

PROCESS DESIGN STUDY FOR THE BENEFICIATION
OF KALUWE PHOSPHATE ORE

BY

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in partial fulfilment of the requirements of the
degree of Master of Mineral Sciences in Metallurgy
and Mineral Processing.

THE UNIVERSITY OF ZAMBIA

LUSAKA

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DECLARATION

I declare that this dissertation was written in accordance with the rules and regulations governing the award of Master of Mineral Sciences Degree of the University of Zambia. I further declare that the dissertation has neither in part nor in whole been presented as substance for award of any degree, either to this or any other University.

Where other peoples' work has been drawn upon, acknowledgement has been made.

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To mum and dad for ensuring my proper upbringing.

A B S T R A C T

Kaluwe phosphate ore constitutes calcite (74.4 wt %) as the major mineral with minor amounts of apatite (5.9 wt %), opaques (13.5 wt %), which are predominantly iron minerals, quartz/feldspar (5.7 wt %) and traces of biotite (0.5 wt %). The chemical composition of the ore averaged 2.2% P_2O_5 , 47.7 % CaO, 1.6 % MgO, 1.2 % Al_2O_3 , 3.3 % Fe_2O_3 and 4.7 % SiO_2 .

Beneficiation studies were performed to investigate methods of concentrating the phosphate values. Preliminary studies involved detailed identification of mineralogical and chemical properties of the constituent minerals which could form the basis of selective separation of apatite from the gangue. The results of these studies showed that liberation of apatite occurred below 425μ m.

On the basis of the identified mineralogical and chemical properties of the minerals, tabling, magnetic concentration and flotation techniques were applied in the beneficiation tests. The results indicated that these techniques could not be individually applied to produce a final phosphate concentrate because of the poor grade of the ore. Encouraging concentrate grades were obtained after combining these techniques.

A treatment flowsheet has been proposed based on the obtained results. The multiplicity of processing steps in the flowsheet implies increased capital and operating costs. This is unavoidable due to the low grade nature of the ore.

A C K N O W L E D G E M E N T S

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INTRODUCTION

Phosphate ores represent the main source of phosphatic fertilizers. The continued growth of the world's population and the subsequent need to increase food production has stimulated efforts to search for new phosphate deposits and beneficiation of low-grade ores to obtain phosphate concentrates suitable for chemical processing. Many technical methods are used to upgrade phosphate ores, but the beneficiation requirements for any given ore are largely determined by ore characteristics and the planned end use of the concentrates. Physical beneficiation, including grinding, wet or dry sizing, desliming, and gravity concentration techniques is the simplest and most widely used technique. More complex procedures involving flotation are required by ores needing high concentration ratios. Phosphate ores containing a high percentage of calcite may be uneconomical for chemical processing because of high acid consumption and foaming during chemical conversion. Generally, $(\text{CaO} + \text{MgO})/\text{P}_2\text{O}_5$ weight ratios higher than 1.61 are considered uneconomical.

Zambia has embarked on an ambitious food production programme aimed at making the country self-sufficient in agricultural products. The success or failure of the programme will depend on a number of factors such as the availability of cheap agricultural inputs, primarily fertilizers. There are several promising phosphate deposits in the country, namely Chilembwe, Kaluwe and

Nkombwa Hill. These deposits if developed could substitute the phosphate component of NPK fertilizers which is currently being imported and enable the country to save some foreign exchange. However, a lot of technical work, ranging from geostatistical information to beneficiation tests need to be done before utilization of these deposits can proceed.

The Kaluwe phosphate deposit lies in the south east of the Central Province of Zambia. The deposit is a very low-grade ore and its mineralogical composition is unfavourable from the beneficiation point of view. However, the large tonnage of the reserve (about 100 million tonnes at an average grade of about 2.55% P_2O_5) has prompted other workers (4,5) to try and find a way of beneficiating this ore. Mineralogical studies on the ore have shown that the dominant mineral is calcite (70-80 wt %) with minor amounts of apatite (4-10 wt %).

The high calcite content of the ore and the similarity in the surface cations (Ca^{2+}) between calcite and apatite present problems in selective separation of the two minerals by flotation.

Preliminary investigations into the possibilities of beneficiating the Kaluwe phosphate deposit have been carried out by Kemira Oy of Finland (2), Warren Spring Laboratories of England (4) and Serran S.A. De Mineracao of Brazil (5). In the first case a reasonable grade of 30% P_2O_5 and a recovery of 75% were reported after using a scheme of one rougher flotation and five cleaner flotation processes. This scheme would involve high operating costs (i.e. labour, maintenance etc.). In the last two cases selective flotation of apatite from calcite was not achieved even

after application of various calcite depressants such as starch, quebracho and sodium silicate. In all cases, flotation conditions were not determined from electro-chemical studies. They were derived from either literature (using conditions for existing flowsheets of similar ores) or determined by trial and error. Some of the problems encountered in these tests could have been solved had surface chemistry studies on the individual ore minerals been considered.

Surface chemistry studies form a logical basis for design of a flotation process for any mineral. In the investigations done by other workers, these studies were not emphasised. This may have contributed to lack of selectivity between apatite and calcite. The importance of surface chemistry studies is that they help in defining the theoretical range of separability of the various ore constituents including what type of collector to use. It must, however, be noted that the water used in flotation would affect the surface chemistry of the minerals in the flotation process. As such, surface chemistry studies should be done using the intended water of use.

Based on the reasons advanced above, the research proposed a process design study on Kaluwe phosphate ore in order to look into the possibility of beneficiating the ore using simpler means. The investigations involved identification and quantification of various mineral phases present in the ore, grain size determination of apatite and gangue minerals, determination of mineral associations as well as assessment as to whether separation was possible by gravity and magnetic concentration.

Surface chemistry studies on the major minerals apatite, calcite and magnetite were done in both distilled and Kaluwe water. Furthermore, the studies were repeated for these minerals in the presence of reagents such as calcium chloride, sodium silicate and orthophosphoric acid. The aim of this was to find out if selectivity of separation could be improved by selective modification of the mineral surfaces.

The flotation tests were based on these studies. Cationic flotation tests using cocoamine were done with and without orthophosphoric acid as a modifier. Reverse anionic flotation tests were also tried using petroleum sulphonate as a collector.

The results of the above investigations are presented in this report. The report is divided into three chapters. Chapter 1 deals with the mineralogical and chemical analyses of the ore. The selection of processes of separation is presented in chapter 2 while chapter 3 deals with testing of the selected processes.

The results from this study demonstrate that Kaluwe phosphate ore is amenable to concentration to a reasonably high grade and recovery after preconcentration of the flotation feed. The biggest problem identified is its low-grade which would require a large throughput to produce a tonne of phosphate concentrate.

CHAPTER ONE

MINERALOGICAL AND CHEMICAL ANALYSES



1.1 Preliminary Studies

1.1.1 Location and size of the Kaluwe phosphate deposit

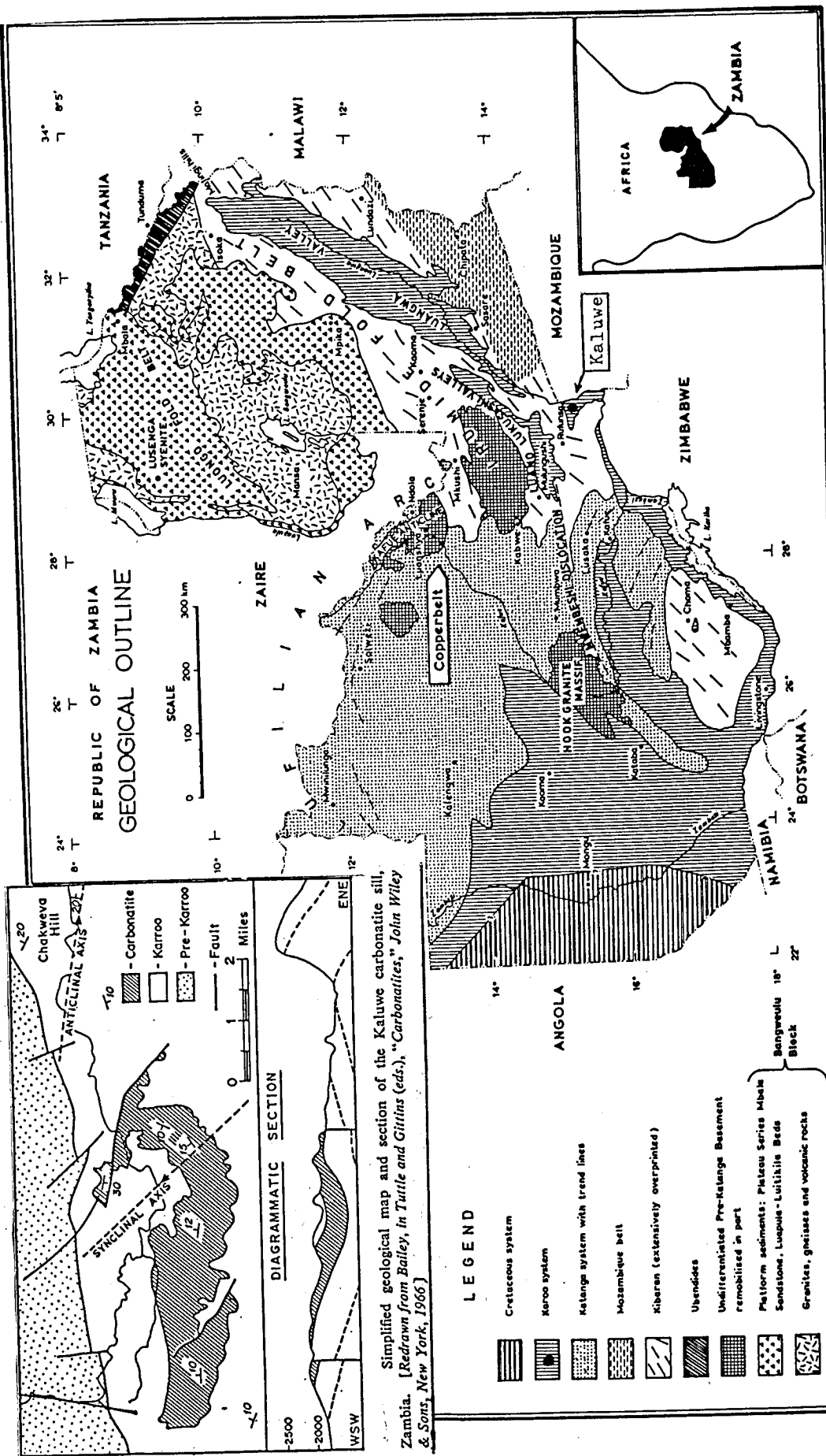
Kaluwe Hill lies south-east of the Central Province of Zambia in an area bounded by the Luangwa River, the Rufunsa River and the Great East Road (Fig. 1). All year round fresh water is available in the Molongoshi River. The source is a spring and a dam would provide enough water for any plant activities. However, the nearest available water in abundant supply is the Rufunsa River, a distance of about 8 km(1)

The Kaluwe phosphate deposit has been estimated at about 100 million tonnes at an average grade of 2.55% P_2O_5 . Although different values have been quoted, the deposit is believed to be large (2).

1.1.2 General Geology of Kaluwe Deposit

The Kaluwe Hill carbonatite forms a north-east dipping, east-west trending curved cuesta. The Hill is extensively faulted and lies about 3 km south of a Karroo / Pre-Karroo fault zone, running from Chakweva to Kasoshi Hill (fig. 2). Kaluwe Hill is approximately 10 km long and rises about 60 m above the surrounding broad river valleys.

The carbonatite was injected into the surrounding Karroo sandstone as a sill-like body. The carbonatite can be separated petrographically into three distinct facies which may or may not reflect separate phases or modes of emplacement. The three facies are of more or less equal thickness (about 80m) but vary slightly in physical appearance and mineralogical assemblage.



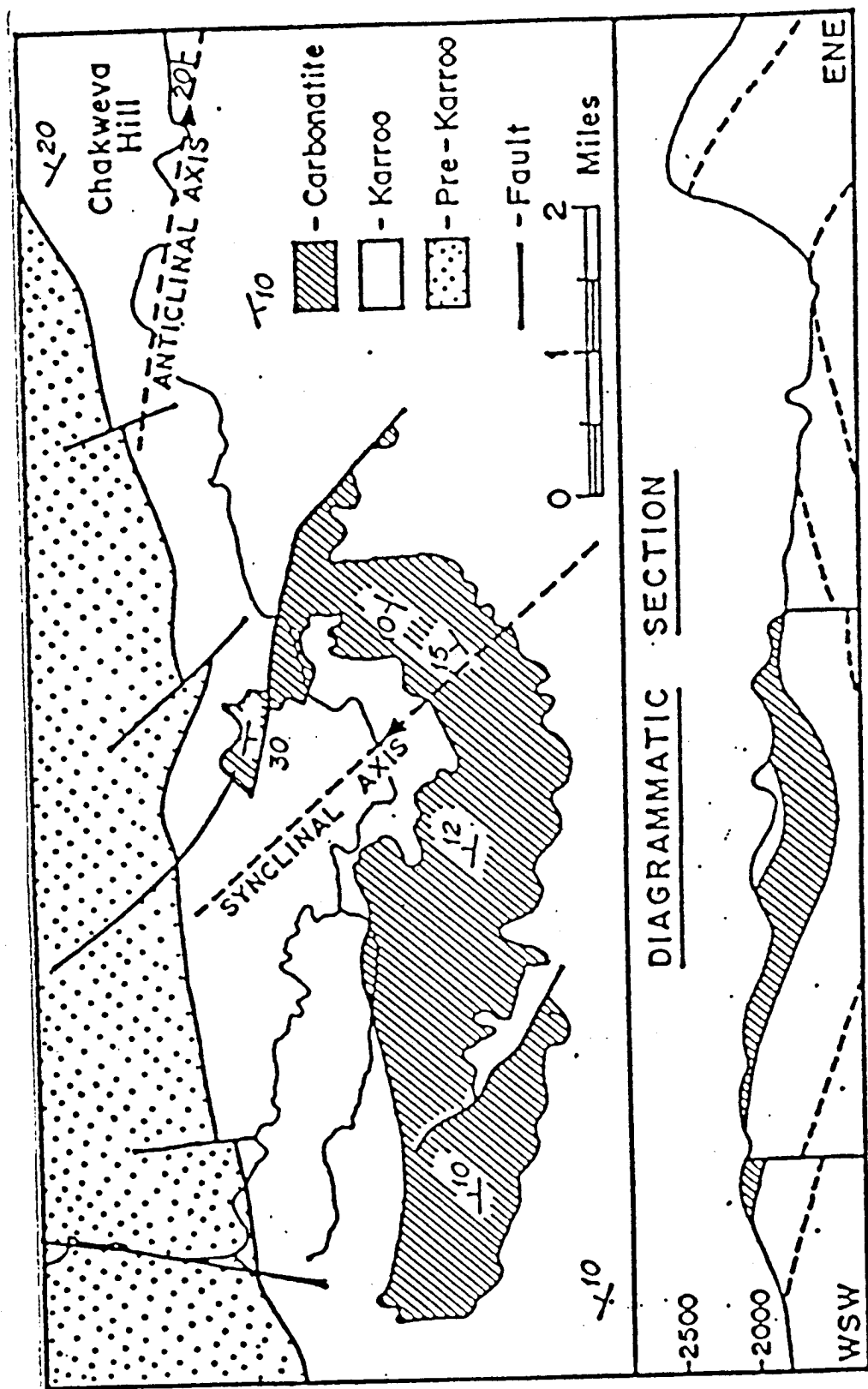


Fig. 2 Simplified geological map and section of the Kaluwe carbonatite sill, Zambia. [Redrawn from Bailey, in Tuttle and Gittins (eds.), "Carbonatites," John Wiley & Sons, New York 1966]

The three facies can be described as follows:

- (a) The Lower Facies is composed of layers of pink carbonatite agglomerate marked by variations in grain and fragment sizes between layers. This facies outcrops higher on the hill on the southern side and is similar to the Middle Facies but generally finer in texture. Quartz grains predominate with some microcline and iron in the matrix.
- (b) The Middle Facies is distinguished from the Lower Facies mainly by the lack of quartz grains. It is a light-grey coloured carbonatite with fragments generally rounded to sub-angular with a great variation in grain size. The Middle Facies, which is a prominent outcrop facies over the hill, displays a characteristic weathering due to calcite-rich particles.
- (c) The Upper Facies outcrops lower down the hill on the northern side and shows similarities with the Lower Facies, but is browner in colour and coarser in texture. Quartz xenoliths are common as are rafts of Karroo sediment.

The ore is massive and the mineral of economic importance is apatite. It is important as a phosphate mineral and is mainly present as fluor-hydroxyapatite. The associated minerals are hematite-magnetite, quartz and calcite, which is the most abundant. Other minerals present in minor or trace amounts include muscovite, feldspars, manganese oxide, garnet, zircon, ilmenite, rutile, mica and barite.

1.1.3 Source of Samples

Several boulders were taken from the Middle Facies, which is the most abundant of the three facies described above. With the help of a Geologist from Minex the boulders were taken from outcrops of carbonatite in a trench and measured approximately 15 to 36 cm in diameter. They were of three types namely, coarse-grained agglomerates, fine-grained agglomerates and carbonate sandstones.

The total sample weighed about 150 kg. About 100 litres of water from the nearest source, in this case the Molongoshi River, was taken for use in the beneficiation studies.

The ore samples were weathered, this is because they were taken slightly below the soil profile. Fresh samples from below the weathering zone may exhibit different mineralogical and chemical characteristics.

1.2 Petrography and Mineralogy of the Ore

Several thin sections were made from the ore sample in order to carry out petrographic and mineralogical studies. The aim of the studies was to identify the mineral constituents of the ore, the proportions in which they occurred and the degree of dissemination of the phosphate values.

The mineral proportions were determined using a polarizing microscope with a Swift Automatic Point-Counter stage unit attached to its stage. This unit was connected to an electronic control box with 11 resetting counters (also from James Swift & Sons Ltd.). The set up was similar to

the one shown in figure 3 (3). The stage unit used had a maximum E-W traverse range of 30mm, operated stepwise by pressing any one of the counter buttons on the control box. The 30mm-traverse limit was rather small and thin sections of length greater than 30mm were counted twice by reversing the slide. The gears in the stage unit were adjusted before beginning to allow a choice of E-W translation steps accordingly. The selection depends on the grain size of the ore. The N-S translation range was 30mm, effected manually by turning a knob, each click being $\frac{1}{6}$ mm.

Each counter was labelled with the name of the mineral to be identified, for example calcite, apatite etc. After identifying the grain under the crosswire, an appropriate button on the control box was pressed and the stage automatically translated one step. This was repeated until one complete line of translation was performed.

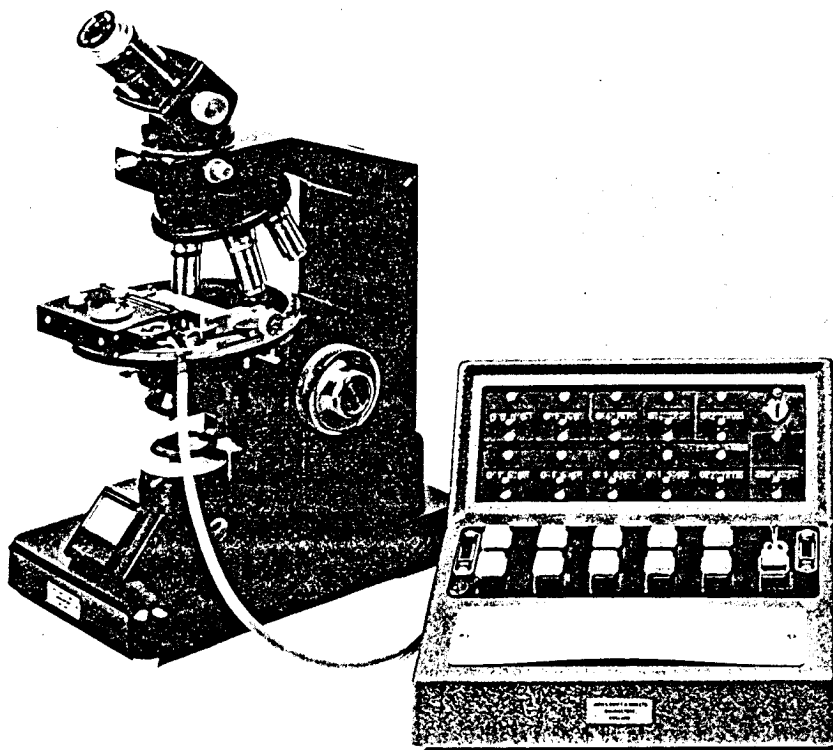


Fig. 3. The Swift automatic thin-section point counter showing the stage unit and the control box with counters (James Swift and Sons, England).

When starting a new translation line, the N-S adjusting knob was turned by one notch. In this way the whole thin section was covered. Approximately 1000 to 2000 points were counted during each operation. The final reading on each mineral counter was noted and all the mineral totals added. The individual mineral counts were then converted to number percentages.

1.2.1 Mineralogy of the ore

The hand specimens were fine and coarse-grained carbonate agglomerates with reddish-brown to grey sandsized, granular matrix and composed primarily of carbonate and iron mineral crystals. Apatite crystals could only be seen under a binocular microscope.

In thin section, crystals of apatite were disseminated in a calcite matrix. Iron oxides (opaques) and flakes of biotite were present. Feldspar and quartz were rare.

Apatite occurred as six-sided prismatic (euhedral) and sub-hedral crystals with a maximum length of $840\mu\text{m}$ and a width of $600\mu\text{m}$. Under crossed nicols apatite exhibited a grey to dark-grey colour with crystals cut perpendicular to the Z-axis being dark. Inclusions in apatite were rare. In some cases apatite occurred as inclusions in opaque minerals. The average grain size of apatite crystals was microscopically determined using a linear measuring technique with a calibrated ocular along the north-south cross hairs. The computed average of the measurements gave a grain size of $390\mu\text{m}$.

Calcite was the major mineral in the ore and occurred as a fine-grained matrix forming the groundmass. In some cases aggregates of calcite grains were observed.

Crystals of opaque minerals occurred as euhedral, sub-hedral and sometimes as anhedral crystals. The size varied from $36\mu\text{m}$ to 1.8mm . Some crystals contained inclusions of apatite.

Biotite occurred as brown lamellar aggregates and was pleochroic with stronger absorption when the cleavage was parallel to the vibration plane of the east-west nicol. Some crystals exhibited replacement by an opaque mineral. The maximum length of the lamellae was 2.3mm and the width was 1.68mm.

A few anhedral crystals of feldspar were observed in some specimens. Quartz occurred mostly as recrystallised grains forming aggregates, but was rare.

Table 1 summarises the proportions of minerals observed in the Kaluwe-Middle facies ore. In the table KM denotes Kaluwe-Middle facies ore. KM-1 to KM-5 denote the thin sections made at random from the sample. There is consistency in the mineralogy of the ore as can be seen from the table. The average abundance of iron oxides (opaques) was 13.5% which is relatively high and as such magnetic separation of the iron minerals should be considered in the treatment of this ore. The average apatite abundance was 5.9% while that for the main gangue mineral, calcite, was 74.4%, the two constituting 80.3% of the sample. Quartz/feldspar averaged 5.7% while biotite was a minor constituent at 0.5% of the sample.

Previous work (6) has shown that apatite in this ore has a refractive index (N_D^{25} mean = 1.642) which indicates that the apatite is a fluor-hydroxyapatite with a composition about half way between pure fluorapatite - hydroxyapatite end members.

TABLE 1: Modal analysis on Kaluwe ore.

SAMPLE NUMBER	MINERAL PROPORTION, %					Total
	Apatite	Calcite	Opauques	Biotite (Mica)	Quartz/Feldspar	
KM-1	4.9	77.1	13.0	0.4	4.6	100
KM-2	9.7	75.6	12.1	0.3	2.3	100
KM-3	4.6	68.2	12.0	0.6	14.6	100
KM-4	6.7	67.4	24.1	0.2	1.6	100
KM-5	3.5	83.9	6.3	1.1	5.2	100
Average Abundance %	5.9	74.4	13.5	0.5	5.7	100

Opauques = Magnetite, Hematite and Goethite and other iron minerals

An XRF scan on the head sample for the heavy elements indicated the presence of calcium as the major element with strontium, iron, phosphorus and silicon as minor elements and traces of barium, niobium, yttrium, zirconium, zinc, manganese, cerium, vanadium and titanium.

An XRD pattern on the head sample is shown in figure 4.

The figure shows that calcite is the most abundant and that Apatite, goethite, muscovite and quartz are minor.

1.3 Chemical and Mineralogical Appraisal

1.3.1 Size Reduction

Although preliminary grain size measurements had indicated that apatite was not coarse-grained, comminution was carried out to produce a size distribution biased towards coarse sizes and a minimum production of fine material. However, production of fines was inevitable in this case since the ore was friable. Fines were created even at large discharge settings.

The hand crushed samples were stage crushed to minus 3mm using a laboratory blake-type jaw crusher.

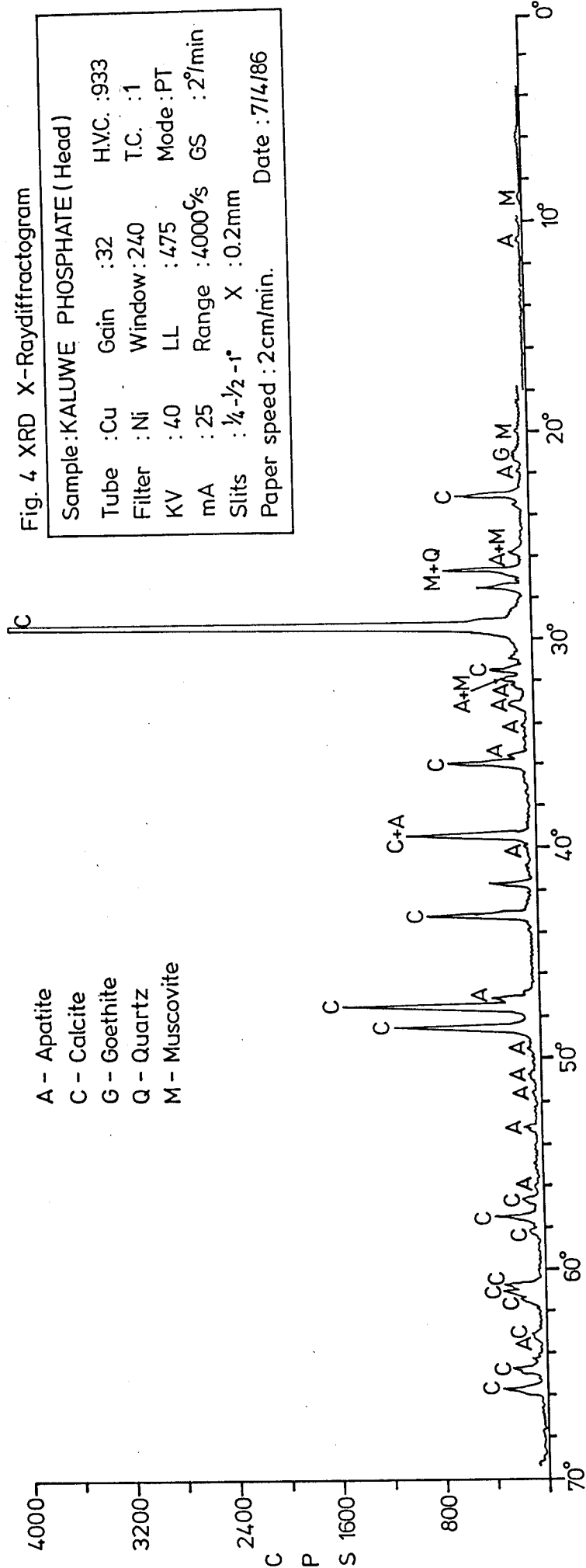
A head sample was collected using a combination of riffle boxes and a rotary sample divider and subjected to chemical analysis.

