

**BRITISH GEOLOGICAL SURVEY**  
**Mineralogy & Petrology Group**

**Technical Report No. WG/94/33R**

**EVALUATION OF LABORATORY METHODOLOGIES FOR  
FROTH FLOTATION OF FELDSPAR AND KAOLIN**

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**CJ Mitchell**

## **1. INTRODUCTION**

### **1.1. Aims**

This report describes work carried out to establish and evaluate methodologies for the laboratory froth flotation of feldspar and kaolin. One of the main aims was to ascertain the feasibility of separating glass-grade feldspar from granite quarry dust and fines. As part of this, a method for HF-free froth flotation of feldspar was evaluated. This work was carried out as part of the ongoing 'Maintenance of capability in Mineral Sciences' project, 75CB

### **1.2. Background**

Since the 1920s, froth flotation has been widely used in the processing of minerals. Approximately 2000 million tonnes of solids are treated every year by flotation. The reasons for its popularity include the capability for high throughput on a continuous basis, relatively low operating and capital costs, and a wide variety of readily available and proven equipment and reagents. Also it is a very robust process to operate and control (Klimpel, 1993). Froth flotation is commonly used in the processing of industrial minerals, especially barite, feldspar, fluorite, graphite, kaolin, phosphate, potash, silica sand and talc. Unlike metallic minerals, industrial minerals have low concentration ratios (waste:valuable mineral) in the order of 2 to 3:1. This encourages the use of gravity, electrostatic, magnetic and heavy media separation, as well as froth flotation (Crozier, 1990).

Minerals with hydrophobic surfaces attach to air bubbles and float to the top of a pulp (mineral suspension) to form a mineral froth (Figure 1). This basic principle of froth flotation is known as coursing bubble or bubble capture flotation (Crozier, 1990). The target mineral is generally recovered from the froth (direct flotation) although the waste minerals can be removed this way leaving the valuable minerals to sink (reverse flotation). Chemical reagents, such as collectors, frothers and modifiers, are used to float specific minerals. Some industrial minerals, such as talc and graphite, have naturally hydrophobic surfaces and therefore require little in the way of chemical treatment to make them float. Other industrial minerals, such as feldspar, fluorite and

calcite, have hydrophilic surfaces and therefore do require chemical treatment. Collectors are used to treat the surfaces of these minerals, these displacing the adsorbed water layers and forming hydrophobic coatings. Commonly used collectors include low-cost soaps, fatty acids, amines, quaternary ammonium compounds, sulphonates and petroleum oils. Frothers are surface active (surfactants) reagents added to the pulp to reduce the surface tension of air bubbles and help stabilise the mineral froth. Commonly used frothers include pine oil, alcohols (eg methyl isobutyl carbinol), polyethers, and amines. Many industrial mineral collectors are surface active and can therefore double as collector and frother. Modifiers are reagents added to control pH (including lime, sodium carbonate and sulphuric acid), enhance flotation of certain minerals (activators such as HF), or prevent the flotation of certain minerals (depressants such as sodium silicate).

Froth flotation is becoming more popular, especially as the economics of mineral extraction demands the exploitation of lower-grade, finer-grained deposits. To partly accommodate this, flotation cells have increased in size (1000+ cubic feet in capacity) which offers reductions in the cost per tonne of ore processed. Also high capacity, non-mechanical flotation devices, such as columns are being used, generally in scavenger and cleaner operations (Crozier, 1990).

Conventional mechanical flotation machines consist of a square cell with a central impeller, which simultaneously stirs and draws air into the pulp. Mechanical agitation is required to keep particles in suspension and promote bubble:particle contact. A relatively undisturbed zone is also required in order for efficient separation of the mineral froth from the pulp (Figure 1). The practical outcome is a compromise between the two. In an industrial plant the cells are arranged in rows, with adjustable weirs between them to allow the passage of mineral froth between cells. The feed is progressively 'cleaned' as it passes from one cell to the next, progressing from rougher, scavenger, cleaner and finally to recleaner cells. This is effective in producing high mineral grade, although inevitably results in a reduction of mineral recovery. Column flotation machines consist of columns up to 10 metres in height with no mechanical agitation. The process relies on the counter-current produced between rising air bubbles and downward moving feed material/wash water. A single column has the capacity of a whole row of conventional cells. Improved mineral recovery and fines separation are attained due to the greater residence time and relatively undisturbed nature of the separation.

## **2. FELDSPAR**

### **2.1. Introduction**

Feldspar is a common rock forming mineral that occurs as Na-, K- and Ca-rich varieties. Na- and K-feldspars (also known as the alkali feldspars) are exploited as raw materials for use in glass and ceramics as a source of alkalis, alumina and silica. Alkali feldspars are major constituents of granite (which contain between 50 and 70% feldspar) related rocks, although these rocks are not normally exploited for their feldspar content. Feldspar of commercial significance generally occurs as relatively large crystals (>3 cm) free of iron-bearing impurities and is easily separated from other minerals. These can be found in coarsely-crystalline igneous rocks such as pegmatite and alaskite.

Industrial-scale processing of feldspar involves the removal of quartz, mica and other iron-bearing impurities. Coarse material may be hand- or photometrically-sorted. Finer material may be processed by a combination of gravity separation, magnetic separation and/or froth flotation. Froth flotation of feldspar is carried out under acidic conditions, using amine-based collectors. HF is generally used as an activator in this process, although the mechanism(s) by which it enhances flotation are poorly understood. HF may remove weathered material and/or coatings and form complex silicofluoride and/or aluminofluoride compounds on the clean surface of feldspar (Manser, 1975). Fluoride salts, such as sodium fluoride, may be used as an alternative to HF. The use of HF is hazardous, requiring careful handling of reagents and management of the flotation process. One of the aims of this study was to evaluate a non-HF flotation route developed by Akzo Nobel, using a specially formulated fatty acid collector.

### **2.2. Samples**

The samples examined as part of this investigation were granite quarry fines from Scotland and feldspar pegmatite fines from Takua Pit Thong mine in Thailand. The Scottish granite contains about 55% alkali feldspar, with the remainder being mainly quartz and a small amount of biotite mica and other ferromagnesian minerals. Production of glass-grade feldspar from the stockpiled quarry fines would have a cost advantage over other commercial feldspar products, as the mining and primary crushing for aggregate production have already been carried out (Mitchell, CJ & Henney, PJ, 1994; Mitchell, CJ and Evans, EJ, 1995).

