

The Recovery of Water from a Colloidally Stable Kimberlite Suspension

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ABSTRACT

A major diamond mining operation in South Africa is faced with the unique problem of recovering usable water from the fine tailings stream. The kimberlite ore hoisted is washed prior to crushing in order to remove adhered fine clay particles. These particles form a colloidally stable suspension from which it has not been possible to recover clear recycle water.

Research has been conducted on this problem for more than 20 years and it is only recently that a breakthrough has been achieved. This paper describes the work done to gain an understanding of the suspension stability and which subsequently led to the testing of a process at pilot-plant scale.

1. INTRODUCTION

Many kimberlite processing operations in Southern Africa incorporate scrubbing of the fresh ground prior to crushing and subsequent diamond recovery. Typically, the washings from this process are dewatered by hydrocyclones and conventional thickeners which yield clear recycle water and a slimes product which is pumped to a suitable impoundment. This is shown by a flowsheet in Figure 1.

One of the oldest operations in the De Beers group has a washing water circuit which, due to the inability of current processes to recover clear recycle water, is markedly different to that shown in Figure 1. In this situation, shown in Figure 2, the fresh ground is washed by slimes from a storage dam and the washings are subsequently de-gritted and returned to the dam. Over the years, the solids concentration in this dam has risen to a dry season high of 18% solids by mass and it is anticipated that these slimes will shortly be too viscous to provide sufficient washing of the fresh ground.

As the mine is situated in a semi-arid area where fresh water is in short supply, the viability of the operation could be jeopardised if a means of recovering usable water from the washings was not found.

2. IDENTIFYING A VIABLE PROCESS

Suspension Characteristics

Much of the early work on the characterisation of these slimes was carried out by O'Gorman and Kitchener [1]. Visible examination of the slimes showed it to be of a uniform grey colour which exhibited streaming double refraction (due to alignment of the plate-like particles) upon stirring. The particles in suspension were ultra-fine as shown by the particle size distribution curve in Figure 3. Transmission electron microscopy showed the slimes to consist of homogeneous fragmented, layer silicates with the largest of the particles being comprised of aggregates.

X-ray diffraction indicated that the solids were 50-60% smectite (swelling clay), 10-20% talc and minor amounts of chlorite, illite and quartz. The most abundant mineral appeared as saponite which is a smectite of the trioctahedral class. It consists of a brucite-like $[\text{Mg}(\text{OH})_2]$ sheet sandwiched between two siloxane sheets and is characterised by the formula $(\text{Si}_{8-x}\text{Al}_x)\text{Mg}_6\text{O}_{20}(\text{OH})_4\text{Na}_x$. This can be regarded as being derived from talc, the difference being that saponite is obtained by substitution of some Si^{4+} ions in the outer layers by Al^{3+} . The substitution leaves a net negative charge on the lattice which is neutralised by cations such as Na^+ and Ca^{2+} .

The cation exchange capacity was found to be 25 mequiv. per 100g by the standard ammonium acetate method (pH7) and the exchangeable cations (in order of decreasing proportion) were Ca, Na, Mg and K. The fixed negative charge works out to an equivalent of 1 charge unit per 7 unit cells.

The stability of the slimes is due to a strong negative electrostatic charge on the particles. Mean zeta-potentials were measured at -45mV which of course, is more than sufficient to provide colloidal stability to the suspension. This high charge is brought about by isomorphous replacement of high charge cations with those of a lower charge e.g. Si^{4+} with Al^{3+} . Mackenzie and Lovell [2] reported a decrease in zeta-potential as the ionic strength of cations such as Ca^{2+} and Mg^{2+} was increased which suggests that these cations could be specifically absorbed by the lattice. It is also reported however [1], that any true lattice uptake is too slow to affect the built-in charge and that clay minerals are not generally in reversible equilibrium with lattice ions.

Coagulation

Laboratory investigations were carried out on coagulation and subsequent gelation of the slimes in an attempt to induce syneresis (i.e. spontaneous separation of the liquid, with shrinkage of the gel). Large additions of electrolytes were found to cause gelation, and the most effective were found to be alkalis such as caustic soda and slaked lime. In both cases, additions of the order of 2g/litre were required and a worthless gel was produced.

