RI 8831

Bureau of Mines Report of Investigations/1984

Alumina Miniplant Operations— Separation of Aluminum Chloride Liquor From Leach Residue by Horizontal Belt Filtration

By Roy T. Sorensen and Dwight L. Sawyer, Jr.

UNITED STATES DEPARTMENT OF THE INTERIOR

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UNITED STATES DEPARTMENT OF THE INTERIOR
William P. Clark, Secretary

BUREAU OF MINES
Robert C. Horton, Director

Library of Congress Cataloging in Publication Data:

Sorensen, Roy T

Alumina miniplant operations.

(Report of investigations / United States Department of the Interior, Bureau of Mines ; 8831)

Bibliography: p. 18.

Supt. of Docs. no.: I 28.23:8831.

1. Aluminum chloride. 2. Kaolin. 3. Leaching. 4. Separation (Technology). I. Sawyer, Dwight L. II. Title. III. Series: Report of investigations (United States. Bureau of Mines) ; 8831.

TN23.U43 [TP245.A4] 622s [699'.722] 83-600275

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

atm	atmosphere, standard
cfm/ft^2	cubic foot per minute per square foot
count/in	count per inch
ft	foot
ft^2	square foot
ft^3	cubic foot
ft/min	foot per minute
ft^2/tpd	square foot per ton per day
g	gram
gal	gallon
g/ft^3	gram per cubic foot
gal/ft^2	gallon per square foot
g/min	gram per minute
g/mL	gram per milliliter
g/L	gram per liter
gal/min	gallon per minute
hp	horsepower
h	hour
h/day	hour per day
in	inch
in^2	square inch
in Hg	inch of mercury
L	liter
lb	pound
lb/ft^2	pound per square foot
lb/ft^3	pound per cubic foot
lb/h	pound per hour
lb/h·ft^{-2}	pound per hour per square foot
lb/gal	pound per gallon
lb solids/hr·ft^{-2}	pound solids per hour per square foot
lb solids/ft^3	pound solids per cubic foot
lb/ton	pound per short ton
L/min	liter per minute
min	minute
min/ft^2	minute per square foot
min/ft^3	minute per cubic foot
min·ft^4/lb·gal	minute foot to the fourth power per pound gal
m/h	minute per hour
mL	milliliter
oz/yd^2	ounce per square yard
pct	percent by weight
pH	hydrogen-ion concentration (logarithm of reciprocal)
ppm	part per million
rpm	revolution per minute
s	second
std ft^3	standard cubic foot
std ft^3/min	standard cubic foot per minute
tpd	short ton per day

ALUMINA MINIPLANT OPERATIONS—SEPARATION OF ALUMINUM CHLORIDE LIQUOR FROM LEACH RESIDUE BY HORIZONTAL BELT FILTRATION

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

ABSTRACT

The Bureau of Mines has investigated the recovery of alumina by hydrochloric acid leaching of calcined kaolinitic clay. Bench-scale studies indicated that horizontal belt filtration was a practical method for separating the siliceous residue from aluminum chloride liquor generated in the leaching step. Miniplant-scale continuous testing was performed to evaluate the filter circuits and to compare filtration response of different calcined kaolin feed materials and of different agitator-baffle designs in the leaching reactors.

Soluble alumina recovery with a single filter ranged from 94 pct with minus 10-mesh feed to more than 96 pct with minus 35-mesh or misted feed. Process advantages were confirmed for the calcined misted kaolin feed material prepared by a Bureau-developed misting process. With two filters in series, an overall filtration rate of 100 lb solids/hr$\cdot$ft^{-2} of filter area was attained with a soluble alumina recovery of more than 99 pct. More than 99.5 pct alumina recovery was obtained with a thickener and a two-filter circuit for either the minus 35-mesh or the misted feed.

Modifications in leaching reactor design made to decrease particle-to-particle attrition were successful and significantly increased filtration rates and decreased flocculant requirements.

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INTRODUCTION

From 1975 through 1980, the Bureau of Mines performed miniplant-scale studies on the production of cell-grade alumina by a clay-hydrochloric acid (HCl) process. Process unit operations are shown in figure 1. As part of this research effort, a series of tests using horizontal belt filtration for solids-liquid separation was made on a variety of acid-leached calcined kaolin feed materials (6).[3] The miniplant test series was preceded by three bench-scale filtration studies:

Study I was performed on miniplant acid leach slurry, and measurements predicted low filtration rates and low washing efficiencies.

Study II was a cooperative effort performed in the research facility of a major filter manufacturer and indicated that higher filtration rates could be obtained on bench-scale-produced slurries, but washing efficiencies were low. Prethickening also resulted in higher filtration rates. Calcined kaolin materials of different sizes were evaluated and included one produced by a "misting" process developed at the Bureau's Albany (OR) Research Center (2, 4-5). In this process, prior to calcination, raw kaolin was crushed to nominal 18 mesh and tumbled on a pelletizing disk fitted with sprays that produced a fine mist of water.

Study III was undertaken to determine if the poor washing efficiency was due to a slow release of aluminum chloride from the porous structure of the leach residue.

Based on results of bench-scale study II, a 10.5-ft by 1-ft horizontal belt filter was designed by a major filter manufacturer for continuous operation on slurry from leaching calcined kaolin with HCl (fig. 2). This filter and a second, smaller unit used for a second stage in some of the tests were commercial units. Although conventional drum filters are usually lower in capital and operating costs, they were not used because of inferior capabilities in cake washing, filter cloth washing, and handling coarse (plus 20-mesh) particles.

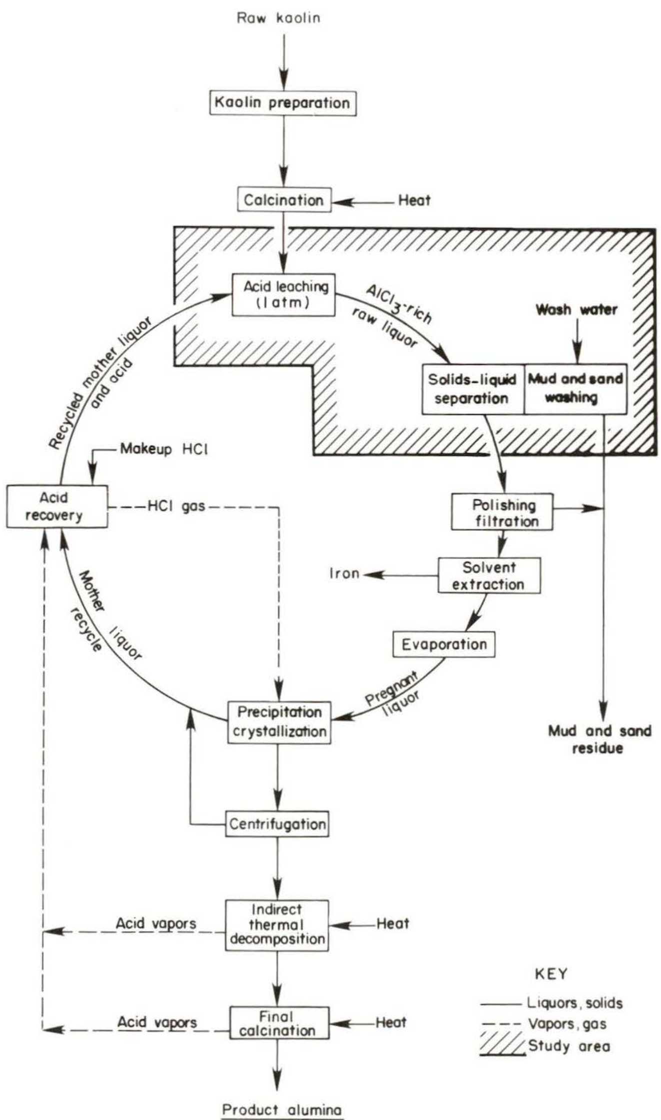

FIGURE 1. - Unit operations of the clay-HCl miniplant process.

[3] Underlined numbers in parentheses refer to items in the list of references preceding the appendixes.

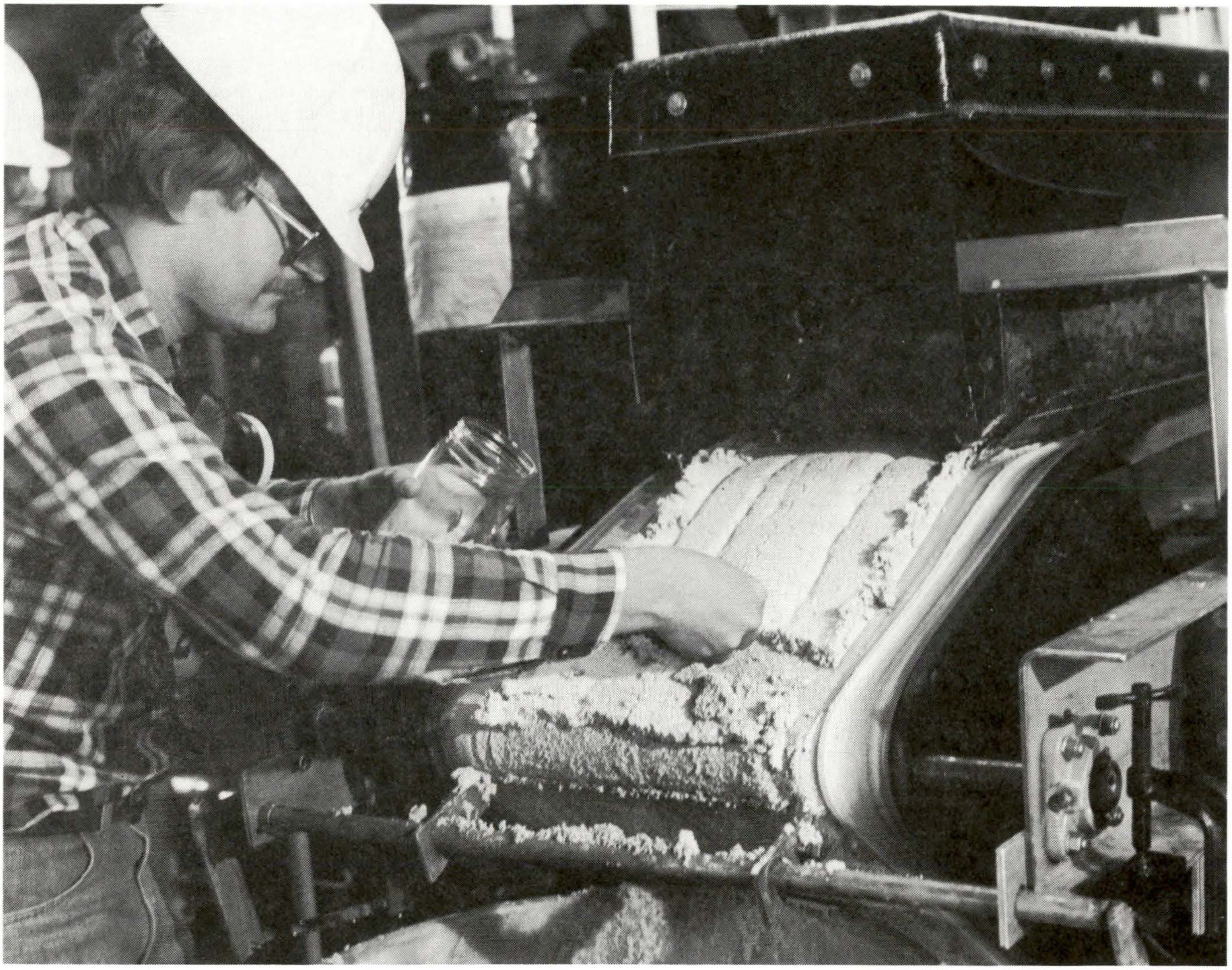

FIGURE 2. - First belt filter in operation.

Miniplant filtration studies were undertaken to--

1. Operate and test the filter circuits using one and two horizontal belt filters with and without feed slurry prethickening.

2. Evaluate the filtration characteristics of slurries produced by HCl leaching different types of calcined kaolin materials.

3. Compare the operation of unbaffled and baffled chemical leaching reactors in the miniplant for producing easily filtered acid leach slurries.

ACKNOWLEDGMENTS

The authors wish to acknowledge the major contributions by E. Scott Robbins, former District Representative, and N. Kelly Brown, former Supervisor of Testing, Envirotech Corp., Salt Lake City, UT, for directing contract testing in bench-scale studies I and II.

BENCH-SCALE EXPERIMENTS AND RESULTS

PRELIMINARY TESTING

Three preliminary filtration studies were conducted to develop characteristic filtration data for designing the mini-plant filtration circuit and test program. Materials, equipment, and procedures used in these tests are presented in appendix A. Test results discussed in the text are supplemented by detailed data in appendix B.

A series of filtration tests (filtration study I) was performed on slurries produced from a clay-HCl miniplant run (6) in which minus 10-mesh calcined kaolin was leached, and a four-classifier, five-thickener circuit was used for solids-liquid separation. Test variables are given in table 1. Form filtration rate for a 3/8-in cake was about 50 lb solids/h$\cdot$ft^{-2} filter area. The estimated full-scale filtration rate (FSFR)[4] obtainable on a commercial horizontal belt filter was 16 lb solids/h$\cdot$ft^{-2} of total filter area. This value was obtained by assuming allowances for cake forming, the application of two 2.0 displacement washes, cake drying to 46-pct free moisture and applying an 0.8 operating factor. Because of low washing efficiency, only about 94-pct recovery of the soluble alumina could be expected by this treatment. A 99-pct recovery could be obtained by adding repulping, a second filter, and more washes, but the FSFR would be halved. Estimated single-stage washing efficiency ranged from 34 pct for 1.0-displacement washing to 47 pct for 2.3-displacement washing. A polypropylene twill filter cloth (Eimco POPR-896)[5] was the most suitable cloth tested.[6] Separan MGL synthetic polymer, a high-molecular-weight, water-soluble, nonionic polyacryamide, was added in a dilute solution as flocculant in all of the filtration studies. It had functioned adequately in an earlier study (6) using thickening in place of filtering; however, other synthetic polymers may perform as well or better.

[4]Full-scale filtration rate is defined as the rate in pounds of solids (dry basis) per square foot of filter area that can be handled per hour in a completely defined filter operation including forming, washing, and drying stages, and allows for baffling between stages.

[5]Reference to specific products does not imply endorsement by the Bureau of Mines.

[6]Much of the filter equipment was supplied by contractor that performed the bench-scale studies. Similar equipment is available from other manufacturers. (See table A-2 for specifications.)

TABLE 1. - Test variables for studies I and II

Operating parameter	Study I	Study II
Filter cloth, Eimco designation[1]..	NY-527F,529F POPR-873, 896, 929	POPR-896,929
Vacuum.....................in Hg..	10, 20	13, 20
Flocculant addition, lb Separan MGL/ton solids..................	0-0.25	0.1-0.7
Cake thickness...............in..	3/32-3/8	3/8-1
Drying time...................s..	15-90	5-60
Wash volume.................[2]N..	1-3	1-4

[1]See table A-2 for description of filter cloths.

[2] $N = \dfrac{\text{volume of washing liquor}}{\text{volume of residual filter cake liquor}}$.

To evaluate the effect of leaching feed particle size, a second test series (filtration study II) was performed in which the leach slurry was produced in a bench-scale reactor. Test variables are listed in table 1. Seven types of calcined kaolin feed were tested:

1. Minus 10 mesh.
2. Minus 18-mesh misted.
3. Minus 20 mesh.
4. Minus 35 mesh.
5. Minus 10 plus 100 mesh.
6. Minus 20 plus 100 mesh.
7. Minus 35 plus 100 mesh.

Estimates of filtration characteristics were developed from these tests and are presented in appendix B.

Filtration response varied not only from that obtained in filtration study I but also among the seven types of filter feed. Without a prethickening step, form filtration rates averaged about 500 lb solids/$h \cdot ft^{-2}$ of form area and FSFR was between 50 and 200 lb solids/$h \cdot ft^{-2}$.

Washing efficiency for the minus 10-mesh leaching feed was about the same as in study I on miniplant slurry produced from the same feed, 30 pct with a 1-displacement wash and 48 pct with a 2.3-displacement wash. Washing efficiencies for the minus 18-mesh misted, minus 20-mesh, and minus 35-mesh feeds were each about 20 pct higher than for the minus 10-mesh material. A single horizontal belt filter[7] with two countercurrent washes (N = 2.3) was predicted to yield a

[7]To avoid repetition, the horizontal belt filter will be referred to as the "filter" or "belt filter" in the remainder of this report.

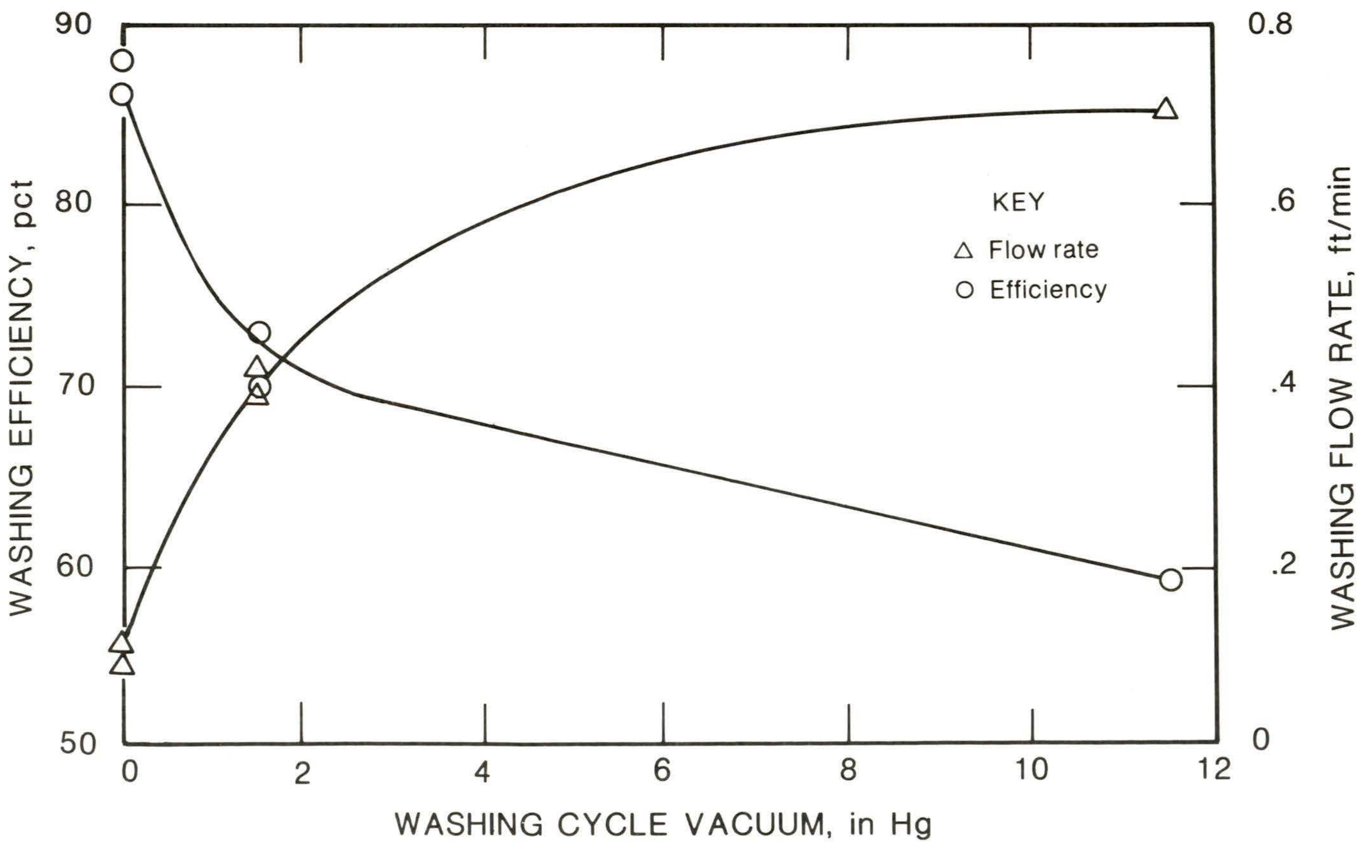

FIGURE 3. - Effect of filtration washing vacuum on washing flow rate through cake and on washing efficiency.