The main constituents of the head sample were: 2.2% P_2O_5 , 47.7% CaO, 1.6% MgO, 1.2% Al_2O_3 , 3.3% Fe_2O_3 and 4.7% SiO_2 . It can be concluded from the chemical analysis data that the ore is very low in grade and as such it needs to be concentrated (as a phosphate) if it has to be subjected

Fig. 4 XRD X-Raydiffractogram

Sample: KALUWE PHOSPHATE (Head)

Tube : Cu Gain : 32 H.V.C. : 933
 Filter : Ni Window : 240 T.C. : 1
 KV : 40 LL : 475 Mode : PT
 mA : 25 Range : 4000 $\frac{1}{s}$ GS : 2 $\frac{1}{min}$
 Slits : $\frac{1}{4}$ - $\frac{1}{2}$ -1 $\frac{1}{2}$ X : 0.2mm
 Paper speed : 2cm/min. Date : 7/14/86



to chemical processing.

1.3.2 Size Classification

The purposes of sizing are to check the quality of crushed or ground products, the extent to which the phosphate values are liberated from the gangue at various particle sizes, and to aid in the specific examination of the distribution of the ore constituents in various particle sizes. Many techniques are employed in laboratory sizing, including screening (wet or dry), infrasizing, elutriation, cyclosizing, and sedimentation.

The minus 3mm product was sampled using riffle boxes and subjected to dry screening. The resulting size fractions were further sampled using a rotary sample divider for chemical and mineralogical analyses. The size distribution data of the jaw crusher product of Kaluwe ore are given in table 1 of the appendices and are summarised in the histogram of figure 5.

About 65% of the material is finer than 850 μ m as shown by the sizing data. The histogram shows that the ore produces two peaks, one in the finer and the other in the coarser size range. This type of behaviour is expected for Kaluwe ore as it is typical of friable ores. Visual examination of the coarse fractions revealed that they consisted predominantly of a black mineral (iron oxides). The black mineral tends to concentrate

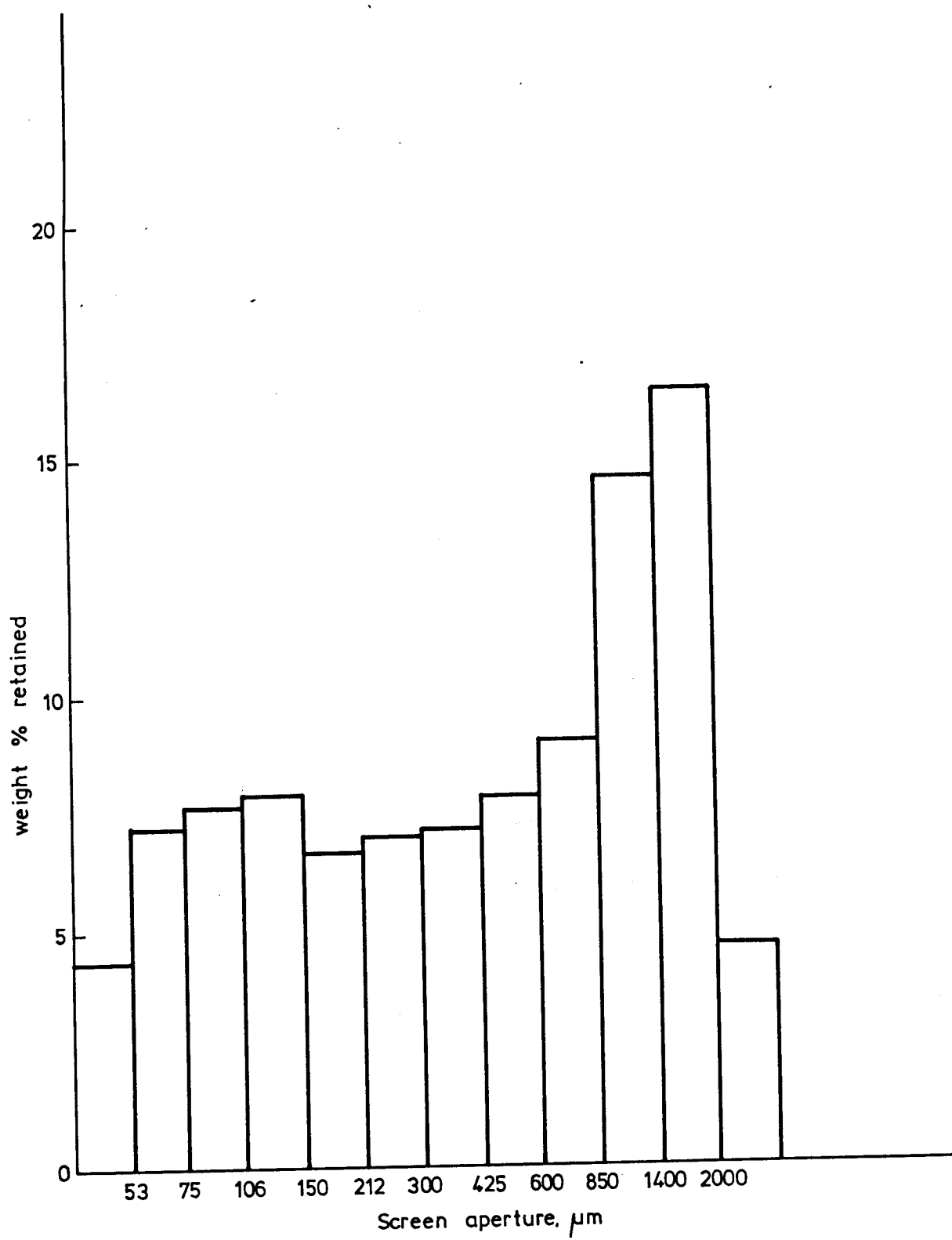


Fig. 5. Size distribution for Kaluwe ore.

in the coarse fraction as it constitutes the harder fraction of the ore compared to calcite and apatite (table 3). The size distribution is skewed toward the coarse sizes.

The degree of upgrading achieved in a concentration process may be measured by the enrichment ratio, ER. In this study, ER was defined as the grade of concentrate or size fraction divided by the head grade i.e. the degree of excess/deficiency to which valuable material is present in the fraction compared to the feed sample. Table 2 gives the calculated enrichment ratios and the same data are presented in figure 6.

Reasonable enrichment ratios were obtained in the size range +106 to +425 μ m. The associated phosphate grade was about 3% P_2O_5 . In this case comminution followed by screening cannot be used to produce a reasonable concentrate because the resultant increase in grade is marginal.

Table 2. Effect of Screening on the phosphate enrichment ratio

Size fraction μm	Grade % P_2O_5	ER
+ 2000	1.66	0.80
+ 1400	1.58	0.76
+ 850	1.73	0.83
+ 600	1.85	0.89
+ 425	2.14	1.03
+ 300	3.02	1.45
+ 212	2.86	1.38
+ 150	2.98	1.43
+ 106	2.74	1.32
+ 75	1.74	0.84
+ 53	1.79	0.86
- 53	1.90	0.91

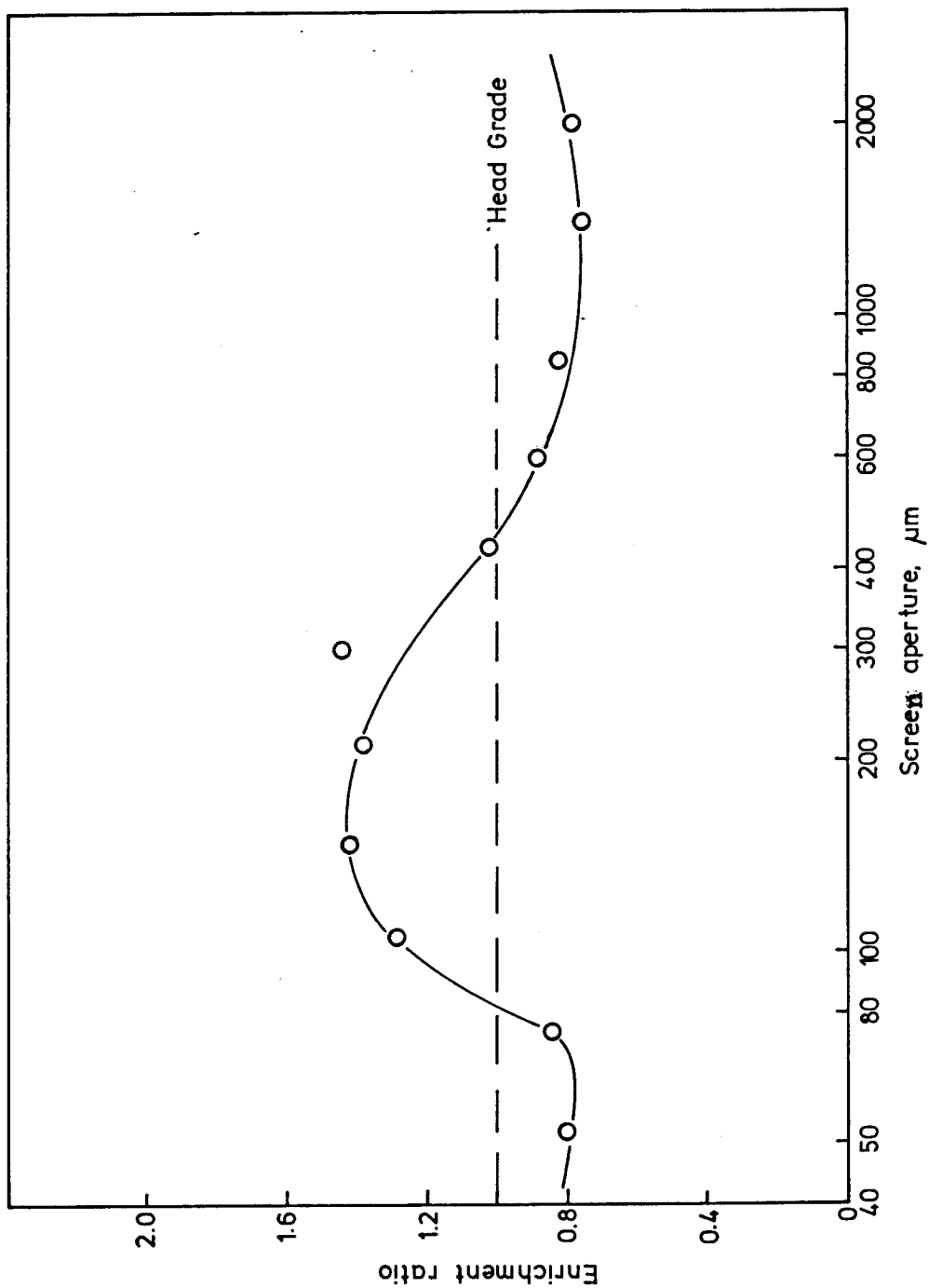


Fig. 6. Effect of particle size on the phosphate enrichment ratio

1.3.3 Size-sorting assay

Table 3 gives chemical analyses of the screen fractions. The table shows that there is a progressive increase in the P_2O_5 grade of the fractions as the size decreases. The coarse and fine size ranges show low P_2O_5 assays whereas there is P_2O_5 enrichment in the +106 to +425 μm size range in relation to the head grade (fig. 7). This is compatible with the earlier observation that the apatite is liberated within this size range. The reduction in the P_2O_5 assay at -106 μm sizes may be attributed to the presence of inevitably produced fines of gangue material and so tend to reduce the grade. In the coarser size fractions the apatite is present as inclusions primarily in massive calcite, due to incomplete liberation, which tends to reduce the P_2O_5 grade. Calcium oxide and silica are evenly distributed throughout the size range and on average CaO represents more than 70% $CaCO_3$ in each size fraction. The other oxides are predominantly distributed in the coarse fractions.

Mineralogical studies were carried out on various size fractions to observe the mineral proportions and their distribution in the size fractions shown. The results are tabulated in table 4. The proportions of minerals in each size fraction were microscopically determined by the point counting technique outlined in section 1.2.

The mineralogical data generally support the observation made from table 3 that there is a progressive increase in the apatite content with decrease in size; that the

Table 3. Sizing-sorting assay for Kaluwe ore

Size Fraction μm	Weight % Retained	P ₂ O ₅		CaO		SiO ₂		Fe ₂ O ₃		Al ₂ O ₃		MgO	
		%	Dist	%	Dist	%	Dist	%	Dist	%	Dist	%	Dist
+2000	4.56	1.7	3.6	44.4	4.4	3.1	3.5	5.3	5.9	0.5	3.3	3.5	5.5
+1400	16.35	1.6	12.3	47.2	16.9	3.0	12.3	4.5	18.2	0.5	11.9	2.8	15.9
+ 850	14.48	1.7	11.9	46.9	14.9	3.5	12.7	4.0	14.1	0.5	10.5	1.6	8.1
+ 600	8.98	1.9	7.9	42.4	8.3	4.1	9.2	4.8	10.6	0.8	11.0	4.0	12.5
+ 425	7.77	2.1	8.0	44.9	7.6	4.0	7.8	4.2	8.1	0.5	5.6	2.2	5.9
+ 300	7.11	3.0	10.3	42.7	6.6	3.7	6.6	4.4	7.6	0.2	2.6	3.5	8.6
+ 212	6.97	2.9	9.5	40.3	6.1	3.9	6.8	4.8	8.2	0.2	2.6	5.0	12.1
+ 150	6.70	3.0	9.6	50.3	7.4	4.1	6.9	4.4	7.2	0.8	8.2	0.2	0.5
+ 106	7.91	2.7	10.4	48.0	8.3	4.9	9.7	3.2	6.1	1.0	12.5	3.5	9.6
+ 75	7.64	1.7	6.4	44.1	7.4	4.9	9.4	3.4	6.3	0.8	9.4	4.8	12.7
+ 53	7.24	1.8	6.2	46.9	7.4	5.4	9.8	3.0	5.3	1.2	14.0	2.8	7.1
- 53	4.29	1.9	3.9	49.4	4.7	4.8	5.3	2.3	2.4	1.2	8.4	1.0	1.5
Total/ Assay	100.0	2.1	100.0	45.7	100	4.0	100.0	4.1	100.0	0.6	100.0	2.9	100.0

N.B. Trace elements not included.

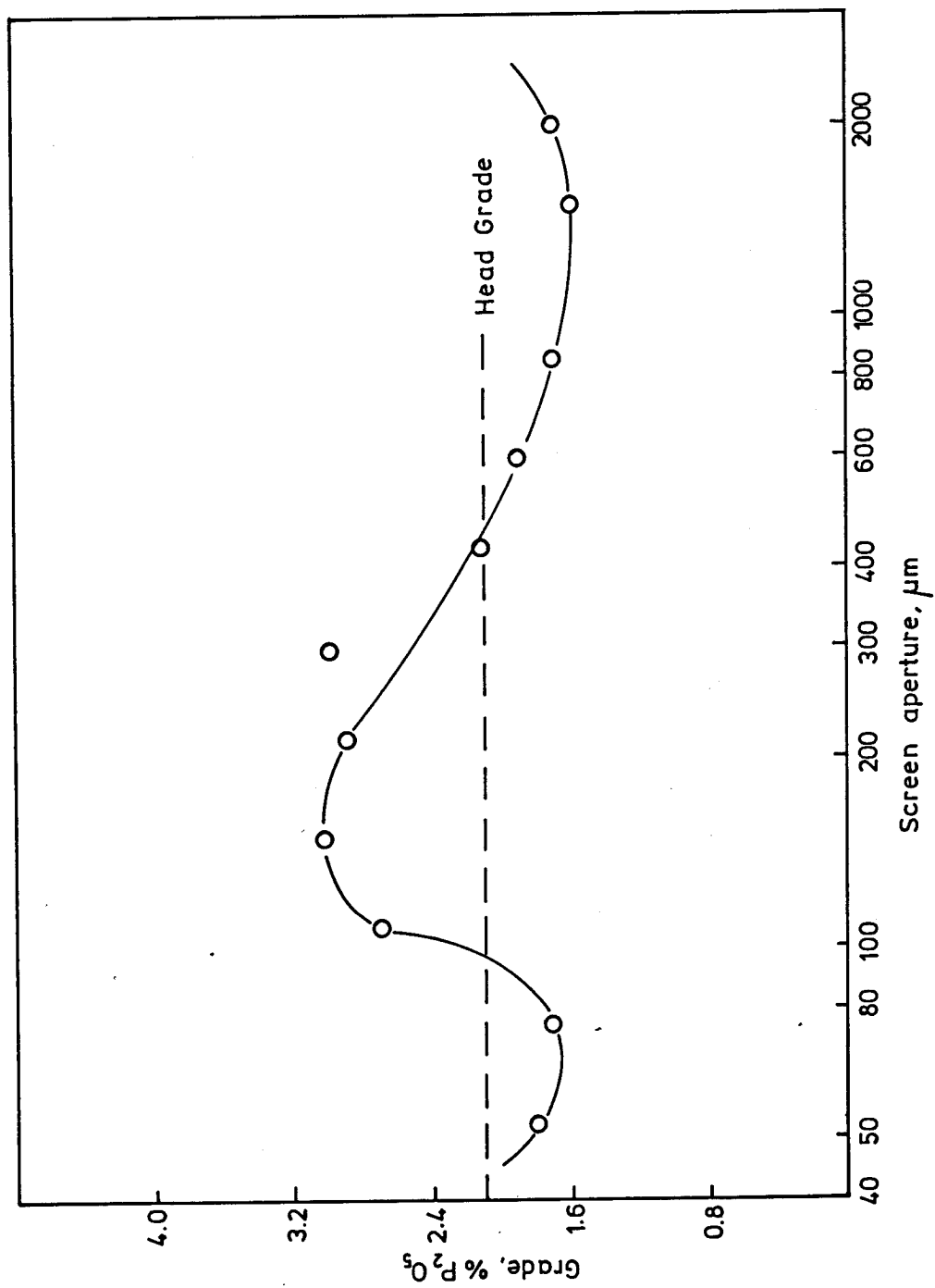


Fig. 7. Effect of particle size on the P_2O_5 grade.

Table 4. Mineral Proportions and Distributions for Kaluwe ore
as a function of size.

Size Fraction µm	Apatite		Calcite		Opakes		Quartz/Feldspar		Biotite	Total of pPropor- tion
	Propor- tion %	Distri- bution %	Propor- tion %	Distri- bution %	Propor- tion %	Distri- bution %	Propor- tion %	Distri- bution %	Propor- tion %	
+2000	6.2	3.4	65.9	4.2	27.2	8.6	0.3	2.9	0.4	100
+1400	5.1	10.0	72.1	16.3	21.7	24.6	0.8	29.5	0.3	100
+ 850	6.6	11.4	71.6	14.3	20.9	21.1	0.6	17.7	0.2	100
+ 600	6.8	7.4	80.9	10.0	11.6	7.2	0.7	14.7	-	100
+ 425	8.3	7.8	74.9	8.0	16.4	8.8	0.4	5.9	-	100
+ 300	9.6	8.3	80.3	7.9	9.5	4.6	0.5	8.8	0.1	100
+ 212	14.0	11.8	76.9	7.4	8.1	3.9	0.6	8.8	0.4	100
+ 150	15.1	12.3	70.4	6.5	14.4	6.7	0.1	2.9	-	100
+ 106	15.3	14.6	69.2	7.6	15.4	8.5	0.1	2.9	-	100
+ 75	7.9	7.3	85.4	9.0	6.3	3.3	0.4	5.9	-	100
+ 53	6.4	5.7	88.2	8.8	5.4	2.7	-	-	-	100
Total of Distribution/ Average Proportion	8.6	100	75.7	100	15.1	100	0.5	100	0.1	100

bulk of apatite mineral is located between 106 and 425 μ m from the apatite distribution and that calcite is evenly distributed throughout the size range with other oxides being predominantly concentrated in the coarse size range due to preferential grinding.

Problems were encountered in identifying biotite due to contamination of fines on the grains making it difficult to optically identify especially at fine sizes.

1.4 Liberation Study

Liberation of the phosphate minerals is important for any subsequent separation techniques. A liberation study was made on the ore to determine the amount of grinding necessary to produce an acceptable percentage of liberated apatite, calcite and opaque minerals.

Various size fractions of the crushed ore were examined with a petrographic microscope to determine the particle size range at which the apatite and the major gangue minerals were liberated. Mineral particles were assigned to each of the following categories according to their degree of liberation; 0-25%, 25-50%, 50-75%, 75-99% and 100%. Using the point counting technique, frequencies of particles in each range were obtained from which an average liberation for each mineral in each size fraction was calculated using a grouped mean average (7).

$$\text{Thus, } \bar{x} = \frac{\sum f_i x_i}{\sum f_i}$$

where x_i - midpoint value of each range

\bar{x} - average liberation

f_i - frequency

Range	x_i
0-25	12.5
25-50	37.5
50-75	62.5
75-99	87.0
100	100

The results are shown in table 2 of the appendices and summarised in figure 8.

An ore is not usually considered "liberated" until at least 80% of the minerals occur as free grains. Therefore, on the basis of the point counting data for Kaluwe ore presented in figure 8, the $-425 + 106\mu\text{m}$ material is the coarsest size fraction with at least 80% liberated grains. Again, this agrees with the earlier finding that apatite was liberated in this size range.

Calcite, being the major gangue mineral and forming the groundmass of the ore was about 60% liberated at coarser fractions though the degree of liberation increased with a decrease in size. The opaques which formed the iron minerals were marginally liberated at coarse fractions until

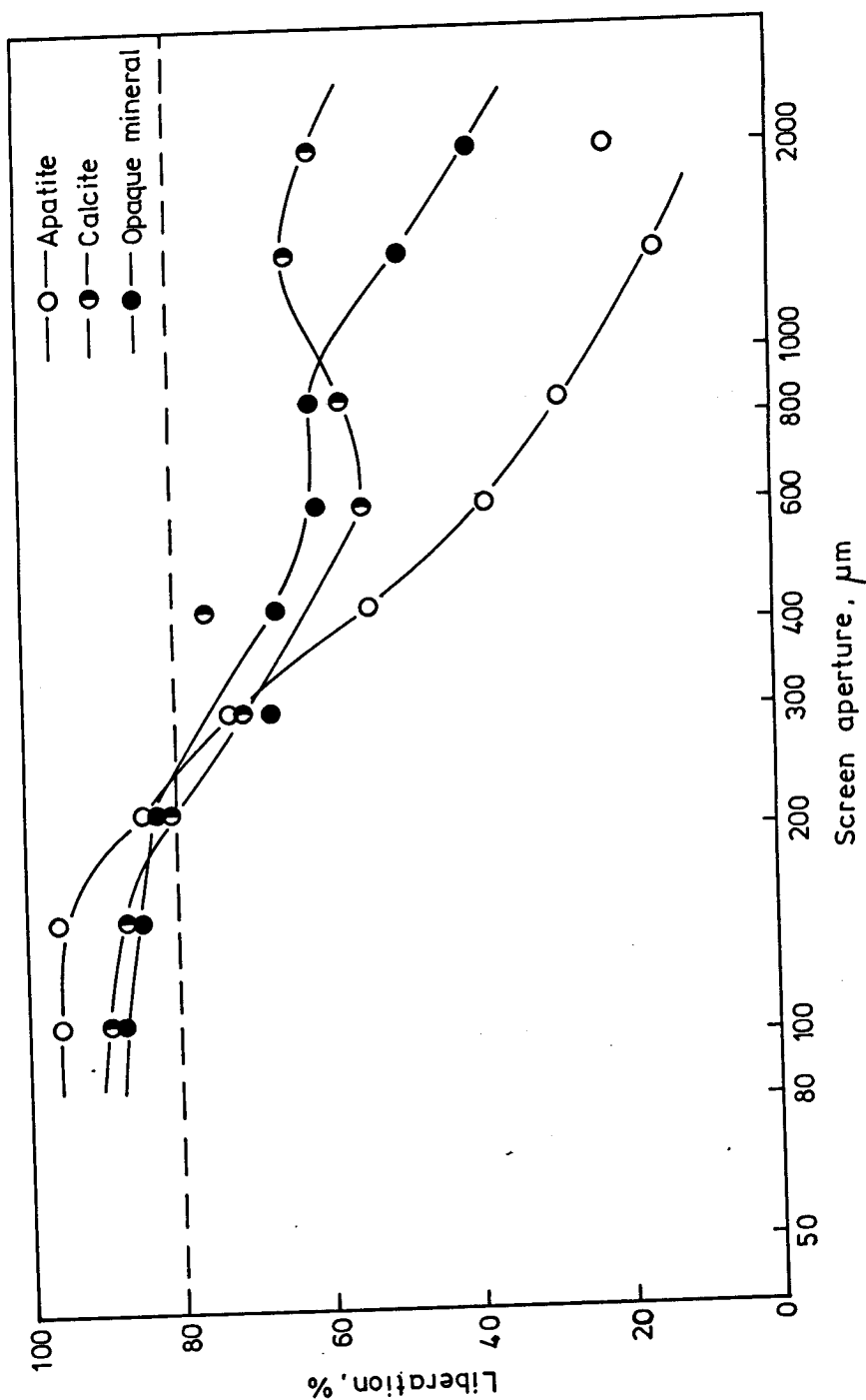


Fig. 8. Effect of particle size on the liberation characteristics of apatite, calcite and opaque minerals.

about $850\mu\text{m}$ when about 60 percent liberation was noted.

Thereafter the degree of liberation increased.