The Thai feldspar fines result from the production of ceramic-grade feldspar from granite pegmatite. The fines consist of dominant microcline feldspar, with major quartz, minor muscovite mica and trace amounts of tourmaline. The raw material is hand-sorted and material finer than 2 cm is being stockpiled. These fines contain a significant amount of muscovite mica

(~10%) which is too fine to remove by hand. Air classification trials were carried out by BGS in 1990 and ceramic-grade feldspar concentrates were produced (Mitchell *et al.*, 1990). However, <500  $\mu\text{m}$  material cannot be processed using this technique. The effectiveness of HF-free feldspar flotation in producing a ceramic-grade feldspar concentrate from this material was tested.

### 2.3. Methods

A sample of the Scottish granite was examined using both petrological microscope and electron microprobe to determine the liberation characteristics and chemistry of the feldspar. The mineralogy, including feldspar liberation size, of the granite was determined by examination of a polished thin section using a petrological microscope. The modal proportions and size range of the minerals present were also determined. The major element chemistry of the feldspar was determined by CAMECA SX50 automated electron microprobe using wavelength dispersive methods to enhance detection limits.

The mineralogy of the Thai feldspar was determined by X-ray diffraction analysis. The modal proportions of the minerals present were determined by examination of the products using a binocular microscope.

The samples were prepared for processing by a combination of screening and/or grinding of oversize material. The granite fines were split into two processing routes, one using material sized to 600 to 106  $\mu\text{m}$  and the other material sized to 300 to 106  $\mu\text{m}$  (Figure 2). Magnetically-susceptible material was removed from the samples by processing using a Carpc high intensity induced-roll magnetic separator. The non-magnetic products formed the feed material for froth flotation. The Thai feldspar fines were sized to 500 to 106  $\mu\text{m}$  (Figure 3).

Froth flotation was carried out to produce feldspar concentrates using a two-stage process to remove the mica and quartz present in the samples (Appendix A). The flotation was carried out in a pyrex cell using a Denver D12 Laboratory flotation machine. Deionised water was used to prepare mineral suspensions. The pH was controlled with weak solutions of sulphuric acid and sodium hydroxide. Excessive frothing during flotation was controlled with heavy distillate (which partially neutralizes the effect of the surfactant). In the first stage mica was floated away from the quartz and feldspar. The reagents used were alkylamine acetate ('Armoflote 64' from Akzo Nobel) as collector and methyl isobutyl carbinol (MIBC) as frother. In the second stage feldspar was floated away from quartz. The reagents used were a fatty acid derivative/Butan-2-ol collector ('Armoflote 543', and 'Lilafлот OT 36' in later trials, from Akzo Nobel) and MIBC as frother. Flotation was repeated several times in order to maximise the amount of separation. Prior to each repeated flotation the samples were washed in hot water and detergent in order to remove reagent residues.

The major element chemistry of the feldspar concentrates was determined using a Phillips PW 1480 X-ray fluorescence (XRF) spectrophotometer.

## **2.4. Results and discussion**

### **2.4.1. Granite feldspar**

The froth flotation trials were successful in producing feldspar concentrates from the granite fines. The 600 to 106  $\mu\text{m}$  concentrate contained at least 95% feldspar and 0.19%  $\text{Fe}_2\text{O}_3$ . The 300 to 106  $\mu\text{m}$  concentrate also contained at least 95% feldspar and 0.26%  $\text{Fe}_2\text{O}_3$ . High-grade by-products were also produced. The 600 to 106 $\mu\text{m}$  route produced a concentrate grading about 95% mica. The 300 to 106  $\mu\text{m}$  route produced a quartz concentrate containing 99.35%  $\text{SiO}_2$  and 0.02%  $\text{Fe}_2\text{O}_3$  (Table 1).

The combined magnetic separation / froth flotation route produced feldspar concentrates from the granite fines with  $\text{Fe}_2\text{O}_3$  contents approaching the specifications of glass-grade feldspar. Although  $\text{Fe}_2\text{O}_3$  contents of <0.1% are generally quoted for glass-grade material, levels as high as 0.2% can be tolerated if the iron content of other raw materials allows. Although initial spot analyses by EPMA of the granite feldspar suggested a very low iron content, the presence of fine-grained biotite inclusions appears to be responsible for the slightly higher levels of iron in the concentrate. Further grinding to liberate this mica would probably produce a concentrate with a lower iron content, although this would then fall outside the particle-size specifications for glass-grade feldspar.

Quartz-rich by-products were also produced from the feldspar flotation. The 300 to 106  $\mu\text{m}$  material produced a quartz by-product containing 99.35%  $\text{SiO}_2$ , 0.02%  $\text{Fe}_2\text{O}_3$  and 0.01%  $\text{TiO}_2$  (Table 1). This is suitable for use in glass making and in the manufacture of sodium silicate.

### **2.4.2. Thai feldspar**

The froth flotation trial was successful in producing a feldspar concentrate from the Thai feldspar fines which meets the published specifications for ceramic-grade feldspar (Table 2). The feldspar concentrate contained at least 95% feldspar with 0.09%  $\text{Fe}_2\text{O}_3$ . High grade by-products were also produced, including a concentrate grading about 95% mica and a quartz concentrate containing 97.21%  $\text{SiO}_2$  and 0.04%  $\text{Fe}_2\text{O}_3$  (Table 2).

### 2.4.3. Best practice for laboratory flotation of feldspar

It was observed that addition of collector reagent to the sample suspensions (worked on in this report) invariably caused vigorous 'flocculation', with large aggregates forming, even during intense stirring of the pulp. This aggregation could be caused by electrostatic attraction between particles. This is known as "charge patch attraction" (Cyanamid, 1989). Collector reagent will often only partially coat the surface of a mineral. This results in a positive charge on those patches of the mineral surface to which the polymer is adsorbed and a negative charge on the uncoated parts, hence the electrostatic attraction between particles. The addition of hydrogen ions (in the form of acid) neutralises the negative surface charge remaining on the mineral surface and eliminates the electrostatic attraction between minerals. Thus, the addition of acid gradually breaks down the aggregates until, at an optimum point (in this case below pH 2), the suspension is fully dispersed. This ensures that when the suspension is aerated that all mineral grain surfaces are available to the bubbles, not just the surfaces of aggregates. This will avoid the flotation of unwanted minerals that would inevitably be entrained in aggregates of the target mineral.

