

NOVEL TECHNIQUE FOR THE SEPARATION OF DOLOMITE FROM PHOSPHATE
ROCK

By

ABBIN ANTONY

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To all who contributed and guided me in the accomplishment of this research work

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LIST OF ABBREVIATIONS

C	Continuous Flow
LF	Line Feed
M	Mixing
P	Pulsating Flow
PF	Point Feed
PVA	Poly Vinyl Alcohol
RF	Reactive Flotation
SC	Spray Coating

Abstract of Thesis Presented to the Graduate School
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By

Abbin Antony

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Co-chair: Hassan El-Shall
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Phosphate is a major ingredient in the manufacture of fertilizers. United States is the second largest phosphate producer and 85% of the total domestic output is from Florida and North Carolina. Depletion of phosphate resources is predicted in the next 100 years. Dolomite is recognized as a major impurity in the ore. Even though several techniques have been suggested by researchers, they have been found to have low separation efficiency in terms of grade and recovery or high process operation cost.

Elaborate research by Ayman A. El-Midany under the guidance of Professor Hassan El-Shall at University of Florida led to discovery and development of a technique to separate dolomite from phosphate employing the property that carbonate rocks reacts with acid and nucleates CO₂ gas. Coating the particles with poly vinyl alcohol (PVA) enables retaining the CO₂ gas generated and enables flotation of dolomite (carbonate) particles. Beaker tests revealed significant separation and recovery.

The work presented here involves the various stages in the translation of the beaker process into a continuous process. It involves analyzing equipment designs

proposed. Development of each design is carried out based on observations in the preceding design starting with the beaker test. Once a design is confirmed, various parameters influencing the performance are also investigated. Designs analyzed in order are :

- i. Column
- ii. Screw Pipe
- iii. Vibrating Sluice

The feed preparation and analysis techniques employed are consistent with those used by Ayman A. El-Midany for his research work.

CHAPTER 1 LITERATURE REVIEW

Introduction

Phosphate is an inorganic chemical and a salt of phosphoric acid. Elemental phosphorous was discovered accidentally in the pursuit to make gold by a German chemist in 1669. Phosphate even though has many applications, as in treatment of potable water and cleaning solutions, it holds greater recognition in the agricultural industry because of its role in fertilizers and animal feed supplements. About 90% of the phosphate rock output in the world is used for fertilizer production.

The United States is recognized as the second largest phosphate producer. Florida and North Carolina account for more than 85 percent of the total domestic output.

The phosphate industry in Florida is suffering a depletion of phosphate rich ore. Based on consumption rate in 2007 the supply of phosphate ore was estimated to run out in 345 years worldwide. However scientists claim that a peak in consumption would occur in 30 years and the reserves will be depleted in 50 to 100 years. Based on the present mining rate, reserves in Central Florida will last for the next 10 years and mining would move further south and southeast where the matrix is leaner in grade and higher in dolomite.

Dolomite (Ca, Mg)CO₃ is identified as a highly taxing impurity in the ore. The proportion of carbonate present is directly proportional to the consumption of sulfuric acid in fertilizer manufacture. Furthermore MgO forms a gel (increases viscosity) and reduces filtration capacity. Hence MgO content of dolomite is an important index in evaluating the quality of phosphate concentrate. Acidulation of phosphate rock requires feed of MgO content less than 1% (by mass).

Phosphate rock is observed to exist as alternate layers of high and low grade. Unavailability of economically feasible dolomite separation techniques has led to mining techniques that bypass high dolomitic zones. This results in wastage of 50% of the phosphate resource and furthermore discarding dolomite pebbles leads to wastage of about 13%.

Separation Techniques

The phosphate industry is interested in a process that is efficient (high removal and recovery), ensures economical feasibility (low operation cost) and is environmentally sound (low toxicity). In the recent decade several separation techniques have been proposed by researchers for the separation of dolomite from phosphate rock [El-Shall et al., 2004].

It is worthy to mention in advance that all of the enlisted processes achieved removal by significantly sacrificing recovery.

I.M.C.F Process

The I.M.C.F process [Snow, 1979; 1982; Lawver and Snow, 1980] (Figure 1-1), is a cationic process for silica-free ores. High MgO content pebbles are initially cycled through rod mills and screens (24 -35 Mesh) (0.707 - 0.420 mm). The screened particles are de-slimed (150 Mesh) (0.105 mm) followed by flotation of silica from the stream using an amine condensate. The dolomite separation feed is dewatered, conditioned (20-30 seconds) at a slightly acid pH with tallow amine acetate and diesel fuel and is then subjected to rougher, cleaner and re-cleaner flotation of dolomite from phosphate.

Resulting phosphate concentrate contained ~ 31% P₂O₅, < 1% MgO and ~ 3% insolubles. Overall P₂O₅ recovery of the process is estimated to be 55 - 60%.

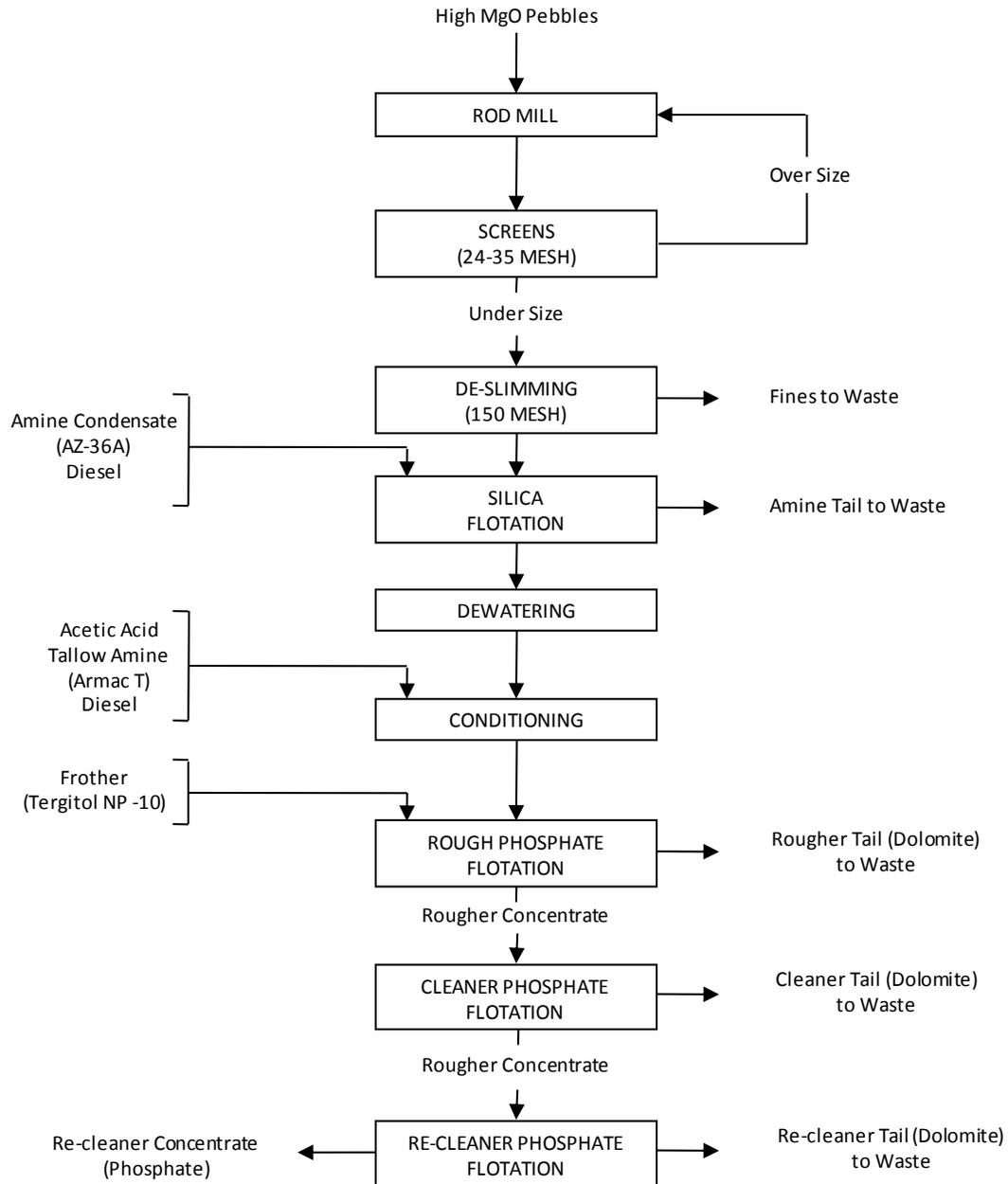


Figure 1-1. Flow sheet of IMCF Process

U.S.B.M Process

In the U.S.B.M (United States Bureau of Mines) process [Davis et al., 1984] (Figure 1-2), high MgO pebbles are cycled through hammer mills and screens (28 Mesh) (0.595 mm). The screened particles are de-slimed (150 Mesh) (0.105 mm) and scrubbed with NaOH (1 lb/ton) for 20 minutes and the stream is again de-slimed (150

Mesh) (0.105 mm). This is followed by conditioning of the stream for 5 minutes with NaOH, oleic acid and fuel oil at pH 9 – 9.2. The stream is subjected to rougher and cleaner flotation to float silica and dolomite from phosphate.

