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MINERAL BENEFICIATION TESTS ON APATITE-BEARING CARBONATITE, TUNDULU, MALAWI

D A Briggs and C J Mitchell

1. INTRODUCTION

Approximately one million tons of the phosphate resources of the Tundulu carbonatite complex, Malawi, are in apatite-carbonatites (10-15% P_2O_5). These rocks are composed of apatite, quartz, calcite plus Fe-oxide pseudomorphs after siderite, accessory late-stage Fe-free calcite, and traces of synchisite, bastnaesite, pyrochlore, barite and strontianite. Mining of the apatite-bearing rocks to provide material for direct application fertilizer would involve selective mining of the paler, Fe-carbonate-free rocks. However, if larger-scale exploitation was required, beneficiation of the lower-grade rocks containing substantial quantities of Fe-carbonates and quartz would be required.

This report describes laboratory testwork on samples of both low-grade (10-15% P_2O_5) and high grade (~30% P_2O_5) apatite-carbonate aimed at establishing a beneficiation scheme for the Tundulu material. The work was carried out for the ODA/BGS R&D project "Agrogeology" which is managed by Dr J D Appleton, BGS.

2. TESTWORK ON LOW-GRADE APATITE-CARBONATITE

2.1. Preliminary treatment

Two samples, labelled T108 and T141, consisting of rock chips about 2 inches in size, were combined after brief mineralogical examination showed them to be of similar composition. Essentially the rocks consisted of apatite-carbonatites with considerable amounts of quartz and iron oxides. The first stage of the investigation was to determine the distribution of iron, silica and phosphate values through the combined sample. Accordingly this sample, totalling about 1500 g, was jaw-crushed and then carefully stage-ground using a laboratory mortar mill until the apatite was beginning to be liberated as individual grains. The was observed to be at around 60 mesh BS (250 µm) so grinding was stopped at this point. The ground material was then subjected to size fractionation, using a combination of screening on test sieves and sizing by air classification for the finer material. The resulting weight distribution was calculated, and is given below in Table 1.

2.2. Chemical analysis

Each sized fraction was subjected to X-ray fluorescence analysis for P_2O_5, SiO_2 and Fe_2O_3, and the distributions of each were calculated. These are also given in Table 1.

Table 1: Weight, assay and chemical distributions

		P_2O_5		SiO_2		Fe_2O_3	
Fraction	**Wt %**	**% assay**	**dist %**	**% assay**	**dist %**	**% assay**	**dist %**
-250+125 µm	8.5	8.5	5.3	23.5	12.1	10.5	11.6
-125+63 µm	25.4	14.2	26.3	19.5	30.1	7.4	24.4
- 63+32 µm	21.7	15.6	24.6	18.8	24.8	6.3	17.8
- 32+20 µm	10.0	16.1	11.7	14.7	8.9	6.4	8.3
- 20+10 µm	17.9	14.3	18.6	14.4	15.7	7.0	16.3
- 10 µm	16.5	11.2	13.5	8.4	8.4	10.1	21.6
Total	100.0		100.0		100.0		100.0
Assay, head		(13.7)		(16.5)		(7.7)	

These results show that the phosphate values have a reasonably even distribution through the size range, with the exception of the coarsest fraction which is relatively impoverished. The iron distribution is similarly regular, this time with the coarsest fraction being anomalous with a higher than average value. The silica distribution, however, has a different pattern and reveals that silica is concentrated in the coarser fractions, and tails off markedly in the fines. It is probable that liberation is incomplete in the -250+125 µm fraction, but obvious that the harder silica grains have concentrated there and have have had the effect of grinding the apatite out of this and into the finer sizes.

However, there is considerable scope for beneficiation work to upgrade the P_2O_5 content, at least of the three coarser fractions. The most obvious improvement could be made by rejecting free silica in the form of quartz, but it was decided to leave this problem for later gravity separation and first to tackle the removal of iron. This would be most conveniently accomplished by magnetic separation.

2.3. Magnetic separation tests

The three coarsest sized fractions were processed on a Carpo Laboratory Induced Roll Magnetic separator to produce three separated fractions. These were:

(i) a magnetic fraction, into which it was intended to concentrate as much of the iron as possible

(ii) a middling fraction which contained intermediate material

(iii) a non-magnetic fraction from which it was intended that iron had been removed.

