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The beneficiation of ultrafine phosphate

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ABSTRACT

This paper discusses a novel method of beneficiation of ultrafine phosphate which allows the recovery of phosphate particles that are less than 20 μm in size and have long been considered in the industry to be unrecoverable. It has been standard practice over many years in the phosphate industry to separate and discard the ultrafine particles, due to unacceptable processing difficulties such as excessively high viscosity and/or poor flotation recovery. In contrast with the established methods of beneficiation of phosphate where classification by hydrocyclone is mainly used to remove ultrafines as tailings, the method described in this paper allows a high proportion of ultrafines to be recovered via flotation without prior separation.

A number of variables and their effect on the flotation recovery of ultrafine phosphate are investigated including the pulp density and water quality during conditioning and flotation, type of flotation machine and reagents used to depress Fe_2O_3 and Al_2O_3 . Some excellent results were achieved using samples containing up to 75 wt% -20 µm particles, including for example 91.2% P_2O_5 recovery to a concentrate grade of 34.7% P_2O_5 from a low feed grade of 6.46% P_2O_5 and 92.4% P_2O_5 recovery to a concentrate grade of 30.2% P_2O_5 from 10.6% P_2O_5 feed. Guar gum was found to be the most effective depressant for Fe_2O_3 , whilst the Al_2O_3 was determined to be hydrophilic, resulting in low amounts being recovered to the concentrate, regardless of whether a depressant was used or not. The results of this work led to the development of the method described herein which is designed to recover phosphate from ores containing particles up to 80 wt% passing 20 µm, by flotation using a Jameson cell.

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1. Introduction

Phosphate beneficiation operations around the world which use flotation as the principal mechanism to concentrate the phosphate bearing minerals, typically discard the "ultrafine" phosphate bearing particles, where for this study "ultrafine" is defined as particles being smaller than 20 µm. It is standard practice in the phosphate industry to separate the ultrafines by scrubbing and hydrocycloning before the remainder of the raw feed is transferred to the flotation plant for concentration (Kogel et al., 2006). Phosphate beneficiation has been carried out this way for many years because of the known poor floatability of the ultrafines and because in the past it has been determined that the larger phosphate bearing particles float and concentrate more efficiently in the absence of ultrafines. After the ultrafines have been separated they are typically discarded and stored in large slimes ponds as tailings, effectively becoming lost revenue to the mining operation.

Legend International Holdings Inc. major phosphate deposits Paradise South and DTree are known to average between 20 wt% and 60 wt% ultrafine particles, containing up to 55 wt% of the phos-

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phate (Teague and Lollback, 2010). After grinding, the proportion of ultrafines can increase to up to 80 wt% of the total feed mass, containing up to 75 wt% of the phosphate. At the beginning of the development of Legend's beneficiation process, it became clear that if standard practice was followed by desliming and discarding the ultrafines, the project would be potentially uneconomical due to the removal of a large portion of the phosphate values. Hence the major aim of this work was to develop a process that could successfully concentrate ore from either of these two major phosphate deposits without discarding any ultrafines.

Bench scale flotation tests were initially carried out in a conventional Denver cell to confirm the poor floatability of the ultrafine phosphate and to investigate the effect of pulp density and water quality during conditioning and flotation, on phosphate recovery. Some bench scale comparison tests between Denver cells and Jameson cells were carried out, which effectively finalised the design of Legend's beneficiation process. Lastly, the entire beneficiation process was set-up at laboratory pilot scale to test diamond drill core and RC chip samples, to enable Legend to calculate a reserve for the Paradise South and a pending reserve for the DTree ore deposits. These tests confirmed the novel process was successful using samples with highly variable phosphate feed grades and mineralogy and also established guar gum as the best depressant for Fe₂O₃.

2. Theory

Examples of methods of desliming and their importance to effective recovery are discussed by Ahmed (2007). The use of screens or hydrocyclones to deslime the crushed ore dramatically improves the efficiency of flotation but in many cases the loss of phosphate fines is over 15% of the total phosphate. A technical report by Luttrell (2004) explains that beneficiation plants in Florida, one of the major areas for phosphate beneficiation in the USA, typically wash and deslime ore matrix at 150 mesh (105 μm) so that finer than 150 mesh particles are considered tailings and pumped to settling ponds so that approximately 30% of phosphate contained in the original ore is lost to tailings ponds. Thus, while flotation is a very effective method for the beneficiation of phosphate, there remains a need for a process which can recover a portion of fines normally discarded as tailings.

There is some reference in the literature to the flotation of ultrafine phosphate with column cells such as that practised at the Barreiro concentrator in Brazil (Wyslouzil, 2009; Wyslouzil et al., 2010) however there are some drawbacks to this process. The feed to the ultrafine circuit has a particle size of 100% passing 30 μm and is deslimed again at approximately 5–10 μm where 60 wt% of the P_2O_5 is still discarded. Also, of the 40 wt% of the P_2O_5 that is fed to the CPT flotation columns, only 7 wt% is recovered to a marketable concentrate grade of 33.5% P_2O_5 and 33 wt% is discarded as the flotation tailing. It can be seen that this process still has major limitations of; not being able to treat and recover ultrafine particles in the range of 0–10 μm ; and having to install additional desliming and flotation circuits for a low additional mass recovery of P_2O_5 . The method discussed in the current paper does not have these limitations.

