

## Selective Coagulation and flocculation of Roper Bar slimes

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## Executive Summary

This report deals with investigations involving the characterization of particle zeta potential as function of pulp pH and selective coagulation and flocculation behaviour of Roper mine ore sample performed under isothermal ( $22 \pm 0.1$  °C) batch conditions. A hydrocyclone overflow T392 powder sample supplied in a well-sealed plastic bag was used in the tests. Discussions with Dr. Vic Absalom indicated that the sample comprised predominantly silica, hematite and siderite as minor mineral phase. Based on this mineralogical information, the main focus of the sample characterization and treatment exercises was to explore the possibility of separating the ore into hematite/siderite and silica based products.

Mineral phase separation approach of selective coagulation and/or flocculation was adopted based upon perceived differences that may exist between silica and hematite in terms of specific gravity (silica ~ 2.6 vs. hematite ~4.0-5.5), isoelectric points (silica ~ pH 2.0-2.5 vs. hematite~ pH 6.0-7.5) and specific affinity for certain high molecular weight ( $>1 \times 10^6$  Da.) polymeric flocculants (e.g., hematite/siderite for starches vs. silica for polyethylene oxide).

Particle size analysis showed that the hydrocyclone overflow T392 sample was polydispersed with size ranging from 0.2 to 160  $\mu\text{m}$  and mode of ~65  $\mu\text{m}$ . 30 wt.% of the particles were less than 20  $\mu\text{m}$  in size, and hence the remaining 70 wt.% coarser fraction spanned 20 – 160  $\mu\text{m}$  size.

Particle zeta potential, determined by pH sweep in the range 2 -10, indicated that the sample's interfacial chemistry displayed in aqueous media of moderate to high ionic strength electrolyte was reminiscent of silica based dispersion rather than that of a mixture of silica and hematite mineral particles. The zeta potential was negative in the pH range 2 – 10, the magnitude of which decreased with decreasing pH. This was accompanied by an isoelectric point (iep) ~ pH 2.2, a value which is substantially the same as that of pure silica (quartz). It appears that despite the presence higher iep Fe-based minerals (hematite/siderite) in the sample, the siliceous phase in the aqueous pulp dominated the interfacial chemistry.

A small but noticeable iep shift to pH 3 occurred in reverse pH sweep from acidic to alkaline values. This is indicative of specific adsorption electropositive species, e.g.,  $\text{K}^+$  ions in the background electrolyte or an hydrolyzable  $\text{Fe(III)}$  ions leached from hematite. Increasing the ionic strength of the background electrolyte from  $10^{-3}$  to  $10^{-1}$  M KCl did not cause a significant electrical double layer compression / particle zeta potential screening effect.

Selective coagulation tests conducted at pH 2.0-2.5 and 6.0-7.0 with dilute (2.5 wt.% solid) dispersions in  $10^{-3}$  or  $10^{-1}$  M KCl background electrolyte did not cause expected differential sedimentation within 1 or 2 h, reflecting poor selective

separation of silica from hematite. In all cases, a fairly rapid sedimentation, predominantly due to the presence of very coarse particle fraction in the pristine sample occurred within, accompanied by a turbid supernatant clarity of colloiddally stable particles. Over a prolonged settling period (>24 h) however, significant sedimentation finally occurred, leaving a poor supernatant clarity. The mineralogical compositions of both the sedimented and suspended solid particles have not as yet been determined.

Selective flocculation of dilute suspension the colloidal size (< 5  $\mu\text{m}$ ) fraction with high mol wt. maize starch as flocculant did not produce expected selectivity for targeted adsorption onto hematite and hence, bridging flocculation. Addition of up to 11 kg polymer/t solid failed to induce selective flocculation of the hematite/siderite component in the plant pulp sample (hydrocyclone overflow T392).

Parallel fundamental investigations performed with model quartz and hematite mineral suspensions showed that their iep's were at pH 2.0-2.5 and 6.0-7.0, respectively. The selective flocculation of 50:50 hematite : quartz mix suspension with the starch flocculant indicated that was also possible to selectively flocculate hematite, leaving quartz in suspension.

The failure of the real mineral ore sample (hydrocyclone overflow T392) to respond to selective, ephemeral coagulation and/or flocculation suggests a number of possible scenarios as a cause. It appears that the polydispersed sample used was not practically ideal for the proof of concept (of selective coagulation or flocculation). The polydispersity is detrimental to the differential sedimentation approach. It is also possible the finer (<20  $\mu\text{m}$ ) size fraction in the dilute suspension used in flocculation was either not liberated or containing substantial amount of liberated hematite without surface contamination by silica slime.