All products were weighed, and again analysed for P_2O_5, SiO_2 and Fe_2O_3. The resulting distributions were calculated and are given below in Table 2. The head assays calculated for each size fraction are the same as those already given in Table 1, from which can also be seen the original distributions of the unseparated material.

Table 2: Results of magnetic separation tests

	Wt%	P_2O_5		SiO_2		Fe_2O_3	
Fraction	**fraction**	**% assay**	**dist %**	**% assay**	**dist %**	**% assay**	**dist %**
Magnetic	33.2	2.7	10.6	8.9	12.6	17.2	54.4
Middling	4.5	3.4	1.8	13.5	2.6	13.5	5.8
Non-magnetic	62.3	11.9	87.6	32.0	84.8	6.7	39.8
-250+125 μm	100.0	(8.5)	100.0	(23.5)	100.0	(10.5)	100.0
Magnetic	48.1	6.5	22.0	9.8	24.2	12.7	82.6
Middling	6.7	10.2	4.8	11.3	3.9	7.0	6.4
Non-magnetic	45.2	23.0	73.2	31.0	71.9	1.8	11.0
-125+63 μm	100.0	(14.2)	100.0	(19.5)	100.0	(7.4)	100.0
Magnetic	51.9	8.7	29.0	9.4	25.9	10.8	88.5
Middling	8.3	22.2	11.8	12.5	5.5	2.5	3.3
Non-magnetic	39.8	23.2	59.2	32.4	68.6	1.3	8.2
-63+32 μm	100.0	(15.6)	100.0	(18.5)	100.0	(6.3)	100.0

From the results given in Table 2, it is clear that magnetic separation has removed the bulk of the iron - well over 80% from the two finer sized fractions - and reduced the iron level of the non-magnetics to less than 2% Fe_2O_3. The results for the -250+125 μm fraction are less good, presumably due to incomplete liberation.

This has been accomplished with satisfactory recoveries of phosphate values to the non-magnetic fractions, namely 87.6% and 73.2% for the two coarser fractions, and 71.0% for the finest fraction when the phosphate-rich (22.2% P_2O_5) middlings are taken into account. Grades of 23.0% P_2O_5 have been attained, a considerable improvement on the 14.0-15% starting grade for the two finest fractions. The phosphate grade of the -250+125 µm non-magnetic fraction is rather poor at only 11.9% P_2O_5 but microscopic examination of this showed that it still contained considerable quantities of free apatite grains available for further concentration. All three non-magnetic fractions contained over 30% of silica, mainly in the form of free quartz. As expected, the bulk of the silica has reported with the apatite into the non-magnetic fractions, and the next stage in the investigation is some tests to determine how this can be removed.

2.4. Gravity separation tests

As there was insufficient non-magnetic material from the above tests for large-scale gravity separation tests on shaking tables, this testwork consisted of heavy-liquid separations only. Apatite has a specific gravity of about 3.15, so it was decided to separate at two specific gravities on either side of this to obtain an appropriate range. The heavy-liquids used were bromoform (SG 2.9) and methylene iodide (SG 3.3). Thus it could be expected that quartz and carbonates would be floated off by the bromoform and that any heavy minerals present would sink in the methylene iodide, leaving more or less pure apatite to concentrate in the specific gravity range between these two limits.

The three non-magnetic fractions were then duly separated in this manner and the apatite concentrates recovered, weighed and analysed for P_2O_5, SiO_2 and Fe_2O_3 as before. The light and heavy fractions were not chemically analysed, but back-calculations were made to relate the apatite concentrates to the non-magnetic starting materials as given below in Table 3.

Table 3: Results of heavy liquid separation tests

	Wt%	P_2O_5		SiO_2	Fe_2O_3
Fraction	**fraction**	**% assay**	**dist %**	**% assay**	**% assay**
2.9 float	41.1				
2.9 - 3.3	57.0	33.4	100.0	7.1	0.6
3.3 sink	1.9				
-250+125 µm	100.0	(11.9)	(100.0)	(32.0)	(6.7)
2.9 float	39.6				
2.9 - 3.3	57.8	33.2	83.4	5.4	0.9
3.3 sink	2.6				
-125+63 µm	100.0	(23.0)	(100.0)	(31.0)	(1.8)
2.9 float	32.6				
2.9 - 3.3	66.6	27.8	79.8	14.6	0.9
3.3 sink	0.8				
-63+32 µm	100.0	23.2	100.00	32.4	1.3

The above results show that considerable upgradings to meet the target grade of around 30% P_2O_5 have been achieved by the heavy-liquid separations. This has been attained with substantial reductions in silica content and further reductions of iron. It should be noted that the coarsest fraction, which it was thought might contain unliberated material, has been upgraded spectacularly. It may be that the 11.9% P_2O_5 assay for the -250+125 µm non-magnetic fraction may be in error on the low side. Certainly the heavy-liquid 2.9 - 3.3 SG fraction prepared from this consists of a high-grade apatite concentrate.

