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# Alumina Miniplant Operations— Separation of Aluminum Chloride Liquor From Leach Residue Solids by Classification and Thickening

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UNITED STATES DEPARTMENT OF THE INTERIOR
James G. Watt, Secretary

BUREAU OF MINES
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| UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT | | | |
|---|---|---|---|
| atm | atmosphere | hr | hour |
| Bé | Baumé | in | inch |
| deg | degree | L | liter |
| °C | degree Celsius | lb | pound |
| ft | foot | $lb/ft^3$ | pound per cubic foot |
| ft/hr | foot per hour | lb/hr | pound per hour |
| ft/min | foot per minute | lb/ton | pound per short ton |
| $ft^2$ | square foot | min | minute |
| $ft^2/lb$ | square foot per pound | mL | milliliter |
| $ft^2/tpd$ | square foot per short ton per day | pct | percent |
| g | gram | rpm | revolution per minute |
| gal | gallon | sec | second |

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# ALUMINA MINIPLANT OPERATIONS—SEPARATION OF ALUMINUM CHLORIDE LIQUOR FROM LEACH RESIDUE SOLIDS BY CLASSIFICATION AND THICKENING

By Roy T. Sorensen,[1] Dwight L. Sawyer, Jr.,[2] and Theodore L. Turner[3]

ABSTRACT

The Bureau of Mines has investigated the recovery of cell-grade alumina by HCl leaching of calcined kaolin in the alumina miniplant at its Boulder City (NV) Engineering Laboratory.

Classification and thickening were used for separating aluminum chloride leach liquor from the siliceous residue generated in the acid leaching step of the clay-HCl process. Coarse solids were classified from fines at 115 mesh and countercurrently washed in three additional spiral classifiers. Fines were treated in a conventional five-thickener, countercurrent decantation circuit. When this method was applied to the solids-liquid separation of slurry from HCl leaching of minus 10-mesh calcined kaolin, 75 pct of the residue reported as classifier sands and 25 pct as thickener underflow. Predicted individual classifier and thickener area requirements were 6 and 25 $ft^2$/tpd, respectively. Total thickener circuit flocculant requirement was 2.4 lb/ton solids. Aluminum chloride pregnant liquor was produced that analyzed 8.4 pct $Al_2O_3$, and contained more than 97 pct of the alumina in the liquid phase of the slurry. Bench-scale settling tests were used to correlate fines content of leach residue to thickener area and flocculant requirements.

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[2]Supervisory chemical engineer.
[3]Research chemist.
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# INTRODUCTION

## BACKGROUND

In 1975, the Bureau of Mines constructed and operated an alumina miniplant to study the production of cell-grade alumina from kaolinitic clay by the clay-HCl process. The process flowsheet shown in figure 1 includes a solids-liquid separation step in which solid residues in the leaching slurry are separated from the aluminum chloride liquor and washed countercurrently. Initially, filtration or thickening were considered suitable, although equipment requirements needed to be established.[4] Earlier miniplant-scale research using nitric acid instead of HCl indicated severe cloth blinding and excessive filter area requirements when pressure or vacuum filtration was used. Consequently, two thickeners in series were selected for solids-liquid separation in the preliminary testing in the clay-HCl miniplant. They were sized on the basis of bench-scale settling tests on leach slurry containing considerable quantities of fine solids.

In actual operation of the miniplant, packing of coarse residue particles in the thickeners caused plugging of the underflow pumping system. To eliminate the plugging condition, two screw classifiers were added to remove coarse solids from the thickener feed. Results from this circuit indicated that a classifier-thickener circuit was feasible, and a new circuit was designed and installed in the miniplant. Five thickeners in series were considered sufficient to indicate operational problems in any multiunit circuit and to predict alumina recovery in relation to the number of units. It was estimated that alumina recovery in four classifiers was the same as in five thickeners.

[4]Peters, F. A., P. W. Johnson, and R. C. Kirby. Methods for Producing Alumina From Clay. An Evaluation of Five Hydrochloric Acid Processes. BuMines RI 6133, 1962, 68 pp.

## MINIPLANT TEST PROGRAM

The first miniplant study (test 1) covered in this report was designed to demonstrate and evaluate the use of a four-classifier and five-thickener solids-liquid separation circuit in the clay-HCl process for the production of cell-grade alumina from kaolinitic clay, and to develop a material balance in terms of liquor, residue, and soluble alumina. A second study (designated tests 2, 3, and 4 of this report) was

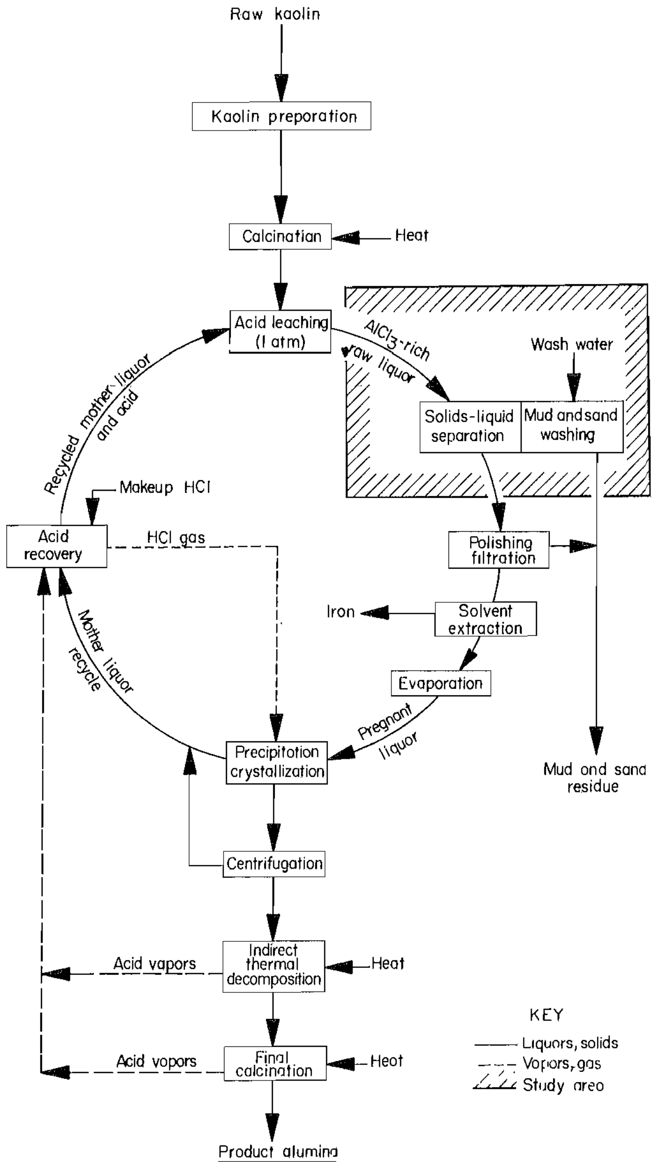


FIGURE 1. - Flowsheet of clay-HCl process.

conducted to determine the effect on aluminum recovery of circuit modifications intended to decrease the generation of fines by particle-to-particle attrition during solids-liquid separation, and to compare circuit response using minus 10-mesh calcined kaolin as the baseline leaching reactor feed with a minus 8-mesh calcined kaolin feed. Thickener area and flocculant requirements in both studies were to be determined by bench-scale settling tests.

The results of these two miniplant studies indicated that an evaluation of the effect of using calcined kaolin finer than minus 10 mesh on leaching slurry settling characteristics was required also. Therefore, the additional bench-scale investigation was conducted by an independent laboratory to compare settling characteristics of slurries produced by leaching different sizes and types of calcined kaolin with HCl.

## ACKNOWLEDGMENTS

The authors wish to acknowledge the valuable assistance provided by Travis Galloway, research engineer, Reynolds Aluminum Co., during the second miniplant study, and by N. Kelly Brown, formerly supervisor of testing, Envirotech Research Center, Envirotech Corp., for directing the independent, bench-scale settling evaluation.

## MATERIALS, EQUIPMENT, AND PROCEDURES

### CALCINED KAOLIN FEED TO LEACHING REACTORS

The raw kaolin used in the miniplant was from the Reedy Creek Mine of the Thiele Kaolin Co., Sandersville, GA, and was crushed and screened to provide a minus 20- plus 4-mesh product; the minus 20-mesh fines were pelletized to about 3/8-in on a conventional pelletizing disk. The mixed material was calcined in a rotary kiln, and was crushed and screened to the desired size. Screen analyses of the calcined kaolin feed products for the miniplant program are given in table 1. Calcined kaolin analyzed 42.7 pct acid-soluble alumina in test 1 and 40.4 pct in tests 2 through 4.

In the first miniplant study, calcined kaolin feed was elevated to an Acrison[5] weightometer by means of a screw elevator. To decrease the formation of fines by particle-to-particle attrition, the elevator was replaced by drum hoisting and a horizontal belt conveyor for miniplant tests 2 through 4.

[5]Reference to specific equipment, products, trade names, or manufacturers does not imply endorsement by the Bureau of Mines.

The calcined kaolin materials that were used in the independent laboratory program were prepared by two methods. The samples designated minus 10 mesh, minus 20 mesh, and minus 35 mesh were prepared by stage-crushing 1/4-in calcined kaolin pellets to size. A "misted" material was produced by mixing a minus 18-mesh raw kaolin clay with a fine mist of water while tumbling the clay on a pelletizing disk.[6] The water addition was sufficient to cause the finest particles to adhere to the coarser ones but was insufficient for pellet formation. Size increase incurred by the coarse particles was negligible. The material was calcined in a laboratory furnace at 750° C without agitation. To obtain the same fines content as that caused by particle-to-particle attrition in fluidized-bed calcination system, calcined material was agitated in an unheated fluidized bed reactor for a similar time period. Screen analyses of the materials before leaching are presented in table 2.

[6]Schaller, J. L., D. B. Hunter, and D. L. Sawyer, Jr. Alumina Miniplant Operations--Production of Misted Raw Kaolin Feed. BuMines RI 8712, 1982, 20 pp.

TABLE 1. - Screen analyses of calcined kaolin miniplant feed

(Cumulative weight retained, percent)

| Tyler standard sieve, mesh | Nominal feed size, mesh | |
|---|---|---|
| | Minus 10, tests 1 and 3 | Minus 8, test 2 |
| 10............ | 6 | 29 |
| 14............ | 29 | 46 |
| 20............ | 49 | 59 |
| 28............ | 62 | 70 |
| 35............ | 71 | 77 |
| 48............ | 78 | 83 |
| 65............ | 85 | 88 |
| 100........... | 90 | 92 |
| 150........... | 94 | 95 |

HCl FEED TO LEACHING REACTORS

Commercial 20 Bé muriatic acid (31 pct HCl) was diluted to 25 pct HCl before mixing with calcined kaolin. Both water and acid additions to the leach reactor train were metered by diaphragm-type pumps. Acid addition was set at 105 pct of the stoichiometric amount estimated for dissolving the acid-soluble alumina and iron in the calcined kaolin feed material.

FLOCCULANT

SEPARAN MGL synthetic polymer, a high-molecular weight, water-soluble nonionic polyacrylamide, was added as a 0.2-pct solution. The manufacturer was Dow Chemical Co., Midland, MI.

MINIPLANT LEACHING REACTORS

Leaching of calcined kaolin was carried out in three jacketed 50-gal, 24-in-ID, glass-lined steel reactors arranged in cascade. Hot oil circulating within the jacket provided temperature control. Acidic vapors from the boiling leaching liquors were condensed by water-cooled reflux condensers and the condensate returned to the reactors. The reactor overflows were located 16 in up the kettle sidewall, and gave about 23 gal effective volume per reactor. Stirrers were three-blade retreating curve design, glass-coated steel, and operated at 120 rpm.