Generally the opaques were intergrown with calcite and in some cases apatite appeared as inclusions in the opaque minerals at coarse size fractions. Determining the liberation characteristics of the three minerals on the +75 and $+53\mu\text{m}$ fractions proved difficult due to the presence of fines which made recognition of the minerals difficult as they appeared as aggregates. The $-425 +53\mu\text{m}$ size range was considered for subsequent beneficiation processes since it was at least 80% liberated.

CHAPTER TWO

PROCESS SELECTION

2.1 Gravity Concentration Methods

Concentration of phosphate ores by gravity methods requires a marked difference in the settling rate between the phosphate and gangue minerals. As the difference in specific gravity of the minerals being separated decreases, the minimum particle size that can be treated increases.

There are many expressions used to determine the amenability of ores to gravity concentration. One expression which consistently appears in discussions of gravity concentration is the concentration criterion⁽⁸⁾. This is usually defined as: The specific gravity of the heavy species minus that of the suspending fluid, divided by the specific gravity of the light species minus that of the suspending fluid. Algebraically this is:

$$\text{Concentration Criterion (CC)} = (S_h - S_f) / (S_l - S_f) \quad (1)$$

where: S_h = specific gravity of the
heavy mineral

S_l = specific gravity of the light
mineral

S_f = specific gravity of the sus-
pending fluid.

In general terms, when the concentration criterion is greater than 2.5, whether positive or negative, then gravity separation is relatively easy. As the value of

the quotient decreases, so the efficiency of separation decreases, and below 1.25 gravity concentration is not generally commercially feasible.

The motion of a particle in a fluid is dependent not only on its specific gravity, but also on its size; large particles will be affected more than smaller ones. The efficiency of gravity processes therefore increases with particle size. In practice, close size control of feeds to gravity processes is required in order to reduce the size effect and make the relative motion of the particles specific gravity dependent.

Heavy medium separation (HMS) is the simplest of all gravity concentration processes and can be closely duplicated in the laboratory. It is a primary tool in the development of gravity concentration flowsheets. Heavy liquids, generally of comparatively high toxicity, are used e.g. bromoform (s.g = 2.89), tetrabromoethane (sg = 2.96) etc. It is impractical to use these in commercial applications and suspensions of fine magnetite and/or ferrosilicon in water are employed.

In a static separatory HMS vessel, the essential separating force is gravitational and the essential counter force is the resistance to viscous shear. If a range of particles of different specific gravity and size are placed in a fully static vessel filled with a true liquid of a density between the highest and lowest specific gravities of particles, then those particles with a specific gravity exactly equal to the liquid density will hover. All other particles will,

given sufficient time, either sink or float.

Except for very large, heavy or light particles, the rate of sinking, or floating, will be determined by Stokes' Law and will be dependent on particle size, the density differential between solid and fluid and inversely dependent on fluid viscosity.

Table 5 shows some of the physical properties of the main minerals in Kaluwe ore. Based on the figures shown, the concentration criterion for separation of apatite from calcite in water is 1.29. This is slightly above the bottom limit showing that gravity separation is possible but the expected efficiency would be low.

Table 5. Physical properties of main minerals present (9).

Mineral name	S.G.	Hardness Mohr
Apatite	3.2	4.5-5.0
Calcite	2.71	3.0
Hematite	5.1	5.5-6.5
Magnetite	5.17	5.5-6.5

Heavy medium separation experiments were carried out on all the size fractions considered. The apparatus was set up as shown in figure 9 using bromoform as the heavy medium (S.g = 2.89) and the procedure outlined in the appendices.

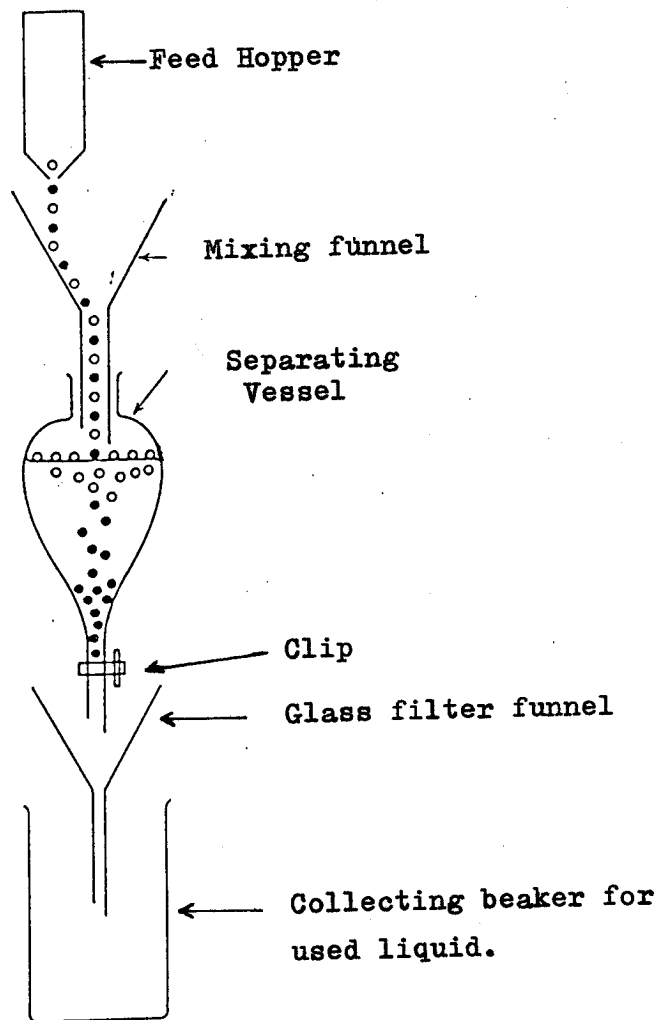


Fig. 9. A stationary density separator for mineral separation
(Retort stand not shown)

The results are shown in tables 6 and 7 and summarised in figure 10. The results show that the P_2O_5 grade of the sinks increased with decrease in particle size. Figure 10 shows that in the size range 106 to $600\mu m$ the recovery was more than 20%. Below $106\mu m$ it decreased. This can be attributed to the fine nature of particles which found it difficult to settle through the heavy liquid and so remained in the float.

Table 6. Effect of particle size on the grade and recovery of P_2O_5 in the sink product.

Size Fraction μm	Amount of Feed, g	Grade of Feed, % P_2O_5	Amount of Sinks, g	Grade of Sinks % P_2O_5	Recovery %	ER
+2000	29.9	1.7	1.9	3.6	13.5	1.71
+1400	30.0	1.6	1.7	4.0	14.2	1.90
+ 850	29.9	1.7	2.1	3.8	15.7	1.81
+ 600	29.8	1.9	2.4	5.0	21.2	2.38
+ 425	29.9	2.1	2.8	6.5	29.0	3.10
+ 300	29.8	3.0	3.4	8.8	33.5	4.19
+ 212	29.8	2.9	4.1	8.0	38.0	3.81
+ 150	29.7	3.0	4.2	6.6	31.1	3.14
+ 106	29.7	2.7	1.8	10.0	22.4	4.76
+ 75	29.6	1.7	1.0	7.9	15.7	3.76
+ 53	29.5	1.8	0.6	3.3	3.7	1.57
Average/ Total	327.6	2.1	26.0	6.5	24.7	3.1

Table 7. Effect of particle size on the grade of floats

Size Fraction μm	Grade of Floats % P_2O_5
+2000	1.3
+1400	2.0
+ 850	1.5
+ 600	1.5
+ 425	1.0
+ 300	0.8
+ 212	0.7
+ 150	0.7
+ 106	2.1
+ 75	2.0
+ 53	1.5
Average	1.5

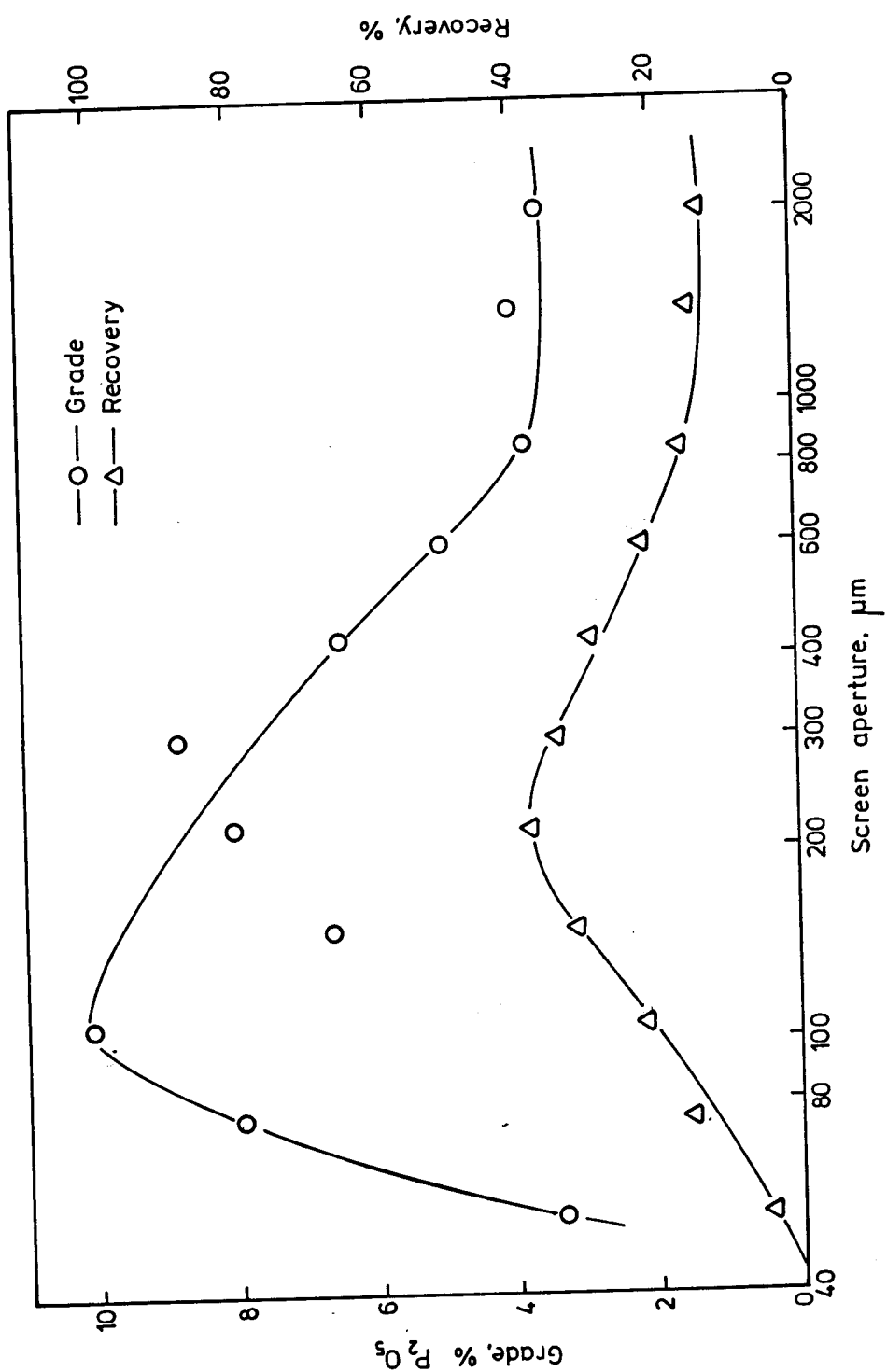


Fig. 10. Effect of particle size on the grade and recovery of phosphate in the sinks (S.G. 2.89).

In the coarser fractions, the decrease in recovery can be attributed to the presence of composite apatite particles in the float. The maximum grade obtained was 10% P_2O_5 in the $+106\mu m$ size fraction and a maximum recovery of 38% was obtained in the $+212\mu m$ size fraction.

Visual examination of the sinks under a binocular microscope revealed that they consisted predominantly of a black mineral (iron oxides) and a pale yellow mineral (apatite). More apatite particles were present as the size decreased and were predominant in the $+106$ to $-425\mu m$ size range. The sinks in the coarse fractions were mainly composite in nature. Difficulties were encountered in separating the $+75$ and $+53\mu m$ fractions and so these were left for long hours in order to effect separation. The decrease in grade at fine fractions can be attributed to "bunching" of fine particles, mainly gangue, which found their way into the sinks thereby decreasing the P_2O_5 grade. There was a marked decrease in the weight of the sinks at finer sizes. This may be due to "crowding" of the particles fed in the vessel. A large mass of low density particles moving upwards will inevitably entrain some high density particles resulting in decreased weight of the sinks.

Mineralogical studies were carried out on the sink products and the results are shown in table 8. The main feature from the table was that the proportion of apatite in the sinks increased with decreasing particle size and was more in the $+106$ to $-425\mu m$ size range. This agreed with the results from chemical analysis.

TABLE 8. Mineral proportions and estimated liberation of apatite in the sink fractions

Size Fraction μm	Apatite		Calcite	Opakes	Quartz/ Feldspar	Biotite	Total
	Proportion %	Estimated liberation %					
+2000	9.2	22.7	54.9	35.0	0.7	0.9	100
+1400	8.9	24.0	52.4	40.0	0.8	0.6	100
+ 850	7.3	31.0	52.8	39.0	0.4	0.5	100
+ 600	8.0	34.5	57.5	33.4	0.4	0.7	100
+ 425	14.7	59.4	32.8	51.8	0.1	0.6	100
+ 300	19.3	70.4	31.3	49.0	0.1	0.3	100
+ 212	29.9	87.3	15.7	53.5	0.9	-	100
+ 150	30.6	96.6	14.8	53.9	0.6	0.4	100
+ 106	31.2	98.7	17.0	51.6	0.2	-	100
+ 75	12.3	nd	21.8	65.9	-	-	100
+ 53	10.7	nd	37.5	51.8	-	-	100
Average Proportion	19.2		32.4	47.5	0.5	0.4	100

The calcite proportion reduced in the same size range.

The opaque minerals showed an increase in proportion with decrease in particle size. This was expected as they constituted the heavier fraction of the minerals present and so reported in the sink fractions.

It is clear from the above tests that gravity concentration of Kaluwe ore is possible and that it should be restricted to the size range +106 to -425 μ m. However, gravity concentration alone cannot achieve high grades since there is the question of iron minerals reporting to the sinks. So gravity concentration should be carried out in combination with magnetic separation in order to achieve meaningful results.

Since beneficiation tests should be restricted to this size range, drum separators would not be suitable since they require coarser sizes although they operate more efficiently if the float constitutes the bulk of the feed as is the case with this ore. Jigs are applicable in the size range 25 mm to 0.20 mm which is a bit higher than the one being considered here. In this case, shaking tables which treat a finer size range (10mm to 50 μ m) and spirals which are limited to 3mm to 76 μ m are suitable to treat this size range.

2.2 Magnetic separation

Mineralogical studies revealed that the ore contained about 10-15% iron minerals. Testing with a hand magnet showed that the ore did contain some magnetic fractions. Since magnetic

susceptibilities for iron minerals lie below that of apatite, magnetic separation can be applied to remove them at current settings less than 1.4 A. Due to the large amount of calcite and the low grade nature of the ore in terms of P_2O_5 , magnetic separation can only be carried out in combination with other beneficiation processes in order to yield positive results. It can either be carried out as a preconcentration step before subsequent beneficiation tests or as a last step in a beneficiation process. Due to the above reasons, it was decided to include magnetic separation in the process tests for Kaluwe ore.

2.3 Flotation

As has been elucidated in chapter 1, liberation of apatite occurs in the medium size range. The low concentration criterion for separation of apatite from calcite using gravity concentration renders it impossible for gravity concentration techniques to be used as the main beneficiation techniques. In this regard, it was decided that flotation would possibly be more efficient in the beneficiation of Kaluwe ore.

One obvious problem with this ore is its friable nature which leads to inevitable production of slimes. These produce slime coatings on larger particles which are

detrimental to flotation in several ways. If the slimes are the valuable mineral and the coarse particles are the gangue minerals, the grade of the concentrate becomes poor. When the slimes consist of gangue minerals and the coarse particles of the valuable mineral, these particles prevent the attachment of air bubbles and recovery of the valuable mineral may then decrease significantly.

Based on this premise, previous work has attempted some interesting variations in flotation techniques. One of the obvious choices is the increase in the particle size by aggregation followed by flotation (10).

Floc-flotation is one such example. If, however, the slimes constitute mainly the gangue minerals, desliming would alleviate some of the problems highlighted above. It was therefore, envisaged to include a desliming stage in the process tests.

2.4 Magnetic/Heavy Media Separation

Due to the reasons advanced earlier, magnetic and heavy medium separations were carried out consecutively.

The magnetic separation procedure is outlined in the next chapter. After magnetic separation at 1.4 A, to remove iron minerals, the non-magnetic fractions which contained most of the apatite were subjected to heavy medium separation using bromoform (s.g. = 2.89). Table 9 shows

Table 9. Effect of particle size on the grade and recovery of P_2O_5 on sink product of the non-magnetic fraction (at 1.4 A).

Size Fraction μm	Amount of feed (non-magnetic), g	Grade of Feed, % P_2O_5	Amount of sinks, g	Grade of sinks, % P_2O_5	Recovery %
+1400	61.8	1.5	0.9	18.8	18.3
+ 850	45.2	1.4	0.7	21.3	23.6
+ 600	57.7	2.3	1.7	21.4	27.4
+ 425	62.1	2.2	1.9	24.2	33.7
+ 300	72.8	2.4	3.6	30.1	62.0
+ 212	78.0	3.6	6.0	29.9	63.9
+ 150	57.1	5.3	5.0	32.1	53.0
+ 106	53.0	3.6	3.0	29.4	46.2
+ 75	49.7	2.8	0.5	25.1	9.2
Average/ Total	537.4	2.8	23.3	28.5	44.1

the results of the combined magnetic/heavy medium separations at various size fractions. It is clear from the table that generally there was a marked increase in the P_2O_5 grade throughout the particle size range investigated with a maximum being 32% P_2O_5 in the +150 μ m size fraction. Figure 11 was drawn from the data in table 9. The main feature from the figure is that there was maximum recovery and grade in the size range 106 to about -425 μ m in which apatite was substantially liberated. The reasons for a drop in grade and recovery at coarser and finer fractions have already been discussed.

Table 10 shows the effect of particle size on the P_2O_5 grade in the float fractions. It can be seen that the grade decreased with size and increased again at finer fractions. This conforms with the earlier observations that the apatite was liberated in the intermediate size range and so most of it reported in the sinks; that at fine fractions some phosphate was entrained in the float and the phosphate values were present as inclusions at coarse fraction.

Mineralogical studies were carried out on the sink and float products and the results are shown in tables 11 and 12 respectively. It is clear from table 11 that the proportion of apatite was substantially increased throughout the size range studied after combining the two processes and it increased with decrease in size. This is in agreement with

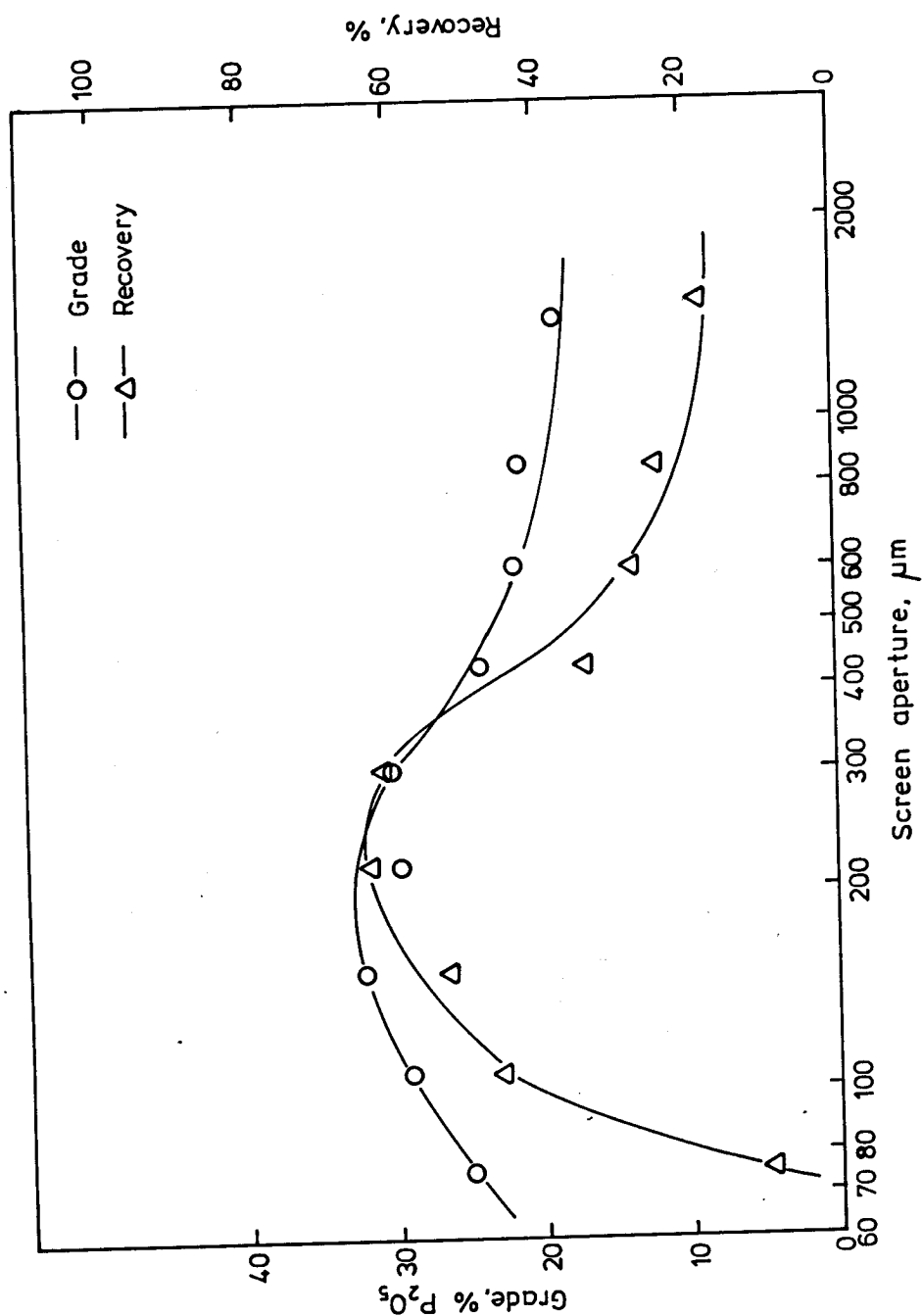


Fig. 11. Effect of particle size on the grade and recovery of phosphate in the sinks (S.G 2.89) of the non-magnetic fraction.

Table 10. Effect of particle size on the grade of floats
of the non-magnetic fraction (at 1.4 A)

Size Fraction μm	Grade of floats % P_2O_5
+1400	3.0
+ 850	1.5
+ 600	1.0
+ 425	0.6
+ 300	0.6
+ 212	0.5
+ 150	0.7
+ 106	1.2
+ 75	2.8
Average	1.4

Table 11. Effect of particle size on the mineral proportions and liberation of apatite in the sink product of the non-magnetic fraction (1.4 A).

Size Fraction μm	Apatite		Calcite	Opakes	uartz/Feldspar	Total of Proportion
	Proportion %	Estimated liberation %	Proportion %	Proportion %	Proportion %	
+1400	17.2	25	57.2	25.0	0.6	100
+ 850	23.2	32	50.8	25.4	0.6	100
+ 600	32.7	53	39.4	26.1	1.8	100
+ 425	35.6	58	34.9	28.1	1.4	100
+ 300	64.6	88	21.2	13.3	0.9	100
+ 212	69.5	94	5.5	24.8	0.2	100
+ 150	83.8	97	5.4	10.8	-	100
+ 106	82.0	97	3.5	14.5	-	100

the chemical analysis results. The proportion of calcite in the sink product was drastically reduced especially at sizes less than $425\mu\text{m}$. As can be seen from table 12, the proportion of calcite in the float product increased and averaged about 88.7 %. Mineralogical studies on the $+75\mu\text{m}$ size fraction were not carried out. This was due to the problems encountered in making a thin section for this fraction.

The results show that a concentrate of about 28.5% P_2O_5 can be produced by combining the two processes at a recovery of about 44.1%.

2.5 Scrubbing

Due to staining of the apatite and calcite by iron oxides, scrubbing tests were done in order to ascertain whether this would clean the surfaces of the minerals. The tests were done using a Denver flotation cell (used as a scrubber) at a pulp density of 75% solids w/v. Results showed that after scrubbing for about 5 minutes the pulp became thicker due to production of fines. This was attributed to the friable nature of the ore. In view of this, including scrubbing in the treatment process would be deleterious in that there would be production of more fines which would lead to increased losses of the phosphate values in the slime waste.

Table 12. Effect of particle size on the mineral proportions on the float
product of the non-magnetic material

Size Fraction μm	Apatite	Calcite	Opakes	Quartz/Feldspar	Total
	Proportion %	Proportion %	Proportion %	Proportion %	
+1400	3.1	90.8	4.6	1.5	100
+ 850	4.9	86.6	8.0	0.5	100
+ 600	5.9	87.2	6.4	0.5	100
+ 425	6.3	86.5	6.6	0.6	100
+ 300	3.2	92.3	3.2	1.3	100
+ 212	6.5	85.0	6.9	1.6	100
+ 150	2.7	92.5	4.6	0.2	100
+ 106	3.5	89.1	7.1	0.3	100
Average	4.6	88.7	5.8	0.9	100

2.6 Electrokinetic studies

Electrokinetics is the general description of phenomena arising when attempts are made to shear off the mobile part of the double layer from the charged surface. If an electric field is applied, the surface and the diffuse layer move in opposite directions. Conversely an electric field is created if the charged surface and the diffuse part of the double layer are made to move relative to each other. The plane of shear is located immediately outside the stern plane. The potential of this plane is known as the zeta potential. It is the only potential that can be measured by electrokinetic methods. There are several methods available. The most popular one is microelectrophoresis in which very fine particles are observed under a microscope during migration to the oppositely charged electrode. The particles are timed between a known distance and the value of the zeta potential obtained from the electrophoretic mobility, (EM) by application of the Helmholtz-Smoluchowski equation:

$$\frac{V}{E} = EM = \frac{\zeta D}{4\pi\eta} \quad (2)$$

where V = velocity of the particle (cm s^{-1})

D = dielectric constant (78 for water)

E = applied potential (esu/cm)

ζ = zeta potential (esu)

η = viscosity of water (0.01 poise)

For aqueous solutions at 25°C, the following equation is used

$$\zeta \text{ (mV)} = 12.83 \times \text{EM} \text{ (}\mu\text{m/sec/V/cm)} \quad (3)$$

Methods of beneficiation of phosphate ores include surface chemical methods such as flotation and flocculation. The performance of such operations depends primarily on the electrochemical properties of the apatite surface and associated gangue minerals. Such information can be applied to the separation of phosphates from the gangue minerals by selective flotation. In view of this, it was decided to carry out surface charge measurements on the major minerals of the ore in order to find out their behaviour in aqueous solutions in the absence or presence of various reagents. These tests were carried out in both distilled and Kaluwe water. The main interest was to find out their behaviour in Kaluwe water since it came from a river near the deposit and that if beneficiation tests worked using this type of water then there would be no problems in using it in a plant in as far as surface chemistry is concerned.

