

Publication No. 02-138-177

NEW TECHNOLOGY FOR CLAY REMOVAL

Prepared by
Jacobs Engineering Group, Inc.

under a grant sponsored by



February 2001

The Florida Institute of Phosphate Research was created in 1978 by the Florida Legislature (Chapter 378.101, Florida Statutes) and empowered to conduct research supportive to the responsible development of the state's phosphate resources. The Institute has targeted areas of research responsibility. These are: reclamation alternatives in mining and processing, including wetlands reclamation, phosphogypsum storage areas and phosphatic clay containment areas; methods for more efficient, economical and environmentally balanced phosphate recovery and processing; disposal and utilization of phosphatic clay; and environmental effects involving the health and welfare of the people, including those effects related to radiation and water consumption.

FIPR is located in Polk County, in the heart of the central Florida phosphate district. The Institute seeks to serve as an information center on phosphate-related topics and welcomes information requests made in person, or by mail, email, or telephone.

Executive Director
Paul R. Clifford

Research Directors

G. Michael Lloyd, Jr.
J. Patrick Zhang
Steven G. Richardson
Brian K. Birky

-Chemical Processing
-Mining & Beneficiation
-Reclamation
-Public Health

Publications Editor
Karen J. Stewart

Florida Institute of Phosphate Research
1855 West Main Street
Bartow, Florida 33830
(863) 534-7160
Fax: (863) 534-7165
<http://www.fipr.state.fl.us>

NEW TECHNOLOGY FOR CLAY REMOVAL

FINAL REPORT

Glenn A. Gruber
Principal Investigator

with

Charles Guan, Kerby Glass, Frank Hicks and Mike Kelahan

JACOBS ENGINEERING GROUP INC.
Lakeland, Florida USA

Prepared for

FLORIDA INSTITUTE OF PHOSPHATE RESEARCH
1855 West Main Street
Bartow, FL 33830 USA

Contract Manager: Patrick Zhang
FIPR Project Number: 99-02-138R

February 2001

DISCLAIMER

The contents of this report are reproduced herein as received from the contractor. The report may have been edited as to format in conformance with the *FIPR Style Manual*.

The opinions, findings and conclusions expressed herein are not necessarily those of the Florida Institute of Phosphate Research, nor does mention of company names or products constitute endorsement by the Florida Institute of Phosphate Research.

PERSPECTIVE

Patrick Zhang, Research Director -- Beneficiation & Mining

On February 26, 1998, FIPR held a workshop to identify research needs in phosphate mining and beneficiation. Better control of desliming cyclones and removal of clay balls/chips from the matrix/flotation feed were given high priority among suggested future research programs. The first objective of the project was to develop a viscosity-based methodology for measuring desliming efficiency of hydrocyclones for optimized operation, thus reducing phosphate loss in phosphatic clays while minimizing clay content in the flotation feed. Another objective was to break down and remove clay chips from the flotation feed using the ultrasonic technique, so that the flotation efficiency can be significantly improved. It also sought to reduce sulfuric acid usage in the de-oiling step of phosphate flotation using ultrasonic treatment.

The Clay Chip Problem

As phosphate mining moves further south, the industry faces three major problems with the phosphate matrix: dolomite contamination, lower grade, and higher clay content. Associated with the clay problem are some difficult-to-break clay chips. Current desliming practices are not adequate to break and remove these clay chips prior to flotation. The toughest chips end up in the flotation concentrate, causing higher Insol content in the product. Some clay chips become secondary slime through attrition in the conditioning and flotation steps, causing higher reagent consumption and lower flotation recovery.

The Cyclone Efficiency Issue

After being desegregated with log washers, the clay minerals are removed from the matrix using hydrocyclones. The cyclones split the feed slurry into a fine, dilute stream (the primary phosphatic clay slurry), and a coarse, thick stream (the flotation feed material). This split size is about 150 mesh. When the cyclones operate properly, they produce an overflow of approximately 3% solids. A higher solid content in the primary slime is generally an indication of significant loss of flotation feed in the phosphatic clays. On the other hand, lower solids (<1%) suggest inefficient removal of clay minerals, a nightmare for flotation engineers.

The Sulfuric Acid Usage Issue

In the current phosphate flotation process, a fatty acid/fuel oil blend is first used to float the phosphate at alkaline pHs, producing a rougher concentrate and a sand tail. The rougher concentrate must be scrubbed with sulfuric acid to remove the fatty acid and fuel oil from the mineral surfaces prior to amine flotation. The industry consumes more than 50,000 tons of sulfuric acid annually. Since sulfuric acid is not the friendliest chemical to work with, operators have a great interest in eliminating its use. But the real problem with sulfuric acid use in this case has to do with the phosphate loss it causes and its environmental implications.

ABSTRACT

A research program was performed to examine new technology for clay removal from Florida phosphate ore.

One aspect of the research tested four applications of ultrasonic treatment:

1. Sonication of pebble to remove mud balls
2. Sonication to clean the surface of flotation feed
3. Sonication to remove reagents from rougher concentrate
4. Sonication of clay slurry to accelerate consolidation

In the first three applications the response to ultrasonic treatment was positive but slight. Sonication of clay slurry appeared to have no effect on clay consolidation. Further testing of ultrasonic treatment to enhance phosphate beneficiation was curtailed because no practical benefits were demonstrated by the four applications tested.

The second aspect of the research program measured the viscosity of primary cyclone overflows from six beneficiation plants. Viscosity measurements for different samples at the same clay % solids varied significantly. A method of controlling cyclone operation using on-line viscosity measurements and pump box level sensors is proposed.

ACKNOWLEDGMENTS

The test program could not have been completed without the cooperation and assistance provided by the following companies:

- Agrifos
- Cargill Fertilizer, Inc.
- CF Industries, Inc.
- IMC Phosphates
- PCS Phosphates

Jacobs gratefully acknowledges these organizations and their personnel who assisted with the collection of samples.

TABLE OF CONTENTS

PERSPECTIVE.....	iii
ABSTRACT.....	v
ACKNOWLEDGMENTS	vi
EXECUTIVE SUMMARY	1
Ultrasonic Treatment	1
Viscosity Testing	2
Engineering Study.....	2
INTRODUCTION	3
Statement of Problem.....	3
Historical Perspective	4
Pertinent Literature and Related Work	4
Viscosity	4
Ultrasonic Treatment	5
Project Scope	6
METHODOLOGY	7
Proof of Concept Testing.....	7
The Ultrasonic Processor	7
Ultrasonic Treatment of Pebble	9
Ultrasonic Treatment of Rougher Flotation Feed	9
Ultrasonic Treatment of Rougher Concentrate	10
Ultrasonic Treatment of Clay Slurry.....	11
Field Sampling and Viscosity Testing	12
Field Sampling.....	12
Viscosity Measurement.....	12
Mineralogical Analysis and Pure Mineral Tests	12
Settling Tests.....	12
RESULTS	15

TABLE OF CONTENTS (CONT.)

Proof of Concept Testing	15
Ultrasonic Treatment of Pebble	15
Ultrasonic Treatment of Rougher Feed.....	19
Ultrasonic Treatment of Rougher Concentrate	32
Ultrasonic Treatment of Clay Slurry.....	42
Field Sampling and Viscosity Testing Results	44
Field Sampling	44
Viscosity Testing Results.....	44
Results of Settling Tests.....	53
Viscosity Control of Cyclone Separation	58
CONCLUSIONS.....	63
REFERENCES	65

LIST OF FIGURES

Figure		Page
1	Pilot Plant Configuration for Ultrasonic Treatment	8
2	Effect of Ultrasonic Treatment on Pebble Size Distribution	17
3	Effect of Ultrasonic Treatment on Pebble Size Distribution (-2 mm Material).....	18
4	Rougher Flotation Performance of HCII Feed (t) With Ultrasonic Treatment and (ut) Without Treatment	26
5	Rougher Flotation Performance of Nichols Feed (t) With Treatment and (ut) Without Treatment	27
6	Rougher Flotation Performance of Refractory Feed (t) With Ultrasonic Treatment and (ut) Without Treatment	28
7	Lab Flotation of HCII Feed, Recovery vs. Grade.....	29
8	Lab Flotation of Nichols Feed, Recovery vs. Grade	30
9	Lab Flotation of Refractory Feed, Recovery vs. Grade.....	31
10	Cleaner Flotation Performance on HCII Rougher Concentrate (t) With Ultrasonic Treatment and (ut) Without Treatment	39
11	Cleaner Flotation Performance on Nichols Rougher Concentrate (t) With Ultrasonic Treatment and (ut) Without Treatment	40
12	Cleaner Flotation Performance on 2-Stage Treated (t) and Untreated (ut) Rougher Concentrate.....	41
13	Viscosity Measurements of Cyclone Overflow Sample 1	46
14	Viscosity Measurements of Cyclone Overflow Sample 2.....	47
15	Viscosity Measurements of Cyclone Overflow Sample 3.....	48
16	Viscosity Measurements of Cyclone Overflow Sample 4.....	49
17	Viscosity Measurements of Cyclone Overflow Sample 5.....	50
18	Viscosity Measurements of Cyclone Overflow Sample 6.....	51
19	Measured and Predicted Clay Slurry Viscosities (Vs. %Solids @ 4,000/Sec. Shear Rate	55
20	A Cyclone Control Scheme	61
21	Example System Curves for Cyclone Control Scheme	62

LIST OF TABLES

Table	Page
1. Screen Analysis of Pebble, Without and With Ultrasonic Treatment.....	16
2. Rougher Flotation of HCII Feed (Without Treatment).....	20
3. Rougher Flotation of HCII Feed (With Treatment).....	21
4. Rougher Flotation of Nichols Feed (Without Treatment).....	22
5. Rougher Flotation of Nichols Feed (With Treatment).....	23
6. Rougher Flotation of Refractory Feed (Without Treatment).....	24
7. Rougher Flotation of Refractory Feed (With Treatment).....	25
8. Amine Flotation of HCII Rougher Concentrate (Without Treatment).....	33
9. Amine Flotation of HCII Rougher Concentrate (With Treatment).....	34
10. Amine Flotation of Nichols Rougher Concentrate (Without Treatment).....	35
11. Amine Flotation of Nichols Rougher Concentrate (With Treatment).....	36
12. Amine Flotation of HCII Rougher Concentrate (Without Treatment).....	37
13. Amine Flotation of HCII Rougher Concentrate (With 2-Stage Treatment).....	38
14. Settling Test Results for 12% Clay.....	42
15. Settling Test Results for 6% Clay.....	43
16. Primary Cyclone Overflow Sample Data.....	44
17. Measured Viscosities of Primary Cyclone Overflow Samples.....	45
18. Measurements of Clay Sample Specific Surface and Viscosity.....	52
19. Settling Rates of Phosphate Particles in Clay Slurries.....	57

EXECUTIVE SUMMARY

The removal of clay from phosphate pebble and flotation feed is an important aspect of phosphate ore beneficiation in Florida. Clay is a natural constituent of the sedimentary phosphate ore and must be removed by attrition and washing. If the clay is not completely removed the value of the pebble product is reduced and the cost of flotation reagents is increased.

The methods currently used to remove clay from the ore have been practiced by the Florida Phosphate Industry for more than 40 years. Log washers attrition the pebble product and wash out disaggregated clay. The flotation feed is classified at nominally 150 mesh by three or more stages of cyclones to remove clay as dilute slurry.

The Florida Institute of Phosphate Research awarded contract # FIPR 99-02-138R to Jacobs Engineering to conduct a research project entitled "New Technology for Clay Removal." An initial scope of work, as listed below, was conditionally approved for the project.

1. Ultrasonic processing
 - Proof of concept testing
 - Survey of ultrasonic applications in mineral processing (conditional)
 - Detailed testing of ultrasonic processing(conditional)
2. Viscosity testing
 - Collection of primary cyclone overflow samples from six plants
 - Viscosity measurements using a Brookfield viscometer and a Nametre 1710 Series Laboratory Viscometer (suitable for on-line measurements)
 - Settling rate measurements in clay slurries
3. Engineering study
 - Flowsheet development for ultrasonic processing (conditional)
 - Control scheme for primary cyclones utilizing on-line viscosity measurement
4. Research report

ULTRASONIC TREATMENT

Proof of concept testing, which included ultrasonic treatment of pebble, rougher feed, and rougher concentrate using a Vibrating TrayTM, was performed to determine if the full ultrasonic test program was warranted. The tray (Model VT-4608), manufactured by Advanced Sonic Processing Systems is available in larger models for commercial applications.

Ultrasonic treatment of coarse pebble (+4 mesh) and fine pebble (-4 mesh) was tested. The ultrasonic energy (1.3 - 2.6 kWh/t) partially disaggregated mud balls, but it was visually evident that ultrasonic treatment was less effective than conventional log washing.

Three samples of flotation feed were treated by ultrasonic vibration and lightly washed, then conditioned with reagents and floated in a laboratory flotation cell. Ultrasonic treatment improved the flotation performance at low dosages of reagent but did not improve performance at the reagent dosage required for acceptable performance.

Three samples of rougher concentrate were treated by ultrasonic vibration prior to acid scrubbing and rinsing (de-oiling) and cleaner flotation. Ultrasonic treatment improved de-oiling at low dosages of sulfuric acid but did not improve de-oiling at the sulfuric acid dosage required for complete de-oiling and acceptable cleaner flotation performance.

Additional testing was performed to determine if ultrasonic treatment of clay slurry would improve the rate of clay settling and consolidation. No significant differences in the initial settling rate or clay consolidation from treated and untreated samples were observed.

The proof of concept testing demonstrated that ultrasonic energy, although helpful under certain conditions, was not a practical substitute for unit operations currently practiced in Florida phosphate beneficiation. Therefore the conditionally approved scope items were not performed.

VISCOSITY TESTING

Samples of primary cyclone overflows from six plants were collected. The viscosities of these slurries were measured at adjusted solids concentrations and a correlation of slurry viscosity to clay % solids was made. The samples, as collected, varied from 3 to 5 % solids by weight. The general equation developed to predict slurry viscosity from clay % solids had to be modified for each sample. Factors such as clay mineralogical composition and surface area influence viscosity more than clay % solids. Consequently, % solids is not a reliable indicator of viscosities for different clay slurries. However, viscosity correlates very well to % solids for a specific clay slurry.

Settling rate tests performed with phosphate particles in clay slurries confirmed that Stokes' Law applies and that slurry viscosity is influential.

ENGINEERING STUDY

A scheme for controlling primary cyclone operation using on-line viscosity measurement and pump tank level control is described. The scheme has potential to minimize pumping energy and improve cyclone performance.

INTRODUCTION

STATEMENT OF PROBLEM

In 1999 the Florida phosphate industry extracted 30.2 million metric tons of phosphate rock and paid \$119 million gross severance, property, sales, and other taxes and fees (Florida Phosphate Council 2000). The recovered phosphate rock consists approximately of equal parts pebble (phosphate particles coarser than 1 mm) and flotation concentrate.

Florida phosphorite ore consists mainly of francolite pellets (phosphate) and quartz grains (sand) in a matrix of clay. Pellets of francolite are typically coarser than 0.1 mm and finer than 16 mm. The ore is initially beneficiated by washing, attritioning, and screening to recover pebble product. The material rejected by washing is prepared for flotation by removing clays (particles finer than 0.1 mm). Finally a phosphate concentrate is recovered from the feed (1 by 0.1 mm) by the Crago flotation process.

Log washers are exclusively utilized to attrition the pebble and wash out disaggregated clays. Desliming cyclones are utilized to remove disaggregated clay from the flotation feed. When hard strata of clay are in the ore, it is common for clay to remain in both the pebble product and the flotation feed as mud balls or clay chips.

Cleaning the surface of the ore particles is an important aspect of phosphate beneficiation because:

- Pebble quality is reduced if clay contaminants are not removed
- Anionic reagent costs are higher if clays are not removed from the flotation feed prior to reagent conditioning
- Cationic reagent consumption and final concentrate grade/recovery can be adversely affected by incomplete de-oiling (cleaning and removal of residual anionic reagents)

The purpose of the research program described in this report was to:

- Investigate ultrasonic treatment as an alternative to log washers, and
- Explore the potential of ultrasonic treatment to clean the particle surfaces of both flotation feed and rougher concentrate, and
- Determine which clay slurry parameter (% solids by weight or viscosity) has the most influence on particle settling velocity and hence cyclone performance.

HISTORICAL PERSPECTIVE

Clays contain minor contaminants such as Fe_2O_3 , Al_2O_3 , and sometimes MgO . When these contaminants exceed tolerable limits in the pebble product, the quality and production costs of subsequently produced fertilizers are penalized. As mining shifts to the Southern Extension reserves, it is probable that increased extraction of lower zone ore will cause pebble quality to deteriorate. The lower zone ore also has a greater clay content than upper zone ore (FIPR 1994). With inherently lower quality pebble, clay contamination will be less tolerable.

Log washers have been used exclusively in Florida plants to remove clay from pebble product. However, log washer modifications have been implemented at one plant to improve clay removal. The removal of clay from pebble by high intensity attrition scrubbing has also been demonstrated (Warneke 1998).

Clays in flotation feed are detrimental because of excessive reagent consumption attributed to their great surface area. Surface area measurements of 15 different as-received phosphate clay samples ranged from 27 to 88 square meters per gram of clay solids (USBOM 1975). Flotation reagents are a significant cost item, amounting to about \$2.31 per short ton of concentrate in 1999. Consequently, phosphate flotation plants are designed with several stages of desliming and dewatering to reject disaggregated clays from the feed prior to conditioning with flotation reagents. Clay chips are not normally removed from the feed by cyclones and can disaggregate in the conditioner and increase reagent consumption.

This research program provides the Florida phosphate industry with information concerning:

- The feasibility of utilizing ultrasonic treatment to:
 - disaggregate mud balls and clay chips,
 - clean flotation feed particle surface,
 - de-oil rougher concentrate
 - enhance clay settling,
- Clay slurry parameters that influence cyclone performance
- A control scheme utilizing on-line viscosity measurement to optimize desliming cyclone operation.

PERTINENT LITERATURE AND RELATED WORK

Viscosity

Slurry viscosity is an important process controlling parameter in mineral processing applications such as slurry pumping, heavy media separation, thickening, and cyclone size classification. The importance of slurry rheology has also been recognized

in other industries. Therefore, extensive efforts have been made to develop laboratory and on-line viscometers suitable for measuring slurries (suspensions).

Three different types of viscometers, the rotational type, the capillary type, and the vibrational type, have been applied to suspensions. Kawatra and Bakshi (1996) studied the problems associated with measuring the viscosity of suspensions for each type of viscometer.

Rotational viscometers are good for measuring viscosities at different shear rates; however, the measurements are sensitive to flow rate and changing rheology due to solids settling. As flow is essential to keep solid particles suspended in a fluid, special designs, such as baffle arrangements, are utilized to reduce swirling and turbulence in the region of the measuring device. Capillary viscometers also measure slurry viscosity at different shear rates; however plugging and slip at the capillary walls are problematic. Wall slip can result when the solids' concentration at the wall is lower than in the slurry.

Vibrating viscometers tolerate slight disturbances in material flow and are rugged enough for on-line use in suspensions. Also, vibrating viscometers are not affected by plugging and wall slip. The disadvantage of vibrating viscometers is operation (measurement) at one shear rate setting only. Many clay suspensions exhibit non-Newtonian flow and their viscosities are shear rate dependent.

The Nametre company manufactures the VISCOLINER torsional in-line viscometer used in both laboratory and industrial environments. Since VISCOLINER employs the theory of torsional oscillation to measure true viscosity, it is able to measure Newtonian and non-Newtonian materials, including solutions, suspensions, emulsions, and slurries.

The VISCOLINER unit consists of a sensor and a digital controller. The sensor, made of 316 stainless steel, has no moving parts, no seals, and is designed with a smooth, round shape, which facilitate its use in slurry applications. VISCOLINER, with a variety of outputs, is designed for process automation. This viscometer has been used for on-line viscosity measurements of a copper concentrate thickener underflow (Kawatra and Bakshi 1998), clay slurry (Nametre), and cyclone feed in a comminution circuit (Kawatra and Bakshi 1995).

Ultrasonic Treatment

Ultrasonic treatment has been found to be effective in many industrial processes. Examples include sludge dewatering, coal beneficiation, foam breaking and cleaning of jet engine parts. It has been reported that ultrasound could enhance recoveries of minerals by disagglomeration and cleaning of the particles (Sobieraj and Farmer 1993).