Frother reagent is generally added after the mineral pulp has been fully conditioned with the collector and immediately before aeration. The collector requires time to disperse and coat mineral grains. The frother however disperses quickly, and if added at the same time as the collector might prevent it from coating all the grains.

## 3. KAOLIN

### 3.1. Introduction

Kaolin is a white clay consisting predominantly of kaolinite. It is mainly used in the filling and coating of paper, as a ceramic raw material, as a filler in paint, rubber and plastic and many other applications (Bloodworth *et al.*, 1994). Kaolin occurs as primary deposits, formed by in situ alteration of feldspathic rocks such as granite, and secondary deposits, formed by erosion of existing deposits and redeposition. The primary kaolins of SW England are extracted by hydraulic mining. The kaolin suspensions are processed by classifiers to remove the coarse sand, hydrocyclones to remove the fine sand and hydroseparators (settling tanks) to remove coarse silt. The <15  $\mu\text{m}$  material is suitable for use as paper filler. Further processing by centrifuges produces very fine material, typically 75% <2  $\mu\text{m}$ , which is suitable for use in paper coating (ECCI).

Kaolinite concentrates in the <2  $\mu\text{m}$  fraction, but it also occurs in coarser size fractions, such as the residue from the settling tanks and the underflows from hydrocyclone separations. Froth



flotation can be used to recover the kaolinite previously lost in these products (often stored in mica tailings lagoons). Low-solids sand grinding of kaolin followed by froth flotation can be used to recover kaolinite for filler or coating grade applications (depending upon the amount of grinding). This involves the attrition of a clay slurry with highly spherical sand in polyurethane lined vessels. Kaolin contaminated with anatase ( $\text{TiO}_2$ ) can be upgraded by 'ultraflotation'. A carrier mineral, usually ground limestone, is added to the pulp and this scavenges out the anatase. The limestone:anatase mixture is removed by flotation using limestone reagents producing a kaolin product with significantly enhanced whiteness (Bloodworth *et al.*, 1993).

### 3.2. Samples

The kaolin-bearing samples used in this investigation are derived from a pegmatite near Chilulwe in the Central Province of Zambia. The head material consists mainly of K-feldspar, with a small amount of kaolinite and muscovite mica. A previous investigation successfully produced ceramic-grade kaolin products from this material (Mitchell, 1994). Wet screening and hydrocycloning were used to remove sand and silt. The kaolin overflow products contained 79-87% kaolinite, with 55-58% <2  $\mu\text{m}$  in size. However, only 20 to 30% of the total kaolinite present in the head sample was recovered in this overflow product. Upwards of 60% of the kaolinite was retained in the hydrocyclone underflow products, and between 10 and 20% in the sieve residues. In order to improve the kaolinite recovery from this material, sieve residues and hydrocyclone underflow products retained from the previous investigation were reprocessed using froth flotation.

### 3.3. Methods

Most of the sieve residues and hydrocyclone underflow samples required no preparation prior to froth flotation. A sub-sample taken from the 1 inch hydrocyclone underflow material was subjected to a form of 'sand grinding' in order to liberate kaolinite from feldspar. This involved shaking the sample with sand (graded to remove <106  $\mu\text{m}$  particles) and water in a Turbula mixer mill for 1 hour. The suspension was wet screened to remove the sand, and <63  $\mu\text{m}$  material was dried and used for processing. The processing flowsheet linking techniques used in the previous investigations and this study is shown in Figure 4.

Froth flotation was carried out to produce kaolinite concentrates (Appendix B). The flotation was carried out in a pyrex cell using a Denver D12 Laboratory flotation machine. Deionised water was used to prepare mineral suspensions. The pH was controlled with weak solutions of sulphuric acid and sodium hydroxide. Excessive frothing during flotation was controlled with heavy distillate (which partially neutralizes the effect of the surfactant). The collector used was

alkylamine ('Armoflote 13' from Akzo Nobel). This collector also acts as a frother. During flotation kaolinite is floated away from the other minerals. Flotation was repeated several times in order to maximise the amount of separation. Prior to each repeated flotation the samples were washed in hot water and detergent in order to remove reagent residues.

The proportions of kaolinite present in the processing products were determined by thermogravimetry (TG) using a Stanton Redcroft 770 thermobalance. The weight losses of the samples heated to 1000°C were recorded and interpreted as mineral percentages. The mineralogy of the kaolinite concentrates was determined by X-ray diffraction (XRD). The samples were loaded into aluminium holders and scanned over the angular range 3 to 50° 2θ using a Phillips PW 1700 X-ray diffractometer. The XRD traces were interpreted with reference to the JCPDS dataset.

### **3.4. Results**

#### **3.4.1. Underflow products, Chilulwe kaolin**

The froth flotation trials produced only a marginal increase in the kaolinite content of the underflow products. The kaolinite content of the 30 mm underflow was increased from 64% to 71% and the 1 inch underflow from 63 to 75%. No appreciable improvement in the grade of the kaolin was produced by the sand-grinding trial (Table 3).

#### **3.4.2. Sieve residues, Chilulwe kaolin**

The froth flotation trials resulted in a modest increase in the kaolinite content of the sieve residues. The kaolinite content of the 250 to 125 µm fraction was increased from 7% to 12% and the 125 to 63 µm fraction from 20% to 26% (Table 3).

### **3.4. Discussion**

The current investigation has shown that froth flotation can be used to separate kaolin from other minerals. However, despite reasonable kaolinite recoveries (70 to 80%) the grades of the kaolinite concentrates were low. Examination of these concentrates by XRD reveals that an appreciable amount of feldspar and mica is still present. Also the middling and sink products still have relatively high kaolinite contents. This indicates that the sand-grinding carried out in this investigation was not aggressive enough to produce complete liberation of the kaolinite. Future laboratory trials might focus on 'stirred media milling' of the wet processing residues. This would involve the use of a silica sand with highly spherical grains, a suspension with a

low solids content and a high torque stirrer / attrition scrubber. This would hopefully liberate the kaolinite present and enable the production of high-grade kaolinite concentrates by froth flotation.

