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RHEOLOGICAL PROPERTIES OF KAOLIN AND CHEMICALLY SIMULATED WASTE

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ABSTRACT

The Savannah River Laboratory is conducting tests to determine the best operating conditions of pumps used to transfer insoluble radioactive sludges from old to new waste tanks. Because it is not feasible to conduct these tests with real or chemically simulated sludges, kaolin clay is being used as a stand-in for the solid waste. The rheology tests described herein were conducted to determine whether the properties of kaolin were sufficiently similar to those of real sludge to permit meaningful pump tests.

The rheology study showed that kaolin can be substituted for real waste to accurately determine pump performance. Once adequately sheared, kaolin properties were found to remain constant. Test results determined that kaolin should not be allowed to settle more than two weeks between pump tests. Water or supernate from the waste tanks can be used to dilute sludge on an equal volume basis because they identically affect the rheological properties of sludge. It was further found that the fluid properties of kaolin and waste are insensitive to temperature.

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RHEOLOGICAL PROPERTIES OF KAOLIN AND CHEMICALLY SIMULATED WASTE

INTRODUCTION

Radioactive waste from Savannah River Plant (SRP) separation processes has been stored in steel waste tanks since plant startup. Due to the age and design and fabrication technologies of these original tanks, SRP is transferring the waste from 23 of these tanks to new stress-relieved tanks for interim storage prior to immobilization and final storage in underground repositories.

The waste is in two basic forms, soluble salt and insoluble sludge. The salts are retrieved from the old waste tanks by dissolving and reconcentrating prior to transfer into new tanks. The sludge phase, a thick gelatinous substance, is slurried (suspended) with long-shaft Bingham™ (Bingham Willamette Co.) pumps, then transferred with conventional liquid transfer pumps.

Sludge is mobilized most efficiently when water or supernate addition is minimized and when the number of pumps is minimized and optimally placed for sludge dispersion. To determine the most efficient conditions, pumps are being tested at the Savannah River Laboratory (SRL) Semiworks in a full-scale mockup tank (Figure 1) and in a 1/12th-scale tank using simulated sludge or kaolin clay (NATKA, National Kaolin Products Co., Aiken, SC) in water slurry.

For the test operations to be meaningful, kaolin and chemical simulants must be rheologically similar to real wastes. To demonstrate this similarity, SRL obtained two Haake Model RV-3 (Haake, Inc.) rotoviscometers (Figure 2), one for operation at SRL Semiworks with kaolin and chemical simulants, and one for operation in the high level caves (HLC) with real waste. The combination of rheological results from the Semiworks and the HLC tests permits formulation of chemically simulated or kaolin sludges with properties duplicating the real wastes. These sludges are used in pump tests so that the slurrying efficiencies and other waste-transfer operations can be determined, permitting accurate assessment of sludge removal and equipment performance.

Although the focus of this report is on kaolin and simulant used to predict tank-cleaning performance, SRL rheological studies are also being conducted in support of sludge washing, intertank and interarea sludge and slurry transfer, and the Defense Waste Processing Facility (DWPF).[1]