Resulting phosphate concentrate contained ~ 29% P₂O₅, < 1.66% MgO and 5.2% insolubles. Overall recovery of the process is estimated to be around 53%.

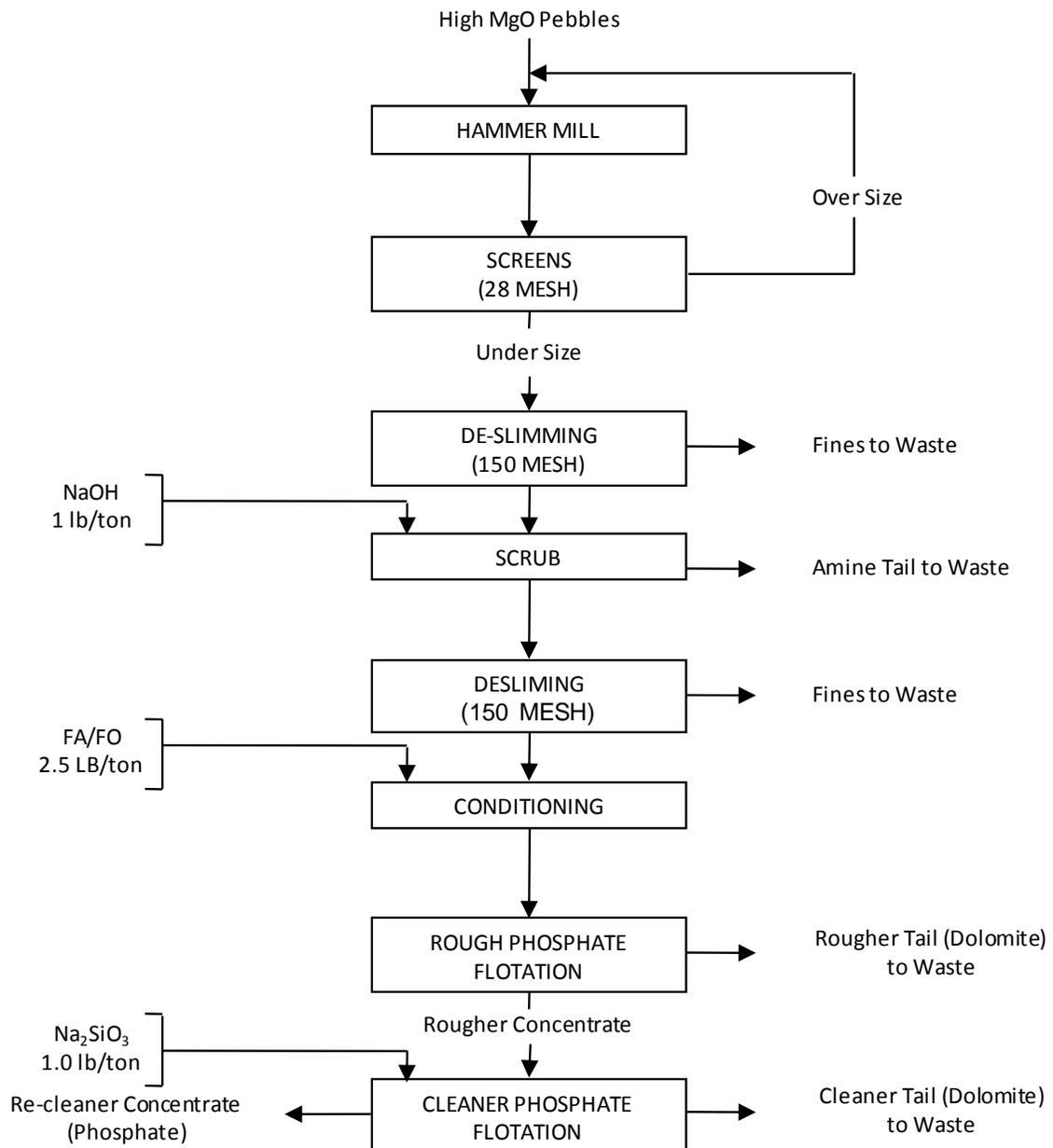


Figure 1-2. Flow sheet of USBM Process

U.F Process

This is a two stage condition process [Moudgil, 1988] (Figure 1-3) where high MgO pebbles are cycled through rod mills and screens (65 Mesh) (0.210 mm). The screened particles are de-slimed (150 Mesh) (0.105 mm) and conditioned (2.5 minutes) at a pH 10. The concentrate is reconditioned (30 seconds) with sulfuric acid (maintains pH 4-5). This is followed by flotation to remove dolomite. Final processing involves cationic flotation of the fine silica from the dewatered dolomite flotation cell.

Resulting phosphate concentrate contained ~ 28% P_2O_5 , ~ 1% MgO and 2.9% insolubles. Overall recovery of the process is estimated to be around 35.7%.

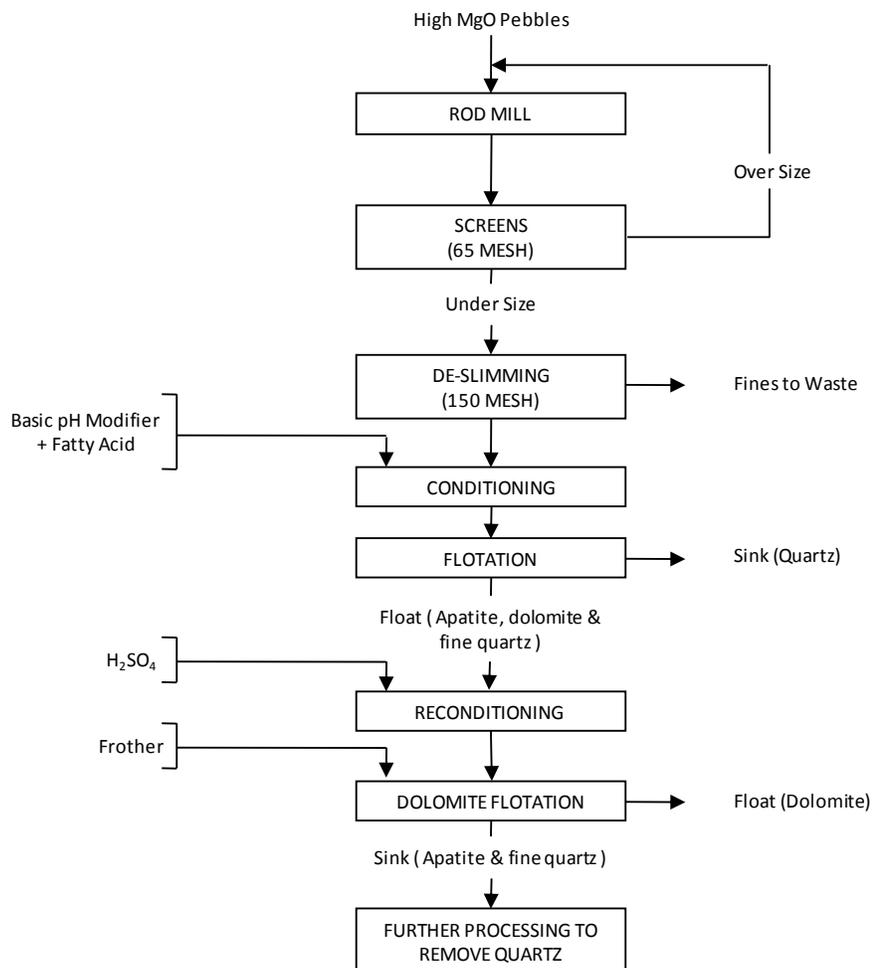


Figure 1-3. Flow sheet of UF Process

U.A Process

In the U.A Process [Hanna and Anazia 1990] (Figure 1-4), high MgO pebbles are cycled through rod mills and screens (35 Mesh) (0.420 mm). The particles are de-slimed (150 Mesh) (0.105 mm), scrubbed for 10 minutes and de-slimed again (325 Mesh) (0.044 mm). The stream is now subjected to conditioning at a pH 5.5 – 6 with fatty acid, sulfuric acid, pine oil and NaOH followed by flotation. The underflow is further conditioned with sodium silicate and fatty acid and exposed to flotation. The results were observed to be inconsistent and were reported [El-Shall et al, 1994].