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**Table 1. Summary of processing, Scottish granite fines**

	Head (wt %)	Feldspar concentrates -600 +106 $\mu\text{m}$ (wt %)	-300 +106 $\mu\text{m}$ (wt %)	Commercial glass-grade feldspar * (wt %)	Quartz concentrate -300+106 $\mu\text{m}$ (wt %)	Mica concentrate -600+106 $\mu\text{m}$ (wt %)
<b>Yield</b> (proportion of head mass)	100.0	47.0	35.6	na	17.2	1.3
<b>Mineralogy</b>						
- Feldspar (Na & K)	55	>95	98-99	na	<1	~1
- Quartz	30	<5	<1	na	99	~1
- Mafics (mica & pyroxene)	5	<<1	<<1	na	<1	98
- Opaques (including magnetite)	<1	nd	nd	na	nd	nd
<b>Recovery</b> (proportion of feldspar from head in product)	100	81 feldspar	64 feldspar	na	57 quartz	35 mica **
<b>Chemistry</b>						
SiO <sub>2</sub>	67.54	67.83	68.45	66 - 69	99.35	not analysed
TiO <sub>2</sub>	0.32	0.02	0.04	na	0.01	
Al <sub>2</sub> O <sub>3</sub>	14.76	19.08	19.15	18 - 20	0.14	
Fe <sub>2</sub> O <sub>3</sub>	1.8	0.19	0.26	<0.1	0.02	
MnO	0.03	0.00	0.00	na	0.00	
MgO	0.71	0.03	0.06	<0.05	0.00	
CaO	1.95	1.59	1.72	0.5 - 2	0.03	
Na <sub>2</sub> O	4.44	6.16	6.89	5 - 7	0.05	
K <sub>2</sub> O	3.77	4.59	3.19	4 - 6	0.08	
P <sub>2</sub> O <sub>5</sub>	0.10	0.01	0.01	na	0.00	
LOI	1.33	0.52	0.48	na	0.07	
<b>Total</b>	99.2	100.02	100.25	100	99.75	

N.B. Mineralogy of head determined by examination of thin section and of products by examination of loose grains using binocular microscope. Chemistry determined by X-ray fluorescence analysis. na =Not available, nd=not detected, \* = Commercial specifications from Harben (1992), \*\* = Large proportion of mica reported to magnetic fraction.

**Table 2. Summary of processing, Thai feldspar**

	Head (wt %)	Feldspar concentrate (wt %)	Commercial ceramic-grade feldspar * (wt %)	Quartz concentrate (wt %)	Mica concentrate (wt %)
<b>Yield</b> (proportion of head mass)	100.0 (1.9)	14.6	na	21.0	10.2
<b>Mineralogy</b>					
- Feldspar	~60	~95	na	~5	~5
- Quartz	~30	~5	na	~95	~5
- Muscovite mica	~10	nd	na	nd	~90
<b>Recovery</b> (proportion of feldspar from head in product)	100	23 ** feldspar	na	67 quartz	92 mica
<b>Chemistry</b>					
SiO <sub>2</sub>	79.66	67.08	66 - 68	97.21	not analysed
TiO <sub>2</sub>	0.02	0.01	0.01	0.01	
Al <sub>2</sub> O <sub>3</sub>	11.12	17.13	18 - 19	1.56	
Fe <sub>2</sub> O <sub>3</sub>	0.44	0.09	<0.1	0.04	
MnO	0.03	0.02	na	0.01	
MgO	0.03	0.06	<0.05	0.01	
CaO	0.11	0.12	0.1 - 0.4	0.04	
Na <sub>2</sub> O	1.96	2.64	2 - 3.3	0.39	
K <sub>2</sub> O	5.63	11.05	5 - 14	0.38	
P <sub>2</sub> O <sub>5</sub>	0.05	0.08	na	0.01	
LOI	0.64	0.74	0.2-0.3	0.42	
<b>Total</b>	99.54	99.02	100	100.08	

N.B. Mineralogy determined by examination of loose grains using binocular microscope. Chemistry determined by X-ray fluorescence analysis na =not available, nd=not detected, \* = Commercial specifications from Harben (1992), \*\* = remainder of feldspar in middling products.

**Table 3. Summary of processing, Zambian kaolin**

		<b>Head</b> (wt %)	<b>Kaolinite concentrate</b> (wt %)	<b>Middling</b> (wt %)	<b>Sink</b> (wt %)
<b>30 mm underflow *</b>	<b>Yield</b>	100	73	4.3	22.7
	<b>Grade</b>	64	71	53	36
	<b>Recovery</b>	58	83	4	13
<b>1" underflow *</b>	<b>Yield</b>	100	54.1	8.6	37.3
	<b>Grade</b>	63	75	57	52
	<b>Recovery</b>	45	66	8	26
<b>1" underflow * (sandground)</b>	<b>Yield</b>	100	54.1		
	<b>Grade</b>	63	75 (rough concentrate only, no further flotation)		
	<b>Recovery</b>	45	66		
<b>-125 +63 <math>\mu</math>m sieve residue</b>	<b>Yield</b>	100	56.4	1.0	42.6
	<b>Grade</b>	20	26	24	14
	<b>Recovery</b>	3	77	2	21
<b>-250 +125 <math>\mu</math>m sieve residue</b>	<b>Yield</b>	100	38.4	1.9	59.7
	<b>Grade</b>	7	12	5	4
	<b>Recovery</b>	2	72	2	26

N.B. Yield = Proportion of head mass, Grade = Kaolinite content, Recovery = Proportion of kaolinite from head that reports to product, \* = Hydrocyclone products.