Advanced Sonic Processing Systems markets equipment for use in mineral processing, specifically an ultrasonic trough and an ultrasonic vibrated tray,

manufactured by the Lewis Corporation. In the case of the vibrating tray, slurry flows over the tray and is ultrasonically treated for several seconds. The acoustical energy causes cavitation of water at particle surfaces, disturbing the particle film boundary layer and accelerating the water/solid interface dynamics. Subsequent rinsing with water alone is very effective in removing many organic and inorganic surface contaminants. Additives become more aggressive as new clean particle surface area becomes available. The tray is reported as especially useful for processes that benefit from low ultrasonic intensity levels with high volume throughput requirements.

The vibrating tray is available in three standard widths, (8 inch, 18 inch & 36 inch). The 8 in. wide model is provided for laboratory experimentation, and the 36 in. wide model is built for installation in the industrial, mining, or manufacturing environments.

PROJECT SCOPE

The five approved components of the research program are:

- **Proof of Concept Testing:** to examine the potential of Ultrasonic treatment
- **Ultrasonic Applications Survey and Testing - Task 1** (conditionally approved)
- **Field Sampling and Viscosity Testing – Task 2**
- **Engineering Study –Task 3** (conditionally approved)
- **Program Report – Task 4**

Tasks 1 and 3 were conditionally approved, pending a favorable outcome from the proof of concept testing. Upon observation and review of the proof of concept testing, a steering committee selected by FIPR recommended these tasks not be executed. The research program was completed, according to schedule, in about 30 weeks after signing the contract.

METHODOLOGY

PROOF OF CONCEPT TESTING

Preliminary tests were conducted to examine ultrasonic treatment of pebble, flotation feed and rougher concentrate. The effectiveness of the ultrasonic treatments were determined as follows:

1. The degree of clay ball disaggregation caused by ultrasonic treatment of pebble samples was measured by sieve analyses.
2. The change in flotation response and reagent consumption from ultrasonic treatment of a flotation feed sample was determined by batch flotation tests.
3. The possible reduction in H₂SO₄ consumption for de-oiling and the subsequent cleaner flotation response caused by ultrasonic treatment of a rougher concentrate sample was determined by laboratory batch testing.

The following samples were collected and prepared for testing:

- **Pebble**
 - feed to the Hardee Complex II No. 2 log washer
 - +4 mesh fraction, and
 - -4 mesh fraction
- **Rougher Flotation Feed**
 - feed to the Hardee Complex II rougher conditioners
 - feed to the Nichols rougher conditioners
 - refractory flotation feed, prepared in Jacobs core washer
- **Rougher Concentrate**
 - Hardee Complex II rougher concentrate
 - two samples
 - Nichols rougher concentrate

The Ultrasonic Processor

The ultrasonic treatment tests were carried out using a Vibrating TrayTM, Model VT-4608, by Advanced Sonic Processing Systems. The general arrangement of the experimental tray for ultrasonic treatment of the samples is shown in Figure 1. The three major components of the Vibrating Tray system are a feeder, a vibrating tray and the ultrasonic generator.

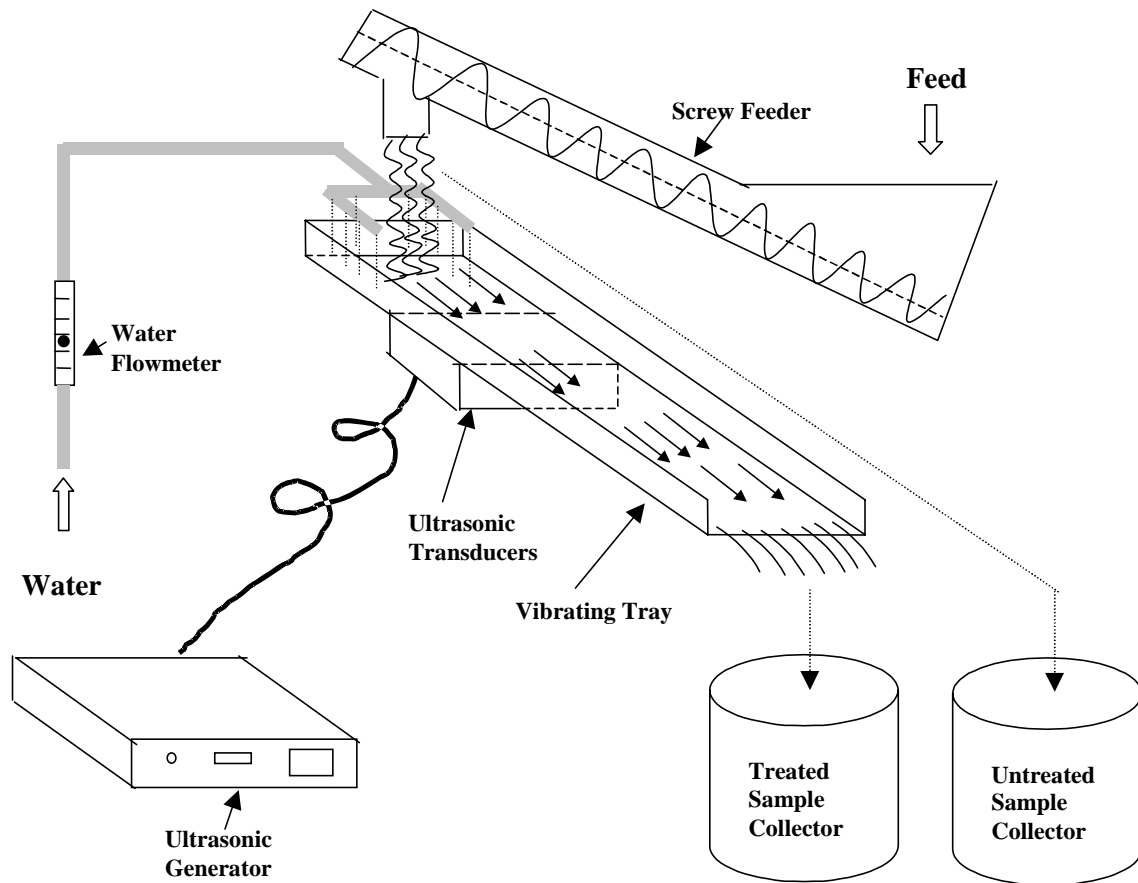


Figure 1. Pilot Plant Configuration for Ultrasonic Treatment.

1. The vibrating tray is a flat resonator with an acoustically energized zone near the feed end. This energized zone, located directly above the transducer, is visible via micro streaming of the carrier solution. The tray length is also acoustically energized thus offering additional capabilities to clean the surfaces of the particles.
2. The ultrasonic generator converts the incoming 60 Hz power into 20,000 Hz ultrasonic power. The generator is equipped with a variable power feature to adjust the ultrasonic energy to the tray.
3. A screw feeder is used to deliver the samples to the inlet of the tray, where tap water is also added to create a uniform slurry flow on the tray.

For all tests, the ultrasonic power was set at the maximum of 500 to 520 Watts. The ultrasonic frequency, also set at its maximum, ranged from 19,270 to 19,450 Hz. The incline angle of the tray was installed at 11 degrees, unless stated otherwise.

Ultrasonic Treatment of Pebble

About 500 pounds of pebble (log washer feed) were collected from CF Industries' Hardee Complex II. The "as received" pebble sample was carefully screened into two fractions, namely +4 mesh and -4 mesh. The +4 mesh and -4 meshed were treated separately using the ultrasonic processor, because it was recognized that the +4 mesh would not be fully submerged and therefore sonication would be less effective. For effective sonication, the particles should be fully submerged in water. Commercial scale vibrating trays accommodate a maximum slurry depth of about 25 mm. The test unit was limited to a slurry depth of about 5 mm, and therefore particles coarser than 4 mesh could not be fully submerged in water on the inclined tray.

The +4 mesh sample was screw fed to the tray and water addition was controlled by a flow meter. The feed rates were about 6.5 pounds/min solids and 4.5 gallons/min water. Five untreated samples were taken at the screw feeder discharge and five treated samples were collected at the tray discharge. The time interval between samples was about 1 minute. The sampling time for each sample, about 15 seconds, was recorded using a stopwatch. The total test time for each sample was about 10 minutes.

Each sample was weighed and then dewatered on a 150 mesh screen. The liquid passing the screen was flocculated, and the flocs were collected, dried, and weighed. The dried + 150 mesh samples, from both before and after ultrasonic treatment, were subjected to size distribution analysis to find out if ultrasonic treatment caused more disaggregation of clay balls.

Similar procedures were tested for the - 4 mesh material.

Ultrasonic Treatment of Rougher Flotation Feed

Three rougher feed samples were tested. Two samples were rougher flotation feed collected directly from Hardee Complex II and the Nichols beneficiation plant. One sample was prepared from run-of-mine (ROM) ore collected from the CF mine. The latter sample was selected because it was considered tough or refractory rougher feed. The ROM ore sample was washed in Jacobs' Laboratory to obtain the rougher feed for the test.

About 200 pounds of each rougher flotation feed sample were fed to the tray using the screw feeder. The feed rates were about 14.9 pounds/minute solids and 4.4 gallons/minute water. Eight untreated samples and eight treated samples were collected at about a half minute interval in an alternate manner such that an untreated sample followed

by a treated sample, then another untreated sample, and so on. The sampling times, targeted at 15 seconds, was measured accurately using a stopwatch.

The samples were weighted and then decanted on a 150 mesh screen. Decants were flocculated and the contained solids were collected for determination of solids in the -150 mesh fraction. The + 150 mesh untreated samples were thoroughly mixed and ten (10) rougher flotation charges (1,211 g wet) were cut out. One charge was used to determine the percent solids and remaining nine were used for flotation tests. Similar procedures were followed for the ultrasonic treated + 150 mesh samples.

Bench flotation tests on the rougher feed samples, prepared with and without ultrasonic treatment, were used to evaluate the effectiveness of ultrasonic equipment to remove clays and clean the feed particle surfaces. Each flotation charge was jet rinsed three times. Excess water and slimes were decanted from treated and untreated feeds through a 200 mesh screen. Flotation charges were conditioned with soda ash and fatty acid in a conditioner for two and a half minutes at about 70% solids, then floated for one and a half minutes in a Denver laboratory cell. The individual flotation concentrates and tailings were dewatered and weighted. Chemical analyses were conducted for both the concentrates and the tailings.

Four fatty acid dosages were tested for untreated and the treated samples. Flotation performance curves, showing %BPL recovery and concentrate %BPL as a function of fatty acid dosage, for untreated and ultrasonic treated samples were established to evaluate of the effectiveness of the ultrasonic treatment. The data were also plotted as recovery versus grade graphs to examine differences in flotation performance for treated and untreated flotation feeds.

Ultrasonic Treatment of Rougher Concentrate

Three rougher concentrate samples were tested. Two samples were collected directly from Hardee Complex II, and one from the Nichols plant. The second Hardee Complex II sample was used to test two-stage ultrasonic treatment. The tests were conducted immediately after the samples were collected, to minimize possible changes to the reagentized particles' surface.

About 200 pounds of each rougher concentrate were fed to the tray using the screw feeder. The feed rates were about 10.64 pounds/minute solids and 4.7 gallons/minute water. Five untreated samples and five treated samples were collected at about a half minute interval in a alternate manner such that an untreated sample followed by a treated sample, then another untreated sample, and so on. The sampling time, targeted at 15 seconds, was measured using a stopwatch. For the two-stage ultrasonic treatment test, only the five untreated samples were taken in the first stage. The first stage treated rougher concentrate was collected and fed back to the tray for the second time. The five second-stage treated samples were collected during the second run.

Both treated and untreated samples were weighted and decanted on a 150 mesh screen. Decants were flocculated and the contained solids were collected for determination of solids in the -150 mesh fraction. The +150 mesh untreated samples were thoroughly mixed. Eleven de-oiling charges (613 g wet) were cut out. One charge was used to determine the percent solids and remaining 10 charges were used for de-oiling and cleaner flotation tests. Similar procedures were for the +150 mesh ultrasonic treated samples.

Bench de-oiling tests and cleaner flotation tests on the charges prepared with and without ultrasonic treatment, were used to evaluate the effectiveness of ultrasonic equipment to de-oil the concentrate.

Each sample was de-oiled by acid scrubbing for three minutes at about 65% solids. The spent reagent and sulfuric acid were removed by jet rinsing. Excess water was decanted through a 150 mesh screen. The de-oiled rougher concentrate was transferred to a flotation cell and diluted with water. Fixed amounts of amine and fuel oil were added and the pulp was conditioned without aeration for about ten seconds. Then the air valve was opened and the pulp was floated for two minutes. The cleaner concentrates and tailings were dewatered and weighted. Chemical analyses were conducted for both the concentrates and the tailings.

Five sulfuric acid dosage levels, ranging from ambient pH to pH 3.0 were tested for untreated and treated de-oiling charges. Flotation performance curves, showing %BPL recovery and concentrate %BPL as a function of sulfuric acid dosage for untreated and ultrasonic treated samples were established as an aid to evaluate the effectiveness of ultrasonic treatment for de-oiling rougher concentrate.

The results of the above preliminary testing were presented to a steering committee selected by FIPR for the purpose of recommending completion or stopping the proposed ultrasonic program.

Ultrasonic Treatment of Clay Slurry

Clay slurry was pumped from a mixing tank to the vibrating tray at about 3 gallons/minute. Ultrasonic treated samples of clay slurry were collected at the end of the energized tray for 30 seconds. An untreated sample was also taken at the end of the tray, but after the ultrasonic power was turned off for 15 seconds. The trough angle for the treatment of clays was set at 5 degrees.

Comparing the settling rates of treated clay and untreated clay assessed the effect of the ultrasonic treatment on clay slurry. The samples ultrasonic treated clay and untreated clay were placed into graduated glass cylinders. The mud line heights in the cylinders were recorded as a function of time. The total time for the settling test comparison was about five weeks.

FIELD SAMPLING AND VISCOSITY TESTING

Field Sampling

Primary cyclone overflow samples from the following six plants were collected.

1. Nichols
2. Hardee Complex II
3. Ft. Green
4. Hookers Prairie
5. Kingsford
6. Swift Creek

The six primary cyclone overflow samples were allowed to thicken at Jacobs laboratory. Each overflow sample was segregated into two components; a concentrated slurry, and a clear decant. Samples of each overflow, varying in solids content from about 3 to 12 percent were then prepared by mixing the concentrated slurry with the decant according to proper ratios.

Viscosity Measurement

Viscosity measurements were performed with a Brookfield Dial Viscometer and a Nametre Viscometer. The rotational type Brookfield viscometer measures slurry viscosity at low shear rates from 0.066 to 13.2 second⁻¹. The vibrating cylinder type Nametre viscometer measures slurry viscosity at a single shear rate, 4,000 second⁻¹. The test temperature was controlled at 25±0.5 °C. Viscosity vs. shear rate curves, were constructed for each cyclone overflow sample, adjusted to different percent solids.

Mineralogical Analysis and Pure Mineral Tests

Samples of the cyclone overflows were also delivered to FIPR for mineralogical analysis and surface area measurements. Similarly, technical quality samples of clay (palygorskite, kaolinite and montmorillonite) were examined to determine the viscosity as a function of solids content and mineral species.

Settling Tests

Settling tests using phosphate particles were performed to correlate settling velocity as a function of the carrier fluid viscosity and percent solids. The phosphate particles used were obtained from sieve fractions of a phosphate concentrate available in Jacobs' laboratory. The particle settling velocity in the clay slurries was measured using a 3.75" diameter by ~50" tall PVC cylinder with a conical bottom for drainage of the

slurry. Because the settling particles of phosphate could not be seen in the opaque clay slurries, an indirect procedure was used to measure particle settling velocity.

The slurry was mixed thoroughly, poured into the cylinder with the drain valve closed. Phosphate grains were added at the top of the cylinder and timer was started simultaneously. After the particles were allowed to settle in the slurry for a predetermined time interval, the drain valve was opened and slurry was drained onto a screen. When the particles appeared on the screen, the slurry level (h_2) was marked and the timer was stopped simultaneously.

The equivalent static column of slurry traversed by the settled particles consisted of cylinder portion and the conical portion. The cylinder portion was obtained from the direct measurement of h_2 . The conical portion was calculated as 4.21 cm, taking an equivalent height of the conical volume to a same cylinder volume. Then, the settling velocity was obtained by dividing the total equivalent slurry column ($h_2+4.21$ cm) by the total settling time.

RESULTS

PROOF OF CONCEPT TESTING

Ultrasonic Treatment of Pebble

Although the +4 mesh pebble particles were larger than optimum for the test vibrating tray, sonication did liberate some feed and clay from the pebble. The size distributions for ultrasonic treated and untreated samples are compared in Table 1 and plotted in Figures 2 and 3. It can be seen that the -14+150 mesh fraction was increased by 0.68% by sonication, while the -150 mesh fraction was increased by 1.58%. The -4 mesh pebble particle size was suitable for the vibrating tray. The -150 mesh fraction was increased by 1.19% by the ultrasonic treatment. The results are given in Table 2 and also shown in Figures 2 and 3. The power usage for the treatment of the pebble samples were in the range of 1.3 to 2.6 kWh/t.

The results confirm that the ultrasonic treatment can decompose clay balls and aggregates to some extent. However, clay balls and aggregates were visually abundant in the pebble samples after sonication. Based on the observed results, ultrasonic treatment is considered less effective than the traditional log washers for removing clay from pebble.

Table 1. Screen Analysis of Pebble, without and with Ultrasonic Treatment

Project No.: 28-V938-00

Date 5/24/00

Test: Ultrasonic Testing of Pebble

Sample Name: HCII Log Washer Feed

Watts 510- 520

Trough angle, degree 11

Frequency 1927

+4 Mesh Fraction of Pebble

	Untreated							Treated							Difference
	weight in grams						Avg. Wt. %	weight in grams						Avg. Wt. %	
	1	2	3	4	5	Avg.		1	2	3	4	5	Avg.		
>3/4"	48.3	73.4	92.3	102.9	46.6	72.7	5.80	21.8	26.7	21	86.7	8.8	33.00	4.49	-1.32
-3/4 + 1/2"	64	160.2	136.6	73.6	107.1	108.3	8.65	20.5	49.9	69.4	89.1	105.6	66.90	9.10	0.45
-1/2" + 4 mesh	650.6	651.2	548.1	528.4	570.4	589.74	47.09	237.2	345.5	345.9	403.9	455.6	357.62	48.62	1.54
-4+8 mesh	353.8	154.8	157.1	138.5	185.3	197.9	15.80	50.6	124.2	95.9	120	125.8	103.30	14.04	-1.76
-8+14 mesh	285	129.5	174.5	160.2	157.8	181.4	14.48	51.6	101.8	92.6	113.7	129.5	97.84	13.30	-1.18
-14 + 35 mesh	41.6	39.3	50.5	52.4	37.8	44.32	3.54	17.3	18.7	27.5	32.4	35.1	26.20	3.56	0.02
-35 + 65 mesh	20.5	23.8	24.7	25.6	26.9	24.3	1.94	14.7	13	18.3	19.3	24.4	17.94	2.44	0.50
-65+150 mesh	15.9	13.3	19.3	18.7	18.8	17.2	1.37	9.7	8.9	10	11.7	16.1	11.28	1.53	0.16
-150 mesh	14.2	16.2	18.2	18.4	16.2	16.64	1.33	16.3	18.7	19.4	26.8	25.9	21.42	2.91	1.58
	1493.9	1261.7	1221.3	1118.7	1166.9	1252.5	100.00	439.7	707.4	700	903.6	926.8	735.50	100.00	

-4 Mesh Fraction of Pebble

	Untreated							Treated							Difference
	weight in grams						Avg. Wt. %	weight in grams						Avg. Wt. %	
	1	2	3	4	5	Avg.		1	2	3	4	5	Avg.		
-4+8 mesh	410.7	448.8	386	466.2	498.5	442.04	49.47	2015.1		560.7			515.16	49.22	-0.25
-8+14 mesh	483.2	388.5	428.2	436	397.3	426.64	47.75	1834.5		613.5			489.60	46.78	-0.96
-14 + 35 mesh	11.5	15.9	20.5	22.5	15.7	17.22	1.93	71.4		34.3			21.14	2.02	0.09
-35 + 65 mesh	1.1	0.8	0.6	3.2	5.6	2.26	0.25	10.7		0.9			2.32	0.22	-0.03
-65+150 mesh	1.2	0.9	0.8	3.7	4	2.12	0.24	9.5		0.9			2.08	0.20	-0.04
-150 mesh	2.4	2.4	2.6	5	4	3.28	0.37	62.8		18.5			16.26	1.55	1.19
	910.1	857.3	838.7	936.6	925.1	893.56	100.00	4004.0	0	1228.8	0	0	1046.56	100.00	