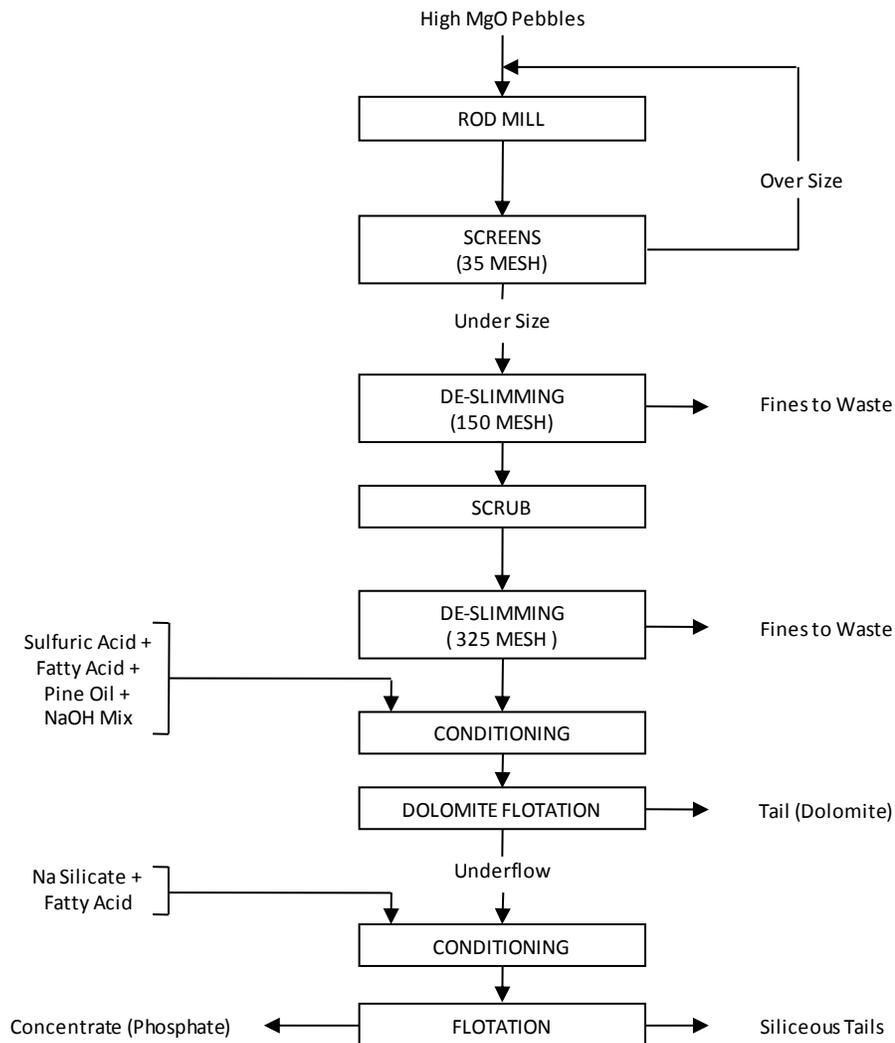


Figure 1-4. Flow sheet of UA Process

T.V.A Process

In the T.V.A process [Hsieh and Lehr, 1985] (Figure 1-5), high MgO pebbles are cycled through ball mills and screens (48 Mesh) (0.297 mm). The particles are de-slimered at 400 Mesh (0.037 mm). The stream is scrubbed for 5 minutes and de-slimered again at 400 Mesh. This is followed by flotation with di-phosphonic acid, oleic acid and pine oil. The concentrate is exposed to silica flotation with an appropriate amine collector.

Resulting phosphate concentrate contained ~ 30% P_2O_5 , ~ 1.1% MgO and 3.2% insolubles. Overall recovery of the process is estimated to be around 64%.

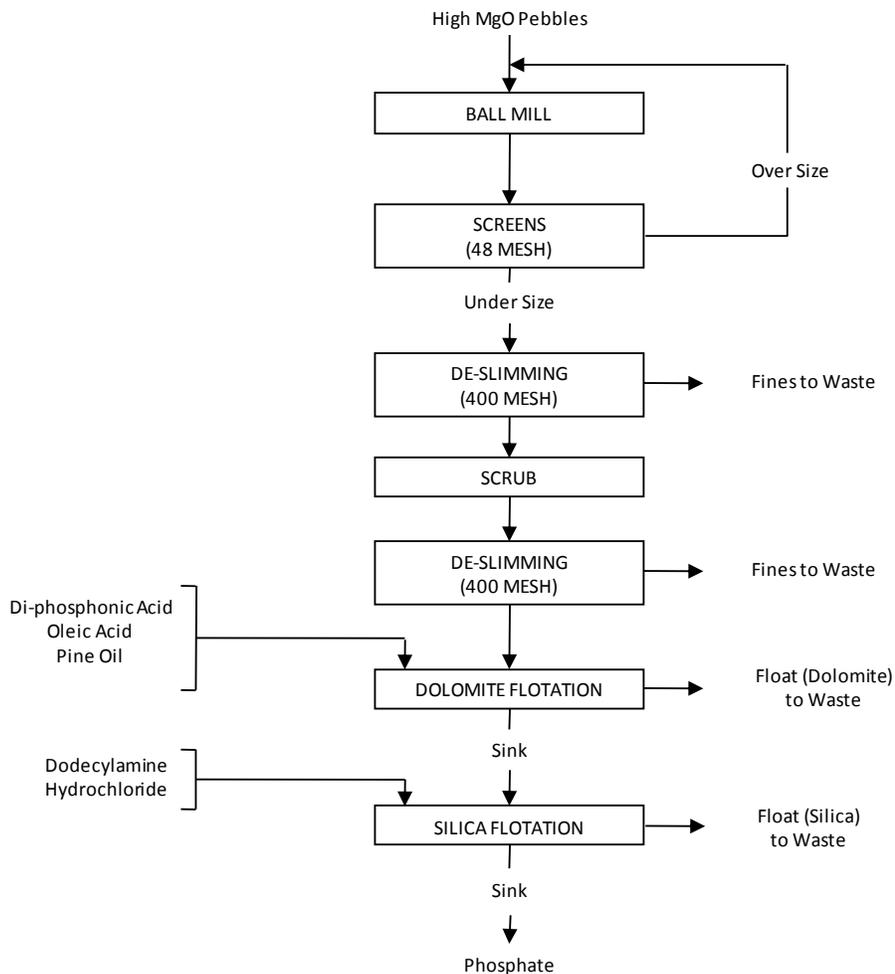


Figure 1-5. Flow sheet of TVA Process

CLDRI Process

China is recognized as the leading producer of phosphate. 90% of the phosphate reserves in China are observed to have high dolomitic content.

The CLDRI Process (Figure 1-6) was developed at China Lianyungang Design and Research Institute and it ensured MgO content less than 0.1% with P₂O₅ loss of 6%. The process was comprised of two steps.

The first step involved grinding of the feed particles for liberation and the second step involved de-sliming which in turn improved flotation recovery and lowered production cost. These flotation technologies have successful industrial applications [El-Shall et al., 1996].

The China Lianyungang Design and Research Institute had conducted extensive studies on Florida high dolomitic pebble with the objective of using the fine particle flotation technology to recover phosphate. Pilot scale testing was carried out jointly by the Research Institute, Jacobs engineering group and IMC phosphate company at Jacob's pilot plant facility. Preliminary economic and technical analysis by Jacob's engineering (based on pilot plant test results) showed that fine-particle flotation technology could be used economically and technically for processing Florida's dolomitic phosphate pebbles [Geo et al., 2002].

In the CLDRI's fine flotation process, by which the percentage of MgO could be reduced to less than 1%, concerns were on handling of the fine concentrates (dewatering and transport), capital costs because of grinding and multiple process steps and operating costs as the reagent consumption is directly proportional to the MgO content in the phosphate rock.

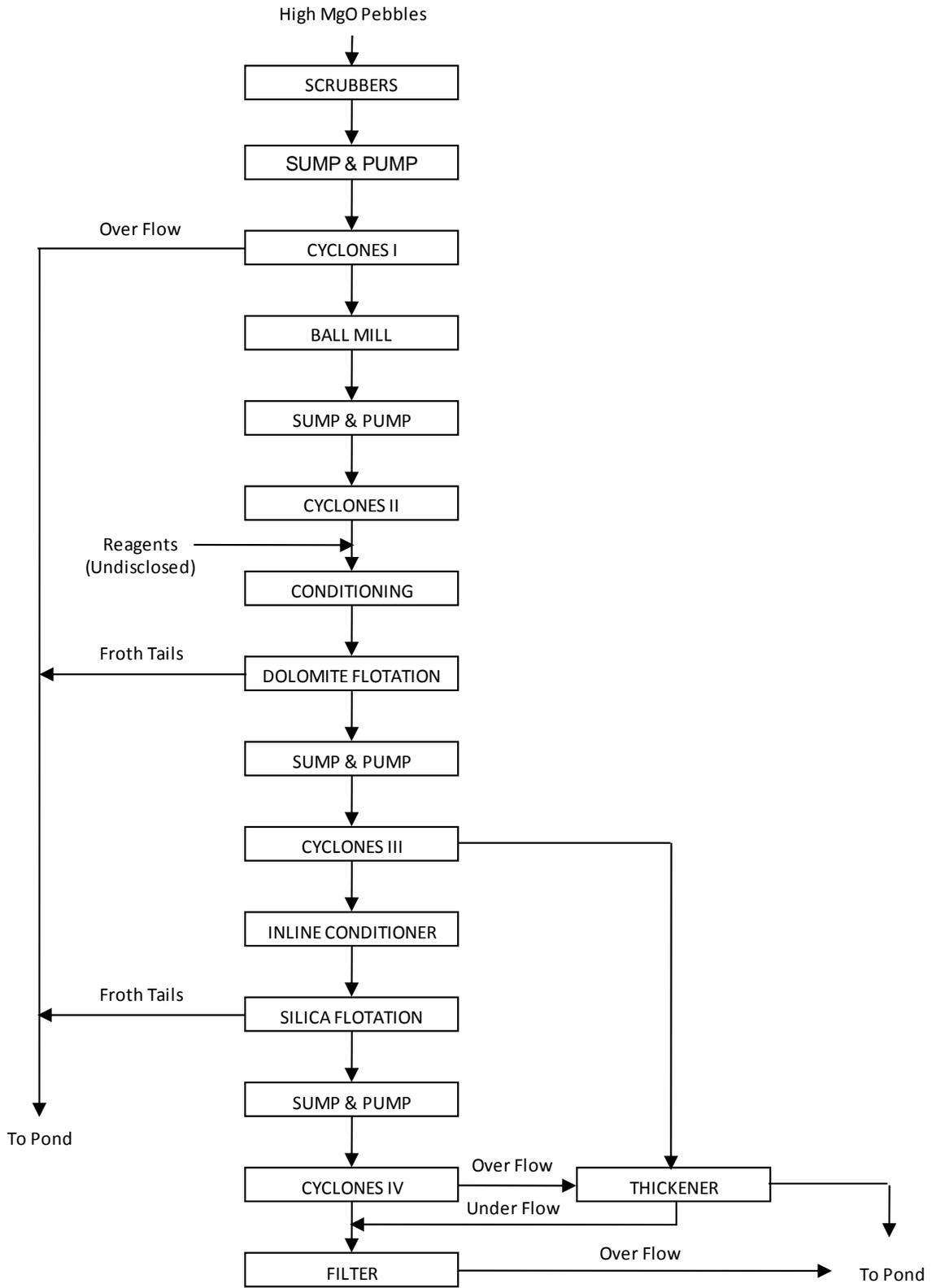
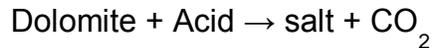