Surface charge measurements were made with a Rank Brothers MK II microelectrophoresis apparatus fitted with a video camera.

Apatite grains (fluor-hydroxyapatite) were, with the help of a binocular microscope, hand picked from ^a -425 + 300 μm size fraction and washed in 0.2M nitric acid to eliminate any trace of calcite present at the mineral surface. Nitric acid was

used for washing because the nitrate ion is commonly known to behave as an indifferent ion for apatite i.e. does not specifically adsorb on the apatite surface. This was followed by washing and rewashing in hot and cold distilled water for four times to remove nitrate ions.

Calcite grains were also hand picked from the ore while the magnetite grains were obtained by passing a hand magnet over the ore sample. The minerals were ground in a clean agate pestle and mortar separately and the powders put in sample containers ready for use.

A supporting electrolyte of 10^{-3} M sodium chloride was used in all the tests. The Na^+ ion does not specifically adsorb, that is, it behaves indifferently for apatites and most oxides. The tests were conducted in distilled water with a conductivity of $0.2 \times 10^{-4} \text{ ohm}^{-1} \text{ cm}^{-1}$ and Kaluwe water with a conductivity of $2.8 \times 10^{-4} \text{ ohm}^{-1} \text{ cm}^{-1}$. A microelectrophoresis technique was used to determine the electrophoretic mobility (EM) values. The microelectrophoresis cell was rectangular with platinum electrodes which were cleaned by washing with water after a run.

The conductivities were measured using a philips R.P. 9500/1000/50 cycles-per-second bridge and a philips P.R 95100 conducting cell.

Samples for microelectrophoresis tests were prepared by making a solution of the desired pH by adding the appropriate volume of standard HCl or NaOH solution. The mineral sample was

added to this solution. The suspension was then conditioned using a magnetic stirrer. The pH values reported in this work are the values measured after the conditioning period and at the time of the microelectrophoresis test.

Twenty particles were timed at each pH value in both directions and the electrophoretic mobility (EM) determined from the mean of the measurements. The electrophoresis cell was used in a room in which the temperature varied from 24-26°C. The cell was flushed several times with distilled water before use and the measurements were done as quickly as possible. A sample of the suspension was used for pH and conductivity measurements at room temperature.

The equation used for computing the electrophoretic mobility was:

$$EM = \frac{XL}{\bar{t}V} \quad (\mu\text{m/sec/V/cm}) \quad (4)$$

where X = distance between the graticule in
binoculars (μm)

L = effective interelectrode distance (cm)

\bar{t} = average of times (sec)

V = applied potential (V)

Figure 12 shows surface charge measurements in distilled water for apatite, calcite and magnetite from Kaluwe ore at a constant ionic strength of 10^{-3}M NaCl. Magnetite was used to represent the iron minerals since they are expected to behave in a similar manner. The iso-electric point of apatite was found to be about 3.8.

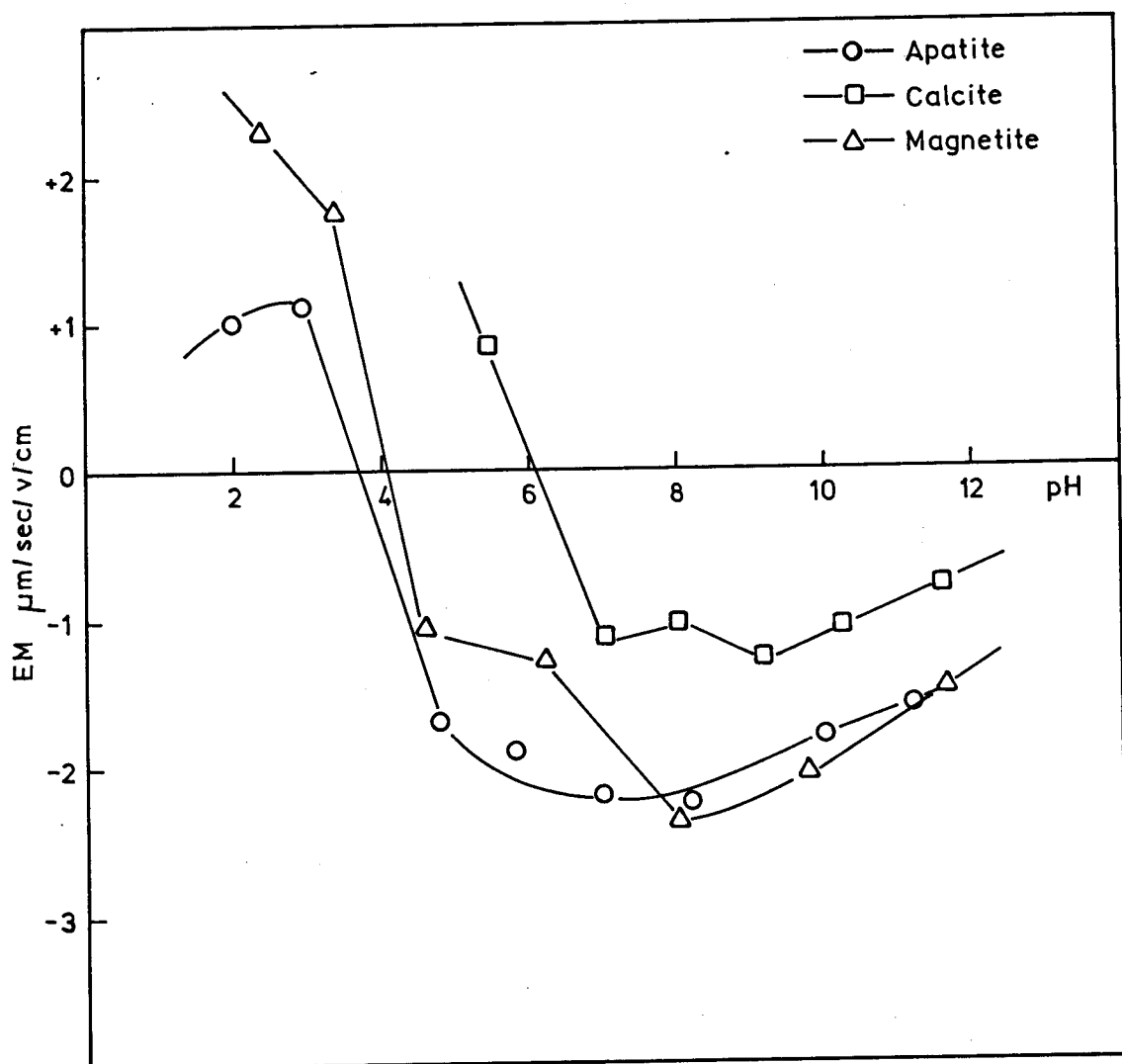


Fig. 12. Effect of pH on EM of apatite, calcite and magnetite from Kaluwe in distilled water at constant ionic strength of 10^{-3} M NaCl.

The iso-electric point (IEP) describes that condition of the system at which the potential at the plane of shear, i.e., the zeta potential (or Electrophoretic mobility,) as obtained from electrokinetic measurements is zero.

Below pH 3.8 apatite was positively charged while above this value it was negatively charged. The IEP of calcite was at pH 6.1. Above this value it was negatively charged and below it the calcite was positively charged. Magnetite gave an IEP of 4.1 above which it was negatively charged and below which it was positively charged.

Electrokinetic results of figure 12 suggested that a theoretical separation could be made by floating apatite with magnetite from calcite using a cationic collector in the pH range 4.1 to 6.1. The iron oxides could then be separated by magnetic separation. However, in practice ores with calcareous gangue, which make up the bulk of world-wide reserves, are difficult to separate by the flotation technique due to the similarity in surface cations (Ca^{2+}). Collectors used for one mineral generally float the other as well, and it is only by use of suitable depressants of phosphates or calcareous gangue that selectivity is obtained. This difficulty in achieving natural selectivity is due to the small range of pH in which the surface charges on the apatite and calcite are different. What the depressants do is to enhance the surface charge on either minerals so that there would be a marked difference which makes selectivity in flotation possible.

In the flotation tests of Kaluwe phosphate deposit done by other workers, the flotation pH range used was 8-10.5. Selective flotation of apatite from the gangue (mainly calcite and iron oxides) would not be possible in distilled water in this pH range since the minerals were negatively charged. The lack of selectivity between apatite and calcite experienced by other workers (4, 5) may have been due to a similar system (fig. 12). Mineralogical studies had revealed that calcite from the ore was stained. This is also supported by previous work (4) that there is limonitic staining on the apatite. To investigate this point further, surface charge measurements were done on the calcite from the ore and relatively pure calcite. The results of these measurements are shown in figure 13. The calcite from the ore showed an IEP of 6.1 while pure calcite gave an IEP of about 8.4. It can therefore be concluded that the calcite was stained with iron oxides; thus the IEP shifted towards the typical IEP values of iron oxides as can be seen in the figure. Owing to the extreme solubility of calcite, no experiments were conducted with this mineral much below pH 5.

2.6.1 Surface charge measurements in Kaluwe water

If a plant for the flotation of Kaluwe ore was to be set up, Kaluwe water would definitely be used in the treatment process. It was for this reason that electrokinetic studies were done in Kaluwe water in order to find out whether the ions present in solution would give surface charge measurements different from those obtained in distilled water.

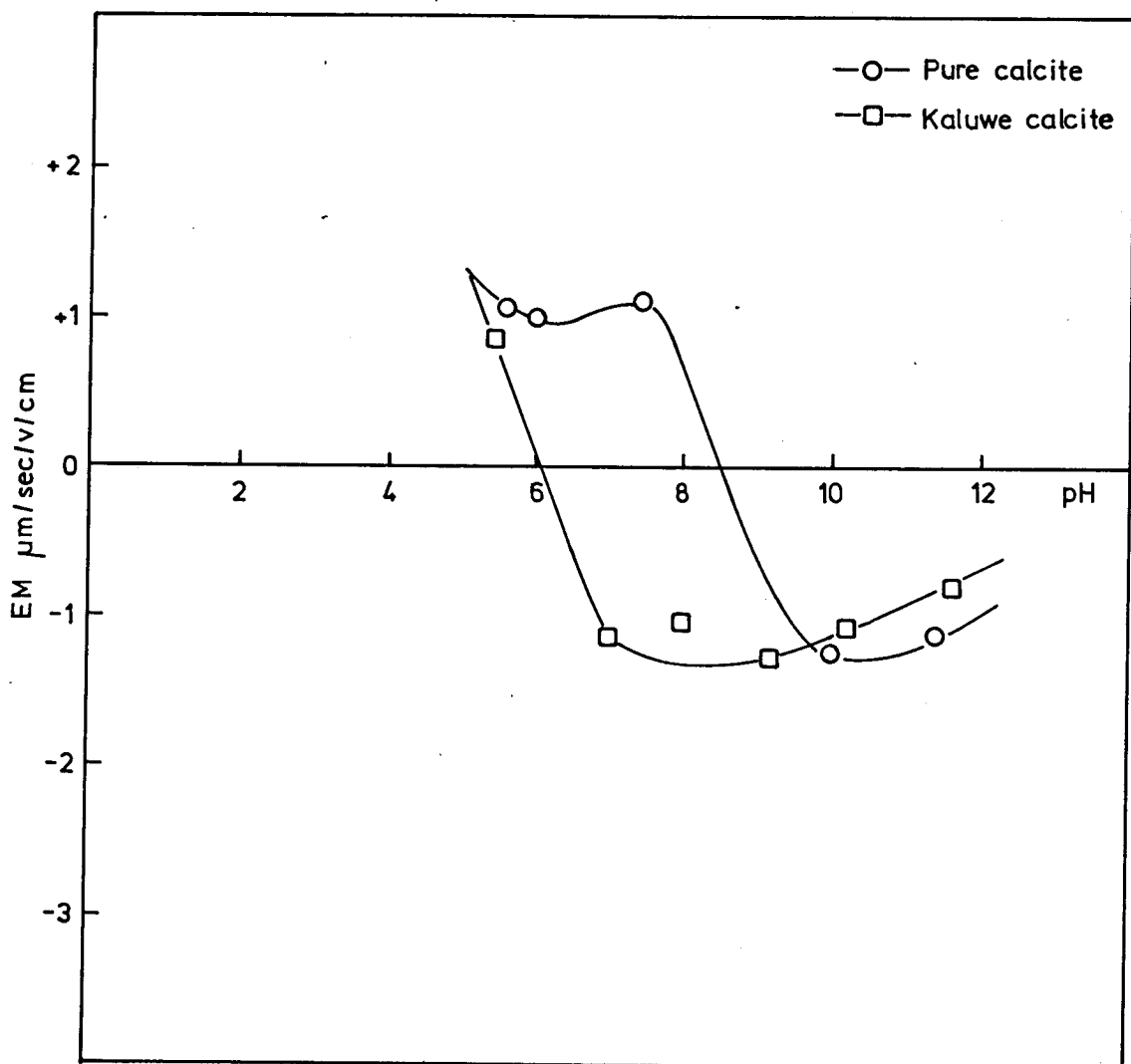


Fig. 13. Effect of pH on EM of pure calcite and Kaluwe calcite in distilled water of constant ionic strength of 10^{-3}M NaCl.

The measurements were done at a constant ionic strength of $10^{-3}M$ NaCl in Kaluwe water. The results are shown in figure 14.

The figure gave an IEP of 3.3 for apatite and 4.4 for magnetite. These results show that there was no marked difference in the IEPs of these minerals from those obtained in distilled water. Calcite showed a charge reversal at pH values less than 6 and above 8.8. The IEP in Kaluwe water was 8.8 above which the surface charge was positive and below which the charge was negative. This meant that there were some cations or anions in Kaluwe water which were specifically adsorbing on the calcite in the pH range of charge reversal. Chemical analysis of the water showed that it had high Ca^{2+} and CO_3^{2-} contents. Both of these ions have been found by other workers / (11, 12, 13) to be potential-determining for calcite. In the pH range where the reversal occurred from positive to negative, the CO_3^{2-} may have been specifically adsorbing on the surface and in the other case the Ca^{2+} may have been active. These ions were presumably dissolved from the abundant calcareous minerals in the region.

These tests showed encouraging results in that at pH 8.8 to 11, there was a marked difference in the surface charge of calcite from that of apatite and magnetite. This suggests that in this pH range, it is possible to selectively float the apatite from the calcite using a cationic collector. The magnetite would also float but this could be removed by magnetic separation.

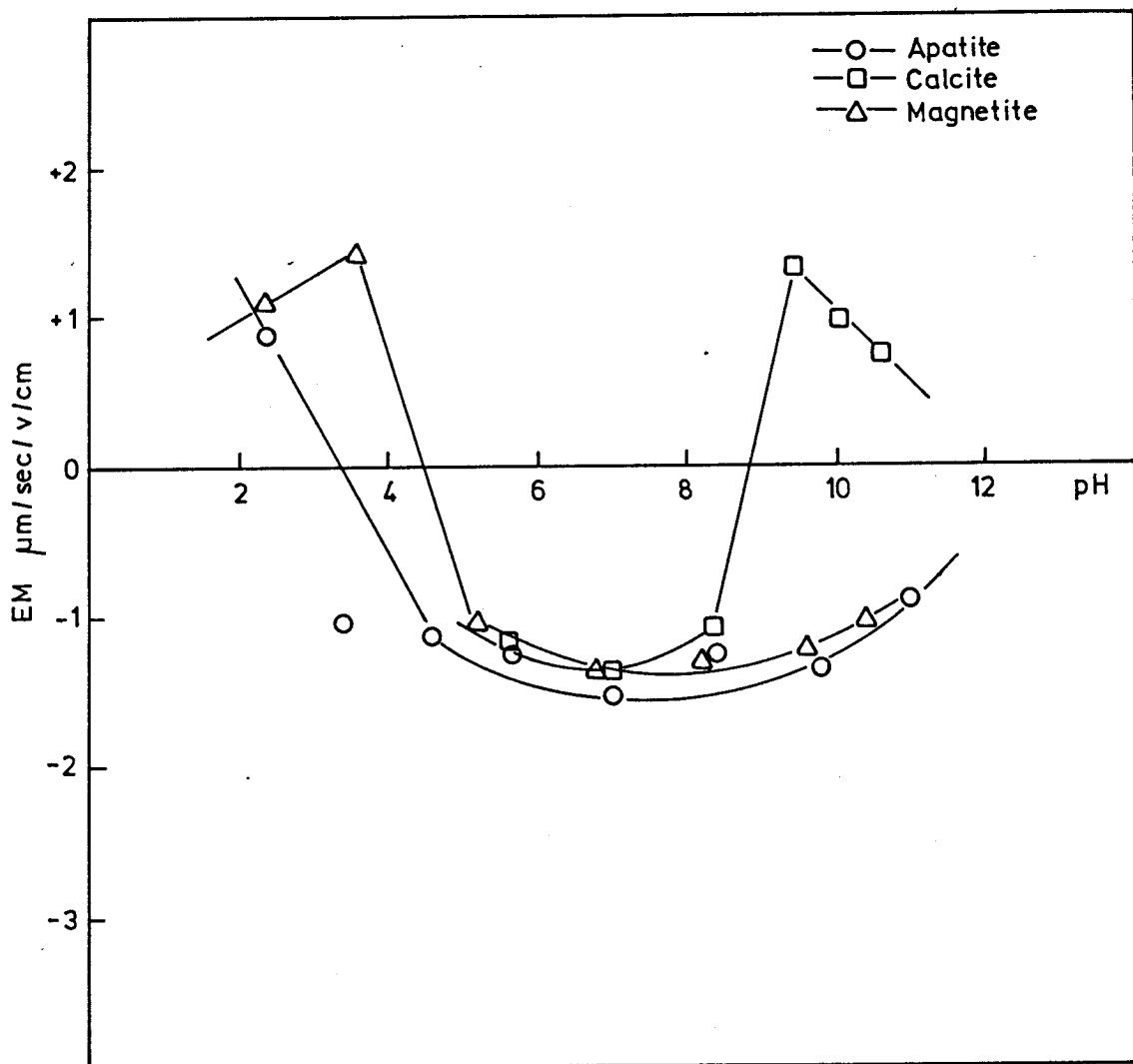


Fig. 14. Effect of pH on EM of apatite, calcite and magnetite from Kaluwe in Kaluwe water at constant ionic strength of 10^{-3} M NaCl.

2.6.1.1 Effect of calcium chloride on the EM of apatite and calcite in Kaluwe water

In the flotation of apatite from a calcareous gangue, various depressants are used some of which may contain Ca^{2+} , CO_3^{2-} or PO_4^{3-} as the active species which when put in solution would modify the surfaces of the minerals in order to selectively separate them. In view of this, surface charge measurements were carried out in an electrolyte containing calcium chloride from which Ca^{2+} cations were derived. The results are shown in figure 15. The results show that, for apatite, there was a charge reversal above pH 6.8 from negative to positive. This was due to Ca^{2+} species which specifically adsorbed on the apatite as counter ions above pH 6.8 thereby reversing the charge. For calcite, charge reversal occurred between pH 6 and 8.8 from negative to positive. However, above pH 8.8 there was an increase in the positive potential on the calcite showing that the Ca^{2+} was specifically adsorbing.

From the above observations it can be concluded that a reagent containing Ca^{2+} as the active species cannot be used as a modifying agent for separations between apatite and calcite by flotation in Kaluwe water. This is because the two minerals responded similarly to the presence of Ca^{2+} species.

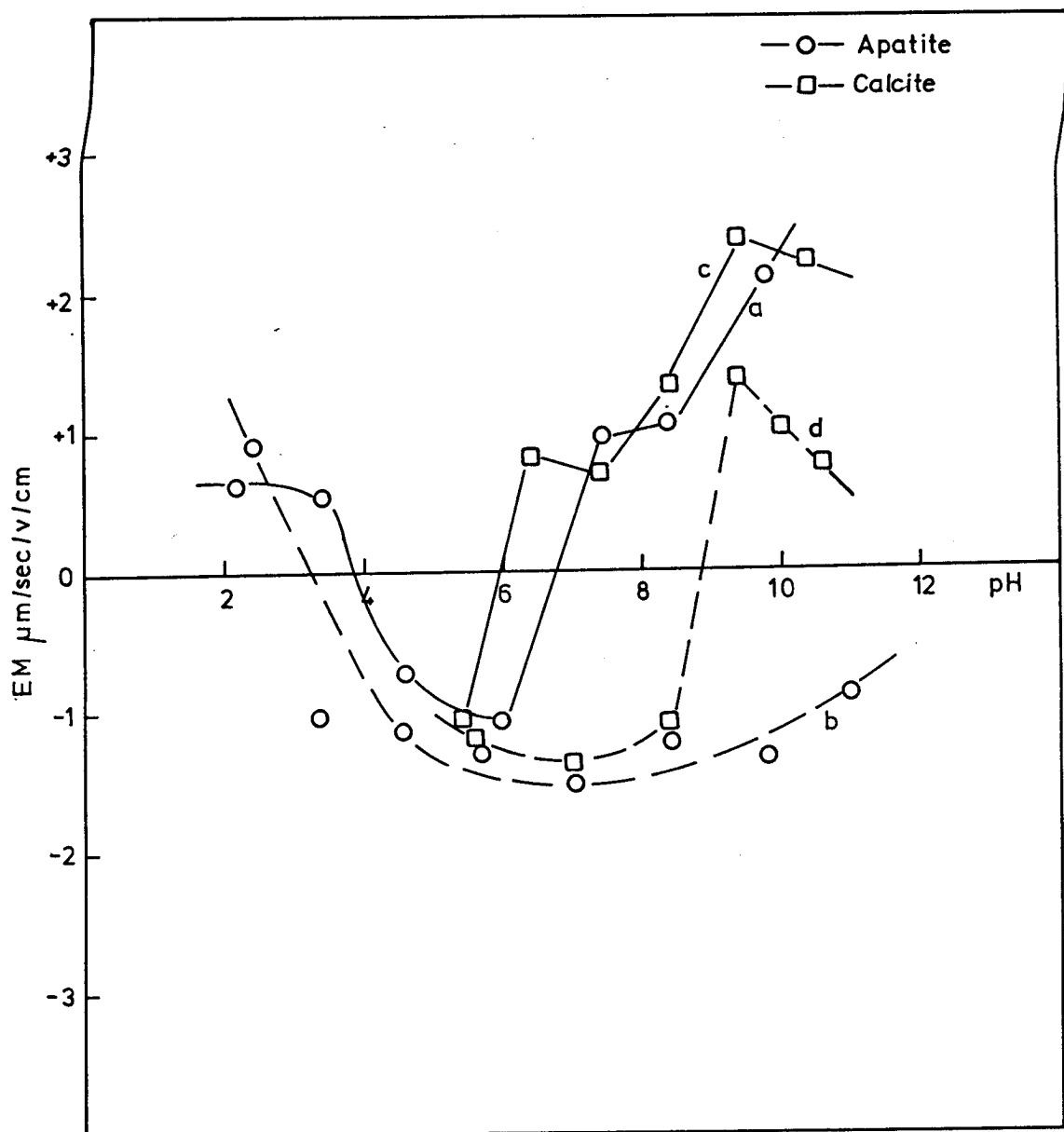


Fig. 15. Effect of pH on EM of apatite and calcite from Kaluwe in Kaluwe water. a,c= containing $2 \times 10^{-4} \text{ M}$ CaCl_2 b,d= Replot in 10^{-3} M NaCl .

2.6.1.2 Effect of sodium silicate on the EM of apatite and calcite in Kaluwe water

Selective flotation of nonmetallic minerals is difficult to achieve with fatty acids or soaps by themselves. As a result, specific reagents are added to aid these separations, and one of the reagents commonly employed for this purpose is sodium silicate. Flotation separations of various calcium-bearing minerals such as fluorite from calcite and scheelite from calcite and apatite, for example, almost always involve the use of sodium silicate. The mechanisms by which sodium silicate functions as a depressant are still not well understood. For one thing, the dissolution process of sodium silicate is complex, giving rise to a number of ionic and colloidal species.

In view of the wide application of sodium silicate in the flotation of calcium-bearing minerals, it was decided to carry out surface charge measurements of calcite and apatite in Kaluwe water in the presence of 2×10^{-4} M sodium silicate. The results of the measurements are shown in figure 16. The results show that between pH 8 and 8.8 the calcite became more negative and charge reversal occurred above pH 8.8. Thus, calcite showed a negative potential

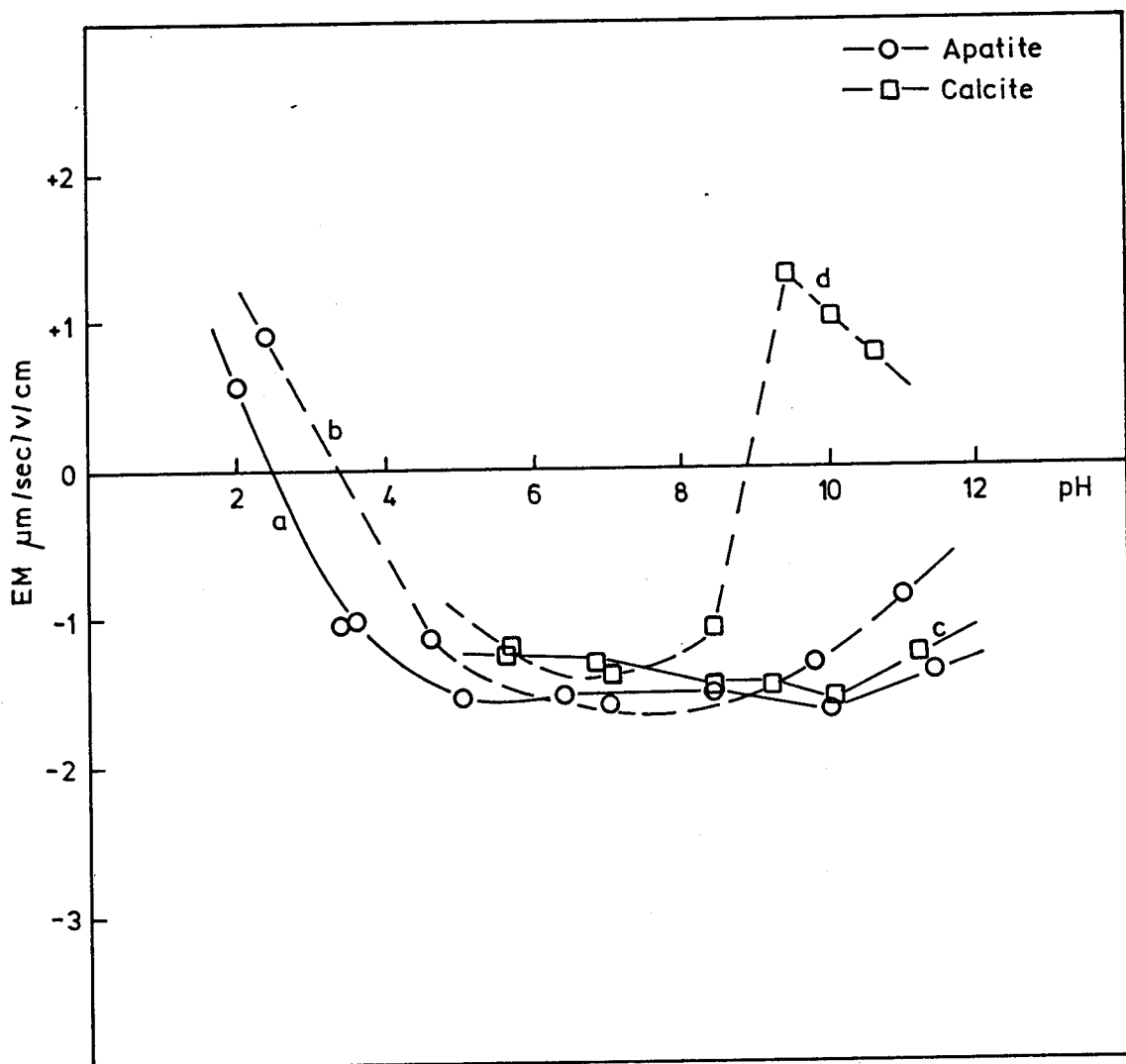


Fig. 16. Effect of pH on EM of apatite and calcite from Kaluwe in Kaluwe water. a,c= containing 2×10^{-4} M sodium silicate; b,d= Replot in 10^{-3} M NaCl.

throughout the pH range studied. Apatite showed an IEP of 2.4.