Jameson cell flotation machines have a number of attributes which have made them a popular choice for facilitating the flotation of fine particles in base metal and coal operations. These have been well described and documented in the past by Jameson et al. (1988) and Young et al. (2006) and others. The average air bubble size is 300 µm compared to 1000 µm for conventional flotation machines and the total surface area of 1 mm³ of air is 20 mm² compared with 6 mm² for conventional machines. This larger bubble surface area generated by the Jameson cell increases the probability of particle-bubble collision and attachment when floating fine particles. It has been documented in the past that fine and ultrafine particles exhibit improved flotation characteristics when floated with small bubbles (Nguyen and Schulze, 2003; Fillipov, 1998), since the collision efficiency increases and there is an increased chance of particle-bubble attachment.

Although conventional and column cells have been used before in phosphate flotation, there is no reference in the literature to Jameson cells ever being used. This is surprising, given they have been used for some time in potash processing (Eisner, 2010) and they are considered to be better suited to the flotation of fine particles than both conventional and column cells since they generate smaller bubbles. With the development of Legend's process to float the ultrafine phosphate, it was thought there would be merit in trialling the Jameson cell, because of its known success in the minerals industry and its ability to generate small bubbles and recover fine particles.

3. Materials and methods

3.1. Bench scale conventional flotation cell tests

Bench scale studies were carried out on two of Legend's phosphate deposits, Paradise South and DTree, to determine the effect of ultrafines removal, pulp density and water quality during

conditioning and flotation and depressants for Fe₂O₃ and Al₂O₃ rejection. Prior to flotation, 1.5 kg of dry solid was wet-ground for 2 min in a 71 laboratory rod mill at 63 wt% solids using distilled water, until a particle size distribution of 80 wt% passing 150 µm was achieved. The slurry was then wet screened at 150 µm and 20 μ m, so that the +150 μ m oversize and $-20~\mu$ m ultrafines were removed respectively. This left 1 kg of solid ($-150 \mu m + 20 \mu m$ fraction)*** available for flotation. The slurry was dewatered to 75 wt% solids for conditioning, using a pressure filter. High intensity conditioning was carried out by vigorously agitating the solids and reagents in a 1 l stainless steel flask, at 1100 rpm, using a drill press with impellor. Soda ash (Na₂CO₃) was added to the slurry at a dose of 0.8 kg/t and stirred for 2 min until a pH of between 9.3 and 9.5 was achieved. Tall oil fatty acid and diesel were then added together as an emulsion at a dose of 1.3 kg/t (ratio of 1:1) and the slurry stirred for a further 6 min. When the amount of Fe₂O₃ in the feed was known to be 4% by weight or greater, guar gum depressant was added to the slurry as a 1 wt% solution and stirred for a further 2 min. After conditioning, each sample was diluted to 30 wt% solids with distilled water and transferred to the flotation cell.

Batch flotation was conducted in an aerated $2.5\,\mathrm{l}$ Denver flotation cell, with a constant impeller speed of 1000 rpm. Froth was scraped at a constant rate during rougher/scavenger flotation until the froth was barren; on average, this took about 4 min. The combined rougher/scavenger concentrate was then re-floated in a cleaning stage using the same flotation cell, without any further reagent or water addition. The flotation products were dried, weighed and assayed for P_2O_5 , Fe_2O_3 , Al_2O_3 , CaO, MgO and SiO_2 by ICP and XRF so that flotation recoveries could be calculated.

3.2. Laboratory pilot plant tests

Continuous flotation tests using drill core and RC chip samples with highly variable mineralogy from the Paradise South and DTree deposits were carried out at laboratory pilot scale. For these tests, the $-20~\mu m$ ultrafine fraction was not removed from the ore samples prior to conditioning and flotation. Samples of between 30 kg and 50 kg each were tumbled in a tumble mill for 15 min and screened at 25 mm to remove oversize silica. Each sample was wet-ground in a 951 rod mill with de-ionised water at 63 wt% solids to achieve a particle size of 80 wt% passing 150 μm , thickened to 75% solids by filtration and conditioned using the same reagent scheme as described in Section 3.1.