The effects of gelation using divalent Ca and Mg cations was investigated over a range of pH values by measuring the mean zeta-potential in each case. The results, shown in Table 1, prove that the zeta-potential is reduced below the critical value of -20mV at the minimum dosage of 100ppm which is confirmed macroscopically by slow coagulation of the particles. It can also be seen that the effect of pH, over the range studied, was minimal.

Coagulant ppm		pH 8	pH 9	pH 10	pH 11
0	Ca	-41.6	-43.3	-45.3	-36.3
	Mg	-44.9	-42.6	-41.2	-38.0
100	Ca	-20.1	-19.6	-19.8	-18.4
	Mg	-19.1	-18.0	-15.2	+27.5
200	Ca	-18.8	-18.0	-15.6	-14.2
	Mg	-15.4	-16.3	+30.8	+25.0
300	Ca	-17.0	-17.0	-15.3	-12.3
	Mg	-15.9	-15.1	+32.4	+18.5
400	Ca	-16.2	-16.5	-15.0	-10.9
	Mg	-14.2	-13.8	+26.2	+28.8
500	Ca	-16.6	-16.0	-14.6	-11.1
	Mg	-13.3	-12.8	+36.1	+17.3

TABLE 1 - Zeta-Potential (mV) for Kimberlite Slimes as a Function of pH and Coagulant Concentration.

Gelation of the slimes by appropriate salts was not considered to be a viable route to recovering water since syneresis was not achieved in any of the cases studied. It was therefore not possible to effect solid/liquid separation with the products of coagulation.

Flocculation

Considering that the stability of the slimes was found to be caused by a high negative surface charge, it was assumed that cationic polymers would be most effective in inducing flocculation. In practise however, it was found that the addition of cationic polymers caused the slimes to gel and that true flocculation was only achieved by using anionic polymers. This was no great surprise however, since all group diamond mines currently use anionic flocculants in this application - despite the fact that the contradictory polarity effect is not understood.

A large range of commercially available anionic polymers was tested and it was shown that none was able to produce a clear supernatant and a dense underflow - even at extremely high dosages. A two-stage process was developed [3] which did produce clear water, but the overall recovery was extremely low due to dilute underflow products. In the first stage of this process, a combination of two high charge anionic polymers was added to the slimes which, upon long standing, gave a cloudy supernatant and an underflow of approximately 15% solids by mass. This supernatant was subsequently treated with a combination of cationic and anionic polymers which clarified the water completely. Despite reasonably low cost reagents, the process was rejected on the grounds that the water recovery was low.

When a partially coagulated slime was treated with anionic polymer, it was observed that dense flocs were produced which settled quickly to leave a cloudy supernatant. Further efforts were thereafter directed towards the combination of coagulation and flocculation.

Coagulation/Flocculation

Preliminary work had already shown the effectiveness of various coagulants in reducing the zeta-potential of the solids. Tests were conducted using a combination of the promising coagulants (at various dosages) and long chain anionic polymers. The polymers used are tabulated below :

Flocculants

Brand Name	Mol.Wt	Charge
Magnafloc 611	Medium	Very high
Magnafloc 156	High	High
Magnafloc 139	Low	Low
Montan PA 103S	V.High	V.High
Montan PA 99S	V.High	High
Montan PA 97S	V.High	Medium
Montan PA 94S	V.High	Medium to low
Montan PA 91S	V.High	Low
Cyanamid A100	High	Low
Cyanamid A130	High	Medium
Cyanamid A150	High	High

TABLE 2 - Anionic Flocculants used in Coagulation/Flocculation Tests

The following coagulants were used :

Ferrous Sulphate, Ferric Chloride, Ferric Sulphate, Aluminium Sulphate, Caustic Soda, Sodium Carbonate, Lime, Calcium Hypochlorite, Sulphuric Acid, Hydrochloric Acid, Sodium Aluminate, Magnesium Sulphate.