Figure 1-6. Flow sheet of CLDRI Process

Reactive Flotation

The term 'Reactive Flotation' as labeled involves combining two unit operations i.e. reaction and flotation into a single unit

Basic Concept: It is well known that carbonate minerals (dolomite) react with acid and nucleate CO₂ gas at the particle solution interface.



Entraining the liberated CO₂ at the surface via a suitable coating agent renders the particle more buoyant providing selective separation i.e. dolomite (carbonate) from phosphate rock (non-carbonate). Extensive research by [El-Midany et al, 2004] identified poly vinyl alcohol (C₂H₄O)_x as an effective coating agent that can retain CO₂ gas. This was because of the impermeability of PVA to CO₂ gas, permeability to sulfuric acid and flexibility of PVA coating. Experimental results reveal 3% PVA solution (coating agent) and 3% H₂SO₄ as optimum parameters. Beaker tests in this research confirmed flotation of particles as large as 10mm and phosphate concentrates of less than 1% MgO with relatively high recovery were obtained.

CHAPTER 2 MATERIALS AND METHODS

Feed Preparation

Phosphate rock received from Mosaic was sampled from the barrels in a manner that ensures uniform distribution. It involved splitting the content into four groups followed by mixing two of the diagonal groups which was split again into 4 groups and the procedure was followed till manageable amount of rock sample is available for grinding. Direct analysis (technique explained later) of a sample revealed the following size distribution and composition

Table 2-1. Size Distribution and composition of Barrel Content

Properties	+9.51mm	-9.51+4.76mm	-4.76+2mm	-2+1mm	-1mm
Size Distribution,%	18.10	36.50	31.80	11.20	02.37
P ₂ O ₅ , %	20.70	20.50	24.90	24.80	19.40
MgO, %	04.20	03.94	01.06	00.81	10.30

Rocks of size +4.76 mm in the sample were observed to have high MgO content. Hence feed preparation for all experiments involved crushing of rocks of size +4.76mm from the sample. The sizes to which the rocks were crushed are -9.51+4.76 mm, -4.76+2mm, -2+1mm and -1mm. Crushing was carried out in a disc mill. Crushed particles were washed to remove fines (de-slimming).

Coating

Crushed particles were coated with 3% Poly Vinyl Alcohol (PVA) [Optimum value determined through research (El-Midany et al)]. Using >3% PVA in experiments was observed to be uneconomical as it further reduced the rate of diffusion of 3% sulfuric acid, whereas <3% PVA revealed significant decrease in flotation due to rupture of PVA coating. For the preparation of 3% PVA solution, a calculated amount of PVA powder was heated in distilled water for the duration of one hour which was followed by

standardization in flasks. Coating was carried out either by mixing or spraying. Experiments carried out revealed spray coating technique more preferable as it increased coverage and ensured uniform coating.

Reactive Flotation

The coated particles were introduced to 3% H_2SO_4 in the process equipment (beaker / column / screw pipe / co-current vibrating sluice / counter current vibrating sluice). Particles that float are referred to as tailings (dolomite rich) and the non-floating particles are referred to as concentrates (phosphate rich). The collected particles were washed, dried, weighed and analyzed.

Analysis

Analysis involves determining percent composition of P_2O_5 , MgO and acid insolubles in the feed, tailings and concentrate. The dried samples were crushed to fine powder and riffled to weights less than 1 gm. These were then dissolved in aqua regia. The resulting solution was filtered, standardized and forwarded for spectroscopy analysis. The filter paper containing insoluble material was dried, charred and weighed for calculating acid insolubles.

CHAPTER 3 EXPERIMENTAL RESULTS WITH BEAKER TESTS

Simple beaker tests have been used as reference. Feed particles coated with PVA were immersed in a beaker containing 3% H₂SO₄ solution. Amount of particles employed were such that they form a monolayer at the bottom of the beaker.

As the reaction proceeded particles were observed to rise to the surface. These particles were identified as tailings (dolomite rich) and continuously collected. The duration of the experiment was roughly 30 minutes. Particles remaining at the bottom were the dolomite lean concentrates. The tailings and concentrate collected were dried and analyzed.

During experimentation it was observed that tailings if not collected quickly tend to drop. However as the reaction proceeded, these particles tended to float again i.e. there was continuous floating and sinking of dolomite particles. Significant amount of dolomite particles at the bottom of the beaker were observed to have bubbles attached to them and did not readily float. These particles tended to float over a longer duration of time or when the monolayer was exposed to slight shocks. These particles were identified as heavy tailings in later equipment designs and contribute significantly towards MgO removal

Beaker tests were carried out for particles of size ranges -9.51mm, -4.76+2mm, -2+1mm and -1mm coated with 3% PVA in the proportion 3 lb PVA/ton feed and introducing to 3% sulfuric acid. The feed was prepared as mentioned earlier by crushing sampled particles from Mosaic barrel of size >4.76 mm in disc mills followed by de-slimming.

[Object 3-1. Video of Beaker test](#)

Table 3-1. Results on beaker test for different particle size

Particle Size Range (mm)		- 9.51	- 9.51 + 4.76	- 4.76 + 2	- 2 + 1	-1
Feed Composition (%)	P ₂ O ₅	19.20	20.60	18.70	19.70	19.90
	MgO	04.12	03.92	04.42	03.89	03.83
	Acid Insoluble	09.02	09.02	15.20	11.7	TBD
Concentrate (%)		62.60	89.70	83.60	69.10	72.00
	P ₂ O ₅	21.70	20.80	19.60	23.70	18.00
	MgO	01.30	01.91	01.72	00.52	02.76
	Acid Insoluble	13.40	09.42	13.10	11.60	TBD
Tailings (%)		32.90	03.47	08.97	25.90	15.35
	P ₂ O ₅	10.10	01.27	01.37	06.95	24.80
	MgO	08.66	13.60	15.20	11.00	06.59
	Acid Insoluble	12.80	10.20	14.90	11.40	10.90
P ₂ O ₅ Recovery		70.70	90.60	87.50	83.20	65.20
MgO Removal		80.30	56.40	67.50	91.90	48.20

Results revealed low removal in large feed particles of size -9.51+2mm and low recovery for particles of size -1mm.

In large particles (-9.51+4.76mm), dolomite maybe entrapped within the complexes of the phosphate rock. This prevented sulfuric acid from interacting with dolomite to generate CO₂ gas. Furthermore, low surface to mass ratio for large particles translated to low bubble generation and hence low removal. Particles of size -1mm tended to have high surface to mass ratio and hence large amount of PVA was required for effective coating (uneconomical). Froth formation was observed during experimentation which resulted in entrapment of phosphate particles in the froth and explained the low recovery.

Particles of size -2+1mm displayed satisfactory removal and recovery and hence have been used as standard feed particle size in all the following designs. It is worthy to note that beaker tests on particles of size -4.76+2 mm yielded removal of just 67.5%.

CHAPTER 4
NEW EQUIPMENT DESIGNS AND EXPERIMENTAL RESULTS

Column

Columns are the most commonly used equipment in industries for floatation processes. Hence a column was the first among the designs investigated. A bench-scale column was installed and feed particles coated with PVA were introduced through a rock feeder at the top of the column and were exposed to a continuous counter current stream of 3% H_2SO_4 which was introduced at the foot of the column at a controlled flow rate employing a centrifugal pump as shown in the figure (Figure 3-1). Tailings overflow whereas the concentrate is collected at the column base.