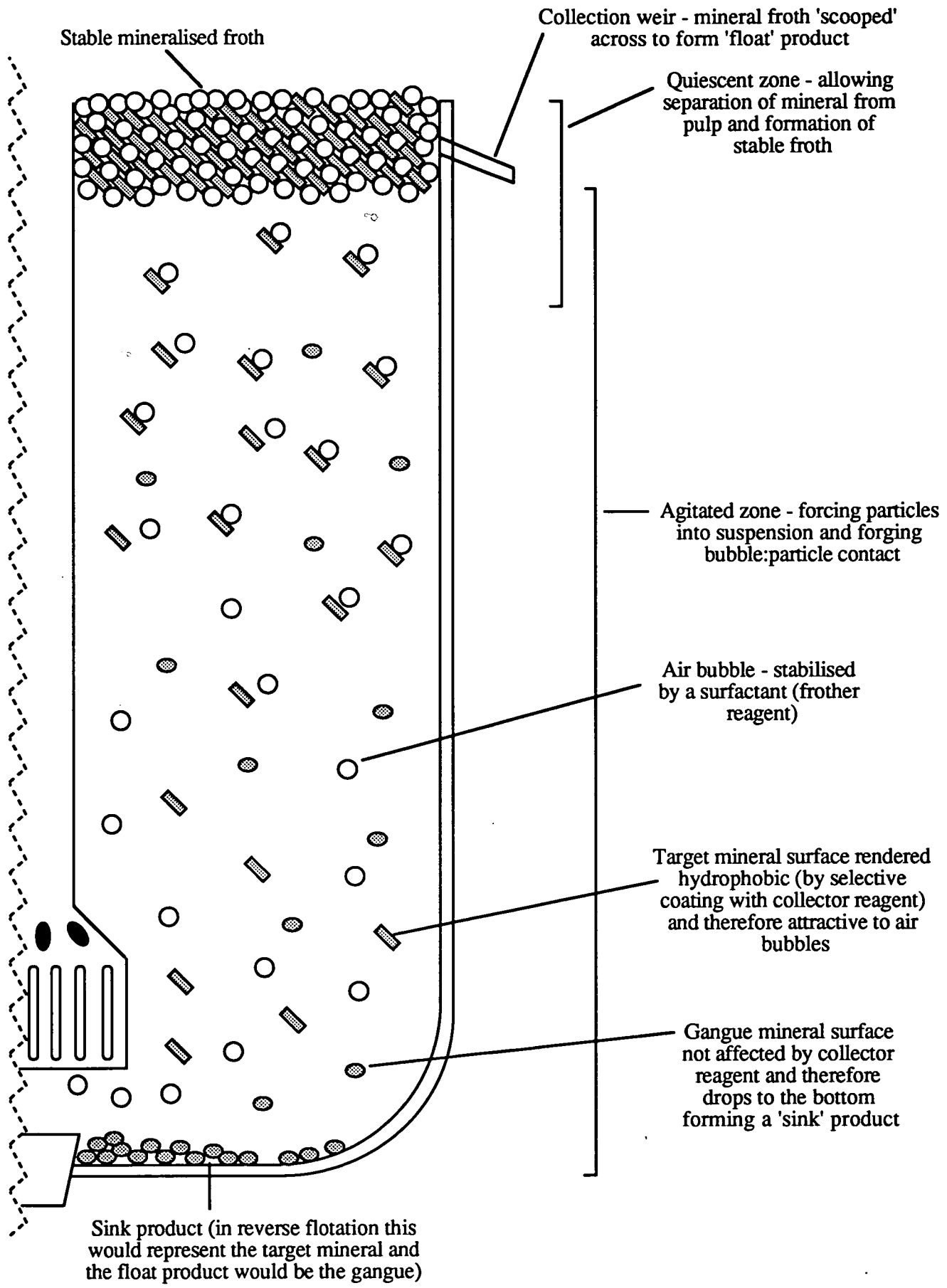


Figure 1. Cut-away diagram illustrating the theory of froth flotation



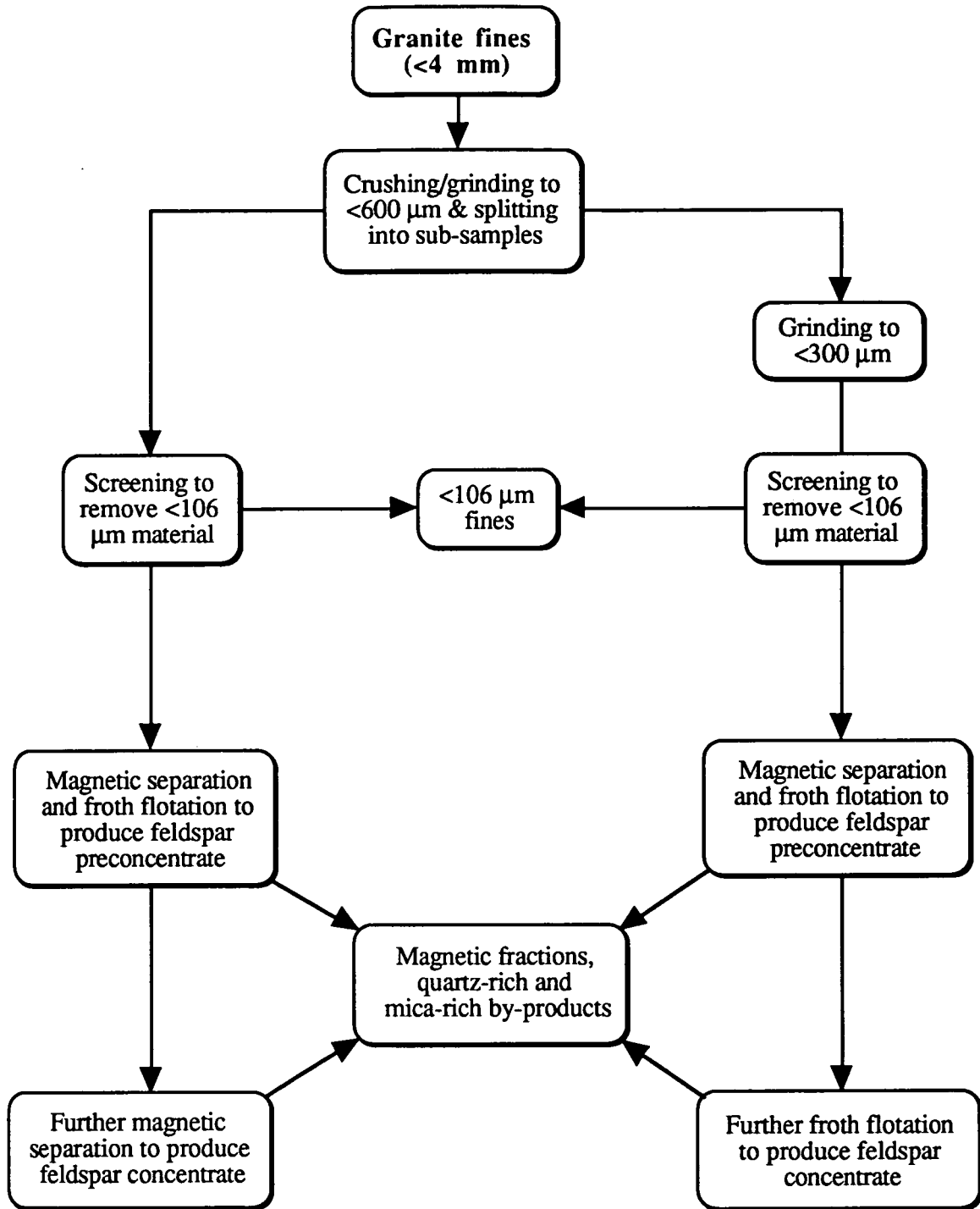