There was an increase in the negative charge of apatite throughout the pH range studied. The behaviour of both calcite and apatite may be attributed to the adsorption of silicate anions. This adsorption has been found to be chemical in nature (14). Negatively charged colloidal silica particles were probably responsible for charge reversal on calcite since adsorption occurred on positively charged calcite surfaces.

The results obtained indicate that sodium silicate cannot be used as a depressant for either of the minerals in Kaluwe water because they possess a similar charge in almost the entire pH range studied. Separation of the two minerals in such a system would be difficult to accomplish.

Warren Spring Laboratory (4) tried to use sodium silicate as a calcite depressant during the flotation of apatite from Kaluwe ore using a fatty acid collector at pH 10. The results showed that there was very little selectivity towards apatite. The reason for this may probably have been due to the fact that at this pH value apatite and calcite were/both negatively charged rendering their separation difficult.

2.6.1.3 Effect of orthophosphoric acid on the EM of apatite, calcite and magnetite in Kaluwe water

In industry orthophosphoric acid is used as either a depressant or activator for phosphate minerals in the flotation process. In order to find out the effect of this acid on the minerals being studied, surface charge measurements were carried out in Kaluwe water in the presence of $2 \times 10^{-4} \text{M}$ orthophosphoric acid (H_3PO_4). The results are shown in figure 17. It is clear from figure 18 that irrespective of the sign of the surface charge, the adsorption of the phosphate species at the apatite surface took place at all pH values studied. This behaviour suggests that the phosphate species are potential-determining ions for apatite. This is in agreement with other workers (11,12, 13, 15). Calcite showed a reduction in potential at pH values higher than 8.8 (fig. 19). This may have been due to the ionic competition between OH^- ions and calcium species. Magnetite (Fig. 20) showed a reduction in the surface charge at pH values less than 6 and thereafter there was an increase in the charge until pH 8.8 when it showed a charge reversal. The reason for this behaviour is not clear.

Figures 18, 19 and 20 illustrate the behaviour of the minerals apatite, calcite and magnetite respectively in Kaluwe water of 10^{-3}NaCl and $2 \times 10^{-4} \text{M H}_3\text{PO}_4$.

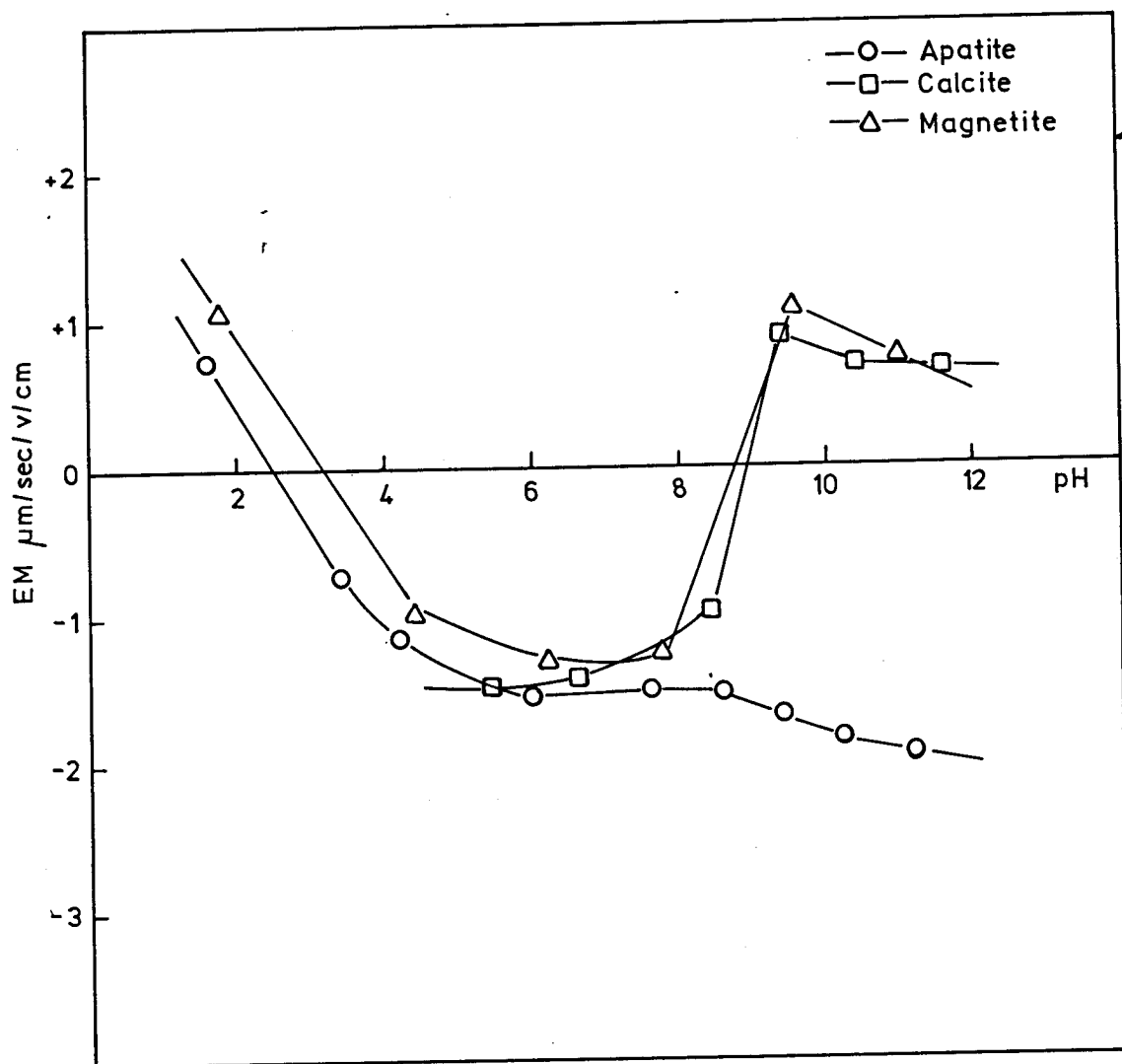


Fig. 17. Effect of pH on EM of apatite, calcite and magnetite from Kaluwe in Kaluwe water containing $2 \times 10^{-4} \text{ M H}_3\text{PO}_4$

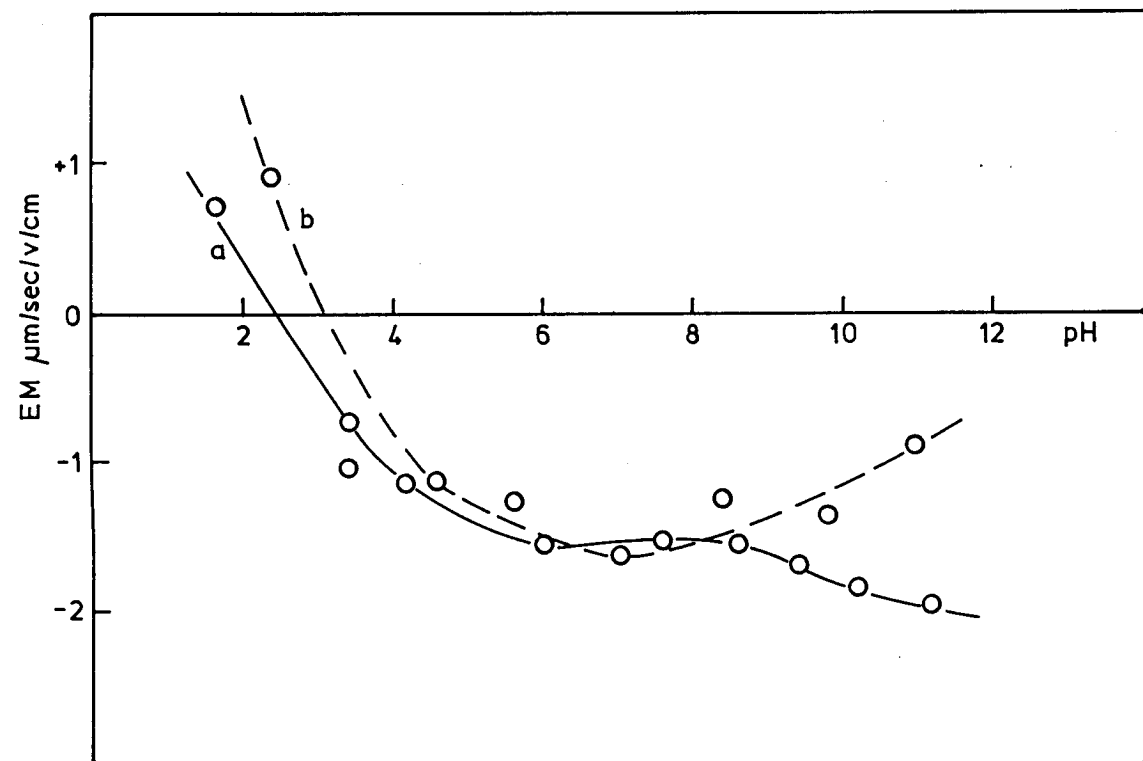


Fig. 18. Effect of pH on EM of apatite from Kaluwe in Kaluwe water a= containing $2 \times 10^{-4} \text{ M H}_3\text{PO}_4$; b=Replot in 10^{-3} M NaCl .

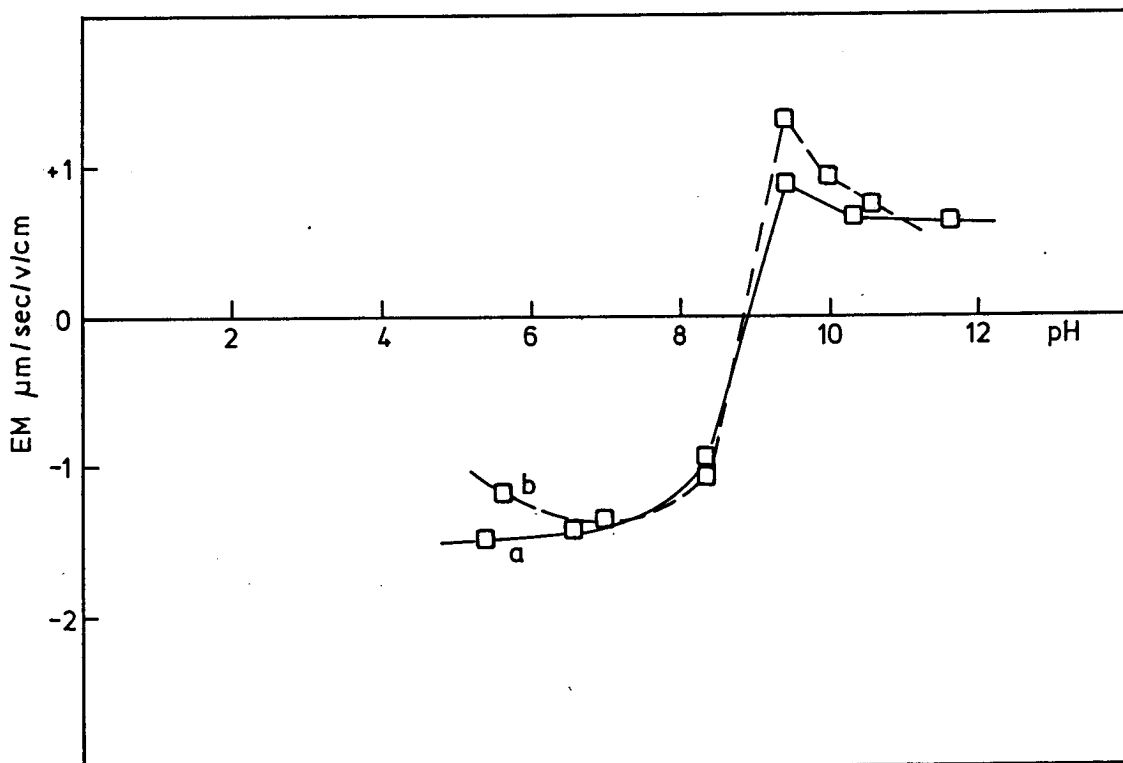


Fig. 19. Effect of pH on EM of calcite from Kaluwe in Kaluwe water. a= containing $2 \times 10^{-4} \text{ M H}_3\text{PO}_4$; b=Replot in 10^{-3} M NaCl .

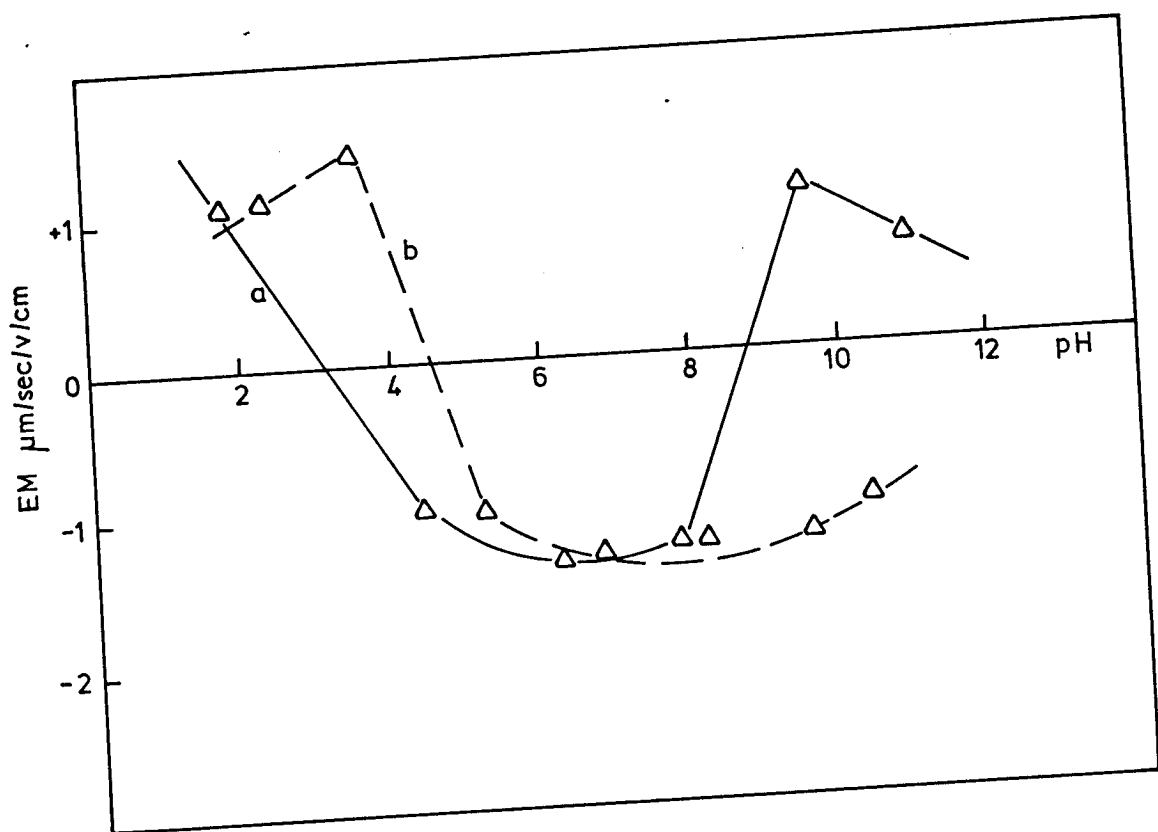


Fig. 20. Effect of pH on EM of magnetite from Kaluwe in Kaluwe water. a= containing $2 \times 10^{-4} \text{ M H}_3\text{PO}_4$; b= Replot in 10^{-3} M NaCl

Electrokinetic studies showed that it is possible to selectively float the apatite in Kaluwe water without applying any depressant. Depressants containing calcium as the active modifying species cannot be used as modifiers in the flotation process involving these minerals.

Furthermore, sodium silicate would also not be applicable in this system as a depressing reagent. Orthophosphoric acid can be applied as an activator for phosphate species.

CHAPTER THREE

PROCESS TESTING

3.1 Shaking table tests

Due to the promising results obtained using heavy medium separation in the last chapter, it was decided to test whether or not such results could be reproduced using devices employed in industry. In this regard, a shaking table was considered for the tests due to the reasons advanced earlier.

The principles of a shaking table can be described as follows: When a film of water flows over a flat, inclined surface, the water closest to the surface is retarded by the friction of the water adsorbed on the surface; the velocity increases towards the top of the film. If mineral particles are introduced into the film, particles of high specific gravity will move more slowly than lighter ones. The flowing film effectively separates light particles from dense particles. The table is vibrated longitudinally using a slow forward stroke, and a rapid return stroke, causing the mineral particles to crawl along the deck parallel to the direction of motion. The minerals are thus subjected to two forces, one due to the table motion and the other at right angles to it due to the flowing film of water. The net effect is that the particles move diagonally across the deck from the feed end. The smallest heavy particles, which resist traverse washing most effectively, ride high toward the concentrate launder at the far end while the larger

light particles are washed in the tailing launder.

The largest heavy particles, which are readily rolled by water, mixed with the fine light particles fall in between.

Shaking table tests were carried out as a function of size using the tabling scheme shown in figure 21.

The notation used is as follows:

F	-	Feed
FC	-	Concentrate from first stage
FT	-	Tailings from first stage
FCC	-	Concentrate, FC as feed
FCT	-	Tailings, FC as feed
FCCC	-	Concentrate, FCC as feed
FCCT	-	Tailings, FCC as feed
FCTC	-	Concentrate, FCT as feed
FCTT	-	Tailings, FCT as feed
FTC	-	Concentrate, FT as feed
FTT	-	Tailings, FT as feed
FTCC	-	Concentrate, FTC as feed
FTCT	-	Tailings, FTC as feed
FTTC	-	Concentrate, FTT as feed
FTTT	-	Tailings, FTT as feed
MFCCC	-	Non-magnetic product from magnetic separation on FCCC

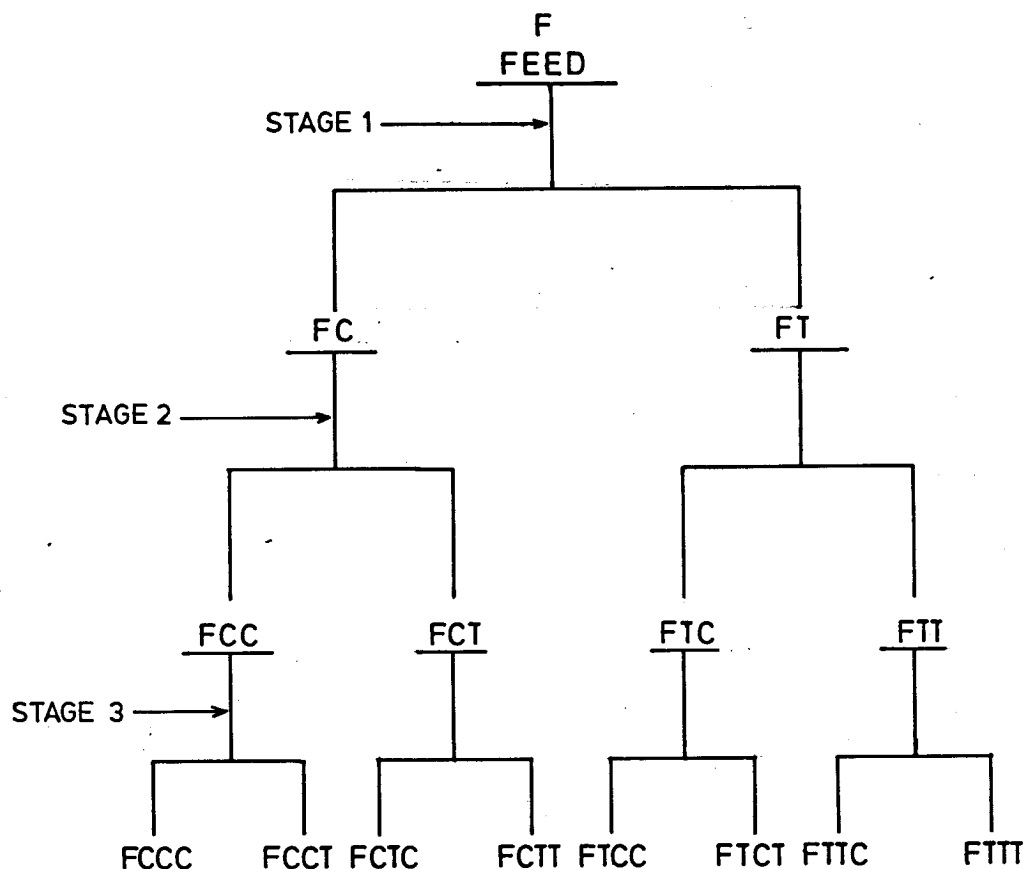


Fig. 21. Diagram showing tabling scheme followed.

As can be seen from the diagram, the concentrate from stage 1 was recycled twice in order to see the extent to which upgrading would occur. The tailings from the first stage was also subjected to the same treatment in order to reduce its P_2O_5 content. The concentrate from the last stage was subjected to magnetic separation in order to remove the iron minerals which would in turn upgrade the concentrate.

The results of these experiments are shown in table 13.

The table shows that there was an increase in the grade of the first stage concentrate (FC row) compared to the feed grade.

Table 13. Effect of particle size on P_2O_5 grade of various tabling product

	% P_2O_5 in size fraction (μm)						
	+600	+425	+300	+212	+150	+106	+75
Feed	1.9	2.1	3.0	2.9	3.0	2.7	1.7
FC	3.0	3.2	4.0	7.0	7.0	8.5	7.0
FT	1.7	1.5	1.1	1.0	0.9	1.1	1.2
FCC	3.0	3.8	7.0	10.0	9.0	10.0	8.6
FCT	2.4	2.1	2.6	4.0	3.1	4.3	3.5
FTC	2.0	2.3	2.1	3.3	4.0	3.0	3.5
FTT	1.6	1.4	1.4	1.3	1.0	1.2	1.0
FCCC	3.4	4.7	8.6	15.0	11.2	10.9	9.2
MECCC	3.3	5.0	10.0	20.0	18.0	13.0	10.0
FCCT	2.4	3.0	4.5	3.2	4.1	3.4	4.0
FTTC	2.5	2.6	3.8	8.0	6.0	6.4	7.0
FCFT	1.6	2.3	2.1	2.8	2.4	2.3	2.0
FTCC	2.4	2.7	4.0	6.0	7.0	2.5	6.0
FTCT	2.7	1.8	1.7	2.5	2.7	2.6	2.4
FTTC	2.4	2.4	1.4	2.2	2.3	3.5	4.2
FTTT	1.0	1.1	0.9	0.8	0.8	1.0	1.1

The increase in grade was prominent in the size range of high liberation ($-425 + 75\mu\text{m}$), the overall average of which was 5% P_2O_5 (table 14). As the concentrate was subjected to further tabling, there was a general increase in grade (rows FCC and FCCC) but the recovery decreased (stage recoveries).

There was an improvement in the grade after magnetic separation with an overall average being 9% P_2O_5 . As can be seen from table 14 the average recovery for the first stage was 61.7% at a grade of 5% P_2O_5 . The second and third stages yielded recoveries of 48.1% and 52.4% with grades of 6.8% P_2O_5 and 7.7 % P_2O_5 respectively.

The lower than expected grades and recoveries were probably due to the following reasons:

- (a) The low grade nature of the ore which resulted in producing a lot of tailings.
- (b) The low concentration criterion of 1.29 which made the separation inefficient.

It was not possible to fully optimise the operating conditions of the shaking table as the unit used had no facilities for measuring the length of the stroke and the slope of the table. However, the slope was visually optimised by trial and error. Nevertheless, the results obtained prove that it is possible to concentrate the ore using tabling but, as has been pointed out earlier, this can only be used as a preconcentration unit before a process such as flotation.

Table 14. Effect of particle size on P_2O_5 grade and recovery of concentrates from various tabling stages

Size Fraction μm	FC		FCC		FCCC		Grade after magnetic separation on (MFCCC)
	P_2O_5 Grade %	Recovery %	P_2O_5 Grade %	Recovery %	P_2O_5 Grade %	Recovery %	
+600	3.0	64.8	3.0	56.5	3.4	43.5	3.3
+425	3.2	64.3	3.8	51.2	4.7	49.0	5.0
+300	4.0	84.2	7.0	52.3	8.6	52.5	10.0
+212	7.0	87.6	10.0	51.4	15.0	61.3	20.0
+150	7.0	88.1	9.0	51.6	11.2	61.6	18.0
+106	8.5	49.5	10.0	46.0	10.9	45.7	13.0
+ 75	7.0	48.2	8.6	52.2	9.2	33.9	10.0
Average Grade/Recovery	5.0	61.7	6.8	48.1	7.7	52.4	9.0

3.2 Magnetic concentration tests

A large variety of minerals may be separated from one another on the basis of their magnetic susceptibilities. As such, it is essential to know which minerals the ore contains and to ascertain from a chart like figure 22 if any of these mineral components have a distinctive magnetic susceptibility that will enable them to be separated from the others. Magnetic separation utilizes a magnetic force field to produce differential responses of mineral grains passing through the field.