The leaching slurry from the final reactor was cooled from 113° to about 60° C before discharging to the first classifier. A stirred mixer with a commercially pure titanium submerged cooling coil, a water-jacketed pipe, and a conventional glass condenser served as heat exchangers.

CLASSIFIER SECTION

Flared-tank screw classifiers were used in the classifier circuit and were rubber lined. Classifier specifications are given in table 3.

TABLE 2. - Screen analyses of calcined kaolin used in independent laboratory test program

(Cumulative weight retained, percent)

| Tyler standard sieve, mesh | Nominal feed size, mesh | | | | | | |
|---|---|---|---|---|---|---|---|
| | Minus 10 | 10 by 100 | Minus 18[1] | Minus 20 | 20 by 100 | Minus 35 | 35 by 100 |
| 14............ | 33 | 36 | NM | NM | NM | NM | NM |
| 20............ | 55 | 59 | NM | NM | NM | NM | NM |
| 28............ | 68 | 73 | 22 | 29 | 34 | NM | NM |
| 35............ | 77 | 82 | 53 | 51 | 60 | NM | NM |
| 48............ | 84 | 90 | 71 | 67 | 79 | 50 | 66 |
| 65............ | 89 | 96 | 81 | 77 | 91 | 64 | 85 |
| 100........... | 93 | NM | 80 | 85 | NM | 75 | NM |
| 150........... | 96 | NM | 94 | 91 | NM | 83 | NM |

NM Not measured. [1]Misted.

TABLE 3. - Classifier specifications

| Classifier number.... | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Cylindrical tank section ID......in.. | 18 | 10 | 10 | 10 |
| Tank length (overflow to discharge)...ft.. | 10 | 9 | 7.5 | 7.5 |
| Pool area.......$ft^2$.. | 9.6 | 6.4 | 7.1 | 6.9 |
| Freeboard length.ft.. | 3.5 | 3 | 2.5 | 2.5 |
| Slope..........deg.. | 29 | 21 | 30 | 30 |
| Screw diameter...in.. | 16 | 8 | 9.5 | 9.5 |
| Screw speed.....rpm.. | 0.09 | 0.40 | 0.86 | 0.86 |
| Screw speed, peripheral..ft/min.. | 0.42 | 0.84 | 2.13 | 2.13 |

## THICKENER SECTION

The thickeners were approximately 5-ft ID by 5 or 6 ft high and had rubber-covered rakes and shafts. The rakes plowed inward to a small conical section on a flat-bottomed tank. Thickener specifications are given in table 4.

TABLE 4. - Thickener specifications

| Thickener number..... | 1 or 2 | 3, 4, or 5 |
|---|---|---|
| Tank construction.... | ([1]) | ([2]) |
| Tank diameter....in.. | 58 | 60 |
| Depth...........ft.. | 6 | 5 |
| Area...........$ft^2$.. | 18.4 | 19.5 |
| Rake speed......rpm.. | 0.38 | 0.53 |
| Rake speed, peripheral..ft/min.. | 5.6 | 7.8 |

[1]Fiber-reinforced plastic.
[2]Rubber-lined steel.

## MIXING TANKS

Flocculant was mixed with process streams (fig. 2) in 3- and 7-gal, fiber reinforced plastic, center-stirred, cylindrical tanks. In the smaller mixer, the flocculant solution was diluted by the thickener overflow. In the larger mixer, the diluted solution was mixed with the advancing thickener underflow. Mixing time varied between 5 and 10 min. Mixing was accomplished by slow-speed stirring (about 60 rpm) with large perforated two-bladed impellers of 90° pitch, 8-in diameter by 1-1/2 in high, which were located several inches from the bottom of the tanks.

Initially, the larger mixers were used for mixing the advancing classifier sands with the returning classifier overflow between each classification stage. To decrease production of fines by particle-to-particle attrition, these mixers were replaced in the second study by inclined, baffled chutes. For the same reason, the smaller mixer initially used for preparing the leach reactor feed slurry by cold premixing of acid solution with calcined kaolin was replaced by an inclined sluice tube.

## MINIPLANT OPERATION

In the circuit shown in figure 2, the leaching reactor discharge, after cooling in a heat exchanger, was split in classifier 1 into a sands and an overflow product. The overflow product, which contained most of the fine residue, was mixed with flocculant and discharged into thickener 1. The classifier 1 sands were washed countercurrently in three classifiers, while the thickener 1 underflow was washed countercurrently in four thickeners. Thickener 1 overflow constituted the raw pregnant liquor product from the combined circuit; whereas, thickener 5 underflow and classifier 4 sands were the waste materials (tailings).

## BENCH-SCALE TESTING PROCEDURES

### General Procedures

Bench-scale tests were divided into two categories--process monitoring tests on miniplant slurries and an independent laboratory study on laboratory-leached slurries.

Settling was carried out in 2-L graduated cylinders with a four-wire stirrer run at 0.1 rpm. The stirrer consisted of four parallel 12-gage titanium wires (17-in long) connected at the top and bottom and diagonally spaced 2-3/8 in apart. The stirrer travel encompassed a 2-3/8-in-diam hollow cylinder within the 3-in-diam cylindrical graduate and extended to 1/2 in above the bottom of the

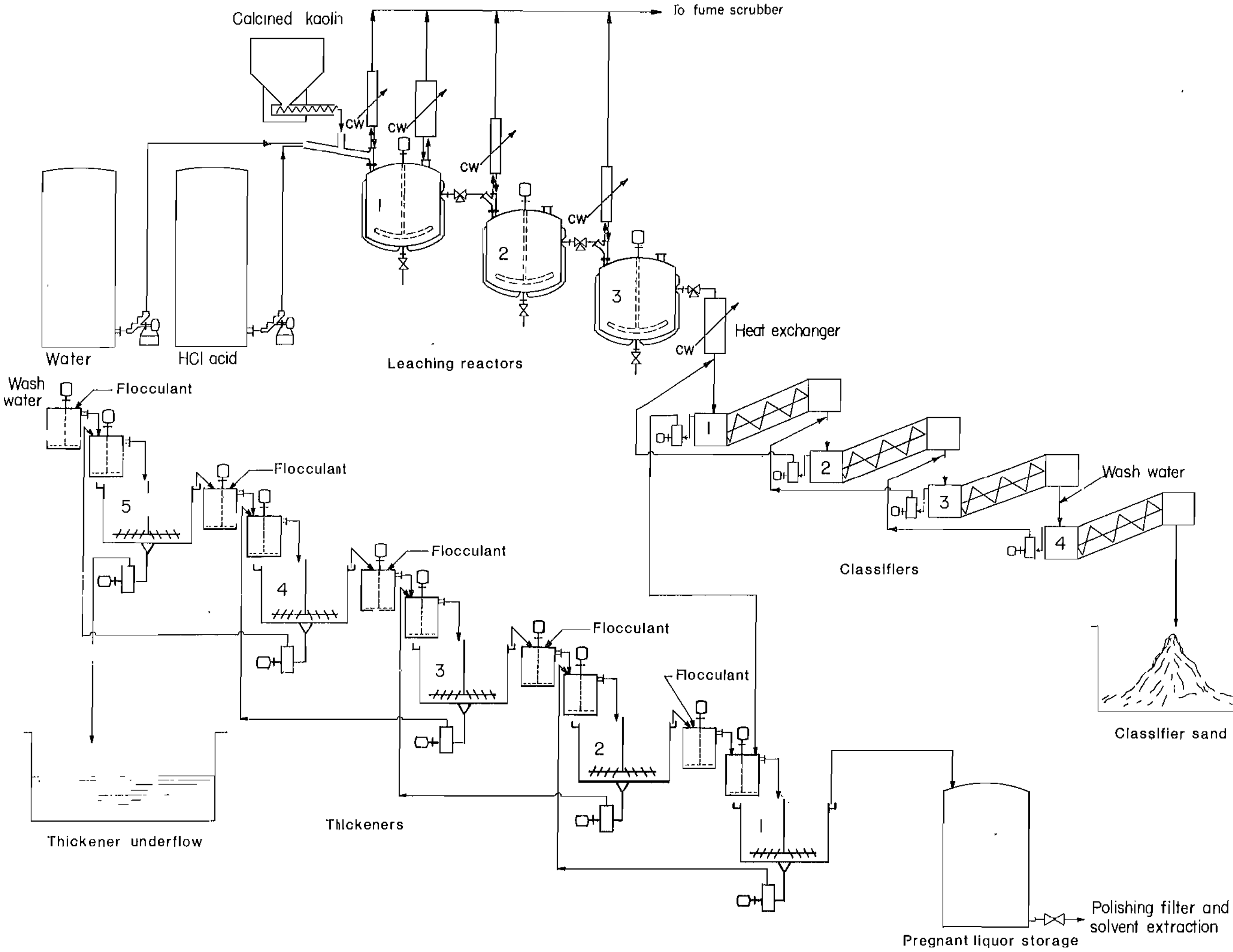


FIGURE 2. - Four-classifier and five-thickener solids-liquid separation circuit.

graduate. The graduate was kept in a hot water bath during the settling period. Interface heights were measured every 30 or 60 sec during initial settling and less frequently when the settling rate began to slow. Height measurement continued for 16 to 24 hr. On completion of the tests, the total sample was weighed. The clear liquor was decanted and its specific gravity measured. The solids were filtered, washed, dried, and weighed.

The settling area requirements were calculated by a standardized Kynch test method.[7] The interface height was plotted versus time. Examples are given in figures 3 and 4. Tangents were extended to the extremities of the curve. The angle formed by the tangents was bisected. The point at which the bisector intersects the curve is the critical point, $C_p$. A line tangential to the curve is drawn through $C_p$. A final concentration line is drawn at the final interface height of the test. If the compression had stopped, the line was labeled $D_\infty$. If cessation of compression was not established definitely, the line was labeled as of the time of the last reading, $D_{16\ hr}$ or $D_{24\ hr}$. The percent solids of the compressed slurry was calculated from the liquor density, solids density (estimated 2.3), and dry solids weight. The volume height line for the design solids ($D_{design}$) was calculated

[7]Kynch, G. J. A Theory of Sedimentation. Trans. Faraday Soc., 48, 1952, p. 166.

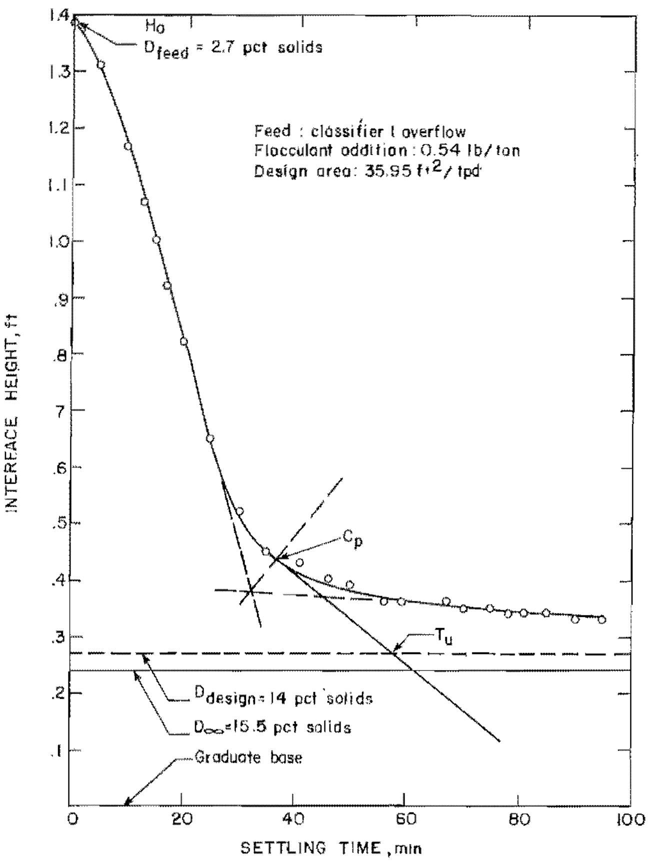


FIGURE 3. - Settling curve and Kynch analysis for miniplant test 3 classifier 1 overflow.