Based on the present outcomes, it is recommended a fresh mineral ore sample of fine particles with the right size (e.g., (<20  $\mu\text{m}$ ) be produced for further selective coagulation and flocculation testing. It will be necessary to gather detailed, specific information on quantitative mineralogy and particle surface chemistry of the test sample, sedimented and suspended solid products upon sedimentation. Characterization techniques such as X-ray photoelectron spectroscopy (XPS), X-ray energy dispersive spectroscopy (EDAX), backscattered SEM imaging and electro-acoustic zeta potential will be of great use here.

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## **INTRODUCTION**

The present Roper mine ore sample being investigated comprises silica, hematite and siderite mineral phases. The main thrust of the investigations performed to was to determine an effective mineral phase separation method to produce hematite-rich stream of interest and quartz based by-product.

In terms of exploring gravity-driven separation between polydispersed silica and hematite/siderite bearing mineral phases, one may expect the coarser particle fraction in aqueous suspensions to display rapid settling behaviour according Stokes' law. The finer fraction, on the other hand, will experience colloid stability and hence poor settling behaviour, particularly at slurry pH values away from the isoelectric points of silica and hematite/siderite. If the mineral liberation is high and the coarser, faster settling particle fraction may constitute a greater portion of the ore, largely consisting of one mineral phase, say silica or hematite. If that was the case, it might be possible to use size/density-mediated differential sedimentation as a means for silica-hematite mineral phase separation. Otherwise, no upgrade will be observed for the sedimented pulp where both silica and hematite/siderite show similar distributions across all the particle size ranges. QEM-Scan size-mineralogy data may shed more light on this issue.

As for the finer, perennially slow settling particles, opportunities may exist for separation by identifying and using selective coagulation or flocculation of either the silica or hematite particles so that one mineral phase is preferentially aggregated to amplify its settling rate in contrast to the other. For selective coagulation, knowledge of pulp interfacial chemistry determined in terms of particle zeta potential as function pH in aqueous environment is necessary. As for selective flocculation, it may be possible to flocculate preferentially one mineral phase with the other remaining in suspension by using a high molecular weight polymeric flocculant that adsorbs selectively onto one of the mineral phases. The challenges which may exist in selective coagulation and flocculation approaches considered in the present work is if mineral liberation is not high enough and/or surface coating of one minerals phase on the other occurs (e.g., silica particle surface contaminated by Fe species/hematite and vice versa).

## **EXPERIMENTAL**

### **Particle Size Analysis**

Particle size and its distribution were measured by laser diffraction using Malvern Mastersizer 2000 static covering the measurement range 0.02 - 2,000  $\mu\text{m}$ . The sample was dispersed in water with no dispersants. Five replicate measurements were taken at 1 min intervals and the results were averaged.

## **Slurry preparation**

2.5 and 15 wt.% solid slurries of plant sample T 392 Hydrocyclone overflow (T392 Hyd O/F) supplied and model hematite and quartz minerals and their 50:50 mixture were prepared in either  $10^{-3}$  or  $10^{-1}$  M KCl electrolyte for sedimentation and electrokinetic zeta potential measurements, respectively. A known mass of the T392 Hyd O/F solid was mixed with a known mass of either  $10^{-3}$  or  $10^{-1}$  M KCl electrolyte. The slurries were then homogenised by agitation at 300 rpm for 20 min and their pH adjusted to 2 or 6-7 (natural) or 10 using either 1.0 M HCl or KOH prior to selective sedimentation (coagulation and flocculation).

## **Selective Coagulation and Flocculation Tests**

To perform selective coagulation or flocculation and sedimentation tests, 2.5 wt.% solid slurries of plant sample T 392 Hyd O/F were made up in either  $10^{-3}$  or  $10^{-1}$  molar KCl. For selective coagulation, sedimentation tests were conducted at pH 2.0-2.5, 6.0-70 or 10 with a dilute (2.5 wt.% solid) dispersion in a  $10^{-1}$  M KCl background electrolyte,

For selective flocculation, the 2.5 wt.% solid suspension was allowed to undergo coarse particle settling for 5 min and aliquot of the turbid supernatant drawn off into small settling tubes. A known volume (e.g.,  $0.3 \text{ cm}^3$ ) of 0.1 wt % of causticized regular maize starch polymeric flocculant solution was added in a single step to known mass (e.g., 5 g) of well-mixed slurry at 0.5 wt.% solid using a syringe the slurry was thoroughly mixed by inversion 3 times.