The efficiency of magnetic separation depends on many variables, such as particle size, density, shape, magnetic permeability, and the intensity of the magnetic field. Magnetic separation may use either low or high intensity magnets. The equipment may be used in either dry or wet processes.

Figure 23 shows the current settings for the observed minerals in Kaluwe phosphate ore used in the magnetic separation tests.

The mineral data are for 25° forward slope and 15° side tilt on a Frantz Isodynamic Magnetic Separator (3).

Initially the samples were deslimed at 20 μ m by beaker decantation in order to remove dust coatings on the grains. This step was important in order to avoid problems presented by slimes during separation like "balling up" of grains. After drying in an oven at about 80°C the magnetic components of the grains were removed using a

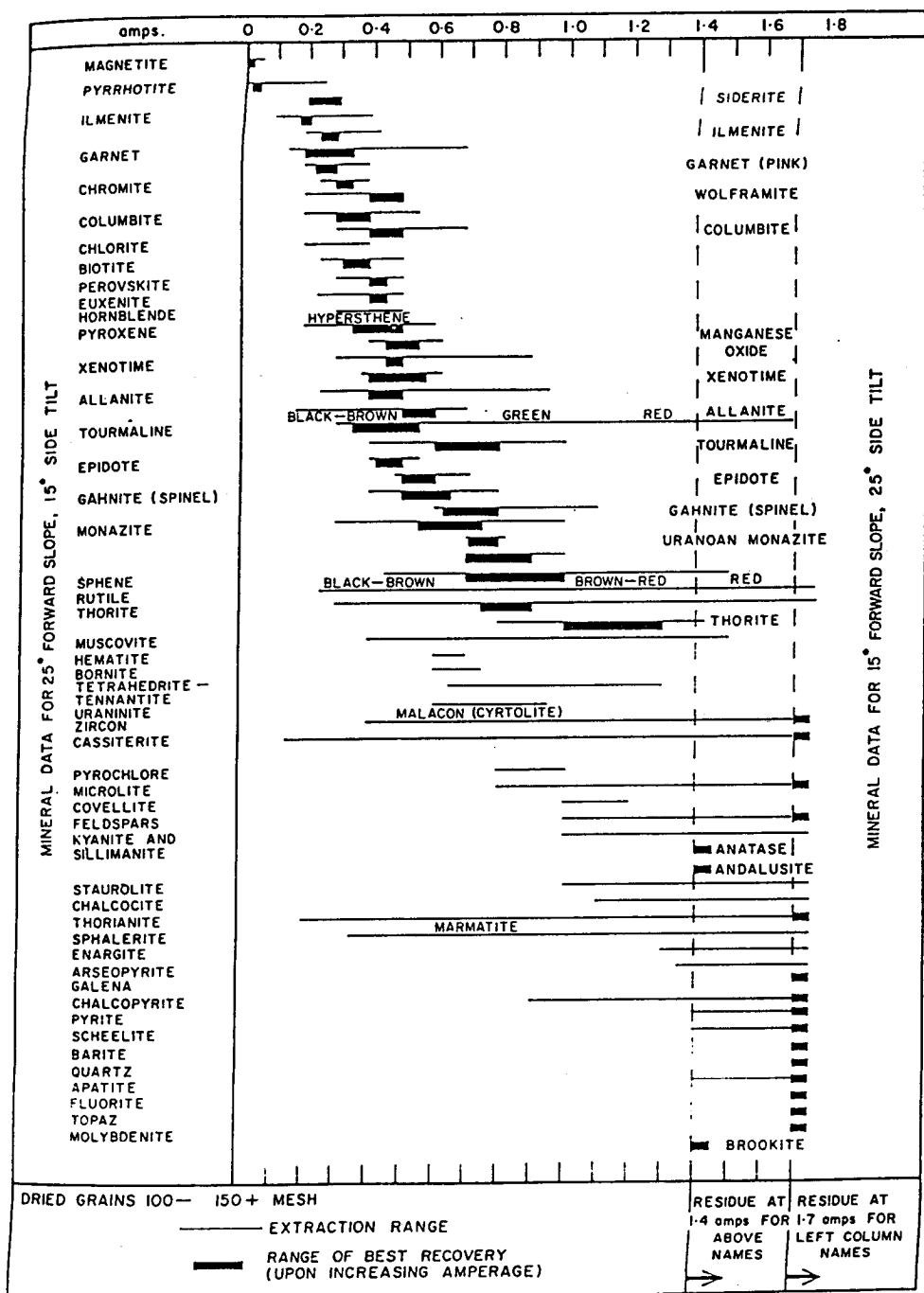


Fig. 22. Current settings for magnetic separation of common minerals. Data from Rosenblum (1958) for the Frantz Isodynamic Magnetic Separator. Thick lines indicate the most frequent range; thin lines indicate the full range that may occasionally be employed (3).

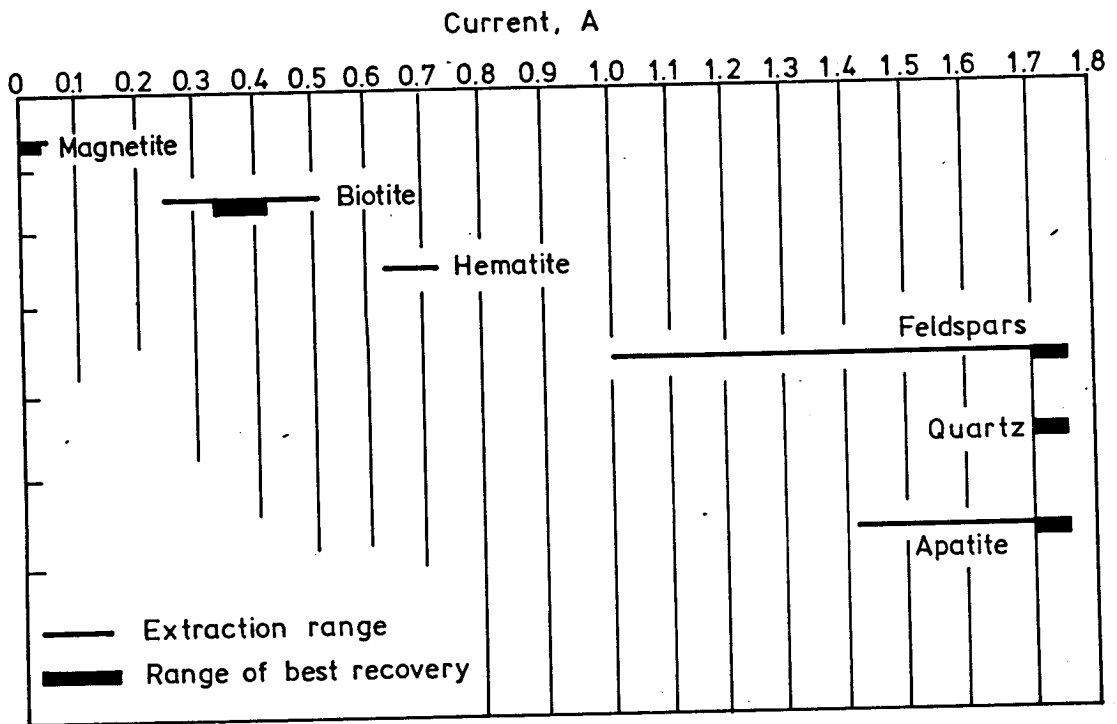


Fig. 23. Current settings for observed minerals in Kaluwe phosphate ore (25° forward slope and 15° side tilt) used in magnetic separation.

weakly magnetized hand magnet. This was done by spreading the grains on a card and passing the magnet over in order to pull out the strongly magnetic ones. These were magnetite belonging to the ore and probably particles of metallic iron that were introduced by the crushing operations. The grains were now ready for separation in a Frantz Isodynamic Magnetic Separator model similar to the one shown in figure 24. It consisted of an inclined vibrating plane, which was set up according to the settings of forward slope and side tilt. The forward slope was adjusted on a goniometer scale set at the back of the instrument and

the side tilt was selected by rotating the black metal wheel on the right. A small aluminium filter funnel was screwed fully clockwise into the top end of the inclined plane in which dry mineral powder was fed.

The principle of operation is that the magnetic field is directed up-slope and pulls the more magnetic particles up the inclined vibrating plane toward the front.

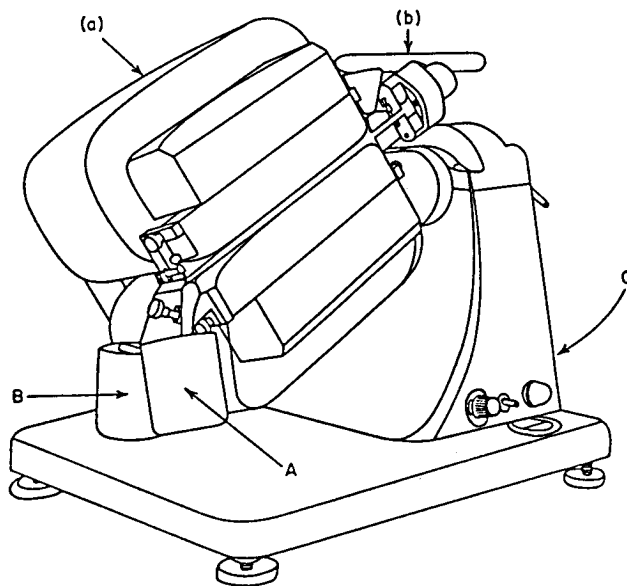


Fig. 24. The Frantz Isodynamic Magnetic Separator, Model L1. The important controls are (a) forward slope, set on a scale at the back; (b) side tilt, set by means of the wheel on the right; and (c) the electric current in amperes, set by an adjustable potentiometer and read on an ammeter. This controls the strength of the magnetic field. Final separation is in two cups: A-more magnetic and B- less magnetic.

Toward the lower end of the inclined plane a longitudinal divider separates the more magnetic grains, which collect in the front cup, from the less magnetic grains, which collect in the cup lower down slope. The amperage on the electromagnet is set at ^a value that would make a clear distinction between one mineral in the powder and the others. Amperage is selected first at low values and minerals are progressively separated from the mixture in the order of highly magnetic.

Although magnetite was removed from the grains using a weak magnet, the samples were run through the isodynamic separator at 0.1 A with a forward slope of 25° and side tilt of 15° . This initial run was necessary to remove all the magnetite. A first run at a higher amperage would otherwise cause clogging of the magnet poles by strongly magnetic grains. The samples were then subjected to the following amperages in the order shown to separate the observed iron minerals:

- (a) At 0.1A to separate any magnetite left.
- (b) At 0.5 A to separate biotite
- (c) At 0.8A to separate hematite
- (d) At 1.4A to remove any of the minerals above and some feldspar.

The apatite was left in the non-magnetic fraction in order to find out whether there would be an improvement in the P_2O_5 grade after magnetic separation. With this regard, an amperage of 1.4A was not exceeded.

Between each run the inclined plane was detached and cleaned with a soft-haired brush, together with the space between the poles of the magnet.

The results of magnetic separation tests are presented in table 15 for magnetic fractions and table 16 for non-magnetic fractions. The $+2000\mu\text{m}$ fraction was not included in the tests due to limitations imposed by the feed aperture. It was difficult to separate the $-106\mu\text{m}$ fractions due to the electrostatic effect of the fines causing the "balling up" effect. The magnetic fractions at different currents were combined and the grade and recovery found for the combined fractions.

Figure 25 was drawn from data contained in table 16. The main feature from the figure was that there was maximum recovery in the $-425 + 150\mu\text{m}$ size range. This was because in this size range the liberation of apatite was high as observed earlier. The $+150\mu\text{m}$ represents the lower limit of efficient treatment for most commonly used commercial machines.

Table 15 shows that the magnetic coarse fractions above $425\mu\text{m}$ had high amounts of P_2O_5 . This could be attributed to the composite nature of the fraction due to incomplete liberation of the major minerals in the ore (calcite, apatite and iron minerals). The increase in the P_2O_5 grade in the non-magnetic fraction was marginal as can be seen from table 16. This means that magnetic separation cannot be carried out alone to produce a final concentrate. It has to be carried out in combination with other processes although acceptable recoveries were obtained in almost all the size fractions considered.

Table 15. Effect of particle size on the recovery and grade of P_2O_5 in magnetic fractions at 1.4 A

Size Fraction μm	Combined Grade % P_2O_5	Combined Recovery %
+1400	3.8	59.4
+ 850	2.3	51.1
+ 600	1.6	28.5
+ 425	2.2	25.5
+ 300	1.8	12.2
+ 212	1.8	10.3
+ 150	1.6	8.0
+ 106	1.5	5.5
Average	1.7	20.9

Table 16. Effect of particle size on the recovery and grade of P_2O_5 in the non-magnetic fraction at 1.4 A

Size Fraction μm	Grade % P_2O_5	Recovery %	Enrichment ratio
+1400	1.5	49.6	0.71
+ 850	1.4	51.2	0.67
+ 600	2.3	80.1	1.10
+ 425	2.2	79.3	1.05
+ 300	2.4	77.9	1.14
+ 212	3.6	72.2	1.71
+ 150	5.3	73.4	2.52
+ 106	3.6	60.7	1.71
Average	2.7	79.1	1.29

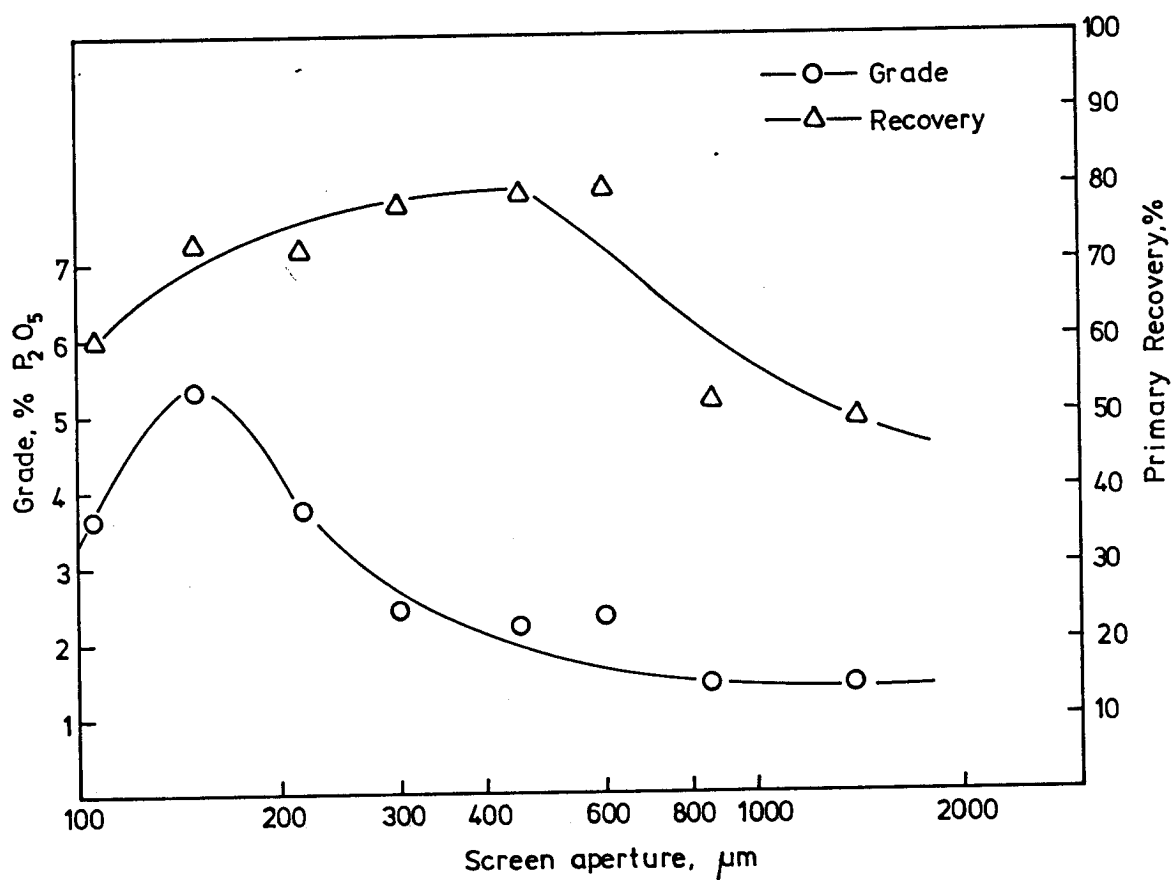


Fig. 25. Effect of particle size on the grade and recovery of phosphate in the primary magnetic separation product (non-magnetic)

Mineralogical studies were carried out on the magnetic and non-magnetic fractions to determine the proportions of minerals in each size fraction. Tables 17 and 18 show the results obtained of the magnetic and non-magnetic fractions respectively. The misplacement of apatite in the coarser fractions of the magnetic fractions (table 17) was due to incomplete liberation of apatite whereas at finer sizes it was due to electrostatic effect of the fine apatite grains which reported in the magnetic fractions.

Table 18 is in agreement with table 16 in that there was an increase in the apatite proportion with decrease in size. The magnetic fractions consisted of an appreciable amount of calcite at coarse fractions. This was due to locking of grains. At fine fractions the calcite present was stained as revealed by visual examination in a binocular microscope. Previous work has shown that this is limonitic staining (4). XRD patterns for some magnetic fractions from a $150\mu\text{m}$ sample at current settings of 0.1, 0.5 and 1 amp are shown in the appendices. The opaques proportion increased as the size decreased. This was due to the high degree of liberation at fine sizes.

The non-magnetic fraction consisted of mostly calcite (more than 80%).

It can be concluded from the results obtained that high intensity magnetic separation can profitably be carried out in the $-425 + 150\mu\text{m}$ size range. However, it must be emphasised that such a separation would only serve to upgrade the ore for subsequent processes and not to produce a final

Table 17. Effect of particle size on the proportions of minerals in the combined magnetic fractions at currents up to 1.4 A

Size Fraction μm	Apatite	Calcite	Opakes	Quartz/Felds- par	Biotite	Total
	Proportion %	Proportion %	Proportion %	Proportion %	Proportion %	
+ 1400	12.2	71.1	15.8	-	0.9	100
+ 850	9.4	69.8	19.5	0.6	0.7	100
+ 600	7.4	71.5	20.5	-	0.6	100
+ 425	7.9	58.8	32.7	0.3	0.3	100
+ 300	5.8	48.2	46.0	-	-	100
+ 212	6.0	42.0	51.5	0.1	0.4	100
+ 150	3.8	43.4	52.6	0.1	0.1	100
+ 106	5.2	23.7	70.4	0.1	0.6	100
Average	8.4	62.2	28.6	0.2	0.6	100

Table 18. Effect of particle size on the mineral proportions and liberation of apatite in the non-magnetic fraction at 1.4 A.

Size Fraction μm	Apatite		Calcite	Opakes	Quartz/ Feldspar	Biotite	Total
	Proportion %	Estimated Liberation %	Proportion %	Proportion %	Proportion %	Proportion %	
+ 1400	10.2	17	83.5	4.9	1.1	0.3	100
+ 850	7.7	26	82.5	8.3	1.0	0.5	100
+ 600	9.6	41	83.9	5.8	0.7	-	100
+ 425	9.8	56	84.8	4.7	0.7	-	100
+ 300	10.8	79	82.1	6.2	0.9	-	100
+ 212	12.1	88	80.8	6.8	0.3	-	100
+ 150	12.7	97	79.7	7.4	0.2	-	100
+ 106	11.8	98	86.8	1.4	-	-	100
Average	10.5		83.2	5.6	0.6	0.1	100

concentrate.

The low average recovery (79.1%) obtained may be due to losses brought about by treatment of large amounts of material. In this regard, magnetic separation could be carried out as a last stage in the beneficiation process to minimise losses because smaller amounts of material would then be used.

3.3. Flotation tests

3.3.1 Preparation of the flotation feed

Based on the liberation study, the crushed ore was dry ground in a rod mill to the liberation size (less than $425\mu\text{m}$) using a stage-grinding technique to minimise the amount of slimes produced. The ground product was deslimed by beaker decantation in batches of 500g each in order to remove the minus $20\mu\text{m}$ as slime waste. The $-425 + 20\mu\text{m}$ (the flotation feed) was then riffled into batches of 250 g each for the flotation tests. The size distribution and chemical analysis of the prepared flotation feed is shown in table 4 of the appendices.

From electrokinetic studies, it was established that there was a marked difference in the surface charges of apatite and magnetite (negative) from that of calcite (positive) in the pH range 8 to 12. This pH range formed the basis for subsequent flotation experiments. All flotation tests were carried out using a Denver flotation machine at an impeller speed of 1200 r.p.m. and at a solids concentration of 20 percent. Various flotation routes were evaluated for the beneficiation of Kaluwe phosphate ore. The general description of the methods tested in this study and the results obtained are outlined in the following paragraphs.

3.3.2 Phosphate Flotation Using a Cationic Collector

An amine(cocoamine-Armeen c from Akzo Chemie, Netherlands) was used in this study since its mineral attachment is mainly due to electrostatic attraction. Flotation tests were first carried out as a function of pH (7 to 12) at a constant collector concentration and then as a function of collector concentration at an optimised pH value. This was done in order to optimise both the pH and collector concentration before considering preconcentration of the flotation feed. Preliminary flotation tests revealed that the first flotation product consisted mainly of sheet silicates (micas). This could be attributed to the high adsorption capacity of the micas for amines which would therefore, tend to float first in the presence of an amine (16). In view of this, it was decided to have a two-stage flotation process in which the first stage would be for flotation of micas (with some phosphates) while the second stage, designated Midlings in this study, would be for phosphate flotation (concentrate). Small dosages of collector were used in the first stage relative to the second.

3.3.2.1. Effect of pH on phosphate grade and recovery

Flotation experiments were carried out as a function of pH at a constant cocoamine concentration. The following flotation conditions were used:

Stage 1

Collector concentration	200g/t
Frother (Dow 250)	0.02 kg/t
Condition for	3 minutes
Float for	2 minutes

The concentrate consisted mainly of sheet silicates as revealed by a binocular microscope.

Stage 2 (MIDLINGS)

Collector concentration	300 g/t
Frother (Dow 250)	0.02 kg/t
Condition for	2 minutes
Float for	3 minutes

The effect of pH on the grade and recovery of Kaluwe ore at a constant cocoamine concentration of 300 g/tonne is presented in figure 26. The recovery showed a peak at pH 10 and a minimum at pH 12. The location of the peak at this pH value was considered to be due to the large difference in surface charges of apatite and calcite, thus allowing apatite to be selectively floated by the coulombic mechanism. The main information from the figure is that a maximum recovery of about 39% could be reached at pH 10. The grade at this pH was 11% P_2O_5 . This low grade obtained can be attributed to the fact that in this pH range the iron oxides were also floated together with the phosphates, so part of the collector

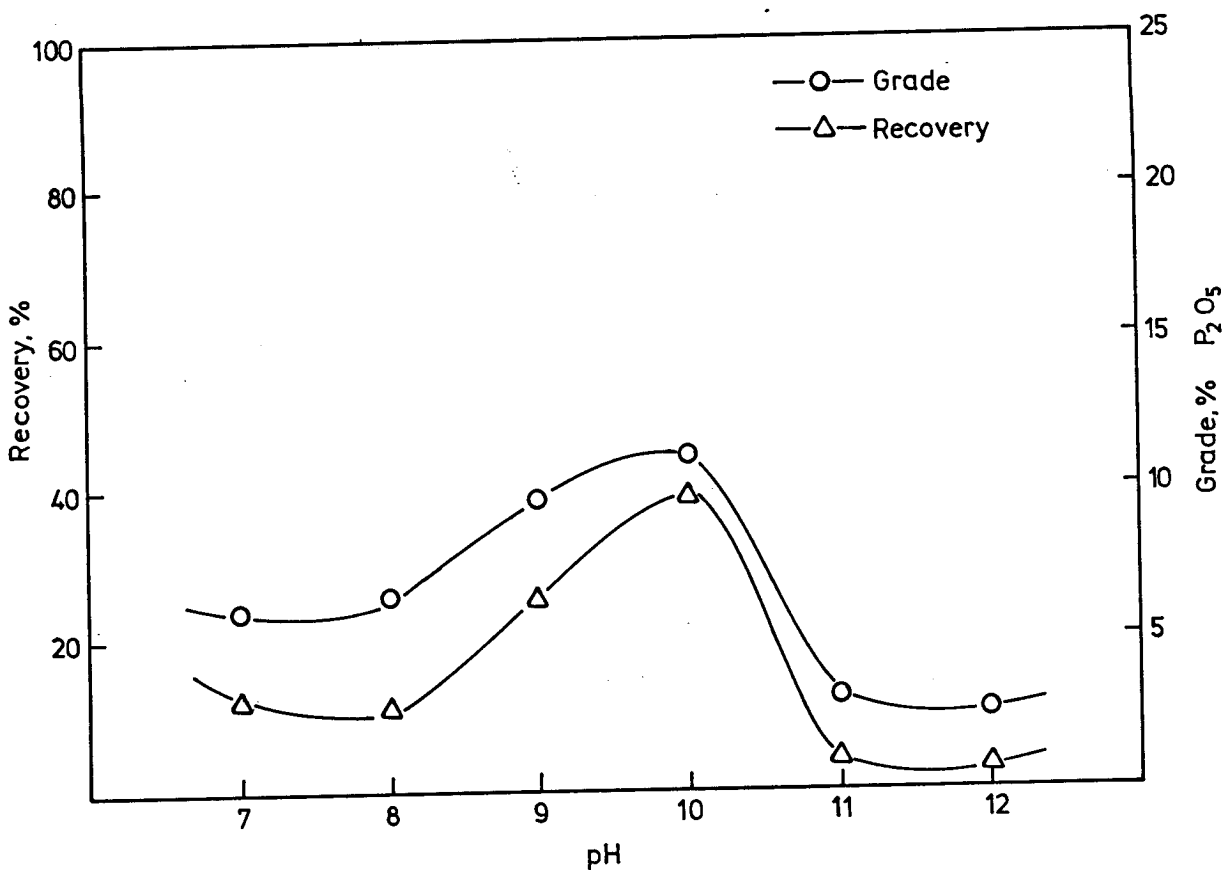


Fig. 26. Effect of pH on phosphate grade and recovery on Kaluwe ore at 300g/t cocoamine.

was used in floating these minerals thereby reducing the grade and recovery.