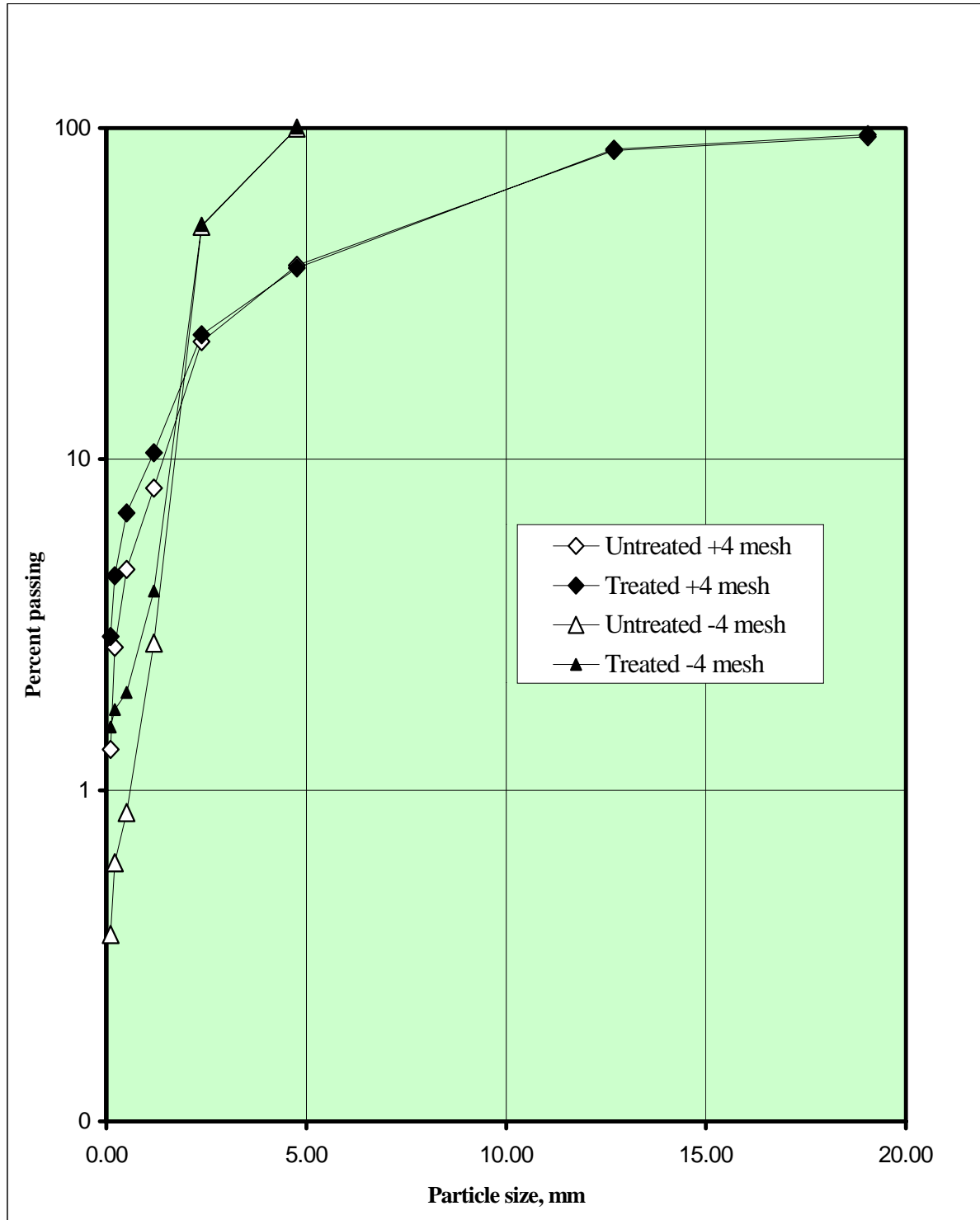


Figure 2. Effect of Ultrasonic Treatment on Pebble Size Distribution.

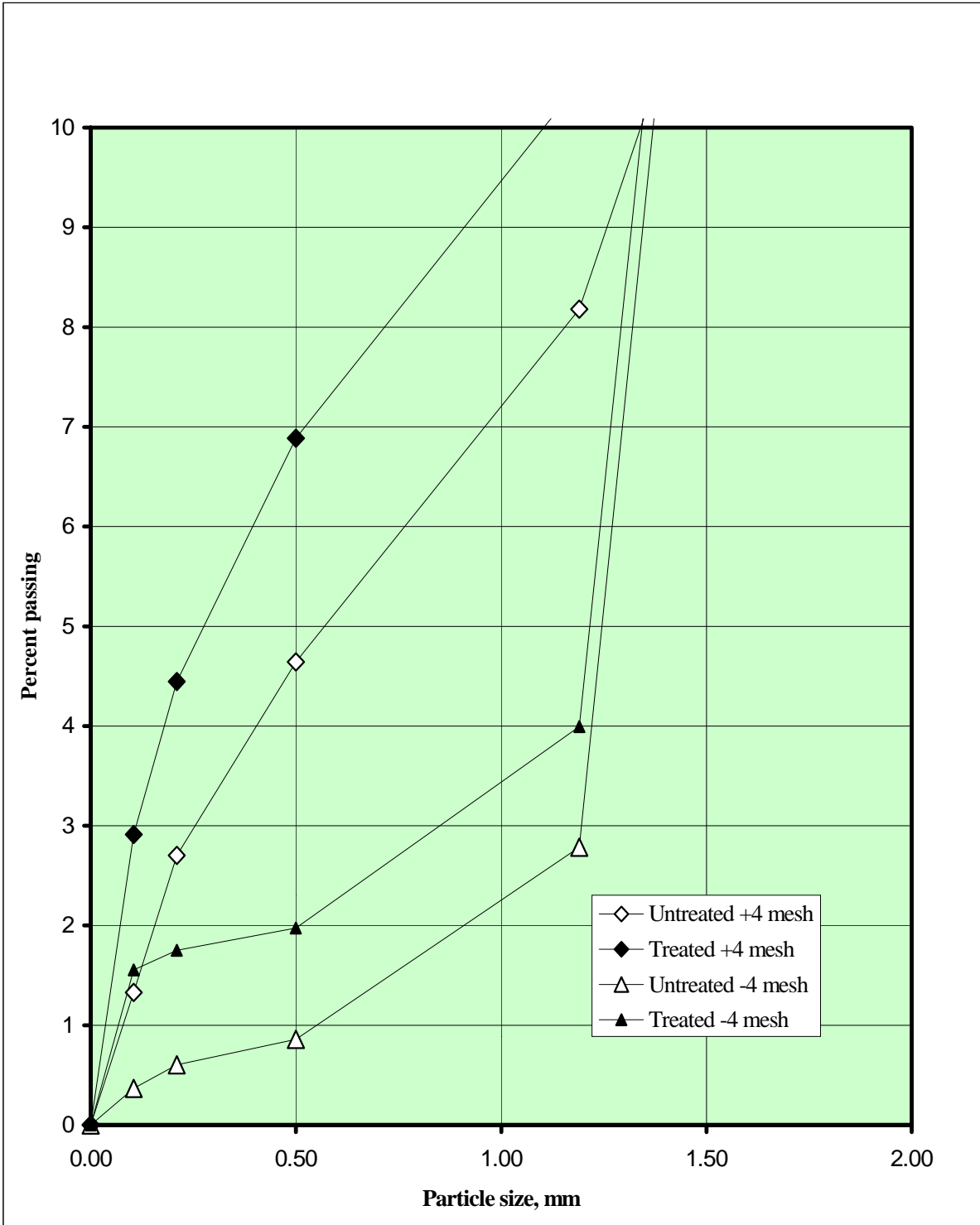


Figure 3. Effect of Ultrasonic Treatment on Pebble Size Distribution (-2 mm Material).

Ultrasonic Treatment of Rougher Feed

Flotation results at different levels of collector dosage for the three untreated rougher feed samples are presented respectively in Tables 2, 4, and 6. Similarly, flotation results for ultrasonic treatment of these three rougher feed samples at different levels of collector dosage are shown in Tables 3, 5, and 7. The flotation test data for each feed, with and without ultrasonic treatment, are compared on Figures 4, 5, and 6. The data show that %BPL recovery increases with collector dosage for treated and untreated feed. At the collector dosage levels required for economic operation (high recovery) there is no appreciable difference in recovery between treated and untreated feeds. At low collector dosage levels the ultrasonically treated feeds gave higher but less than satisfactory BPL recovery. Reagent consumption, %BPL recovery and concentrate %BPL were not significantly improved by ultrasonic treatment.

The clays removed from two rougher feed samples, with and without ultrasonic treatment is tabulated below. The data show that the amount of clays removed from the treated feeds were not consistently or significantly more than for untreated feeds. Therefore both flotation performance and the amount of clay removed indicate the ultrasonic treatment of rougher flotation feed was not practically effective.

	Untreated Feed	Ultrasonically Treated Feed
Rougher feed #1	6.27 gm	7.00 gm
Rougher feed #2	0.51 gm	0.44 gm
Average	3.39 gm	3.72 gm

Concentrate BPL recoveries were plotted against concentrate %BPL for each feed sample, as shown in Figures 7, 8, and 9. A common characteristic of these plots is that at lower fatty acid dosage, the ultrasonic treatment resulted in higher BPL recovery and grade than untreated samples. This confirms that ultrasonic may improve rougher flotation recovery and concentrate BPL grade, but only at starvation levels of fatty acid.

Table 2. Rougher Flotation of HCII Feed (Without Treatment).

3 jet rinses, Soda ash to pH 9.6, 2.5 minutes conditioning; 1.5 minutes flotation

0.34 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	154.30	31.23	6.01	68.19	62.31	1.23	15.48
Tails	842.50	3.46	88.05	7.55	37.69	98.77	84.52
Feed	996.80	7.76	75.35	16.94	100.00	100.00	100.00

0.60 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	232.80	28.70	13.11	62.66	89.78	4.02	23.43
Tails	760.70	1.00	95.75	2.18	10.22	95.98	76.57
Feed	993.50	7.49	76.39	16.36	100.00	100.00	100.00

0.85 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	258.60	28.06	16.33	61.27	92.77	5.66	26.04
Tails	734.40	0.77	95.92	1.68	7.23	94.34	73.96
Feed	993.00	7.88	75.19	17.20	100.00	100.00	100.00

1.11 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	281.20	26.59	19.44	58.06	95.36	7.28	28.26
Tails	714.00	0.51	97.51	1.11	4.64	92.72	71.74
Feed	995.20	7.88	75.45	17.20	100.00	100.00	100.00

Table 3. Rougher Flotation of HCII Feed (With Treatment).

3 jet rinses, Soda ash to pH 9.6, 2.5 minutes conditioning; 1.5 minutes flotation

0.34 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	149.70	31.37	6.46	68.49	64.84	1.25	14.95
Tails	851.50	2.99	90.08	6.53	35.16	98.75	85.05
Feed	1001.20	7.23	77.58	15.79	100.00	100.00	100.00

0.60 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	236.90	28.75	12.63	62.77	89.97	3.94	23.79
Tails	759.00	1.00	95.99	2.18	10.03	96.06	76.21
Feed	995.90	7.60	76.16	16.60	100.00	100.00	100.00

0.85 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	259.30	27.28	18.10	59.56	93.46	6.15	25.99
Tails	738.50	0.67	96.93	1.46	6.54	93.85	74.01
Feed	997.80	7.59	76.44	16.56	100.00	100.00	100.00

1.11 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	273.70	25.81	20.26	56.35	95.01	7.27	27.36
Tails	726.80	0.51	97.32	1.11	4.99	92.73	72.64
Feed	1000.50	7.43	76.24	16.23	100.00	100.00	100.00

Table 4. Rougher Flotation of Nichols Feed (Without Treatment).

3 jet rinses, Soda ash to pH 9.6, 2.5 minutes conditioning; 1.5 minutes flotation

0.34 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	243.60	30.19	9.34	65.92	89.64	2.85	23.13
Tails	809.70	1.05	95.75	2.29	10.36	97.15	76.87
Feed	1053.30	7.79	75.77	17.01	100.00	100.00	100.00

0.60 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	275.50	28.47	16.57	62.16	96.39	5.70	26.30
Tails	772.10	0.38	97.75	0.83	3.61	94.30	73.70
Feed	1047.60	7.77	76.40	16.96	100.00	100.00	100.00

0.85 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	273.20	28.76	16.02	62.79	97.14	5.47	26.18
Tails	770.50	0.30	98.15	0.66	2.86	94.53	73.82
Feed	1043.70	7.75	76.65	16.92	100.00	100.00	100.00

1.11 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	293.80	26.89	20.52	58.71	97.26	7.70	28.39
Tails	740.90	0.30	97.58	0.66	2.74	92.30	71.61
Feed	1034.70	7.85	75.70	17.14	100.00	100.00	100.00

Table 5. Rougher Flotation of Nichols Feed (With Treatment).

3 jet rinses, Soda ash to pH 9.6, 2.5 minutes conditioning; 1.5 minutes flotation

0.34 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	234.20	31.39	7.85	68.54	92.02	2.29	22.27
Tails	817.40	0.78	96.10	1.70	7.98	97.71	77.73
Feed	1051.60	7.60	76.45	16.59	100.00	100.00	100.00

0.60 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	274.90	28.04	16.90	61.22	96.63	5.80	26.33
Tails	769.20	0.35	98.10	0.76	3.37	94.20	73.67
Feed	1044.10	7.64	76.72	16.68	100.00	100.00	100.00

0.85 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	297.90	26.51	21.18	57.88	97.24	7.93	28.51
Tails	747.10	0.30	98.00	0.66	2.76	92.07	71.49
Feed	1045.00	7.77	76.10	16.97	100.00	100.00	100.00

1.11 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	318.00	25.99	22.64	56.75	97.56	9.03	30.11
Tails	738.10	0.28	9.32	0.61	2.44	90.97	69.89
Feed	1056.10	8.02	75.53	17.51	100.00	100.00	100.00

Table 6. Rougher Flotation of Refractory Feed (Without Treatment).

3 jet rinses, Soda ash to pH 9.6, 2.5 minutes conditioning; 1.5 minutes flotation

0.34 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	82.00	29.62	4.51	64.67	23.84	0.57	8.26
Tails	910.60	8.52	70.28	18.60	76.16	99.43	91.74
Feed	992.60	10.26	64.85	22.41	100.00	100.00	100.00

0.60 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	390.70	25.54	13.59	55.76	95.60	8.28	38.96
Tails	612.20	0.75	96.08	1.64	4.40	91.72	61.04
Feed	1002.90	10.41	63.94	22.72	100.00	100.00	100.00

0.85 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	405.20	25.05	15.87	54.69	96.83	10.07	40.60
Tails	592.90	0.56	96.85	1.22	3.17	89.93	59.40
Feed	998.10	10.50	63.97	22.93	100.00	100.00	100.00

1.11 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	428.90	23.68	19.83	51.70	97.86	13.28	42.98
Tails	569.10	0.39	97.60	0.85	2.14	86.72	57.02
Feed	998.00	10.40	64.18	22.71	100.00	100.00	100.00

Table 7. Rougher Flotation of Refractory Feed (With Treatment).

3 jet rinses, Soda ash to pH 9.6, 2.5 minutes conditioning; 1.5 minutes flotation

0.34 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	241.30	28.54	6.24	62.31	65.94	2.37	24.37
Tails	748.90	4.75	82.70	10.37	34.06	97.63	75.63
Feed	990.20	10.55	64.07	23.03	100.00	100.00	100.00

0.60 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	347.00	27.26	10.46	59.52	90.05	5.72	35.12
Tails	641.00	1.63	93.30	3.56	9.95	94.28	64.88
Feed	988.00	10.63	64.21	23.21	100.00	100.00	100.00

0.85 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	396.70	25.30	15.13	55.24	96.46	9.48	40.06
Tails	593.50	0.62	96.52	1.35	3.54	90.52	59.94
Feed	990.20	10.51	63.91	22.94	100.00	100.00	100.00

1.11 ml Fatty acid/fuel oil

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	%Weight
Concentrate	430.20	23.78	19.53	51.92	97.85	13.35	43.31
Tails	563.20	0.40	96.84	0.87	2.15	86.65	56.69
Feed	993.40	10.52	63.36	22.98	100.00	100.00	100.00

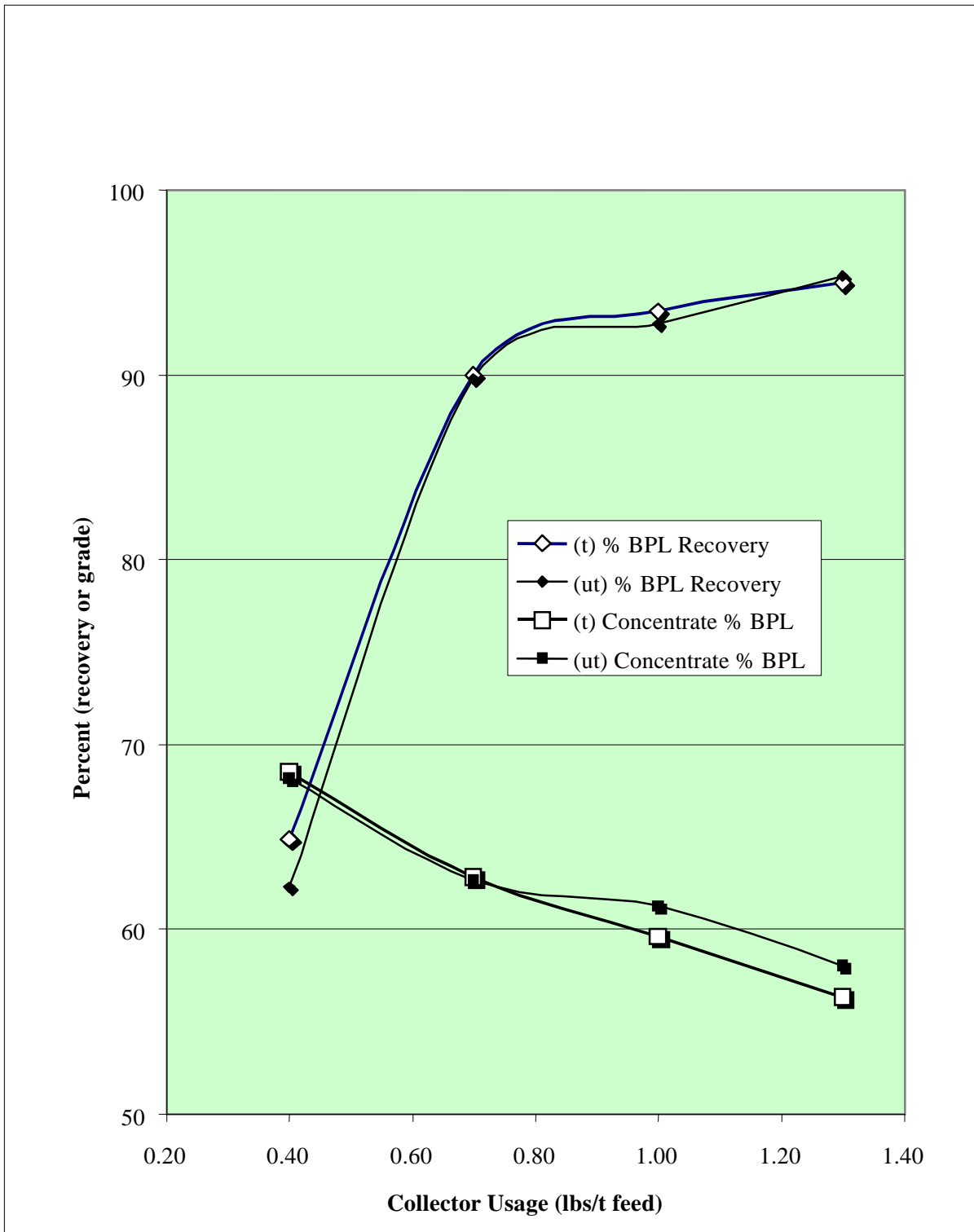


Figure 4. Rougher Flotation Performance of HCII Feed (t) With Ultrasonic Treatment and (ut) Without Treatment.