The initial settling rate of the coagulated or flocculated suspension was determined by recording the time taken for the “mud line” (solid-liquid interface) in the  $500 \text{ cm}^3$  cylinder to pass between the  $450 \text{ cm}^3$  and  $350 \text{ cm}^3$  marks (over a distance of  $\sim 5.5 \text{ cm}$ ). The sediment was then allowed to stand quiescently for a period of 24 h after which the consolidated bed volume recorded. All tests were conducted at  $22.0 \pm 0.1^\circ \text{C}$ . Photographs were taken initially and after 1 and 24 h.

## **Zeta potential measurements**

Particle zeta potentials ( $\zeta$ ) were determined in the pH range 2-10 from dynamic mobility measurements using an Acoustosizer (Colloidal Dynamics Inc, Australia). The measurements were carried out on agitated suspensions (15% w/w) T392 Hyd O/F particles dispersed in  $10^{-3}$  or  $10^{-1}$  M KCl. All experiments were conducted with the suspension pH progressing from 6-7 (pristine) to pH 10, followed by decreasing pH from 10 to 2. Prior to measurements and analysis, calibration of the Electrokinetic Sonic Amplitude (ESA) probe was carried out by measuring the ESA signal for a standard electrolyte of potassium tungstosilicate  $\text{K}_4(\text{SiW}_{12}\text{O}_{40})$ . This calibrant was selected as a standard because of its very high

chemical stability and the strong electroacoustic signal it provides. The procedure was performed once a day at  $22\text{ }^{\circ}\text{C} \pm 0.1$ . Background corrections were performed for electrolytes at high ionic strength ( $> 0.1\text{ M}$ ) by analysing the electrolyte solution without any particles in it. This background analysis was then subtracted from the final analysis of the dispersion with particles. Depending upon the state or flow behaviour of the dispersion, agitation rates between 500 and 2000 rpm were used. The conductivity, temperature and pH were continuously monitored *in situ*.

## RESULTS AND DISCUSSION

### Average Particle Size

Particle size analysis by laser diffraction indicated that about 20 and 30 wt.% of the particles are less than 10 and 20  $\mu\text{m}$  in size, respectively, with the remaining 70 % being coarser particle whose sizes range from 20 to 160  $\mu\text{m}$ , as shown in Figure 1 below. The data suggest that 2 particle populations of finer size fraction with uniform distribution in 1 – 20  $\mu\text{m}$  size range and coarser fraction displaying an approximate normal distribution with modal size of  $\sim 65\text{ }\mu\text{m}$  exist, and these will have different sedimentation behaviour.