96 pct Al_2O_3[8] recovery of the alumina in solution with minus 10-mesh feed and 98 pct with the finer feeds. Thickening would increase these recoveries slightly more than 1 pct.

Filtration study II also included two special tests that simulated filtration with four countercurrent washes. The first test was made on slurry from leaching 20- by 100-mesh calcined kaolin. Vacuum was maintained at about 10 in Hg, and each countercurrent stage received 1.6-displacement washes. In the second test, in which minus 20-mesh feed was used, washes were of 2.6 displacements. The two multistage washing tests indicated that the washing efficiencies obtained in the single washing tests were 10 pct higher than those attained for each wash in a multistage circuit. This may be attributable to washing liquor channeling through the cake in the later washing stages. A soluble alumina recovery of 96 pct was obtained in the concurrent washing test with four 1.6-displacement washes and 99 pct with four 2.6-displacement washes. The FSFR was 120 lb solids/$h \cdot ft^{-2}$ of total filter area for filtering slurry from leaching 20- by 100-mesh calcined kaolin and 55 lb solids/$h \cdot ft^{-2}$ with minus 20-mesh calcined kaolin.

Filtration study III was undertaken to determine the cause of poor efficiency in washing. Five filter tests using 5-displacement washes were made on slurry from a bench-scale leach of the 20- by 100-mesh calcined kaolin. Wash liquor flow rate through the cake was controlled by varying the vacuum in the washing cycle. Figure 3 shows that the flow rate was 0.1 ft/min at no applied vacuum and 0.7 ft/min at 11.5 in Hg vacuum. Washing efficiency ranged from 88 pct at no vacuum to 59 pct at 11.5 in Hg vacuum.

Four special washing tests were performed to determine the effect of particle size on release of aluminum chloride. Two sizes of leach residue were immersed in water and the rate of aluminum chloride release was monitored. Release from 1/4-in pellets was slower than from 20- by 100-mesh cake (fig. B-6).

CONCURRENT TESTING

Filtration study IV was conducted concurrently with the miniplant filtration tests. Bench-scale tests conducted on the clay-HCl miniplant leach slurry were made to verify miniplant filtration rates. This testing is discussed with the results of the miniplant filtration testing, and estimates of filtration characteristics are presented in appendix B.

MINIPLANT RESULTS AND DISCUSSION

The bench-scale studies indicated the need for miniplant testing to develop data concerning--

1. Alumina recovery and equipment requirements for various filtration circuits.

2. The effect of feed characteristics on filtration response.

3. The effect on filtration response of using baffled reactors for acid leaching compared with using essentially unbaffled reactors.

Four continuous solids-liquid separation circuits were evaluated in the miniplant:

1. Single (one) filter.

2. Thickener and one filter.

[8]The objective of the clay-HCl process is to produce a cell-grade alumina (Al_2O_3) product from kaolin. To maintain continuity of usage throughout the entire series of Bureau reports devoted to the alumina miniplant operations, extracted or soluble aluminum is considered as alumina (Al_2O_3), or soluble alumina (soluble Al_2O_3), respectively.

3. Two filters with intermediate repulping step.

4. Thickener and two filters with intermediate repulping step.

Miniplant equipment is described in appendix C, sampling procedures in appendix D, and calculation methods in appendix E.

Filtration data of individual tests were developed as material balances (table E-1) and as plots of cake moisture versus length of filter (fig. E-1). The plots were necessary for developing filter requirement and performance characteristics because the filters were oversize to miniplant capacity as shown in figure 4. Although only 12 lb solids/$h \cdot ft^{-2}$ of total filter area were processed in the first filter at the miniplant leaching rate of 200 lb/h calcined kaolin, higher rates were calculated from the cake moisture plots (fig. E-1) using the calculation methods described in appendix E. Maximum FSFR were developed from the curves assuming forming to 61 pct moisture and drying to 50 pct. Miniplant results discussed in the text are supplemented by detailed data in appendix F, which include the following:

1. Factors for calculating washing area for any cake-loading and for any wash-addition rate.

2. Curves relating drying area to cake loading and final cake moisture.

3. Curves relating final cake moisture to FSFR for specific filtration circuits.

Leaching slurry conditions common to all tests are given in table 2; operating conditions are given in table 3. Four types of calcined kaolin were used in filter circuit testing:

1. Minus 10 mesh (baseline).

2. Minus 18-mesh misted.

3. Minus 20 mesh.

4. Minus 35-mesh spray-dried.

The preparation of miniplant feed materials is described in appendix C.

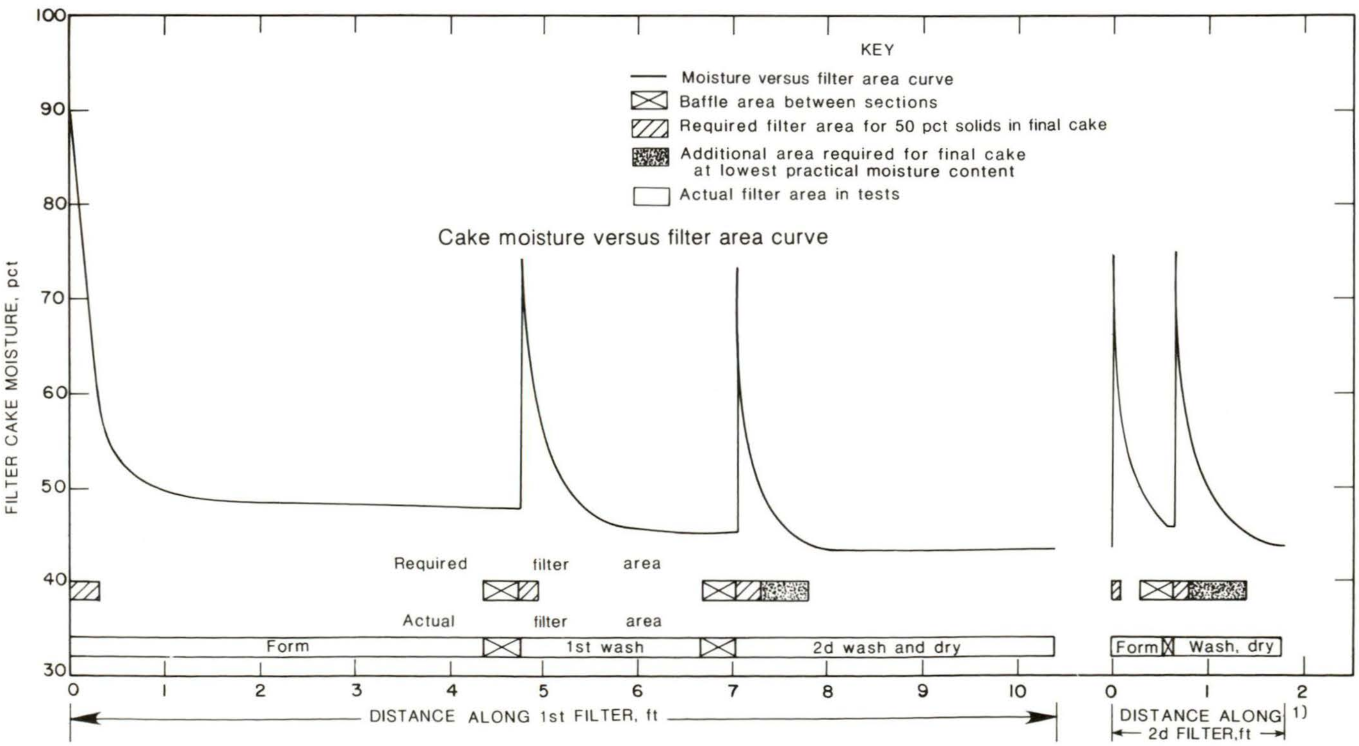

FIGURE 4. - Comparison between actual belt area used and minimum belt area required for miniplant filtration test 11.

TABLE 2. - Leaching slurry flow data for miniplant tests

Slurry:	
Rate....................lb/h..	890
Temperature..............°C..	60
Liquor:	
Rate....................lb/h..	770
Al_2O_3 concentration.....pct..	10
Solids:	
Rate....................lb/h..	120
Concentration...........pct..	13.5

TABLE 3. - Standard operating conditions for filters

Filtration	1st filter	2d filter
Feed[1] temperature.....°C..	50	25-35
Vacuum, in Hg:		
Form....................	4-12	2-5
1st washing.............	2-10	1-3
2d washing..............	1-8	NAp
Form filtrate.........°C..	40-50	25-35
Belt speed........ft/min..	1.5-1.6	2.2
Cake thickness........in..	0.41	0.41
Cake loading......lb/ft^2..	1.31	1.31

NAp Not applicable.

[1]Leach slurry and 1st washing filtrate for 1st filter, repulper discharge for 2d filter.

ONE-FILTER CIRCUIT

The one-filter circuit was operated with two washes. The flowsheet is shown in figure 5. Numbers encircled in flow streams are for correlation with flowsheets of other filter circuits, sample locations shown in figure C-1, and stream numbers in table E-1. Tests (summarized in tables F-1 and F-2) were conducted on all four types of calcined kaolin. Soluble alumina recovery ranged from 95 to 96 pct, and average stage washing efficiency ranged from 28 to 46 pct. These responses were related to the type of feed material and the wash dilution, which was varied from 1.6 to 3.1 displacements. Modifying the wash addition to short circuit part of the wash back on itself (fig. 5) increased alumina recovery about 1 pct and washing efficiency by at least 5 pct. A specially prepared minus 35-mesh spray-dried material was also tested with the circuit. The solids content of the pregnant liquor (form filtrate) ranged from 200 to 450 ppm, and cake moisture ranged from 42 to 57 pct, depending on the feed material.

TWO-FILTER CIRCUIT

In the two-filter circuit, the second filter was preceded by a repulper and had only one countercurrent wash (fig. 6). Tests (summarized in tables F-4 and F-5) were run on all but the minus 35-mesh spray-dried calcined kaolin. Soluble alumina recovery ranged from 98.8 to 99.6 pct, washing efficiencies from 33 to 49 pct, and solids content of the pregnant liquor from 190 to 420 ppm; cake moistures were about 45 pct.

THICKENER-AND-FILTER CIRCUIT

In the thickener-and-filter circuit, one filter (fig. 7) and two filters (fig. 8) were tested using the minus 20-mesh calcined kaolin (data are summarized in tables F-6 and F-7). At a wash addition of 2.5 displacements, soluble alumina recovery was 98 pct for one filter and 99.7 pct for two filters.

WASHING EFFICIENCY

Because of more channeling in the miniplant filters, washing efficiencies were lower than in bench-scale testing (table F-8). The maximum practical washing efficiency is independent of the washing addition volume between N = 1.5 and 3. Washing efficiency increased as the particle size of the material decreased, and was higher in the second filter than in the first (table 4) because the lower vacuum level used in the second filter caused a slower draw of the wash through the cake. Washing efficiency was higher when the area of wash addition was flooded using a special wash-recycling procedure (fig. 5).

TABLE 4. - Comparison of washing efficiencies in first and second miniplant filters, percent

Calcined kaolin feed size, mesh	1st filter		2d filter,
	Unflooded wash	Flooded wash	Unflooded wash
Minus 10 (baseline)....	32	33	57
Minus 18 misted........	39	47	65
Minus 20...............	44	NR	50
Minus 20 thickened.....	50	NR	46
Minus 35 spray-dried...	NR	56	NR

NR Not run.

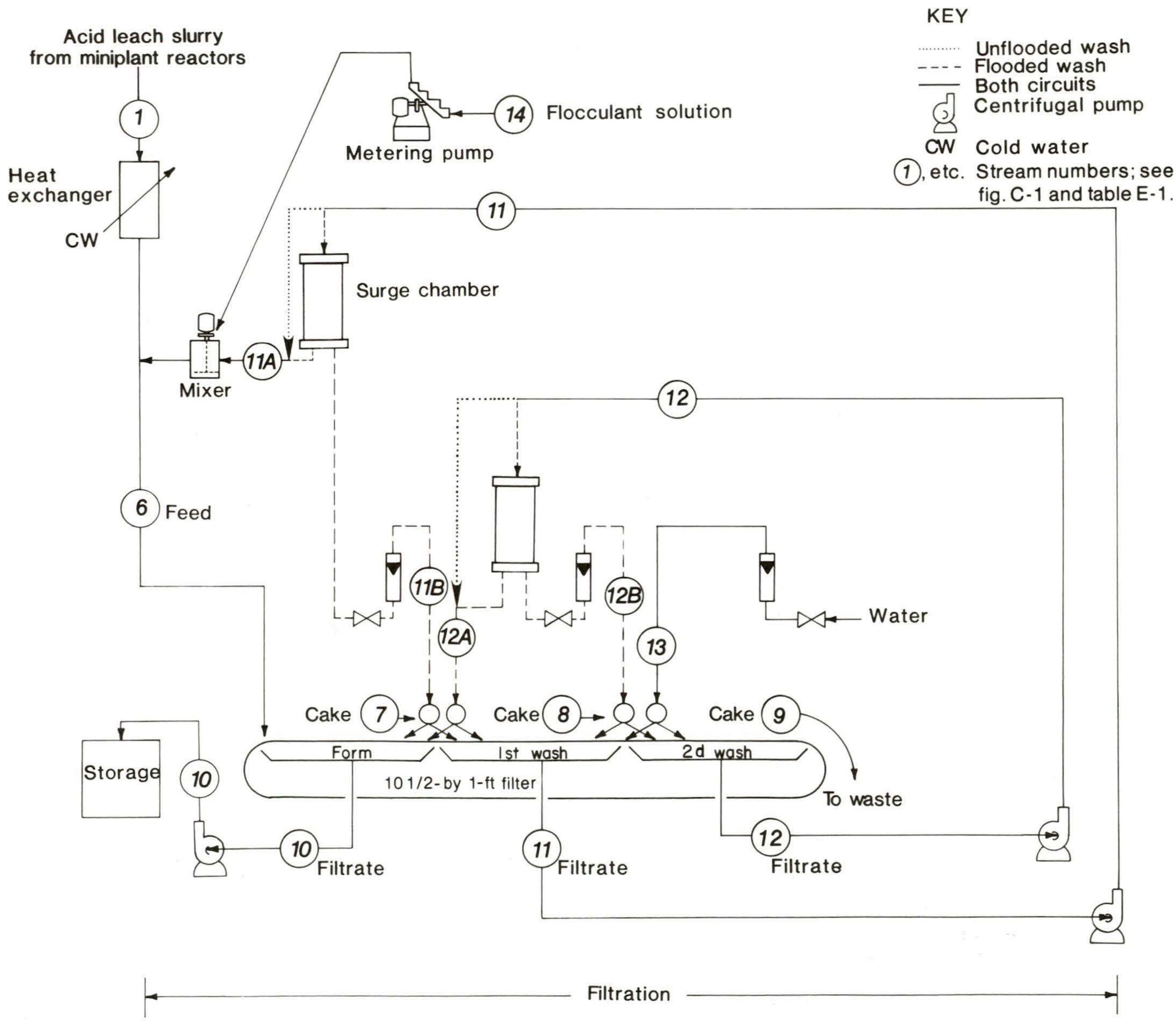

FIGURE 5. - One-filter circuit.

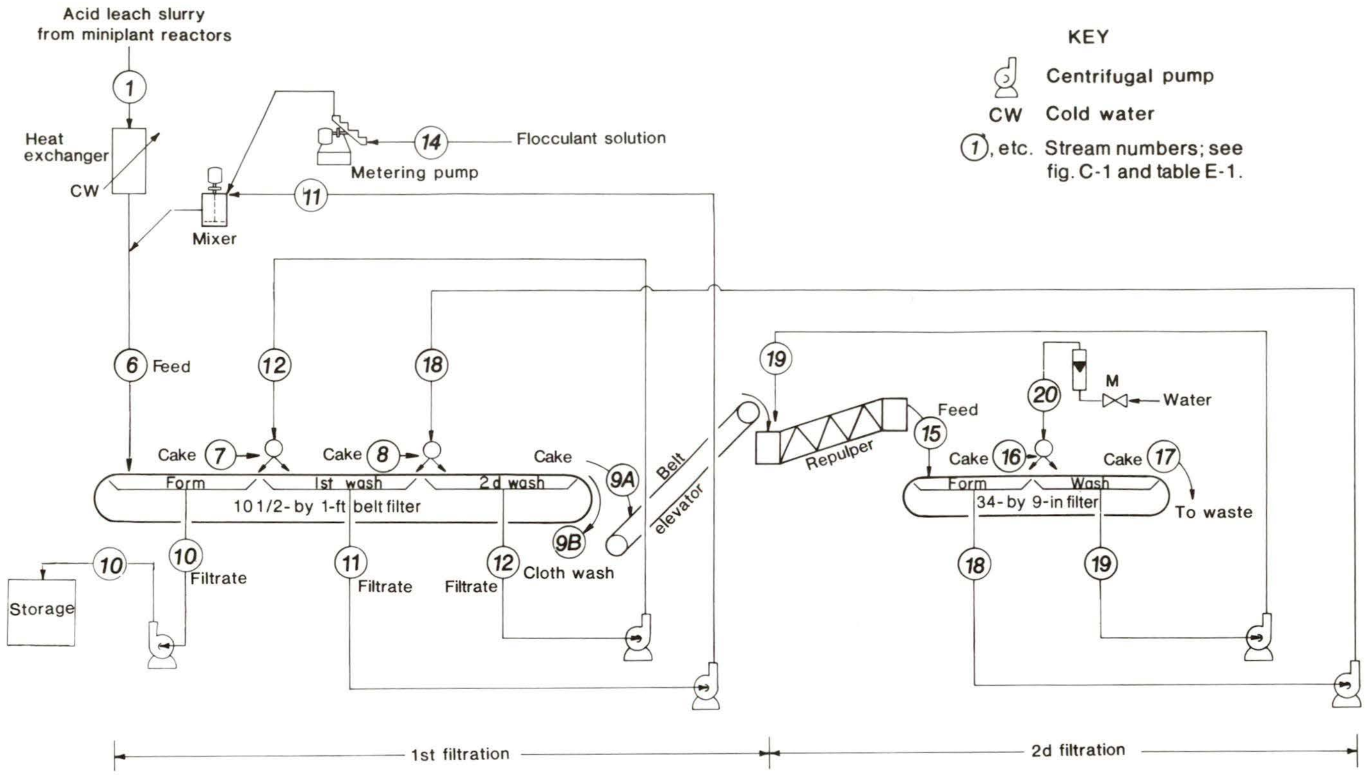

FIGURE 6. - Two-filter circuit.