Of the flocculants used, the best results were achieved using the Montan PA series - possibly due to the fact that these had the highest chain length of all those tested. The most effective coagulants were magnesium sulphate and lime. When coagulant was added prior to the flocculant, good flocs were formed which settled rapidly upon slow stirring. The supernatant in each case was cloudy. It was found that the coagulant would cause the slimes to gel unless the

concentration was below 10% solids by mass, so dilution with distilled water was carried out before the coagulant was added.

Although the coagulation/flocculation approach was considerably more successful than any previous methods, the question of clarifying the supernatant and dewatering the underflow still required attention.

It was suspected that an higher initial coagulant dosage would be sufficient to clarify the supernatant, but when this was done, it was found that the slimes gelled. An excess of coagulant was added to the products of the first stage and the water was found to clear but to the detriment of the settled flocs, which appeared to reduce in size - even at extremely low shear rates. A second addition of flocculant however provided a spectacular result as the smaller flocs, already present, were bonded together to form one large, spongy mass. This mass could be lifted from the beaker and squeezed by hand to release a considerable volume of water.

Of the two most promising coagulants tested, lime was selected for the process in view of its relative cheapness. The most effective anionic flocculant was Montan PA 97S - a very high molecular weight polymer with a medium charge (approx 50%).

The final process is summarised for slimes at 10% solids as follows:-

Stage 1	Add 125g/litre lime (slaked at approx. 40g/litre)
Stage 2	Add 0.018g/litre PA 97S flocculant (as 0.025% solution)
Stage 3	Add 125g/litre lime as before
Stage 4	Add 0.009g/litre PA 97S flocculant (as 0.025% solution)

TABLE 3 : Coagulation/Flocculation Process for Clarifying the Slimes

Many variants of the above process were tried in an attempt to reduce it to two stages. This was not found to be possible however, since a two stage lime addition was necessary to avoid gelation and a two stage flocculant addition was necessary to provide an expressible floc for dewatering.

The reagent costs of this process, calculated from bench-scale results, was R0.25 per m³ clear water recovered.

3. TESTING THE PROCESS

Continuous Dewatering Tests

Having identified a low-cost process, it was decided to examine various process equipment options in the laboratory prior to pilot-scale tests. Until now, the clarification process had only been carried out in beakers and measuring cylinders - the question of how to dewater the flocs to maximise recovery remained unanswered.

As stated earlier, the flocs could be described as large and gelatinous but with very little shear strength. Considering the nature of these flocs, the following unit process options were considered :

- Gravity settling, thickening
- Stirred vessel thickening (sludge compaction)
- Vacuum and pressure filtration
- Expression filtration
- Dewatering screens
- Centrifugation

Flocculation of the slimes in a measuring cylinder did not produce a rapid-settling mudline due to the size and voluminous nature of the flocs. A semi-batch test was carried out in a laboratory deep cone settler. This failed to produce a thickened underflow since the density of the flocs was insufficient to provide significant compression forces in the cone section of the unit. Gentle stirring in this zone did improve the underflow concentration considerably however, and for this reason pilot-scale work was carried out on the mine using a Neyertec Tasster Sludge Compactor.

The flowsheet for the sludge compaction pilot-scale unit is shown in Figure 4.

Feed to the plant was supplied via a T-piece in one of the dam return water lines to a variable speed positive displacement pump, thereby providing a controllable and constant feed rate. Two injection points were placed in this line prior to the feed pump. The first point was used for the addition of plant water, using a variable speed pump to dilute the incoming feed. The second dosing point was used to add the primary lime. The distance from the dosing points to the feed pump was ± 5 m, thereby allowing adequate mixing.

Primary flocculant was added at the feed pump outlet, from where the slurry passed into a small mixing box at which point the secondary lime was added before passing into a 3000 l tank equipped with a low shear gate impeller mixer. The residence time in this tank was ± 65 minutes. From the large mixing tank, the slurry was gravity fed (thereby

minimising shear) to the Tasster, with the secondary flocculant being added in this line \pm 1m before the discharge.