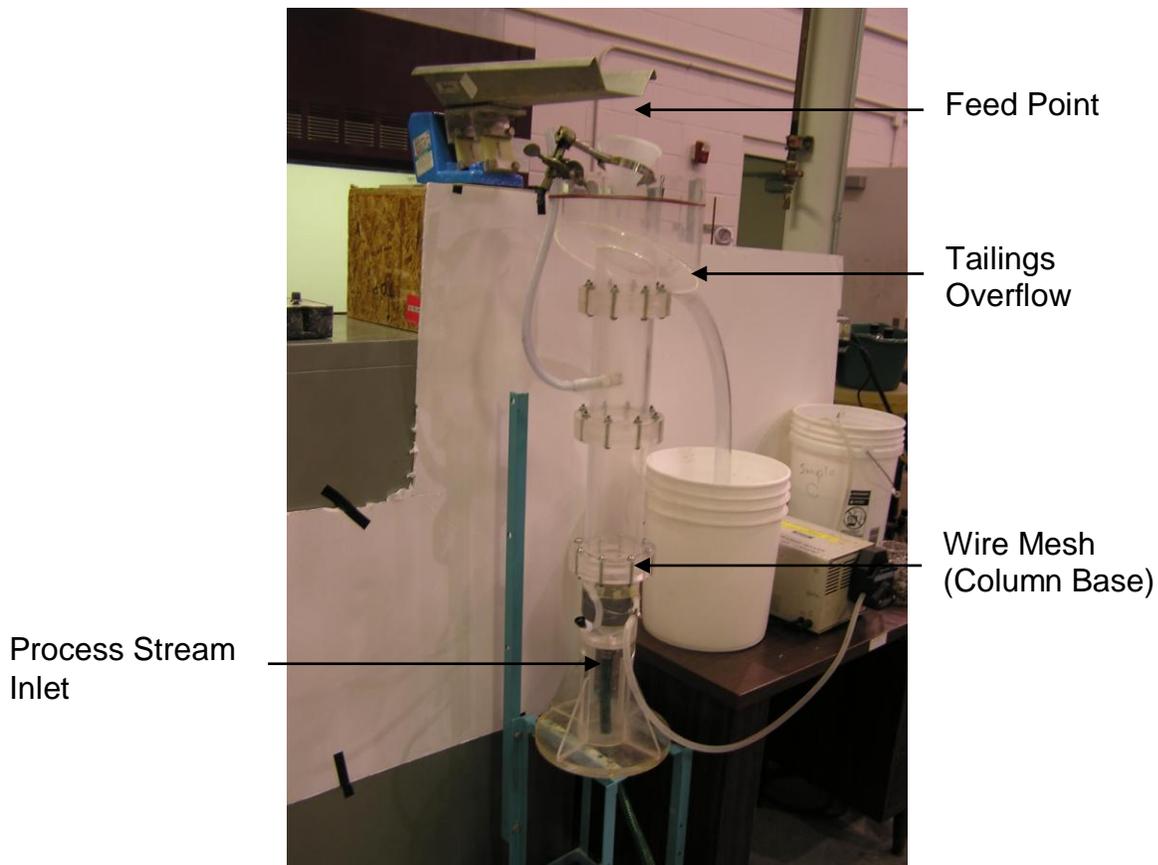


Figure 4-1. Experimental setup of column for reactive flotation

Two models of actual column base were tried out

- i. Fluidized bed
- ii. Wire Mesh

Experimentation on the model employing the concept of a fluidized bed to generate the effective base revealed significant attrition between the packing material of the bed and the coated particles which disrupted the stability of the bubbles and resulted in almost no flotation. Hence experimentation on the column was carried out with the installation of a wire mesh as the effective base of the column i.e. while the tailings overflowed from the top of the column, concentrate was collected on the wire mesh. The effective height of the column was recognized as the length from the top of the column (overflow point) to the wire mesh and not to the actual foot of the column (inlet for 3% H_2SO_4).

During experimentation with effective column length of ~75 cm it was observed that particles floated to a height of around 15-20 cm from the column base, remained suspended for a few seconds and then tended to sink. A possible explanation could be that the pressure exerted by the liquid column (~ 55 cm) accompanied with the flow to overcome this pressure tends to rupture the bubbles generated on the particles at the column base. Reducing the effective length improved the situation.

Experiments were carried out for an effective column length of ~15cm at different flow rates with coated particles of size -2+1mm prepared by crushing barrel sample of size >4.76 mm followed by de-slimming.

Note that appreciable losses were recorded in column test because of extensive dissolution by sulfuric acid.

Table 4-1. Results on column test for different flow rates

Flow Rate (LPM)		5	10	15	20
Feed Composition (%)	P ₂ O ₅	22.60	22.60	22.60	22.60
	MgO	03.01	03.01	03.01	03.01
	Acid Insoluble	07.03	07.03	07.03	07.03
Concentrate (%)		88.60	86.90	84.20	81.30
	P ₂ O ₅	21.30	20.10	22.80	23.40
	MgO	01.93	01.92	02.17	02.03
	Acid Insoluble	07.50	08.48	08.43	07.96
Tailings (%)		02.53	02.97	03.05	04.79
	P ₂ O ₅	15.70	17.40	11.50	12.20
	MgO	05.40	04.46	06.95	06.65
	Acid Insoluble	07.33	07.01	09.27	07.48
P ₂ O ₅ Recovery (%)		83.60	77.10	85.10	84.20
MgO Removal (%)		43.30	44.50	39.10	45.10

Higher flow rates tend to rupture the bubbles and the tailings consisted of particles that have been carried solely by the flow and not due to bubble formation. Generally unsatisfactory results made us abandon the column and we moved forward to analyze horizontal equipment designs.

Screw Pipe

To avoid influence of pressure of liquid column and high flow rate to overcome the pressure, equipment with horizontal flow was proposed. A screw pipe with internal threading and of length ~1.25 m was installed at an angle of ~5° and was connected to a mechanism that sets the pipe rotating in a manner such that particles introduced at the lower end are carried to the upper end as shown in Figure 3- 2. The process liquid was introduced at the upper end of the screw pipe and a circular weir installed at the lower end regulated the liquid level in the pipe. The feed of coated particles introduced at the lower end under the influence of rotation and the presence of screw threading inside the pipe moves to the upper end and is exposed to a counter current stream of

process liquid (3% H_2SO_4). The process liquid which flows towards the lower end under the influence of gravity carries the tailings over the circular weir and the concentrate is obtained at the upper end.

The actual distance travelled by the coated particles is $\gg 1.25$ m because of the internal threading. As the coated feed particles travel between the threading there was significant contact between the particles and the pipe surface which led to rupture of bubbles. Hardly any tailings were collected during the experimentation. Furthermore the pipe threading proved a hindrance for heavy tailings, a concept coined later in the next horizontal equipment design tested i.e. a vibrating sluice.