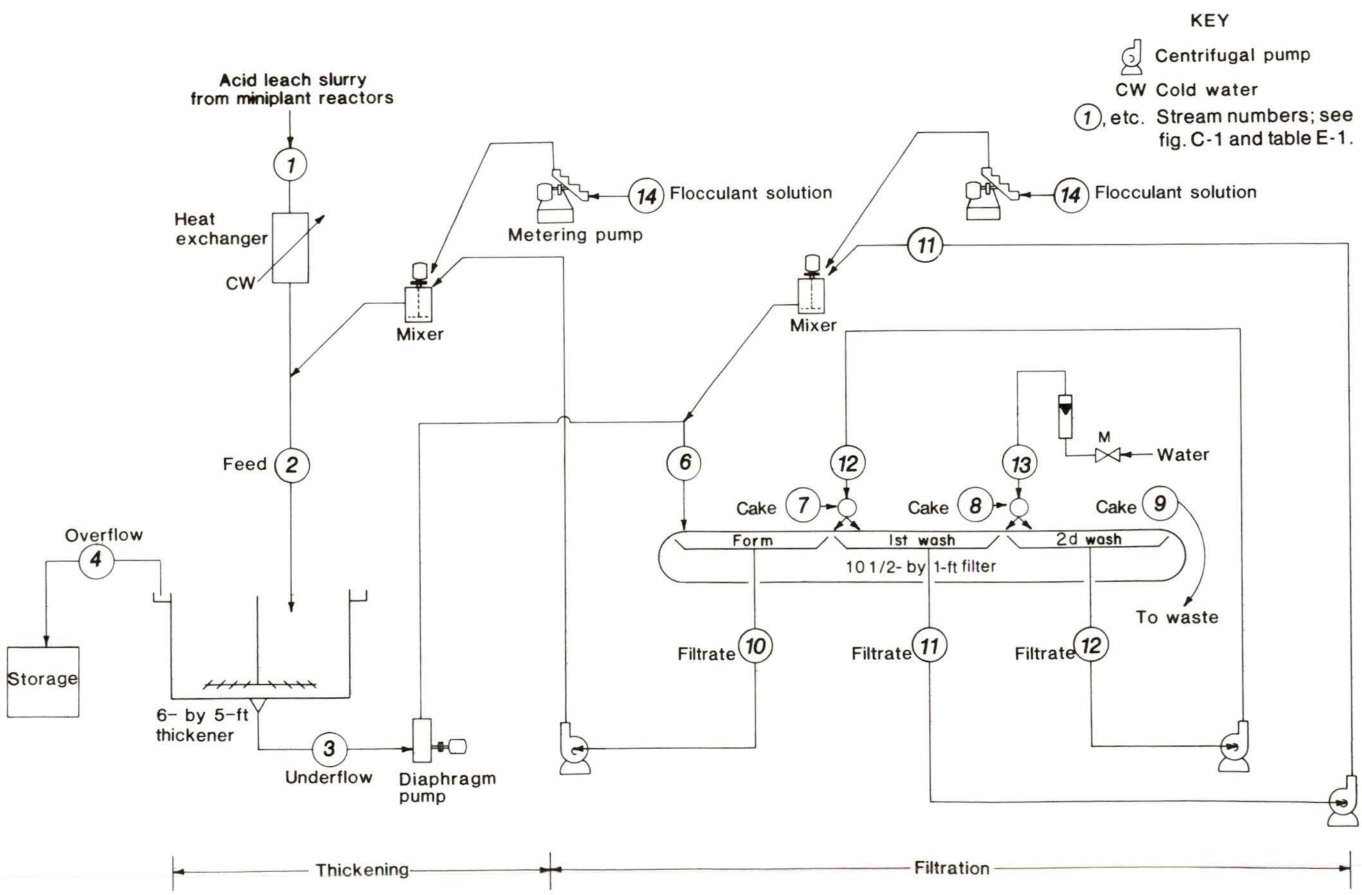

FIGURE 7. - Thickener-and-one-filter circuit.

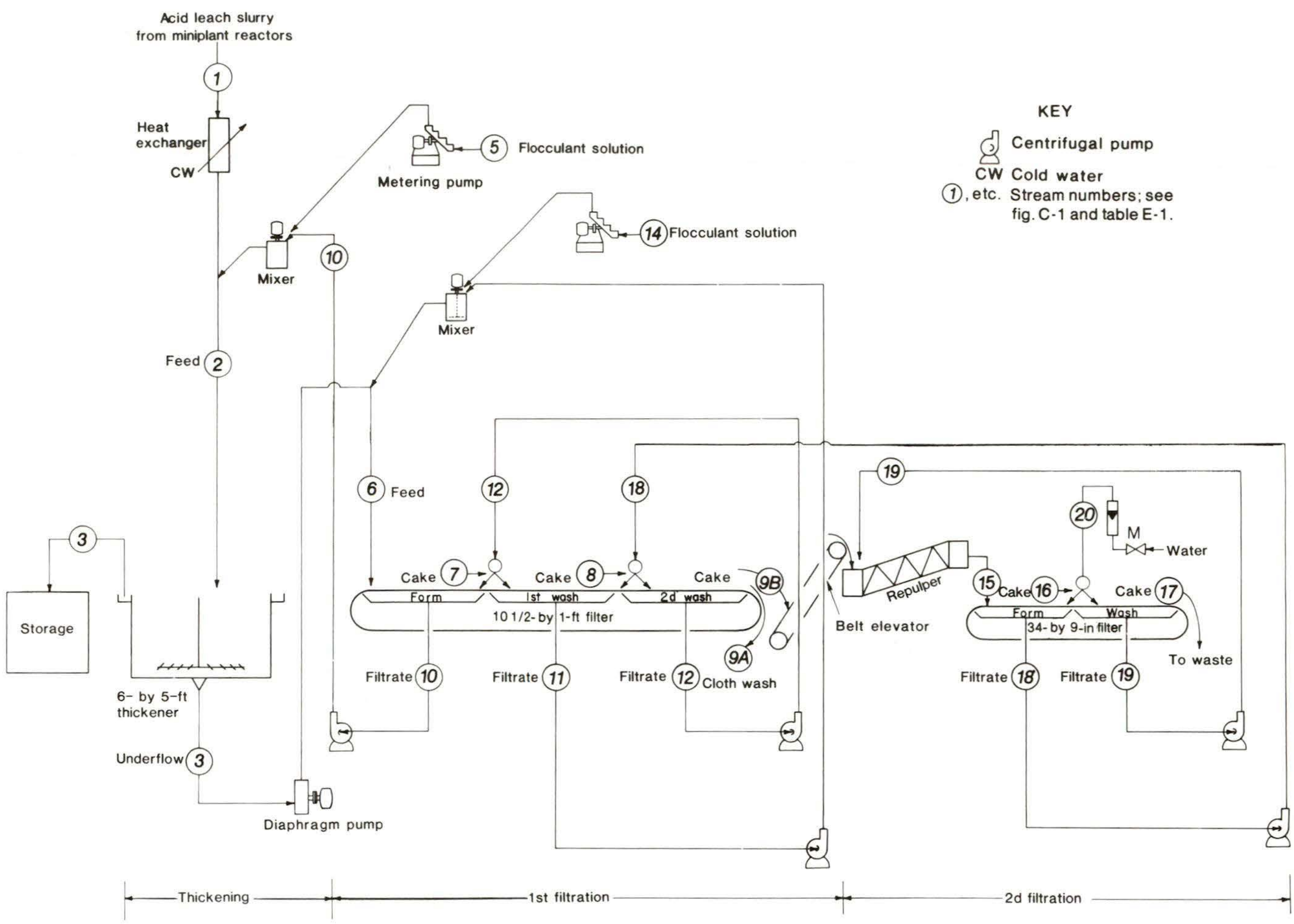

FIGURE 8. - Thickener-and-two-filter circuit.

PREGNANT LIQUOR CLARITY

About 200 ppm of finely divided solids were suspended in the pregnant liquor from filtration in the testing on minus 10- or 18-mesh calcined misted kaolin but about 450 ppm for minus 20- or 35-mesh spray-dried material. With prethickening, the pregnant liquor contained 140 ppm.

MOISTURE REMOVAL FROM FILTER CAKE

One-half minute (a Θ_d/w of 0.35) is the best drying time and provides cake moistures from a single filter (fig. F-1) as follows:

Calcined kaolin feed size, mesh	Moisture, pct
Minus 10..................	49
Minus 18 misted...........	43
Minus 20..................	48
Minus 35 spray-dried.......	46

In a two-filter circuit, cake moistures at one-half minute are about 45 pct for each material tested (fig. F-3). Filter cake loading averaged about 1.31 lb solids/ft^2.

FILTRATION RATE

Form filtration rate depends on the amount of minus 150-mesh solids in the filter feed (tables F-1 and F-2), and the flocculant addition. Plots of form filtration rate versus flocculant addition for minus 10-mesh, minus 18-mesh misted, and minus 35-mesh spray-dried calcined kaolin are given in figures 9, 10, and 11, respectively. High forming rates are attained at low flocculant addition when leaching was done in baffled reactors because fewer fines were created by particle-to-particle attrition. Washing rates, in general, were proportional to forming rates, but the FSFR is also dependent on the number of filters, number of washing stages, and the final cake moisture (figs. F-1 and F-3). The FSFR, as previously defined, can be estimated for single- and two-filter circuits from figures F-2 and F-4, respectively.

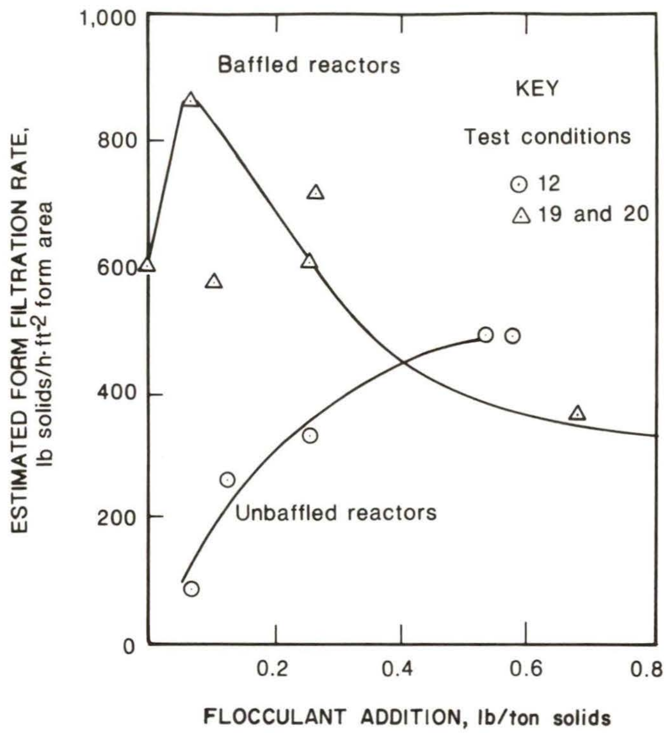

FIGURE 10. - Form filtration rate versus flocculant addition for minus 18-mesh calcined misted kaolin.

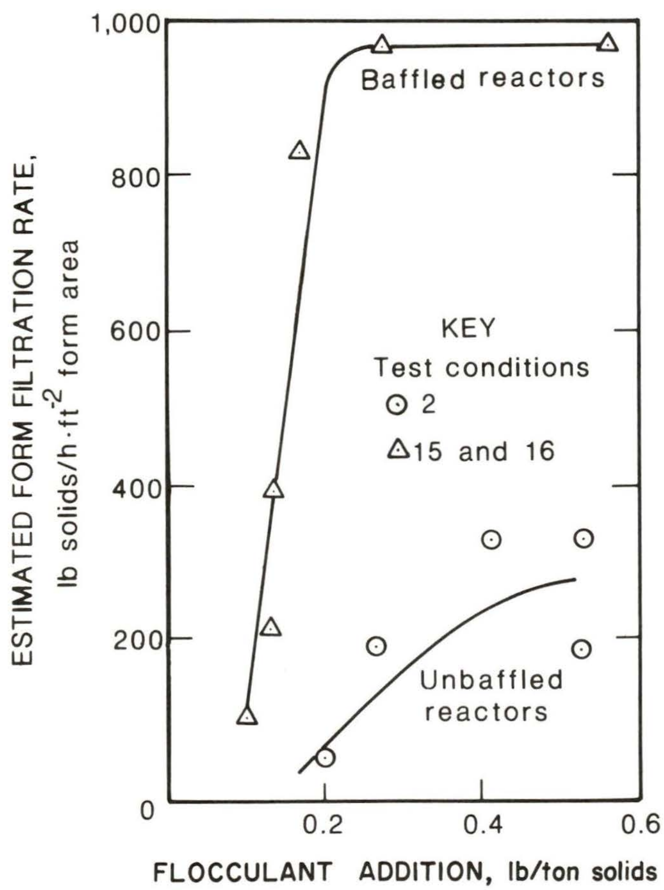

FIGURE 9. - Form filtration rate versus flocculant addition for minus 10-mesh calcined kaolin.

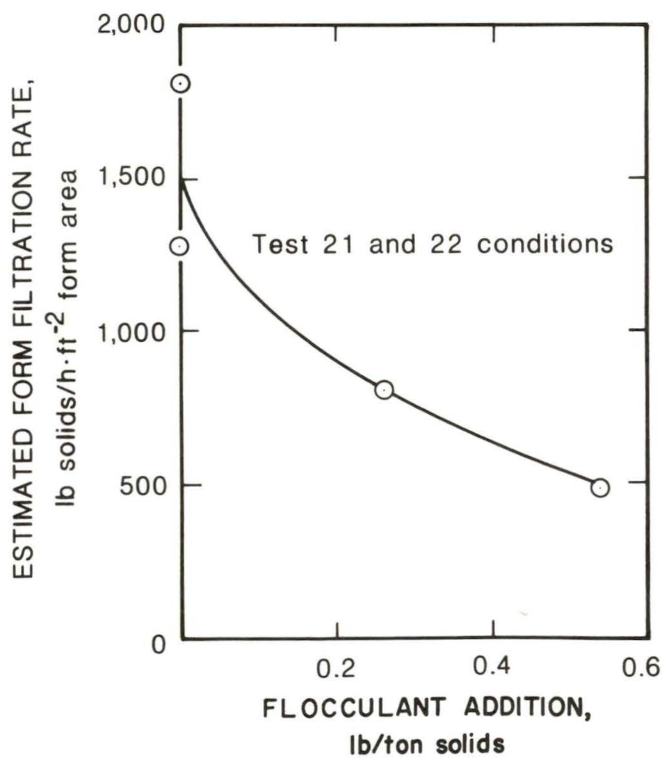

FIGURE 11. - Form filtration rate versus flocculant addition for minus 35-mesh calcined spray-dried kaolin.

ESTIMATES OF BEST FILTRATION CONDITIONS AND PERFORMANCE

The most efficient filtration separation was obtained with the minus 18-mesh calcined misted kaolin. Estimates of final product analyses, alumina distribution, and process requirements are presented in table 5 (response for other materials in table F-9). These data are estimated for the lowest practical cake moisture, i.e., at a drying time of 0.5 min (figs. F-1 and F-3), washing-liquor addition of 1.5 lb per pound of residue (flooded wash), cake thickness of 0.41 in, and cake loading of 1.31 lb/ft^2. Alumina recovery was estimated at 99.2 pct for two filters with unthickened feed and 99.6 pct with prethickening. The FSFR was 51 $lb/h{\cdot}ft^{-2}$. Filtration rate could be increased about 50 pct by drying to 50 pct moisture, but recovery of soluble alumina would be decreased by about 0.5 pct.

Recovery of soluble alumina in the final liquor is an important factor. Figure 12 illustrates that highest alumina recovery is attained with the minus 18-mesh calcined misted kaolin. More washing stages could have been used in the one-belt filter. Figure 13 illustrates the effect of adding or subtracting washing stages in the first filter. More than 99.2 pct Al_2O_3 recovery could be achieved with slurry from leaching minus 18-mesh kaolin in the following hypothetical circuits:

1. Thickener, first filter with one wash and repulper; second filter with one wash.

2. First filter with two washes and repulper; second filter with one wash (miniplant circuit).

3. Thickener and one filter with four washes.

4. One filter with six washes.

These predictions assume that each washing stage in the first filter has the same washing efficiency. The second bench-scale study (filtration study II) indicated that wash liquor may begin to channel through the cake in later washing stages. Therefore, 99.2 pct Al_2O_3 recovery may not be possible with one filter and six countercurrent washes.

TABLE 5. - Predicted best results for filtration of minus 18-mesh misted calcined kaolin[1]

Number of filters	Flocculant addition, lb Separan MGL/ton of solids	Liquor product[2]			Final filter cake			FSFR,[3] $lb/h{\cdot}ft^{-2}$
		Solids, ppm	Al_2O_3, pct	Al_2O_3 recovery, pct	Moisture,[3] pct	Al_2O_3, pct	Al_2O_3 loss, pct	
Unthickened:[4]								
1..........	0.07	190	8.6	96.8	43	2.7	3.2	64
2..........	.07	200	8.9	99.2	44	.6	.8	51
Thickened:[5]								
1..........	.20	150	8.8	98.3	43	1.4	1.7	64
2..........	.20	130	8.9	99.6	44	.3	.4	51

[1]Assuming a slurry from acid leaching of calcined kaolin which contains 10.0 pct soluble Al_2O_3 and 13.8 pct solids, a total wash addition of 1.5 lb fresh water per pound residue solids, and 0.375 ft^2 baffle area between washing stages.

[2]Depending on mode of operation, the liquor product from the filtration operation is form filtrate when the acid leach slurry is not prethickened, and is thickener overflow liquor when prethickening is employed.

[3]Lowest practical drying time, $\Theta_d/w = 0.5$ (figs. F-1 and F-3).

[4]Summarized from material balances made from actual tests.

[5]Estimated in part from testing on minus 20-mesh calcined kaolin in which filter feed was prethickened.