The Tasster unit consisted of a deep conical bottomed tank, \pm 1.2 m in diameter and \pm 3 m deep, in which slow moving (0,75 rpm) perforated rakes were used to compact the sludge, the latter being removed from the bottom via a variable speed positive displacement pump. Clarified water gravitated from the overflow to a sump.

Although performed on a relatively small scale ($2\text{m}^3/\text{hour}$), the tests showed that the unit was capable of dewatering the slimes to produce an underflow of 31% solids by mass.

Laboratory centrifugation proved that a thickened cake of up to 50% solids by mass could be produced by this method. One of the unknowns however, is the amount of shear the flocculated feed must endure as it enters a continuous machine and is accelerated to the speed of the bowl. A KHD S2-1 solid bowl decanter centrifuge was installed at the mine adjacent to the Tasster plant and tested in the configuration as shown in Figure 5. The machine was tested in two ways - the first being the centrifugation of the underflow produced by the Tasster, and the second was the centrifugation of the flocculated slimes without any prior thickening.

Both centrifuge operating scenarios failed. The shear forces imparted on the feed material in both cases were too great for the flocs to withstand. Furthermore, as the torque on the centrifuge scroll was very great, it was suspected that the machine was experiencing difficulty in discharging the cake owing to its sticky nature.

Vacuum and pressure filtration of the flocculated slimes were examined using the apparatus shown in Figure 6. An example of the typical results is shown in Table 4.

Test	Pressure	Cake % solids by mass	Specific Resistance $\text{cm/g} * 10^{12}$	Filtrate Volume cm^3	Comments
1	340mm Hg	58	3.26	23.2	Vacuum unit
2	340mm Hg	57	2.90	23.5	Vacuum unit
3	340mm Hg	57	3.19	24.0	Vacuum unit
4	650 kPa	65	4.49	15.2	Pressure unit
5	800 kPa	58	5.11	15.0	Pressure unit

TABLE 4 : Typical Results from Pressure and Vacuum Filtration of Flocculated Slimes over a 30 minute period

The results above clearly demonstrate that the filtration of these slimes as a dewatering option would not be viable due to the high specific resistance values obtained. It is interesting to note that when the driving force is increased, as in Test 5, the specific resistance also increases. This is because under increased forces, a highly resistant coating forms on the filtration membrane due to alignment of the clay platelets.

For air to enter a filter cake (thus replacing expelled water), the strength of the cake must exceed the capillary suction pressure of the water in the pores. For 1 micron particles, the capillary pressure is about 140 kPa which is apparently more than the flocculated network can withstand. For these reasons, filtration was rejected as a dewatering possibility.

It was reported during bench-scale flocculation work, that the flocs could be removed from the beaker and squeezed gently by hand in order to expel considerable amounts of water. Various woven cloths were obtained from expression press manufacturers and simple laboratory expression tests showed that it this could be a promising route to obtaining high concentration sludges.

An Eimco Expressor unit as shown schematically in Figure 7, was obtained for pilot-scale tests. Flocculated material was placed on the feed section of the unit from where it passed through 4 pre-drainage roll where the pressure profile gradually increased. The material then entered the high pressure zone where pressures up to 500 psi (35.2 kg/cm²) were exerted. Following this, the cake was discharged from the machine via discharge rolls. Filtrate was collected on an underpan which was situated below the pre-drainage and high pressure zones.

The results from the expression tests were promising and plans are currently being made to test a full-scale unit on the mine.

Simple laboratory tests showed that the flocculated slimes had sufficient strength to withstand the shear forces experienced when they were introduced to a trommel screen. A schematic of the trommel apparatus is shown in Figure 8. Trommel meshes varying from 5.0 to 0.5 mm were tested and it was shown that the larger sizes fared best since the surface tension of the clarified water was too high to allow liquid passage with the -1.0 mm meshes. The discharge from the trommel screen contained approximately 20% solids by mass which was considered to be too low to allow further work on this concept. However, it should be noted that this could be an attractive dewatering method should the floc conditions favour sufficient water release under these conditions.