After conditioning, the slurry was transferred to a baffled 300 l drum and diluted to between 15 and 20 wt% solids using de-ionised water for flotation in a L150 (6 m high) Jameson cell. The slurry was continuously fed to the Jameson cell, at a constant flow rate of 0.35 m³/h, for rougher flotation. Air to the cell was adjusted to create a vacuum of between 15 and 20 kPa, which produced a feed pressure of between 120 and 150 kPa. When the operation was stable at the set process conditions, a survey was conducted by collecting 5 l feed, concentrate and tailing slurry samples. When the rougher stage was completed, the tailing was collected and fed continuously to the Jameson cell at 0.35 m³/h, to simulate scavenger flotation. Surveys were again conducted upon reaching stable operation. After the scavenger stage, the Jameson cell was drained and cleaned, and the rougher and scavenger concentrates were collected and fed though the cell to simulate cleaner flotation. The enrichment ratios for each flotation stage were found to vary with feed grade; typically they averaged between 1.8 and 2.7 for the rougher, 3 and 5 for the scavenger and between 1 and 1.5 for cleaner flotation. Similar to the bench scale tests, no further reagent additions were made to the scavenger and cleaner flotation stages. The flotation products were each dried, weighed and assayed for P₂O₅, Fe₂O₃, Al₂O₃, CaO, MgO and SiO₂ by ICP and XRF so that flotation recoveries could be calculated.

During operation of the Jameson cell, only one problem was encountered with short-circuiting. Occasionally the feed pressure to the cell would drop due to airlocks in the pump caused by excess froth in the slurry. When this occurred, it was necessary to stop-start the pump so that the feed pressure and vacuum in the downcomer were restored. Although this problem was encountered occasionally, it was quickly managed and did not affect the flotation results.

3.3. Mineralogy

Phosphorite at Legend's DTree deposit occurs predominantly as mudstone and silty mudstone phosphorite (also known as microsphorite) with some minor occurrences of peloidal grainstone phosphorite (also known as pelletal phosphorite). At Paradise South the phosphorite is made up of pelletal phosphorites almost completely comprised of collophane (carbonate fluorapatite), mudstone phosphorite and replacement phosphorite, which is a more indurated, porcellanous phosphorite (Hough, 2010). Mineralogical studies of DTree and Paradise South ore by QEMSCAN (Jones, 2011) and quantitative XRD (Johnson, 2009) determined the major mineral species to be apatite, quartz, kaolinite and goethite. Average head assays taken from the test samples that were used in this study for DTree and Paradise South ore are given in Table 1.

4. Results and discussion

4.1. Ultrafines removal - conventional flotation cell tests

Table 2 shows results that were obtained from bench scale flotation tests on DTree ore using a conventional Denver cell, with and without ultrafines. These tests were designed to investigate and confirm the effect ultrafine particles have on P_2O_5 concentrate grade and recovery when floated at different pulp densities.

It can be seen from the results that leaving the ultrafines in the ore has a negative impact on the amount of P_2O_5 recovered as well as the concentrate grade, when flotation is carried out in a conventional Denver cell. The difference in P_2O_5 recovery is more pronounced at 20 wt% solids (51.1%) compared to 30 wt% and 40 wt% solids (25.2% and 31.6%), nevertheless the difference is significant at each pulp density. Given the phosphate industry has used conventional flotation cells for many years, the poor recoveries in these tests demonstrate why the ultrafines are commonly separated from the ore and discarded before flotation. The results also show that in this case, 30 wt% solids is the optimum pulp density to achieve the highest P_2O_5 recovery and grade for DTree ore, marginally better recovery (6.7%) than 40 wt% solids with ultrafines and the same recovery (94%) with slightly higher grade (2.3%) without ultrafines.

4.2. Effect of pulp density during conditioning

Bench scale tests using Paradise South ore, were designed to investigate the effect of pulp density during conditioning on the flotation recovery of P_2O_5 . It has been previously documented that conditioning at high wt% solids promotes collector adsorption onto phosphate surfaces and reduces the activation of silica (Gruber et al., 1995). Table 3 shows the results. It can be seen that

conditioning at low wt% solids yields poor P2O5 recoveries and grades. The P₂O₅ recoveries increase from a low 10.3% at 30 wt% solids to a high of 97.8% when the slurry is conditioned at 75 wt% solids. There is not much difference in concentrate grade when the slurry is conditioned at 51 or 75 wt% solids but the P₂O₅ recovery is over 50% greater when conditioned at 75 wt% solids indicating that the hydrophobicity of the P₂O₅ in the feed has increased markedly under these conditions, resulting in improved floatability. Gruber et al. (1995) explain that the benefits of conditioning at high wt% solids are twofold; firstly, it increases the concentration (moles per litre) of the collector and therefore the force driving chemisorption of collector onto phosphate is increased and secondly, the effects of dissolved cations such as Ca2+ and Mg2+ are reduced when the quantity of water is reduced so that less reagent is lost due to bulk precipitation and there are fewer cations available to activate quartz. The results in Table 3 confirm this hypothesis.