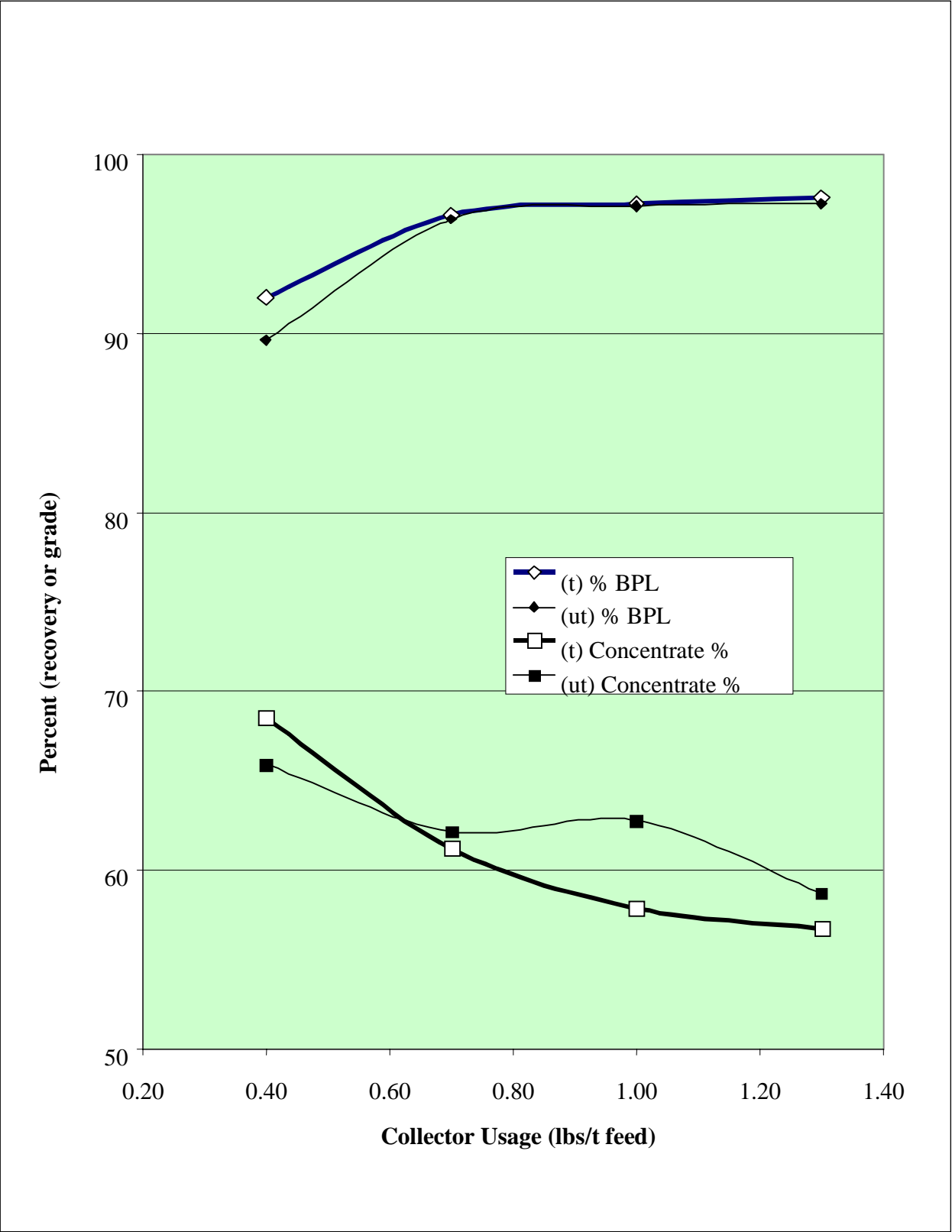


Figure 5. Rougher Flotation Performance of Nichols Feed (t) With Treatment & (ut) Without Treatment.

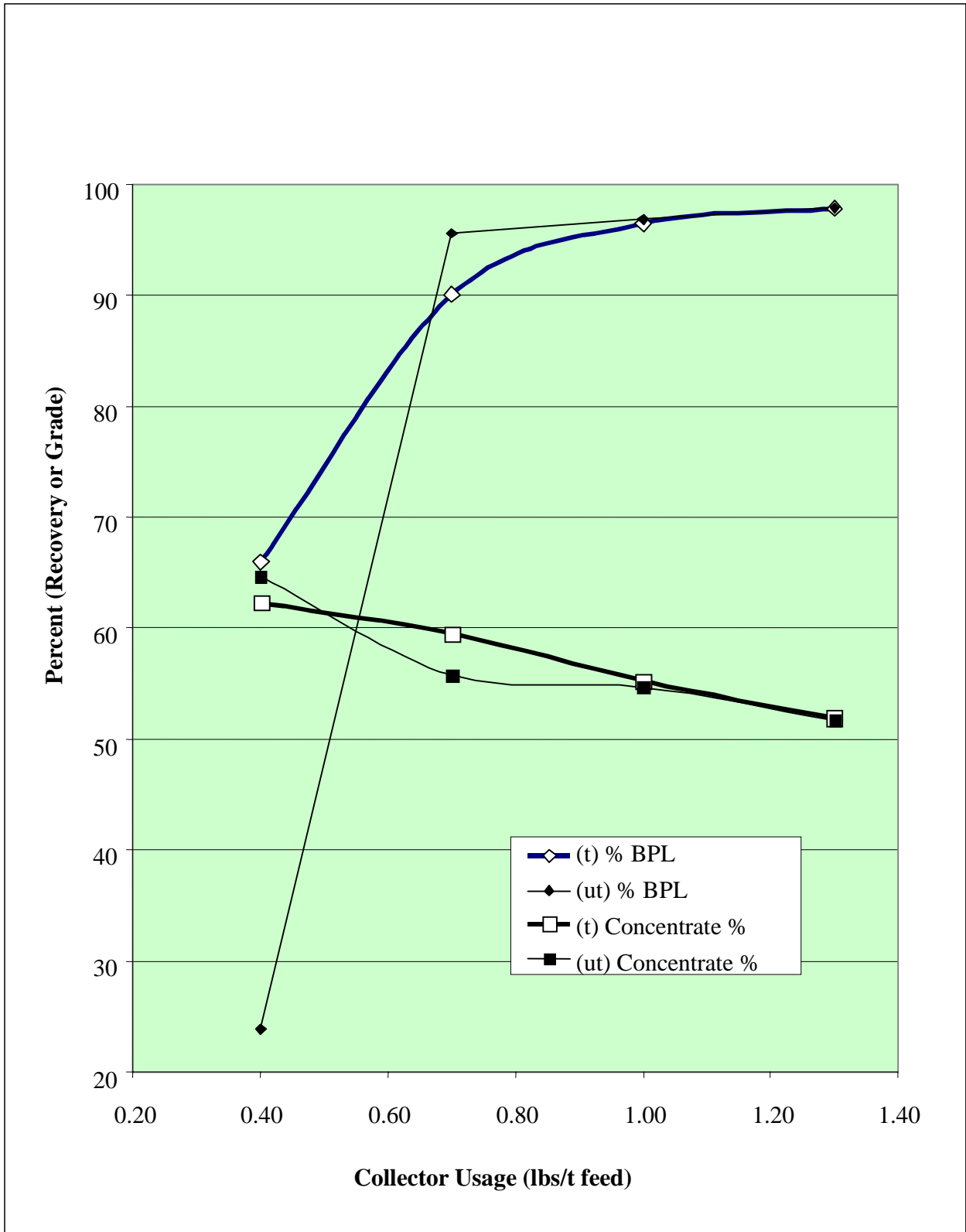


Figure 6. Rougher Flotation Performance of Refractory Feed (t) With Ultrasonic Treatment & (ut) Without Treatment.

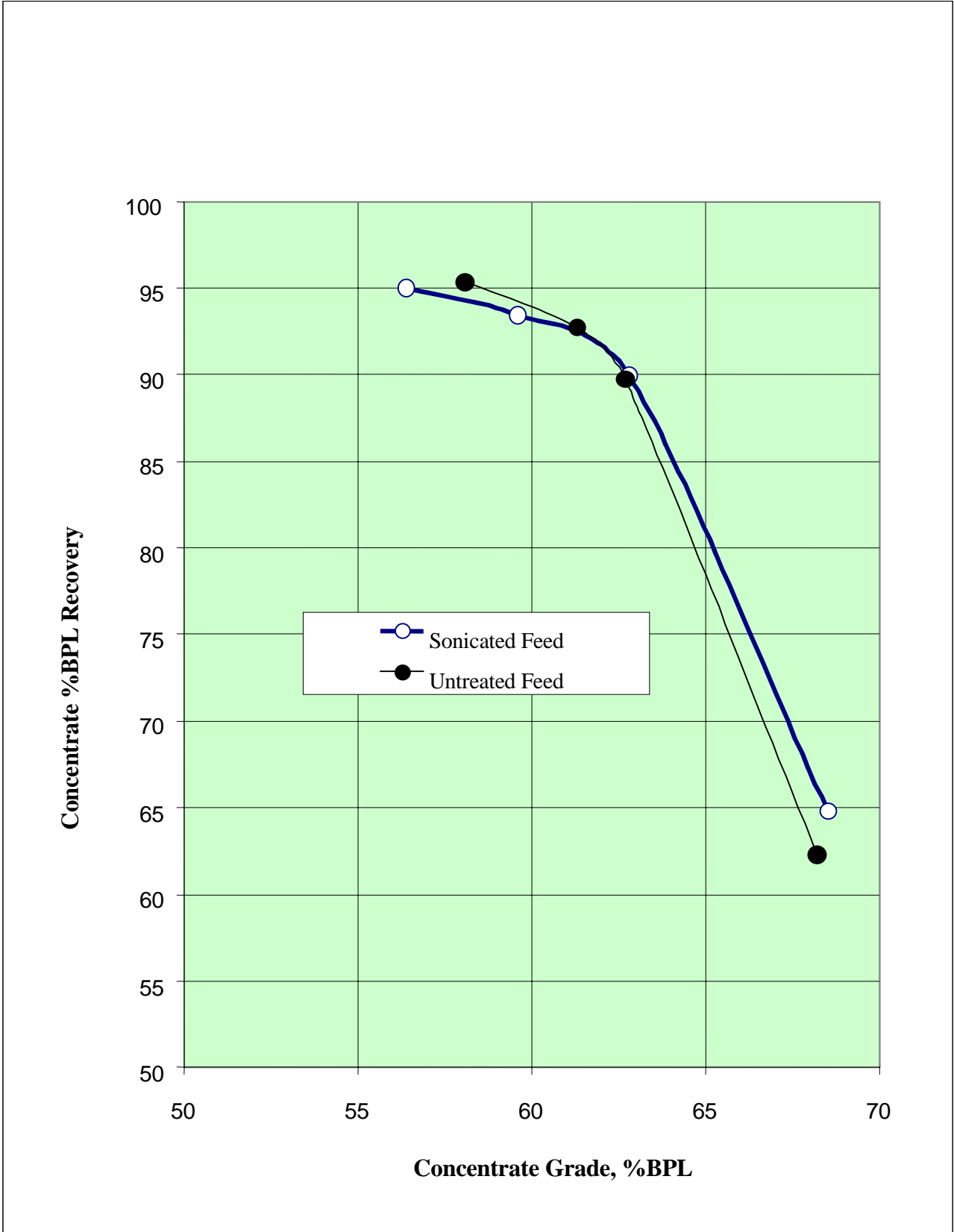


Figure 7. Lab Flotation of HCII Feed, Recovery Vs. Grade.

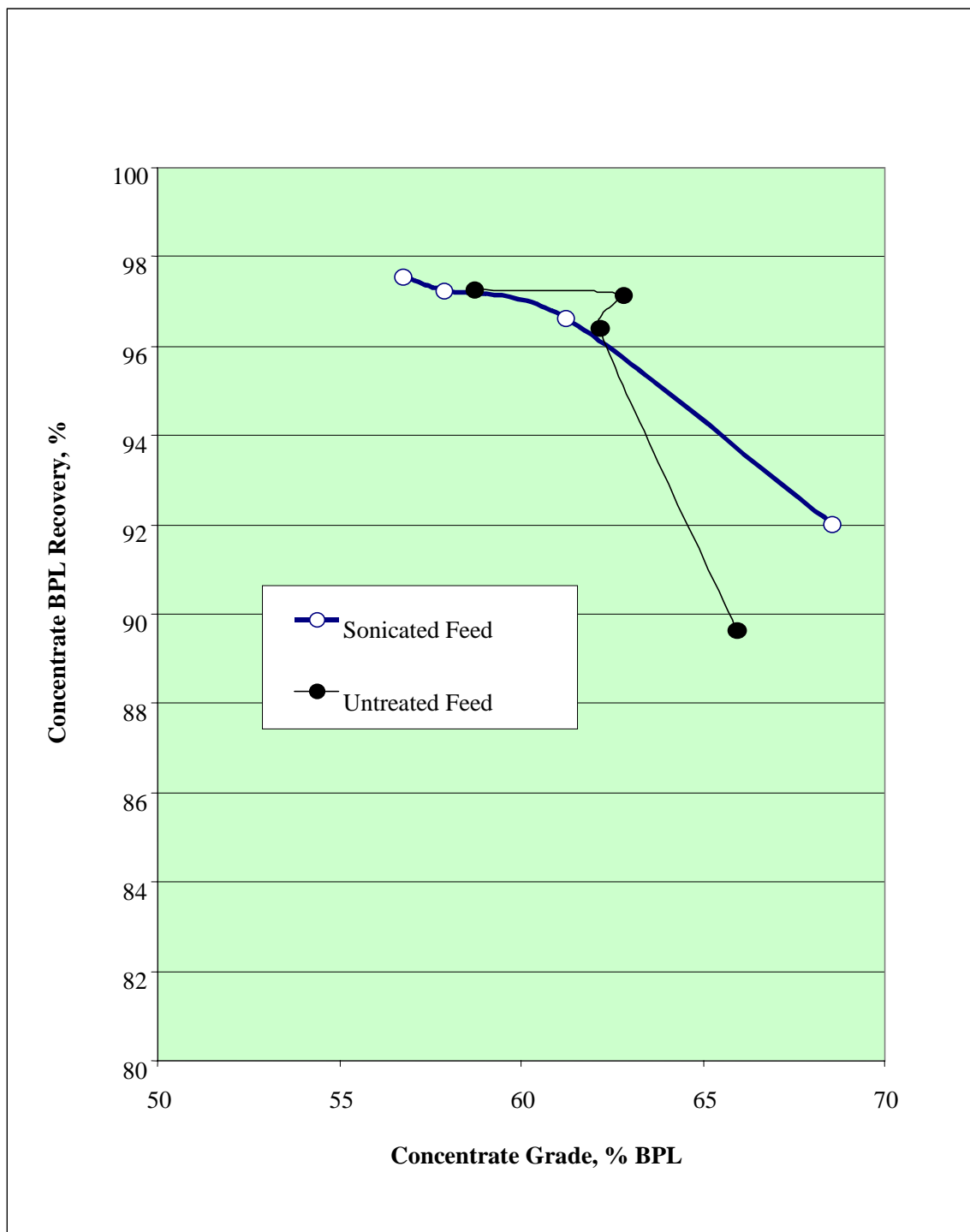


Figure 8. Lab Flotation of Nichols Feed, Recovery Vs. Grade.

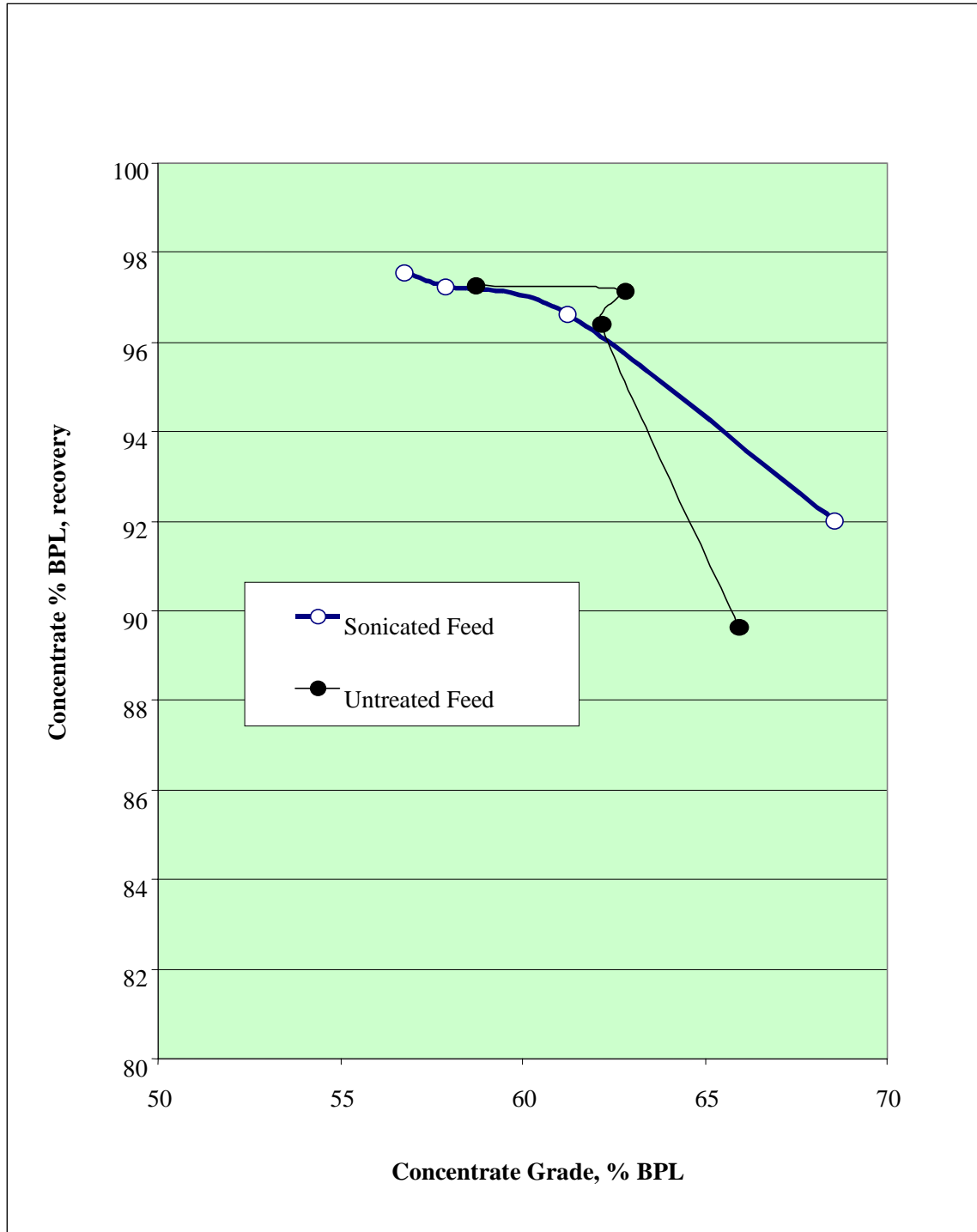


Figure 9. Lab Flotation of Refractory Feed, Recovery Vs. Grade.

Ultrasonic Treatment of Rougher Concentrate

Cleaner flotation results for three rougher concentrate samples with and without ultrasonic treatment are given in Tables 8 to 13 and Figures 10, 11, and 12. It can be seen from Figure 10 that one stage ultrasonic treatment improved flotation recovery and concentrate grade slightly at lower sulfuric acid dosage for HCII rougher concentrate. For the Nichols rougher concentrate, as shown by Figure 11, ultrasonic treatment made no improvement for de-oiling and cleaner flotation. When two stages of ultrasonic treatment were applied the recovery improvement was increased for all but the highest sulfuric acid dosage (5.2 lb./t), as shown on Figure 12.

It is worthwhile to point out that ultrasonic treatment of rougher concentrate without sulfuric acid did not result in satisfactory de-oiling. The results of the de-oiling and cleaner flotation tests confirmed that sulfuric acid is required for de-oiling. Ultrasonic treatment before acid scrubbing can improve the efficiency of de-oiling, but does not replace sulfuric acid.

Table 8. Amine Flotation of HCII Rougher Concentrate (Without Treatment).