1. SPECIFYING A PROCESS PLANT

At this stage of writing, the two most promising dewatering options are Sludge Compaction and Expression - the former having been tested on sufficiently large scale to allow full scale-up. Once expression has been tested on a similar scale, it will be possible to specify a plant by December 1988.

Flowsheet Considerations

The washing water reticulation flowsheet (Figure 2) was examined to determine how a clarification step should be incorporated into the operation. As the dam contains already 60 million megalitres of slimes, it was considered uneconomic to continue drawing water from this source and simultaneously return clarified water. Various options were modelled and it was shown that the cost of treatment would increase by 100% during the wet season owing to classification within the dam which caused the particle d_{50} to reduce from 5.2 microns to 2.5 microns.

The isolated washing water circuit, as shown in Figure 8, emerged as the most promising option for incorporation into the current operation. The advantages of this option are that scrubbing efficiencies would be improved and that the properties of the slimes would not change seasonally. The plant design therefore, would adopt this option as a basis.

Disposal Considerations

The final disposal of the fine tailing sludge deserves some mention here. As the existing operation comprises the pumping of the scrubber washings to the dam, there is no fine tailings impoundment (in the usual form) on the mine. The design of a suitable tailings transport and impoundment system depends obviously on the physical characteristics of the final sludge. This, in turn, would not be known until full-scale tests are carried out using the expression press.

The transport method for the final sludge will depend likewise on the properties of the product from the selected dewatering method. The underflow from the Tasster Sludge Compactor was shown to be pumpable over short distances using a Mono-type pump, but no pressure loss measurements were made which would facilitate scale-up. Since it is likely that the tailings impoundment would be situated a considerable distance from the treatment plant, the method and cost of transporting the tailings would be an important factor in selecting a site for the clarification plant.

The transport and tailings impoundment considerations will be examined in more detail once the final plant configuration and properties of the sludge are known.

Water Quality

An analysis of the clarified water is shown below in Table 5.

TEST	TA2	TA3	TA4	TA5
pH	12.2	12.0	12.0	11.9
Total dissolved solids (TDS) ppm	1397	1019	1160	887
Total alkalinity as ppm CaCO ₃	1250	600	820	580
Total calcium as ppm CaCO ₃	140	60	70	70
Langelier Stability Index at 25 C	1.0	2.6	2.2	2.6

TABLE 5 : Analysis of Clarified Water using the Coagulation/Flocculation Technique

It can be seen from Table 5 that the clarified water is extremely hard and that the possibilities of equipment scaling are extremely high. The use of anti-scalants is under consideration at present and it is hoped that this will not add significantly to the overall cost of the process. It is hoped that, during full-scale operation of a clarification plant, the residual calcium in the water will assist in the reduction of zeta-potential thus allowing a reduction in lime dosage. The scaling nature of the clarified water will be monitored during the evaluation of the pilot plant.

5. DISCUSSION

This work has demonstrated the importance of applying mineralogy and surface chemistry fundamentals to the solution of a severe solid/liquid separation problem. Although a solution was identified as early as 1972 [1], it was considered to be cost-prohibitive because magnesium sulphate was selected as the coagulant and the flocculant dosage required was high. With the introduction of extremely high molecular weight polymers in recent years, the dosage required in this process is far less than that recommended in 1972 using shorter-chain polymers.

The evaluation of unit process equipment is still ongoing. The ultimate sludge product desired is one which is transportable by belt-conveyors, but considering the water retention properties of such fine clays (particularly when flocculated), it appears unlikely that this will be achieved.

The impoundment of the final sludge will require careful study once the properties of the material are known. It is proposed, in collaboration with civil engineering consultants, to construct test impoundments during the evaluation of a large scale pilot-plant.

It is anticipated that the required design parameters for a pilot-plant will be available by December 1988 and that a decision to adopt the isolated scrubber water circuit can be made in the following year.

6. CONCLUSION

A viable dewatering process was developed for a hitherto non-settling, fine tailings stream from a kimberlite diamond mine providing the opportunity for recycling water and saving what is becoming a valuable and potentially scarce resource.