Figure 2. Flowsheet outlining the mineral processing of the Scottish granite fines

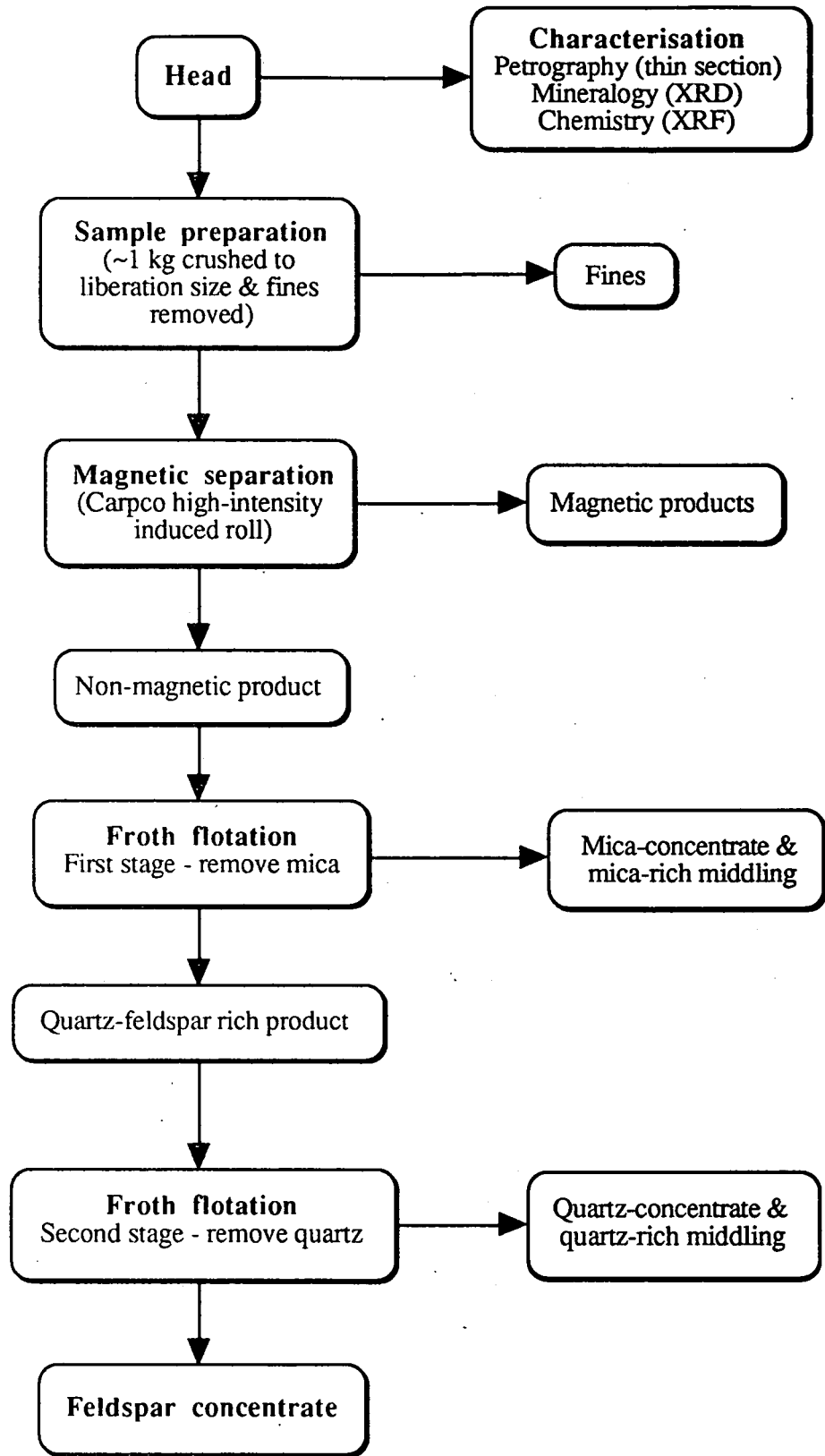


Figure 3. Flowsheet outlining mineral processing of feldspar (see Appendix A)