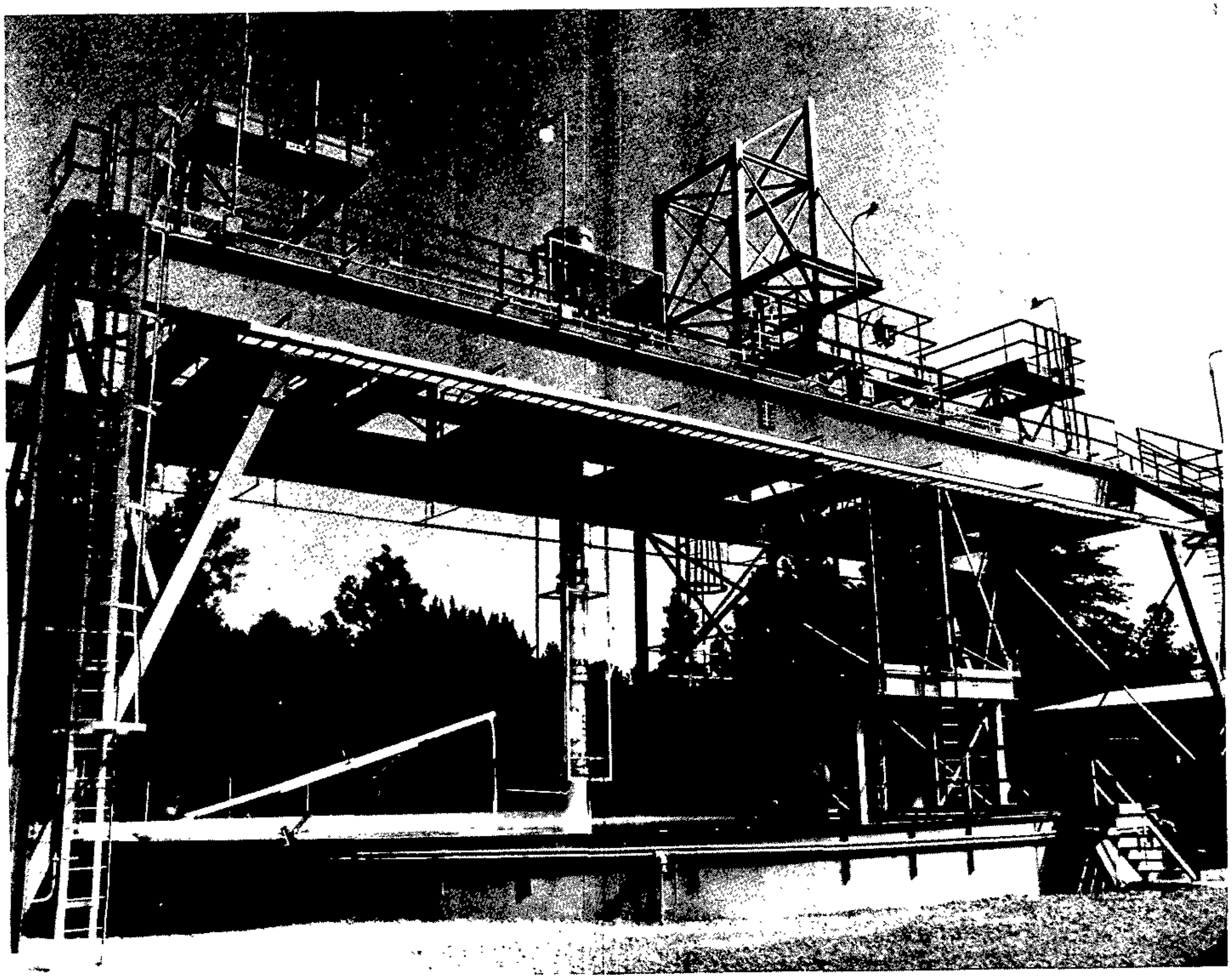


FIGURE 1. Full-Scale Mockup Tank

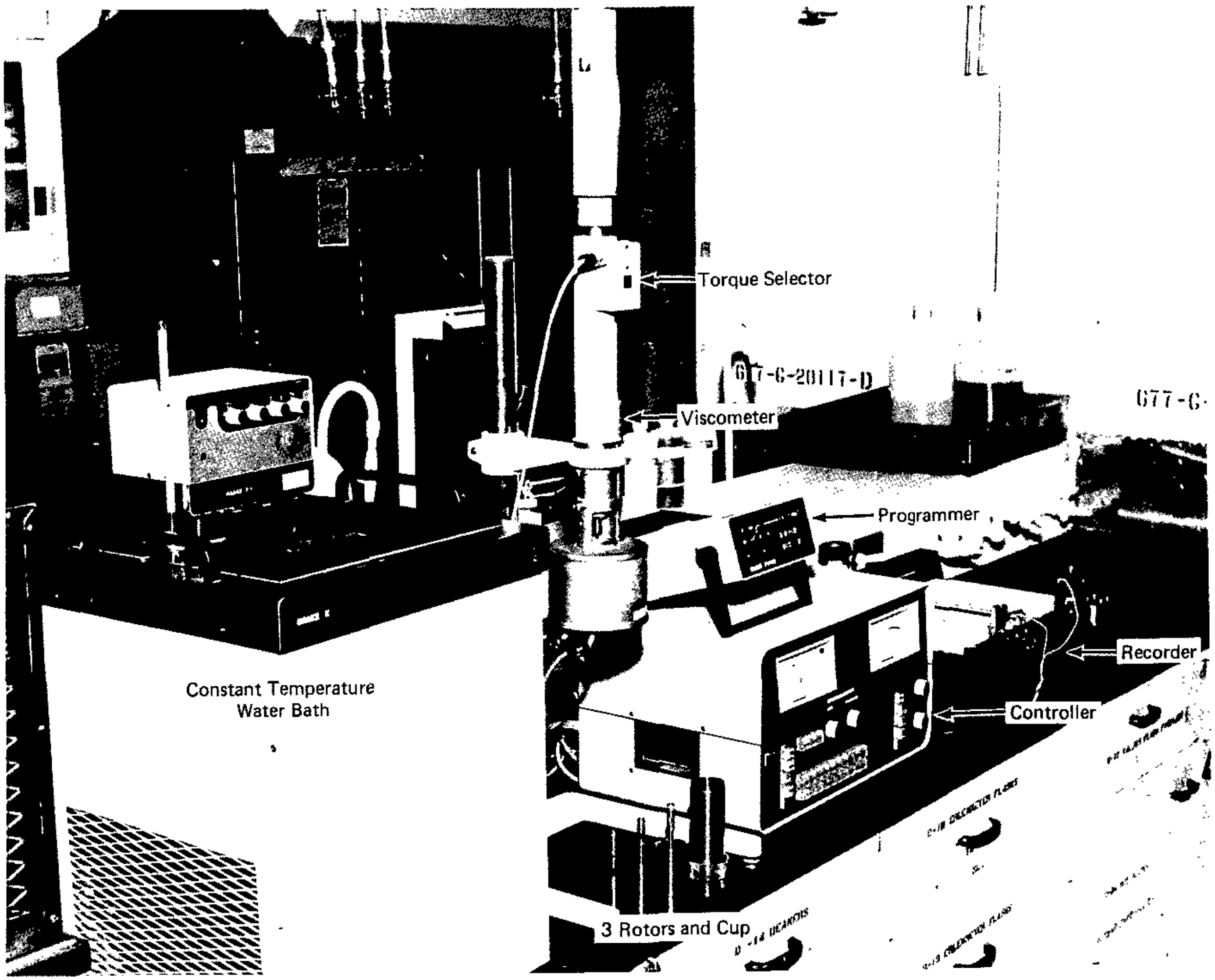


FIGURE 2. Haake Rotoviscometer and Support Components

SLUDGE RHEOLOGY PROGRAM

Viscosity Theory

As mentioned, two Haake rotoviscometer systems were purchased. The viscometer consists of a rotor which rotates inside of a cup containing the test fluid. Four rotor sizes are provided, plus two torque adjustments in the drive mechanism (one ten times the other) to permit analysis of a wide range of viscosities. The torque or shear stress required to rotate the rotor against the drag of the fluid in the cup is continuously recorded while the rotation or shear rate is slowly increased from zero to a predetermined value, held at the maximum value, and slowly returned to zero.

The maximum shear stress used was 300 sec^{-1}. Although kaolin slurry exits the pump at 1600 sec^{-1}, its shear stress declines with distance due to encounters with stagnant or returning slurry. The range of zero to 300 sec^{-1} covers the shear stresses which exist at the sludge-slurry interface during the final stages of slurrying where the effective cleaning radius is established. This range also includes the shear stresses likely to be encountered in a pipe.

From this shear rate vs. shear stress curve, a yield stress is conservatively (over) estimated by extrapolating a line tangent to the high shear rate portion of the curve back to shear rate zero (Figures 3 and 4). The slope of this tangent line is the consistency.[2]

For a known fluid flow or fluid shear rate, the yield point and shear rate are combined into a single value, the apparent viscosity.[3] This is the slope of a line from the rheology curve back to origin. The slope of this line is the viscosity of a Newtonian fluid which would produce an identical amount of fluid motion at the same pump torque.

Rheological analysis demonstrated that kaolin ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) and simulated waste (material having the same elemental composition as real waste) are similar Bingham plastics (Figures 3 and 4). This means that large increases in pump torque are required to produce small increases in fluid motion until a yield point is reached. Thereafter, small increases in torque produce large increases in fluid motion. Above the yield stress, the shear stress vs. shear rate behavior is much like a Newtonion fluid (motion proportional to torque) with a displaced origin.[4] The slope of the shear stress vs. shear rate curve is called the viscosity for Newtonian (ideal) fluids and the consistency for Bingham plastic fluids under applied stresses greater than the yield stress. Both viscosity and consistency are reported in centipoise.