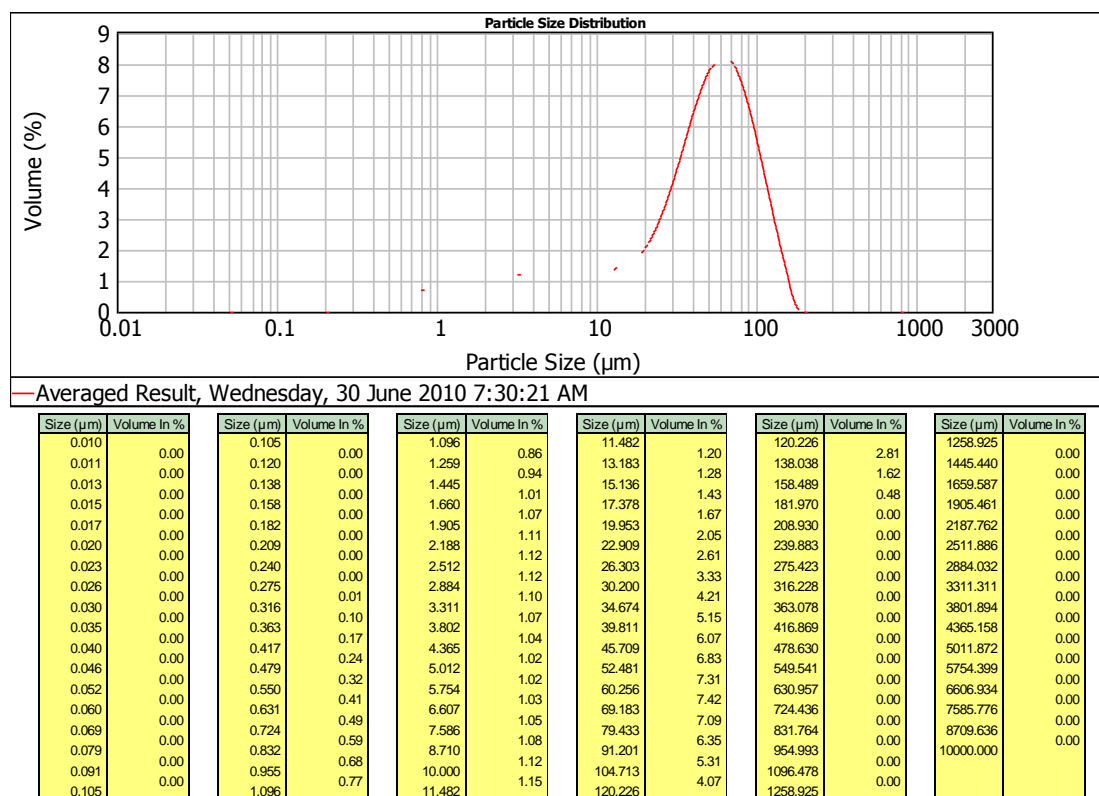


Figure 1: Particle size distribution of Roper mine T392 hydrocyclone overflow solid sample.

### Interfacial Chemistry: Particle zeta potential

Particle zeta potential determined as a function of pH at room temperature for 15 wt.% solid Roper Mine T392 ore slurry showed that in the range 2-10, the particles were negatively charged, the magnitude of which decreased with decreasing pH (Figure 2). An iep of  $\sim$  pH 2.2 was indicated at  $10^{-3}$  to  $10^{-1}$  M KCl ionic strength in the high to low pH sweep. The zeta potential and the iep trends displayed in the pH range 2 – 10 are substantially the same as that of pure silica (quartz). This suggests a siliceous mineral dominated interfacial chemistry system, with the presence of higher iep mineral phases, hematite and siderite, exerting no significant influence. Upon the reverse low to high pH sweep, an iep shift from pH 2.2 to pH 3.0 was observed, suggestive of specific adsorption of  $K^+$  ions in the background electrolyte or an hydrolyzable metal ions (e.g. Fe(III)) leached from hematite.

Increasing the ionic strength of the background electrolyte from  $10^{-3}$  to  $10^{-1}$  M KCl did not cause either a significant electrical double layer compression / particle zeta potential screening effect or a noticeable iep shift.

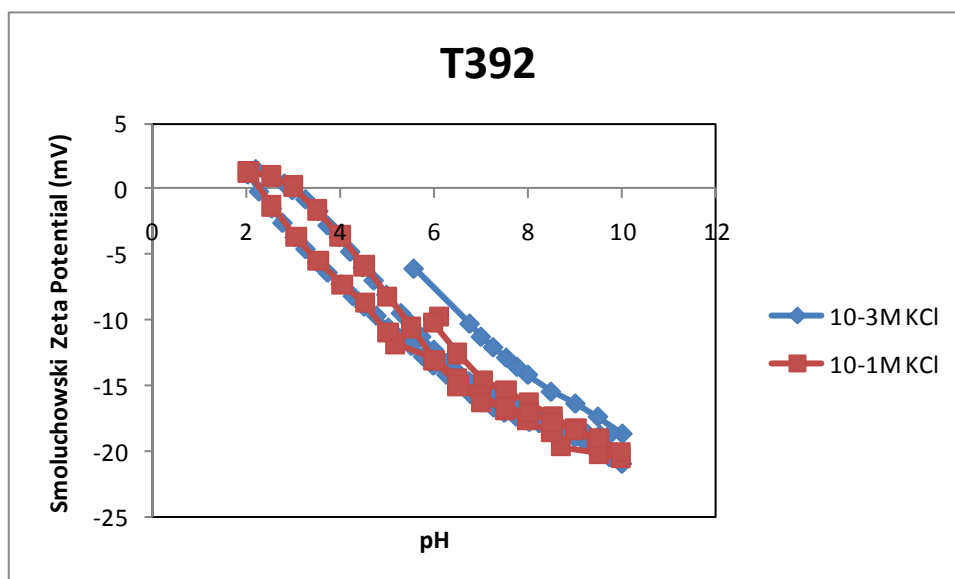


Figure 1: Particle zeta potential versus pH of 15 wt.% solid hydrocyclone O/V T392 dispersion