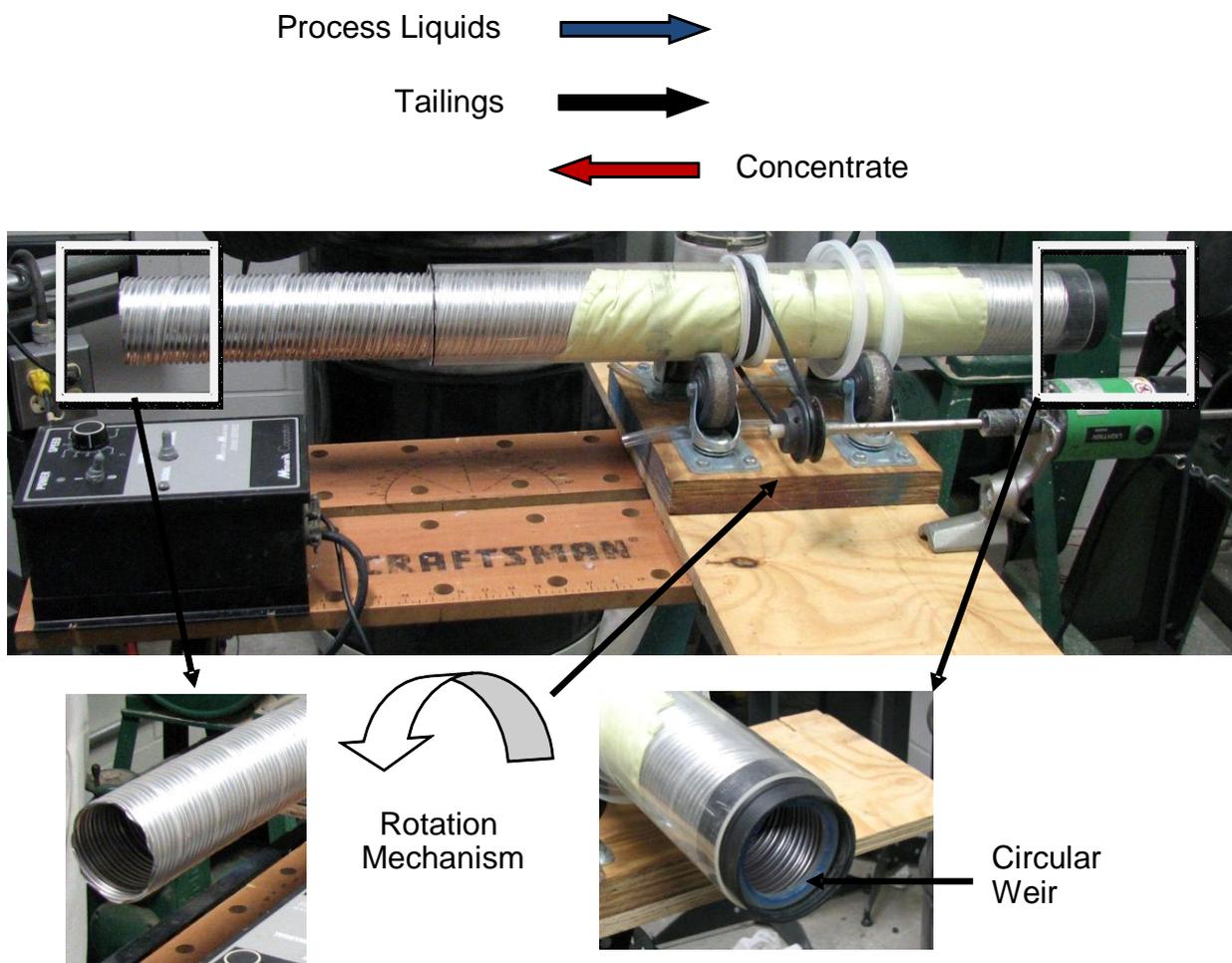


Figure 4-2. Experimental setup of screw pipe for reactive flotation.

Vibrating Sluice

Two concepts of flow pattern were investigated in the Vibrating Sluice

- i. Co-Current Flow
- ii. Counter Current Flow

Co-Current

Co-current flow is that in which the channel vibrations that influenced the movement of the concentrate and the process stream (3% H_2SO_4) flow that influenced the movement of the tailings is in the same direction

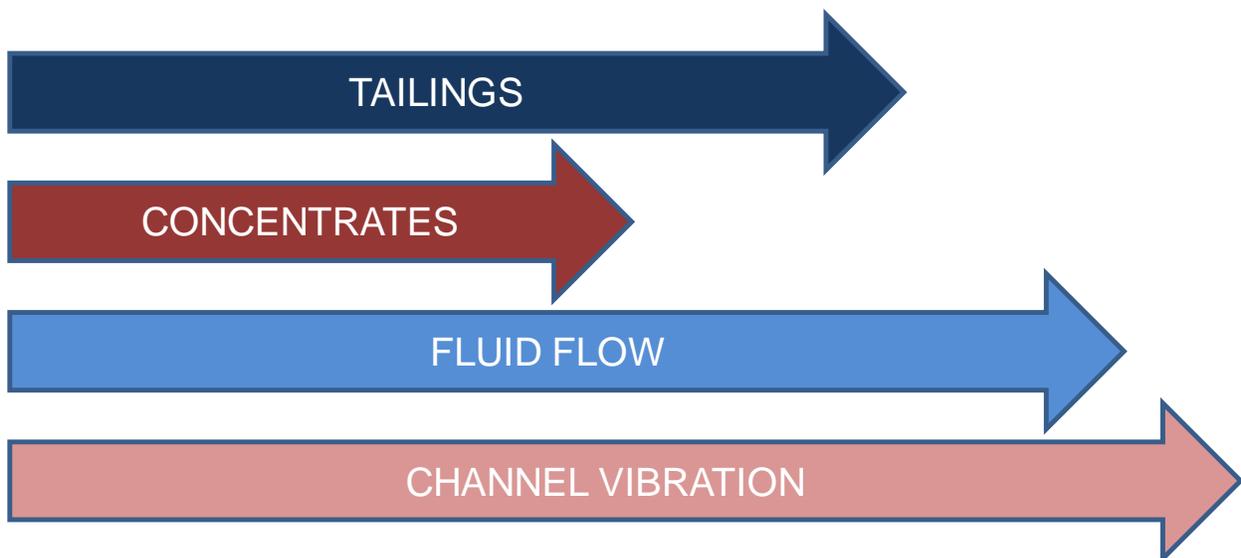


Figure 4-3. Concept of co-current flow

A channel protected with acrylic material (H_2SO_4 resistant) was attached to a vibrating rock feeder. The channel was placed at an inclination of $\sim 1^\circ$ and the process liquid was introduced at the upper end of the channel through a flow distributor. PVA coated feed particles were introduced at the upper end through a rock feeder. A skimmer was placed at the lower end to collect the tailings. Vibration of the sluice in the direction of the liquid flow tended to carry the particles down the channel along with the process liquid. During the movement of the feed particles in the channel it was

separated into tailings and concentrate which were collected separately at the lower end with the assistance of a skimmer

There was significant engineering difficulty to maintain the liquid level in the channel and hence in placing the skimmer. Appreciable amount of particles with small bubbles that do not float to the gas liquid interface and hence getting collected among concentrate were observed. It is here that the significance of these particles were recognized. The term tailings were to be sub categorized into light tailings and heavy tailings. Dolomite particles that clustered to large bubbles and readily floated have been termed as light tailings whereas those with small bubbles (signifies some presence of dolomite) were referred to as heavy tailings. Among the particles that do not reach the gas liquid interface (concentrates and heavy tailings), heavy tailings were observed to be significantly influenced by fluid flow in relative to channel vibrations, unlike concentrate which was more influenced by the channel vibrations. Reconsidering the beaker test, particles referred to as heavy tailings can be identified as those particles that tend to float over a longer duration of time. Ideal performance of our equipment would involve the capture of these heavy tailings. This led to the concept of employing counter current flow in the channel.

Counter Current

The concept of counter current flow involved employing channel vibration that influenced the movement of concentrate in the opposite direction with respect to the direction of process stream flow that influenced the tailings. Tailings as mentioned earlier have been categorized as light and heavy tailings. Light tailings were solely influenced by the liquid flow whereas the heavy tailings were influenced by both channel vibrations and process stream flow but with a greater inclination to the latter.

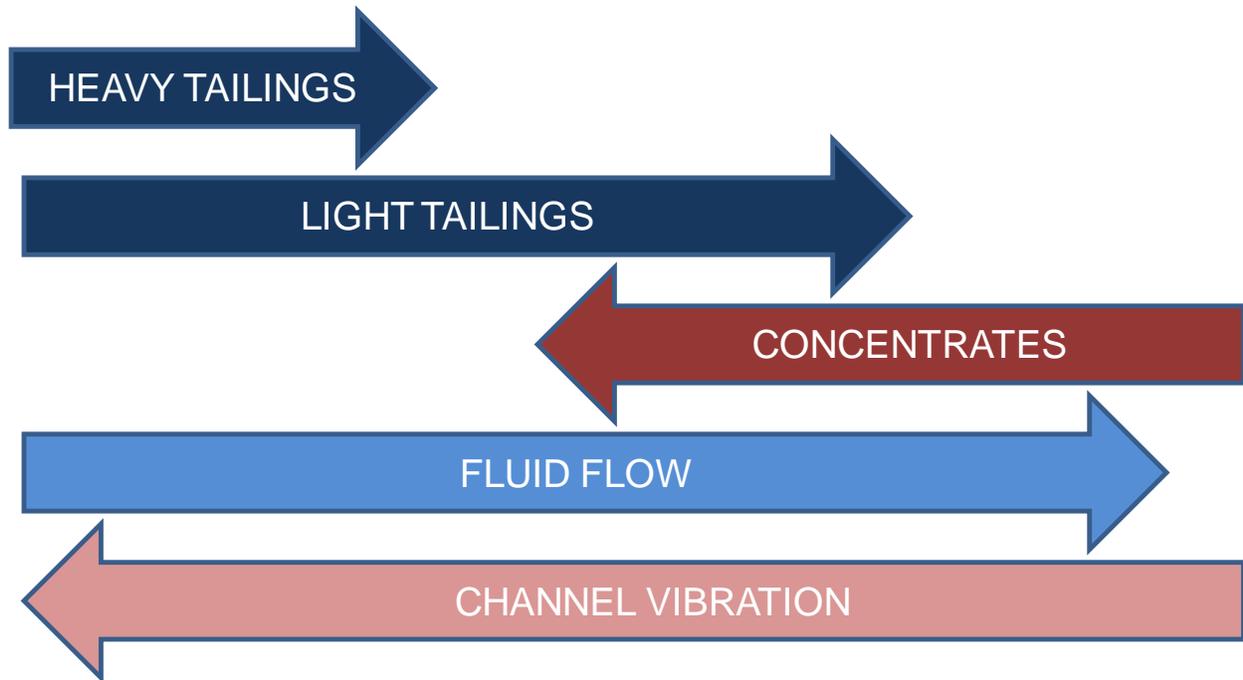
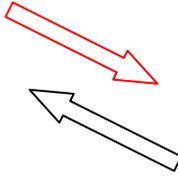


Figure 4-4. Concept of counter-current flow

A channel protected with acrylic material (H_2SO_4 resistant) is attached to the base of a vibrating rock feeder as shown in Figure 3-5. The channel was placed at an inclination of $\sim 3^\circ$ and the process liquid was introduced as a combination of continuous (employing a centrifugal pump) and pulsating (employing a peristaltic pump) flow at the upper end of the channel (Figure 3-6) through a liquid distributor that doesn't disrupt the liquid level in the channel nor the movement of the concentrate about to leave the channel. Both the liquid flows (continuous and pulsating) were against the vibration of the channel. Pulsating flow was introduced to damp the vibrations transmitted to the liquid surface by the vibrating channel.