4.3. Effect of water quality

Further to the discussion in the previous Section 4.2, the study undertaken by Jacobs Engineering (Gruber et al., 1995) clearly showed that the use of distilled or de-ionised water in phosphate conditioning and flotation, instead of plant or tap (hard) water, increased the recovery of phosphate and reduced the recovery of quartz. They suggested that the Ca²⁺ ions in the hard water activated the quartz and residual oleate ions from the collector were physically adsorbed onto the quartz in the flotation cell. In the current study, bench scale tests were carried out using DTree ore to confirm this effect and the results are given in Table 4, whilst Fig. 1 shows a plot of water hardness as CaCO₃ equivalent versus P₂O₅ recovery. The results demonstrate that the flotation recovery of P₂O₅ is inversely proportional to the hardness of the water that is used in grinding, conditioning and flotation, for instance 97.6% P₂O₅ recovery for distilled water with <1 mg/L hardness and only 59.9% P₂O₅ recovery for fresh site water with 410 mg/L hardness. Interestingly, the result for Test 4 of 97.1% P₂O₅ recovery using IX softened site water, confirmed that the ion-exchange apparatus successfully removed hard cations to a level <0.1 mg/L hardness, making it similar in quality to distilled water. The P2O5 grade of the concentrates was found to increase from 26.6% P₂O₅ to 30.9% P₂O₅ as the water hardness decreased, although this did not appear to be as linear as the relationship between water hardness and P₂O₅ recovery as illustrated in Fig. 1. When hard water was used during conditioning, a pH change in the slurry was observed, although more soda ash was needed to raise the pH from approximately eight to the target of between 9.3 and 9.5 and hence neutralise the concentration of Ca²⁺ in the water. Elevated levels of water hardness resulted in more silica reporting to the phosphate concentrate which agrees with the investigations carried out with Gruber et al. (1995) and their hypothesis of Ca²⁺ activating silica as discussed previously in this section. The water hardness may have also contributed to the lower phosphate recovery and concentrate grade by the reaction between fatty acid collector and Ca²⁺ and Mg²⁺, reducing the efficiency of the collector as found by Gruber et al. (1995).

In a full scale operation it is expected that all the recycled process water would be collected into a common collection tank which would then pass through a filter to remove fine particulate

Table 1Average head assays for DTree and Paradise South ore.

Ore type	P ₂ O ₅ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	MgO (%)	CaO (%)	SiO ₂ (%)
DTree	15.52	5.45	6.13	0.35	21.43	47.28
Paradise South	15.58	6.29	5.40	0.70	22.91	39.92

Table 2DTree conventional flotation cell tests with and without ultrafines.

Pulp wt% solids	DTree with ultrafines		DTree without ultrafines			
	Concentrate grade P ₂ O ₅ (%)	P ₂ O ₅ recovery to concentrate (%)	Concentrate grade P ₂ O ₅ (%)	P ₂ O ₅ recovery to concentrate (%)		
20	29.3	37.1	34.2	88.2		
30	27.4	69.2	36.4	94.4		
40	26.5	62.5	34.1	94.1		

Table 3Effect of pulp density during conditioning on flotation of Paradise South ore.

Test	Pulp wt% solids in conditioning	Feed grade P ₂ O ₅ (%)	Concentrate grade P_2O_5 (%)	P_2O_5 recovery to concentrate (%)
1	30	17.63	20.76	62.45
2	30	17.63	30.09	10.30
3	51	19.50	31.50	45.50
4	51	19.30	32.50	41.40
5	75	19.80	30.80	97.80
6	75	19.60	32.00	97.40

solids and ion exchange system to remove Ca^{2+} and Mg^{2+} , such as that facilitated by industrial water providers.

4.4. Conventional flotation cell versus Jameson cell

Table 5 shows results for the flotation of DTree ore with ultrafines in a conventional Denver cell versus a L150 Jameson cell at a pulp density of 20 wt% solids. From these results it can be seen that even though the cell types were tested using DTree samples of varying P₂O₅ feed grades, the difference in performance of the Denver cell and Jameson cell in terms of P₂O₅ recovery (30–50%) is obvious and significant. The Jameson cell tests were found to be very encouraging since they were the first trials ever attempted using phosphate ore and as such the process conditions were far from optimised, yet they still gave an outstanding result on material with 75 wt% ultrafines. The high P₂O₅ recoveries of 82.9% and 90% for Tests 3 and 4 at grades of 27.5% P_2O_5 and 29.7% P_2O_5 respectively, demonstrate that on a grade/recovery curve, the recovery could potentially be reduced to improve the grade to >30% P₂O₅. The results demonstrate that the use of the Jameson cell has a significant impact on the recovery of ultrafine phosphate particles compared to the conventional Denver cell, most likely due to the presence of small bubbles in the mixing zone which increase the probability of particle-bubble collision and attachment. Even though the average bubble size was not measured in this study, over the last twenty years it has been a major focus in Jameson cell research; Evans et al. (1992, 1994) measured and determined the average bubble size to be between 360 µm and 950 µm compared to conventional column cells that had an average bubble size between 2 mm and 3 mm. This was proven for full scale and pilot cells of various sizes. In the current study, it has been assumed that the bubble size generated during operation of the L150 pilot cell is not significantly different to that measured in other Jameson cells.