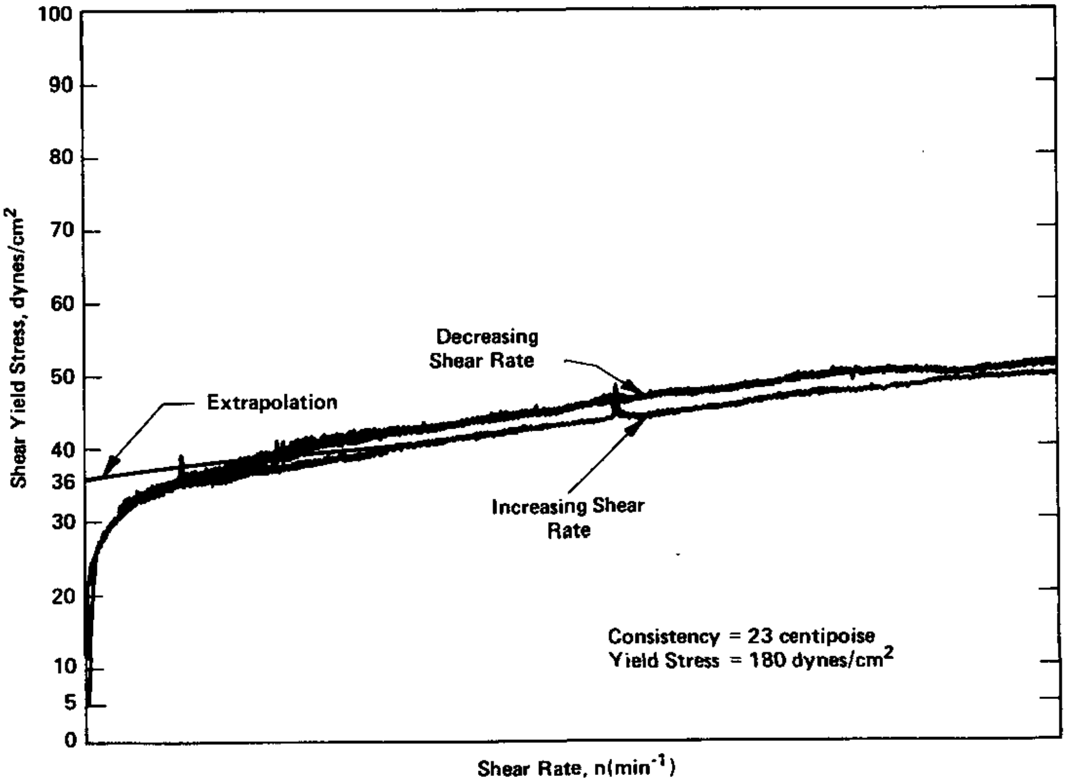


FIGURE 3. Rheology Curve for Kaolin-Water Slurry

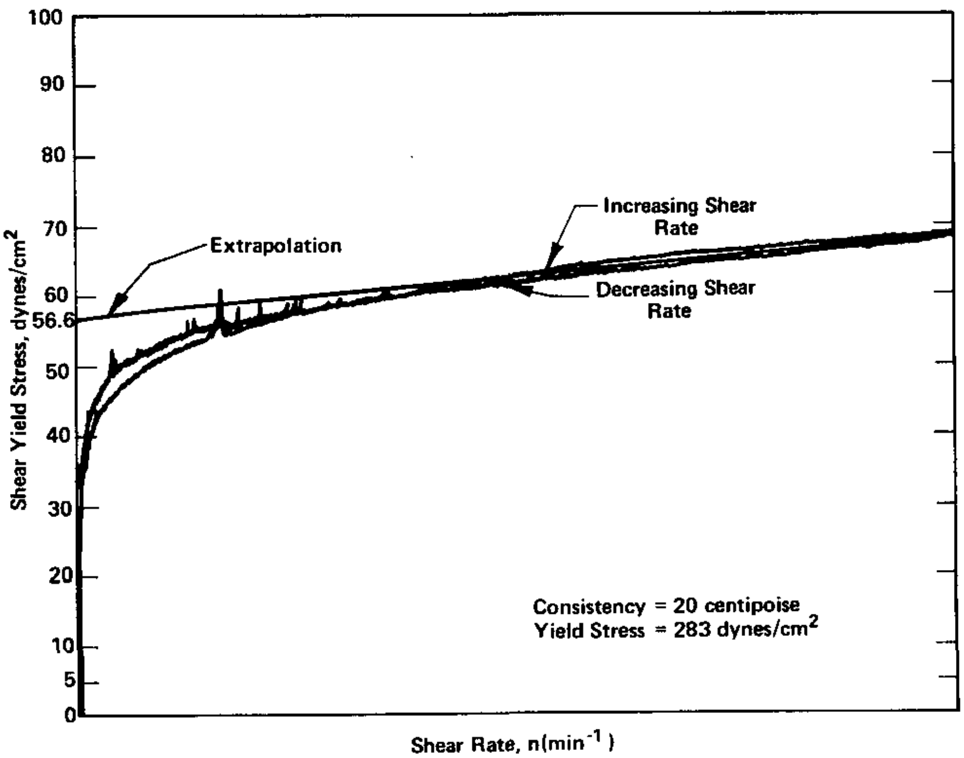


FIGURE 4. Rheology Curve for Simulated Waste

The yield stress results from the ability of kaolin to become electrostatically charged. Kaolin platelets are hexagonal and thin in comparison with length and breadth. The body of a platelet becomes negatively charged and the edges positively charged. When the platelets are still, they stick to each other, edge to center, forming a "house of cards" structure.[3] This structure must be broken apart before the kaolin will flow. A constant force or torque must be supplied at all times to keep the platelets from becoming reattached, and additional torque must be supplied to increase the rate of fluid motion.

The ability to directly measure the shear yield stress is an advantage of the Haake rotoviscometer over the Brookfield viscometer (Brookfield Engineering Laboratories, Inc.). However, the Brookfield viscometer was used to measure viscosity in past programs including SRP Tank 16-H sludge removal.[5]

Yield stress was shown to be such an overwhelming effect compared with consistency (or its equivalent, viscosity) that yield stress alone was found to accurately predict the behavior of chemical sludge in piping systems. Without the yield stress determination, serious underestimates of pump requirements for slurry transfer operations result.

The reproducibility of the viscometer system for measuring kaolin, including variation in sample preparation, was determined as shown in Table 1.