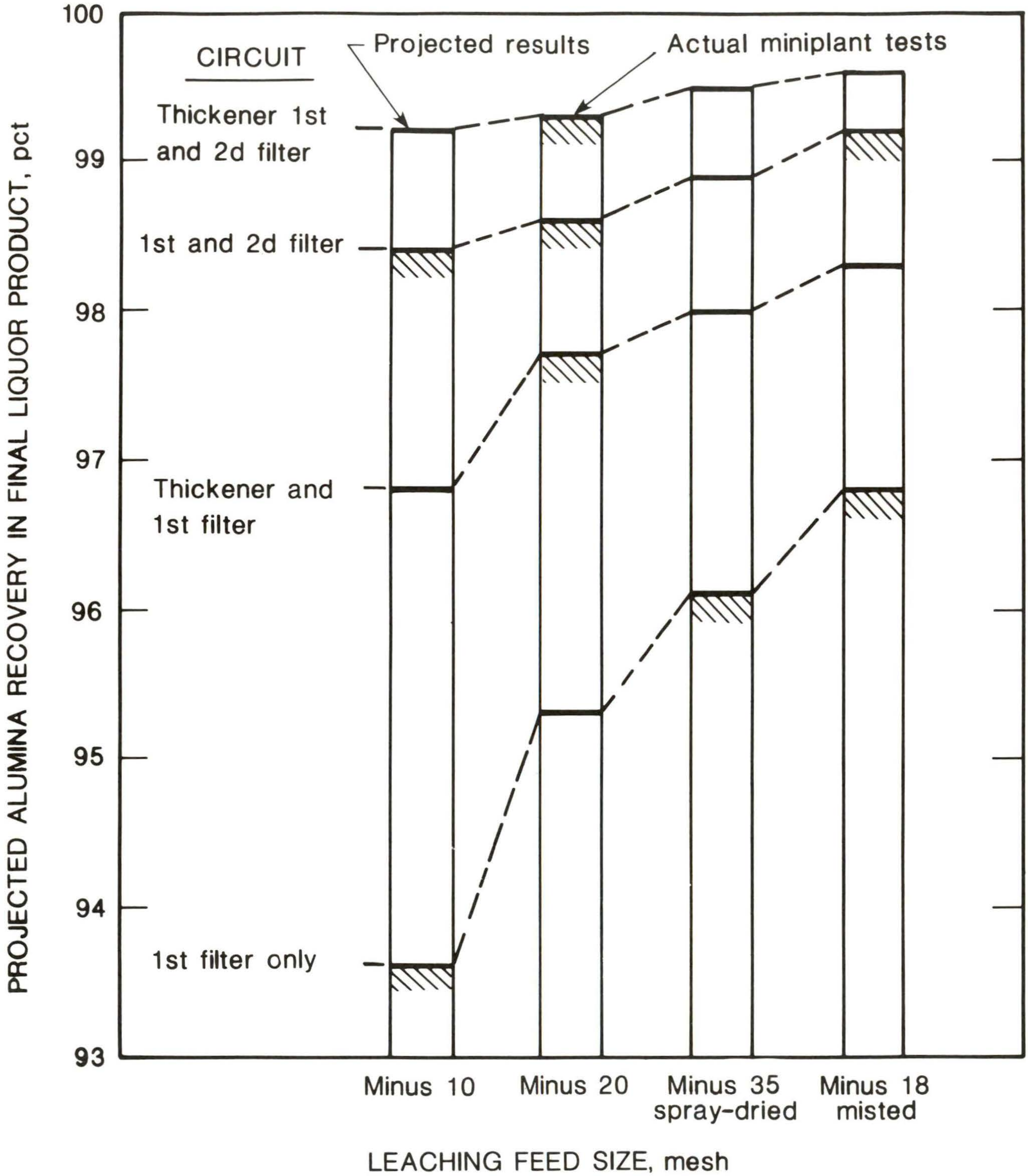

FIGURE 12. - Effect of feed size on recovery of soluble alumina for various filter circuits.

A four-classifier, five-thickener solids-liquid separation circuit previously tested in the miniplant (6) provided a 97.3-pct recovery of soluble alumina. The final waste product contained 57 pct moisture, 1.3 pct of which was soluble Al_2O_3. Equipment requirements were 12 ft^2/tpd (7 lb/h·ft^{-2}) for the total classifier pool area and 31 ft^2/tpd (2.7 lb/h·ft^{-2}) for the total thickener area. Seven thickeners in series would provide 96.7 to 99.0 pct recovery of

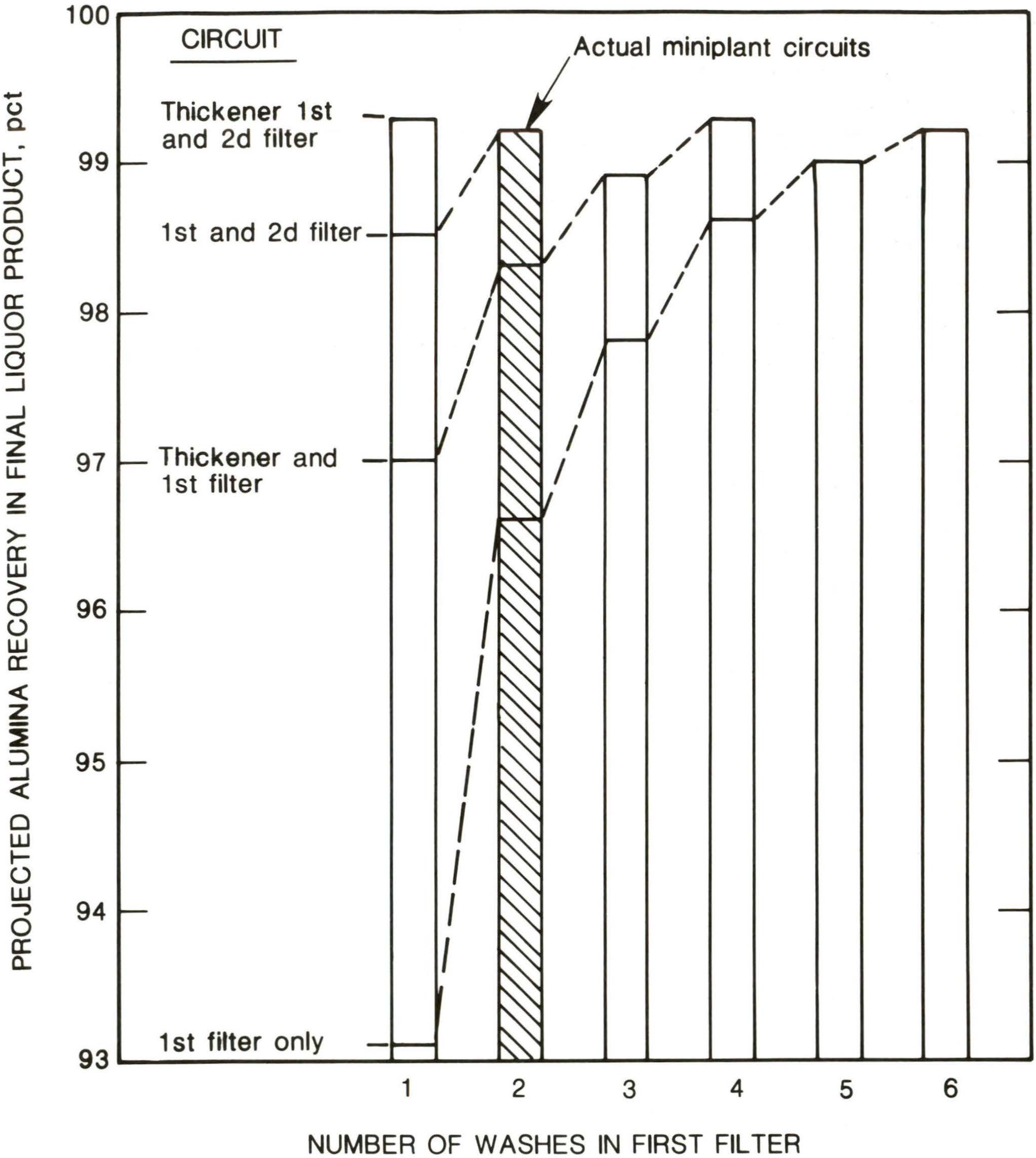

FIGURE 13. - Effect of number of washes in first filter on soluble alumina recovery for minus 18-mesh calcined misted kaolin.

soluble alumina and 10 thickeners, 99.0 to 99.7 pct.

The data in this filtration study can be used for setting specifications for using a horizontal belt filter with any of the calcined kaolin feed materials tested, at varying production rates, and for a specified soluble alumina recovery. The following example is provided:

Assumptions:

1. 1,000-tpd alumina production.
2. Misted feed.
3. 96.8-pct recovery of soluble alumina.

Specifications:

1. One filter with two washes (table 5, fig. 12 or 13).
2. Residue

$$\frac{122.88 \text{ lb residue/h} \times 1{,}000 \text{ tpd} \times 2{,}000 \text{ lb/ton}}{76.47 \text{ lb } Al_2O_3\text{/h} \times 0.968 \times 24 \text{ h/day}} \simeq 138{,}000 \text{ lb/h}.$$

3. Wash liquor (N = 1.5):

$$\frac{138{,}000 \text{ lb/h} \times 1.5}{60 \text{ min/h} \times 8.34 \text{ lb/gal}} = 414 \text{ gal/min}.$$

4. Form area (fig. 10):

$$\frac{138{,}000 \text{ lb/h}}{860 \text{ lb/h}\cdot\text{ft}^{-2}} = 160 \text{ ft}^2.$$

5. First wash area (table F-3):

$$414 \text{ gal/min} \times 1.31 \text{ lb/ft}^2 \times 0.29 \frac{\text{min}\cdot\text{ft}^4}{\text{lb}\cdot\text{gal}} = 157 \text{ ft}^2.$$

6. Second wash area (table F-3):

$$414 \text{ gal/min} \times 1.31 \text{ lb/ft}^2 \times 0.21 \frac{\text{min}\cdot\text{ft}^4}{\text{lb}\cdot\text{gal}} = 157 \text{ ft}^2.$$

7. Drying rate (fig. F-1):

$$\frac{60 \text{ min/h}}{0.5 \text{ min}\cdot\text{ft}^2\text{/lb}} = 120 \text{ lb/h}\cdot\text{ft}^{-2}.$$

8. Drying area:

$$\frac{138{,}000 \text{ lb/h}}{120 \text{ lb/h}\cdot\text{ft}^{-2}} = 1{,}150 \text{ ft}^2.$$

9. Total operational area:

   $160 \text{ ft}^2 + 157 \text{ ft}^2 + 114 \text{ ft}^2 + 1{,}150 \text{ ft}^2 = 1{,}581 \text{ ft}^2.$

10. Baffle area:

    (assume $0.375/10.5 \text{ ft}^2$ baffle area per baffle per ft^2 filter area)

    $1{,}581 \text{ ft}^2 \times 2 \times 0.375 \text{ ft}^2/10.5 \text{ ft}^2 = 113 \text{ ft}^2.$

11. Total filter area = $1{,}694 \text{ ft}^2$.

SUMMARY AND CONCLUSIONS

Three preliminary studies on filtration of slurries from acid-leaching of calcined kaolin were made to develop characteristic data for designing the miniplant circuit and test program: The tests showed the following:

1. The higher the fines content in the filter feed, the higher were both the filter area and the flocculant requirement except in the case of misted feed.

2. Bench-scale leaching slurry filtered considerably faster than miniplant leaching slurry.

3. Washing efficiency was higher when filtering leach slurry of finer particle size.

4. Only the misted feed yielded high filtration rate and high washing efficiency.

5. Prethickening increased filtration rate by 300 pct for the misted and minus 10-mesh kaolin, but only by 50 pct for the minus 20-mesh and minus 35-mesh kaolin.

6. Removing the minus 100-mesh material from the leaching feed increased filtration rate but decreased washing efficiency.

7. Efficiency of a single-stage wash was 10 pct higher than the average efficiency per wash in a four-stage wash.

8. Poor washing efficiency is caused by slow release of $AlCl_3$ in the filter cake to the washing solution.

9. The rate of $AlCl_3$ release is slower for larger particle sizes.

The following conclusions were reached in subsequent miniplant studies using commercial horizontal belt filters:

1. The horizontal belt filter is a practical method for solids-liquid separation in the production of cell-grade alumina by the clay-HCl process. Except for low washing efficiencies, no problems were encountered in operation or maintenance of the units tested or in the solids-liquid separation.

2. Filtration performance on a calcined kaolin that had been prepared by a Bureau-developed misting process was characterized by high filter capacity and low flocculant requirements.

3. Soluble alumina recovery of about 95 pct can be obtained with a single filter with two countercurrent washes and at an overall filtration rate as high as 100 lb solids/$h \cdot ft^{-2}$ of filter area.

4. Soluble alumina recovery as high as 99.2 pct can be achieved by two filters in series with an intermedite repulper and a total of three countercurrent washing steps. The final pregnant liquor would contain about 8.9 pct Al_2O_3 and 200 ppm of fine, suspended solids. The final filter cake would contain approximately 43 pct moisture, analyze 0.6 pct Al_2O_3, and have a volume of 4 pct more than the volume of the as-mined kaolin. Overall filtration rate would be about 60 lb solids/$h \cdot ft^{-2}$ of filter area.

5. Addition of a thickening step before filtration would decrease alumina loss in the final filter cake by 50 pct, but would not increase the overall filtration rate.

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APPENDIX A.--BENCH-SCALE MATERIALS, EQUIPMENT, AND PROCEDURES

FEED

Discharge slurry samples taken from the miniplant acid leaching circuit during a classifier-thickener study (6) were used for filter feed in filtration study I. The leaching circuit is described in appendix C. Screen analyses of the calcined feed to the miniplant acid-leaching reactors and of the washed residue from the slurry discharge are presented in table A-1.

In filtration study II, samples of calcined kaolin were batch leached in a bench-scale reactor. The samples designated minus 10 mesh, minus 20 mesh, and minus 35 mesh were obtained by stage-crushing calcined 1/4-inch pellets made from minus 20-mesh kaolin. The samples designated 10 by 100 mesh, 20 by 100 mesh, and 35 by 100 mesh were attained by screening out the minus 100-mesh material from the above products. The misted material was produced by spraying a fine mist of water onto crushed kaolin while tumbling it in a pelletizing drum (2, 5). The misted material was calcined in a furnace and subsequently agitated with air to simulate the particle-to-particle attrition that would have occurred in a fluidized-bed calciner. Screen analyses of the materials before and after leaching are presented in table A-1.

LEACHING

A 4-L, resin-kettle reactor with a hemispherical bottom was used for leaching and was stirred by a 3/4- by 4-in flat-blade impeller of "half-moon" shape. The impeller was located 1-1/4-in above the bottom of the reactor and turned at 185 rpm, which was the minimum speed for keeping the solids suspended. Leaching was performed at 107° C for 30 min after

TABLE A-1. - Dry-screen analyses of calcined kaolin feeds and residues for studies I and II

(Cumulative weight retained, percent)

Tyler standard sieve, mesh	Nominal feed size, mesh							
	Minus 10[1]	Minus 10	10 by 100	Minus 18[2]	Minus 20	20 by 100	Minus 35	35 by 100
KAOLIN FEED								
14............	28	33	36	NM	NM	NM	NM	NM
20............	49	55	59	NM	NM	NM	NM	NM
28............	61	68	73	22	29	34	NM	NM
35............	70	77	82	53	51	60	NM	NM
48............	79	84	90	71	67	79	50	66
65............	86	89	96	81	77	91	64	85
100...........	92	93	NM	80	85	NM	75	NM
150...........	95	96	NM	94	91	NM	83	NM
WASHED ACID-LEACHED RESIDUE								
14............	16	35	35	NM	NM	NM	NM	NM
20............	30	55	59	NM	NM	NM	NM	NM
28............	46	68	72	22	32	43	NM	NM
35............	58	76	82	43	52	62	29	43
48............	67	83	89	70	69	79	50	67
65............	73	88	95	81	77	90	66	87
100...........	77	92	98	89	82	96	76	88
150...........	79	94	99	94	86	97	81	94

NM Not measured.
[1]Filtration study I; all others are for filtration study II.
[2]Misted.

addition of solids to the boiling liquor. The reactor and the leaching procedure approximated the way a continuous commercial-scale leaching operation might be conducted. After leaching, the slurry was rapidly cooled to 60° C in a water bath and divided in a rotary splitter into a number of samples. A one-eighth split provided the amount of slurry used for filter feed in most of the individual tests. Samples were kept in a 60° C bath before filtration. This temperature is typical of a commercial filter feed temperature.

The charge to each leaching batch was 1,655 g of 26-pct HCl and 461 g of calcined kaolin. The recovered leaching slurry for testing contained an average of 284 g of residue solids and 1,820 g of liquor that analyzed 10 pct Al_2O_3. The one-eighth-split samples each contained 200 mL of slurry to which 47 mL of aluminum chloride liquor containing 6.7 pct Al_2O_3 was added to simulate recycling the first wash to the filter feed.

THICKENING

Thickening before filtration was carried out in a 2-L graduate containing a four-wire stirrer run at 1 rpm. The stirrer, which simulated a thickener rake and consisted of a cage that had four 12-gage titanium wires connected at the top and bottom, was 17 in long, encompassed a diameter of 2-3/8 in, and extended to 1/2 in above the bottom of the graduate. The graduate was kept in an 80° C bath during thickening. Before thickening, 378 mL of aluminum chloride liquor (6.7 pct Al_2O_3) was added to the slurry from the leaching step to simulate form filtrate recycle in a continuous operation. The total volume of slurry plus recycle was about 1,900 mL. The flocculant solution was added by stroking a blunger consisting of an inverted rubber stopper on a glass rod up and down for five strokes and injecting one-fifth of the flocculant to the face of the stopper on each stroke. After flocculant addition and mixing, which took 15 s, the blunger was removed, the stirrer inserted, and thickening commenced. After about 16 h of settling, approximately 1,200 mL of clear liquor was decanted. The thickened product containing about 30 pct solids was split into eight 90-mL samples. Each sample was diluted with 47 mL of the 6.7-pct-Al_2O_3 liquor to simulate recycling the first washing filtrate and to provide a filter feed of 20 pct solids.

FILTRATION

A cylindrical filter having 7.2 in^2 (0.05 ft^2) of filtration area, a grooved base for filtrate drainage and 4-in-high sides (3) was used. The drainage cavity was connected through a bottle trap to a small centrifugal vacuum pump with vacuum level controls and a bypass system. The polypropylene filter cloth (Eimco POPR 896) was stretched over the grooved base, and cemented between the outside wall of the base and the inside wall of the cylinder. Separan MGL flocculant was diluted to between 0.02 and 0.2 pct in water. Plastic beakers (2-1/2 in diam by 3-1/2-in high) were used for storing filter feed and as mixing vessels. A flat-bladed, 1-1/4-in stirrer operating at about 100 rpm was used to mix the flocculant with the slurry sample for 15 s before pouring into the filter test cylinder. Cake-washing solution was added from a 2.7-in-ID cylinder with a perforated bottom with 25 1/16-in holes (designed to add the average volume of washing solution in 2 s). Batch filtration testing procedures (3) were the same for miniplant and bench-scale kaolin and included a single-stage wash. Repulping the final filter cake with a fixed amount of water (3) provided an analytical sample for use in residual cake liquor analyses.[1] Filter cloth selection was made by checking for cake cracking and comparing filtrates for clarity and cloths for blinding. Cloths tested are described in table A-2.

[1]Analytical and calculation methods for the bench-scale filtration studies are the same as in appendix E except that the FSFR estimated from bench-scale tests will have an 0.8 operating factor applied to allow for briding between sections and variations in operating conditions that occur in continuous filtration.