4.5. Depression of Fe₂O₃ and Al₂O₃

Bench scale flotation tests were carried out on Paradise South ore using a Denver cell to compare a number of reagents known to depress Fe_2O_3 and Al_2O_3 during phosphate flotation (Finch, 2009). These included corn starch, potato starch and guar gum. It was necessary for Legend to find a suitable depressant for these species so that the rock concentrate would be more amenable to downstream manufacture of the fertilizers mono-ammonium-phosphate (MAP) and di-ammonium-phosphate (DAP). On average, Paradise South and DTree run of mine (ROM) ore contain

approximately 5-6% each of Fe₂O₃ and Al₂O₃ by weight, so the aim of these tests was to find a reagent that could reduce these contaminants to as low a level as possible in the flotation concentrate. From the results in Table 6 it can be seen that the best depression of Fe₂O₃ was achieved in Test 7, using 500 g/t of guar gum. This test reduced the grade of Fe₂O₃ by the greatest margin, compared to the other depressants, from 4.40% to 2.89%, whilst the Al₂O₃ grade was reduced from 0.93% to 0.74%. The next best results were Tests 4 and 5, using 1500 g/t of corn starch and 500 g/t of potato starch respectively. In Test 4, the corn starch produced the lowest concentrate grade of Fe₂O₃ and Al₂O₃ out of all the tests $(2.78\% \text{ Fe}_2\text{O}_3 \text{ and } 0.72\% \text{ Al}_2\text{O}_3)$ and in Test 5, the potato starch gave a similar margin of Fe₂O₃ grade reduction to Test 4 (1.47% compared to 1.48%). The P₂O₅ recoveries to the concentrate are all consistently between 97% and 98% which is a good result, given that starch and guar gum are known to also adsorb onto phosphate surfaces and cause flotation depression if added in excessive quantities. From these tests, guar gum was selected as the overall best depressant because it performed the best in reducing the concentrate grade of Fe₂O₃ and Al₂O₃ at a minimal dosage of between 250 g/t and 500 g/t, compared to the other reagents which used between 500 g/t and 1500 g/t.

4.6. Laboratory pilot plant

The results from the bench scale work led to the refinement and finalisation of the process flowsheet. This flowsheet was then constructed at laboratory pilot plant scale at Amdel laboratories in Adelaide, Australia to carry out beneficiation tests on Paradise South and DTree ore, to enable Legend to calculate a mineable reserve for these deposits. All tests were completed without the removal of any $-20\,\mu m$ particles. The results of this work proved the novel process worked effectively using diamond drill core and RC chip samples with highly variable feed mineralogy and wt% proportions of $-20\,\mu m$ particles.

4.6.1. Paradise South

Table 7 shows a selection of flotation results from the Paradise South reserve determination tests whilst Figs. 2 and 3 illustrate the relationship between the grade of Fe₂O₃ and Al₂O₃ in the feed versus concentrate respectively.

The results in Table 7 indicate excellent P_2O_5 recoveries of between 76% and 97.3% from a variety of feed grades ranging from a low 6.46% P_2O_5 to 23.8% P_2O_5 . The result for Test 7, giving a concentrate grade of 34.7% P_2O_5 from 6.46% P_2O_5 feed whilst

Table 4The effect of water quality on flotation of DTree ore.

Test	Water type	Hardness as CaCO ₃ equivalent (mg/L)	Feed grade P ₂ O ₅ (%)	Concentrate grade P ₂ O ₅ (%)	Concentrate grade SiO ₂ (%)	P ₂ O ₅ recovery to concentrate (%)
1	Distilled	<0.1	17.1	30.9	17.6	97.6
2	IX softened site	<0.1	17.1	29.8	18.2	97.1
3	Recycled softened site	110	18.7	22.3	28.0	78.8
4	Brisbane tap	120	20.2	29.5	22.3	76.2
5	Fresh site	410	17.5	26.6	20.9	59.0

recovering 91.2% P_2O_5 , is outstanding. Tests 4 and 6 also gave excellent results from low grade feeds of about 10% P_2O_5 ; they recovered between 85% and 86% P_2O_5 to concentrate grades between 33% and 34% P_2O_5 from feed samples that consisted of between 47 wt% and 54 wt% $-20\,\mu m$ particles. Most phosphate operations around the world would consider the feed grades of Tests 4, 6 and 7 to be too low to process using established methods, so these results really highlight the benefits of the using the new beneficiation process described in this paper. These results indicate that this method of high wt% solids conditioning, de-ionised water and flotation with Jameson cells is very efficient and selective in recovering phosphate particles, even when the feed consists of a high wt% proportion of $-20\,\mu m$ particles.