These results are very encouraging, even although they were obtained by means of heavy-liquid separations under laboratory conditions. Shaking tables and certainly the modern gravity concentration machines such as the Mozley Multi-G machine ought to be capable of reproducing these results, or even improve on them with fine tuning.

3. TESTWORK ON HIGH-GRADE APATITE-CARBONATITE

3.1 Preliminary treatment, magnetic separation and chemical analysis

A 1200 g high-grade sample, T301, containing about 30% P_20_5, was also subjected to an investigation similar to the one described above for the low-grade sample. The test material, which had previously been ball-milled to pass a nominal 100 mesh (125 µm) size, was fractionated as before by a combination of sizing and air classification. The three coarsest size fractions, with the exception of the very small +125 µm oversize, were then magnetically separated in the same way as before. All the products were analysed for P_20_5, $Si0_2$ and Fe_20_3, and the results are given below in Table 4.

The weight distribution for the head sample shows that the ball milling has been over-vigorous as 30% of the total weight has been ground into the -10 µm size fraction, which is too fine to respond to any normal method of benefication. This contrasts with a weight loss of only 15% to fines for the low-grade sample which was ground in a more controlled way. Careful stage-grinding must obviously be carried out in any future treatment.

The results of the magnetic separation are very disappointing. Although the iron content has been reduced from around 3% Fe_20_3 in the starting material to about 1% in the non-magnetics, the phosphate values lost in the magnetic rejects are unacceptably high. In short, the magnetic separation has not achieved much in the way of a positive result.

Table 4: Magnetic separation - weight distributions and assays

Fraction	Wt% fraction	Wt% head	P_2O_5 % assay	SiO_2 % assay	Fe_2O_3 % assay
+125 μm unseparated	100.0	2.4	25.2	36.7	3.1
Mangetic	7.1		20.8	27.1	14.2
Middling	3.9		28.7	24.4	2.6
Non-magnetic	89.0		28.9	30.4	1.2
-125+63 μm	100.0	15.4			
Magnetic	17.7		26.8	24.8	6.5
Middling	10.4		33.1	12.2	1.4
Non-magnetic	71.9		28.5	33.2	1.1
-63+32 μm	100.0	20.6			
Magnetic	31.4		29.1	21.4	4.5
Middling	23.9		31.0	21.3	2.0
Non-magnetic	44.7		29.7	29.1	1.3
-32+20 μm	100.0	15.3			
-20+10 μm unseparated	100.0	15.9	29.9	24.3	2.8
-10 μm unseparated	100.0	30.4	30.3	17.1	4.1
Total		100.0	(29.3)	(21.0)	(3.1)

Microscope examination of the magnetic fractions revealed the presence of free apatite grains which were coated with a thin film of iron oxide. This could possibly be an effect of the ball milling, and it has rendered the apatite grains magnetic and led to their removal with free iron oxide particles into the magnetic fractions. This would indicate that future treatment should include an attrition scrubbing stage to remove the iron oxide films and thus facilitate their removal without excessive loss of apatite. Wet as opposed to dry magnetic separation might be expected to produce improved results.

3.2 Gravity separation tests

Heavy-liquid separation tests were carried out on the -125+63 µm and -63+32 µm non-magnetic products in the same way as for the low-grade material to determine the extent to which they could be upgraded by gravity means. Back-calculations were made as before and the results are given below in Table 5.

Also given in Table 5 are the results of similar separations on the -250+125 µm and the -125+63 µm fractions derived from a hard rock sample equivalent to T301. This sample, T300, was carefully stage ground through 60 mesh (250 µm), fractionated and magnetically separated as before prior to heavy-liquid separation of the two sized fractions.

These results show that the high-grade material can be upgraded successfully from just below 30% to over 36% P_2O_5 with recoveries from the non-magnetic fractions of around 90%. Again these improvements are accompanied by significant reductions in iron and silica content of the concentrates. Even the coarsest -250+125 µm fraction, derived after careful stage grinding, has improved markedly which shows that this fraction is substantially well liberated and confirms that the ball-milling had been over-vigorous. The results from the hard rock sample also show higher recoveries, and indicate good scope for improved beneficiation.