3 minutes scrubbing @ ~65% solids; 2.5 ml 5% amine, 10 seconds conditioning; 2 minutes flotation

13.0 ml 10% sulfuric acid scrubbing**Starting pH: 2.10****End pH: 4.21**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	375.70	31.98	2.90	69.83	97.08	9.64	76.67
Tails	114.30	3.16	89.33	6.90	2.92	90.36	23.33
Feed	490.00	25.26	23.06	55.15	100.00	100.00	100.0

9.75 ml 10% sulfuric acid scrubbing**Starting pH: 2.10****End pH: 4.69**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	359.50	32.36	2.24	70.66	93.66	7.01	73.16
Tails	131.90	5.97	80.95	13.03	6.34	92.99	26.84
Feed	491.40	25.28	23.37	55.19	100.00	100.00	100.0

6.5 ml 10% sulfuric acid scrubbing**Starting pH: 2.21****End pH: 5.33**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	320.30	33.01	2.11	72.07	84.17	6.03	65.39
Tails	169.50	11.73	62.18	25.61	15.83	93.97	34.61
Feed	489.80	25.65	22.90	56.00	100.00	100.00	100.0

3.25 ml 10% sulfuric acid scrubbing**Starting pH: 2.50****End pH: 5.90**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	135.00	32.50	2.21	70.96	34.76	2.84	27.34
Tails	358.80	22.95	28.43	50.11	65.24	97.16	72.66
Feed	493.80	25.56	21.26	55.81	100.00	100.00	100.0

0 ml 10% sulfuric acid scrubbing**Starting pH: 7.59****End pH: 7.69**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	49.30	31.14	3.71	67.99	12.11	1.66	9.88
Tails	449.70	24.77	24.07	54.08	87.89	98.34	90.12
Feed	499.00	25.40	22.06	55.46	100.00	100.00	100.0

Table 9. Amine Flotation of HCII Rougher Concentrate (With Treatment).

3 minutes scrubbing @ ~65% solids; 2.5 ml 5% amine, 10 seconds conditioning; 2 minutes flotation

13.0 ml 10% sulfuric acid scrubbing**Starting pH: 2.05****End pH: 4.14**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	374.90	32.54	2.78	71.05	97.23	9.19	76.56
Tails	114.80	3.03	89.68	6.62	2.77	90.81	23.44
Feed	489.70	25.62	23.15	55.94	100.00	100.00	100.00

9.75 ml 10% sulfuric acid scrubbing**Starting pH: 1.99****End pH: 4.60**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	363.20	32.45	2.28	70.85	94.68	7.19	73.91
Tails	128.20	5.17	83.41	11.29	5.32	92.81	26.09
Feed	491.40	25.33	23.45	55.31	100.00	100.00	100.0

6.50 ml 10% sulfuric acid scrubbing**Starting pH: 2.05****End pH: 5.28**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	338.60	32.97	2.25	71.99	88.92	6.54	68.89
Tails	152.90	9.10	71.24	19.87	11.08	93.46	31.11
Feed	491.50	25.54	23.71	55.77	100.00	100.00	100.0

3.25 ml 10% sulfuric acid scrubbing**Starting pH: 2.12****End pH: 5.82**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	155.50	32.68	2.02	71.35	40.82	2.69	31.30
Tails	341.30	21.59	33.26	47.14	59.18	97.31	68.70
Feed	496.80	25.06	23.48	54.72	100.00	100.00	100.0

0 ml 10% sulfuric acid scrubbing**Starting pH: 7.48****End pH: 7.59**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	49.70	31.23	3.25	68.19	11.88	1.49	10.02
Tails	446.30	25.80	23.97	56.33	88.12	98.51	89.98
Feed	496.00	26.34	21.89	57.52	100.00	100.00	100.0

Table 10. Amine Flotation of Nichols Rougher Concentration (Without Treatment).

3 minutes scrubbing @ ~65% solids; 2.5 ml 5% amine, 10 seconds conditioning; 2 minutes flotation

13.0 ml 10% sulfuric acid scrubbing**End pH: 3.32**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	479.00	34.20	3.14	74.67	98.96	14.44	83.30
Tails	96.00	1.79	92.80	3.91	1.04	85.56	16.70
Feed	575.00	28.79	18.11	62.86	100.00	100.00	100.00

9.75 ml 10% sulfuric acid scrubbing**End pH: 3.77**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	484.50	33.64	3.12	73.45	98.60	14.34	83.06
Tails	98.80	2.34	91.43	5.11	1.40	85.66	16.94
Feed	583.30	28.34	18.08	61.87	100.00	100.00	100.0

6.5 ml 10% sulfuric acid scrubbing**End pH: 4.35**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	447.50	34.11	2.70	74.48	86.15	11.57	77.52
Tails	129.80	18.91	71.16	41.29	13.85	88.43	22.48
Feed	577.30	30.69	18.09	67.01	100.00	100.00	100.0

3.25 ml 10% sulfuric acid scrubbing**End pH: 5.32**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	156.80	33.44	2.88	73.01	31.78	4.32	26.79
Tails	428.50	26.27	23.34	57.36	68.22	95.68	73.21
Feed	585.30	28.19	17.86	61.55	100.00	100.00	100.0

0 ml 10% sulfuric acid scrubbing**End pH: 7.11**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	2.70	23.69	30.36	51.72	0.39	0.77	0.47
Tails	577.50	28.14	18.37	61.44	99.61	99.23	99.53
Feed	580.20	28.12	18.43	61.40	100.00	100.00	100.0

Table 11. Amine Flotation of Nichols Rougher Concentrate (With Treatment).

3 minutes scrubbing @ ~65% solids; 2.5 ml 5% amine, 10 seconds conditioning; 2 minutes flotation

13.0 ml 10% sulfuric acid scrubbing**End pH: 3.30**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	479.30	33.11	3.12	72.29	99.01	15.19	84.18
Tails	90.10	1.77	92.70	3.86	0.99	84.81	15.82
Feed	569.40	28.15	17.29	61.46	100.00	100.00	100.00

9.75 ml 10% sulfuric acid scrubbing**End pH: 3.75**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	473.20	33.88	2.82	73.97	97.82	13.18	82.53
Tails	100.20	3.57	87.73	7.79	2.18	86.82	17.47
Feed	573.40	28.58	17.66	62.41	100.00	100.00	100.0

6.50 ml 10% sulfuric acid scrubbing**End pH: 4.31**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	447.40	33.54	2.40	73.23	86.32	10.41	77.62
Tails	129.00	18.43	71.62	40.24	13.68	89.59	22.38
Feed	576.40	30.16	17.89	65.85	100.00	100.00	100.0

3.25 ml 10% sulfuric acid scrubbing**End pH: 5.28**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	132.90	33.40	2.40	72.93	28.07	2.80	23.19
Tails	440.10	25.84	25.20	56.42	71.93	97.20	76.81
Feed	573.00	27.59	19.91	60.25	100.00	100.00	100.0

0 ml 10% sulfuric acid scrubbing**End pH: 7.03**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL	%A.I.	% Weight
Concentrate	3.00	24.41	28.37	53.30	0.45	0.82	0.52
Tails	569.20	28.28	18.04	61.75	99.55	99.18	99.48
Feed	572.20	28.26	18.09	61.70	100.00	100.00	100.0

Table 12. Amine Flotation of HCII Rougher Concentrate (Without Treatment).

3 minutes scrubbing @ ~65% solids; 2.5 ml 5 % amine, 10 seconds conditioning; 2 minutes flotation

7.0 ml 10% sulfuric acid scrubbing**End pH: 5.20**

	Grams	%P ₂ O ₅	% A.I.	%BPL	%BPL Recovery	% A.I. Recovery	% Weight
Concentrate	423.70	31.40	3.84	68.56	97.05	23.32	86.45
Tails	66.40	6.10	80.58	13.32	2.95	76.68	13.55
Feed	490.10	27.97	14.24	61.07	100.00	100.00	100.00

5.25 ml 10% sulfuric acid scrubbing**End pH: 5.40**

	Grams	%P ₂ O ₅	% A.I.	%BPL	%BPL Recovery	% A.I. Recovery	% Weight
Concentrate	379.90	31.40	2.46	68.56	87.95	13.38	77.37
Tails	111.10	14.71	54.45	32.12	12.05	86.62	22.63
Feed	491.00	27.62	14.22	60.31	100.00	100.00	100.00

3.5 ml 10% sulfuric acid scrubbing**End pH: 5.90**

	Grams	%P ₂ O ₅	% A.I.	%BPL	%BPL Recovery	% A.I. Recovery	% Weight
Concentrate	299.30	31.44	2.58	68.65	68.68	11.55	60.81
Tails	192.90	22.25	30.65	48.58	31.32	88.45	39.19
Feed	492.20	27.84	13.58	60.78	100.00	100.00	100.00

1.75 ml 10% sulfuric acid scrubbing**End pH: 6.30**

	Grams	%P ₂ O ₅	% A.I.	%BPL	%BPL Recovery	% A.I. Recovery	% Weight
Concentrate	198.10	31.78	2.94	69.39	45.32	8.71	40.18
Tails	294.90	25.76	20.71	56.24	54.68	91.29	59.82
Feed	493.00	28.18	13.57	61.53	100.00	100.00	100.00

0 ml 10% sulfuric acid scrubbing**End pH: 7.50**

	Grams	%P ₂ O ₅	% A.I.	%BPL	%BPL Recovery	% A.I. Recovery	% Weight
Concentrate	189.40	31.14	3.59	67.99	43.53	8.96	38.17
Tails	306.80	24.94	22.53	54.45	56.47	91.04	61.83
Feed	496.20	27.31	15.30	59.62	100.00	100.00	100.00

Table 13. Amine Flotation of HCII Rougher Concentrate (With 2-Stage Treatment).

3 minutes scrubbing @ ~65% solids; 2.5 ml 5 % amine, 10 seconds conditioning; 2 minutes flotation

7.0 ml 10% sulfuric acid scrubbing**End pH: 5.10**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	416.70	31.63	2.69	69.06	96.64	16.04	84.99
Tails	73.60	6.22	79.73	13.58	3.36	83.96	15.01
Feed	490.30	27.82	14.25	60.73	100.00	100.00	100.00

5.25 ml 10% sulfuric acid scrubbing**End pH: 5.35**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	402.90	31.73	2.50	69.28	93.06	14.44	81.81
Tails	89.60	10.64	66.60	23.23	6.94	85.56	18.19
Feed	492.50	27.89	14.16	60.90	100.00	100.00	100.00

3.50 ml 10% sulfuric acid scrubbing**End pH: 5.85**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	355.10	31.88	2.51	69.61	82.17	13.12	72.13
Tails	137.20	17.90	43.00	39.08	17.83	86.88	27.87
Feed	492.30	27.98	13.79	61.10	100.00	100.00	100.00

1.75 ml 10% sulfuric acid scrubbing**End pH: 6.25**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	255.20	31.60	2.53	69.00	59.19	8.96	51.50
Tails	240.30	23.14	27.30	50.52	40.81	91.04	48.50
Feed	495.50	27.50	14.54	60.04	100.00	100.00	100.00

0 ml 10% sulfuric acid scrubbing**End pH: 7.50**

	Grams	%P ₂ O ₅	%A.I.	%BPL	%BPL Recovery	%A.I. Recovery	% Weight
Concentrate	181.10	31.10	3.09	67.90	41.52	7.78	36.70
Tails	312.30	25.40	21.25	55.46	58.48	92.22	63.30
Feed	493.40	27.49	14.58	60.03	100.00	100.00	100.00

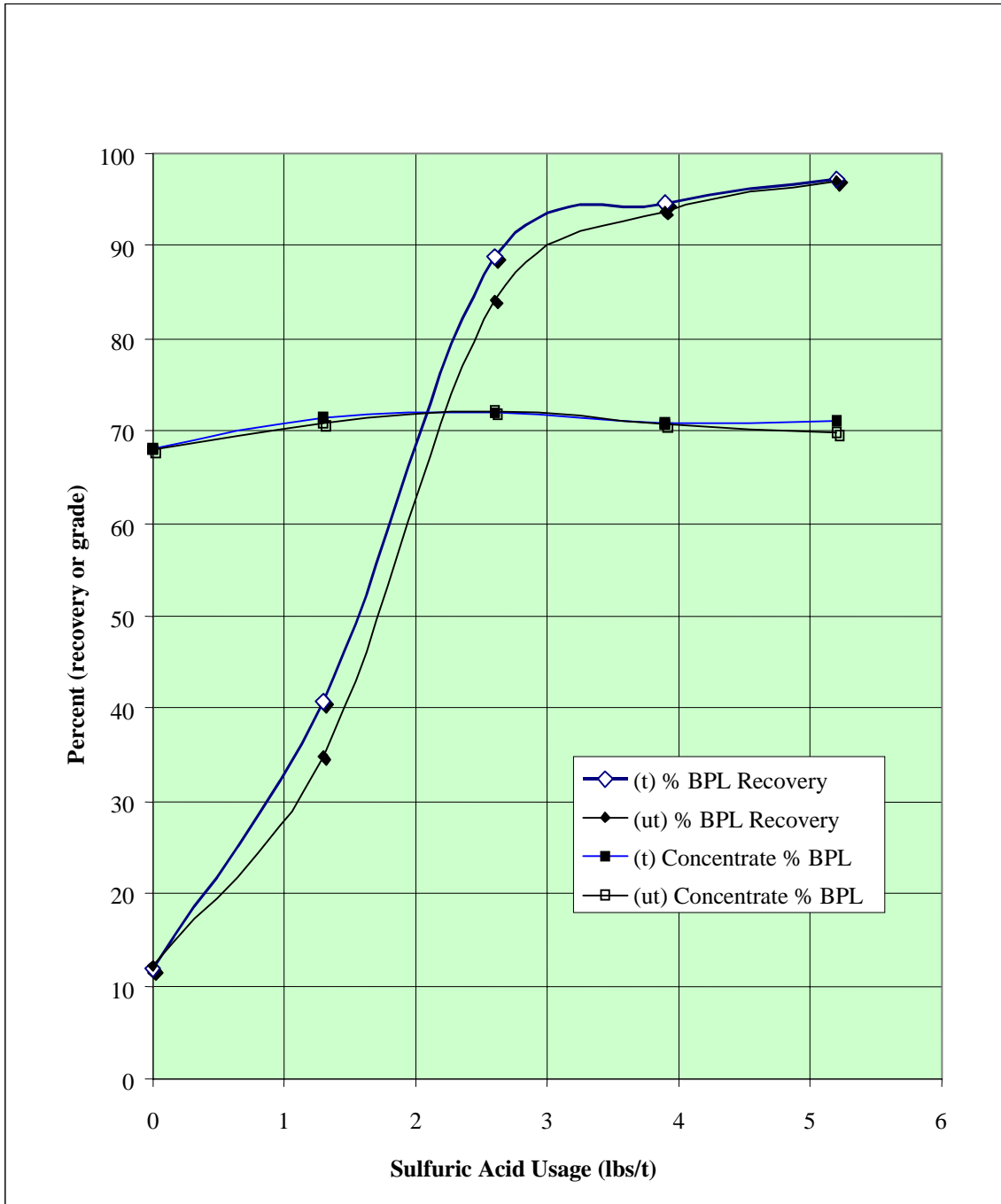


Figure 10. Cleaner Flotation Performance on HCII Rougher Concentrate (t) With Ultrasonic Treatment & (ut) Without Treatment.

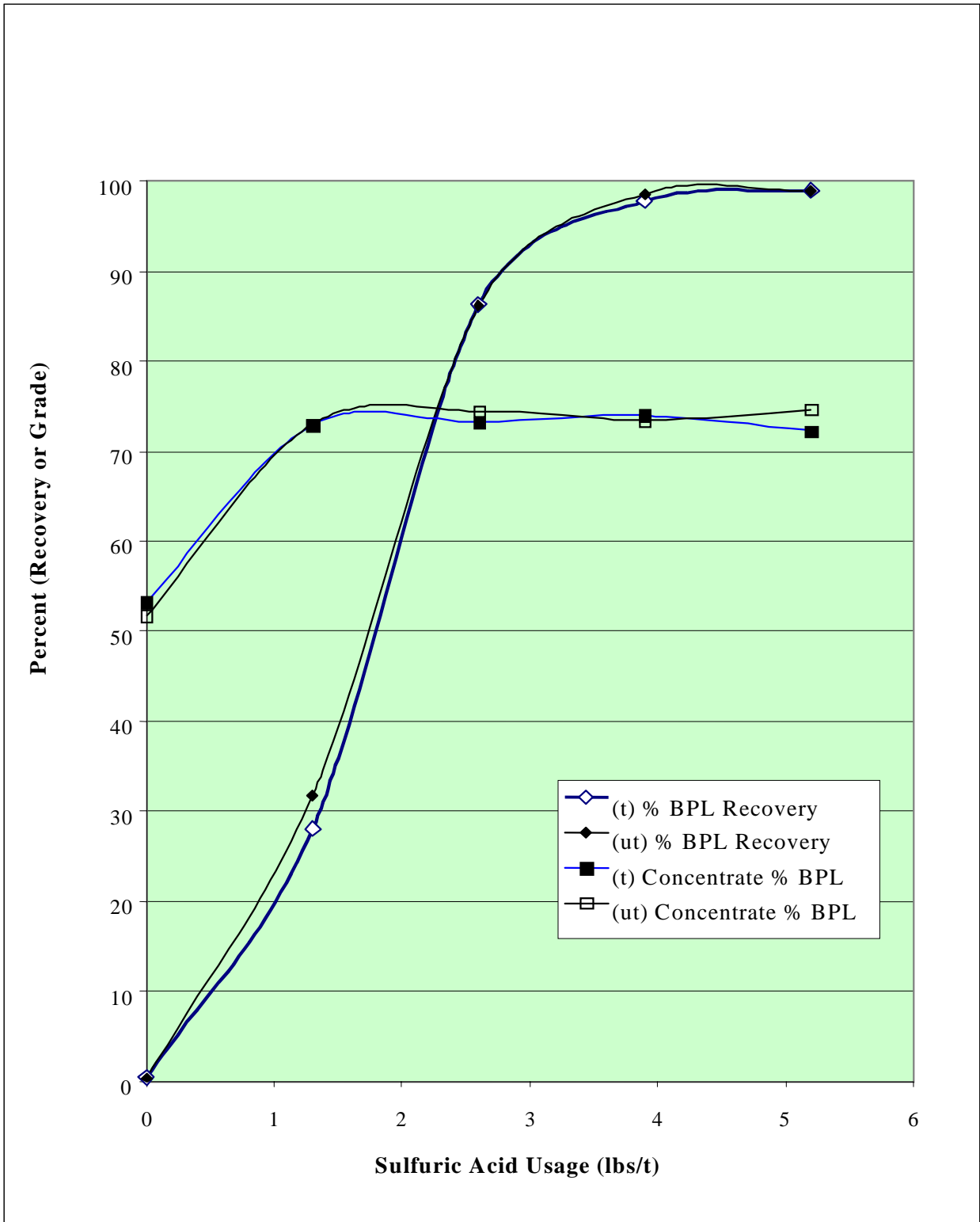


Figure 11. Cleaner Flotation Performance on Nichols Rougher Concentrate (t) With Ultrasonic Treatment & (ut) Without Treatment.

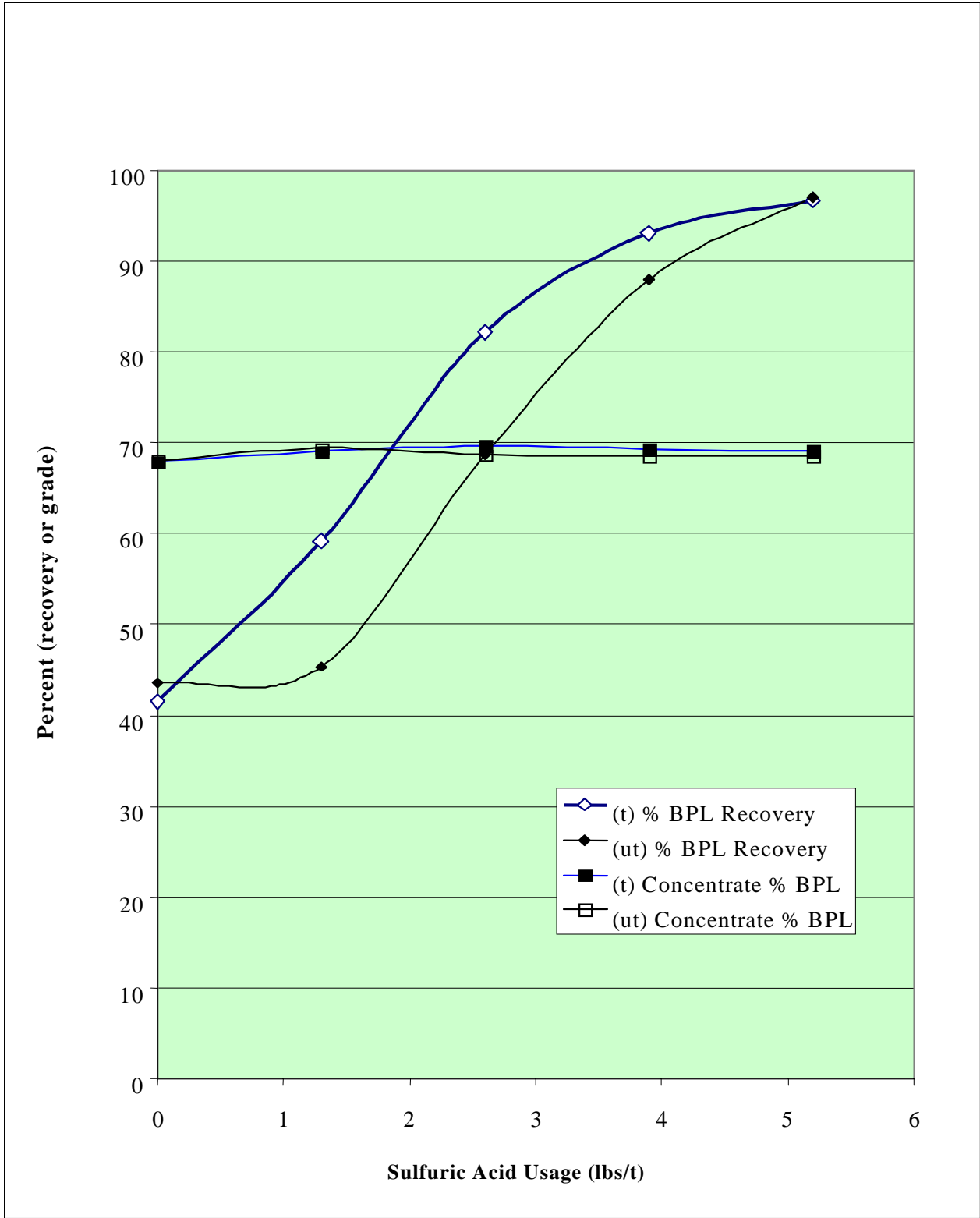


Figure 12. Cleaner Flotation Performance on 2-Stage Treated (t) & Untreated (ut) Rougher Concentrate.