3.3.2.2 Effect of collector concentration on grade and recovery

The effect of collector concentration on the flotation characteristics of the ore was investigated in order to optimise the addition rates of the collector. In the experiments, the pH was held constant at the optimised level (pH 10) established in the preceeding section. The flotation conditions were similar to those outlined in the last section in which the first stage flotation was for the removal of sheet silicates at 200 g/t collector. The effect of collector concentration was studied in the second stage in which the collector concentration was varied. The concentration range investigated was 500 to 1200 g/t. The results of these investigations are shown in figure 27. As can be seen from the figure there are two peaks on the recovery curve, one at 58% corresponding to a collector concentration of 1000 g/t and a lower one at 40% corresponding to a collector concentration of 700 g/t. Since the difference in grade at the two peaks was not marked, it was decided to take 700 g/t as the optimum concentration although it gave a low recovery. The aim was to work at lower reagent dosages. The concentrate grade at this concentration was 11.5% P_2O_5 with the tailings at 1.4 % P_2O_5 .

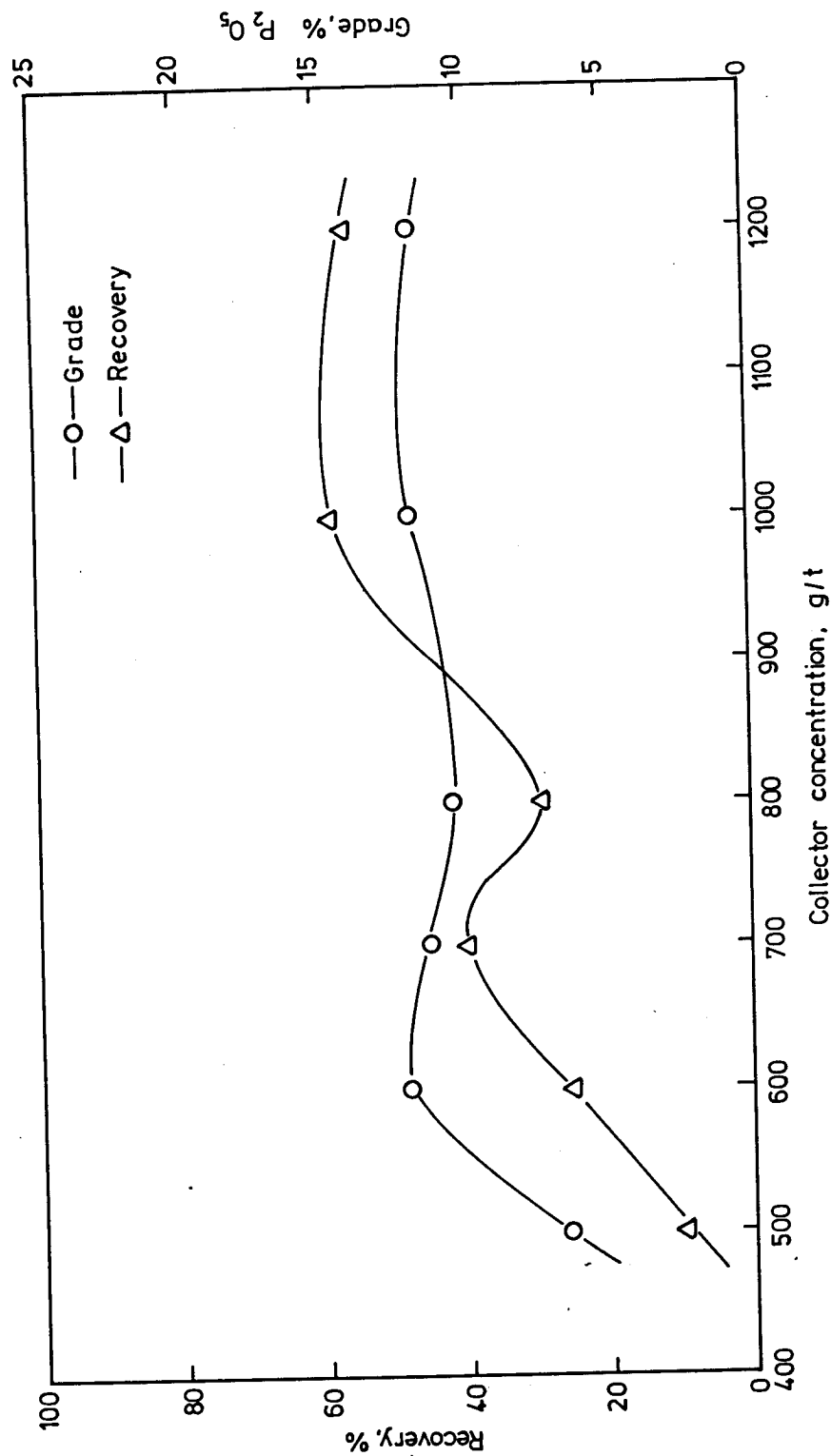


Fig. 27. Effect of collector concentration on grade and recovery on Kaluwe ore at pH 10 (midlings)

The main conclusion from the above flotation tests is that it is possible to concentrate Kaluwe ore using a cocoamine. The low grades and recoveries obtained can be attributed to the low grade nature of the ore. A preconcentrated flotation feed could give positive results. In view of this, it was decided to preconcentrate the ore before flotation.

3.3.3 Flotation of preconcentrated product

Two preconcentration routes were used. The $-425 + 75\mu\text{m}$ size fraction was subjected to the shaking table while the $-75 + 20\mu\text{m}$ fraction was bulk floated. Applying flotation as a preconcentration stage for the $-75 + 20\mu\text{m}$ fraction was necessary as the shaking table proved to be rather inefficient for this size fraction. The concentrates from the two routes assayed both at about 6% P_2O_5 . These were combined and subjected to flotation. The flotation conditions were as described earlier for the first stage except that a pH of 10 was used. The second stage flotation was conducted at pH 10 with 700 g/t cocoamine collector concentration. This was done in duplicate and the results are shown in tables 19, 20 and 21. A phosphate concentrate of about 27.6 % P_2O_5 , 19.1 % CaO , 3.5 % MgO , 2.2 % SiO_2 , 5.8 % Fe_2O_3 and 0.4 % Al_2O_3 was produced at a recovery of 62.6%. After subjecting the concentrate to magnetic separation, the grade increased to about 29.5 % P_2O_5 and the Fe_2O_3 content reduced to 3.7 %. The change

FLOTATION OF PRECONCENTRATED PRODUCT

Table 19. Final Concentrate

	Assay, %					
	P ₂ O ₅	CaO	MgO	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃
Concentrate I	28.0	20.4	2.8	2.3	5.8	0.3
Concentrate II	27.0	17.4	4.5	2.1	5.7	0.5
Average	27.6	19.1	3.5	2.2	5.8	0.4

Table 20. AFTER MAGNETIC SEPARATION

	Assay, %					
	P ₂ O ₅	CaO	MgO	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃
Concentrate I	29.0	25.2	2.2	2.0	4.3	0.6
Concentrate II	30.0	21.9	4.1	2.0	3.2	0.3
Average	29.5	23.5	3.1	2.0	3.7	0.4

Table 21. FINAL TAILINGS

	Assay, %					
	P ₂ O ₅	CaO	MgO	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃
Tailings I	1.1	44.6	4.2	2.8	5.7	0.2
Tailings II	0.9	39.3	4.8	3.1	6.5	0.2
Average	1.0	41.9	4.5	3.0	6.1	0.2

in the phosphate recovery was marginal. The grade of the final tailings was 1.0 % P_2O_5 . Figure 30 shows a proposed flowsheet for the beneficiation of Kaluwe ore while figure 31 gives a material balance on part of the flowsheet.

A fresh ore (not weathered) would probably behave in a similar manner, if not better, in that staining of the apatite and calcite would not be a problem. This would improve collector-mineral attachment resulting in good concentrate grades and recoveries.

3.3.4 Anionic (reverse) Flotation Process

In this process the carbonate was floated using a sulphonated fatty acid (petroleum sulphonate). No reagent was used to depress the phosphate mineral. The sulphonated fatty acids are preferred to normal fatty acids because they are self-frothing, have strong collecting properties for the alkaline earth metal carbonates, have a strong carbon-sulphur linkage that makes them ~~more~~ stable to pH fluctuations and water hardness has little effect on their wetting properties.

The concentrate from these tests was the flotation cell underflow and the tailings were the overflow.

3.3.4.1 Effect of pH on phosphate grade and recovery

Flotation tests were carried out as a function of pH at a constant petroleum sulphonate concentration of 900 g/t. The pH range chosen, from electrokinetic

results, was 7 to 11.

Figure 28 shows the results obtained from these tests. The recovery showed a maximum of 46.7% at pH 8. The phosphate grade at pH 8 was 3.5 % P_2O_5 . The low phosphate grade obtained can be attributed to the large amount of carbonates which had to be floated, but results showed that a substantial amount remained unfloated. Some of the phosphate was floated together with the carbonates as can be seen from the grades of the tailings in table 7 of the appendices.

3.3.4.2 Effect of petroleum sulphonate concentration on phosphate grade and recovery

Flotation tests were carried ^{out} at a constant pH (pH=8) as a function of collector concentration. Due to the fact that the carbonate to be floated was abundant, collector dosages of less than 900 g/t were not tried. The collector concentration range tried was from 900 - 1400 g/t. Figure 29 shows the results obtained. It is clear from the figure that both the recovery and grade showed their peaks at a collector concentration of 1200 g/t. The maximum recovery and grade obtained was 67.1% and 4.8 % P_2O_5 respectively. Again the tailings contained a relatively high amount of phosphates (table 8 in appendices). Due to the poor results obtained from the reverse flotation experiments, the tests were not extended to the flotation of the preconcentrated feed.

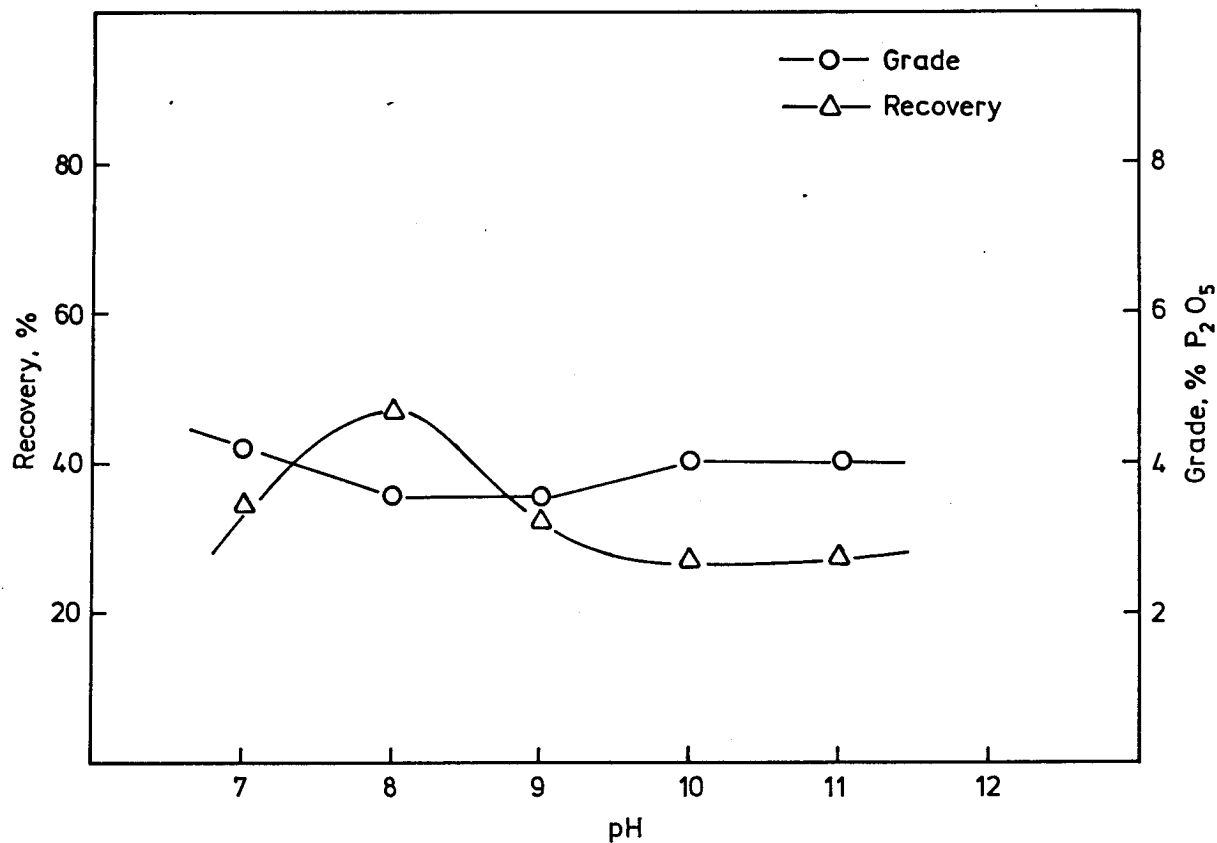


Fig. 28. Effect of pH on phosphate grade and recovery in reverse anionic flotation of calcite on Kaluwe ore at 900g/t petroleum sulphonate concentration.

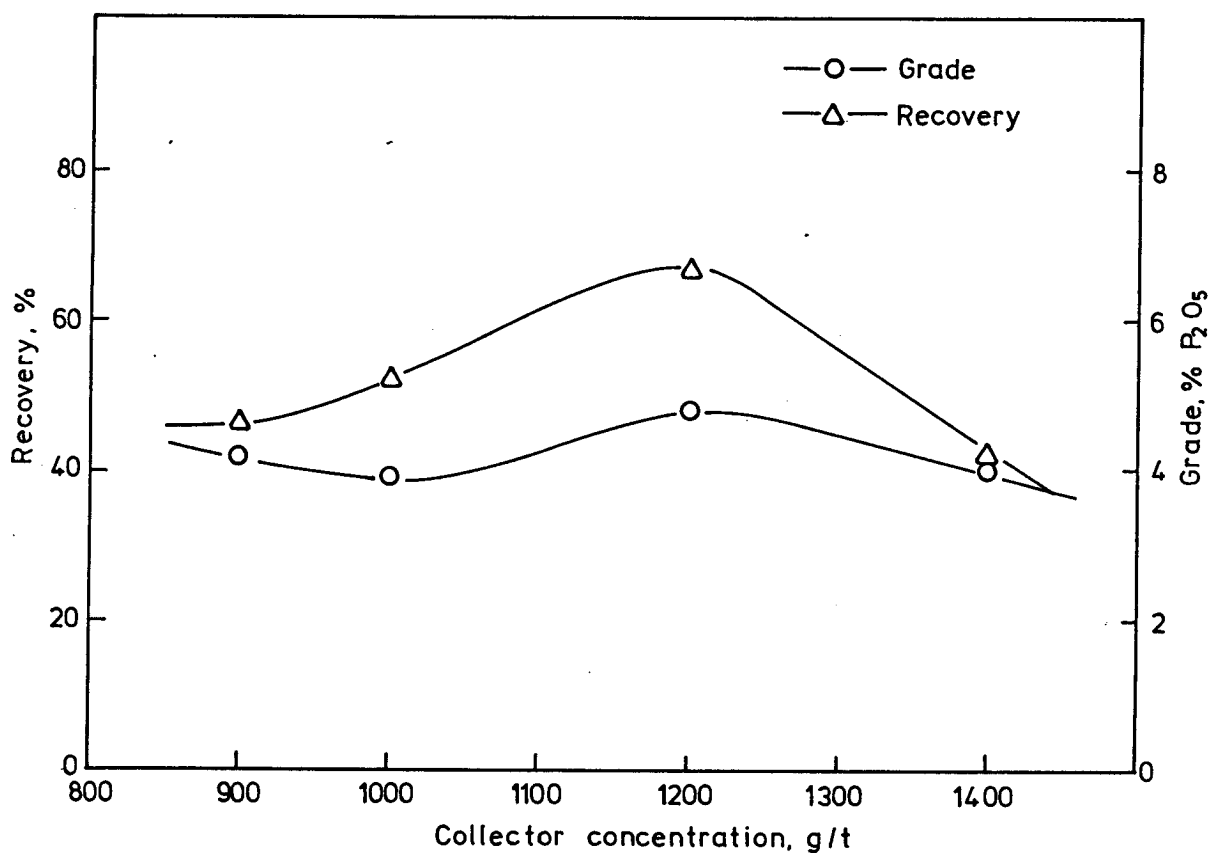


Fig. 29. Effect of collector concentration on phosphate grade and recovery for Kaluwe ore in reverse anionic flotation of calcite at pH 8.

The low grades obtained can be attributed to the low grade nature of the ore which means that a large amount of carbonate has to be floated. This is not feasible in a flotation process because usually the mineral floated does not constitute the major mineral of the ore. Flotation of carbonates using this process would require high reagent consumption and long flotation times in order to improve the phosphate grade.

3.3.5 Orthophosphoric Acid Process

Based on the electrokinetic results, orthophosphoric acid was used to enhance the negative charge on the apatite before flotation with cocoamine.

Flotation tests were done on a preconcentrated feed using the same procedure as in section 3.3.3 except that in this case orthophosphoric acid was added to the pulp before the second stage of flotation. The dosages of orthophosphoric acid tried were 7 and 9 kg/t respectively which are within the dosage range of some industrial plants (6.0 - 10.0 kg/t). The aim of these experiments was to see if there would be an improvement in the grade of the concentrate using orthophosphoric acid as a phosphate modifier. The results obtained from these tests showed an improvement in the P_2O_5 grade but the recovery decreased. The 7 and 9 kg/t acid dosages gave grades of 29.6 % P_2O_5 and 31.8% P_2O_5 and recoveries of 49.5 % and 46.2 % respectively. Although there was an increase in grade after application of orthophosphoric acid, the recovery

decreased. A decrease in recovery occurred because there was a decrease in the amount of concentrate produced due to improved selectivity. Table 22 shows results obtained from these tests.

Table 22. Results from orthophosphoric acid process (concentrates)

Dosage kg/t H_3PO_4	Assay, %					
	P_2O_5	CaO	MgO	SiO_2	Fe_2O_3	Al_2O_3
7	29.6	19.4	1.8	1.4	2.1	0.2
9	31.8	16.1	2.1	1.9	2.9	0.3
Average	29.1	17.9	1.9	1.6	2.5	0.2

3.4 Mineralogical examination on various products

A mineralogical examination was done on the following products from the treatment process (see figure 30):

- (1) Tailings from the shaking table
- (2) Tailings from the primary fines flotation
- (3) Final concentrate (before magnetic separation)
- (4) Final Tailings (before magnetic separation)
- (5) Magnetic concentration product (non-magnetic)
- (6) Final concentrate using H_3PO_4 as modifier.

A description of the observations is presented below:

- (1) Tailings from shaking table:

Very little but coarse-grained apatite was observed

averaging $400\mu\text{m}$. In some cases apatite was present as inclusions in calcite. Calcite was also coarse-grained. A shaking table feed of less than $400\mu\text{m}$ would reduce losses of coarse-grained apatite. Mostly coarse-grained opaques were present.

(2) Tailings from primary fines flotation:

Apatite was rare and was fine-grained $<60\mu\text{m}$.

Opaques were also rare. The tailings contained mostly calcite.

(3) Final concentrate (before magnetic separation):

Apatite occurred as liberated grains with the grain size ranging from 60 to $360\mu\text{m}$. The average grain size was $250\mu\text{m}$. Fine-grained stained calcite was also present. Opaque minerals were fine grained.

(4) Final tailings (before magnetic separation):

Apatite was mainly present as stained coarse grains. The average grain size of apatite was $344\mu\text{m}$. In some cases apatite occurred as composite grains with either calcite or opaques. Calcite and opaques also occurred as coarse grains. Scrubbing could clean the apatite surface and make it amenable to flotation but, as was observed in section 2.5, it is not possible to scrub this ore due to its friable nature which would lead to production of more fines.

(5) Magnetic concentration product (non-magnetic):

There was an increase in the apatite abundance compared to that prior to this process. Opaques were rare.

Calcite was present as fine grains. As was observed in section 3.3.3, there was an increase in the P_2O_5 grade after magnetic separation. This agrees with the observed increase in the apatite abundance.

- (6) Final concentrate using orthophosphoric acid as modifier:

The average grain size of apatite was $360\mu m$. An increase in the average grain size of apatite compared to the flotation without application of orthophosphoric acid could be attributed to the increase in the negative charge on the coarse-grained apatite which made the grains to float easily thereby increasing the phosphate grade as seen earlier. The grain size of apatite ranged from $60-437\mu m$. Calcite appeared as fine-grained and stained. Very few opaque minerals were present. Although from electrokinetic studies opaque minerals were expected not to float, some of them floated. This was probably due to the fact that some of them were negatively charged at the flotation pH.

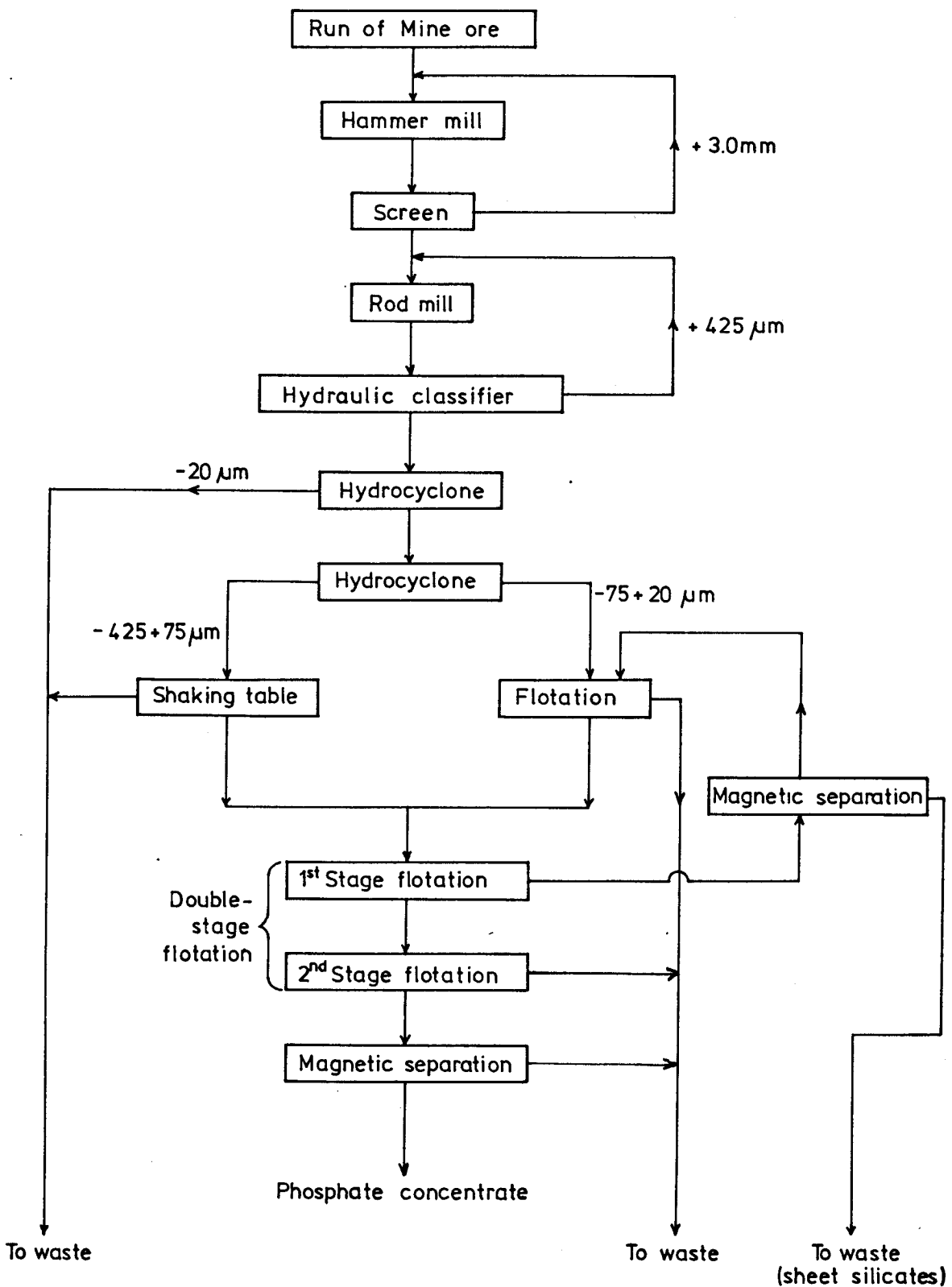


Fig.30. Proposed flowsheet

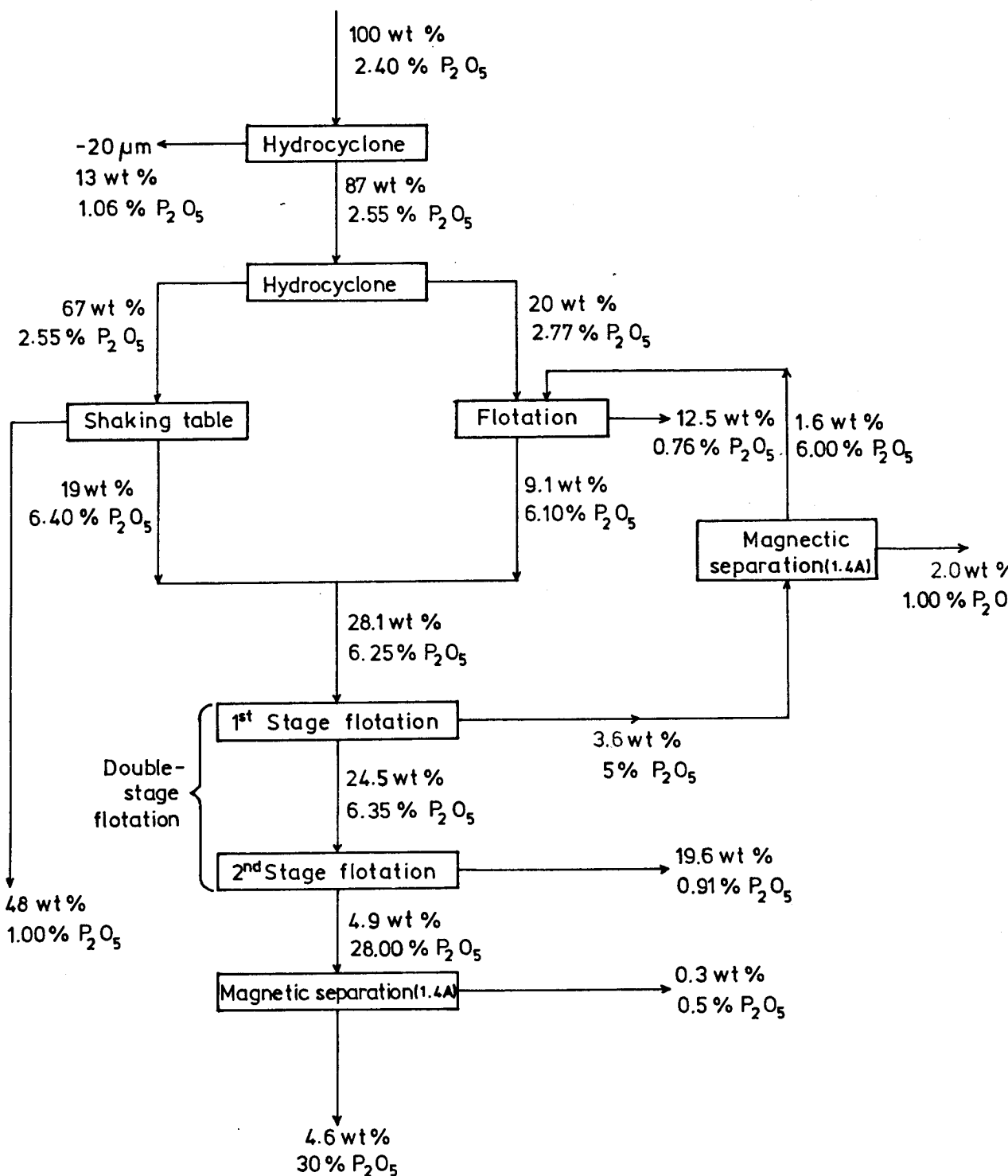


Fig. 31. Material balance on part of beneficiation flowsheet.