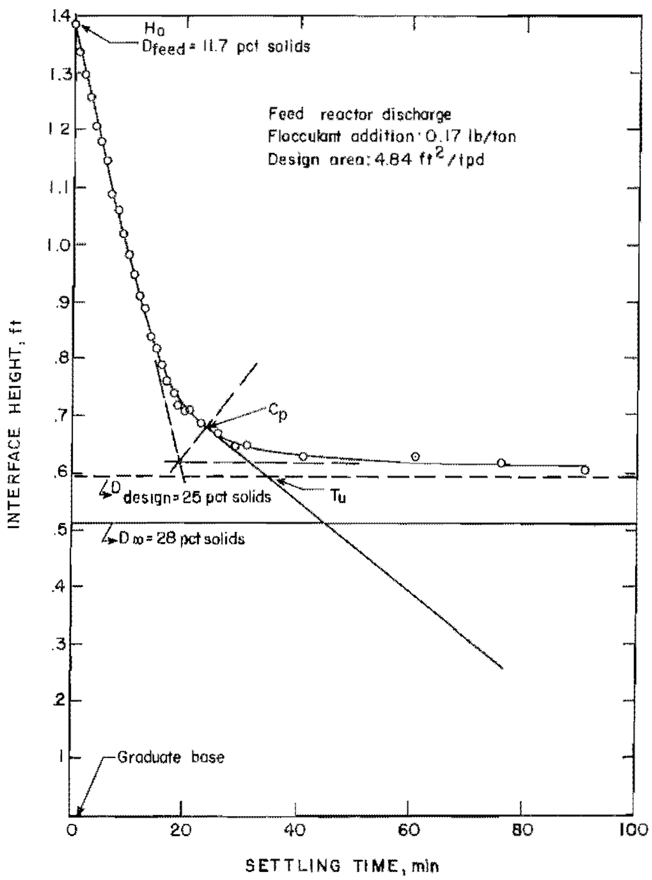


FIGURE 4. - Settling curve and Kynch analysis for miniplant test 3 reactor discharge slurry.

and plotted on the curve. The design solids was always less than the final individual test solids or average replicate group final solids.

The intersection of the curve tangent and the $D_{design}$ line is designated $T_u$. Its value on the time axis is converted from minutes to days. The design unit area ($A_{design}$) in square feet of thickener area per tons of solids per day is calculated from the values obtained and the following equation:

$$A_{design} = \frac{1.33 \times T_u}{C_o \ H_o},$$

where

$H_o$ = initial slurry height,

$$C_o = \frac{\text{weight of dry solids in feed, ton}}{\text{feed volume, cubic feet}},$$

and

1.33 = design factor.

The procedure was supplied by the contractor that made the independent laboratory study.

## Process Monitoring Testing Procedures

Settling tests were made on the following types of miniplant slurries:

1. Reactor 3 discharge.
2. Classifier 1 overflow.
3. Thickener 1, 2, 3, 4, and 5 feed.

A 1,450-mL sample of reactor 3 discharge slurry was diluted with 550 mL of a mixture of 0.2 pct SEPARAN MGL solution and actual miniplant thickener 2 overflow. If 10 mL of the flocculant solution was used, 540 mL of the thickener overflow solution was used. These volumes were set by the anticipated material balance flows. The thickener 2 overflow represented the countercurrent wash. The ratio of the volumes was close to the ratios of the actual miniplant flowstreams. The diluted flocculant solution and the reactor 3 discharge sample were blunged for 60 sec in a 2-L graduate before the settling test was started.

For settling tests on classifier 1 overflow, the ratio of classifier overflow to the mixture of flocculant solution and thickener 2 overflow was 1,470 mL to 530 mL, and was close to that of plant flowstreams.

The constant temperature bath was maintained at 80° C for settling tests on the reactor 3 discharge; this is the temperature expected in the first thickener handling leach slurry in a commercial operation. The temperature was maintained at 40° C for settling tests on the classifier 1 overflow; this is the temperature expected in the first thickener following heat exchange treatment and classification of leach slurry in a commercial operation.

In the tests run on feed slurry of the individual thickeners, approximately 2,000-mL samples were taken. No flocculant was added and settling was carried out in a 40° C constant temperature bath.

### Independent Laboratory Study Testing Procedures

Settling tests in the independent laboratory study were carried out on acid-leached slurries from batch leaching of different sizes and types of calcined kaolin. A 4-L glass resin kettle with a hemispherical bottom was used as the leaching reactor. The 3/4- by 4-in flat bladed, crescent-shaped, paddle-type impeller was set about 1 in above the bottom of the reactor and rotated at 185 rpm, which was the minimum speed for keeping the solids in suspension. The charge was 1,655 g of 26-pct HCl and 460 g of calcined kaolin. After addition of solids to the boiling liquor, leaching was carried out at boiling (about 107° C) for 30 min. The resulting slurry had a volume of about 1,520 mL and was diluted before each settling test with 380 mL of liquor containing about 6.7 pct $Al_2O_3$ (commercial aluminum chloride) to simulate a system using a countercurrent wash. The relative quantities of these bench-scale products and their concentrations were similar to those in the miniplant operation. For instance, miniplant thickener 2 overflow averaged about 6.3 pct $Al_2O_3$. The 0.2-pct flocculant was added and mixed by blunging with an inverted rubber stopper attached to a glass rod (five up-and-down strokes). An injection of one-fifth of the flocculant to the face of the stopper on each upstroke was made with a hypodermic syringe connected to a thin plastic tube that directed the solution to the face of the stopper. A thin titanium metal sheet over the end of the stopper dispersed the flocculant to the sides of the stopper. After mixing, which required about 15 sec, the plunger was removed. A stirrer operating at 0.1 rpm was inserted and settling begun.

# EXPERIMENTAL RESULTS

## CLASSIFIER-THICKENER CIRCUIT

### Test Parameters

Two miniplant studies were made covering four different sets of test conditions shown in table 5. As described in the "Materials, Equipment, and Procedures" section, a number of circuit modifications were made and process throughput was increased in the second study (tests 2, 3, and 4) to decrease production of fines by particle-to-particle attrition. Only one set of conditions (test 1) was used in the first study. In the second study, the major

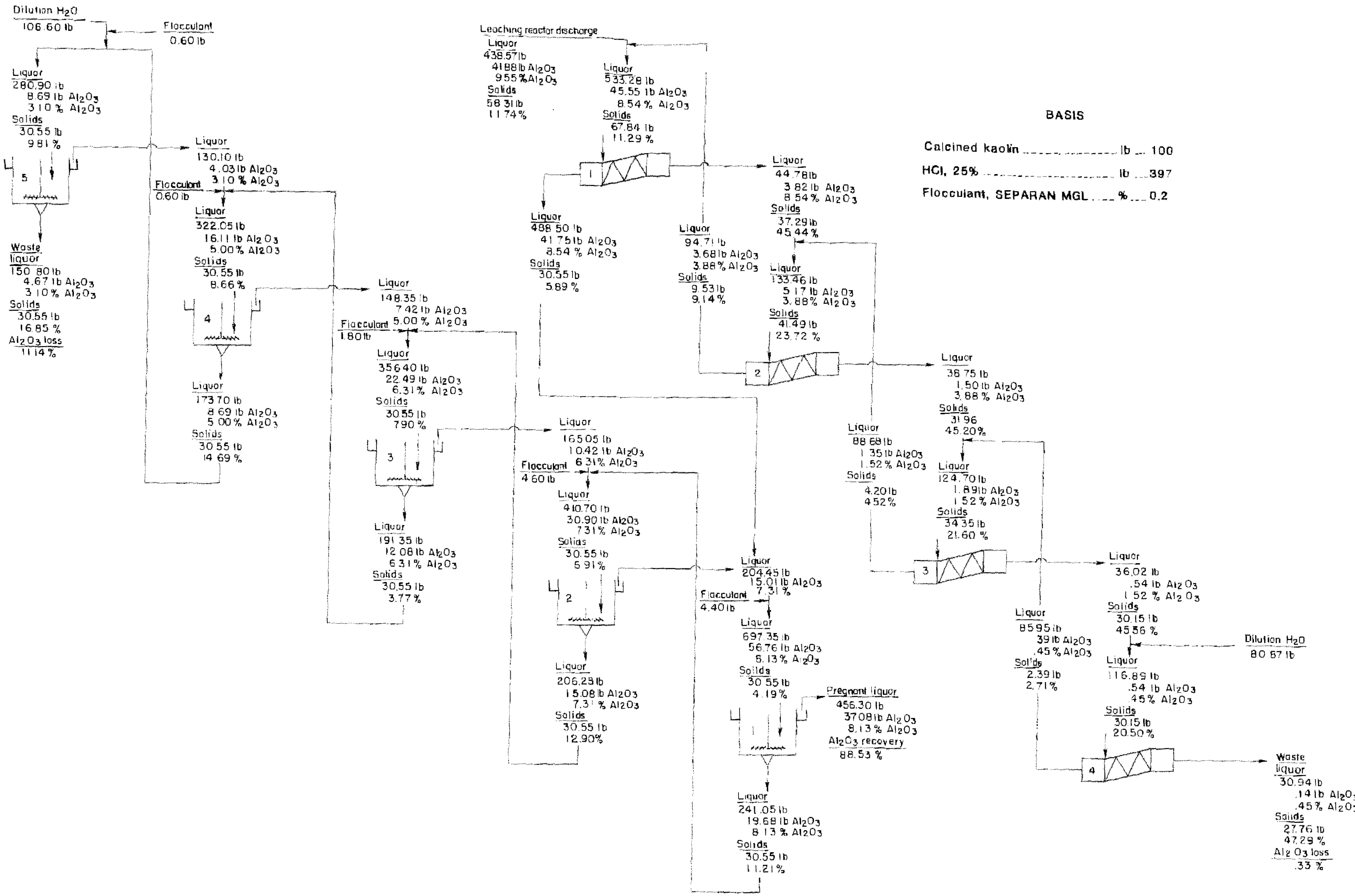


FIGURE 5. - Material balance for solids-liquid separation in miniplant test 1. Best fit of sample analyses and flow measurements.