A weir with a small opening at the bottom was installed at the lower end to facilitate exposure of heavy tailings to significant flow rate. A differential level trap was set up at the lower end before the weir to entrap the heavy tailings (Figure 3-7).

Process Liquid +
Light Tailings +
Heavy Tailings



Concentrate

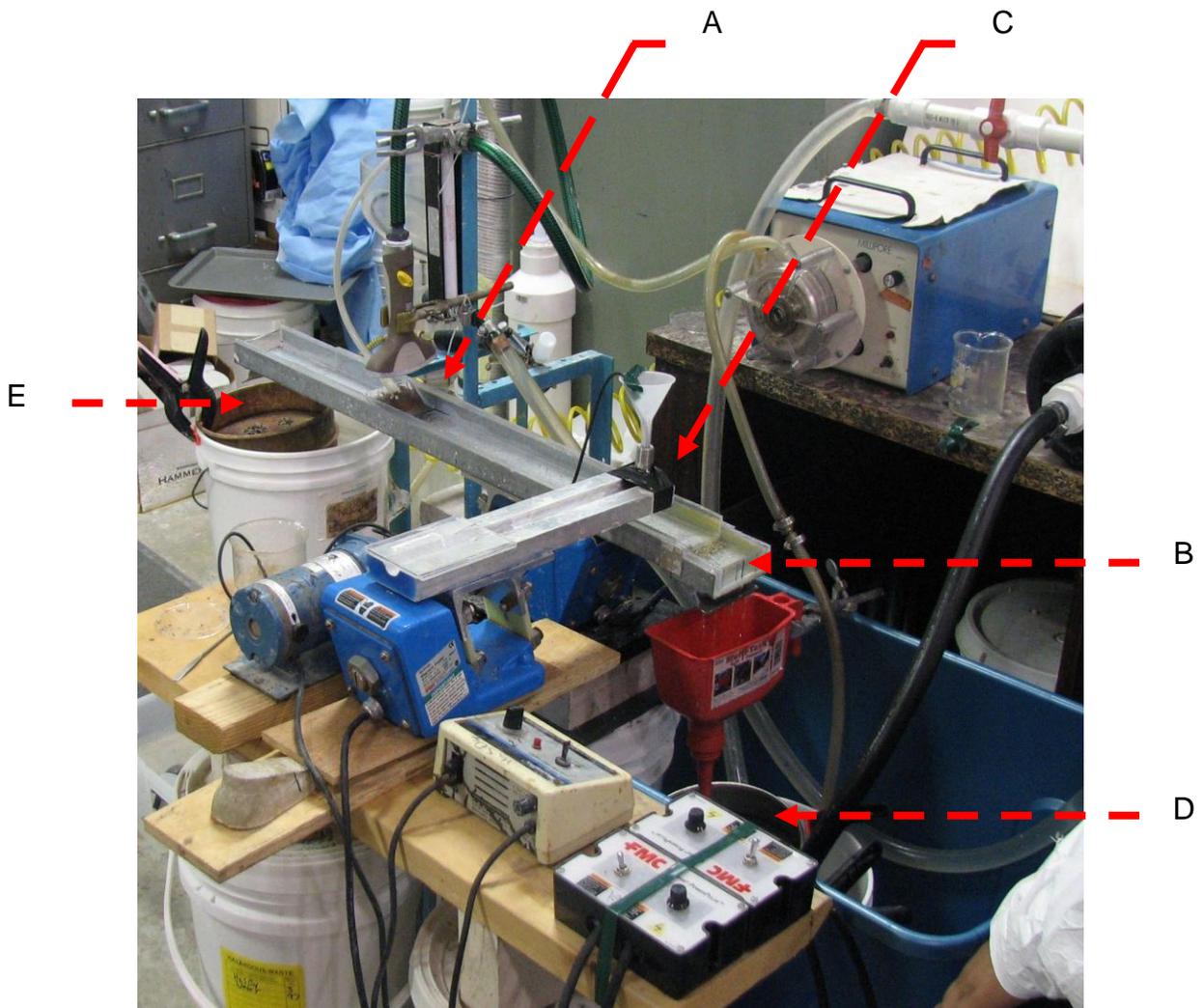


Figure 4-5. Experimental setup of vibrating sluice for reactive flotation. A) Process stream inlet. B) Weir and Trap. C) Feed Point. D) Tailings. E) Concentrate.

[Object 4-1. Video of Counter current vibrating sluice](#)

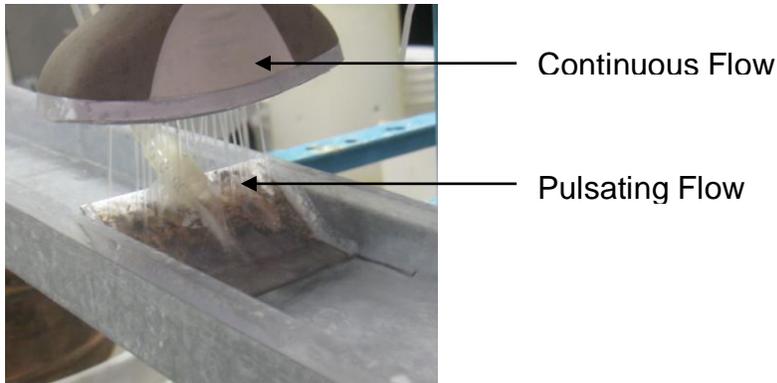


Figure 4-6. Process stream inlet

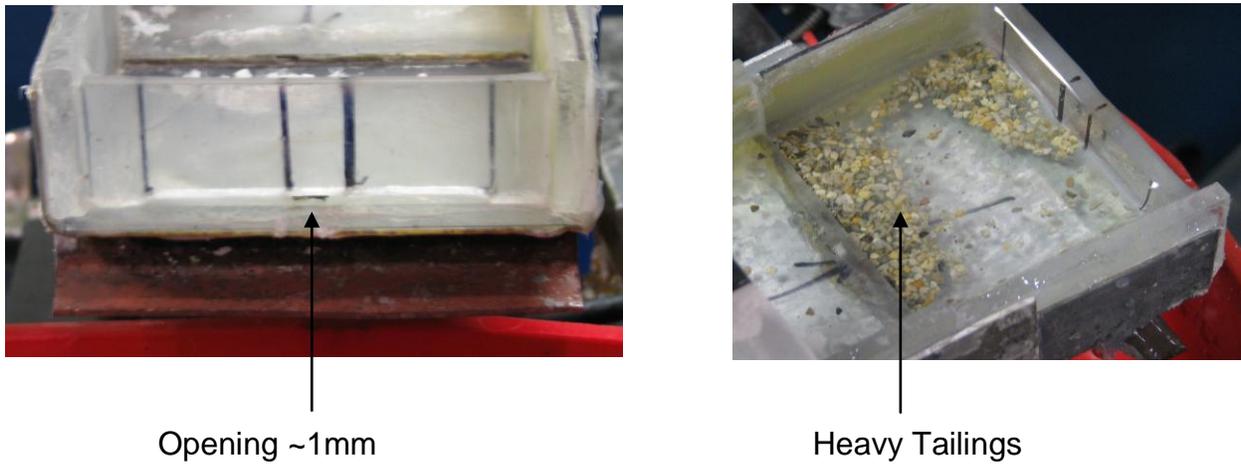


Figure 4-7. Weir and trap

The feed particles coated with PVA were pre-treated with the process liquid and were introduced at the lower end at a distance $\sim 7\text{cm}$ in front of the trap. Pre-treatment of the feed particles was observed to enhance the separation and reduced build up of particles in the channel (Figure 3-8). It was observed the introduction of the feed as a line hindered the movement of the heavy tailings and hence point feeding was adopted (Figure 3-8). The effective length of the vibrating sluice is the distance from the liquid distributor to the feed point ($\sim 50\text{ cm}$).

As the feed particles moved to the upper end, they were exposed to a counter current process stream ($3\% \text{H}_2\text{SO}_4$). The stream carried the light tailings (Figure 3-9)

over the weir and the heavy tailings (Figure 3-7) into the trap. The concentrate moved to the upper end and was collected across the liquid distributor (Figure 3-9).