Ultrasonic Treatment of Clay Slurry

The settling results of clay slurry with and without ultrasonic treatment are shown in Tables 14 and 15. It can be seen from these tables that there were slight differences between the treated and untreated slurries. In general, the settling rate was faster for the ultrasonic treated sample than for the untreated sample at the beginning of the test. However, the difference was very minimal and by the end of the test the untreated sample was slightly more consolidated.

Table 14. Settling Test Results for 12% Clay.

(with and without ultrasonic treatment)

Time Hour	Height, cm		%Solids of Settled Clay		
	Treated	Untreated	Treated	Untreated	Difference
0.00	39.30	40.40	12.00	12.00	0.00
24.50	38.10	38.50	12.38	12.59	-0.21
47.00	34.55	36.18	13.65	13.40	0.25
160.00	32.90	34.30	14.33	14.13	0.20
168.00	32.60	34.10	14.47	14.22	0.25
184.00	32.20	33.60	14.65	14.43	0.22
280.00	29.70	31.00	15.88	15.64	0.24
304.00	29.00	30.40	16.26	15.95	0.31
328.00	28.50	29.90	16.55	16.21	0.33
336.00	28.40	29.80	16.61	16.27	0.34
352.00	28.00	29.30	16.84	16.55	0.30
448.00	26.50	27.50	17.80	17.63	0.17
472.00	26.10	27.00	18.07	17.96	0.11
496.00	25.90	26.70	18.21	18.16	0.05
520.00	25.70	26.50	18.35	18.29	0.06
616.00	24.70	25.50	19.09	19.01	0.08
640.00	24.60	25.40	19.17	19.09	0.08
664.00	24.50	25.30	19.25	19.16	0.09
672.00	24.40	25.20	19.33	19.24	0.09
768.00	24.00	24.50	19.65	19.79	-0.14
792.00	23.90	24.40	19.73	19.87	-0.14
816.00	23.80	24.30	19.82	19.95	-0.14
840.00	23.80	24.20	19.82	20.03	-0.22

Table 15. Settling Test Results for 6% Clay.
(with and without ultrasonic treatment)

Time Hour	Height, cm		%Solids of Settled Clay		
	Treated	Untreated	Treated	Untreated	Difference
0.00	40.50	39.50	6.00	6.00	0.00
0.12	40.30	39.40	6.03	6.02	0.01
0.33	39.80	39.10	6.11	6.06	0.04
0.50	39.00	38.70	6.23	6.12	0.11
1.00	37.70	37.60	6.45	6.30	0.14
1.50	37.00	36.80	6.57	6.44	0.13
2.00	36.20	36.10	6.71	6.57	0.15
18.00	29.70	30.00	8.18	7.90	0.28
19.00	29.30	29.60	8.29	8.01	0.29
20.00	29.10	29.40	8.35	8.06	0.29
24.00	28.80	28.20	8.44	8.40	0.03
41.00	25.00	25.20	9.72	9.40	0.32
48.00	23.80	24.00	10.21	9.88	0.34
65.00	22.10	22.00	11.00	10.77	0.22
72.00	21.40	21.40	11.36	11.07	0.28
74.00	21.20	21.20	11.46	11.18	0.28
161.00	16.60	16.00	14.64	14.81	-0.17
168.00	16.50	15.90	14.73	14.91	-0.18
185.00	16.00	15.30	15.19	15.49	-0.30
209.00	15.50	14.80	15.68	16.01	-0.34
233.00	15.10	14.40	16.09	16.46	-0.37
329.00	14.20	13.40	17.11	17.69	-0.57
336.00	14.00	13.40	17.36	17.69	-0.33
352.00	13.90	13.30	17.48	17.82	-0.34
377.00	13.80	13.20	17.61	17.95	-0.35
401.00	13.50	13.10	18.00	18.09	-0.09
497.00	13.40	12.90	18.13	18.37	-0.24
521.00	13.40	12.80	18.13	18.52	-0.38
528.00	13.40	12.80	18.13	18.52	-0.38
545.00	13.40	12.80	18.13	18.52	-0.38
569.00	13.30	12.70	18.27	18.66	-0.39
665.00	13.20	12.50	18.41	18.96	-0.55
672.00	13.20	12.50	18.41	18.96	-0.55
689.00	13.20	12.50	18.41	18.96	-0.55
713.00	13.20	12.50	18.41	18.96	-0.55
737.00	13.20	12.50	18.41	18.96	-0.55

FIELD SAMPLING AND VISCOSITY TESTING RESULTS

Field Sampling

Primary cyclone overflow samples were collected from six phosphate beneficiation plants in Florida. Table 16 shows the quantity, percent solids, and size distribution for each sample. It can be seen from Table 16 that the solids content of the primary cyclone overflow samples ranged from 3 to 5 percent, and the contained solids were 92.7% to 99.9% passing 150 mesh (105 microns).

Table 16. Primary Cyclone Overflow Sample Data.

Sample No	1	2	3	4	5	6
Quantity, gal.	~70	~70	~70	~70	~70	~70
Clay % Solids	3.54	4.40	3.86	3.31	5.03	3.79
Mesh	Cumulative % Passing					
-20	100.00	100.00	100.00	100.00	100.00	100.00
-35	100.00	99.90	100.00	100.00	100.00	98.29
-48	99.98	99.78	100.00	100.00	99.96	96.67
-65	99.93	99.54	100.00	100.00	99.85	95.08
-100	99.50	99.99	100.00	100.00	99.39	93.18
-150	98.12	97.69	99.89	99.94	98.07	92.72
-200	95.49	95.59	99.56	99.60	94.21	92.23
-270	92.39	92.80	99.23	99.12	88.96	91.06
-325	90.54	91.81	98.85	98.78	87.41	90.45

Clay rejection and feed losses were not measured directly; however, feed losses are indicated by the size distributions of the primary cyclone overflows. The higher losses indicated for some samples may have resulted from mechanical problems or overloaded conditions.

Viscosity Testing Results

The viscosity measurements were carried out in the lab using two viscometers. A Brookfield viscometer was used to measure viscosities at shear rates from 0.066 to 13.2 Sec⁻¹. The Nametre viscometer measured clay slurry viscosities at a single shear rate of 4,000 Sec⁻¹. The viscosities of the primary cyclone overflow samples, measured at adjusted percent solids and controlled shear rates, are given in Table 17. Viscosity versus shear rate at different percent solids for different samples were plotted and the results are shown in Figures 13 to 18.

Table 17. Measured Viscosities of Primary Cyclone Overflow Samples.

Name	Solids	Shear Rate, S ⁻¹								
		0.066	0.132	0.33	0.66	1.32	2.64	6.6	13.2	4000
Viscosity, reported as centipoise, cP										
Sample 1	3.35%							30	21	9
	6.09%						205	161	104	58
	9.20%						1,550	730	426	283
Sample 2	1.69%								9	1
	3.10%								12	2
	6.09%								15	2
	9.00%				250	130	75	33	22	4
Sample 3	3.00%						51	25	15	2
	6.00%				225	110	73	34	22	5
	9.00%					115	88	62	47	22
	12.45%		1,600			850	550	270	158	39
Sample 4	2.96%								9	3
	6.48%							29	21	9
	9.50%	12,000	6,375	2,850	1,625	1,013	713	470	348	28
Sample 5	3.16%							23	15	9
	6.29%				210	175	150	110	80	55
	8.76%	21,250	14,875	7,700	4,250	2,363	1,313	595	330	261
Sample 6	3.46%							24	16	3
	6.48%	2,250	1,325	540	290	163	94	55	37	14
	9.38%				350	335	301	220	135	62
	12.96%				44,00	3,000	1,675	750	405	223

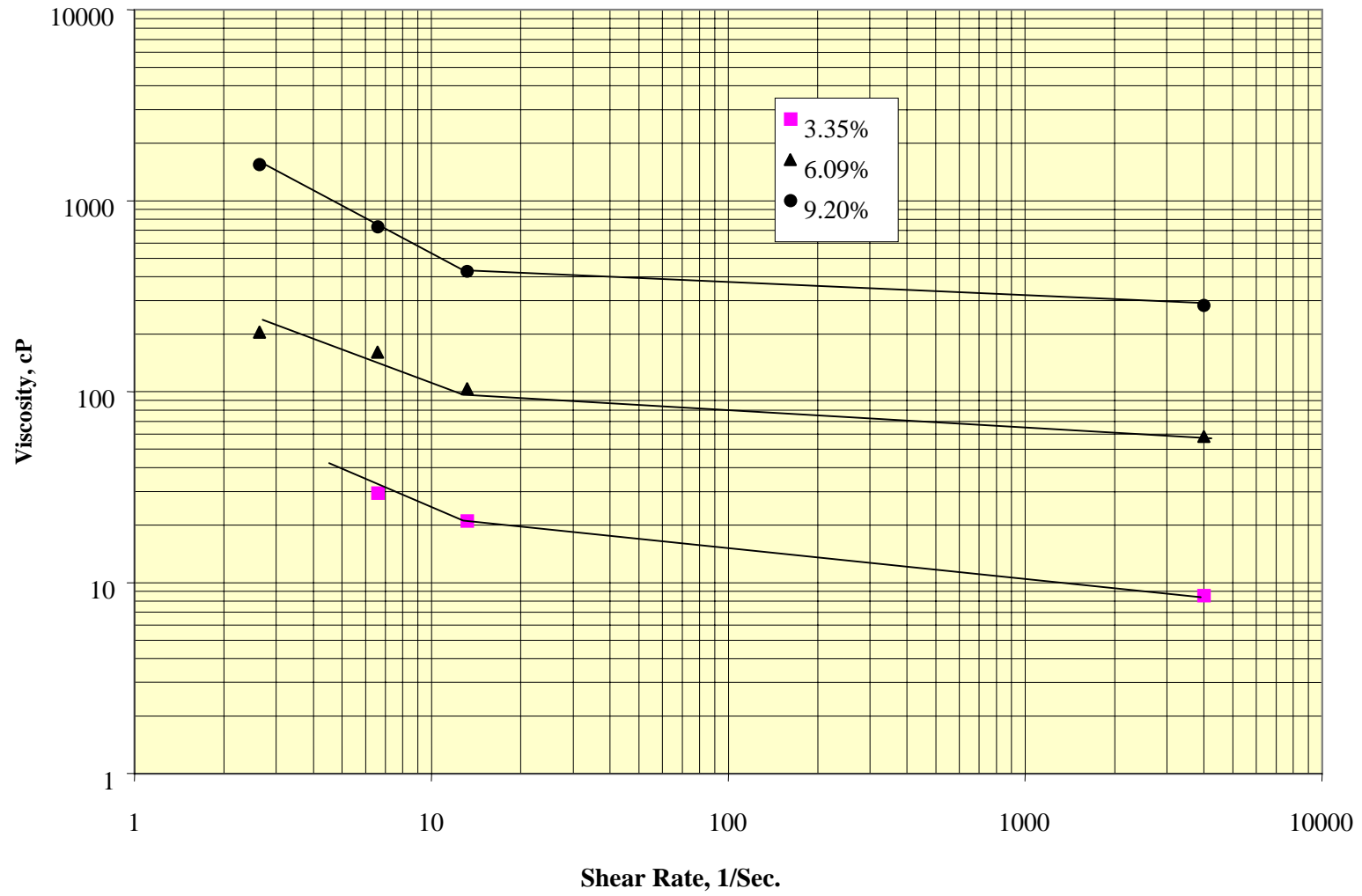


Figure 13. Viscosity Measurements of Cyclone Overflow Sample 1.

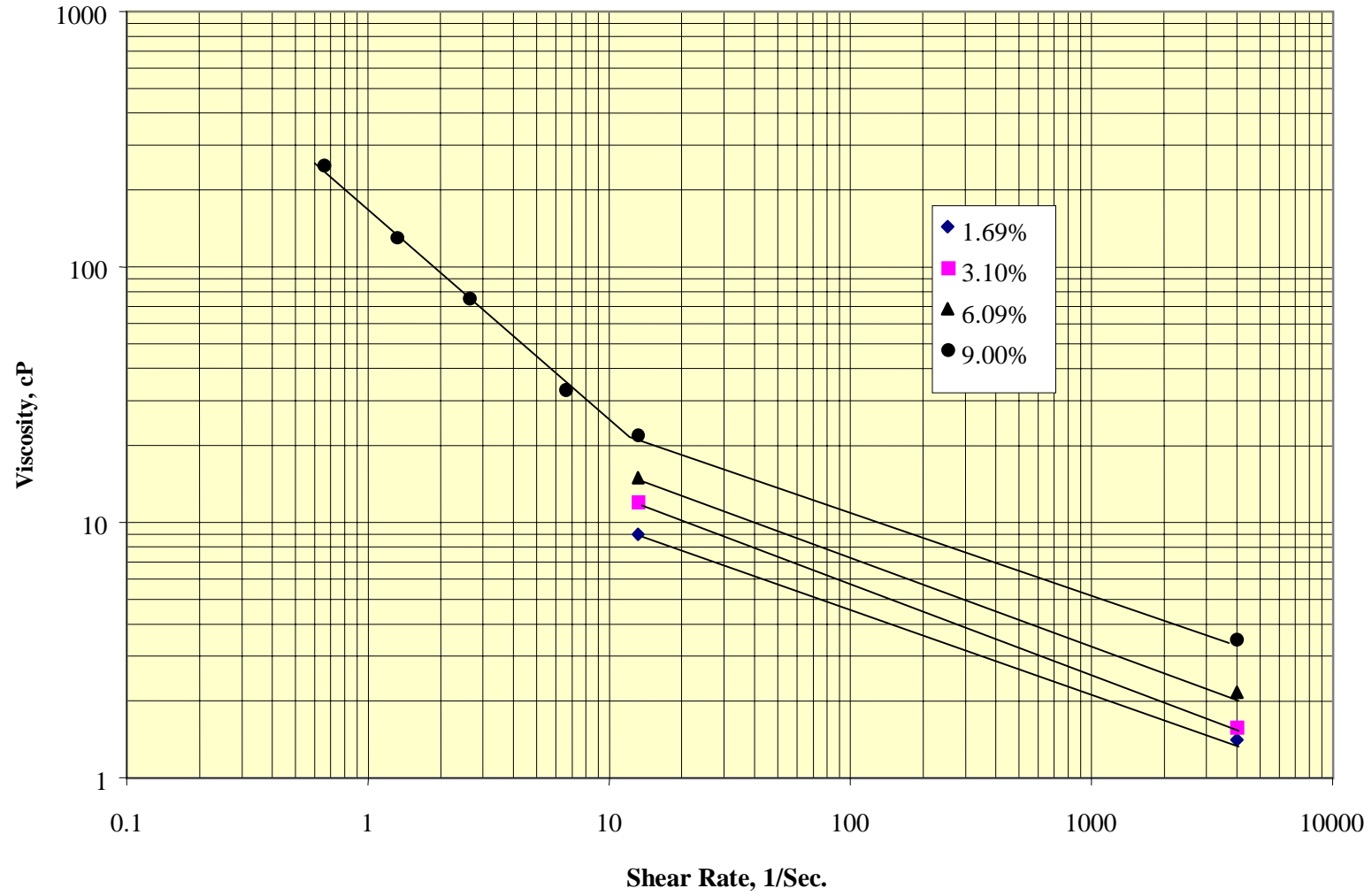


Figure 14. Viscosity Measurements of Cyclone Overflow Sample 2.

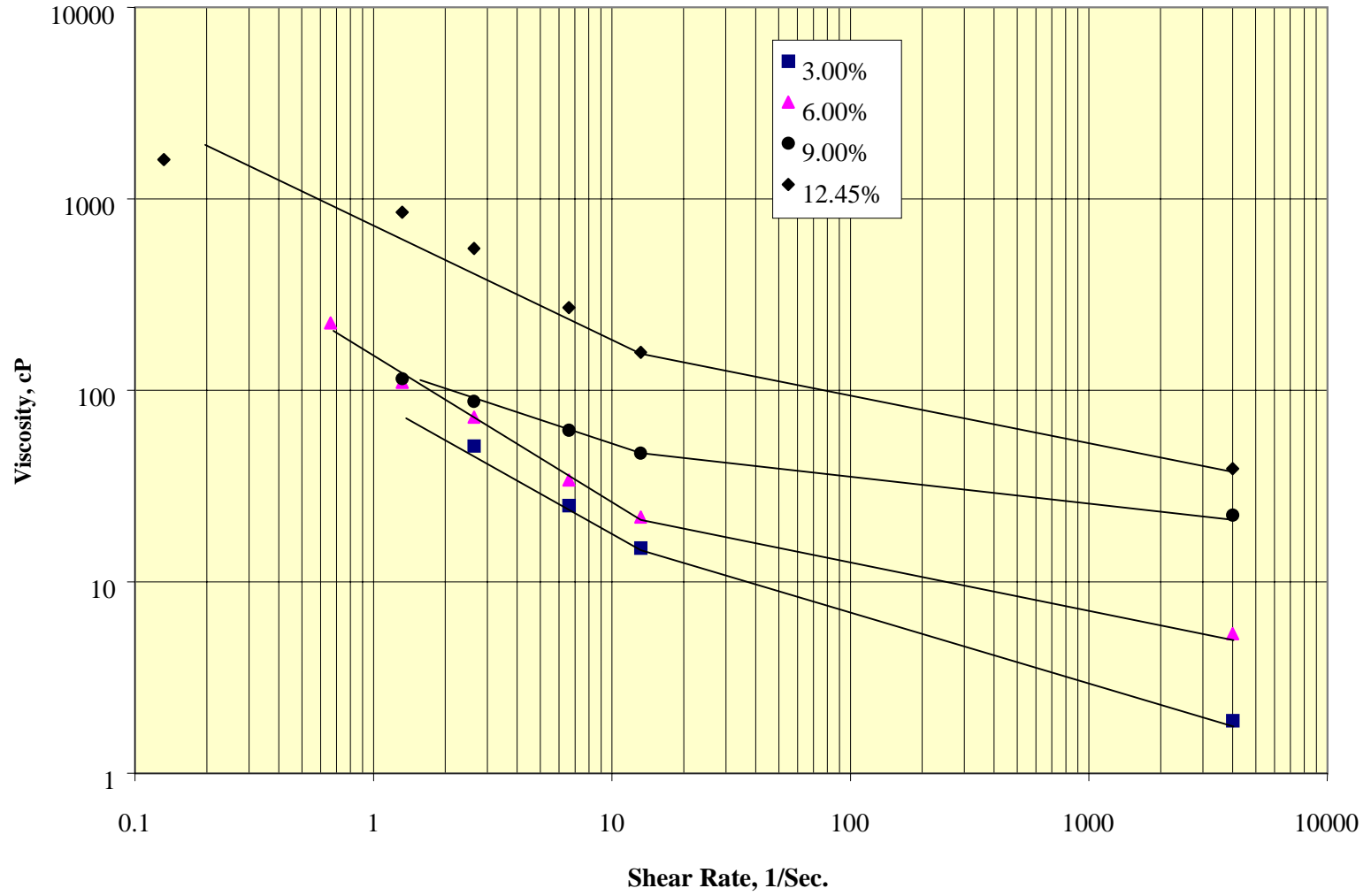


Figure 15. Viscosity Measurements of Cyclone Overflow Sample 3.