The particle zeta potential determined as a function of pH in the range 2-10 at room temperature for 15 wt.% solid pure silica (quartz) and hematite slurries are shown in Figures 3 and 4, respectively. It evident from Figure 3 that in the entire pH range 2-10, the quartz particles were negatively charged, the magnitude of which decreased with decreasing pH (Figure 3). An iep of  $\sim$  pH 2.0 was indicated at  $10^{-3}$  and  $10^{-1}$  M KCl ionic strength in the pH sweep, with a noticeable electrical double layer screening effect shown at the higher ionic strength.



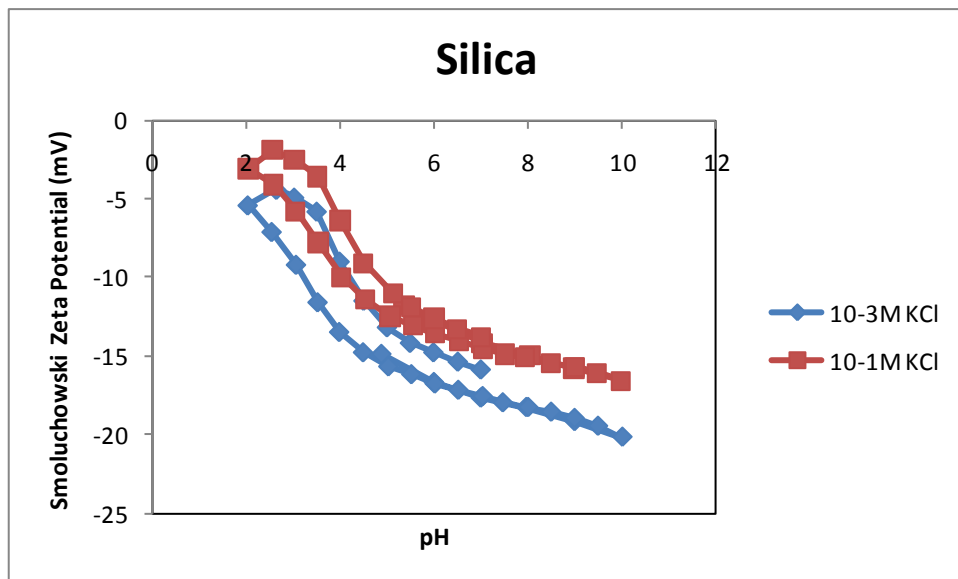


Figure 3: Particle zeta potential as a function pH of 15 wt.% silica (quartz) dispersion.

In Figure 4, it clearly shown the hematite particles exhibit an iep in the pH range 6.0-7.0, depending upon pH history and ionic strength of the KCl supernatant. Given this particle zeta and iep information of both quartz and hematite, a strategy for pH conditioning of ore sample T392 slurries for selective coagulation and flocculation was formulated.