With regard to Fe_2O_3 depression, the results show that 250 g/t guar gum performed well when it was used in 5 out of the 7 Tests, reducing the Fe_2O_3 grade by margins between 17% and 70%. The best result was achieved in Test 5 which reduced the Fe_2O_3 grade from 7.98% in the feed to 2.38% in the concentrate. Fig. 2 illustrates the relationship between Fe_2O_3 in the feed versus Fe_2O_3 in the concentrate for the entire campaign of tests using Paradise South ore. It can be seen that there is a straight line relationship between the Fe_2O_3 grade in the feed and concentrate, indicating consistent depression of Fe_2O_3 by the guar gum.

In terms of Al₂O₃ depression, it can be seen from the feed and concentrate grades in Table 7, that the process is extremely efficient in rejecting Al₂O₃ regardless of the feed grade and whether guar gum depressant has been used or not. The Al₂O₃ grade is consistently reduced by between 82% and 87% to levels ranging from 0.66% Al₂O₃ to 1.14% Al₂O₃ with the exception of Test 7 which reduced the grade from 5.34% Al₂O₃ to 2.23% Al₂O₃. The best individual result was in Test 4 where 7.22% Al₂O₃ in the feed was reduced to 0.94% Al₂O₃ in the concentrate. Fig. 3 shows a scatter plot of Al₂O₃ grade in the feed versus Al₂O₃ grade in the concentrate. The amount of scatter in the data indicates that there is no relationship between the Al₂O₃ in the feed and concentrate, because regardless of the feed grade, only minimal Al₂O₃ is recovered to the concentrate. The Al₂O₃ appears to behave differently to the Fe₂O₃ during flotation; it is postulated that it does not adsorb fatty acid collector and become hydrophobic like the Fe₂O₃, hence the guar gum has no effect on its depression, since it is already hydrophilic. It is also thought that the unique operating mechanism of the Jameson cell has added to the excellent Al₂O₃ rejection because of its small bubble generation and froth washing, resulting in only minimal entrainment of Al₂O₃ into the froth.

4.6.2. DTree

Table 8 shows a selection of flotation results from the entire campaign of DTree pending reserve determination tests, whilst Figs. 4 and 5 illustrate the relationship between the grade of Fe_2O_3 and Al_2O_3 in the feed versus concentrate respectively.

Similar to the results discussed in Section 4.6.1 for Paradise South, the DTree results shown in Table 8 demonstrate excellent P_2O_5 recoveries ranging from 76.7% to 95.6% from samples of

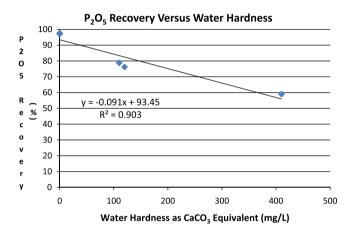


Fig. 1. P₂O₅ recovery versus water hardness.

variable feed grades (10.6% P_2O_5 –19.1% P_2O_5) and wt% proportions of $-20~\mu m$ particles (40.7–65.2%). In general the wt% proportions of $-20~\mu m$ particles in the milled DTree samples were higher than for the Paradise South samples, nevertheless, the beneficiation method still produced excellent flotation recoveries and concentrate grades. Some of the best results included Test 7 which achieved the highest P_2O_5 recovery of 95.6% at a concentrate grade of 33.3% P_2O_5 from a feed grade of 16.9% P_2O_5 with 53.4 wt% $-20~\mu m$ particles and Test 5 which gave 92.4% P_2O_5 recovery at a concentrate grade of 30.2% P_2O_5 from a low feed grade of 10.6% P_2O_5 with 49.7 wt% $-20~\mu m$ particles.

The Fe $_2$ O $_3$ grades in the feed and concentrate for Tests 2, 3, 5, 6 and 8 indicate that the guar gum at 250 g/t worked effectively as a depressant. The reductions in Fe $_2$ O $_3$ grade range from 6.6% to 67%, although on average the reductions are less than they were for the Paradise South tests, which may have been due to the presence of fine goethite needing further liberation. The best individual result was Test 6 which reduced the Fe $_2$ O $_3$ feed grade of 8.36% to a concentrate grade of 2.74%.

Fig. 4 illustrates the relationship between Fe_2O_3 in the feed versus Fe_2O_3 in the concentrate and similar to Fig. 2, gives a straight line relationship. This result suggests that the Fe_2O_3 mineralogy in both DTree and Paradise South is similar and responds to the guar gum depressant in the same way, although the R^2 value for DTree is slightly lower than for Paradise South (0.61 compared to 0.73) which could mean some of the goethite in the DTree samples was not as well liberated as it was for the Paradise South tests.

From the results in Table 8, it can be seen that similar to the Paradise South results, the process rejects Al_2O_3 from the phosphate concentrate extremely well, regardless of the feed grade and whether guar gum has been used or not. The Al_2O_3 grade is consistently reduced by between 66% and 88% to grades ranging from 0.64% Al_2O_3 to 1.67% Al_2O_3 . The best individual result was achieved in Test 8 which gave a concentrate grade of 0.64% Al_2O_3 from a feed grade of 7.75% Al_2O_3 . Fig. 5 shows a scatter plot of Al_2O_3 grade in

Table 5 Flotation of DTree ore using a Denver cell and Jameson cell.