7. ACKNOWLEDGEMENTS

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REFERENCES

1. O'Gorman, J.V. and Kitchener, J.A. The Flocculation and Dewatering of Kimberlite Clay Slimes. Int. Journal Min. Proc., 1 (1974) 33-49.
2. Mackenzie, J.M.W. and Lovell, V.M. The Coagulation and Flocculation of Suspensions of Kimberlite. Report 1403 (Restricted), National Institute of Metallurgy, Johannesburg, South Africa.
3. Batson, R.G. Progress Reports "Treatment of Slimes from No.7 Dam". November 1986 - November 1987. Water Renovation (Pty) Ltd.

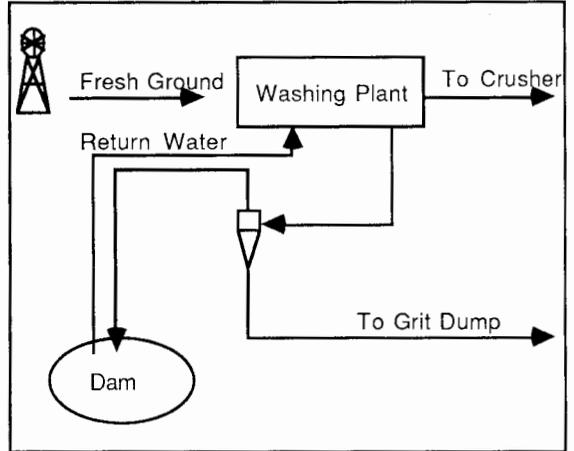
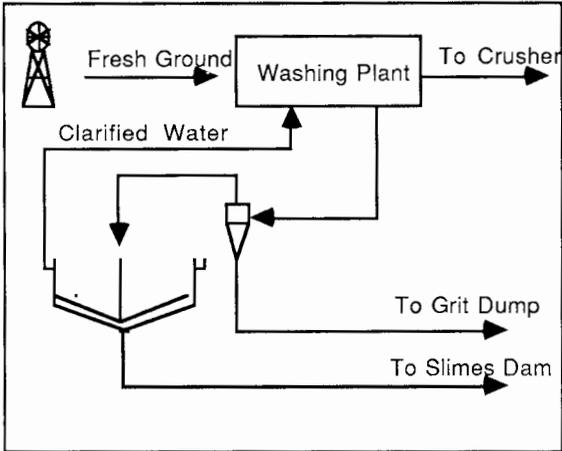


Figure 1 : Typical Wash Water Reticulation

Figure 2 : Reticulation for Non-Settling Slimes.

