236 wall (50%, 50% and 58% in A, B and C respectively). The recovery decreases towards the centre of the
 237 column until 6.8 mm from the centre within which there is little variation.

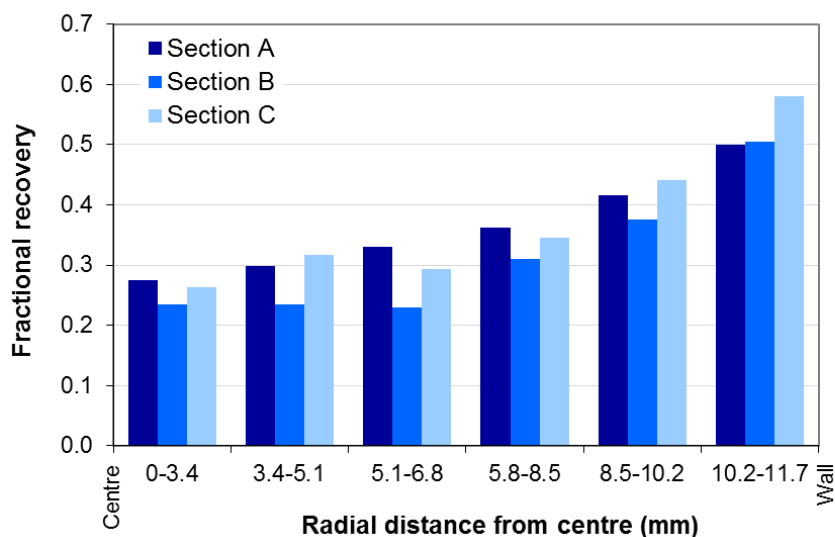
238 The voidage also varied as a function of radial distance from the centre of the column as is shown in
 239 Figure 7. This is a common characteristic of packed beds as particles must conform to the curvature of
 240 the column wall (Benenati and Brosilow, 1962). The highest voidage in all three sections was at the
 241 wall of the column (64±8%, 61±6% and 55±9% in A, B and C respectively). The voidage within
 242 10.2 mm of the centre was significantly lower, on average less than half the voidage of the outer ring.
 243 No clear trend was identifiable in this central region because variations in the voidage were caused by
 244 the random orientation of ore particles. In section B in particular there was a large standard deviation
 245 in the voidage in the central 3.4 mm of the column. This was due to the large particles known to be
 246 present in the centre of this section of the column, resulting in areas of very low and very high voidage.

247 The voidage and mineral leaching therefore follow a similar radial trend (decreasing towards the centre
 248 of the column). This may be because the higher voidage at the column walls may have caused the
 249 leaching solution to flow preferentially along the column walls, resulting in relatively inefficient
 250 leaching in the less irrigated centre of the column. This type of behaviour would be expected in a system

251 with a high flow rate (relative to the particle size distribution), when liquid will preferentially flow
 252 through a region of coarser packing (O’Kane Consultants Inc., 2000). Preferential liquid flow along the
 253 column walls was supported by visual observation.

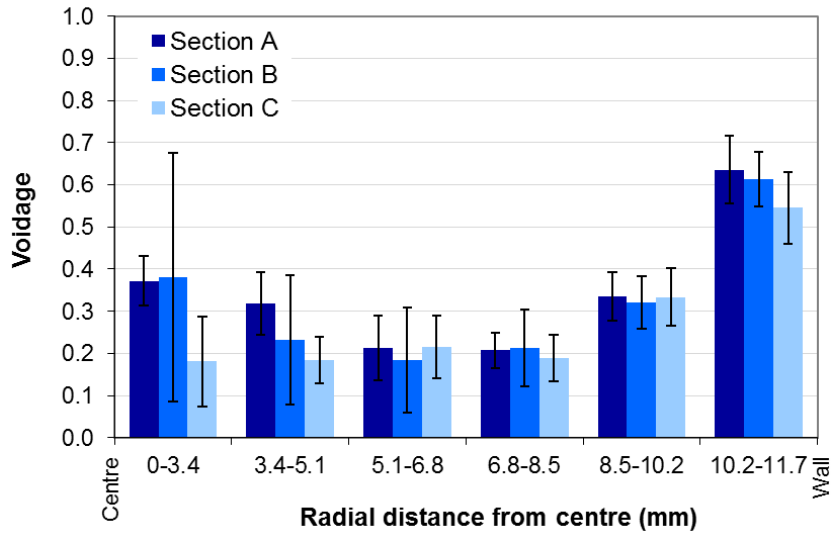
254 Alternatively this could correspond to mineral exposure to the surface in different regions of the column.
 255 The original volume of mineral per cubic millimetre is presented as a function of distance from the
 256 centre of the column in Figure 8. Figure 9 shows what fraction of this mineral is within 2 mm of the ore
 257 surface as the results in Section 3.3 showed that this was the mineral that was preferentially recovered.
 258 In sections B and C the fraction of mineral within 2 mm of the ore surface decreased towards the centre
 259 of the column, with the exception of the central 3.4 mm radius circle in section C where it increases.
 260 This was because of the large particles in these sections of the column whose edges, and so too the
 261 mineral within 2 mm of the ore surface, were located nearer to the walls of the column. This trend is
 262 complementary to the decrease in recovery from the outside to the centre of the column and suggests
 263 that the radial variations in recovery is because less mineral is accessible for leaching towards the centre
 264 of the column and not due to voidage effects. However, the results for section A do not fit this trend,
 265 confirming that there were factors other than the proximity of the mineral to the ore surface that
 266 contributed to the mineral leaching efficiency. Barring the existence of a higher concentration of non-
 267 recoverable mineral in the 6.8 mm closest to the centre of section A, the decrease in recovery towards
 268 the centre of the column was therefore most likely a combination of the proximity of the mineral to the
 269 ore surface and liquid distribution variations due to preferential flow into the areas of higher voidage
 270 near the column wall.