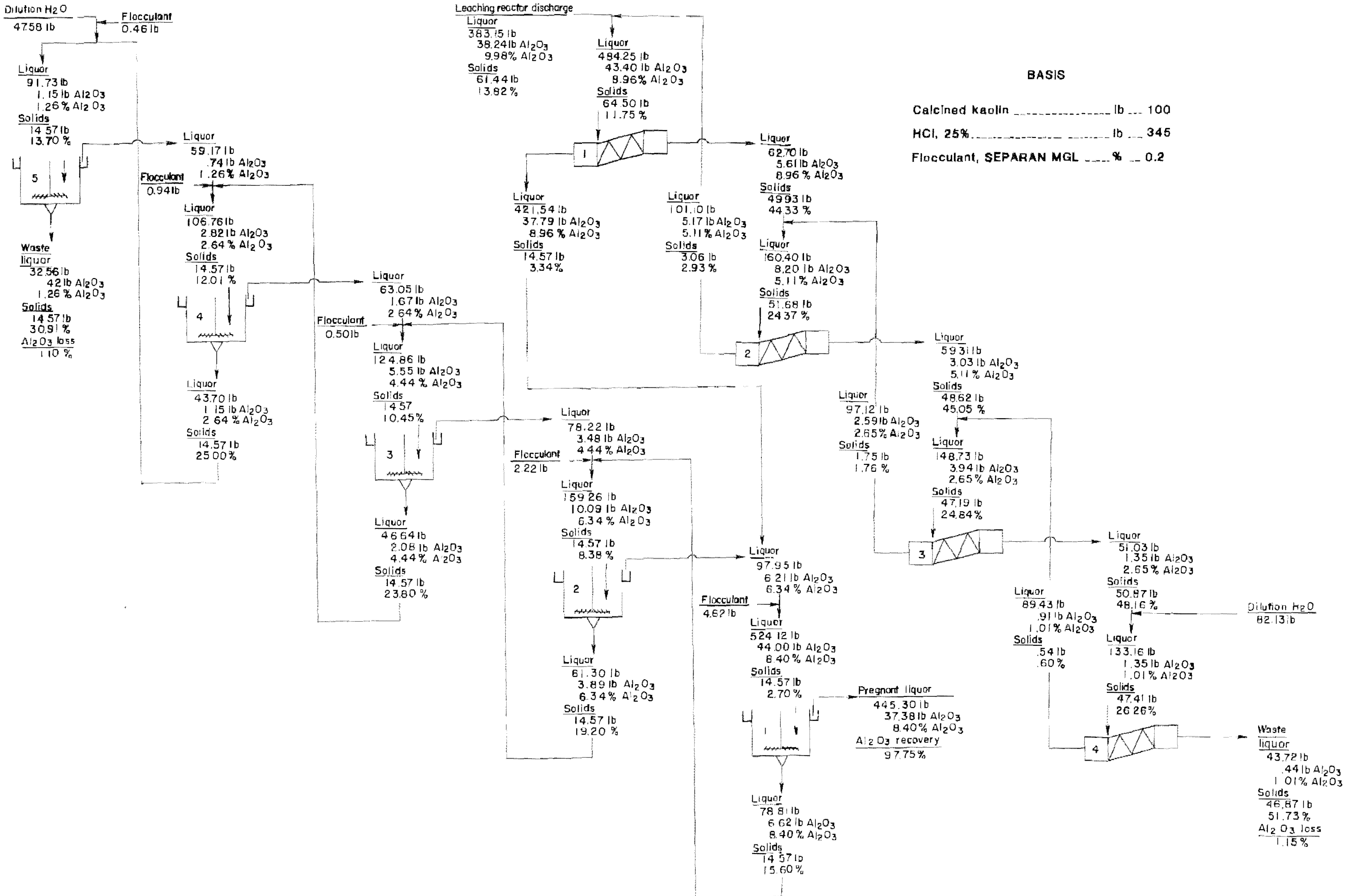


FIGURE 6. - Material balance for solids-liquid separation in miniplant test 3. Best fit of sample analyses and flow measurements.

TABLE 5. - Miniplant test schedule

| Test and study.... | 1, I | 2, II | 3, II | 4, II |
|---|---|---|---|---|
| Duration....days.. | 10 | 2 | 6 | 1.5 |
| Miniplant circuit. | U | M | M | M |
| Calcined kaolin feed: | | | | |
| Nominal top size.....mesh.. | 10 | 8 | 10 | 10 |
| Rate.....lb/hr.. | 100 | 173 | 175 | 243 |

M Modified to minimize production of fines by particle-to-particle attrition.
U Unmodified.

sampling and process evaluation effort was made for test 3. Test 2, which used minus 8-mesh calcined clay, was aborted after 2 days because of classification problems. Test 4 was run for 1-1/2 days to investigate circuit capacity. Fresh water addition to the thickeners was set about equal to the amount of liquor expected in the thickener 5 underflow. To obtain a steady operation, the wash addition to the classifiers had to be set considerably above the amount of liquor in the classifier 4 sands.

## Best Fit Material Balance

Solids-liquid separation material balances were made by best-fitting the average circuit flow rates and sample analyses to the constraints of the flowsheet in figure 2. The balances for tests 1 and 3 are presented in figures 5 and 6, respectively, and are on a basis of 100 lb/hr of calcined kaolin feed to the leaching section. The higher process throughput in test 3 and the circuit modifications to decrease particle-to-particle attrition resulted in increasing the aluminum recovery in the pregnant liquor from the solids-liquid separation from 88.5 pct in test 1 to 97.8 pct in test 3. $Al_2O_3$ contents in the pregnant liquors were 8.1 and 8.4 pct, respectively.

The increase in recovery resulted from the smaller amount of solids reporting to the thickeners and the higher thickener underflow densities in test 3. This is because the higher the underflow or sands fraction solids in a countercurrent washing system, the higher the washing efficiency or solute (in this case, aluminum chloride reported as $Al_2O_3$) recovery. These best fit balances represent the best estimate of average circuit conditions during the tests. Because thickeners 2 through 5 were improperly operated in test 1, and thickener pulp was pasty and contaminated with coarse particles; in test 3, it was necessary to modify these balances to predict results under proper operating conditions. The predicted balances are discussed later in this report.

## Classifier Separation

Classifier 1 separated both the fine residue solids and the pregnant liquor from coarse solids. Both classifier functions were dependent on residue solids particle size distribution, leaching slurry throughput, and slurry viscosity. Classifier performance is summarized in table 6.

The minus 150-mesh fines fraction of the leaching slurry solids fed to the classifier section varied from a high of 36 pct in test 1 to a low of 16 pct in test 4. Fines production was decreased owing to circuit modification and higher throughput, which also resulted in a

TABLE 6. - Classifier 1 performance summary

| Test[1].................. | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Feed solids: | | | | |
| Rate............lb/hr.. | 58 | 107 | 107 | 150 |
| Minus 150 mesh....pct.. | 36 | [2]24 | 21 | 16 |
| Overflow, settling velocity[3].......ft/min.. | 41 | 65 | 60 | 81 |
| Split, solids to thickener: | | | | |
| Pct of total........... | 51 | [2]77 | 25 | 24 |
| Size[4]...........mesh.. | 115 | [2]20 | 115 | 100 |
| Area requirement ft²/tpd solids.. | 12 | 7 | 7 | 5 |

[1]See table 5 for test conditions.
[2]Extremely viscous classifier feed slurry.
[3]Average upward flow velocity in pool area.
[4]Tyler standard sieve.

smaller fraction of the solids reporting to the thickener section. The decrease in fines from test 1 to test 3 is illustrated in figure 7 and resulted in a corresponding decrease of from 51 to 25 pct in the percentage of residue reporting to the thickeners. The least fines were contained in the classifier feed to test 4, when only 24 pct of the residue reported to the thickeners.

The classification size made in each test is illustrated by bar graphs (figs. 8 through 11) that were developed from flow rates in the material balances and screen analyses of the classifier products. The bars compare flow rates of

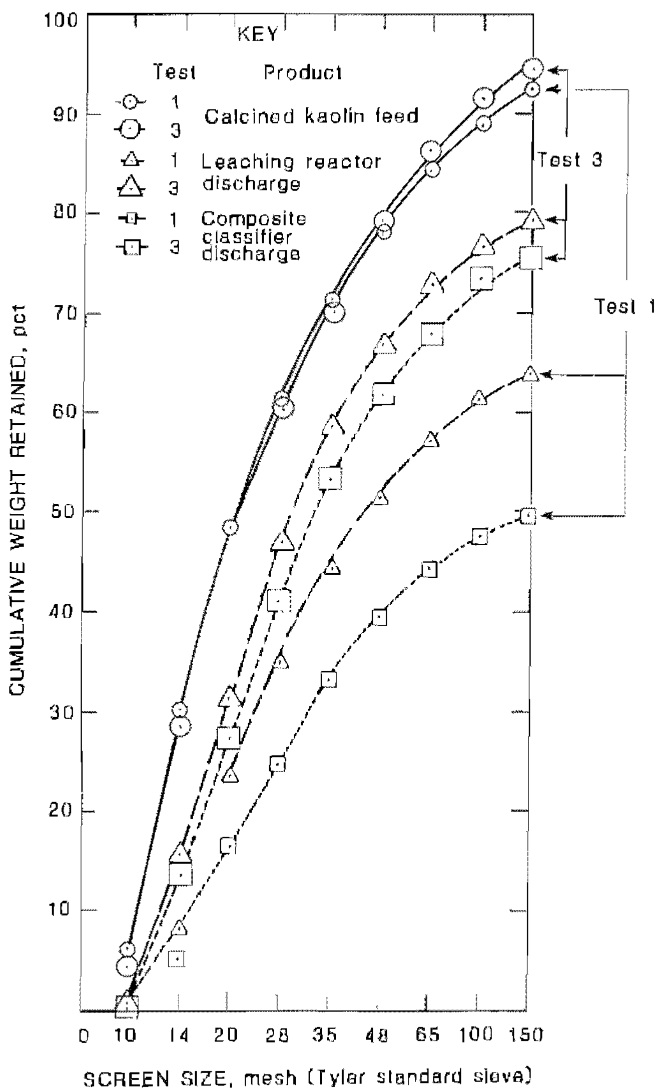


FIGURE 7. - Effect of leaching and classifier circuits on residue particle size, tests 1 and 3.

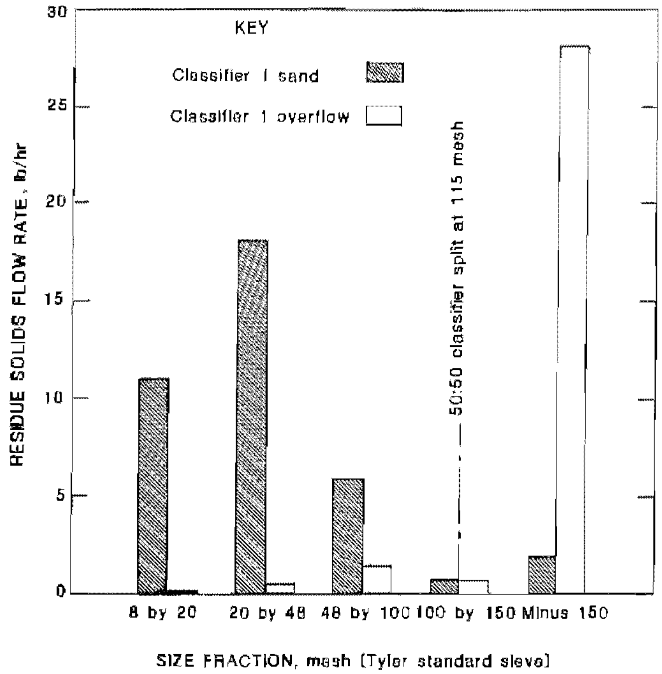


FIGURE 8. - Distribution of solids between classifier 1 overflow and sand products by size fraction, test 1.