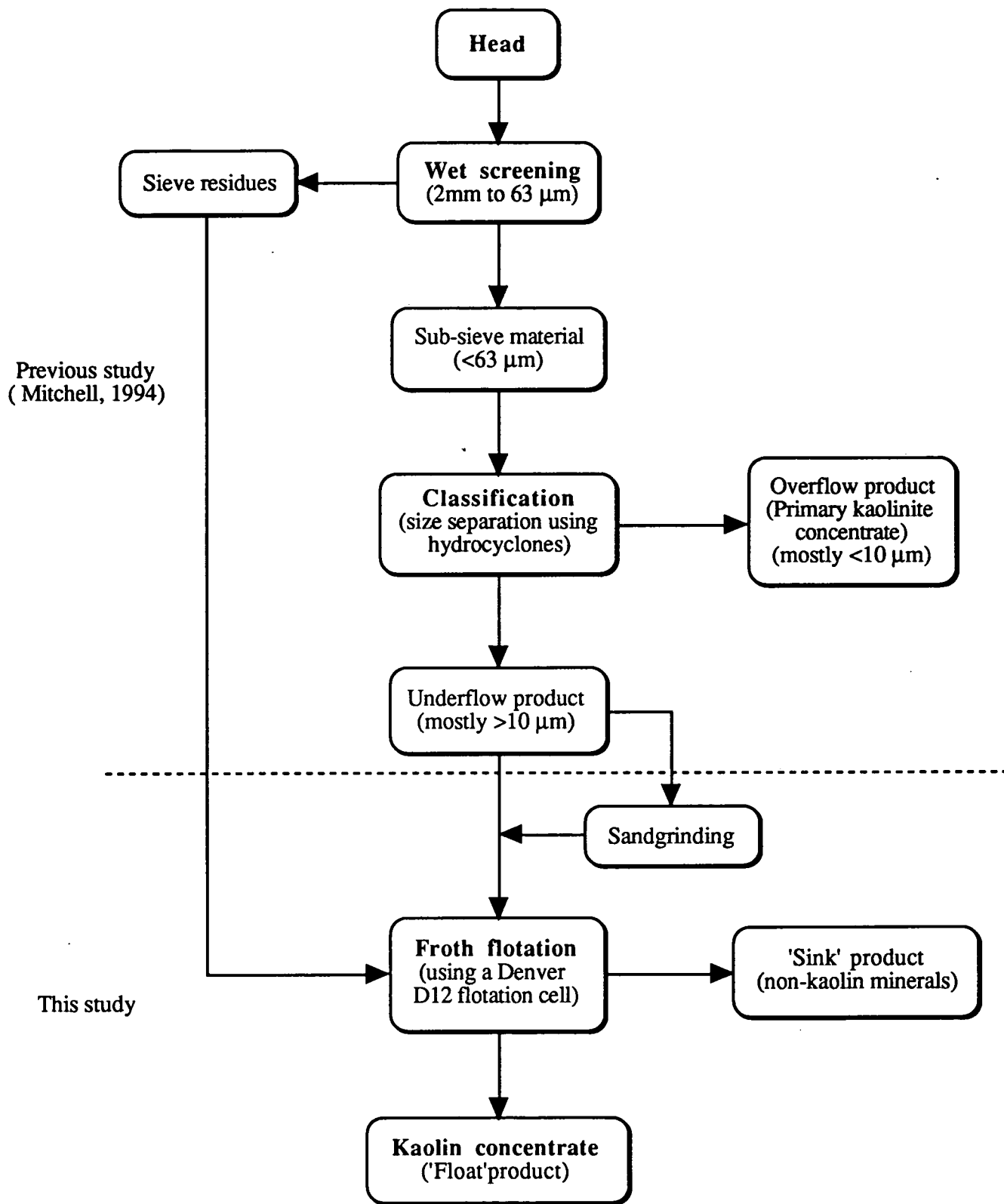


Figure 4. Flowsheet outlining mineral processing of kaolin in this and previous investigations (Mitchell, 1994)

## **APPENDIX A. Procedure for laboratory separation of feldspar**

This laboratory methodology has been developed to produce feldspar concentrates which can be evaluated against specifications for ceramic- and glass-grade material. It also enables the processing characteristics of a particular rock type to be assessed. The process route is summarised in Figure 3.

### **1. Sample characterisation**

Factors likely to affect processing properties and product quality are assessed during initial characterisation of the sample. These include mineralogy, modal proportion, size range (liberation size), inclusions and the nature of any coatings or staining. Chemistry of the feldspar may be determined at this stage by EPMA.

### **2. Sample preparation**

A representative sub-sample of approximately 1 kilogram is crushed (jaw crushing, roller milling and/or cone grinding) down to the chosen liberation size. For glass-grade material a preferred upper-size between 850 and 600  $\mu\text{m}$ ; for ceramic-grade material 75  $\mu\text{m}$  (although for the latter a coarser upper size could be used, with milling to  $<75 \mu\text{m}$  after processing). The crushed material is then processed to remove fines (for glass-grade material this entails removing sub-100  $\mu\text{m}$  material by wet screening; for ceramic-grade material the sub-10  $\mu\text{m}$  material should be removed by air classification). Retain a sub-sample of the prepared feldspar as representative of the mineral processing feed material (head).

**N.B.** If coatings are present then attrition scrub the feed material (2 hours at approximately 250 rpm using a high-torque stirrer) and process to remove any fines generated.

### **3. Magnetic separation**

Magnetically susceptible minerals are removed by processing with a Carpco high-intensity induced-roll magnetic separator.

3.1. The feed material is fed (25% maximum feed hopper vibration) over the drum (set at 180 rpm) at an electromagnet current of 0.1 A (1400 Gauss). The magnetic product is retained.

3.2. The non-magnetic product is reprocessed by repeating step 3.1. with an incremental increase in electromagnet current (1.0 A = 11000 Gauss, 2.0 A = 14600 Gauss and 3.45 A = 15700 Gauss).

3.3. The final non-magnetic product is used as the feed for froth flotation. A sub-sample is retained as representative of the non-magnetic product.

#### 4. Froth flotation

Mica and quartz are removed from the feldspar by two stage flotation using a Denver D12 Laboratory flotation machine. A maximum of 500 grams of sample should be used for any single flotation trial. Ideally, the sample should be composed of particles between 100 and 300  $\mu\text{m}$  in size (an upper limit of 500  $\mu\text{m}$  and a lower limit of 10  $\mu\text{m}$  are achievable).

##### 4.1. Mica removal

4.1.1. The feed material is placed into a glass cell with 1 litre of deionised water (ideally a solids concentration of 50 to 60% is required). The pH is adjusted to 2.5 to 2.7 using weak solutions of sulphuric acid and sodium hydroxide. Add approximately 20 drops of alkylamine acetate ('Armoflote 64' from Akzo Nobel) as a collector. The mineral suspension (pulp) is stirred (conditioned) for 4 minutes (the speed at which the pulp should be stirred, and subsequently floated, depends on the particle size of the feed; 1000 to 1200 rpm for material  $<100 \mu\text{m}$  and 1200 to 1500 rpm for material  $>100 \mu\text{m}$ ).