This research on clay rheology was limited to kaolin because kaolin has a nonexpanding crystal lattice that is free flowing in water slurry. Bentonite clays have expanding crystal lattices that are colloidal and hydrate to produce plastic and gel-like substances.[6]

TABLE 1

Reproducibility of Viscometry

	Nominal	Standard Deviation
Yield Stress, dynes/cm^2	134	4.7
Consistency, centipoise	13	1.6

Rheology vs. Composition

The similar rheology of kaolin and chemically simulated waste means that kaolin can be effectively substituted for waste. This can be done by choosing an equivalent composition from Figure 5 or from the constitutive equations shown in Table 2. Then, the rheograms and tank performance of the two substances will be almost identical. The curves for chemically simulated waste in Figure 5 can be replaced by curves developed for real waste when real waste data are available. Then, the cleaning radius of a pump operating in real waste can be predicted from the operation of the pump in kaolin slurry with the same yield point and consistency. The consistency rises with solids content just as yield stress does (Figure 6).

Figure 5 displays the yield stress variation of fresh kaolin that has not been exposed to "shear" in the Bingham™ pumps. Kaolin slurry properties change rapidly during pumping. Once the shear values represented by the top curve are attained, the properties do not change further and do not revert when the fluid stops moving. The kaolin is then said to be shear-stabilized. This stabilization makes kaolin practical as a waste stand-in and a means for pump evaluation.

The yield stress value for fresh 30 wt % kaolin slurry (Figure 5) does not fall into line with the values for lower compositions. This is believed to occur because the higher composition platelets are crowded together causing shear to take place during the rheology test.

An alternate explanation for the break in the curve is that the concentration is so high that close packing of kaolin atoms prohibits normal rotation of particles that occurs during normal flow.[6] Above such a critical point, viscosity increases rapidly with further increases in particle concentration. This explanation is believed to be unlikely because such a break does not occur in sheared kaolin or the simulated sludges at equal compositions.

TABLE 2

Equations for Yield Stress in Terms of Solids Content

Substance	Constitutive Equation
Kaolin	ln (yield stress) = 0.16 (wt % kaolin) + 2.2
Unwashed Simulant	ln (yield stress) = 0.12 (wt % solids) + 1.5
Washed Simulant	ln (yield stress) = 0.17 (wt % solids) + 1.2