Notes on proposed flowsheetSize Reduction:

The friable nature of the ore suggests that a multiplicity of sizing equipment would be a main feature in order to avoid production of fines during comminution. For a single stage crushing the reduction ratio would be too high to achieve with one crusher. A hammer mill has been suggested due to its high ratio of reduction (usually 10/1 or more) and high capacity.

Comminution itself can be accomplished with hammer mills and rod mills since the ore is soft. The use of hammer mills is, however, dependent on the size of run of mine ore. If this is large then a jaw crusher may be used for primary crushing. Each crushing stage is in closed circuit with a sizing stage. This is to avoid overproduction of fine material.

Sizing:

Due to the friable nature of the ore, production of fines would be inevitable. Hydrocyclones would be suitable for sizing in the medium to fine size range which in this case is the treatment size range.

Preconcentration:

This step is necessary to upgrade the ore before flotation. The $-425 + 75\mu\text{m}$ can be preconcentrated using a shaking table and the $-75 + 20\mu\text{m}$ using a primary flotation process since it would be inefficient to preconcentrate this size range using a shaking table.

Flotation:

Using an amine as a collector would require a double-stage process in which the first stage would be for flotation of sheet silicates while the second stage would be for phosphate flotation.

Magnetic separation:

This may be carried out as a last stage in the treatment process since the iron minerals are also floated with apatite.

General:

The low grade nature of the ore indicates that several processing stages are inevitable. This is because it is not possible in practice to achieve a high concentrate grade and at a reasonable recovery in one pass through any process of concentration.

CONCLUSIONS

A study has been made on the beneficiation of the Kaluwe phosphate deposit. The techniques used in the study included mineralogical and chemical analyses of the ore, gravity concentration techniques, magnetic concentration tests, electrokinetic studies of the major minerals: apatite, clacite and magnetite which formed the basis of the flotation tests.

From the results obtained the following conclusions can be made:

Mineralogical and chemical analyses

- (a) Calcite was the most abundant mineral (74.4 %) in the ore with minor amounts of apatite (5.9%), opaques (13.5%), quartz/feldspar (5.7%) and biotite (0.5%). The ore was weathered.
- (b) An XRF scan on the head sample indicated trace elements of Ba, Nb, Yt, Zr, Zn, Mn, Ce, V and Ti.
- (c) The apatite in the ore was medium-grained. The average grain size of the apatite was $390\mu\text{m}$.
- (d) The head grade of the sample used was 2.2 % P_2O_5 with other constituents being 47.7% CaO, 1.6 % MgO, 1.2% Al_2O_3 , 3.3 % Fe_2O_3 and 4.7 % SiO_2 .
- (e) The apatite from the ore was more than 80% liberated at sizes below $425\mu\text{m}$.

Process Selection

- (a) Heavy medium tests showed that it was possible to apply gravity concentration techniques to upgrade the feed. It was noted that a process using a gravity concentration

technique could not be used to produce a final concentrate product. This could only be used in combination with other processes in order to yield acceptable concentrates. Gravity concentration was possible in the size range of high liberation ($-425\mu\text{m}$) and shaking tables and spirals were suitable to treat this size range.

- (b) Magnetic concentration should be part of a treatment process since the ore contained more than 10% opaques (iron minerals).
- (c) Since liberation of apatite occurred in the medium size range and due to the anticipated inefficiency of gravity concentration techniques caused by the low concentration criterion, flotation would be the best technique for the beneficiation of this ore.
- (d) Combination of magnetic and heavy medium techniques indicated that an average grade of 28.5 % P_2O_5 could be obtained with a recovery of 44.1 %.
- (e) Electrokinetic measurements using Kaluwe water showed that apatite could be successfully concentrated using a cationic collector in the alkaline pH range.
- (f) Reagents containing Ca^{2+} as the active modifying species could not be used as calcite depressants in Kaluwe water as both apatite and calcite behaved similarly rendering their separation impossible.

- (g) Sodium silicate could not be used as a calcite depressant in Kaluwe water as it made calcite negatively charged throughout the pH range studied and the pH range where apatite was negative. This could make it difficult to selectively concentrate apatite.
- (h) Using orthophosphoric acid as a modifier made the apatite surface more negative throughout the pH range studied. This was marked at alkaline pH range indicating that in this range it could be used to enhance the negative charge on the apatite surface thereby improving selectivity.

Process Testing

- (a) A shaking table could be used to upgrade the ore before flotation. The ore was upgraded to an average of about 5.0 % P_2O_5 in one stage. The average recovery for a single stage was 61.7 %. The low grades obtained were attributed to the low grade nature of the ore and the low concentration criterion for gravity concentration which made the process inefficient.
- (b) The average low grade of 2.7% P_2O_5 obtained after magnetic separation suggested that magnetic concentration could only be used to upgrade the ore. It was further concluded that magnetic separation could be carried out as a last stage in order to minimise P_2O_5 losses in the magnetic fractions due to treatment of small amounts of material.
- (c) The -425 + 20 μ m was considered as the flotation feed since liberation study had indicated that maximum liberation of apatite occurred in this size range.

- (d) Kaluwe phosphate ore could successfully be treated by flotation after preconcentration of the flotation feed by a shaking table ($-425 + 75\mu\text{m}$) and a primary flotation ($-75 + 20\mu\text{m}$). The cationic collector tried, cocoamine, gave satisfactory results in the alkaline pH range. The optimum flotation conditions for this collector were between pH 9.5 and 10. The collector concentration used was 700 g/t. A recovery of 62.6% with a grade of about 27.6 % P_2O_5 were obtained. Magnetic separation improved the grade marginally to 30% P_2O_5 .
- (e) Reverse anionic flotation of calcite by a sulphonated fatty acid was not successful. The collector tried, petroleum sulphonate, gave poor results. The optimum flotation conditions were between pH 7 and 9 and collector concentration of 1200 g/t. The low grades of about 4.8 % P_2O_5 obtained were attributed to the large amount of calcite to be floated which led to inefficient flotation.
- (f) Orthophosphoric acid could be used as a phosphate modifier to enhance the negative charge on the surface thereby improving selectivity in flotation. With the application of this reagent at 7 and 9 kg/t, grades of 29.6 % P_2O_5 and 31.8 % P_2O_5 were obtained with recoveries of 49.5 % and 46.2 % respectively. The increase in grade could be attributed to the flotation of coarse grained apatite as well. The low recoveries obtained were attributed to the decrease in the amount of concentrate obtained.

As a general conclusion it is considered that recovery of apatite from Kaluwe ore is likely to be viable. But, it must be emphasised that this can only be achieved after preconcentration of the flotation feed and carrying out flotation in Kaluwe water.

From an economic point of view the multiplicity of processing steps implies increased capital and operating costs. Although the ore characteristics suggested straightforward separations, low recoveries were expected because of the many routes through which phosphate values were lost.

APPENDICES

Table 1. Size distribution of Kaluwe Ore

Screen Aperture μm	Weight Retained kg	Weight % Retained	Cumulative weight %	
			Retained	Passing
+ 2000	0.34	4.56	4.56	95.44
+ 1400	1.22	16.35	20.91	79.09
+ 850	1.08	14.48	35.39	64.61
+ 600	0.67	8.98	44.37	55.63
+ 425	0.58	7.77	52.14	47.86
+ 300	0.53	7.11	59.25	40.75
+ 212	0.52	6.97	66.22	33.78
+ 150	0.50	6.70	72.92	27.08
+ 106	0.59	7.91	80.83	19.17
+ 75	0.57	7.64	88.47	11.53
+ 53	0.54	7.24	95.71	4.29
- 53	0.32	4.29		
Total	7.46	100		

Table 2. Liberation characteristics of apatite, opaques
and calcite as a function of particle size.

Size Aparture μ m	Liberation, %		
	Apatite	Calcite	Opaques
+2000	21.9	60.5	39.9
+1400	15.6	64.5	49.7
+ 850	28.2	57.9	61.1
+ 600	38.0	54.4	60.2
+ 425	53.5	76.0	66.0
+ 300	72.8	70.5	67.4
+ 212	84.5	81.6	82.5
+ 150	96.6	87.2	86.4
+ 106	96.2	88.5	87.0
+ 75	n.d	n.d	n.d
+ 53	n.d	n.d	n.d

n.d = not determined

Table 3. Heavy media separation (Bromoform s.g. = 2.89)

Particle size μm	Sinks g	Floats g	Particle recovery %
+2000	2.67	27.21	99.6
+1400	2.21	27.61	99.4
+ 850	2.70	27.17	99.6
+ 600	3.02	26.79	99.4
+ 425	2.77	26.97	99.1
+ 300	3.38	26.46	99.5
+ 212	4.16	25.75	99.7
+ 150	4.25	25.54	99.3
+ 106	2.10	27.65	99.2
+ 75	0.55	28.97	98.4

Table 4. Size Distribution and chemical Analysis of the prepared flotation feed.

Size Aperture μm	Weight %	Assay, %						Distribution, %					
		P ₂ O ₅	CaO	MgO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	P ₂ O ₅	CaO	MgO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃
-425+300	20.09	2.4	46.8	1.8	3.8	0.3	5.8	19.9	19.2	14.2	18.4	17.6	20.2
-300 +212	15.20	2.6	47.9	3.4	3.4	0.2	5.6	15.1	14.9	20.8	12.5	8.8	14.7
-212 +150	10.31	2.8	45.7	4.4	4.1	0.3	7.6	10.7	9.6	18.3	10.2	8.8	13.6
-150 +106	10.62	2.8	50.5	2.2	5.2	0.3	6.2	12.3	11.0	9.4	13.3	8.8	11.4
-106 + 75	10.70	2.5	50.5	2.0	3.5	0.4	5.7	11.1	11.1	8.6	9.0	11.8	10.6
-75 + 53	12.08	2.9	51.6	1.8	4.9	0.4	5.1	14.5	12.8	8.7	14.3	14.7	10.7
-53 + 20	8.00	2.6	49.9	1.2	3.7	0.4	6.1	7.3	8.2	3.9	7.1	8.8	8.5
-425+20 (Flotation feed)	87.00	2.6	48.7	2.4	4.0	0.3	6.0	90.9	86.8	84.3	84.9	79.3	89.7
- 20	13.00	1.0	49.9	3.0	4.8	0.5	4.6	9.1	13.2	15.7	15.1	20.7	10.3
Calculated Head	100.00	2.4	48.9	2.5	4.1	0.3	5.8	100.0	100.0	100.0	100.0	100.0	100.0

Table 5: Effect of pH on the phosphate grade and recovery for Kaluwe ore at a cocoamine concentration of 300 g/t

pH	Recovery, %		Grade, % P_2O_5			P_2O_5 Distribution, %		
	CONS	MIDS	CONS	MIDS	TAILS	CONS	MIDS	TAILS
7	2.1	11.9	2.0	6.0	2.3	1.8	10.2	88.0
8	3.8	9.5	2.6	6.5	1.9	3.8	9.7	86.5
9	4.1	24.8	3.0	9.5	1.8	3.8	23.1	73.1
10	5.4	39.0	3.0	11.0	1.6	4.8	34.9	60.3
11	3.0	3.6	3.1	3.0	2.1	3.0	3.5	93.5
12	1.2	1.9	3.0	2.5	2.0	1.2	2.0	96.8

Table 6. Effect of collector concentration on the phosphate grade and recovery for Kaluwe ore at pH 10.

Collector Concentration g/t	Recovery, %		Grade, % P_2O_5			P_2O_5 Distribution, %		
	CONS	MIDS	CONS	MIDS	TAILS	CONS	MIDS	TAILS
500	5.8	9.8	3.0	6.4	1.9	6.8	11.6	81.6
600	8.7	25.8	4.8	12.0	2.1	7.9	23.4	68.7
700	2.7	40.0	2.7	11.5	1.4	3.1	36.9	60.0
800	2.8	29.2	2.9	10.4	1.7	3.0	31.2	65.8
1000	4.1	58.0	3.3	11.8	1.3	3.7	39.3	57.0
1200	3.8	55.7	2.9	11.7	1.6	3.3	47.7	49.0

Table 7. Effect of pH on the phosphate grade and recovery for Kaluwe ore in reverse anionic flotation of calcite at a petroleum sulphonate concentration of 900 g/t

pH	Recovery, %	Grade, % P_2O_5		Distribution, %	
	CONS	CONS	TAILS	CONS	TAILS
7	34.0	4.2	2.1	34.2	65.8
8	46.7	3.5	2.0	47.3	52.7
9	35.6	3.5	2.2	36.1	63.9
10	26.7	4.0	2.2	26.4	73.6
11	28.0	4.0	1.9	29.2	70.8

Table 8. Effect of collector concentration on the phosphate grade and recovery for Kaluwe ore in reverse anionic flotation of calcite at pH 8

Collector Concentration g/t	Recovery, %	Grade, % P_2O_5		Distribution, %	
	CONS	CONS	TAILS	CONS	TAILS
900	46.7	4.2	2.1	47.3	52.7
1000	52.7	3.9	1.7	51.0	49.0
1200	67.1	4.8	1.7	64.3	35.7
1400	41.9	4.0	1.9	40.9	59.1

Heavy liquid separation procedure

Using Bromoform CHBr_3 , s.g. 2.89 at 20°C

Storage: In a dark bottle away from strong light and at a low temperature.

Diluent: Acetone (also for washing out vessels)

Warning: (1) Keep vessels closed and check s.g. regularly, because of high rate of acetone evaporation
(2) The solution must be kept away from open flames
(3) Work in an extraction chamber to avoid inhaling the fumes.

Apparatus:

- (1) A retort stand with funnel support and clamp
- (2) A glass separating vessel
- (3) Glass filter funnel
- (4) Watch glass
- (5) Collecting beaker for used liquid.

Procedure:

- (1) Assemble apparatus, check clip is in position and closed.
- (2) Pour about 200 ml of bromoform (2.89 s.g) into the glass separating vessel and place watch glass in position to minimise evaporation
- (3) Weigh out a portion of perfectly dry, cleaned and sieved sample;

10g/100 ml (bromoform)	if grain size	0.06 mm ($60\mu\text{m}$)
15g/ "	" " "	60-100 μm
20 / "	" " "	100-500 μm
30 / "	" " "	500-1000 μm

- (4) Empty weighed sample into the heavy liquid in the vessel and stir using a glass rod, ensuring all the grains are fully immersed and well agitated.
- (5) Wash all grains that stick to the glass vessel and stirring rod back into the heavy liquid by using a stream of heavy liquid from a squeezable plastic wash bottle and replace the watch glass.
- (6) Allow heavy grains to settle to the bottom and, when no further grains seem to be sinking, stir the light fraction again and wash down the sides of the vessel and stirring rod as before.
- (7) Place a fluted filter paper in the lower filter funnel.
- (8) When there are at least a few centimeters of clear liquid between floating grains and sunken grains, open the clip and let the liquid run out of the separating vessel into the filter paper below.
- (9) As soon as all the heavy grains are out close the clip.
- (10) Wash the heavy grains in the filter paper with acetone and take for drying.
- (11) Repeat 7, 8, 9 and 10 for the lighter grains (floats)
- (12) Weight the two fractions. Fractions are ready for subsequent analyses.

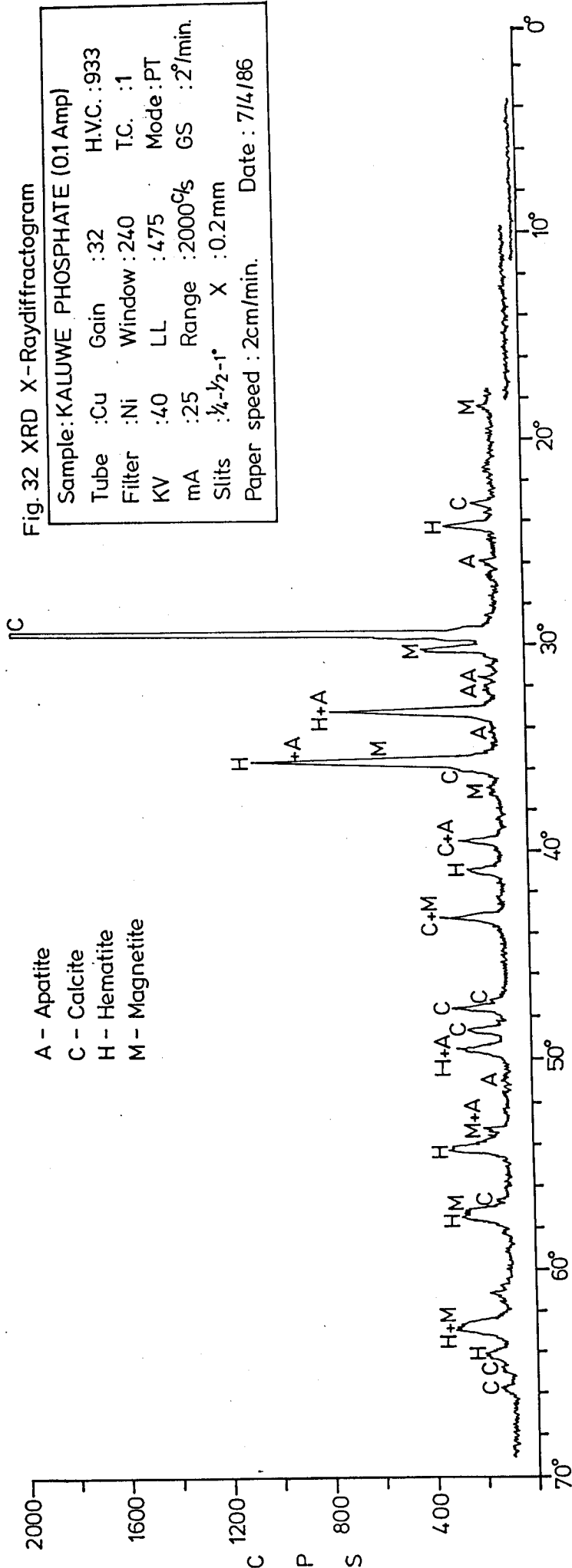
X-ray diffraction patterns of some magnetic fractions from Kaluwe ore.

Fig. 32 - at 0.1 A

Fig. 33 - at 0.5 A

Fig. 34 - at 1.0 A

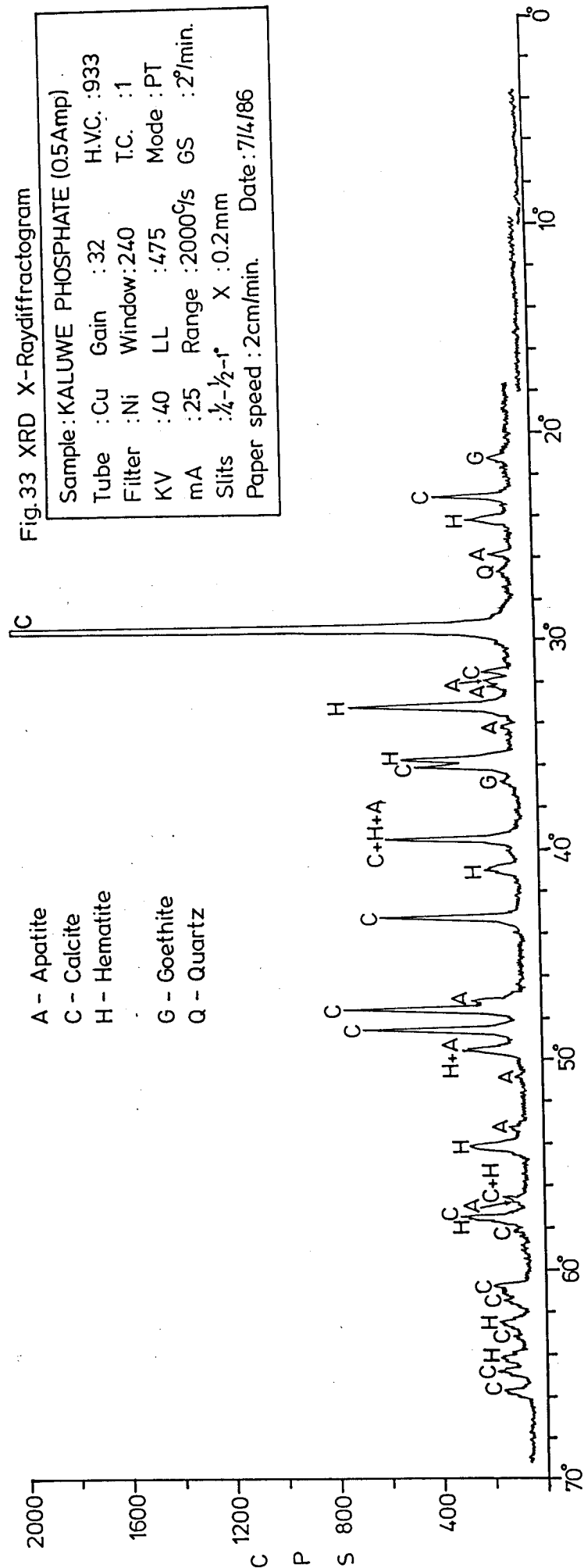
Minor phases are not labelled for clarity. All the patterns show the presence of calcite in the magnetic fractions. This results from the stained calcite which become magnetic and so report in the magnetic fraction.



A - Apatite
 C - Calcite
 H - Hematite
 G - Goethite
 Q - Quartz

Fig. 33 XRD X-Raydiffractogram

Sample: KALUWE PHOSPHATE (0.5Amp)
 Tube : Cu Gain : 32 H.V.C. : 933
 Filter : Ni Window: 240 T.C. : 1
 KV : 40 LL : 475 Mode : PT
 mA : 25 Range : 2000 G/s GS : 2°/min.
 Slits : $\frac{1}{4}$ - $\frac{1}{2}$ -r X : 0.2mm
 Paper speed : 2cm/min. Date : 7/4/86



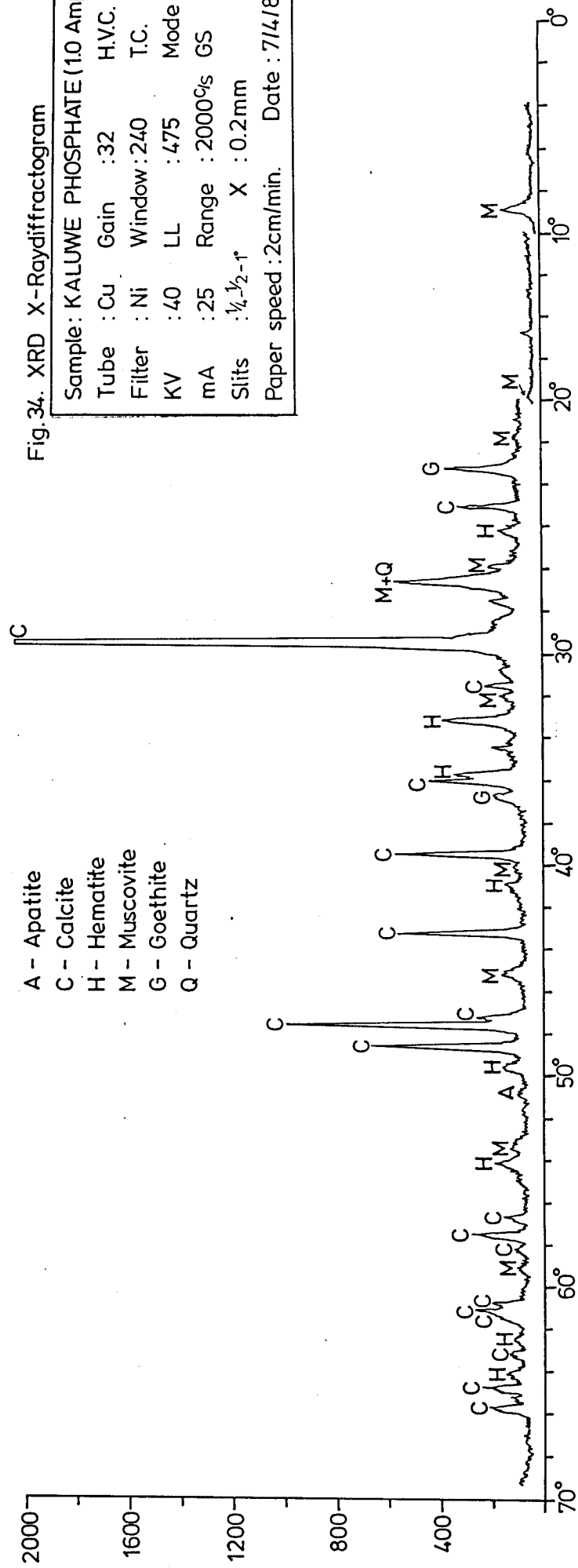


Fig. 34. XRD X-Ray diffractogram

Sample: KALUWE PHOSPHATE (10 Amp)									
Tube	: Cu	Gain	: 32	H.V.C.	: 933				
Filter	: Ni	Window	: 240	T.C.	: 1				
KV	: 40	LL	: 475	Mode	: PT				
mA	: 25	Range	: 2000°s	GS	: 2°/min				
Slits	: $\frac{1}{4}$ - $\frac{1}{2}$ -1"		X	: 0.2mm					
Paper speed			: 2cm/min.	Date	: 7/4/86				

- A - Apatite
- C - Calcite
- H - Hematite
- M - Muscovite
- G - Goethite
- Q - Quartz

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