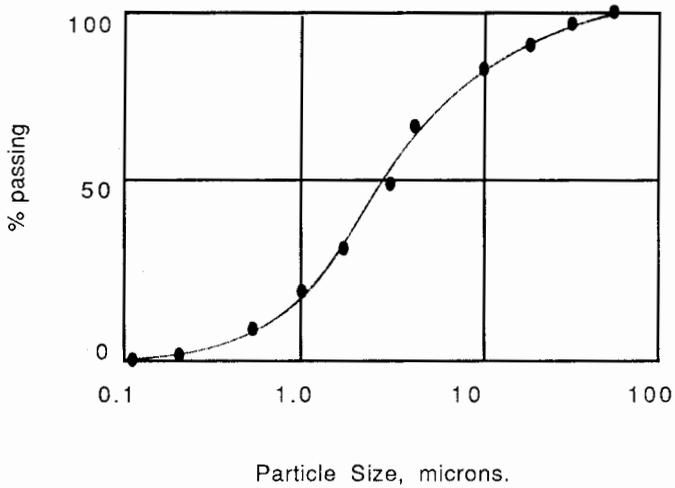


Figure 3 : Particle Size Distribution of Non-Settling Slimes

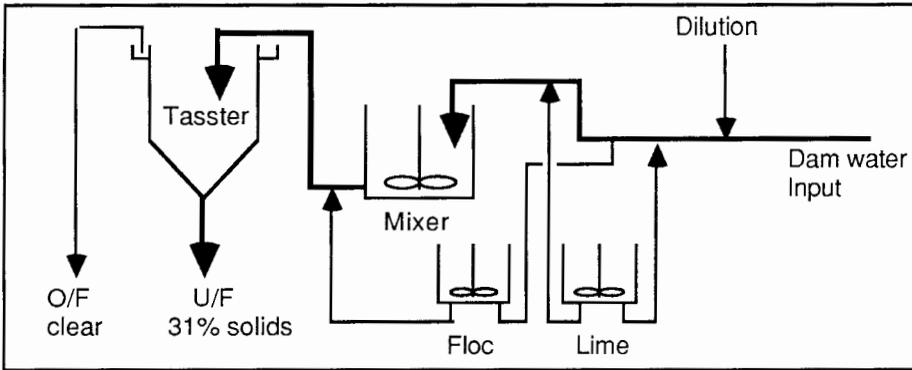


Figure 4 : Tasster Sludge Compactor Pilot Plant

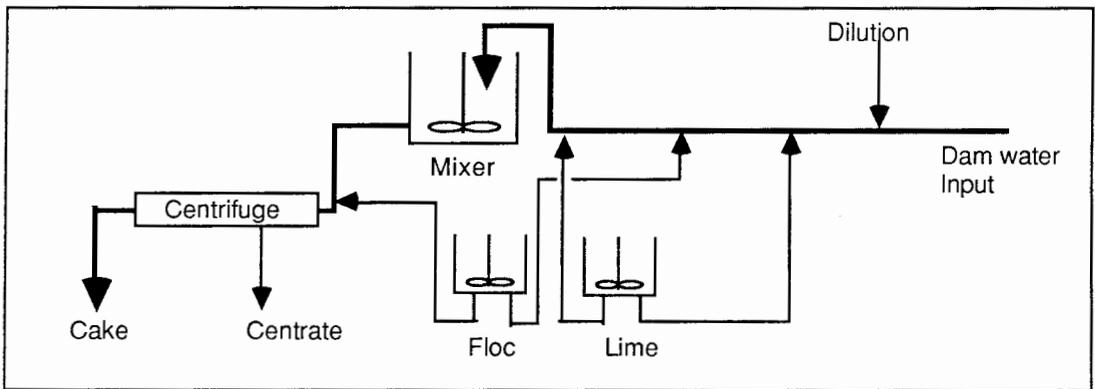


Figure 5 : Centrifuge Pilot Plant Layout

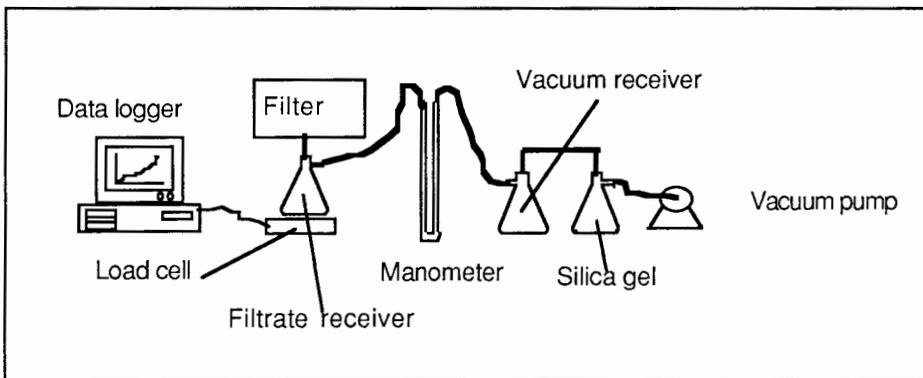