TABLE A-2. - Description of filter cloths tested in studies I and II

Eimco designation	Weave	Yarn filament	Thread, count/in	Weight, oz/yd^2	Airflow, cfm/ft^2	Finish[1]
NY527F.............	Twill.....	Mono......	157×62	5.75	176	Calendered.
NY529F.............	...do.....	Multi.....	144×54	8.0	39- 65	Heat-set.
POPR873............	Plain.....	Mono......	166×34	6.15	25- 35	Calendered.
POPR896............	Twill.....	...do.....	68×30	8.5	120-150	Greige.[2]
POPR929............	Basket....	...do.....	110×45	7.25	60	Calendered.

[1]Standard cloth industry terminology for cloth finish.
[2]Means no treatment after weaving.

Two bench-scale filtration tests that simulated multistage countercurrent washing were performed. The apparatus (shown in figure A-1) had a multiport valve that separated washing filtrates without having to stop the test to switch filtrate flasks.

Initially, an unwashed cake is formed in the test cylinder. After a short prewash dry, a present volume of synthesized first-stage washing liquor is applied and the time noted to incipient disappearance of the washing liquor from the surface. After a preselected dry period, a like volume of synthesized second-stage washing liquor is applied. In a similar manner, volumes of synthesized third- and fourth-stage washing liquors are applied.

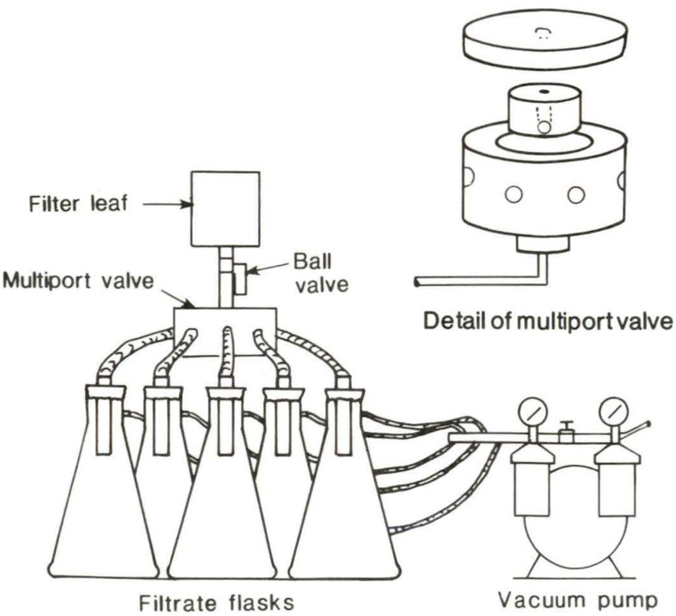

FIGURE A-1. - Filter apparatus for simulating multistage countercurrent washing.

The cake is permitted to dewater during the final drying period. When each washing volume is applied, the multiport valve is turned to direct the filtrate flow into another flask, so that forming- and washing-stage filtrates are collected separately. The synthesized washes containing decanted pregnant liquor are diluted with distilled water so that preselected washing volumes and estimated Al_2O_3 concentrations representative of a continuous four-stage washing filter at steady state can be obtained. It is not important that the estimated concentrations be exact because the wash filtrates obtained in sequence 1 are used in additional test sequences exactly representative of a countercurrent operation.

In test sequence 2, an unwashed filter cake is formed and the liquor used for the first washing is the second-stage washing filtrate from sequence 1; the liquor used for the second-stage washing is the third-stage washing filtrate from sequence 1, etc. Fresh water is used for the fourth-stage washing and at the same volume as in sequence 1. By repeating the above procedure five times, filtrate concentrations of each individual stage should be representative of steady-state conditions on a continuous filter operating at a similar volume of fresh water to the fourth-stage wash. After each sequence, the washed cake is weighed, repulped, filtered, washed and reweighed, and the repulp liquor analyzed. The cake is discarded and a fresh cake is formed for the following test sequence. The filtrates from each washing stage are only analyzed for the final test sequence (sequence 5).

APPENDIX B.--DETAILED RESULTS OF BENCH-SCALE STUDIES

Estimates of filtration characteristics[1] developed from studies I and II are as follows:

1. Form time (Θ_f) versus cake loading (w) for unthickened and thickened slurries (figs. B-1 and B-2).

2. Correlating factor or drying time divided by cake loading (Θ_d/w) versus filter cake moisture content (figs. B-3 and B-4).

3. Multiplication factor for determining washing time from product of cake loading times wash volume (table B-1).

[1]Because the removal of fines before leaching was not adequately offset by improved filtration, development of filtration characteristics for specialty feeds (10 by 100, 20 by 100, and 35 by 100 mesh) was not warranted.

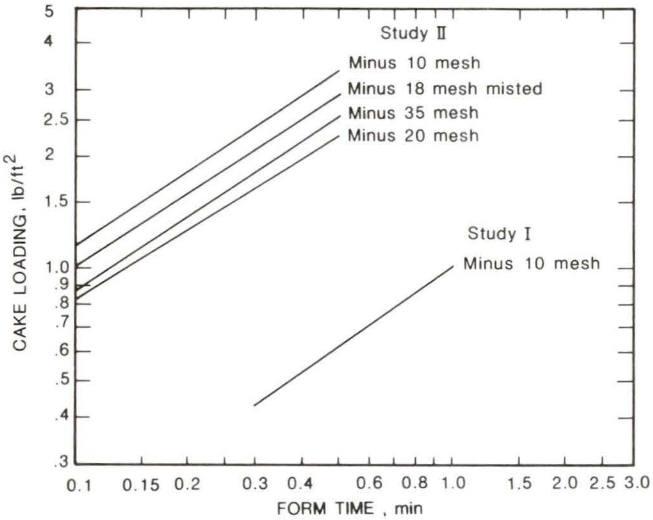

FIGURE B-1. - Form time versus cake loading for unthickened slurry in studies I and II.

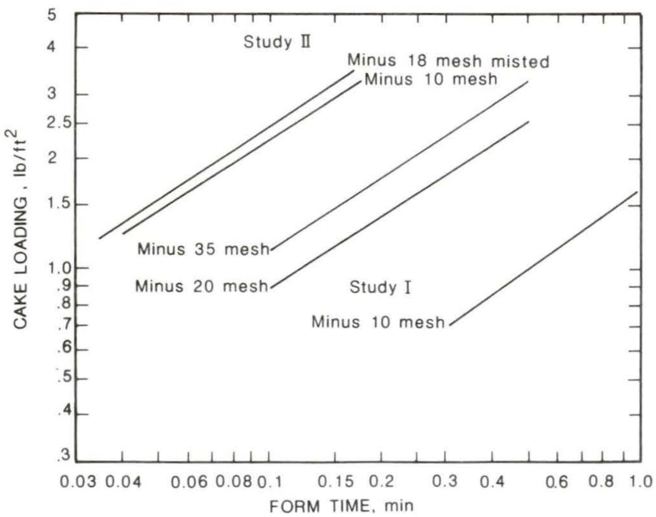

FIGURE B-2. - Form time versus cake loading for thickened slurry in studies I and II.

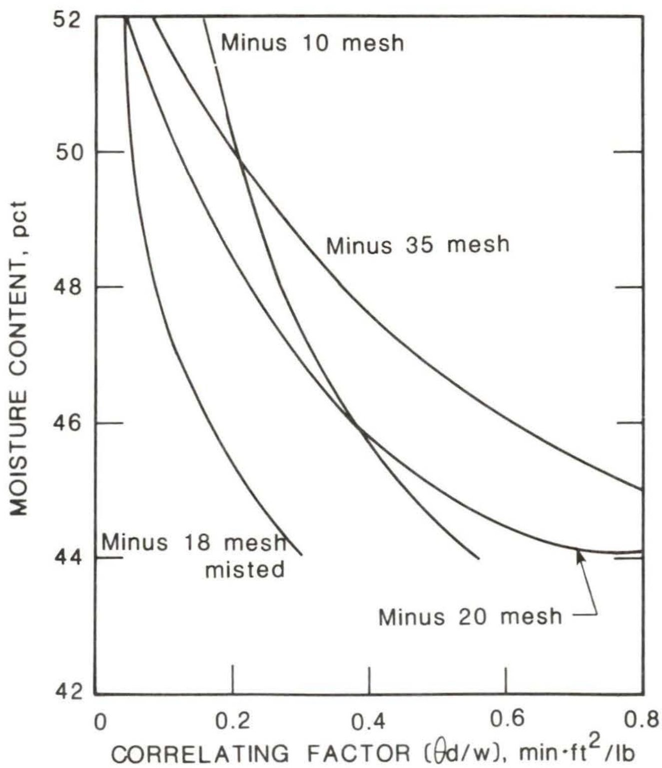

FIGURE B-3. - Correlating factor, θ_d/w, versus final filter cake moisture for unthickened slurry in study II.

TABLE B-1. - Factors from study II for calculating washing-area requirements

Calcined kaolin[1] feed size, mesh	Multiplication factor,[2] (min•ft^4/lb•gal)	
	Unthickened	Thickened
Minus 10........	0.21	0.11
Minus 18 misted.	.11	.11
Minus 20........	.17	.17
Minus 35........	.14	.18

[1]Specialty screened products (10 by 100, 20 by 100, and 35 by 100 mesh) are not included.

[2]To obtain washing area requirements (ft^2) multiply factor by cake loading (lb solids/ft^2) times wash addition (gal/min).

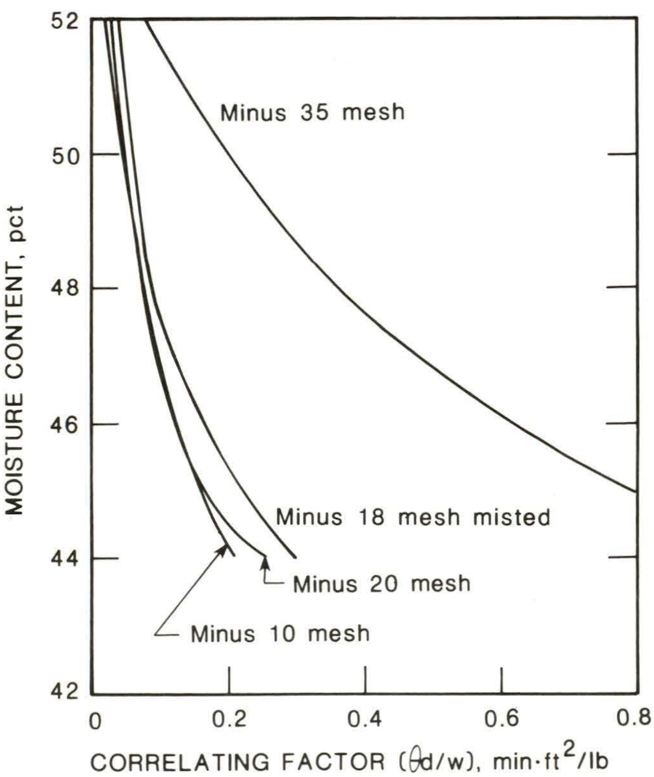

FIGURE B-4. - Correlating factor, θ_d/w, versus final filter cake moisture for thickened slurry in study II.

4. Wash displacements (N) versus washing efficiency (fig. B-5).

5. Estimated filter performance indicated by the above filtration characteristics (table B-2).

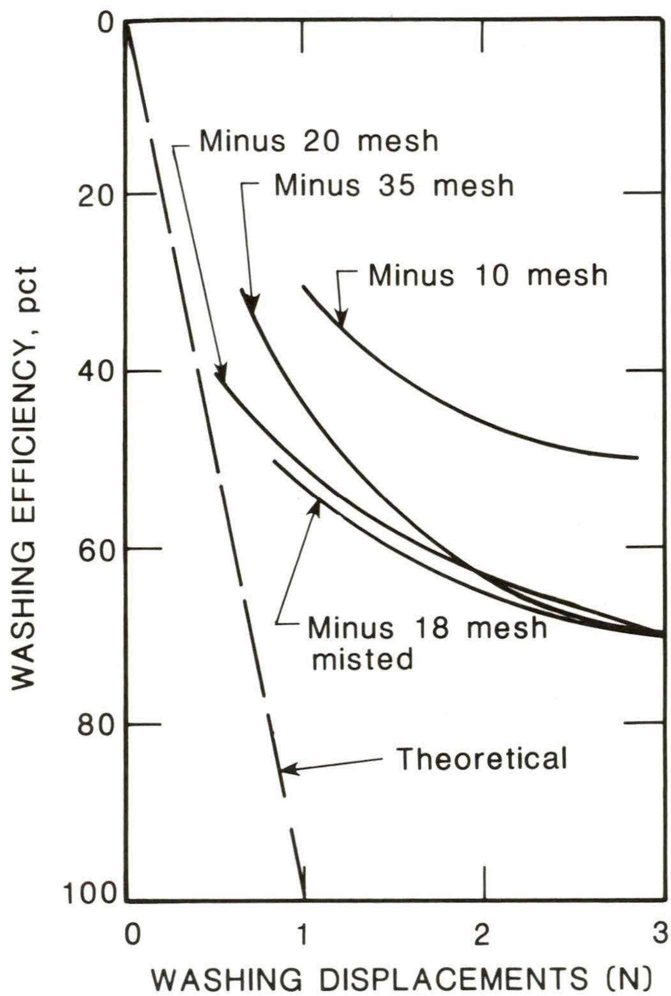

FIGURE B-5. - Number of washing displacements, N, versus washing efficiency for thickened and unthickened slurry in study II.

TABLE B-2. - Critical measurements and filtration response for study II

Calcined kaolin feed size, mesh	Filter feed solids, pct minus 150 mesh	Flocculant requirements, lb Separan MGL/ton solids[1]	Washing efficiency, pct (N = 2.3)	Al_2O_3 recovery,[2] pct	Filtration rate, $lb/h \cdot ft^{-2}$ Forming	Full-scale[2]
Unthickened:						
Minus 10.....	5.5	0.2	48	96	670	107
Minus 18[3]....	6.0	.2	67	98	540	192
Minus 20.....	14.0	.2	65	98	380	119
Minus 35.....	19.3	.5	66	98	430	109
10 by 100....	.9	.1	56	97	1,020	137
20 by 100....	3.2	.2	54	97	2,990	253
35 by 100....	5.6	.2	55	97	2,970	168
Thickened:						
Minus 10.....	5.5	.4	48	98	1,830	290
Minus 18[3]....	6.0	.3	67	99	2,020	283
Minus 20.....	14.0	.5	65	99	440	156
Minus 35.....	19.3	.7	66	99	630	112
10 by 100....	.9	.2	56	98.5	2,770	153
20 by 100....	3.2	.3	54	98.5	5,890	266
35 by 100....	5.6	.3	55	98.5	5,890	182
Multistage washing (unthickened):						
20 by 100....	3.2	.2	[4]48	[4]96	3,080	[4]120
Minus 20.....	14.0	.2	[5]55	[5]92	670	[5]55

[1]Includes flocculant for thickening when thickening was used.
[2]Estimated for forming, 2 2.3-displacement washes, and final cake dried to 50 pct moisture except where noted otherwise.
[3]Misted feed.
[4]Obtained by forming, 4 1.6-displacement washes, final cake dried to 43 pct moisture.
[5]Obtained by forming, 4 2.6-displacement washes, final cake dried to 40 pct moisture.

Figure B-6 (study III) indicates the release with time of aluminum chloride to a diluting solution from leached 1/4-in pellets and from leached 20- by 100-mesh particles.

Bench-scale tests run on the clay-HCl miniplant leaching slurry were made to verify miniplant filtration rates in study IV. Characteristic cake-loading versus form-time curves are given in figures B-7 and B-8; filtration characteristic curves for cake moisture versus drying time are given in figure B-9.

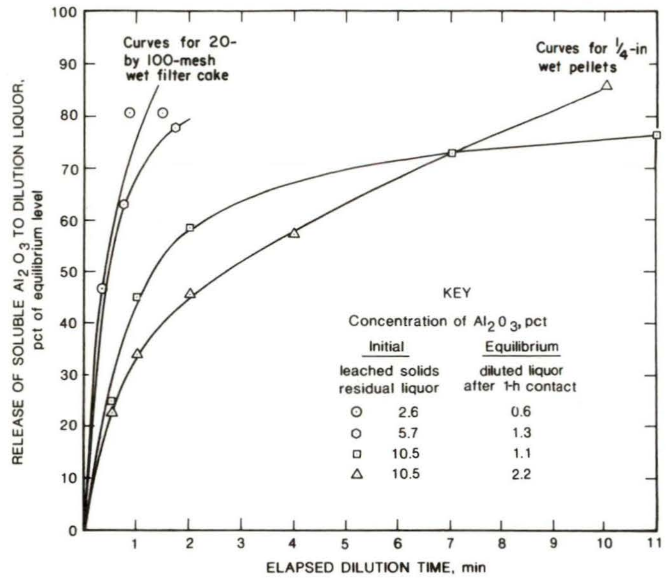

FIGURE B-6. - Effect of particle size on rate of soluble alumina release from the residue to the washing solution.

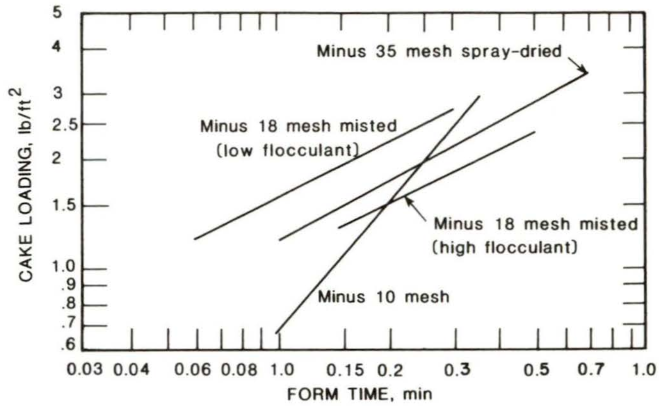

FIGURE B-7. - Log-log plot of form time versus cake loading for unthickened slurry in study IV using unbaffled leaching reactors.

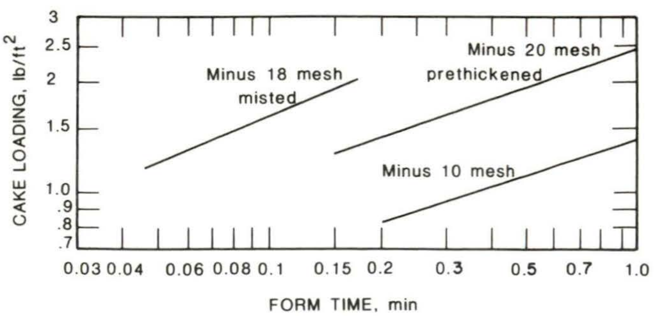

FIGURE B-8. - Log-log plot of form time versus cake loading for unthickened slurry in study IV using baffled leaching reactors.