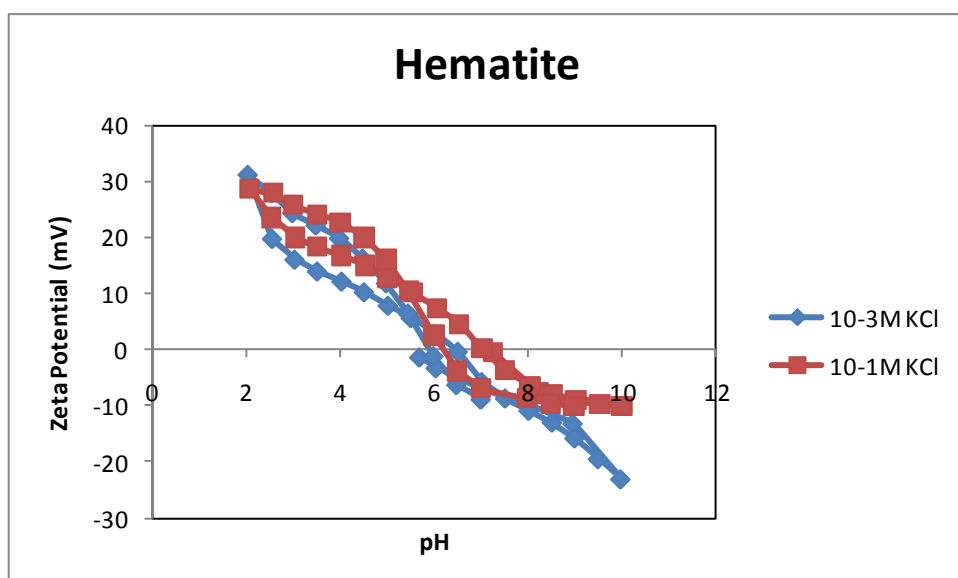


Figure 4: particle zeta potential as a function pH of 15wt.% hematite dispersion

### **Selective Coagulation**

Coagulation and settling behaviour of 2.5 wt.% T392 suspensions undergoing quiescent, isothermal batch sedimentation as a function time at 3 different pH values and at  $10^{-3}$  and  $10^{-1}$  M KCl ionic strength over 24 h are shown in Figure 5. In all cases, the photographs of cylinders with  $10^{-3}$  and  $10^{-1}$  M KCl are displayed on the left and right, respectively. Although fairly rapid settling of the very coarse particles occurred within 5 min, at no noticeable selective coagulation observed in the first one or two hours, contrary to expectation.

Over a prolonged settling period (>24 h) however, significant sedimentation finally occurred, accompanied by poor supernatant clarity. The mineralogical compositions of both the sedimented and suspended solid have not as yet been determined. As shown in the photographs, the suspension at  $10^{-3}$  M KCl at pH 10 was the most stable. At lower pH values however, the magnitude of the ionic strength of the background electrolyte had no significant effect on the selective coagulation rate.

### **Selective Flocculation**

To investigate selective flocculation, settling tests involving dilute ore T392 sample and fine, pure hematite and quartz and their 50:50 mix dispersions were undertaken. Subsequent to the starch flocculant addition, the suspensions were allowed to stand quiescently and their photographs taken at the time intervals shown below in Figure 6. The order of samples in the tubes (left to right) is: hematite, quartz, and hematite + quartz and T 392 ore samples.

The photographs shown in Figure 6 indicate that the fine model quartz particles remained dispersed whilst the model hematite particles were flocculated. This, evidently, demonstrates the strong efficacy of the corn starch to selectively flocculate hematite. For the T392 ore sample, however, no such selective flocculation of the hematite fraction was observed. Clearly, these photographs show that corn starch used is very effective in flocculating hematite from a hematite:quartz mixture but not hematite from the Roper Bar T392 ore sample.

The observed failure may, possibly, be ascribed to the poor liberation of the hematite in the current T392 ore sample. Alternatively, the incidence silica coatings onto the hematite particle surface might be a mitigating factor for the starch flocculant adsorption onto the hematite particles, suppressing bridging flocculation under the conditions where hematite is typically flocculated. The quest for the exact cause or mechanism underpinning the lack of selective starch adsorption and flocculation of the Roper Bar T392 pulp is beyond the scope of the current work, and warrants further research.




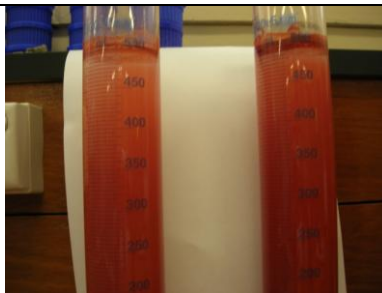
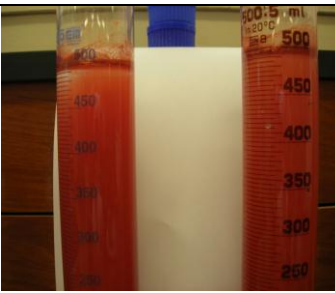
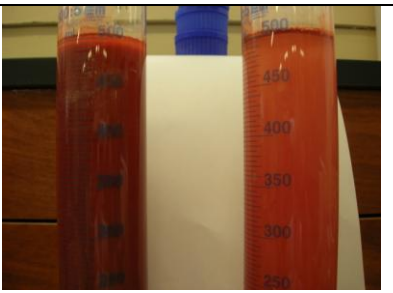
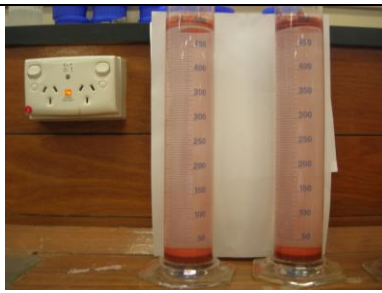
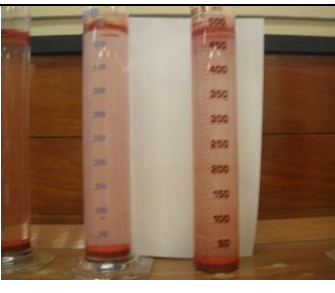