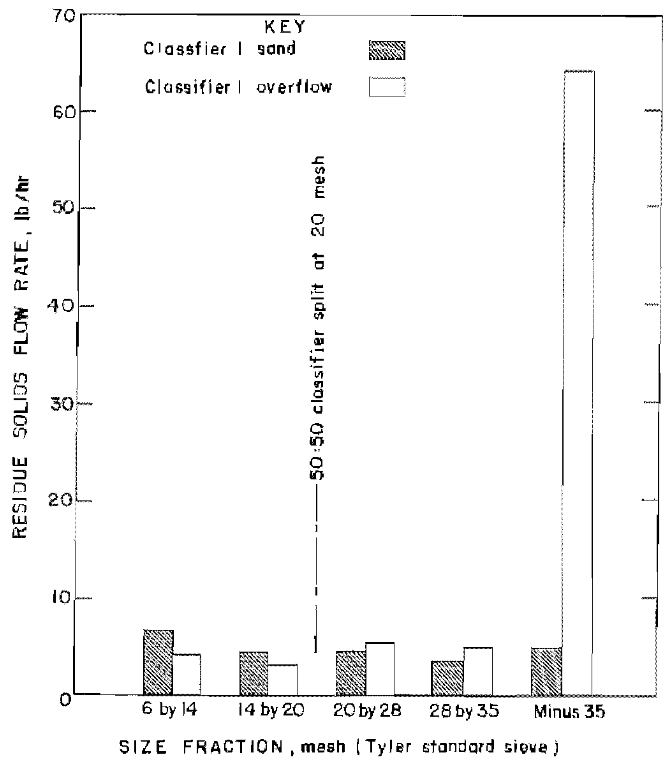


FIGURE 9. - Distribution of solids between classifier 1 overflow and sand products by size fraction, test 2.

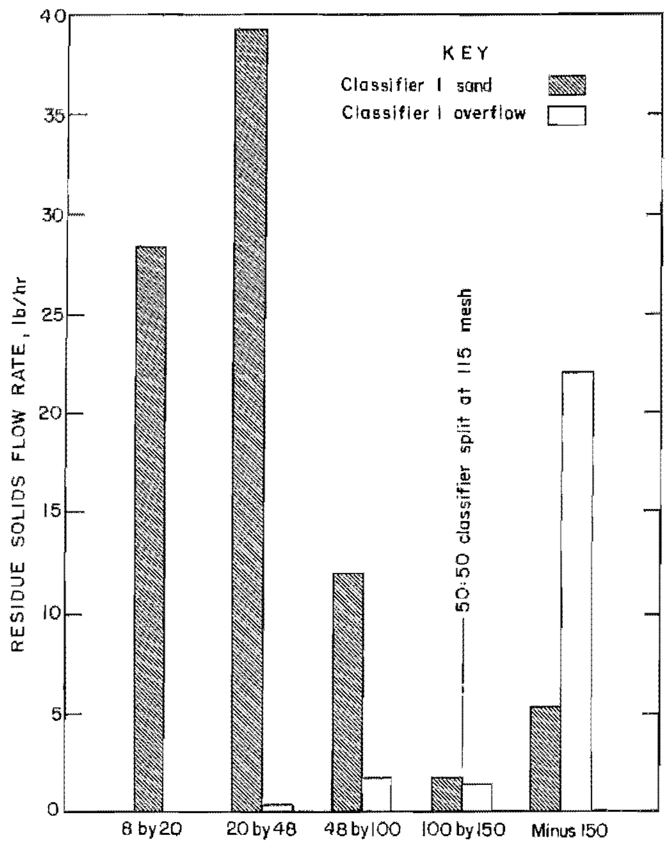


FIGURE 10. - Distribution of solids between classifier 1 overflow and sand products by size fraction, test 3.

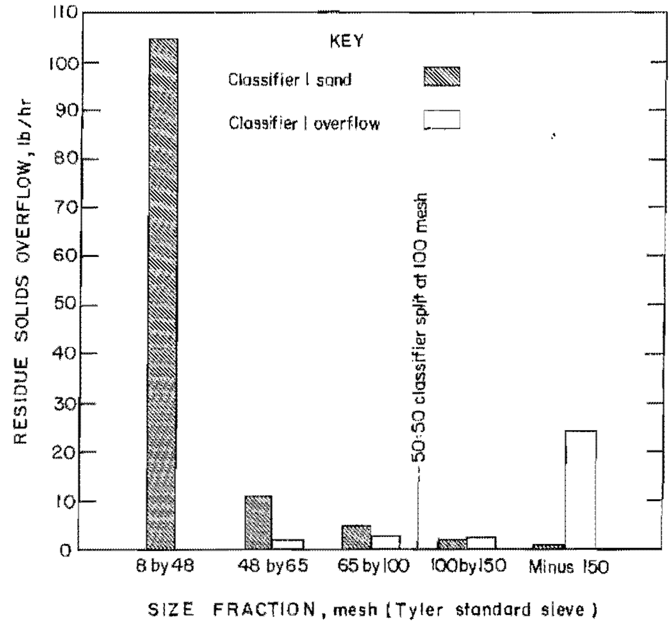


FIGURE 11. - Distribution of solids between classifier 1 overflow and sand products by size fraction, test 4.

specific size fractions in classifier 1 overflow with the same size fraction in classifier 1 sand. The bar graphs were used to determine the mesh size of the classifier split shown previously in table 6. The size at which particles reported to both overflow and sand in equal amounts was the split size. (In both tests 1 and 3 these equal amounts occurred in the 100- by 150-mesh fraction whose median size can best be represented by the Tyler fourth-root-of-2 series size of 115 mesh. In test 4, the equal amount fraction must be coarser than 100 by 150 mesh but finer than 65 by 100 mesh and can be best represented by 100 mesh.) Sharply defined splits were made in all tests except 2. In test 2 a poorly defined split at 20 mesh was obtained, and 77 pct of the residue reported to the thickeners. The inefficient classification was caused by a very viscous reactor discharge slurry. Since the minus 8-mesh calcined kaolin feed was too coarse for the reactors, solids built up and particle-to-particle attrition increased. The presence of very coarse material in the thickeners decreased overall $Al_2O_3$ recovery, caused underflow pumping problems, and negated the preclassification step.

Classifier 1 pool area requirements pertain to the actual miniplant pool area which, under the conditions listed in table 6, provided the solids splits described in figures 8 through 11. The decrease in classifier 1 pool area requirements from test 1 to 3 of 12 to 7 $ft^2$ per ton of solids treated per day reflected the corresponding increase in classifier throughput shown in table 6. This increase occurred without coarsening the size of the classifier split and was due to the decrease in fines content of classifier 1 feed from test 1 to test 3.

Assuming that particle size settling velocity in a classifier varies with the square of the particle diameter,[8] the classifier pool area requirements can

[8]Gaudin, A. M. Principles of Mineral Dressing. McGraw Hill Book Co., Inc., New York, 1939, p. 217.

be decreased by increasing the size of splits as follows:

| | | | | |
|---|---|---|---|---|
| Mesh of split........ | 115 | 100 | 65 | 48 |
| Percent of classifier pool area requirement at 115-mesh split............... | 100 | 71 | 36 | 18 |

In confirmation, the coarser (100-mesh) size split in test 4 accompanied a greater classifier input, which corresponded to an area requirement of 5 $ft^2$ per ton of solids per day, or about 71 pct of the 7 $ft^2$ of pool area used per ton per day in test 3.

On a basis of relative size of the actual miniplant classifiers, classifiers 2, 3, and 4 pool area requirements would be about three-fourths of that of classifier 1. On a basis of holding rising overflow velocities in classifiers 2, 3, and 4 the same as in classifier 1, about one-fourth of the classifier 1 pool area would be required.

## Thickener Compression

Miniplant thickener operation, testing, and evaluation were correlated to test 1 (first study) and 3 (second study) conditions.

Alumina recovery in the thickener section was proportional to the percent solids in the underflow streams. Estimates of the underflow solids under test 1 conditions are given in figure 12. The values obtained in the "best fit" material balance were a compromise between observed solids and the rest of the miniplant test measurements and were low. The low percent solids in thickeners 3 through 5 were due to an inability to pump the underflow at slow rates so that a buildup of settled solids in the thickeners could occur. Underflow pumping rates slow enough to maintain a 1-1/2-ft depth of settled pulp could be maintained in thickeners 1 and 2 because slurry in these thickener stages inherently settled to lower percent solids. Lower solids gave greater pump discharge volume when equal solids flow rates were maintained between thickeners. The measured thickener 1 underflow was 13.3 pct solids, was attained with the test 1 circuitry, and represents the expected value when operating with a sufficient inventory of settled solids.

When test 1 sampling was completed, an on-off timer was installed on thickener 5 underflow pump, and the inventory was increased from the 3 in it had held for days to 1 ft by maintaining slow pumping rates. The underflow held steady at 22 pct solids for several hours at an equilibrium pumping rate. If timers had been used, 22 pct solids could have been sustained in test 1 and also higher solids in thickeners 3 and 4.

Only the 13 pct solids for thickener 1 underflow and the 22 pct solids for thickener 5 were obtained with adequate pulp inventories. These two values and interpolated percent solids for the intermediate thickeners (fig. 12) are the only estimates for predicting thickener operation at adequate pulp inventories. A predicted material balance (fig. 13) developed by modifying the test 1 data and using these new values should provide a more reliable estimate of expected solids-liquid separation than the best fit material balance (fig. 5).

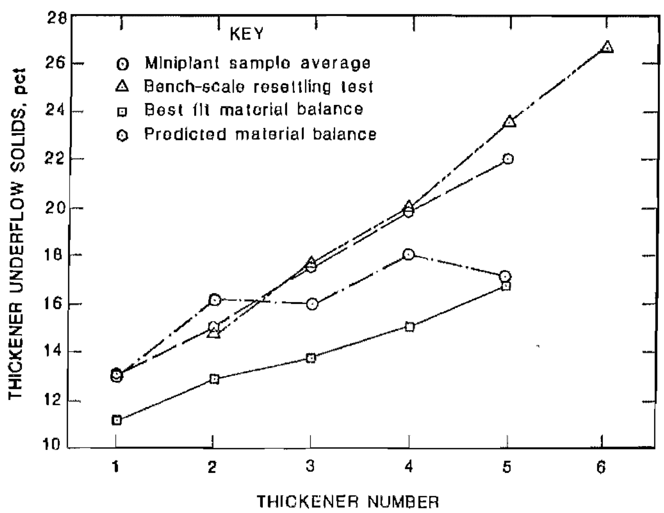


FIGURE 12. - Measured and calculated values for thickener underflow, percent solids, test 1.