Test	Cell type	Wt% passing 20 μm	Feed grade P ₂ O ₅ (%)	Concentrate §	Recovery		
				P ₂ O ₅ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	P ₂ O ₅ (%)
1	Denver	72	15.8	29.3	7.3	2.7	37.1
2	Denver	52	16.4	33.8	2.9	1.2	52.0
3	Jameson	75	21.4	27.5	1.8	1.4	82.9
4	Jameson	75	21.7	29.7	1.6	1.7	90.0

Table 6 Flotation of Paradise South ore comparing depressants.

Test	Reagent	Addition rate (g/t)	Head grade			Final grade	Recovery		
			P ₂ O ₅ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	P ₂ O ₅ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	P ₂ O ₅ (%)
1	Baseline	_	19.8	4.42	1.03	30.8	3.49	0.94	97.8
2	Corn starch	500	19.6	4.35	0.94	31.3	2.91	0.79	97.4
3	Corn starch	1000	19.7	4.35	0.95	31.1	2.89	0.80	97.7
4	Corn starch	1500	19.9	4.25	0.91	32.4	2.78	0.72	98.0
5	Potato starch	500	19.5	4.42	0.97	32.0	2.94	0.75	97.6
6	Guar gum	250	19.7	4.31	0.96	30.5	2.97	0.82	97.7
7	Guar gum	500	19.5	4.40	0.93	31.4	2.89	0.74	97.3

Table 7Paradise South laboratory pilot plant flotation results.

Test	Wt% passing 20 μm	Head grade				Concentrate grade				Recovery
		P ₂ O ₅ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	SiO ₂ (%)	P ₂ O ₅ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	SiO ₂ (%)	P ₂ O ₅ (%)
1	46.7	20.9	5.63	5.77	29.8	36.4	2.19	0.86	8.30	96.4
2*	48.6	23.8	3.55	3.65	30.9	36.7	2.13	0.66	4.30	97.3
3	41.3	16.2	5.35	7.01	38.4	33.0	3.33	1.14	6.30	94.1
4	47.2	10.1	2.75	7.22	56.2	34.3	2.05	0.94	6.10	85.2
5	55.3	10.2	7.98	4.47	51.3	30.4	2.38	0.91	11.0	76.0
6	54.3	10.4	4.80	4.48	59.5	33.0	3.99	0.98	10.7	86.5
7*	35.5	6.46	0.92	5.34	75.1	34.7	1.17	2.23	9.13	91.2

 $^{^{\}ast}$ Indicates tests where no guar gum was added during conditioning.

Fe,O, in Feed Versus Fe,O, in Concentrate

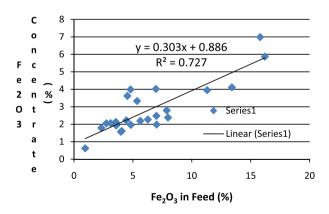
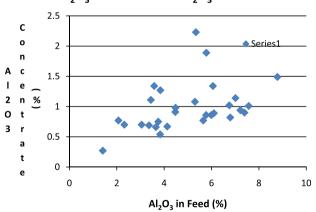


Fig. 2. Fe_2O_3 grade in feed versus Fe_2O_3 grade in concentrate for Paradise South laboratory pilot plant tests.

the feed versus Al_2O_3 grade in the concentrate. Again similar to the Paradise South tests, the amount of scatter in the data demonstrates there is no relationship between the Al_2O_3 in the feed and concentrate, because the beneficiation process has performed so well it has rejected Al_2O_3 regardless of the feed grade. Overall, the similarity of the P_2O_5 , Fe_2O_3 and Al_2O_3 flotation results between DTree and Paradise South ore indicate the excellent repeatability and robust nature of the designed beneficiation process.

Al₂O₃ in Feed Versus Al₂O₃ in Concentrate



 ${\bf Fig.~3.~Al_2O_3}$ grade in feed versus ${\rm Al_2O_3}$ grade in concentrate for Paradise South laboratory pilot plant tests.

4.6.3. Final process method

The results from all of the test work discussed in this paper culminated in the design of Legend's beneficiation process which is summarised here and is currently in patent pending status following the submission of an international patent application (Teague, 2011). This method allows the successful flotation recovery of phosphate from ore with a particle size distribution of up to $80\,\text{wt}\%$ passing $20\,\mu\text{m}$.

Table 8DTree laboratory pilot plant flotation results.