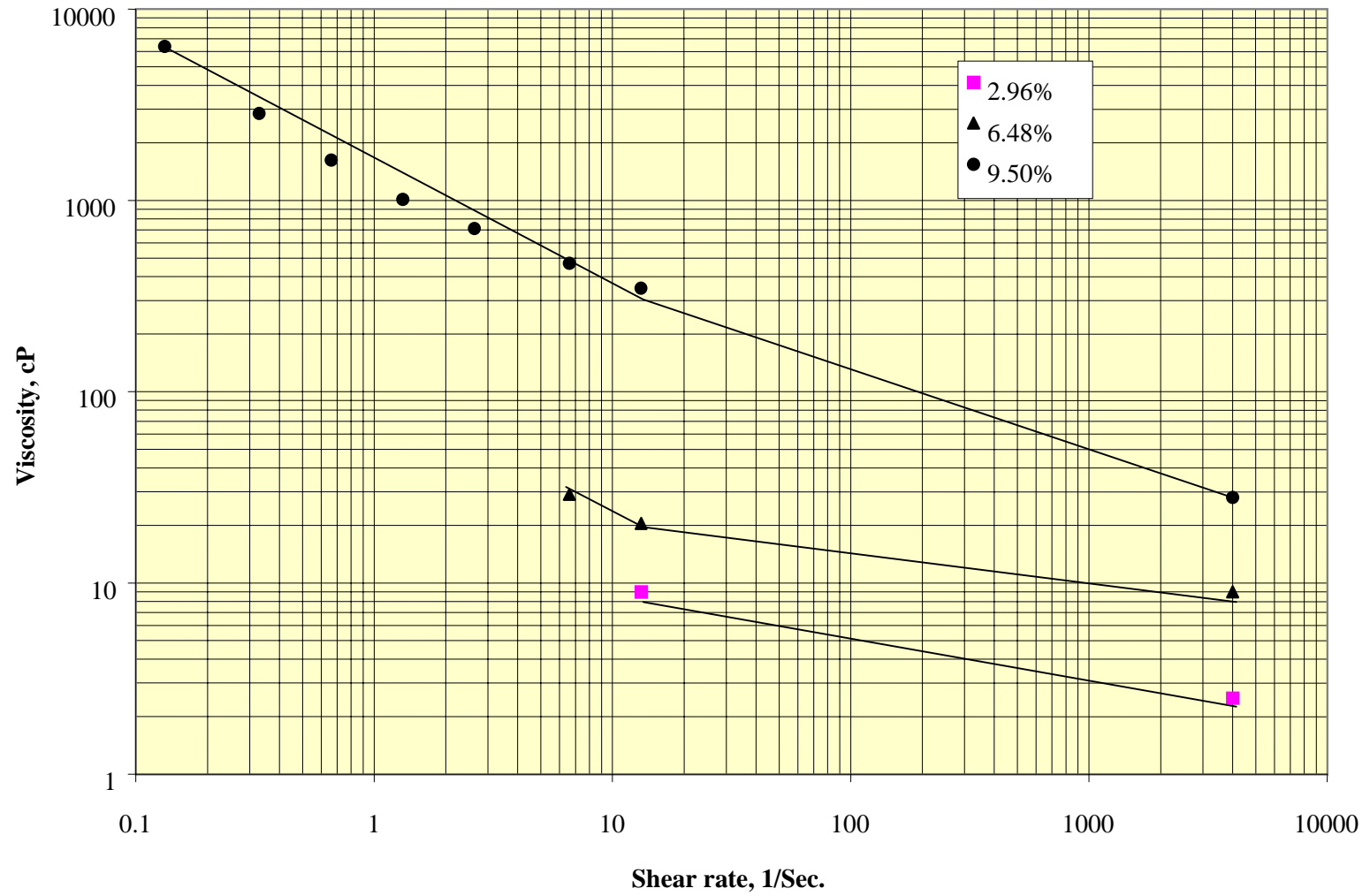


Figure 16. Viscosity Measurements of Cyclone Overflow Sample 4.

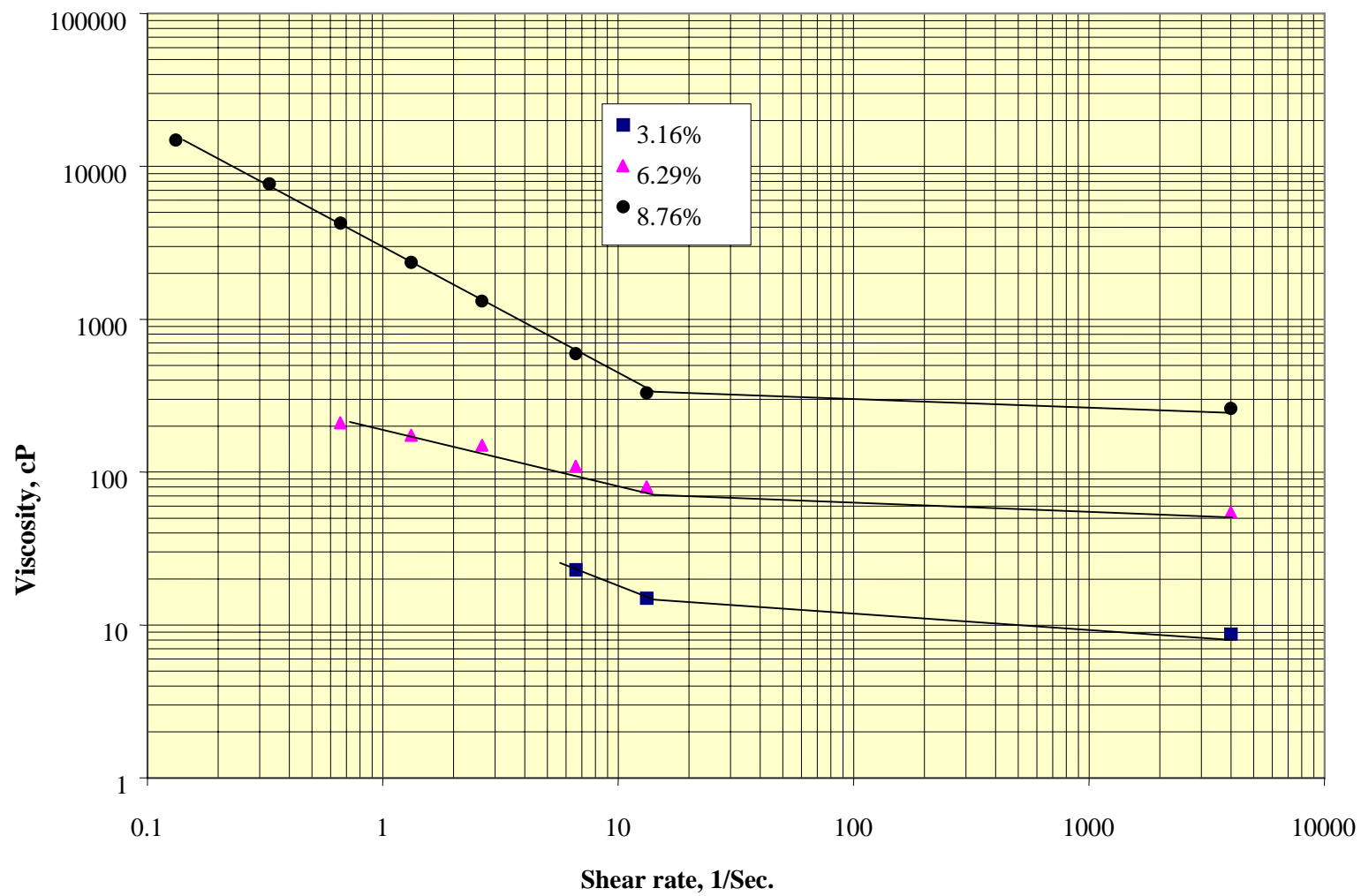


Figure 17. Viscosity Measurements of Cyclone Overflow Sample 5.

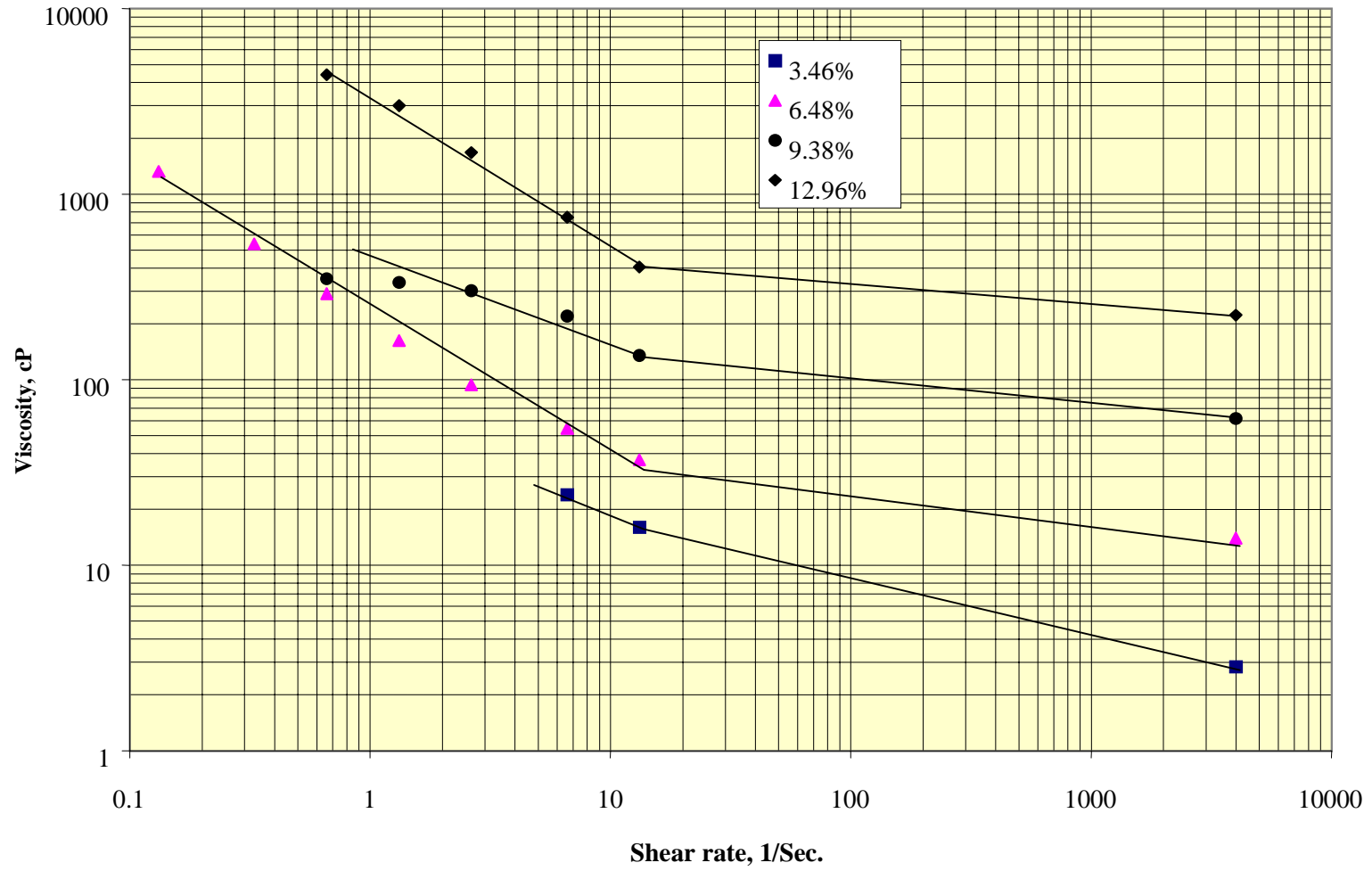


Figure 18. Viscosity Measurements of Cyclone Overflow Sample 6.

Figures 13 through 18 clearly show non-Newtonian characteristics of the slurries. Since the viscosity of all samples decreased with increased shear rate, the tested slurries may be considered pseudoplastic fluids. Size analysis data showed that the clays were about 90% less than 44 microns. Specific surface measurements (surface area per unit weight of solids) of the cyclone overflow solids and pure clay samples are tabulated below, along with viscosity data for slurries with nominally 6% solids. The measured viscosities were obtained with the Nametre viscometer. Calculated viscosity data derived from particle settling rates as described in the following section are also shown. The prepared slurries of kaolinite and palygorskite settled much more rapidly than all but one primary cyclone overflow sample. The montmorillonite clay was not amenable to testing because it was a balling clay that would not dispense.

Table 18. Measurements of Clay Sample Specific Surface and Viscosity.

Sample Identity	Specific Surface m²/gram	Viscometer cP @ 4000 /Sec.	Settling Rate cP (calculated)
Sample 1	34.03	58	229
Sample 2	16.99	2	2
Sample 3	36.87	5	10
Sample 4	25.08	9	3
Sample 5	55.85	55	1171
Sample 6	43.23	14	15
Kaolinite	16.67	2	na
Palygorskite	108.50	4	na

The pseudoplastic behavior may be attributed, at least in part, to the high specific surface area and corresponding exaggerated influence of the double layer surrounding the clay particles. If the volume fraction of solids includes both the volume of solid particles and the volume of their double layer, it is obvious that the volume contributed by the double layer increases with an increase in particle specific surface. Stern (1924) suggested that the double layer could be divided into a compact inner layer and a diffuse outer layer. It is suggested that viscosity increases as the volume fraction occupied by solids and their double layer increases. When slurry is subjected to shear, the thickness of the diffuse component of the double layer may decrease with an increase in shear rate. Reducing the double layer thickness also reduces the volume fraction, which may explain why clay slurry viscosity decreases with increased shear rate.

The above speculation on pseudoplastic behavior is neither refuted nor strongly supported by the data presented in Table 18. It should be noted that the viscosities calculated from settling rates show more variability because the shear rates vary between the samples. Additional discussion of the settling tests is presented in the next section. The above viscosity data were obtained from primary cyclone overflow samples adjusted to about 6% solids by weight. The data show that weight % solids alone is not a reliable predictor of slurry viscosity. Cyclone vendors, in the absence of reliable slurry viscosity

data, rely on volume % solids and mineral characteristics to specify cyclone dimensions and operating parameters for a given application.

Figure 19 shows the effect of solids content on viscosity at high shear rate (4,000 Sec-1) for different samples. As shown, viscosity increases exponentially with solids content. The relationship between viscosity and solid content, for the six samples tested, may be expressed by the following general equation:

$$\mu_s = A \exp(BC),$$

where μ_s is the slurry viscosity in centipoise, A and B are constants for a specific slurry, and C is the solids weight fraction in the slurry. Regression of the experimental data gives A and B the following values:

	A	B
Sample 1	1.280	59.628
Sample 2	1.093	12.353
Sample 3	0.765	33.343
Sample 4	0.835	36.886
Sample 5	1.265	60.560
Sample 6	0.653	46.210

As shown on Figure 19, the correlation between slurry viscosity and slurry weight % solids is very good for the individual samples; however, between samples there are significant variations in viscosity at a given weight % solids. To use the above general equation, it appears necessary to have A and B constants for the specific slurry.

Results of Settling Tests

The settling rates of phosphate particles in the samples of primary cyclone overflows at different clay contents are given in Table 18. The settling velocity was calculated by dividing the distance settled by the measured settling time. Slurry viscosity was calculated from the measured settling velocity according to Stokes' Equation:

$$\mu_f = \frac{d^2(\rho_p - \rho_f)g}{18v_s},$$

where μ_f is the slurry viscosity, d is the particle diameter, ρ_p is the density of the particle, ρ_f is the density of the slurry, g is the gravity acceleration, v_s is the settling velocity. Reynolds number (Re) was derived from the calculated viscosity according to:

$$\text{Re} = \frac{dv_s \rho_f}{\mu_f}$$

The derived Reynolds numbers are all less than 10, indicating settling at laminar or near laminar conditions. Stokes' equation is therefore considered valid for the phosphate particles settling in the clay slurries under the test conditions.

The viscosities of clay slurries derived from the settling tests were in reasonable agreement with those measured by viscometers when shear rate was taken into account. The maximum shear rate existing in a Newtonian fluid flowing about a sphere at Reynolds numbers of no more than 40 can be expressed by the equation below (Dallon and Christiansen 1968):

$$\gamma_{Max} = \chi \left(3 \frac{v_s}{d} \right),$$

where

$$\chi = 1, \quad \text{Re} \leq 1$$

$$\chi = \text{Re}^{0.36}, \quad 1 < \text{Re} \leq 40$$

The calculated maximum shear rates are recorded in Table 19 for the conditions of the settling tests.

Solid particles in a fluid in a centrifugal force field, such as might exist inside a cyclone, also have a settling velocity. However, in this case the acceleration due to gravity (g) is replaced by a centrifugal acceleration (g_c),

$$g_c = \frac{v_t^2}{r},$$

where v_t is the tangential velocity at radius r .

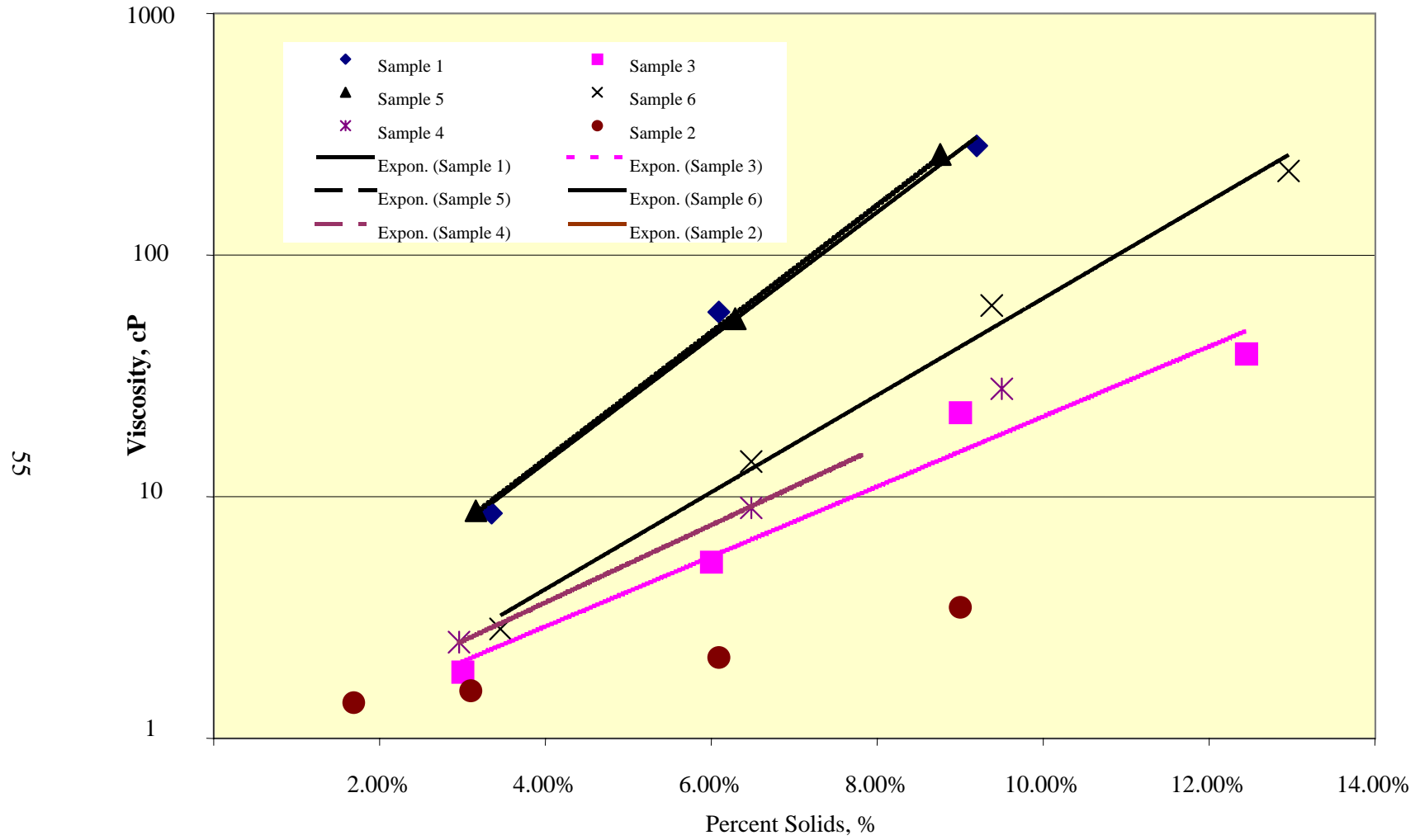


Figure 19. Measured & Predicted Clay Slurry Viscosities (vs. %Solids @ 4,000/Sec. Shear Rate).