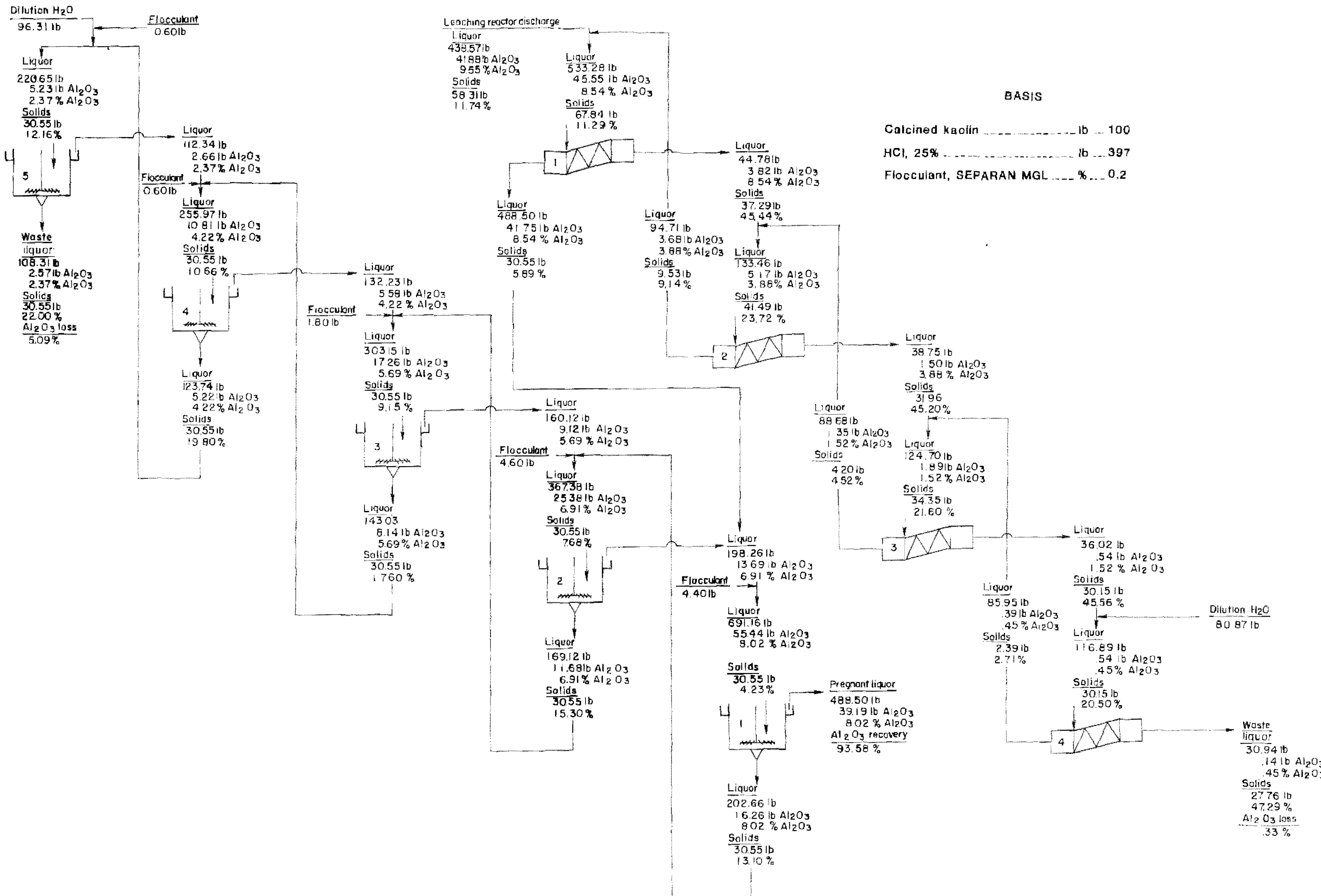


FIGURE 13. - Predicted best circuit material balance for solids-liquid separation in miniplant test 1.

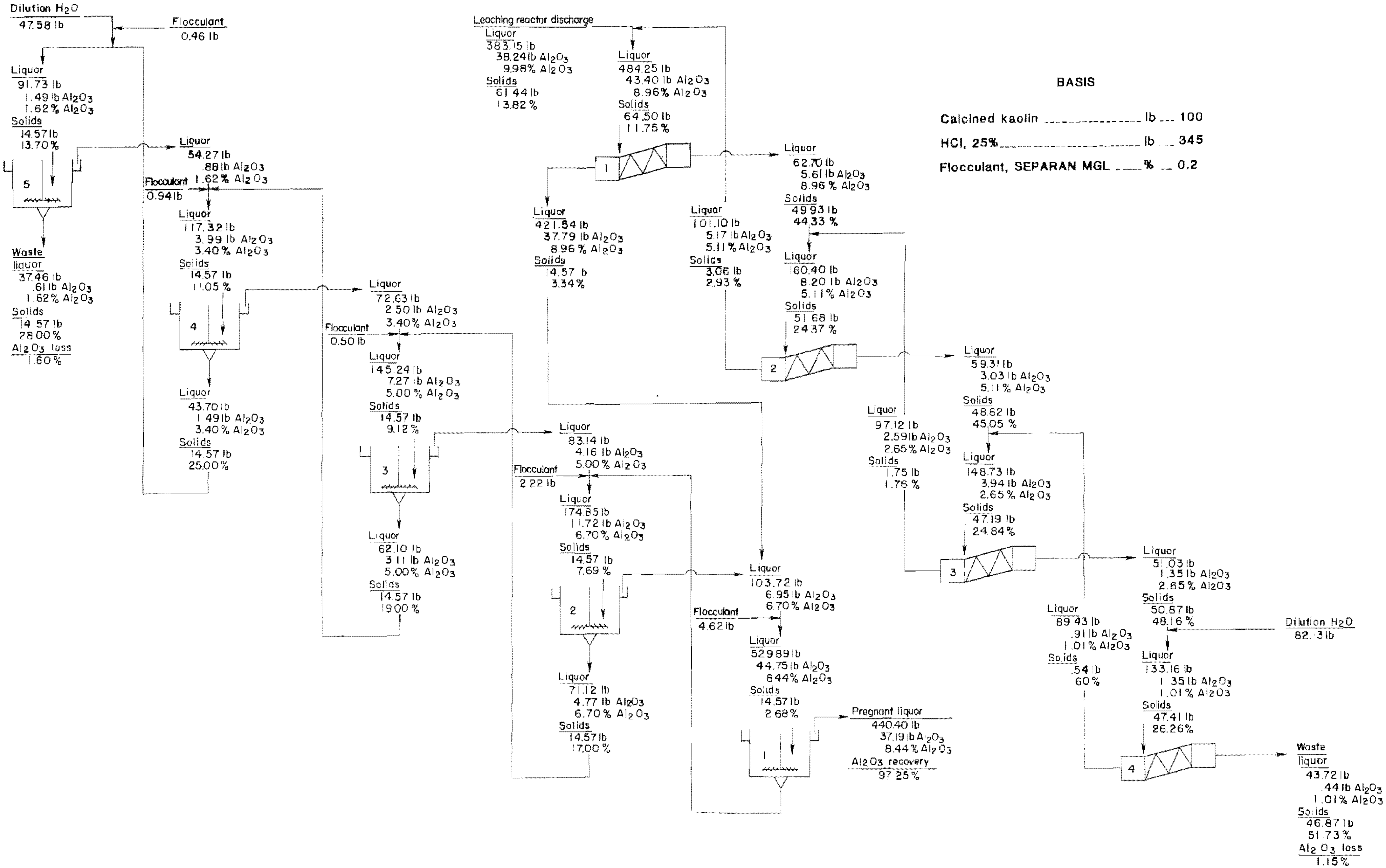


FIGURE 14. - Predicted best circuit material balance for solids-liquid separation in miniplant test 3.

An additional estimate of underflow solids was obtained by resettling 2-L samples of thickener underflows (fig. 12). Resettling of thickener 1 underflow gave an estimate for percent solids from thickener 2, and thickener 2 underflow resettling for thickener 3, etc. These data closely agreed with the underflow percent solids in the predicted material balance (fig. 14).

In the second study, all thickener underflow pumps were equipped with on-off timers so that slow pumping rates could be maintained to permit pulp inventories of 2 to 2-1/2 ft per thickener.

At the end of test 3 of the second study and when most of the minus 8-mesh material from test 2 had gone through the thickener circuit, bench-scale settling tests were made on feed to individual thickeners (table 7). The final underflow solids observed in the tests agreed with the average underflow solids from plant sampling and those obtained in developing the best fit material balance (fig. 15). However, miniplant thickener 4 and 5 underflows had a pasty consistency similar to their counterparts in the settling tests. Steady pumping of these products required a degree of operator attention intolerable in a commercial operation. Bench-scale observations indicated that a small amount of liquor markedly increased the fluidity of these products. Decreasing the underflow percent solids in a material balance for predicted operation (fig. 14) to the level of the $D_{design}$ values (table 7) would provide an underflow that is fluid enough to be tolerated in a commercial operation.

TABLE 7. - Settling tests using miniplant test 3 thickener feed slurries[1]

| Thickener | Solids, pct: Feed slurry | Solids, pct: Thickener underflow (design) | Average thickener area requirements,[2] $ft^2$/tpd |
|---|---|---|---|
| 1....... | 2.4 | 14.0 | 26.74 |
| 2....... | 6.7 | 17.0 | 26.08 |
| 3....... | NR | NR | NR |
| 4....... | 11.7 | 25.0 | 24.04 |
| 5....... | 14.8 | 28.0 | 32.99 |
| Av.... | NAp | NAp | 25.21 |

NAp Not applicable. NR Not run.

[1]Included miniplant recycle wash liquor and miniplant flocculant addition.

[2]Kynch method of analyses was used. The area requirements are in terms of tons of total solids to the thickener; would be 1.15 times as high in terms of minus 150-mesh solids, and 0.15 times as high in terms of calcined kaolin leaching feed.

## Predicted Material Balance

Predicted solids-liquid separation for the classifier-thickener circuit under the conditions of test 1 and 3 are presented in figures 13 and 14. Data summarized in table 8 show that predicted alumina recovery increased from 93.6 pct in test 1 to 97.3 pct in test 3. $Al_2O_3$ contents in the pregnant liquors were 8.1 and 8.4 pct, respectively. As in the best fit material balance, the higher recovery in test 3 was due to increases in the solids fraction reporting as classifier sands and in the thickener underflow solids. To attain a 99-pct $Al_2O_3$

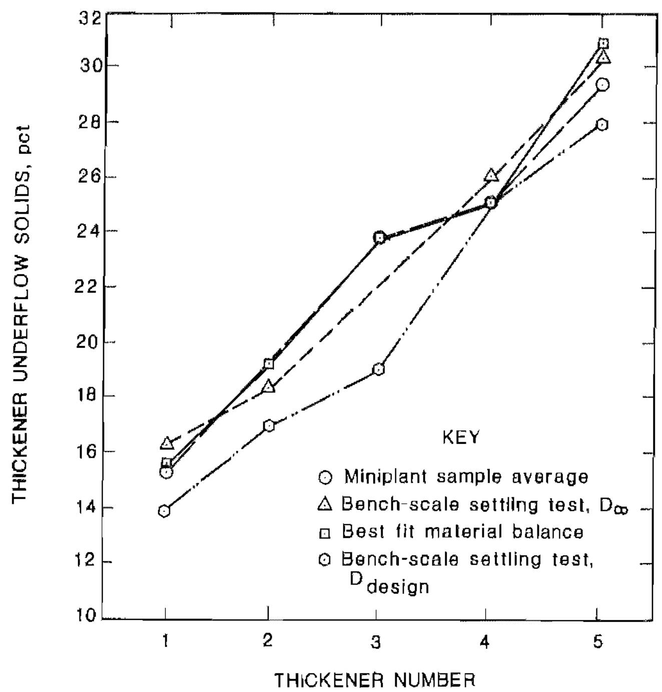


FIGURE 15. - Measured and calculated values for thickener underflow, percent solids, test 3.

recovery, it can be calculated with the test 3 material balance as a basis, that five classifiers and seven thickeners are required. A pregnant liquor product containing 8.6 pct $Al_2O_3$ would be obtained.

### Waste Disposal

The waste product from the solids-liquid separation consisted of thickener 5 underflow and classifier 4 sand. Half of the residue solids reported to the thickener 5 underflow in test 1 and one-quarter in test 3. As shown in table 8, thickener 5 underflow was 22 pct solids and classifier 4 sand was 47 pct solids in test 1 and significantly higher in test 3, 28 and 52 pct solids, respectively. The composite tailings in test 1 contained 29 pct solids and occupied 69 pct more volume than the as-mined clay. The composite tailings from test 3 contained 43 pct solids and theoretically occupied only 11 pct more volume than the as-mined clay if no air was entrapped in the mixture.