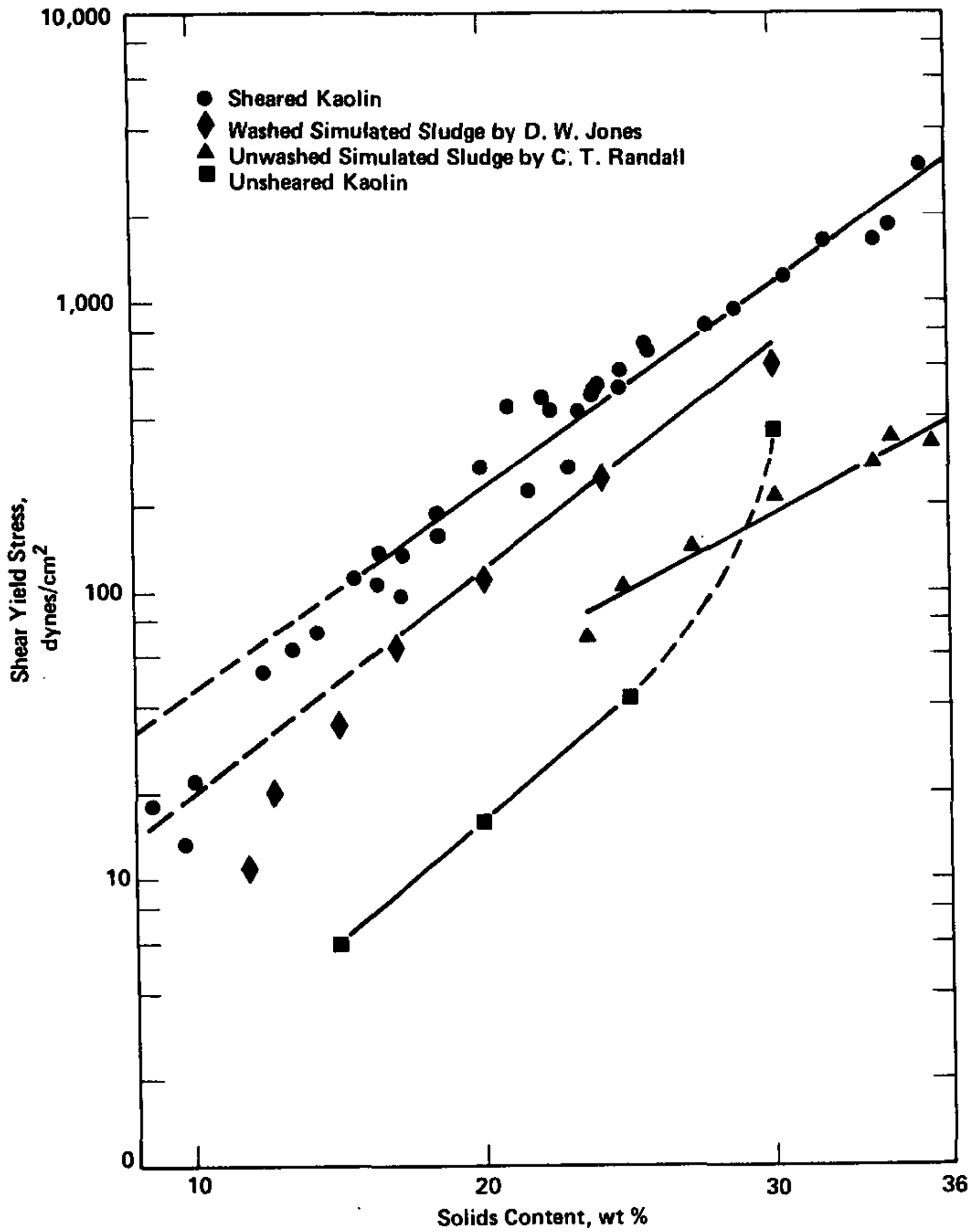


FIGURE 5. Rheology of Sludges

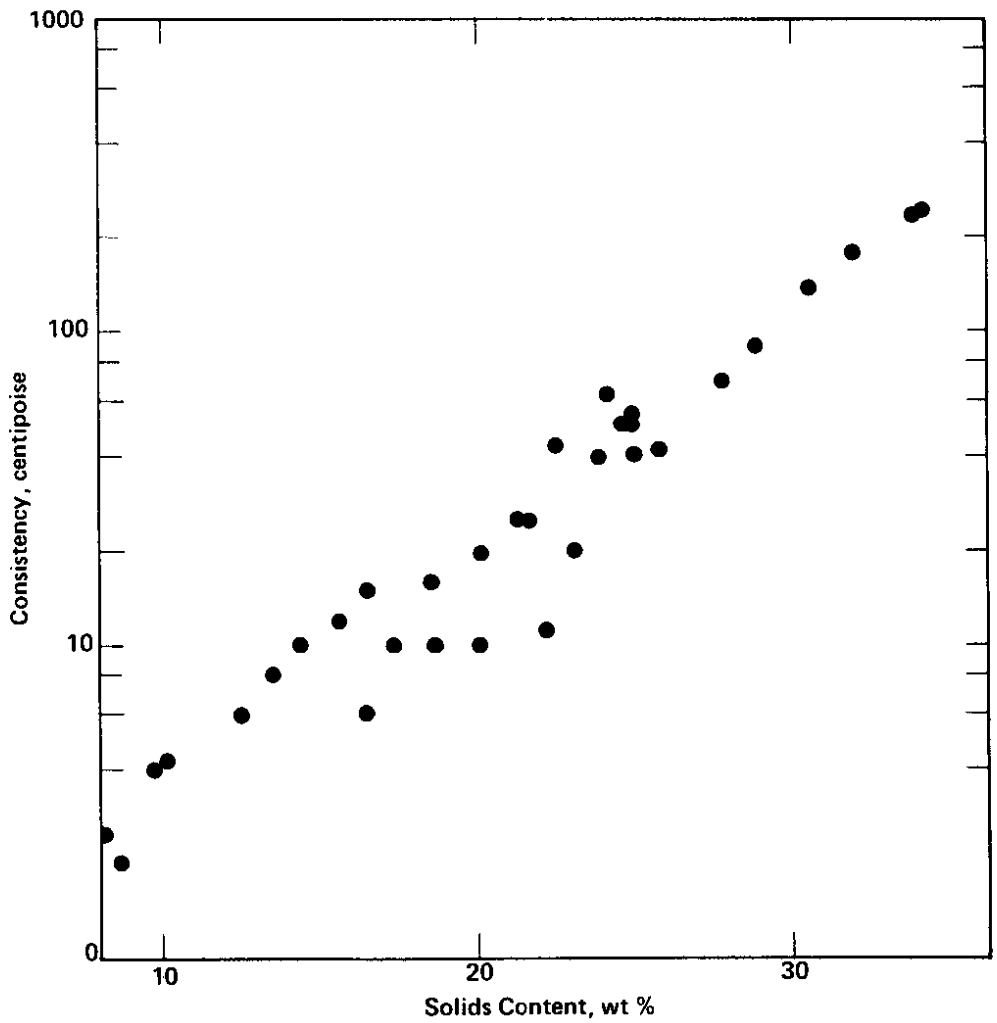


FIGURE 6. Rheology of Kaolin

Because kaolin properties change with shear, it is important that kaolin be fully sheared before it is used in pump tests. If the more fluid unsheared kaolin is used, predicted cleaning radii will be much greater than with fully sheared material. If shear continued during extended pump operation or changed significantly when higher shear pumps were used or when pumps were operated at higher speed, results would be inconsistent.

Attainment of shear stability occurred within three hours at a low shear rate of 714 sec^{-1} in a water-cooled Waring™ (Testing Machines, Inc.) blender (Figure 7). Shear rates at the Bingham™ pump discharge nozzles are estimated to be 1600 sec^{-1} when the standard sludge-removal pump (Table 3) is operated at full speed. However, the pumps shear the kaolin only intermittently, whereas the Waring™ blender exposes the kaolin to continuous shear. The shear yield stress of fresh kaolin, which was stirred for 22 hours with a Bingham™ pump in the full tank, allowed to settle to a sludge, and decanted, was 228 $dynes/cm^2$. The shear stress of a fully sheared sludge of the same composition would be expected to be 800 $dynes/cm^2$. The predicted yield stress was attained some time between 22 and 34 hours of pump operation. No further rheological change had occurred in samples taken after 46, 58, and 70 hours of pump operation. Hysteresis, which was recorded in the rheogram of the 22-hour sample due to shear occurring during analysis, was absent in the later samples.

TABLE 3

Slurry Pump Specifications

	Tank 16 Type Pump
Estimated Cleaning Radius, ft	25
Capacity, gpm	1200
DV (Momentum), inch-ft/sec	165
Nozzle Size, inches	1.5
Number of Nozzles	2
Pump Diameter, inches	22.5
Impeller Type	Radial
Number of Volutes	2
Speed, rpm	1760
Discharge Pressure, psi	87
Application	Sludge and Salt