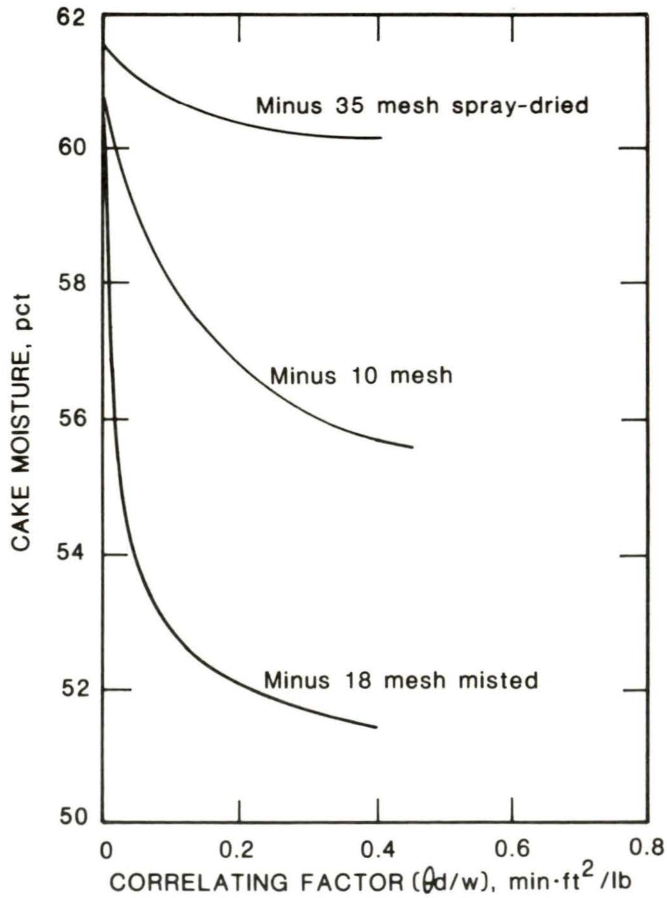

FIGURE B-9. - Correlating factor, θ_d/w, versus final cake moisture for unthickened slurry in study IV.

APPENDIX C.--MINIPLANT MATERIALS AND EQUIPMENT

FEED

Four calcined kaolin materials were used as leaching feed. Screen analyses of the feed materials and of the washed solids from the leach residue are presented in table C-1. The materials differed in particle size, method of preparation, and particle degradation during leaching as stated below:

1. Minus 10-mesh (baseline)--Raw kaolin was crushed to one-quarter inch, the minus 20-mesh fines were pelletized, and the combined material was calcined in a rotary kiln. The calcined material was stage-crushed to size. This material degraded appreciably during leaching in unbaffled reactors but much less in baffled reactors. The reactors are discussed in the section, "Miniplant Leaching." The minus 10-mesh material was used in other miniplant and bench-scale solids-liquid separation studies (4, 6).

2. Minus 18-mesh misted--Raw kaolin was crushed to 18 mesh, tumbled on a pelletizing disk, and sprayed with a mist of water in an amount less than that necessary for pellet formation. The product was calcined in either a fluidized-bed reactor or rotary kiln and was not crushed after calcination (5, 7). The material degraded very little during leaching and gave superior filtration in preliminary bench-scale testing and studies made at Albany Research Center during development of the misting process (2).

3. Minus 20-mesh--Preparation was similar to that of the minus 10-mesh material. The material degraded appreciably during leaching in unbaffled reactors.

4. Minus 35-mesh spray-dried--A 64-pct suspension of kaolin in water was spray dried and calcined in a rotary kiln. Unlike crushed calcined kaolin feed materials, the calcined spray-dried kaolin degraded almost imperceptibly during leaching in the unbaffled reactors. The remarkably smooth particle surfaces of the spray-dried material and the narrow range of particle size distribution (table C-2) are factors that would enhance filtration of the leaching slurry.

TABLE C-1. - Wet-screen analyses of calcined kaolin feeds and residues from leaching in unbaffled reactors

(Cumulative weight retained, percent)

Tyler standard size, mesh	Nominal feed size, mesh		
	Minus 10[1]	Minus 18[2]	Minus 20
CALCINED KAOLIN FEED			
10.........	8	<1	NM
14.........	30	1	NM
20.........	53	21	2
28.........	68	43	38
35.........	76	60	57
48.........	82	75	71
65.........	86	85	79
100........	89	91	84
150........	92	94	88
WASHED ACID-LEACHED RESIDUE			
10.........	6	NM	NM
14.........	24	1	NM
20.........	45	20	1
28.........	59	40	32
35.........	66	57	51
48.........	73	72	64
65.........	78	82	72
100........	82	89	77
150........	84	92	81

NM Not measured.
[1]Baseline.
[2]Misted.

TABLE C-2. - Wet-screen analyses of calcined kaolin feeds and residues from leaching in baffled reactors

(Cumulative weight retained, percent)

Tyler standard size, mesh	Nominal feed size, mesh		
	Minus 10[1]	Minus 18[2]	Minus 35[3]
CALCINED KAOLIN FEED			
10........	8	NM	NM
14........	30	NM	NM
20........	53	3	NM
28........	68	23	NM
35........	76	42	<1
48........	82	62	12
65........	86	78	50
100.......	89	87	78
150.......	92	92	90
WASHED ACID-LEACHED RESIDUE			
10........	6	NM	NM
14........	24	NM	NM
20........	47	3	NM
28........	61	20	NM
35........	71	39	<1
48........	78	58	10
65........	83	73	50
100.......	86	83	78
150.......	89	90	90

[1]Baseline.
[2]Misted.
[3]Spray dried.

LEACHING SECTION

The clay-HCl miniplant leaching circuit consisted of three 50-gal, glass-lined steel reactors (24-in ID) in cascade. Hot oil circulating within the jackets provided temperature control. Reflux condensers for HCl-H_2O vapor condensation were water cooled. Before filtration, slurry temperature was decreased from 108° C to less than 60° C by a water-cooled glass condenser. For filtration tests 1 through 14, the leaching reactor circuit was the same as that used in a classifier-thickener study (6) and in bench-scale filtration study I. The reactor overflows were located 16 in above the kettle bottom and provided about 22 gal of effective volume per reactor. Slurry mixers were three-blade, glass-coated steel, retreating curve stirrers operating at 120 rpm.

In miniplant filtration tests 15 through 22, the leaching reactor circuit was modified to decrease the generation of fines during leaching. The modifications were as follows:

1. Wider impeller blades were used.

2. Four baffles were added in place of a single vortex breaker.

3. A subsurface slurry transfer system was installed, which increased effective volume of reactors from 22 to about 27 gal.

4. Impeller speed was decreased from 120 to 50 rpm.

FIRST FILTER

The first of two filters used in the filtration circuit was a 10-1/2- by 1-ft unit (fig. 2) that was designed to handle pregnant liquor at 60° C or less and as many as three washes. The filter was set up for two washes. Section arrangement is shown in figure C-1. Belt drive and tension were supplied to Neoprene-covered, 18-in head and tail pulleys. The drive mechanism was a 2-hp ac motor with a variable-speed drive, which provided belt speeds from about 1 to 10 ft/min. The elastomer belt material (Eimco EPDM polyester type 1023) was selected after immersion tests of 1- and 2-month duration on three types of elastomers in a solution containing 20 pct $AlCl_3$, 0.5 pct $FeCl_3$, and 5 pct free HCl at 60° C. On the basis of measurements and microscopic examination and of the samples indicating little or no deterioration, the belt manufacturer predicted excellent elastomer life. The filter cloth (Eimco POPR 896) was selected from previous bench-scale testing (table A-2). The filter was made for continuous industrial application. All wetted parts were Neoprene, titanium, Hastalloy B, or fiberglass. Filtrate receivers, filtrate

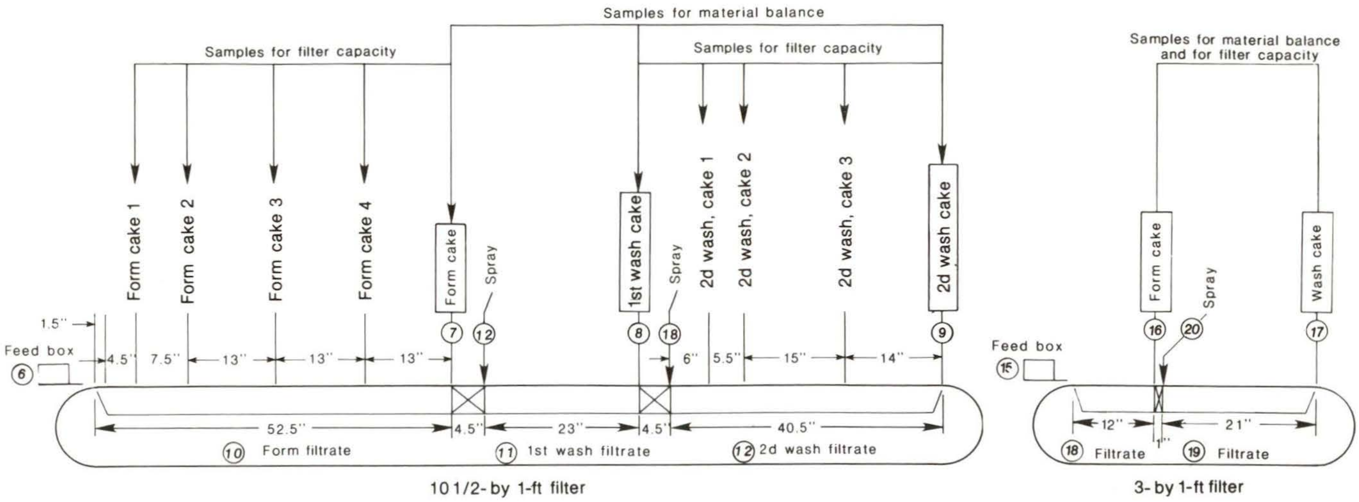

FIGURE C-1. - Section arrangement of filter and cake-sampling locations.

lines, and vacuum lines were fiberglass. Depending on location, nonwetted parts were 316 stainless steel, fiberglass, or painted mild steel. Filtrate pumps, with titanium sleeves and double mechanical seals, were made of Kynar, a polyvinylidene fluoride.

VACUUM SYSTEM

The liquid-ring vacuum pump was of all-iron construction. A 45-L/min water supply and prior removal of any substantial quantity of HCl vapor in the airflow coming from the filtrate receivers were required. A cascade condenser-scrubber was inserted between the vacuum pump and the filtrate receivers. The unit was a 24-in-diam by 60-in-high fiberglass tank in which a curtain of water was cascaded in a series of three rings down to a filter pump connected to the bottom of the tank and then routed to waste. The air flow from the top of the filtrate receivers entered at the bottom of the condenser and flowed up through three successive water curtains and to the liquid-ring vacuum pump. The three water curtains were formed by water cascading from the upper water inlet over a disk in a ringed pattern, laundered to cascade over a second ring and then a third. A 2-1/2-in, rubber-lined filtrate pump rated at 570 L/min (50 ft total discharge head at 1,750 rpm) removed water from the cascade condenser. The vacuum system air flow was measured and analyzed for HCl content in two tests (table C-3). At least 114 L/min of water fed to the cascade condenser was needed to remove between 60 and 80 pct of the HCl from the air flowing through the vacuum system. The HCl concentration in the vacuum system was recorded at two levels, 0.0009 and 0.003 g/ft^3 (STP). Estimates based on the pH of the liquid-ring water indicated that the condenser decreased the HCl concentration in the vacuum system from the above levels to 0.0002 and 0.001 g/ft^3, respectively.

The pH of the liquid-ring water discharging from the vacuum pump was an index of the vacuum pump corrosion environment. Plant water pH was 7.7. The liquid-ring water discharge ranged from pH 6.5 to 7.0 without the cascade condenser and from 7.0 to 7.5 with the cascade condenser. Craig Gilmore, service engineer, Nash Engineering Co., Norwalk, CT, reported that satisfactory pump life would be obtained in the pH range 6.5 to 7.0 without the cascade condenser. He recommended half the liquid-ring water addition should be added as a spray just ahead of the vacuum pump and the other half directly to the liquid ring. If higher HCl concentrations are encountered, vacuum pump water addition should be put on a recirculation system with caustic added.

TABLE C-3. - Distribution of HCl in air and water flow streams in the first filter vacuum system

Cascade condenser test..	A[1]	B[1]	C[1]	D[1]	E[2]	F[2]	G[2]	H[2]
Cascade condenser water:								
Water flow.....L/min..	0	45	114	228	0	45	114	228
HCl removal[3].....pct..	0	0	63	63	0	0	80	80
Vacuum system air:								
Vacuum level...in Hg..	12	12	12	12	20	20	20	20
Airflow from pump curve...std ft^3/min..	425	425	425	425	435	435	435	435
Airflow measured std ft^3/min..	474	474	474	474	336	336	336	336
HCl content (std ft^3).....g/ft^3..	0.0033	0.0033	0.0033	0.0033	0.00094	0.00094	0.00094	0.00094
Vacuum pump liquid-ring water:								
Water flow.....L/min..	45.4	45.4	45.4	45.4	45.4	45.4	45.4	45.4
pH..................	6.5	6.5	7.0	7.0	7.0	7.0	7.5	7.5
HCl content[4].....g/L..	0.031	0.031	0.0115	0.0115	0.0115	0.0115	0.0017	0.0017
HCl flow.......g/min..	1.41	1.41	0.52	0.52	0.52	0.52	0.08	0.08

[1]Run during miniplant tests 6-8.

[2]Run during miniplant test 14.

[3]Assuming that any HCl not picked up by the cascade condenser reports to the liquid-ring water.

[4]Estimated by determining the amount of HCl necessary to lower the same plant water being fed to the liquid ring to the same pH as the actual water discharging from the liquid ring.

The cascade condenser manufacturer reported that failure of the wash water to remove HCl at the 45-L/min flow rate occurred because the rate was below the recommended 114 to 228 L/min; gaps occurred in the water curtain and allowed air to bypass. The manufacturer recommended using a commercial demister if lower water requirements were desired. This is a scrubber but with polypropylene mattes in place of the packed column. The unacceptably high water flow rate specified for the cascade condenser reflects the owners' concern about corrosion of their rental filter. The problem of gaps at low flow rates was not known until testing was completed. A recirculating system to maintain the required flow rate or a spray-type scrubber should have been used.

SECOND FILTER

A 3- by 1-ft vacuum filter designed for use with HCl was modified to include only a form section and a single wash section. Section arrangement is shown in figure C-1. All wetted parts were made of fiberglass. The same polypropylene filter cloth (Eimco POPR 896) was used as in the larger filter unit.

The filtrate pump had casing and impellers of silica-filled epoxy with a ceramic shaft and double mechanical seals. The liquid-ring vacuum pump was of all iron construction and required an 11-L/min water supply. Because of dilution of the residual cake liquor in the first filter, a scrubber or condenser system was not needed in the vacuum system. The pH of the liquid-ring water discharge was not measurably different from that of the supply water.

THICKENER

The thickener was a flat-bottom, fiberglass cylinder 58 in ID and 6 ft deep and had a cross-sectional area of 18.4 ft^2.

The rake, which was inward ploughing, and rake shaft were rubber-covered. Rake speed was originally 0.38 rpm and provided a peripheral speed of 5.6 ft/min. At this rate, settled solids packed into the underflow pump intake piping and caused plugging. Underflow pumping was substantially improved by adding an on-off timer and running the rakes intermittently so that the peripheral travel was only 1.4 ft/min.

REPULPER

The repulper was a 9-in-diam by 8-ft-long rubber-lined screw classifier, which was set at about a 3° slope instead of the usual 10° to 14° slope. The screw was 8-in in diam, rotated at 3.7 rpm, and when filled to overflowing with water, had a volume of 2 ft^3. Effective volume was only about 1 ft^3, or 10 lb of residue at a retention time of 10 min.

APPENDIX D.--MINIPLANT SAMPLING

Two sets of samples were taken for each filtration test. The filtration circuit was started 1 h after the reactor circuit began discharging leaching slurry. The filtration circuit was sampled after a 2-h wait for the filter circuit to attain equilibrium. The following samples were taken:

1. Leaching discharge slurry.

2. Each filtrate liquor (see figs. 5-8).

3. Each filter cake as designated in figure C-1.

4. Repulper discharge when the 2d filter was used.

5. Thickener overflow liquor and underflow slurry when thickener was used.

Samples of about 500 mL were taken over a period of 15 to 20 min in a sequence that minimized upset of the circuit equilibrium. The individual sets of samples were taken between 45 and 60 min apart. When more than one filter test was made during a 7- to 9-h leaching run, 60 min was allowed for the changed filtration circuit to come to equilibrium before sampling was undertaken. Slurry, liquor, and filter cake samples taken for material balances (fig. C-1) were analyzed for percent or parts per million solids, percent Al_2O_3 in liquor fraction, and density (grams per milliliter) of the liquor fraction. Filter cake samples required for capacity measurements (fig. C-1) were analyzed for percent solids.

APPENDIX E.--MINIPLANT CALCULATION METHODS

RESIDUAL LIQUOR ANALYSES

The density and Al_2O_3 analyses of the residual liquor in the filter cakes were determined by a repulping method. The wet cake was weighed, repulped by shaking in a sealed jar with at least one-half the wet cake weight of distilled water. The sample was settled for 1 h. An aliquot of clear liquor was decanted or filtered off and analyzed for Al_2O_3 and density. Al_2O_3 was washed from the remaining sample. The washed cake was then dried and weighed. The weight of the residual cake liquor was the difference between the wet and dry cake weights. Either the density or the percent Al_2O_3 of the residual cake liquor was obtained by multiplying the corresponding analysis figure for the repulp liquor by a correction factor. The factor was the ratio of the weight of the distilled water added in repulping plus the weight of the residual cake liquor divided by the weight of the residual cake liquor.

MATERIAL BALANCE

A material balance for soluble Al_2O_3, liquor, and solids was made for each test. Table E-1 is a typical example of these material balances. This balance for test 20 is used to explain the subsequent calculations. In calculating the test 20 material balance, flow rates of the leaching slurry components were calculated first. The leaching slurry produced from a 200-lb/h calcined kaolin feed rate over a 6-day run (6) was used as a reference composition for calculating the material balance for each test and is as follows:

Slurry.................lb/h..	889.18
Liquor.................lb/h..	766.30
Solids.................lb/h..	122.88
Liquor...........pct Al_2O_3..	9.98
Soluble Al_2O_3.........lb/h..	76.47

To calculate a leaching-slurry composition for any specific test, only the percent Al_2O_3 in the leaching slurry liquor for that test and reference composition is used. The test composition is calculated by (1) keeping the slurry flow rate at 889.18 lb/h, (2) setting the percent Al_2O_3 in the leaching slurry liquor at the test value, and (3) setting new rates for the liquor, solids, and soluble Al_2O_3 such that the new flow rate for Al_2O_3 is changed from the reference value by an equal but offsetting change in the flow rate for the solids. If the leaching slurry liquor analysis for the test of table E-1 was 10.20 pct Al_2O_3, the new slurry flow rates calculated by successive approximations are as follows:

Slurry.................lb/h..	889.18
Liquor.................lb/h..	768.18
Solids.................lb/h..	121.00
Liquor...........pct Al_2O_3..	10.20
Soluble Al_2O_3..........lb/h..	78.35

The solids and liquor materials balance for any test can be calculated using the following:

1. The leaching slurry rates and composition as calculated above.

2. Percent solids of final filter cake for each section.

3. Fresh water and flocculant additions, in pounds per hour.

To develop the Al_2O_3 analyses in the material balance, Al_2O_3 analyses of the following products were used and balanced in a "best fit" solution:

1. Residual liquor in all filter cakes that were samples for material balance (fig. C-1). Analytical samples were obtained with the repulping technique (3).