**N.B.** During conditioning the water level should be high enough to ensure that all of the sample is in suspension. Ideally a solids content of 50 to 60% is required however it is important that all grains are available for coating with collector reagent. Therefore more deionised water should be added to ensure complete sample suspension.

4.1.2. Add more deionised water to the cell to bring the level of the pulp to approx. 1 to 2 cm below the weir height (ideally this should be 20 to 30% solids concentration). Adjust the pH to 3 to 3.3. Add 1 to 2 drops of methyl isobutyl carbinol (MIBC) as a frother.

4.1.3. Commence flotation by reducing the stirrer speed to 1000 rpm and introducing air into the stirred pulp. Allow the froth to develop, this should be a stable layer of bubbles with a 'firm' looking appearance. (In some instances the bubbles will not be stable, bursting upon contact with the air above. This is indicative of a lack of collector reagent. If this occurs add more collector and recondition the pulp. At this stage it is not important to readjust the pH back to 2.5 to 2.7, but it is important to adjust the pH to 3 to 3.3 prior to flotation). Start collecting mineralised froth by scooping it across the weir (taking care not to remove any of the pulp at the same time). Continue collecting froth until no more is generated from the pulp. In extreme circumstances frothing may become excessive. If this occurs add heavy distillate (1 drop) to the pulp to control the frothing. However this may have the effect of eliminating any further flotation response, i.e. no more mica will float.

- 4.1.4. Add more collector (and more frother if required) and repeat conditioning/flotation.
- 4.1.5. Repeat step 4.1.4. until no more mineral is recovered from the froth (the pulp may become murky/dark coloured).
- 4.1.6. Dewater the pulp (glass-grade material should be screened using a 63  $\mu\text{m}$  sieve; ceramic-grade material should be dewatered using a Buchner funnel or a filter press). Wash the sink material in hot water and detergent ('Teepol') to remove the flotation reagents. Thoroughly wash the sink product using deionised water to remove detergent residues. Removal of flotation reagents from the products may require repeated washing. However washing is crucial as any residue of reagent remaining in the sample will adversely affect further flotation response.
- 4.1.7. Repeat steps 4.1.1. to 4.1.6. until all the mica is removed from the sample. Thoroughly wash all the float products in hot water and detergent prior to drying. Examine and combine appropriate float products into a mica concentrate and a mica-rich middling.
- 4.1.8. The sink product is used as the feed material for the second stage of flotation (4.2.). Retain a sub-sample of this as representative of the mica flotation sink product.

#### 4.2. Quartz-feldspar separation

- 4.2.1. The flotation was carried out as for mica, with the following exceptions: The pH is adjusted to 1.8 to 2.0. Add approximately 20 drops of fatty acid derivative: Butan-2-ol ('Armoflote 543' from Akzo Nobel) as a collector. The pulp is conditioned for 4 minutes.
- 4.2.2. Add more deionised water to the cell to bring the level of the pulp to approx. 1 to 2 cm below the weir height (ideally this should be 20 to 30% solids concentration). Add 1 to 2 drops of MIBC as a frother. **N.B.** No pH modification is required after conditioning.
- 4.2.3. Carry out steps 4.1.3 to 4.1.6. as for the mica removal procedure. The pH should not be adjusted.
- 4.2.4. Repeat steps 4.2.1. to 4.2.3. until all the feldspar is removed from the sample. Thoroughly wash all the float products in hot water and detergent prior to drying. Examine and combine appropriate float products into a feldspar concentrate and a quartz-rich middling. The sink product is the quartz concentrate.

## **5. Further processing**

To remove any remaining impurities (such as feldspar with iron-bearing inclusions) further processing may be considered, including high-intensity magnetic separation (wet or dry), froth flotation (using reagents tailored to iron-bearing minerals) and chemical treatment (such as acid leaching).

## APPENDIX B. Procedure for the froth flotation of kaolin (Fig 4)

1. This procedure is designed to recover coarse kaolinite from hydrocyclone underflow products and sieve residues from the wet processing of kaolin (Bloodworth *et al.*, 1994). Samples to be processed should ideally be composed of particles between 10 and 300  $\mu\text{m}$  in size. A maximum of 500 grams should be used per flotation trial.
2. The sample is placed into a glass cell with 1 litre of deionised water. The pH is adjusted to 2 to 3 (using weak solutions of sulphuric acid and sodium hydroxide). Add approximately 10 drops of alkylamine ('Armoflote 13' from Akzo Nobel) as a collector and condition for 4 minutes. The mineral suspension (pulp) is stirred (conditioned) for 4 minutes (using stirrer speeds of 1000 to 1200 for material  $<100 \mu\text{m}$  in particle size and 1200 to 1500 rpm for material  $>100 \mu\text{m}$  in particle size). Through out conditioning the pH should be monitored and maintained at the required range. **N.B.** During conditioning the water level should be high enough to ensure that all of the sample is in suspension. Therefore more deionised water should be added to ensure complete sample suspension.
3. Add more deionised water to the cell to bring the level of the pulp to approx. 1 to 2 cm below the weir height. No frother is required.
4. Commence flotation by reducing the stirrer speed to 1000 rpm and introducing air into the stirred pulp. Allow the froth to develop, this should be a stable layer of bubbles with a 'firm' looking appearance. (In some instances the bubbles will not be stable, bursting upon contact with the air above. This is indicative of a lack of collector reagent. If this occurs add more collector and recondition the pulp). Start collecting mineralised froth by scooping it across the weir (taking care not to remove any of the pulp at the same time). Continue collecting froth until no more is generated from the pulp. In extreme circumstances frothing may become excessive. If this occurs add heavy distillate (1 drop ) to the pulp to control the frothing. However this may have the effect of eliminating any further flotation response, i.e. no more kaolin will float.
5. Allow the pulp to settle and decant the clear supernatant. The sink product is dried and weighed.
6. Dry the float product (retain a sub-sample for assay) and record its weigh. This is the rougher concentrate and it is the feed for the second flotation stage.
7. Repeat steps 2 to 5. The sink product is known as the middling. The float product (final kaolin concentrate) is dried and weighed.