The diameter ratio of particles settling under gravitational and centrifugal forces with the same settling velocity can be derived from the following equation.

$$\frac{d_g}{d_c} = \sqrt{\frac{g_c \mu_{fg}}{g \mu_{fc}}},$$

where d_g is the diameter of a particle settling by gravity, d_c is the diameter of particle settling in a centrifugal field, μ_{fg} is the slurry viscosity reacting on the particle settling by gravity, and μ_{fc} is slurry viscosity reacting on the particle settling in the centrifugal field.

The separation size for Florida phosphate desliming cyclones is nominally 105 microns (150 mesh). The particles used in the gravity settling tests ranged from 105 to 4,760 microns in diameter, with mostly a size of 297 microns for ~3% clay solids and 595 microns for ~6% clay solids. These particle diameters are 3 to 6 times larger than the nominal desliming size., which implies that the test results can be used to demonstrate settling conditions of 150 mesh particles in a centrifugal field of 10 to 40 times gravity.

Table 19. Settling Rates of Phosphate Particles in Clay Slurries.

Clay Slurry		Particle Size		Settling			Calculated Values		
Cw	SpG	Mesh	Micron	Distance	Time	Velocity	Centipoise		Max. Shear
				cm	sec	cm/sec	cP	Re	Sec ⁻¹
HCI									
3.00%	1.019	48X65	297 X 210	94.21	21.5	4.38	1.5	7.3	907.43
6.00%	1.040	28X35	595 X 420	74.51	28.5	2.61	10.2	1.3	146.71
9.00%	1.061	14X16	1190 X 1000	72.91	28.0	2.60	47.4	0.6	55.86
12.45%	1.086	14X16	1190 X 1000	21.21	96.0	0.22	550.7	0.0	0.81
Kingsford									
3.16%	1.021	48X65	297 X 210	77.51	48.0	1.61	4.2	1.0	163.07
6.29%	1.042	14X16	1190 X 1000	36.16	340.0	0.11	1171.3	0.0	0.23
8.76%	1.059	14X16	1190 X 1000	14.21	653.0	0.02	5672.6	0.0	0.01
Nichols									
3.35%	1.022	28X35	595 X 420	94.21	22.5	4.19	6.5	3.4	326.67
6.09%	1.040	14X16	1190 X 1000	50.61	93.0	0.54	229.1	0.0	3.74
9.20%	1.062	4X5	4760 X 4000	22.21	311.0	0.07	27610.4	0.0	0.02
Swift Creek									
3.46%	1.023	48X65	297 X 210	85.11	25.7	3.31	2.0	4.2	561.51
6.48%	1.043	28X35	595 X 420	61.81	35.0	1.77	15.1	0.6	74.85
9.38%	1.063	14X16	1190 X 1000	66.01	52.0	1.27	97.0	0.2	16.26
12.96%	1.090	4X5	4760 X 4000	40.96	307.5	0.13	14585.7	0.0	0.05
Ft. Green									
2.96%	1.019	48X65	297 X 210	93.91	21.0	4.47	1.5	7.6	939.60
6.48%	1.043	48X65	297X210	76.81	28.3	2.71	2.5	2.9	403.18
9.50%	1.064	14X16	1190 X 1000	69.41	36.7	1.89	65.1	0.3	32.29
Hookers Prairie									
1.69%	1.011	100X150	149 X 105	86.91	24.3	3.58	0.5	9.6	1628.29
3.10%	1.020	100X150	149 X 105	84.31	25.3	3.33	0.5	8.5	1449.08
6.09%	1.040	48X65	297 X 210	84.51	24.0	3.52	1.9	4.9	630.00
9.00%	1.061	48X65	297 X 210	69.51	30.7	2.26	2.9	2.1	297.98

Density of Settling Particles = 2.95

Cw = clay % solids, SpG = slurry specific gravity

Re = Reynolds Number

Viscosity Control of Cyclone Separation

Many theoretical and empirical correlations used to predict the 50% passing size (d_{50}) for cyclone separations have the following common form (Kelly and Spottiswood 1982):

$$d_{50} \propto \left(\frac{\mu_f}{\rho_s - \rho_f} \right)^{\frac{1}{2}}$$

It has been shown that the above equation can be used for slurries up to about 35% solids by weight.

Feed dilution is often used to control cyclone performance in operating plants. The addition of dilution water, other things being equal, always results in a smaller d_{50} and sharper classification. A control loop incorporating a nuclear density gauge and a controller to modulate a water valve is often used to control the slurry density to cyclones. However, the feed percent solids is an indirect measure of fluid viscosity (μ_f) and density (ρ_f). The cyclone overflow stream, particularly for desliming applications, is the closest representation of the carrier fluid.

The desliming cyclone overflow density increases from 1.00 to 1.08 (8 % increase) when the weight percent solids increases from 0 to 12% (+1200 % increase). The density variation corresponding to a change of one-percent solids is only 0.0067 (g/cm^3), indicating that sensitivity would be very low if density measurements were used to control the cyclone operation.

The viscosity measurement results presented in this report show that the viscosity increases exponentially with cyclone overflow percent solids. In other words, the viscosity is very sensitive to changes in percent solids. Similarly, carrier fluid viscosity is a variable that influences cyclone performance because of its influence on settling velocity. The cyclone overflow viscosity is therefore an ideal parameter for control of dilution water addition to optimize cyclone performance.

With constant speed slurry pumps, the modulation of dilution water will cause corresponding variation of slurry level in the cyclone feed tank. When the slurry level increases, the pump's total dynamic head is reduced and pumping capacity and pressure drop across the cyclones are increased. The opposite occurs when slurry level decreases. For desliming cyclones, the cut point is changed only about one Tyler mesh size for a three-fold change in cyclone pressure drop (Tarr 1985).

An automatic control system proposed for desliming cyclones is shown in Figure 20. The system consists of two loops, one for viscosity control and a second for level control. Only two cyclones are shown in Figure 20; however, in commercial plants 12, 18, or 24 cyclones may be operated in parallel.

For viscosity control, a Nametre Viscoliner monitors the cyclone overflow viscosity. Deviations from the target viscosity cause the controller to adjust the process water valves. Lower viscosities would cause the dilution water to be reduced, while higher viscosities would cause the dilution water to be increased. Changes in dilution water addition rate cause changes in the cyclone feed tank slurry level.

An automatic level control maintains the slurry level in the pump feed tank between high (HL) and low (LL) levels. Opening or closing cyclone inlet valves controls the slurry level. An example pump curve is combined with system curves for HL, mid level (ML), and LL tank conditions in Figure 21. The slurry is distributed to a relatively large number of cyclones to minimize cyclone pressure drop changes as inlet valves are opened and closed. The example system curves assume that LL, ML, and HL are 4 ft., 10 ft., and 16 ft. respectively above the pump suction.

Starting with system curve 2 on Figure 21, the system is stable at about 31,500 gpm and 89 feet of head, with 20 cyclones operating. If the viscosity control system calls for more dilution water, the slurry level in the tank will reach ML+3 ft., and the level sensor will open the inlet valve of the 21st cyclone. If input still exceeds output, the tank will fill to HL and the level sensor will open the inlet valve of the 22nd cyclone. At this point the system has reached its maximum flow capacity of about 35,500 gpm.

Now, if the cyclone overflow viscosity is reduced because of a reduction in solids or a change in clay mineralogy, the viscosity control system will reduce dilution water until either the target viscosity or minimum system capacity is reached. Again, starting with system curve 2 on Figure 21, the system is stable at about 31,500 gpm and 89 feet of head, with 20 cyclones operating. As output exceeds input, the level drops below ML, and the 20th cyclone inlet valve is closed. If the level continues to fall, the inlet valve to the 19th cyclone will also close. With 18 cyclones operating, the system reaches its maximum head capacity of 94 ft. when the tank level falls to LL.

The system described above, operates with 18 to 22 cyclones, ranging in capacity from 28,000 gpm at 94 ft. of head to 35,500 gpm at 84 ft. of head. If the latter flow represents normal design conditions, then the former flow represents potential energy savings.

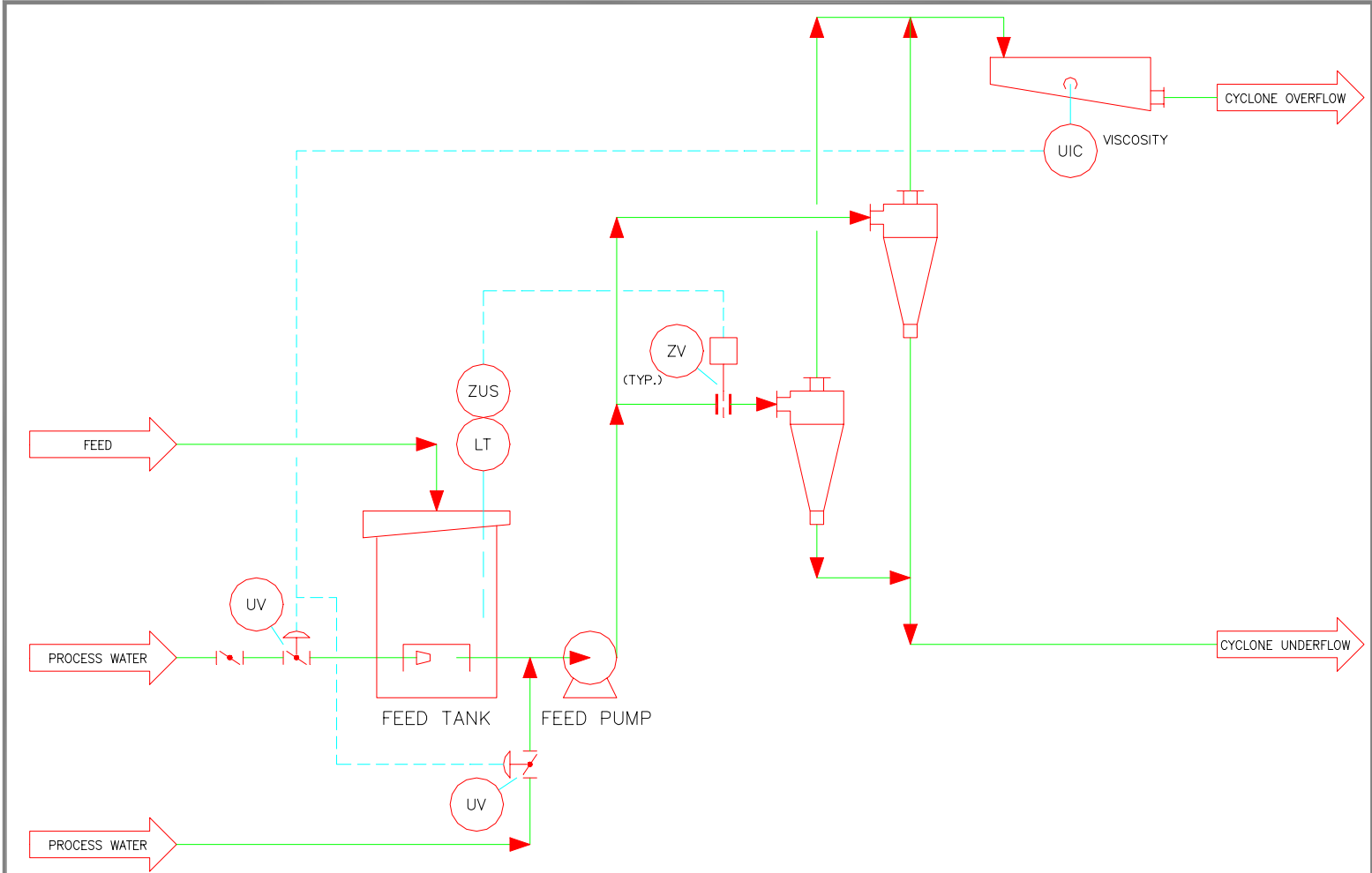
$$\text{Annual pump energy cost} = \$0.04/\text{kW.h} \times \text{hr/yr} \times 0.746 \times Q \times H \times G / (3960 \times E)$$

$$\text{Design condition} = 0.04 \times 6,000 \times 0.746 \times 35,500 \times 84 \times 1.129 / (3960 \times .6) = \$253,691$$

$$\text{Optimized condition} = 0.04 \times 6,000 \times 0.746 \times 28,000 \times 94 \times 1.164 / (3960 \times .6) = \\ \$230,856$$

The maximum annual energy savings for the example case is about \$22,000. Because the energy savings are accomplished by reducing the number of operating cyclones, the volumetric split between cyclone overflow and underflow should maintain good clay rejection to the overflow. Similarly, because viscosity is maintained at acceptable levels,

the losses of feed to the cyclone overflow should be minimized. The valuation of feed losses is influenced by many factors; however, for this exercise it is estimated that each ton of feed recovered from the cyclone overflow earns \$2.00 profit. For a plant producing 2 million tons concentrate per year, a 2% feed loss amounts to about 200,000 tons per year. Recovering a quarter of the loss, or 50,000 tons, could improve profits by \$100,000 annually.



PN:	28-V938-00
Prepared By:	FSH/CMP
Date:	9/27/00
Ref:	V93801A

JE JACOBS ENGINEERING GROUP INC.
LAKELAND FLORIDA

FIGURE 20
FIPR
A CYCLONE CONTROL SCHEME

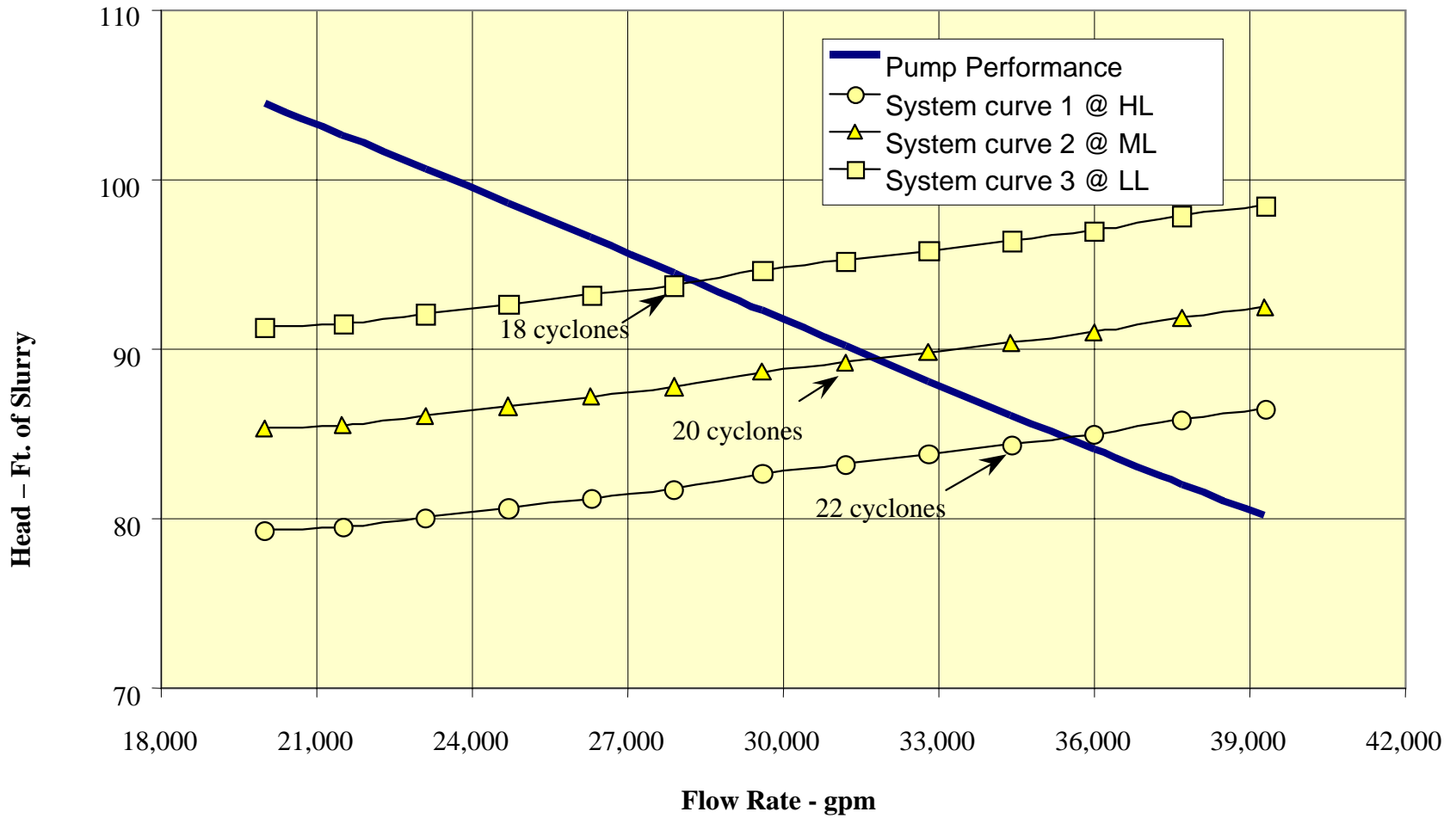


Figure 21. Example System Curves for Cyclone Control Scheme.

CONCLUSIONS

The following conclusions are based on the test results obtained from this study.

- Ultrasonic treatment of pebble partially decomposed clay balls and aggregates, but is less effective than the traditional log washing.
- Ultrasonic treatment of flotation feed only improved rougher flotation recovery and concentrate BPL grade with starvation levels of fatty acid. At the levels of fatty acid required for economic operation, ultrasonic treatment of feed did not significantly change flotation performance.
- Ultrasonic treatment of rougher concentrate before acid scrubbing improved de-oiling at low levels of sulfuric acid use but gave completely unsatisfactory de-oiling when no acid was used. De-oiling was enhanced by increased sonication time
- Ultrasonic treatment of clay slurry did not improve clay settling or consolidation.
- The viscosity of the tested clay slurries decreased with increased shear rate, which is characteristic for pseudoplastic fluids.
- The viscosity of the tested slurries increased exponentially with solids content according to the general equation $\mu_s = A \exp(BC)$. The A and B values changed for each sample, because the viscosity at a given % solids changed from sample to sample.
- Settling tests with phosphate particles in clay slurries confirmed that Stokes' Law applies. Fluid viscosity and particle diameter are major factors influencing settling velocity. For cyclone separations fluid viscosity and particle diameter remain important; however, centrifugal force is substituted for gravitational force.
- A control scheme to optimize primary cyclone performance and reduce pumping energy has been proposed. The scheme utilizes on-line viscosity measurement and tank level control.

REFERENCES

- Dallon DS, Christiansen EB. 1968. A settling correlation between drag coefficient and a newly-defined Reynolds number for single spheres in non-Newtonian liquids. Preprint 24C, Symposium on Selected Papers, Part III, 61st Ann. Mtg. AIChE, Los Angeles, Dec. 1-5, 1968.
- El-Shall H, Bogan M. 1994. Characterization of future Florida phosphate resources. Bartow (FL): Florida Institute of Phosphate Research. Report nr 02-082-105.
- Florida Phosphate Council. 1999. Florida phosphate facts. Lakeland (FL): Florida Phosphate Council.
- Gruber G, Moudgil BM, Somasundaran, P. 1995. Survey of anionic conditioning practice. In: Gruber G, Moudgil BM, Somasundaran P. Understanding the basics of anionic conditioning in phosphate flotation. Bartow (FL): Florida Institute of Phosphate Research. FIPR Contract nr 92-02-090. Tasks 1.3 and 1.4. p 4-1 to 4-17.
- Kelly EG, Spottiswood DJ. 1982. Introduction to mineral processing. New York: John Wiley & Sons. p 213-23.
- Lamont WE and others. 1975. Characterization studies of Florida phosphate slimes. Pittsburgh (PA): U.S. Bureau of Mines. Report of Investigations No. 8089.
- Sobieraj S, Farmer AD. 1993. The use of ultrasonics in mineral processing. Australian Minerals Industries Research Association. Ltd., Project No. 397, November 1993.
- Tarr DT. 1985. Hydrocyclones. In: Weiss, NL, editor. SME mineral processing handbook. New York: Society of Mining Engineers of the American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc. Chapter 2, Section 3D. p 3D10 – 3D45.
- Warneke W. 1998. Presentation to Beneficiation Technical Advisory Committee, Florida Institute of Phosphate Research, Bartow, FL, December, 1998.