2. All filtrate liquors.

3. Liquor in repulp slurry when second filter was used.

4. Liquor in thickener underflow when thickener was used.

TABLE E-1. - Material balance[1] for test 20[2]

Stream name	Discharge slurry	Feed	Form cake	1st wash cake	2d wash cake	Form filtrate	1st wash filtrate	2d wash filtrate	Fresh water wash	Flocculant solution
Stream No.[3]	1	6	7	8	9	10	11	12	13	14
Rate, lb/h:										
Liquor	768.18	961.41	102.08	98.92	88.85	859.33	185.17	182.01	171.94	8.06
Solids	121.00	121.00	121.00	121.00	121.00	[4]0	[4]0	[4]0	0	0
Al_2O_3	78.35	85.14	9.04	4.81	2.25	76.10	6.79	2.56	0	0
Separan MGL	0	0.016	NA	NA	NA	NA	NA	NA	NA	0.016
Composition:										
Solids........pct of stream	13.61	11.18	54.24	55.02	57.66	[4]0	[4]0	[4]0	0	0
Al_2O_3.........pct of liquor	10.20	8.86	8.86	4.86	2.53	8.86	3.67	1.41	0	0
Separan MGL...pct of liquor	0	0.002	NA	NA	NA	NA	NA	NA	0	0.20
Distribution of Al_2O_3: Fraction reporting..........pct	100.00	108.67	11.54	6.14	2.87	97.13	8.67	3.27	0	0

NA Not available.

[1]Data carried out to hundredths, but accuracy is less for most values.

[2]Operating conditions given in tables 2 and F-1.

[3]All streams are as in 1-filter circuit (fig. 5) except for the discharge slurry, which is part of the leaching operation (fig. 2).

[4]Assumed zero for calculation purposes.

Additional constraints made on the balance were that (1) the form section filter feed liquor, filtrate, and residual cake liquor all had identical Al_2O_3 analyses, and (2) the solids in all the filtrates (form filtrates ranged from 100 to 500 ppm) were to be neglected.

The resulting material balances (such as the example in table E-1) provided final product specification and Al_2O_3 recovery for each test. In addition, the washing efficiency and washing-displacement index (N) for each filter stage were calculated from these balances.

WASHING EFFICIENCY

Washing efficiency is defined as the percent of soluble alumina removed in washing, and is a direct measure of the effectiveness of washing. The washing index (R) used in commercial filtration testing (3), is defined as the percent of soluble alumina left after washing. R is a misleading term because it is an inverse function of washing effectiveness; therefore, washing efficiency is the only index used in this report to indicate soluble alumina removal by washing.

To calculate R for any stage of washing, the following values are used:

A = pct Al_2O_3 in residual cake liquor after washing.

B = pct Al_2O_3 in residual cake liquor before washing.

C = pct Al_2O_3 in washing liquor.

For single-stage washing using fresh wash water,

$$R = 100 \times \frac{A}{B},$$

and washing efficiency $= 100 \times \frac{(B-A)}{B}$.

For any stage washing using other than fresh wash water,

$$R = 100 \times \frac{A-C}{B-C},$$

and washing efficiency $= 100 \times \frac{(B-A)}{B-C}$.

The washing displacement index N is the number of times that the residual cake liquor volume is displaced by a single-stage washing and is equal to the wash volume divided by the volume of the residual cake liquor. In a perfect spray washing system, i.e., plug flow, washing efficiency = 100 × N, and R = 100 × (1-N) when $N \leq 1$.

FILTRATION RATE

Three operations, cake filtration, washing, and drying, occur in horizontal belt filtration. The total filtration rate for a filter incorporates all of these operations and is called full-scale filtration rate (FSFR). An estimate of form filtration rate in the miniplant filter can be made by measuring the distance from the feed box to the furthest point at which liquor still shows on top of the filter cake. This distance is converted to belt area (square feet) and divided into the rate at which solids are fed to the filter. The distance measurements were made for each filtration test and also for the short tests (which only compared the effect of flocculant addition on form filtration rate).

Filtration rates were determined from the measured moisture contents of the cake samples. For each test, the percent moisture versus the cake travel within the particular filter section was plotted as in figure E-1. Conventional Θ_d/w versus cake moisture curves in bench-scale testing established 61 pct as a form cake moisture (fig. B-9). If each filtration stage has the same moisture content by volume as the form cake, the calculated moisture content by weight for each stage would be as follows:

Stage	Pct moisture
1st filter form.......	61
1st wash.........	60
2d wash..........	59
2d filter form........	58
Wash............	57

By equating filter area used to attain these moistures (fig. 12) and setting a

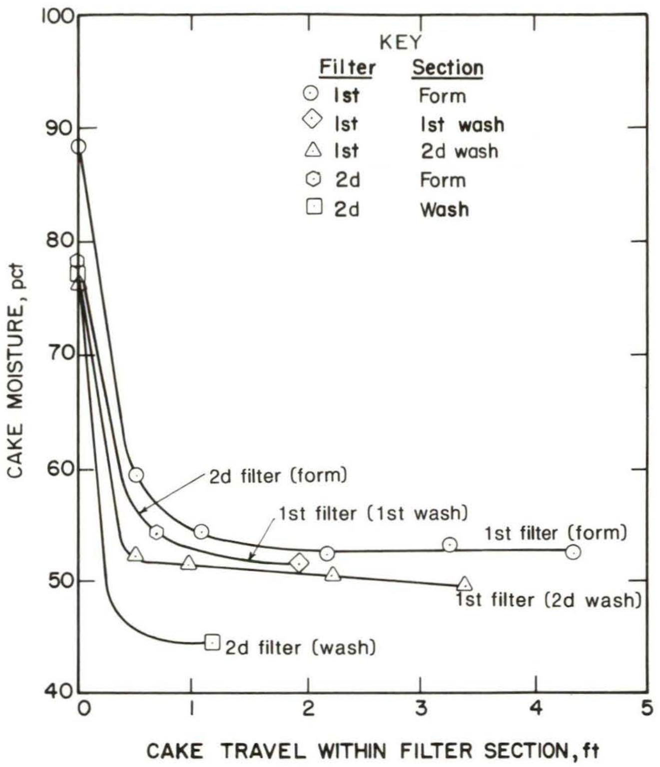

FIGURE E-1. - Plot of cake moisture versus cake travel for filter test 9.

final desired cake moisture, a total filter area requirement or a FSFR can be developed for each test. In the example shown in figure 4, the filter overcapacity is apparent when the actual filter area used is compared to the required filter area. The bar graphs and the accompanying plot of cake moisture versus filter area illustrate that an accurate prediction of filter requirements is possible.

The filtration characteristics plot of cake moisture versus drying stage time divided by cake loading (θ_d/w) is standard for filtration testing (3). For the miniplant study, the curves were calculated from the individual plots of cake moisture versus belt distance (e.g., fig. E-1), the belt speed, and the residue solids feed rate. Results were averaged so that a single curve was provided for each major test condition.

The filtration characteristic for washing rate (3) is developed from bench-scale data as a plot of washing time (minutes) versus the product of cake loading (pounds solids per square foot) times wash addition volume per area (gallons per square foot) and is usually a straight line. Data for developing these plots from miniplant data were calculated from curves in the individual plots of cake moisture versus belt distance (fig. E-1) and the wash addition volume (gallons) for each type of feed material and for the first, second, or third washing stage. The best curve fit for the washing-rate plots were straight lines; therefore, the curves can be replaced by a factor equal to the slope of the straight line plots. The factor can be calculated without plotting by dividing the washing time (minutes) by the product of the cake loading (pounds solids per square foot) times the wash addition volume per area (gallons per square foot). An average of these values is the washing factor. Washing area requirements are calculated by multiplying the washing factor times the cake loading (pounds per square foot) and the wash addition rate (gallons per minute).

APPENDIX F.--DETAILED RESULTS OF MINIPLANT TESTS

ONE-FILTER CIRCUIT

Nine tests were conducted using a 10-1/2- by 1-ft filter with two conventional countercurrent washes, and six tests were conducted in which part of the wash filtrate from the same wash was recycled to flood the washing area (fig. 4). Operating conditions that were standard for the 15 tests are presented in table 2; conditions that were varied in the study are presented in table F-1. Four leaching feeds were tested, and wash dilutions ranged from 1.6 to 3.1 displacements. Because both sides of the cloth are washed on a horizontal belt filter, blinding of the filter cloth was not observed. Operating problems that decreased overall wash efficiency slightly were intermittent unevenness of cake bedding caused by uneven application of wash addition, and occasional plugging of some holes in wash headers. A summary of the filtration performance for the 15 tests is presented in table F-2.

TABLE F-1. - Operating variables for one filter

Calcined kaolin feed size, mesh	Test[1]	Filter feed, pct: Minus 150 mesh (Tyler) standard sieve)	Filter feed, pct: Solids	Flocculant addition, lb Separan MGL/ton solids	Cake washing: washing displacements (N), average per stage
Unbaffled reactors:					
Minus 10............	3	14.2	9.5	0.55	3.1
Do................	2	14.7	10.3	.56	2.2
Do................	1	14.9	11.0	.53	2.1
Do................	[2]14	18.0	11.6	.68	2.3
Minus 18 misted.....	12	7.9	10.5	.58	2.7
Do................	[2]13	7.5	10.6	.58	2.7
Minus 20............	10	20.6	10.3	NA	2.3
Do................	[2,3]11	13.6	10.1	NA	2.4
Baffled reactors:					
Minus 10............	15	11.4	10.4	.52	2.3
Do................	16	11.4	10.6	.13	2.5
Do................	17	10.4	10.6	.52	[1]2.2
Do................	18	10.4	11.3	.51	[1]1.6
Minus 18 misted.....	19	9.8	10.7	.26	[1]2.8
Do................	20	9.8	11.2	.26	[1]2.0
Minus 35 spray-dried	21	9.6	10.8	0	[1]2.8
Do................	22	9.6	11.2	0	[1]1.8

NA Not available.

[1]Tests 17-22 flooded washing (fig. 5), all others unflooded washing.

[2]1st filter only of a 2-filter test.

[3]Calcined kaolin feed for the first half of test 11 was calcined in a rotary kiln; that for the second half was calcined in a direct, coal-fired fluo-solids reactor. The only accompanying change noted in filtration performance was that the filter cake color abruptly changed from light to dark. Measurements and analyses used for calculation of filtration rate, washing efficiency, and aluminum recovery were nearly identical for both halves of the test.

TABLE F-2. - Critical measurements and responses using one filter[1]

Test	Washing efficiency, pct (av/stage)	Pregnant liquor product			Final filter cake			Filtration rate, $lb/h \cdot ft^{-2}$	
		Solids, ppm	Al_2O_3, pct	Al_2O_3 recovery, pct	Moisture, pct	Soluble Al_2O_3, pct	Al_2O_3 loss, pct	Forming	Full-scale[2]
1	34	203	7.7	94.3	48.2	3.6	5.7	NA	<21
2	32	350	8.4	93.9	50.1	4.1	6.1	190-350	49
3	33	NA	7.3	94.5	51.0	3.4	5.5	190	<19
10	44	497	7.7	96.1	47.3	2.7	3.9	[3]200	39
11[4]	46	421	NAp	NAp	47.5	NAp	NAp	[3]200	39
12	39	191	7.9	96.1	43.4	3.1	3.9	490	88
13[4]	41	181	NAp	NAp	40.8	NAp	NAp	490	88
14[4]	22	241	NAp	NAp	50.1	NAp	NAp	330	28
15	28	157	8.1	93.6	48.4	4.4	6.4	970	60
16	41	490	8.1	96.1	43.9	3.1	3.9	220	64
17	31	132	8.1	94.1	47.7	4.1	5.9	1,330	73
18	34	171	8.5	93.9	47.9	4.6	6.1	1,160	84
19	43	194	8.1	96.7	41.6	2.9	3.3	610	96
20	51	217	8.9	97.1	42.3	2.5	2.9	740	93
21	63	449	8.6	98.1	44.6	1.5	1.9	1,280	71
22	48	450	8.8	96.3	45.0	2.8	3.7	1,810	75

NA Not available.
NAp Not applicable.
[1]Constant operating conditions are given in table 3 and variable conditions in table F-1.
[2]Assumes forming to 61 pct moisture, 1st wash to 60 pct, 2d wash to 59 pct, and drying to 50 pct.
[3]Flocculant addition not known.
[4]1st filter only of a 2-filter test.

Three of the 15 tests (tests 11, 13-14) were run using the two-filter circuit. Response data for the first filter on washing efficiency, cake moisture, and filtration rate is valid for a one-filter test, and is included in table F-2.

Data obtained from these one-filter tests are--

1. Effect of flocculant addition on form filtration rate is plotted in figures 9, 10, and 11 for minus 10-mesh, minus 18-mesh, and minus 35-mesh leaching feeds, respectively.

2. Factors for calculating washing area required for any cake loading (pounds solids per square foot) and wash addition rate (gallons per minute) are listed in table F-3.

TABLE F-3. - Factors from miniplant data for calculating washing-area requirements

Calcined kaolin feed size, mesh	Multiplication factor[1] $\left(\frac{min \cdot ft^4}{lb \cdot gal}\right)$		
	1st wash	2d wash	3d wash[2]
Unbaffled leaching reactors:			
Minus 10 baseline.........	0.55	0.37	0.34
Minus 18 misted...........	.29	.18	.22
Minus 20 prethickened.....	.39	.31	.51
Baffled leaching reactors:			
Minus 10 baseline.........	.34	.26	NR
Minus 18 misted...........	.29	.21	NR
Minus 35 spray-dried......	.46	.20	NR

NR Not run.

[1]To obtain washing area requirements (ft^2), multiply factor by the product of cake loading (lb solids/ft^2) times wash addition rate (gal/min). See appendix E.

[2]On 2d filter.

3. Effect of drying cycle time divided by cake loading (Θ_d/w) on final cake moisture content is plotted in figure F-1.

4. Effect of final cake moisture content on the FSFR is plotted in figure F-2.

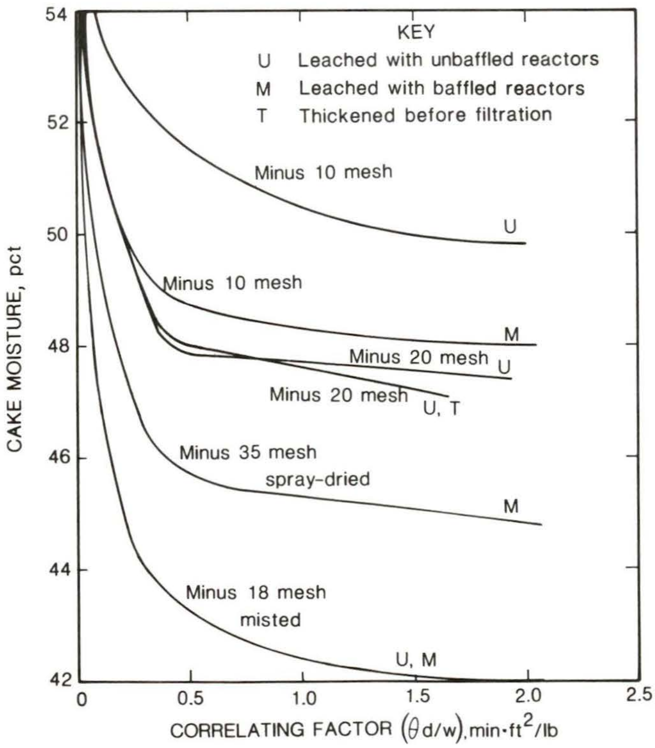

FIGURE F-1. - Correlating factor, θ_d/w, versus cake moisture using one filter.

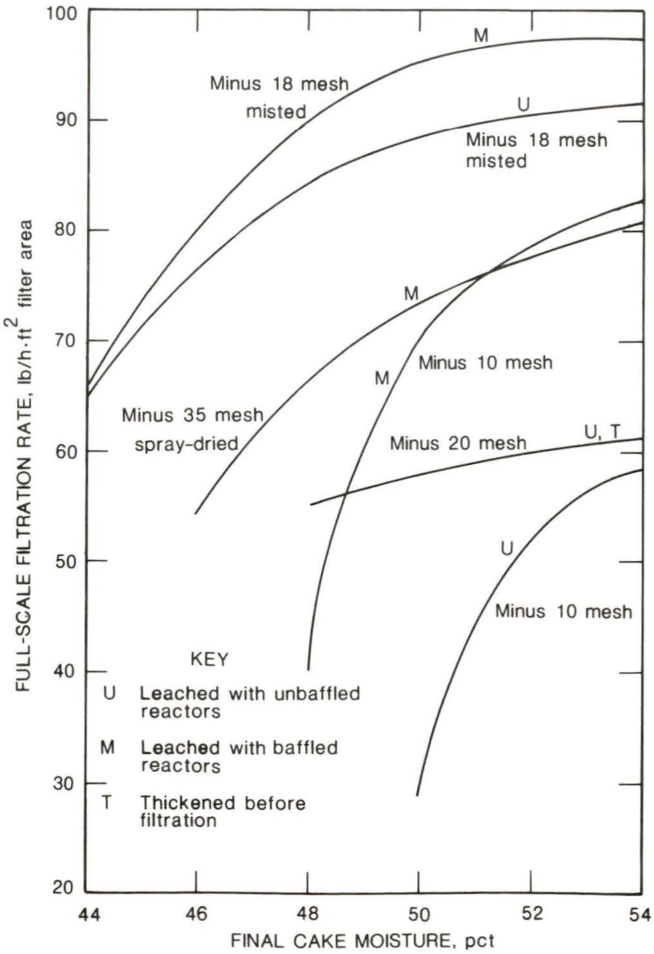

FIGURE F-2. - Full-scale filtration rate versus final cake percent moisture using one filter.

TWO-FILTER CIRCUIT

Three tests were conducted using two filters in series. The first filter had two conventional countercurrent washes, but the second filter was preceded by a repulper and had only one countercurrent wash (fig. 5). Constant operating conditions are presented in table 3. The variables were flocculant and wash water addition and nominal size of leaching feed (table F-4; filtration performance in table F-5). The following data were obtained

1. Effect of flocculant addition on form filtration rate is plotted in figures 9-11.

2. Factors for calculating washing area are listed in table F-3.

3. Effect of Θ_d/w on final cake moisture content is plotted in figure F-3.

4. Effect of final cake moisture on FSFR is plotted in figure F-4.

The only operating problem attributable to the additional filter was that about 10 pct of the cake fed to the first filter stuck to the cloth; it was removed by the belt wash. This loss can be eliminated by an air jet at the cake-discharge point.