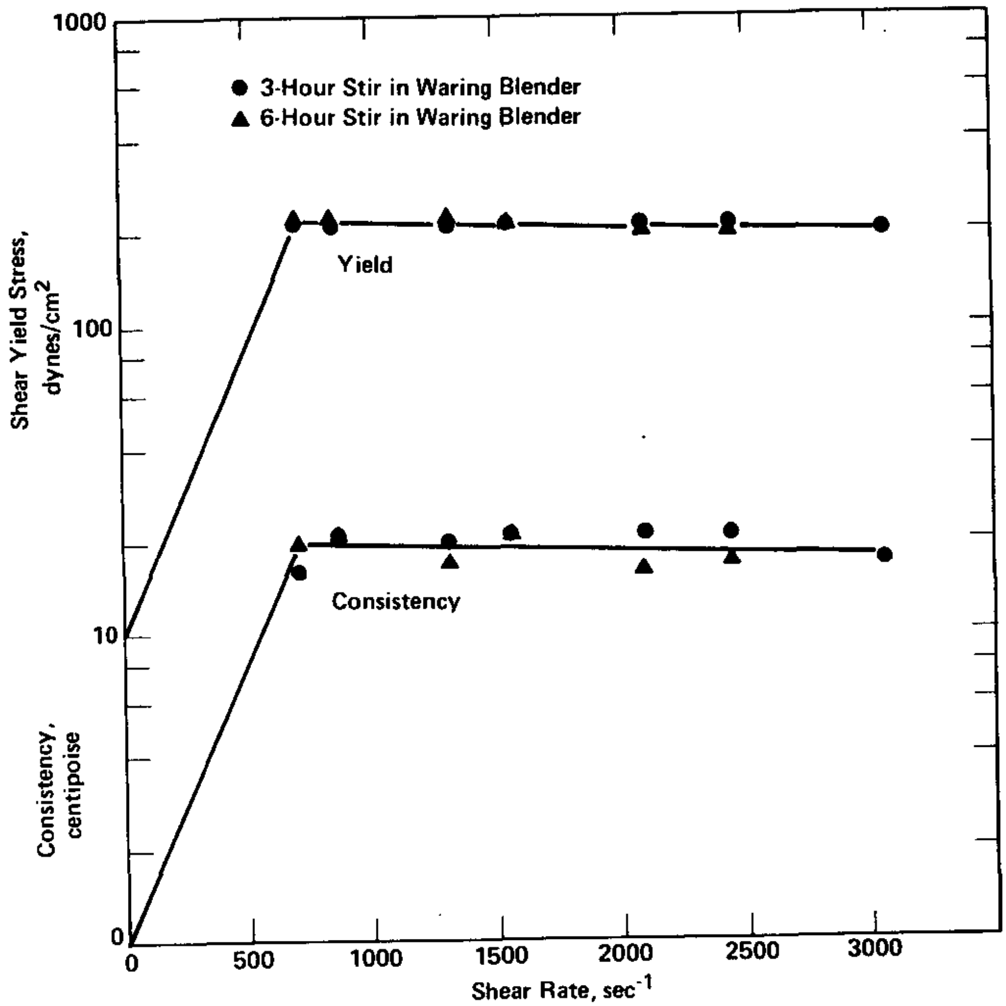


FIGURE 7. Effect of Shear on Kaolin Rheology

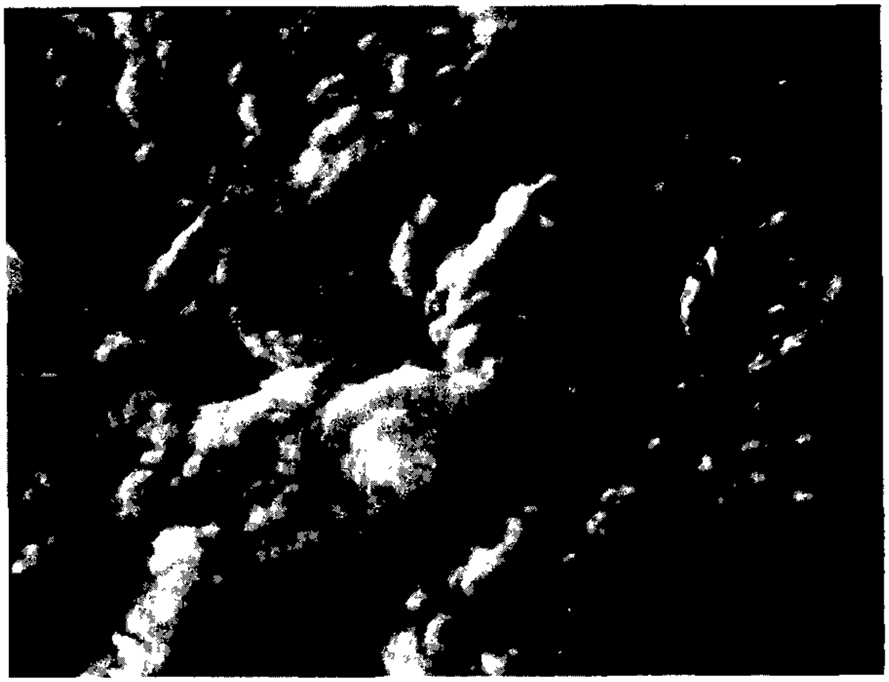
The mechanism of shear is unknown. An obvious mechanism is breakup of particles during pumping. However, unsheared and sheared kaolin platelets, which were examined in the electron microscope, (Figures 8 and 9) appeared to have roughly equal plate size even though the kaolin viscosity increased during shearing in the Bingham™ pump. The viscosity change may result from breaking up of agglomerates, adsorption of water instead of particle fracture, or a particle shape change, such as rounding of corners. The electron microscopy studies also indicated that kaolin particles are much smaller than simulant (Figures 9 and 10) even though the rheological behaviors are similar.

If an analysis of real waste shows rheological properties unlike kaolin, a match may be possible by the addition of resins to the kaolin. Two groups of resins, one produced by B. F. Goodrich called Carbopol® and one by Hercules Inc., were investigated. The Carbopol® series produced changes in yield stress and consistency, but these properties changed with shear. Similar changes would be produced in pump operation (Figure 11). These resins may be used to simulate a sludge which does change during pumping but would not be satisfactory for simulating stable fluids. The Hercules series were shear stable and produced unusual property changes as shown in Figure 12. Additional types of resins remain to be tested if waste analyses indicate that the waste cannot be rheologically simulated with kaolin.

Sludge Settling

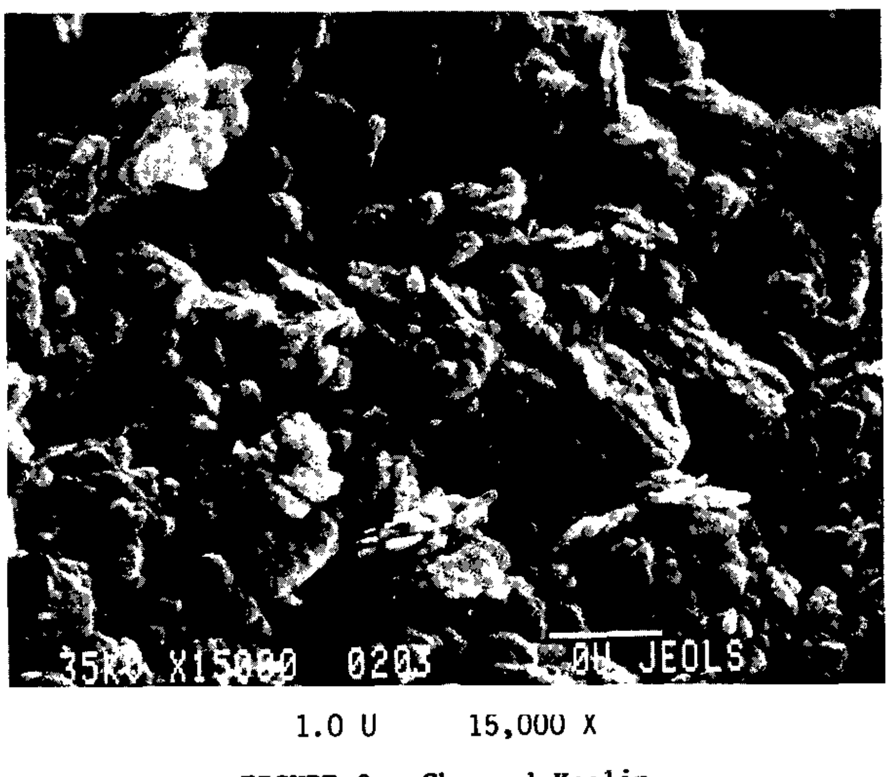
A time lapse between Bingham™ pump tests in the full-scale mockup tank could affect the cleaning radius by making the sludge more difficult to slurry. Laboratory tests showed that kaolin sludges continue to increase in viscosity for two months (Figure 13) due to water exclusion even without pressure by significant overburden.