The thickener 5 underflow was very pasty during test 3 when its solids content approached or exceeded 30 pct. As the composited waste product from test 3 containing 43 pct solids could probably not be pumped, especially not as a thickener underflow, a thickener-only circuit could not provide a waste product as compact as that obtained in test 3. Because of the handling problem at high solids content in the thickener underflow, the combined tailings volume predicted from a hypothetical five-classifier and seven-thickener circuit should be at least 111 pct of the as-mined clay volume.

### Miniplant Thickener Area and Flocculant Requirements

In both miniplant studies, flocculant addition was first set by preliminary settling tests, modified by visual observation, and then held at the rates shown in table 9. The settling area requirements for test 3 as shown in table 7 are from settling tests on thickener feed that had the flocculant added in the miniplant. The relationship between thickener 1 area requirements and flocculant addition as shown in figure 16 is from bench-scale settling tests on classifier 1 overflow that had the flocculant added at the bench. With the 1.3 lb SEPARAN MGL per ton of solids added in the miniplant, thickener 1 requirements were 27 $ft^2$ per ton of solids per day in test 3 (table 7). If the flocculant were

TABLE 8. - Predicted final products from solids-liquid separation--miniplant tests 1 and 3

| Test | Product | Liquor, pct[1] | | Slurry | |
|---|---|---|---|---|---|
| | | $Al_2O_3$ analyses | $Al_2O_3$ distribution | Solids, pct[1] | Percent of mined clay volume[2] |
| 1.... | Raw pregnant liquor...... | 8.0 | 93.6 | 0.01 | NAp |
| | Classifier 4 sand........ | .5 | .3 | 47.3 | 45 |
| | Thickener 5 underflow.... | 2.4 | 6.1 | 22.0 | 121 |
| | Composite waste product.. | 1.9 | 6.4 | 29.5 | 169 |
| 3.... | Raw pregnant liquor...... | 8.4 | 97.3 | .01 | NAp |
| | Classifier 4 sand........ | 1.0 | 1.1 | 51.7 | 46 |
| | Thickener 5 underflow.... | 1.6 | 1.6 | 28.0 | 65 |
| | Composite waste product.. | 1.3 | 2.7 | 43.1 | 111 |

NAp Not applicable.
[1]Based on predicted material balances, figures 13 and 14.
[2]Based on 76 $lb/ft^3$ of dry kaolin in place as mined and 1.163 lb of dry kaolin per pound of calcined kaolin. These are calculated from values supplied by D. White, former Georgia State Liasion Officer, Bureau of Mines, and from Dana's Manual of Mineralogy, 7th ed., 1951. Residue solids density of 2.3 and liquor densities estimated from $Al_2O_3$ analyses were used.

TABLE 9. - Average flocculant addition per thickener in miniplant tests 1 and 3, pound SEPARAN MGL per ton of solids thickened

| Thickener | Test 1 | Test 3 |
|---|---|---|
| 1............. | 0.58 | 1.27 |
| 2............. | .60 | .61 |
| 3............. | .24 | .14 |
| 4............. | .08 | .25 |
| 5............. | .08 | .12 |
| Total....... | 1.58 | 2.39 |

added at the bench, figure 16 shows that the area requirements of 27 $ft^2$ per ton of solids per day is attained with only 0.07 lb/ton. The longer retention time in the miniplant mixer (5 min versus 1 min) and/or the different miniplant mixing agitation (stirrer versus blunger) may have caused less efficient conditions in the miniplant.

Bench-scale settling tests on individual miniplant thickener feeds (table 7) show that the thickeners have almost identical area requirements of 25 $ft^2$/tpd at the miniplant flocculant additions shown in table 9.

## THICKENER-ONLY CIRCUIT

### Tests on Miniplant Leached Slurry

Preclassification was required in the miniplant before thickening of leaching slurry from minus 10-mesh calcined kaolin because the thickener underflow pumps could not handle the coarsest material. Larger scale equipment or even a finer sized leaching feed would make the use of classifiers optional. To evaluate a thickener-only circuit, bench-scale settling tests were made on unclassified miniplant leaching reactor discharge produced during tests 1 (study I) and 3 (study II) and are summarized in figure 17. Lower fines content of the reactor discharge in test 3 than in test 1 resulted in a drastic decrease in the bench-scale flocculant requirements and the calculated (Kynch method) area requirements.

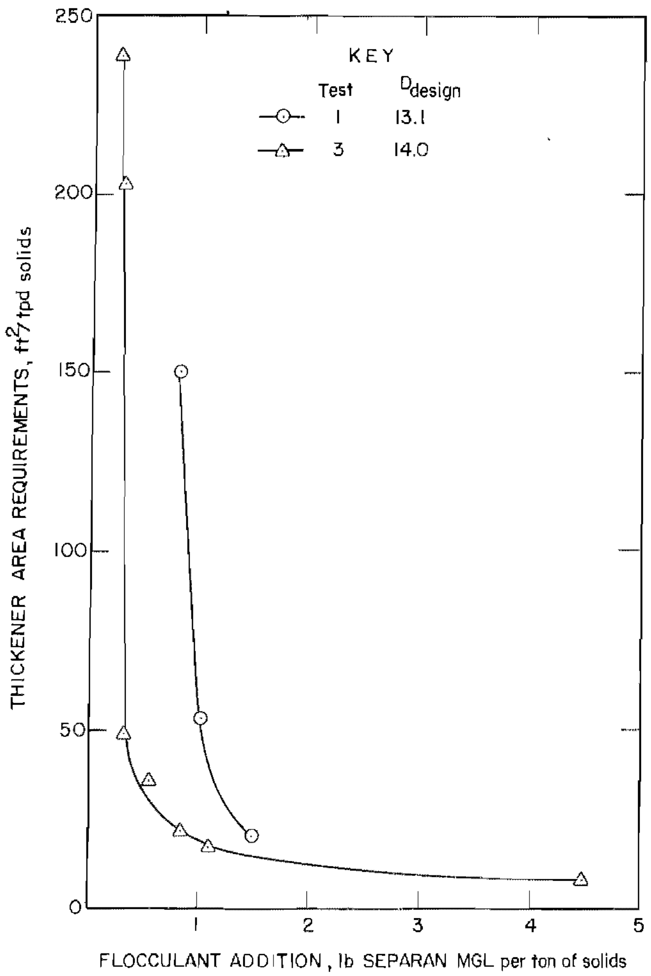


FIGURE 16. - Flocculant addition versus thickener area requirements for bench-scale settling of miniplant tests 1 and 3 classifier 1 overflow.

A comparison with testing on classifier 1 overflow (figure 17 with figure 16) indicates an appreciable decrease in thickening requirements when the classifiers were eliminated. Underflow solids were higher when preclassification was eliminated (20 to 25 pct solids as opposed to 13 to 14 pct solids). These differences were due to the amounts of fines in the thickener feed. The fines in the thickener feed were critical to the relation between flocculant addition and thickener area requirement. If the SEPARAN MGL addition in pounds per ton of minus 150-mesh solids is plotted against thickener area requirements in square feet per ton of minus 150-mesh solids per day, figure 18, the classifier 1 overflow

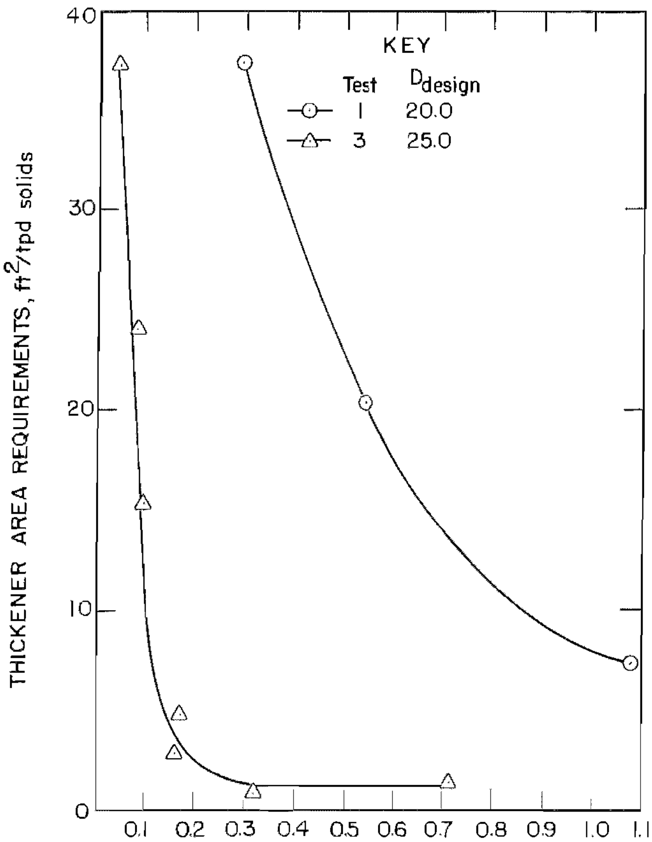


FIGURE 17. - Flocculant addition versus thickener area requirements for bench-scale settling of mini-plant tests 1 and 3 leaching reactor discharge.

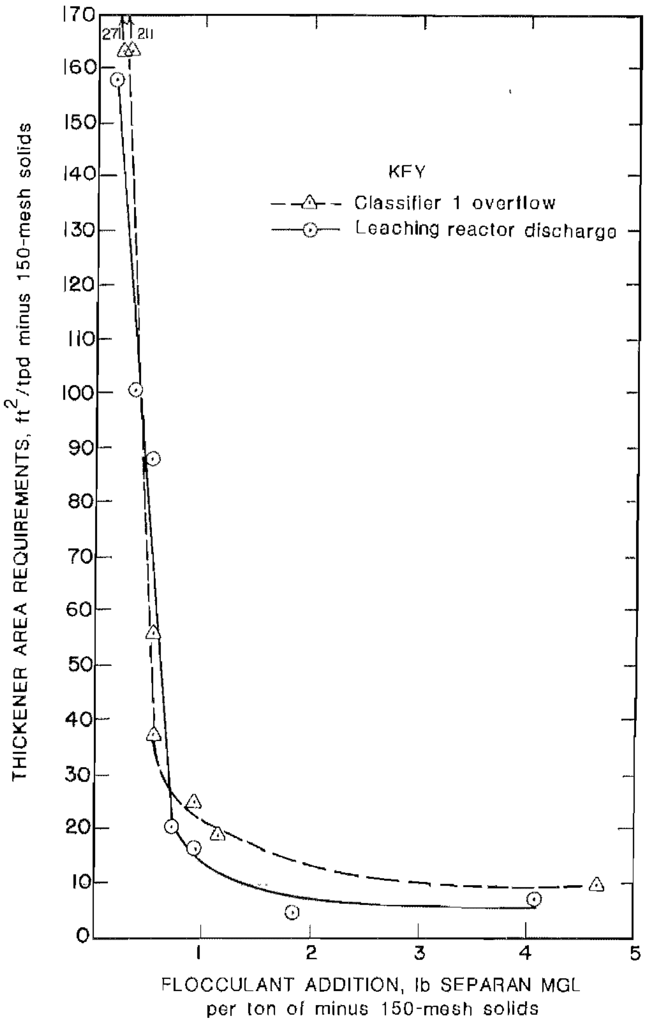


FIGURE 18. - Flocculant addition versus thickener area requirements in terms of minus 150-mesh solids for bench-scale settling of miniplant test 3 products.

settling curve coincides with the leaching reactor discharge settling curve. Settling area and flocculant requirements are a function of the minus 150-mesh fraction in the thickener feed slurry and will not be affected by previous removal of coarse material. In terms of tons of minus 150-mesh solids fed to the thickener, 1.5 lb of flocculant per ton corresponds to a thickener area requirement of 15 $ft^2$/tpd. A flocculant addition of about 0.2 lb of SEPARAN MGL per ton of calcined kaolin fed to the leaching circuit corresponds to a thickener requirement of 2 $ft^2$/tpd.