THICKENER-AND-FILTER CIRCUIT

Five tests were conducted using a thickener followed by a filter with two countercurrent washes (fig. 7), and one test was conducted using the same circuit with the addition of a repulper and second filter in series (fig. 8). Flocculant addition to the thickener was 0.55 lb Separan MGL per ton solids. Operating conditions are listed in table 2. Tests were limited to minus 20-mesh calcined kaolin because the minus 10-mesh material was too coarse for the thickener underflow pumping system, and the inventories of minus 18-mesh misted and minus 35-mesh spray-dried calcined kaolin were inadequate. In addition to determining the effect of prethickening on filter

TABLE F-4. - Operating variables for two filters

Calcined kaolin feed size, mesh	Test	First filter feed, pct: Minus 150 mesh (Tyler standard sieve)	First filter feed, pct: Solids	2d filter feed, pct solids	Flocculant addition, lb Separan MGL/ton solids	Cake washing: washing displacements (N), av/stage
Minus 10........	14	18.0	11.6	21.8	0.68	2.4
Minus 18 misted.	13	7.5	10.6	25.6	.58	2.7
Minus 20........	11	13.6	10.1	22.9	NA	2.5

NA Not available.

TABLE F-5. - Critical measurements and responses using two filters[1]

Test	Washing efficiency, pct (av/stage)	Pregnant liquor product: Solids, ppm	Pregnant liquor product: Al_2O_3, pct	Pregnant liquor product: Al_2O_3 recovery, pct	Final filter cake, pct: Moisture	Final filter cake, pct: Soluble Al_2O_3	Final filter cake, pct: Al_2O_3 loss	Filtration rate, $lb/h \cdot ft^{-2}$: Forming	Filtration rate, $lb/h \cdot ft^{-2}$: Full-scale[2]
11	47	421	8.0	99.3	45.5	0.6	0.7	[3]200	[3]20
13	49	149	8.2	99.6	43.9	.4	.4	490	59
14	33	241	8.2	98.9	45.2	1.0	1.2	330	42

[1]Constant operating conditions are given in table 3, and variable operating conditions in table F-4.

[2]Assumes forming to 61 pct moisture, 1st wash to 60 pct, 2d wash to 59 pct, form to 58 pct, 3d wash to 57 pct, and drying to 50 pct.

[3]Flocculant addition not known.

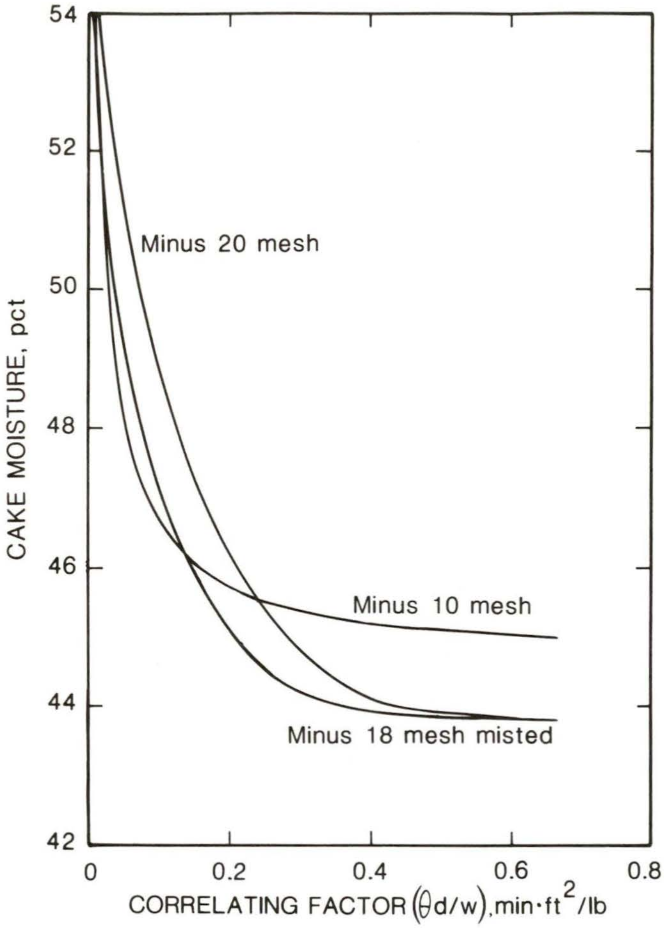

FIGURE F-3. - Correlating factor, θ_d/w, versus final cake moisture using two filters.

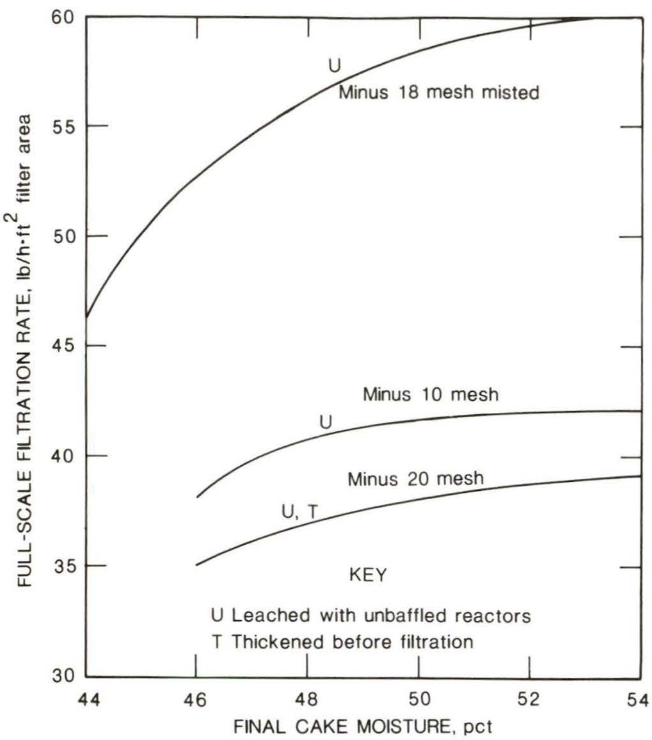

FIGURE F-4. - Full-scale filtration rate versus final cake percent moisture using two filters.

performance, the tests were used to evaluate the effect of filter feed rate. Thickener underflow feed rates to the filter were varied from 100 to 310 lb/h of residue solids. The effect of cake-washing volume over a range of 1.4 to 4.2 displacements was also evaluated. The variables investigated are listed in table F-6.

Two startup methods were used:

1. In tests 4 through 8, leaching slurry was fed to the thickener for about 7 hours until several feet of settled pulp accumulated; then the underflow pump was started and filtration begun.

TABLE F-6. - Operating variables using thickener and filter

Test	Thickener feed solids, pct minus 150 mesh (Tyler standard sieve)	Thickener underflow slurry, pct solids	Final filter feed slurry		Flocculant addition to filter, lb Separan MGL/ton solids	Cake washing: washing displacement (N), av/stage
			Solids, lb/h	Solids, pct		
7	19.9	16.0	143	9.9	0.41	4.2
6	21.0	20.4	171	11.3	.24	3.9
8	21.8	21.9	181	13.9	.32	2.8
4	18.4	24.3	100	15.0	.25	2.5
5	16.1	21.6	310	16.4	.08	1.4
9	20.0	23.1	114	15.1	.51	[1]2.6, 2.7

[1]Test 9 was a 2-filter test; displacement was 2.6 for the 1st filter and 2.7 for the 2d filter.

2. In test 9,[1] the underflow pumps were started immediately at a low rate with the discharge routed to waste. After 4 h, the pumps were set at the test rate and the discharge pumped to the filter.

Only the second technique (test 9) provided a steady flow of feed to the filter. Results from tests 4 through 8 are reported because they provide an approximate evaluation of the effect of feed rate and wash addition on filtration.

Filtration performance is summarized in table F-7. Material balances were calculated with the rate of the leaching slurry fed to the thickener changed to provide the same pounds per hour of dry solids as actually fed to the filter. The thickener underflow and overflow rates were modified proportional to this change, but the filter flow rates were not changed. Form filtration rates and the FSFR's averaged 500 and 55 lb solids/h$\cdot$ft^{-2} of area, respectively. Data obtained from tests using thickening before horizontal belt filtration were as follows:

1. Effect of flocculant addition on form filtration rate (plotted in figures 9-11).

2. Factors for calculating washing area (table F-3).

3. Effect of Θ_d/w on final cake moisture content (figs. F-1 and F-3).

4. Effect of final cake moisture on FSFR (figs. F-2 and F-4).

[1] Test 9 was run using 2 filters; 1st-filter data on washing efficiency, cake moisture, and filtration rate are valid for a 1-filter test. Tables F-6 and F-7 present test 9 data in both 1- and 2-filter form.

WASHING EFFICIENCY

Data on washing efficiency in the miniplant testing is contained in tables F-2, F-5, and F-7 and summarized in table F-8. Washing efficiency in the first filter averaged 42 pct for both the first and second stages. The washing efficiency in the second filter (third wash) was significantly higher than in the first filter. In four tests, the third wash averaged 55 pct efficiency and the first two washes averaged 41 pct. Data comparing the average washing efficiency for the different feed materials in the first filter to those in the second filter are listed in table 4.

TABLE F-7. - Critical measurements and responses using thickener and filter[1]

Test[2]	Washing efficiency, pct (av/stage)	Pregnant liquor product: Solids, ppm	Pregnant liquor product: Al_2O_3, pct	Pregnant liquor product: Al_2O_3 recovery, pct	Final filter cake, pct: Moisture	Final filter cake, pct: Soluble Al_2O_3	Final filter cake, pct: Al_2O_3 loss	Filtration rate, lb/h$\cdot$ft^{-2}: Forming	Filtration rate, lb/h$\cdot$ft^{-2}: Full-scale[3]
4	53	148	7.6	98.1	49.4	1.2	1.9	800	56
5	50	NA	8.9	96.5	47.0	2.4	2.4	410	119
6	51	NA	6.9	98.8	48.8	.8	1.2	460	49
7	63	NA	6.9	99.1	47.2	.6	.9	380	46
8	33	NA	7.9	97.2	46.5	2.1	2.8	480	57
9[4]	52	133	NAp	NAp	43.8	NAp	NAp	680	58
9	53	133	8.1	99.7	43.9	.3	.3	680	38

NA Not available.
NAp Not applicable.

[1] Constant operating conditions are given in table 3 and variable operating conditions in table F-6.

[2] Test 9 was run using 2 filters; 1st-filter data on washing efficiency, cake moisture, and filtration rate are valid for a 1-filter test.

[3] Assumes forming to 61 pct moisture, 1st wash to 60 pct, 2d wash to 59 pct, and drying to 50 pct; on 2d filter forming to 58 pct moisture and washing to 57 pct.

[4] 1st filter only of a 2-filter test.

TABLE F-8. - Relationships between washing efficiency and wash addition (N) in bench-scale and miniplant testing

Calcined kaolin feed size, mesh	Washing displacements (N)	Washing efficiency, pct	
		Miniplant[1]	Bench-scale[2]
Unflooded wash:			
Minus 10............	2.1	34	46
	2.2	32	47
	2.3	28	48
	3.1	33	50
Minus 20 thickened..	1.4	50	56
	2.5	53	68
	2.8	33	69
	3.9	51	70
	4.2	63	70
Flooded wash:			
Minus 10............	1.6	34	41
	2.2	31	47
Minus 18 misted.....	2.0	51	64
	2.8	43	69
Minus 35 spray-dried	1.8	48	60
	2.2	63	65

[1]From tables F-2 and F-7.
[2]Estimated from curves in figure B-5.

The flocculant had no significant effect on washing efficiency except to speed up or slow down the filtration process. In the filtration of minus 10-mesh material, a large decrease of the flocculant level in test 15 compared with test 16 (tables F-1 and F-2) caused a large decrease in form filtration rate. Washing efficiency increased from 28 to 41 pct. In similar testing on minus 35-mesh spray-dried material, a large increase in flocculant (test 22 run with no flocculant and a special short test run with 0.5 lb/ton flocculant) caused a large decrease in form rate, but washing efficiency increased from 48 to 54 pct. Decreasing the filtration rate increases washing efficiency because of slower fluid flow through the filter cake. In one case, lowering the flocculant addition decreased the filtration rate and increased washing efficiency; in the other case, raising the flocculant addition decreased the filtration rate and increased washing efficiency. Prethickening has little effect on filter washing efficiency (table 4).

PREGNANT LIQUOR CLARITY

Solids content in pregnant liquor filtrate is given for each test in tables F-2, F-5, and F-7. Decreasing flocculant addition to less than that needed for rapid filtration markedly increased solids contamination in the form filtrate (tests 15-16, tables F-1 and F-2). In these tests, a decrease in flocculant from 0.5 to 0.1 lb/ton solids caused an increase in the form filtrate solids from 160 ppm to 490 ppm.

MOISTURE REMOVAL FROM FILTER CAKE

For any of the test conditions, the cake moisture could be decreased to 50 pct or less in the drying stage, which followed cake washing. The effect of test conditions on moisture removal is summarized in the cake moisture versus Θ_d/w plots presented in figures F-1 and F-3, which show the following:

1. Cake moisture removal in a one-filter circuit was fastest (i.e., filter

drying area requirements were smallest) for minus 18-mesh misted residues followed by minus 35 mesh spray-dried, minus 20 mesh, and minus 10 mesh.

2. Cake moisture removal in a two-filter circuit was the same for all materials.

3. After 0.5 min, or a Θ_d/w of 0.35, rate of moisture removal decreased appreciably, indicating that 0.5 min was the best drying time.

4. Prethickening had little effect on moisture removal in the drying stage.

5. With minus 10-mesh feed, when baffled reactors were used to decrease particle degradation in leaching, moisture removal in the filter drying stage increased.

6. Cake loading averaged 1.31 lb solids/ft^2 and cake thickness 0.41 in for a volumetric density of 38.3 lb solids/ft^3, which is 4 pct more volume than the as-mined kaolin.[1]

FILTRATION RATES

Forming filtration rates for individual tests are listed in tables F-2, F-5, and F-7, and are correlated to flocculant addition in figures 9-11. Both overflocculation and underflocculation decreased the forming rate. When fully baffled reactors were used and 0.1 lb of Separan MGL per ton of solids was added to leached minus 18-mesh misted material, a maximum forming rate 900 lb/h·ft^{-2} was obtained. The maximum forming rate for the minus 35-mesh spray-dried material (1,600 lb/h·ft^{-2}) was obtained at zero flocculant addition, but 0.3 lb/ton flocculant addition was required to obtain the maximum rate (1,000 lb/h·ft^{-2}) for minus 10-mesh material.

The washing factors provided in table B-1 for the bench-scale study II and in table F-3 for the miniplant study are proportional to the washing area requirements and inversely proportional to the rate of washing. Tables B-2 and F-2 indicate that washing factors and form filtration rates are inversely proportional. Forming and washing rates are proportional and have the same relationship to flocculant addition, type of feed, and amount of fines. Washing-area requirements were not increased when the "flooded" washing circuit (fig. 5) was used. By recycling part of the first and second wash filtrates back to their respective wash additions, the volume of wash going through the cake was doubled, but this composite liquor pulled through the cake twice as fast so no more washing area was required than that indicated for the normal circuit.

FSFR's are dependent on the forming, washing, and drying stage rates. In the miniplant study, the major factors influencing the FSFR, as illustrated in figures F-2 and F-4 were as follows:

1. Type of feed.

2. Amount of flocculant addition.

3. Number of filters and stages of washing.

4. Final cake moisture.

The figures indicated a 30- to 60-pct increase in filtration rate when the final cake moisture was increased from between 44 and 48 pct to 50 and 52 pct. The minus 18-mesh material attains the highest FSFR and the addition of a second filter decreased the FSFR by up to 50 pct.

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[1]This is calculated from (1) clay-HCl miniplant data indicating that the leach residue solids represent 61 pct of the original calcined clay, (2) data from Doss White, former Georgia State Liaison officer, Bureau of Mines, indicating that "in-place" kaolin density is 95 lb/ft^3 and free H_2O content of in-place kaolin is 18 to 22 pct, and (3) combined H_2O content of dry kaolin is 14 pct (1).

ESTIMATES OF BEST FILTRATION CONDITIONS AND PERFORMANCE

Final product analyses, alumina distribution, and corresponding process requirements are presented in tables 5 and F-9 for each type of calcined kaolin. The four materials rank as follows, in terms of desirable filtration characteristics:

Filtration characteristics	Nominal size, mesh
Highest filtration rate................	Minus 10, minus 18 misted.
Lowest final cake moisture.............	Minus 18 misted.
Lowest final cake liquor Al_2O_3 analyses	Minus 18 misted, minus 35 spray-dried.
Highest final liquor Al_2O_3 analyses....	Minus 35 spray-dried.
Highest average washing efficiency.....	Do.
Highest filtrate Al_2O_3 recovery........	Minus 18 misted, minus 35 spray-dried.
Lowest flocculant requirements.........	Do.

These characteristics are interrelated. Washing efficiency and final cake moisture influence the final liquor and cake analyses and the alumina recovery.

TABLE F-9. - Predicted best results for belt filtration[1]

Slurry feed and number of filters	Flocculant addition, lb Separan MGL/ton solids	Pregnant liquor product: Solids, ppm	Pregnant liquor product: Al_2O_3, pct	Pregnant liquor product: Al_2O_3 recovery, pct	Final filter cake, pct: Moisture,[2]	Final filter cake, pct: Soluble Al_2O_3	Final filter cake, pct: Al_2O_3 loss	FSFR, $lb/h \cdot ft^{-2}$
MINUS 10 MESH								
Unthickened:[3]								
1..........	0.27	150	8.6	93.6	49	4.2	6.4	63
2..........	.27	240	8.8	98.4	45	1.2	1.6	53
Thickened:[4]								
1..........	.73	150	8.8	96.8	49	2.1	3.2	63
2..........	.73	130	8.9	99.2	45	.6	.8	53
MINUS 20 MESH								
Unthickened:[3]								
1..........	0.25	ND	8.6	95.3	48	3.2	4.7	59
2..........	.25	ND	8.8	98.6	44	1.1	1.4	45
Thickened:[4]								
1..........	.80	150	8.9	97.7	48	1.6	2.3	59
2..........	.80	130	8.8	99.3	44	.5	.7	45
MINUS 35 MESH SPRAY DRIED								
Unthickened:[3]								
1..........	0	450	8.7	96.1	46	2.9	3.9	63
2..........	0	450	9.0	98.9	46	.8	1.1	50
Thickened:[4]								
1..........	ND	150	8.9	98.0	46	1.5	2.0	63
2..........	ND	130	9.0	99.5	46	.4	.5	50

ND Not determined.

[1]Assuming a slurry from acid leaching of calcined kaolin contains 10.0 pct soluble Al_2O_3 and 13.8 pct solids, a total wash addition of 1.5 lb fresh water per pound residue solids, and 0.375 ft^2 baffle area between washing stages. Results for the minus 18-mesh material are given in table 5.

[2]Lowest practical drying time, $\Theta_d/w = 0.5$ (figs. F-1 and F-3).

[3]Summarized from material balances made from the actual tests.

[4]Estimated in part from testing on minus 20-mesh material in which filter feed was prethickened.