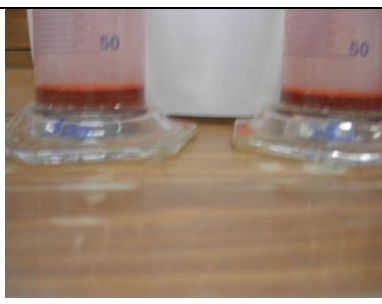

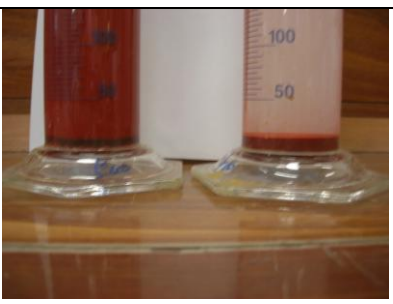
pH 2	Natural pH6-7	pH 10
Initial	Initial	Initial
		
After 1 hour	After 1 hour	After 1 hour
		
After 24 hours	After 24 hours	After 24 hours
		
Bottom after 24 hours	Bottom after 24 hours	After 24 hours
		

Figure 5: Photographs of 2.5 wt.% Roper Mine ore T392 suspensions undergoing quiescent isothermal batch sedimentation as a function time at 3 different pH values.

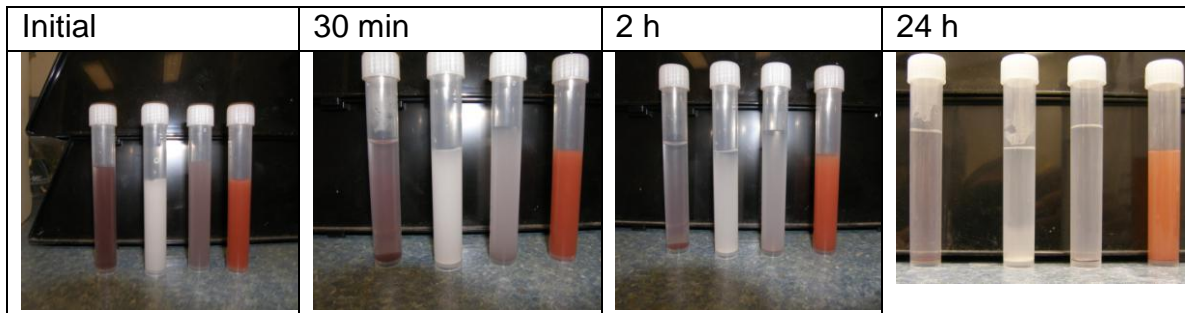


Figure 6: Photographs of 0.5 wt.% hematite, quartz, hematite + quartz and Roper mine ore T392 suspensions undergoing quiescent isothermal batch sedimentation as a function time.

## CONCLUSIONS

Particle size analysis indicated that the Roper Bar T392 hydrocyclone overflow sample used in the current work is polydispersed with particles in the size range 0.2 – 160.0  $\mu\text{m}$ . Electrokinetic zeta potential analysis of the particles which is believed to be a mixture of silica and hematite show that the interfacial chemistry is dominated by siliceous mineral species.

Selective coagulation of the ore sample was perennially slow, with pulp pH and high ionic strength of supernatant showing no marked effect, contrary to expectation. High molecular weight maize starch is demonstrated to be very effective in flocculating hematite from a hematite:quartz mixture but not hematite from the Roper Bar T392 ore sample.

## RECOMMENDATION

Based on the present outcomes, it is recommended a fresh mineral ore sample of fine particles with the right size (e.g., ( $<20 \mu\text{m}$ )) be produced for further selective coagulation and flocculation testing. It will be necessary to gather detailed, specific information on quantitative mineralogy and particle surface chemistry of the test sample, sedimented and suspended solid products upon sedimentation. Characterization techniques such as X-ray photoelectron spectroscopy (XPS), X-ray energy dispersive spectroscopy (EDAX), backscattered SEM imaging and electro-acoustic zeta potential will be of great use here.