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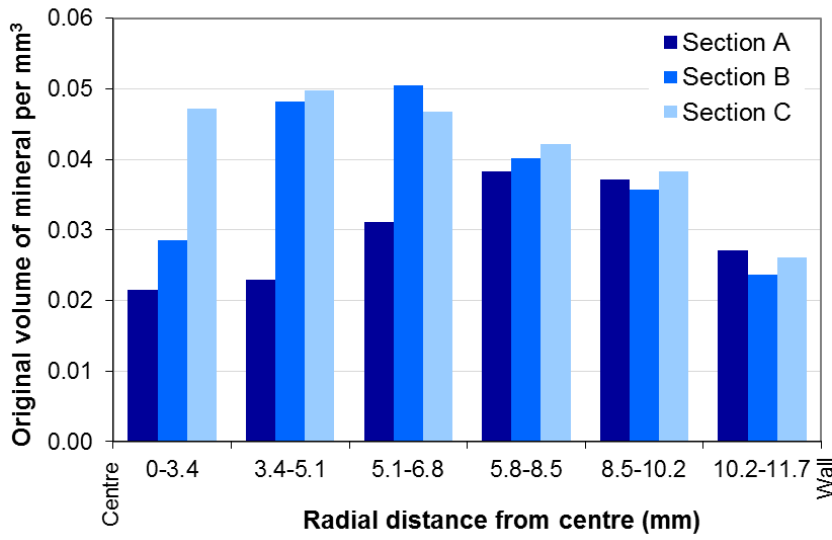
273 Figure 6. Recovery of the mineral after 102 days of leaching as a function of radial position in the
 274 column.



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276 Figure 7. Radial variation in the voidage where the error bars indicate the standard deviation in the
 277 voidage in each section.

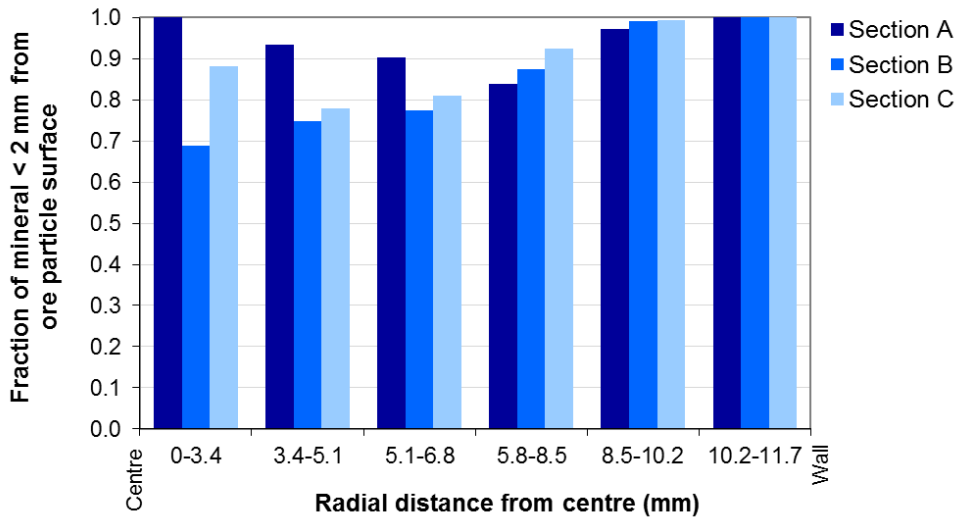
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280 Figure 8. Volume of sulfide mineral per mm³ on day 0 as a function of radial position in the column.

281



282

283 Figure 9. Fraction of the sulfide mineral particles less than 2 mm from the ore surface on day 0 as a
 284 function of radial position in the column.

285

286 **4. CONCLUSIONS**

287 The proximity of the mineral grains to the ore particle surface had a pronounced influence on recovery
 288 for this chalcopyrite ore, with leaching recoveries found to be highest on the ore particle surface and
 289 then decreasing as the distance from the ore particle surface increased. This was coupled with a higher
 290 leaching variability as the distance from the ore particle surface increased, proposed to be because the
 291 recovery of mineral located further from the ore surface has a higher dependence on the surrounding
 292 environment, such as whether it is in contact with other reactive minerals or cracks that link it to the
 293 surface. A maximum leaching penetration distance of 2 mm was observed to exist for the ore, not seen
 294 for other ores previously studied using X-ray μ CT; thus showing full metal recovery is not possible
 295 with the ore in its given state. Further particle size reduction to ensure access to these mineral grains is
 296 often not practicable for heap leaching due to both increased costs as well as liquid flow issues that may
 297 result due to decreased ore bed permeability. This motivates the need to identify mechanisms for the
 298 improved permeability of the ore particles of interest, for example through the selection of appropriate
 299 comminution methods for internal crack propagation during ore preparation or through degradation of
 300 the support material during the leaching process, highlighting the importance of improved
 301 understanding of an ore's full mineralogy and the composition of the gangue.

302 The radial variations in the mineral leaching showed that the proximity of the mineral to the ore surface
 303 was not the only phenomenon that affected mineral recovery. The high recovery at the column wall was
 304 attributable to preferential flow of the leaching solution in the higher voidage regions at the column
 305 wall. Therefore variations in inter-particle pore size (voidage) effected through preferential liquid flow
 306 paths were found to have some influence on the mineral recovery. Further investigations into the effect

307 of liquid distribution and flow on mineral leaching are being pursued using a combined Magnetic
308 Resonance Imaging (MRI) – X-ray μ CT approach.

309

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315

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