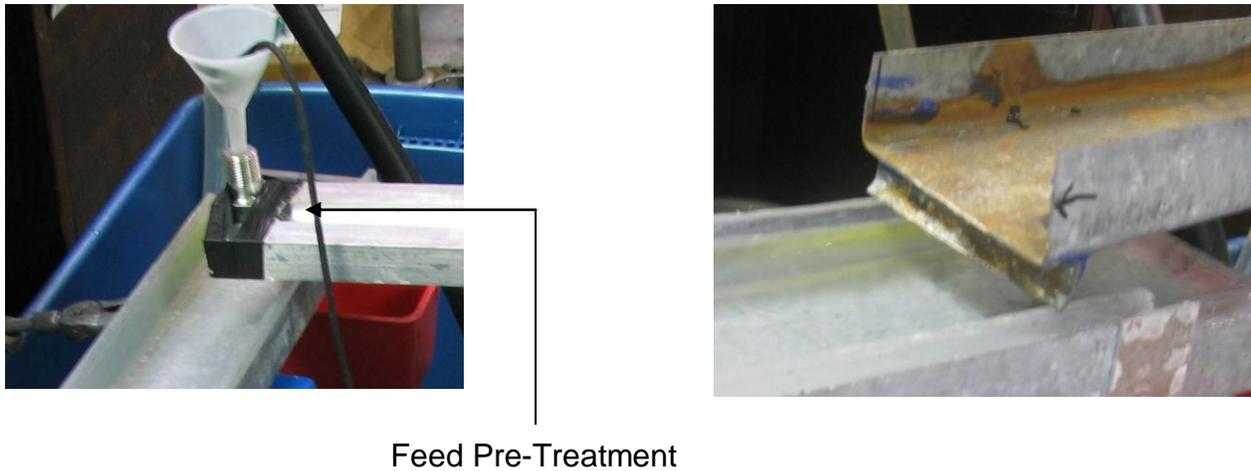


Figure 4-8. Point feed and line feed techniques respectively.



Figure 4-9. Tailings and concentrate collected during experimentation.

Experiments were carried out employing crushed particles of size $-2+1\text{mm}$ and $-4.76+2\text{mm}$, varying the feeding technique, flow technique, coating technique, coating agent and amount of coating employed.

LF- Line Feed; PF- Point Feed

P-Pulsating Flow; C-Continuous Flow

M- Mixing; SC-Spray Coating

Table 4-2. Results on counter current vibrating sluice for different feeding techniques, flow techniques, coating techniques and amount of coating

Feed Particle Size		-2+1mm	-2+1mm	-2+1mm	-2+1mm
Feed Technique		LF	LF	PF	PF
Flow Type		P	P+C	P+C	P+C
Coating Technique		M	M	SC	SC
Coating Material (lb PVA / ton feed)		PVA (3)	PVA (3)	PVA (3)	PVA (6)
Feed Composition (%)	P ₂ O ₅	21.90	21.90	21.90	21.90
	MgO	03.75	03.75	03.75	03.75
	Acid Insoluble	11.30	11.30	11.30	11.30
Concentrate (%)		79.70	69.40	58.48	67.80
	P ₂ O ₅	24.10	23.20	25.82	25.23
	MgO	02.36	01.59	01.46	01.19
	Acid Insoluble	10.00	12.39	11.69	11.13
Tailings (%)		13.30	22.00	33.64	24.71
	P ₂ O ₅	13.90	11.60	16.09	12.06
	MgO	07.08	07.73	06.13	08.22
	Acid Insoluble	11.80	14.26	10.73	11.06
P ₂ O ₅ Recovery (%)		87.80	73.80	69.10	78.20
MgO Removal (%)		49.80	70.50	77.00	78.40

Table 4-3. Results on counter current vibrating sluice for different coating agents

Feed Particle Size		-2+1mm	-2+1mm	-4.76+2mm
Feed Technique		PF	PF	PF
Flow Type		P+C	P+C	P+C
Coating Technique		NA	M	SC
Coating Material (lb PVA / ton feed)		NIL	Fatty Acid	PVA (3)
Feed Composition (%)	P ₂ O ₅	21.90	21.90	22.30
	MgO	03.75	04.74	04.38
	Acid Insoluble	11.30	10.13	10.13
Concentrate (%)		90.00	63.32	67.00
	P ₂ O ₅	17.80	22.01	23.29
	MgO	01.98	03.42	01.62
	Acid Insoluble	12.12	10.23	11.23
Tailings (%)		03.47	19.70	28.53
	P ₂ O ₅	09.25	08.36	11.71
	MgO	08.13	10.47	08.73
	Acid Insoluble	06.56	06.32	09.70
P ₂ O ₅ Recovery (%)		73.15	63.63	70.27
MgO Removal (%)		51.20	54.38	75.23

Experiments reveal point feed more effective than line feed. Combination of pulsating and continuous flow was found to be more effective than pulsating flow alone. Pulsating flow alone tends to leave the tailings suspended and do not move with the flow. Spray coating of feed particles was found to be more effective than mixing as it ensures uniform and effective coating of all particles. Increasing the amount of PVA used to 6 lb PVA/ton of feed brought about dramatic improvement in recovery and removal. This however is not economically feasible. In the experiment involving the use of no coating the feed was introduced at a distance of ~1cm from the trap. Tailings collected were mainly heavy tailings and this reveals the performance capability of this equipment. Use of fatty acid seems to be counter-productive. The process stream is observed to strip the particle surface of fatty acid and resulted in an oily stream. Experiments on the equipment with particles of size $-4.76+2\text{mm}$ revealed significant removal in comparison to beaker tests. This result revealed the effectiveness of the equipment as it is capable of removing particles that tend to slightly float (Heavy tailings). The presence of significant amount of dolomite in heavy tailings is further confirmed by this result. Ability to use particles larger than 2 mm is highly advantageous from the industrial perspective as it reduces crushing cost. Experiment were not carried with particles larger than 4.76 because of the engineering limitations in maintaining the desired liquid level in the channel for this model

Parameters observed to influence the performance of the process are the angle of the channel, flow rate, pulse rate, feed rate, vibration rate of the channel, size of the opening at the bottom of the weir (regulates the flow rate exposed to the heavy tailings),

fluid level in the channel, distance of the feed point from the trap and the extent of pre-treatment of the coated feed particles.

Experiments were also tried for different concentrations of PVA coating employing crushed particles of size -2+1mm.

LF- Line Feed; PF- Point Feed

P-Pulsating Flow; C-Continuous Flow

M- Mixing; SC-Spray Coating

Table 4-4. Results on counter current vibrating sluice for different concentrations of PVA

Feed Particle Size		-2+1mm	-2+1mm	-2+1mm
Feed Technique		PF	PF	PF
Flow Type		P+C	P+C	P+C
Coating Technique		SC	SC	SC
Coating Material (lb/ton feed)		PVA (2)	PVA (2)	PVA (3)
Concentration of Coating PVA (%)		1	2	2
Feed Composition (%)	P ₂ O ₅	22.01	22.01	22.01
	MgO	03.50	03.50	03.50
	Acid Insoluble	10.13	10.13	10.13
Concentrate (%)		77.96	76.08	74.50
	P ₂ O ₅	22.11	21.73	21.60
	MgO	02.45	02.20	02.03
	Acid Insoluble	10.17	09.75	10.78
Tailings (%)		13.89	15.97	18.01
	P ₂ O ₅	08.97	09.01	08.99
	MgO	09.80	09.65	10.23
	Acid Insoluble	07.01	07.95	08.75
P ₂ O ₅ Recovery (%)		78.31	75.11	73.11
MgO Removal (%)		45.42	52.23	57.83

The results were unimpressive because low concentration of PVA does not guarantee strong binding of bubbles on the particle surface. Furthermore, rupture of coating takes place. The results also revealed that coating of the particles prevents dissolution of phosphate by sulfuric acid.

CHAPTER 5 CONCLUSIONS AND RECCOMENDATIONS FOR FUTURE WORK

The equipment designing was carried out crudely. One of the major concerns is the amount of PVA used. Optimum results were obtained for 6 lb PVA / ton feed. Reducing this would enhance the favorability of the process. The model employed is crude and has limitations. However impressive results were observed for particles with no PVA coating and particles of size $-4.76+2$ mm (relative to beaker tests). Design limitations hindered testing of particles larger than 4.76 mm.

Coating and mixing technique should be further investigated to bring about optimum and uniform coverage. Various parameters such as flow rate (continuous + pulsating), frequency of vibration, feed rate, channel length and liquid level in channel are potential parameters influencing the performance of the equipment and should be investigated on a better pilot scale equipment that readily allows the manipulation of these parameters.

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BIOGRAPHICAL SKETCH

Abbin Antony was born in Hofuf, Saudi Arabia in November 1985. He was enrolled at TKM College of Engineering affiliated to University of Kerala in India from 2003 to 2007. During his bachelor's degree he underwent In-Plant Training at Hindustan Organic Chemical Ltd. and also worked on a project involving optimization of a production unit at Kochi Refineries- Bharat Petroleum Corp Ltd. in Kerala, India. He completed his bachelor's degree in chemical engineering in 2007 and enrolled for master's degree at University of Florida in 2008. He worked with Professor Spyros A. Svoronos and Professor Hassan El-Shall on translating a batch technique for separation of dolomite from phosphate developed by a student of Professor Hassan El-Shall into a continuous process.