Table 5: Results of heavy liquid separation tests

Fraction	Wt% fraction	P_2O_5 % assay	P_2O_5 dist %	SiO_2 % assay	Fe_2O_3 % assay
2.9 float	28.6				
2.9 - 3.3	71.1	36.4	89.6	6.4	0.1
3.3 sink	0.3				
-125+63 µm	100.0	(28.9)	(100.0)	(30.4)	(1.2)
2.9 float	17.0				
2.9 - 3.3	82.8	32.5	94.4	20.3	0.1
3.3 sink	0.2				
-63+32 µm	100.0	(28.5)	(100.0)	(33.2)	(1.1)
2.9 float	16.5				
2.9 - 3.3	83.3	36.3	(96.6)	6.7	0.3
3.3 sink	0.2				
-250+125 µm ex-hard rock	100.0	(31.3)	(100.0)	(19.0)	(0.6)
2.9 float	6.2				
2.9 - 3.3	93.5	34.6	(100.0)	14.0	0.5
3.3 sink	0.3				
-125+63 µm ex-hard rock	100.0	(30.9)	(100.0)	(17.6)	(0.9)

Finally, the two hard rock fractions were subjected to a new method of mineral separation known as the Magstream system. This consists of a combination of gravity and magnetic separation in ferromagnetic fluids. The effect is that highly selective separations can be made on the basis of small differences in specific gravity. Batch tests were carried out on the sample using a machine made available by the manufacturers and these save promising results. The specific gravity parameters were set at 3.0 and 3.4 so that the apatite was concentrated between these two limits, whereas the heavy liquid range used was 2.9-3.3. This resulted in a lower weight distribution to apatite concentrate of 70% and 80% as against the heavy liquid results of 80% and 90% for the coarse and fine fractions respectively. The products have not yet been analysed chemically, but the concentrates appear microscopically to be of even better grade than the heavy liquid products. The Magstream system is expensive, but the results obtained bear out the observation made earlier in this report about the need for fine tuning of gravity separation processes.

4. CONCLUSIONS AND RECOMMENDATIONS

1. The results of the investigation show that controlled grinding through 60 mesh (250 μm) is required to liberate the bulk of the apatite. Care must be taken not to produce excessive untreatable fines.

2. Magnetic separation has been shown to be effective in removing iron oxide from the low-grade sample, reducing the levels present from 10% to about 2%, without serious loss in phosphate recovery to the non-magnetic fractions. The process worked less well on the high-grade material due to excessive ball milling and the presence of iron oxide coatings on the apatite grains. Magnetic separation was carried out dry, but wet separation would give a greater degree of dispersion and selectivity, particularly if preceded by attrition scrubbing.

3. There is considerable scope for gravity separation which would remove much of the silica present, reducing it from 30% to around 10% or better. This upgrades the phosphate content to over 30% in the case of the low-grade ore (from 12-15%) and to around 36% for the high grade ore. These results are achieved at recoveries of 80-90% from the non-magnetic fractions.

4. The Magstream tests give an indication that the grades of the apatite concentrates could be improved still further. Although the system is available for commercial scale separations, it is expensive. Some preliminary trials were conducted on the Mozley Multi-Gravity concentrator but unfortunately these were done before the separation parameters were established by heavy-liquid separation, and thus were somewhat unsatisfactory and are not reported here. Now that some basic data have been obtained, it is suggested that larger-scale trials are conducted on this machine, and also on the GEC Duplex concentrator to attempt to match the encouraging results obtained by means of the laboratory separations.

5. It is recommended that both low and high grade materials are carefully ground through 60 mesh (250 µm), sized by screening on 125 µm and 63 µm, then air classified or hydrocycloned to remove untreatable -10 µm fines. Each size fraction should then be treated by dry or wet magnetic separation, preferably after attrition scrubbing, to remove iron oxides. Finally the non-magnetic fractions should be treated by some form of low-cost gravity separation using shaking tables or preferably one of the newer methods such as the GEC Duplex concentrator or the Mozley Multi-Gravity concentrator. These separators will remove much of the silica, and produce apatite concentrates containing around 35% P_2O_5 or better.

6. The separation data obtained in this investigation could apply equally well to processing by froth flotation as far as grades and recoveries are concerned. But this method was considered to be more expensive than a gravity processing route and has not been pursued.

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