Percent solids in the thickener underflow are inversely proportional to the percent minus 150-mesh solids in the thickener feed. If the final settled solids ($D_\infty$) from the settling tests and the design settled solids ($D_{design}$) are plotted as a function of the minus 150-mesh in the thickener feed, a curvilinear relationship is established as shown in figure 19.

## Tests on Bench-Scale Leached Slurry

Figure 19 includes data from tests on bench-scale leached slurry. In these tests, calcined kaolin of different sizes (table 2) were batch leached in a bench-scale reactor. Samples with the minus 100-mesh material screened out were tested to evaluate a circuit in which fines are removed by air classification and returned to the calcination operation after pelletizing. Settling tests were made on the leaching slurry, using the Kynch technique of analysis. The solids

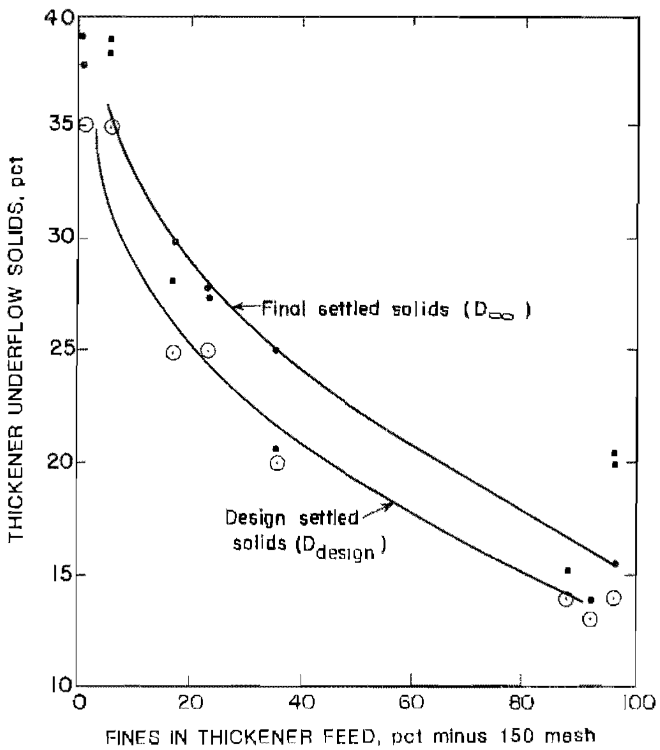


FIGURE 19. - Effect of fines in thickener feed on final settled solids and design settled solids.

in the slurries from the bench-scale leaches varied considerably in fines content as is shown in table 10 along with a summary of the settling tests. Flocculant and thickener area requirements for the bench-scale leached minus 10-mesh calcined kaolin were less than for the miniplant leached slurry (table 10). Less fines were produced in bench-scale leaching.

Flocculant and thickener area requirements in table 10 were higher when settling the finer sized material except for the minus 18-mesh misted material for which minimal thickener area and flocculant requirements were obtained. Screening out fines before leaching decreased flocculant requirements but not thickener area requirements.

## Predicted Thickener-Only Material Balance

Alumina recovery in a thickener-only solids-liquid separation circuit is dependent on the number of thickeners, the

TABLE 10. - Summary of thickener parameters and requirements from bench-scale settling tests on both miniplant and bench-scale leaching slurry

| Nominal calcined kaolin size mesh.. | Miniplant Test 1 | Miniplant Test 3 | Bench-scale reactor Minus 10 | Minus 18[2] | Minus 20 | Minus 35 | 10 by 100 | 20 by 100 | 35 by 100 |
|---|---|---|---|---|---|---|---|---|---|
| | Minus 10[1] | | | | | | | | |
| REACTOR DISCHARGE SETTLING | | | | | | | | | |
| Feed fines content pct minus 150 mesh.. | 36 | 21 | 6 | 6 | 15 | 19 | 1 | 3 | 6 |
| Flocculant requirements lb SEPARAN MGL per ton solids.. | 0.5 | 0.5 | 0.23 | 0.04 | 0.17 | 0.27 | 0.06 | 0.04 | 0.08 |
| Area requirements $ft^2$/tpd solids.. | 20 | 1 | 0.86 | 0.80 | 2.00 | 2.41 | 0.85 | 2.18 | 1.15 |
| Design underflow density pct solids.. | 20 | 25 | 35 | 35 | 35 | 35 | 35 | 35 | 35 |
| CLASSIFIER 1 OVERFLOW SETTLING | | | | | | | | | |
| Feed fines content pct minus 150 mesh.. | 92 | 87 | NR | NR | NR | NR | NR | NR | NR |
| Flocculant requirements lb SEPARAN MGL per ton solids.. | 1.5 | 1.0 | NR | NR | NR | NR | NR | NR | NR |
| Area requirements $ft^2$/tpd solids.. | 21 | 20 | NR | NR | NR | NR | NR | NR | NR |
| Design underflow density pct solids.. | 13.1 | 14.0 | NR | NR | NR | NR | NR | NR | NR |

NR Not run. [1]Baseline. [2]Misted.

underflow solids content, and the dilution wash. Table 11 compares the alumina recovery and content in the pregnant liquor predicted for thickening systems with 28 and 35 pct underflow solids. Ten thickeners are required to attain 99 pct $Al_2O_3$ recovery at 28 pct solids underflow but only seven at 35 pct underflow. All multistage settling tests on HCl-leached kaolin have indicated a significant increase in underflow percent solids with each thickening stage. The bench-scale testing on miniplant leached slurry indicated underflows of 20 to 25 pct solids. The 28- to 35-pct solids levels represent estimates for the underflow after seven or more stages.

TABLE 11. - Effect of thickener underflow solids and number of stages on estimated pregnant liquor alumina recovery and content in a thickener-only circuit

| Underflow solids, pct | Stages[1] | $Al_2O_3$, pct | |
|---|---|---|---|
| | | Analysis | Recovery |
| 28......... | 7 | 8.4 | 96.7 |
| 28......... | 10 | 8.6 | 99.0 |
| 35......... | 7 | 8.6 | 99.0 |
| 35......... | 10 | 8.7 | 99.7 |

[1]Washing water and flocculant additions set at about same amount as in clay-HCl acid miniplant test 3 (138 lb per 100 lb calcined kaolin leached).

## SUMMARY AND CONCLUSIONS

Solids-liquid separation tests were made in the clay-HCl miniplant to evaluate a four-classifier and five-thickener circuit at feed rates of from 100 to 243 lb/hr of calcined kaolin. Material balances were made for two different test conditions. Test 1 was performed at a feed rate of 100 lb of minus 10-mesh calcined kaolin per hour, while test 3 used the same leaching circuit, a feed rate of 173.5 lb/hr and inclined chutes in lieu of stirred mixers. Decreased production of fines by attrition in test 3 improved alumina recovery in the pregnant liquor as shown in the following, in percent:

| Test | $Al_2O_3$ recovery | $Al_2O_3$ content |
|---|---|---|
| 1... | 88.5 | 8.1 |
| 3... | 97.8 | 8.4 |

Both batch and miniplant-scale ancillary testing indicated that percent solids in the thickener underflow would have been higher in test 1 if adequate settled solids inventory in the thickeners had been maintained. Conversely, percent solids in the thickener underflow would have been less in test 3 if the settled solids had not been contaminated with coarse solids from the preceding test. Predicted final residue solids which are corrected for thickener operating conditions are

| Test | Unit | Solids, pct |
|---|---|---|
| 1 | Classifier | 47 |
| 3 | ...do.... | 52 |
| 1 | Thickener | 22 |
| 3 | ...do.... | 28 |
| 1 | Composite | 28 |
| 3 | ...do.... | 43 |

Predicted material balances based on revised discharge solids provided more meaningful estimates of alumina recovery in the pregnant liquor as follows, in percent:

| Test | $Al_2O_3$ recovery | $Al_2O_3$ content |
|---|---|---|
| 1... | 93.6 | 8.1 |
| 3... | 97.3 | 8.4 |

The combined tailings predicted from test 3 occupied 11 pct more volume than the as-mined clay compared with 67 pct more in test 1. The differences are related to the decrease in fines production which decreased the amount of solids reporting to the thickeners. The solids splits are summarized below for the two comprehensive tests (1 and 3) and two shorter tests (2 and 4).

| Test | Calcined kaolin, lb/hr | Solids to thickener, pct |
|---|---|---|
| 1... | 100 | 52 |
| 2... | 175 | 77 |
| 3... | 173 | 26 |
| 4... | 243 | 24 |

In test 2, calcined kaolin too coarse (minus 8 mesh) for adequate suspension in the leaching reactors caused excessive fines production and prevented efficient classification. Operating the system with minus 10-mesh calcined kaolin at a higher feed rate (243 lb/hr versus 173 lb/hr) did not change the solids split significantly.

The particle size of classifier split was 115 mesh (Tyler) for the two comprehensive tests (1 and 3), but increased to about 20 mesh with the minus 8-mesh calcined kaolin (test 2). Increasing the minus 10-mesh calcined kaolin feed rate to 243 lb/hr increased the particle size of split from 115 to 100 mesh.

Classifier 1 area requirements for the classifier-thickener circuit are dependent on the size of separation, as shown in the following:

| | | |
|---|---|---|
| Split, mesh (Tyler sieve).. | 115 | 100 |
| Area, square feet per ton per day.................. | 7 | 5 |

Classifiers 2, 3, and 4 area requirements would be three-fourths of these values on a basis of the ratio of miniplant classifier 1 pool area to classifier 2, 3, or 4 pool area or one-fourth of these values on a basis of equal rising velocities in the classifier pool.

Flocculant addition and thickener area requirements were developed from bench-scale tests on slurry produced in the miniplant. The requirements summarized in table 10 indicated the following:

1. Low fines content in the leaching slurry required low thickener area and thickener flocculant additions.

2. Settling fine solids in the classifier 1 overflow required more thickener area and more flocculant per ton of solids settled than for thickening the leaching slurry directly.

3. Flocculant addition and area requirements per ton of calcined kaolin treated were the same for classified and unclassified feeds. If the classifiers were eliminated, no additional thickener area would be required.

4. Percent soilds in the thickener underflow were shown to vary inversely with the percent fines in the thickener feed.

5. Bench-scale reactor discharge from leaching minus 10-mesh calcined clay contained less fines than the miniplant leaching reactor discharge (miniplant test 3), required less flocculant, and gave greater thickener percent solids.

6. Minus 18-mesh misted calcined kaolin leaching feed resulted in significantly lower flocculant requirements than for minus 10-, 20-, or 35-mesh calcined kaolin.

7. Minus 20-mesh and minus 35-mesh calcined kaolin leaching feed required increased thickener area and flocculant additions.

The miniplant tests demonstrated that thickener underflow percent solids increased significantly with each successive thickening stage. Predicted solids for test 3 varied from 14 pct for thickener 1 underflow to 28 pct for thickener 5.

At the predicted underflow percent solids, thickener area requirements were similar for each thickener and averaged 25 $ft^2$/tpd solids or 3.7 $ft^2$/tpd calcined kaolin. Total flocculant addition was 2.4 lb/ton of solids (0.35 lb/ton calcined kaolin), of which 1.3 lb/ton was added to the first thickener.

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