Test	Wt% Passing 20 μm	Head grade				Concentrat	Recovery			
		P ₂ O ₅ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	SiO ₂ (%)	P ₂ O ₅ (%)	Fe ₂ O ₃ (%)	Al ₂ O ₃ (%)	SiO ₂ (%)	P ₂ O ₅ (%)
1*	52.4	19.1	2.99	5.84	37.1	33.8	2.76	1.65	8.70	83.7
2	40.7	17.6	4.20	4.93	43.1	30.6	3.68	1.67	15.5	92.4
3	44.9	17.3	5.18	5.09	40.6	32.0	4.62	1.64	11.2	88.4
4*	54.3	18.7	2.41	6.10	39.1	31.8	2.25	1.15	13.7	92.1
5	49.7	10.6	5.84	6.05	54.9	30.2	3.82	1.11	15.2	92.4
6	56.5	15.3	8.36	7.12	40.8	35.1	2.74	0.84	6.40	79.4
7*	53.4	16.9	3.45	5.54	45.6	33.3	3.74	0.91	12.2	95.6
8	65.2	16.3	3.24	7.75	43.3	36.2	1.09	0.64	8.10	76.7

^{*} Indicates tests where no guar gum was added during conditioning.

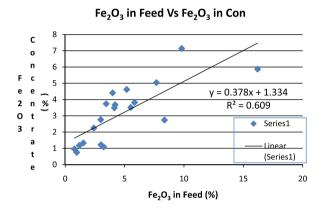


Fig. 4. Fe_2O_3 grade in feed versus Fe_2O_3 grade in concentrate for DTree laboratory pilot plant tests.

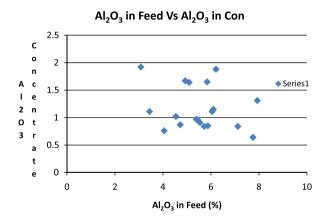


Fig. 5. Al_2O_3 grade in feed versus Al_2O_3 grade in concentrate for DTree laboratory pilot plant tests.

The slurry is formed by milling the ore in a rod mill, followed by classification using a hydrocyclone to provide a size fraction of at least 80 wt% passing 150 μ m, then it is dewatered to at least 70 wt% solids using a belt filter. Conditioning is carried out by contacting the slurry at 70 wt% solids with at least one collecting agent selected from fatty acids and salts thereof such as tall oil fatty acid (C12–C36) and at least one hydrocarbon such as diesel. Typically the dose of collector and hydrocarbon are in the range of 0.5–3 kg per tonne of dry solid contained in the slurry. The collector and hydrocarbon may be added to the aqueous slurry together or separately. They can be added neat or in the presence of diluents such as alkaline aqueous diluents if desired. In cases where the

content of Fe_2O_3 and Al_2O_3 is relatively high such as at least 4% each by weight of the solids content of the slurry, guar gum can be used to depress these constituents. The guar gum may be added to the slurry at a dosage of 0.05-1 kg/t.

A pH adjusting agent such as soda ash (Na_2CO_3) may be added at the milling stage or up to or including the conditioning tank so that a pH of between 9.3 and 10 is achieved. The soda ash may be added and mixed with the slurry in the conditioning tank, for example for 2 min and the fatty acid collector and diesel together or separately added and mixed with the slurry for a further period of, for example, 6 min. Where used, the guar gum may then be added and mixed with the slurry for a period (for example 2 min). Typically when it is used, the guar gum is mixed with the slurry after the phosphate particles have been made hydrophobic by the collector and hydrocarbon.

The method comprises a step of diluting the conditioned aqueous slurry to provide a solids content of no more than 35% by weight and preferably 20% solids by weight. The diluted conditioned slurry is introduced to the one or more downcomers of a Jameson flotation cell. Rougher, scavenger and cleaner flotation is carried out using Jameson cells to obtain a 32% P_2O_5 concentrate with at least 80% P_2O_5 recovery.

The water used in the method may be fresh water, recycled water or a mixture. It is particularly preferred that the water used in the method has a concentration of no more than 10 mg/L combined Ca²⁺ and Mg²⁺ and most preferably no more than 1 mg/L. The water can be treated using an ion exchange unit to reduce the concentration of Ca²⁺ and Mg²⁺ ions in the water, for example by exchange with Na⁺ ions. The reduction in the combined concentration of calcium and magnesium significantly improves adsorption of the collector onto the phosphate during conditioning and this improves flotation performance.

5. Conclusions

A process has been designed which successfully beneficiates phosphate ore with highly variable mineralogy and ultrafine ($-20~\mu m$) size fractions of up to 80~wt% of the feed. The process uses conditioning with reagents at high wt% solids (at least 70~wt%) and flotation with Jameson cells in a rougher, scavenger, cleaner configuration to recover at least $80\%~P_2O_5$ at a grade of $32\%~P_2O_5$ or greater. The Jameson cell was found to have an advantage over conventional flotation cells when treating ultrafine particles, due to their intense mixing zone and propensity to form small bubbles. The use of de-ionised water in the process was also found to be important to minimize the concentration of hard cations that could activate silica and hence adsorb collector, thereby interfering with collector adsorption onto phosphate, decreasing its floatability.

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