Figure 6 : Laboratory Filtration Apparatus

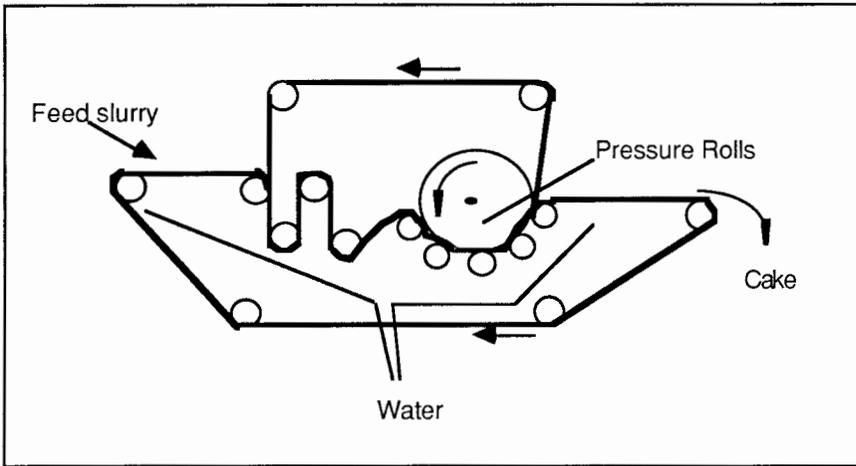


Figure 7 : Schematic of Expression Press used in Testwork

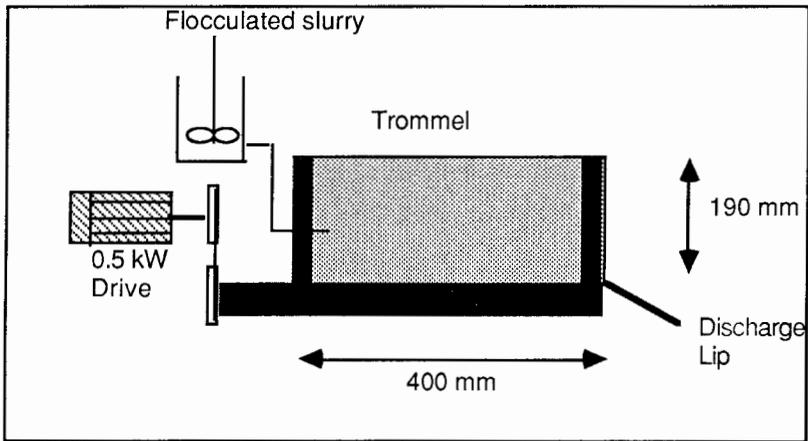


Figure 8 : Dewatering Trommel used in Testwork

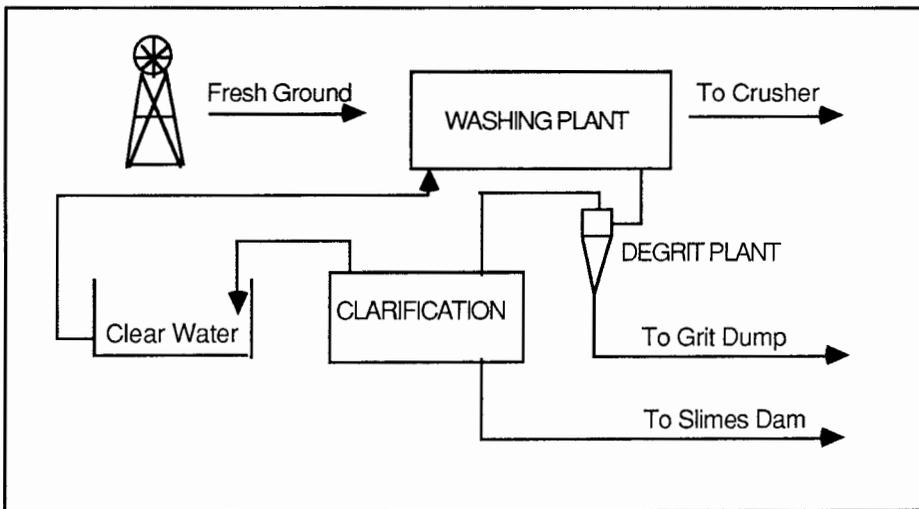


Figure 9 : Isolated Wash Water Circuit