An overburden increases the water exclusion from kaolin but does not promote water exclusion from simulated sludge. The water exclusion from kaolin is accompanied by a corresponding increase in viscosity (Figures 14 and 15). These data were obtained from kaolin sludge which had been allowed to settle for five and one-half months and from simulated waste allowed to settle for one year. The supernate water was decanted, and successive layers of sludge were removed and analyzed for viscosity and concentration. The kaolin-viscosity data are highly correlated so that the fitted curve is presented instead of the data in Figures 14 and 15. The kaolin concentration paralleled the viscosity rise. The simulated waste data which appear in the figures show no significant change with depth.



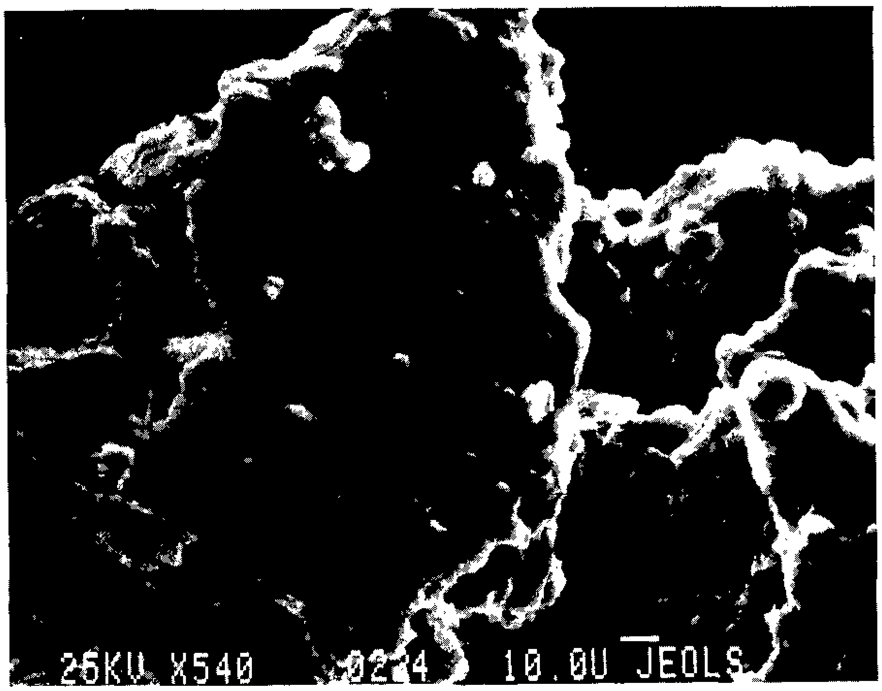
1.0 U 4,000 X

FIGURE 8. Fresh (Unsheared) Kaolin



1.0 U 15,000 X

FIGURE 9. Sheared Kaolin



10.0 U 540 X

FIGURE 10. Simulated Sludge

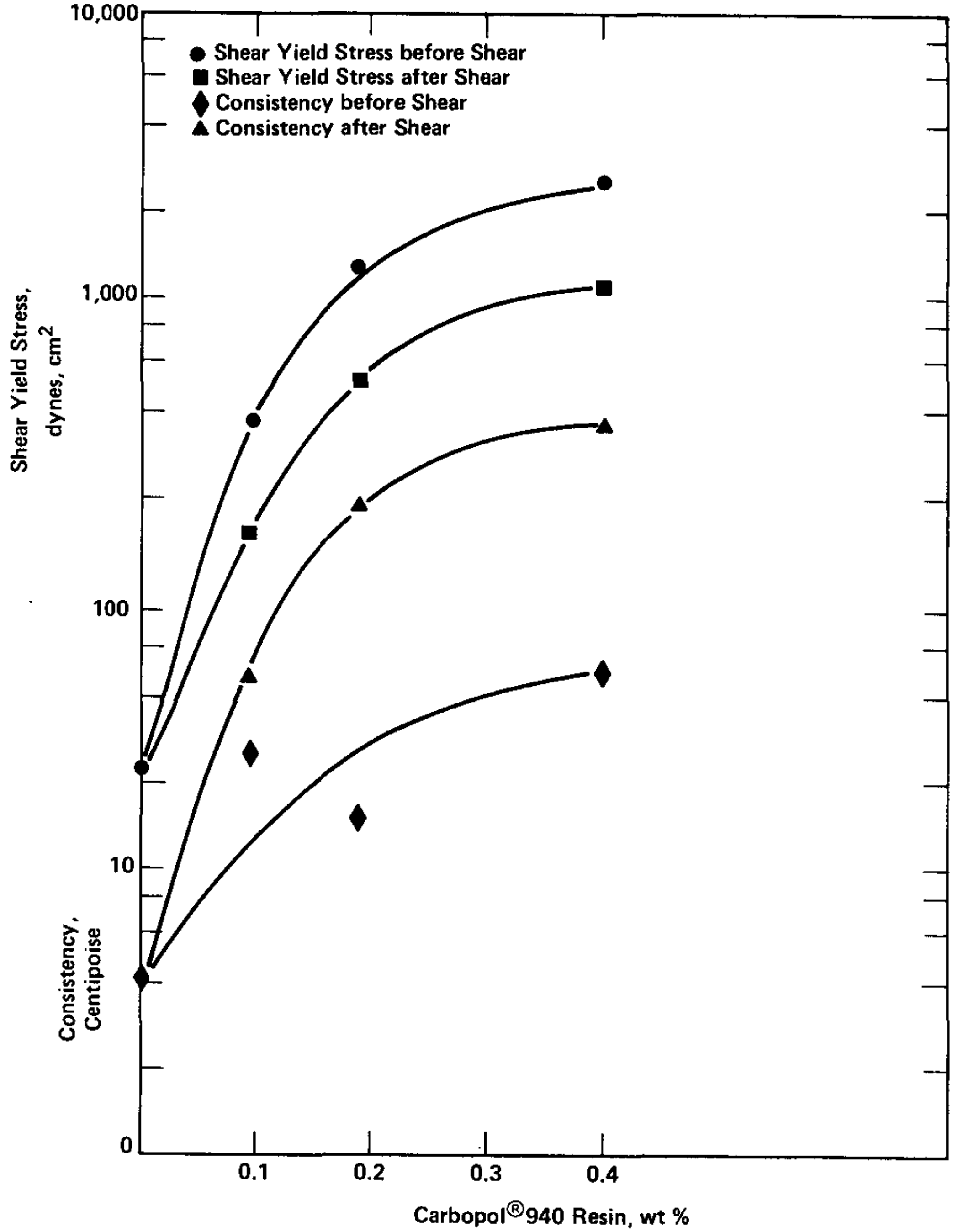


FIGURE 11. Effect of Carbopol® Resins on Viscosity

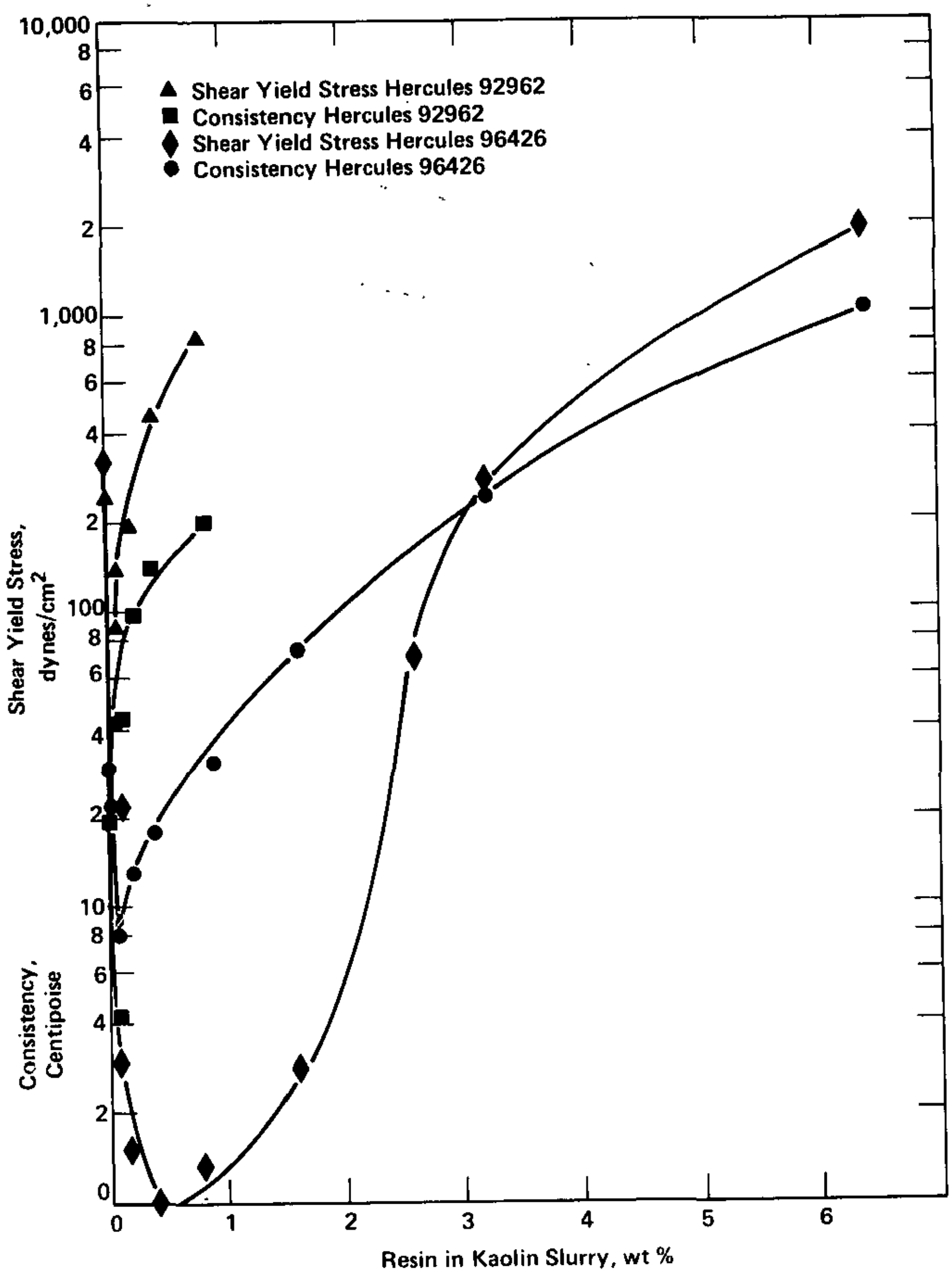


FIGURE 12. Effect of Hercules Resins on Viscosity of Kaolin

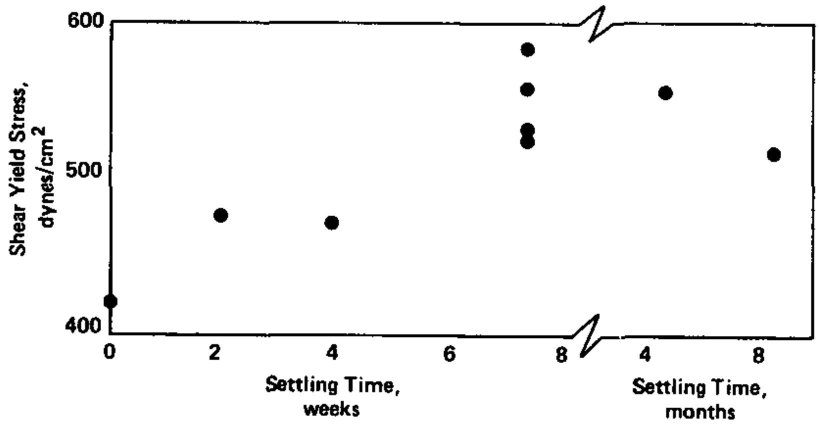


FIGURE 13. Rheological Effect of Unburdened Settling

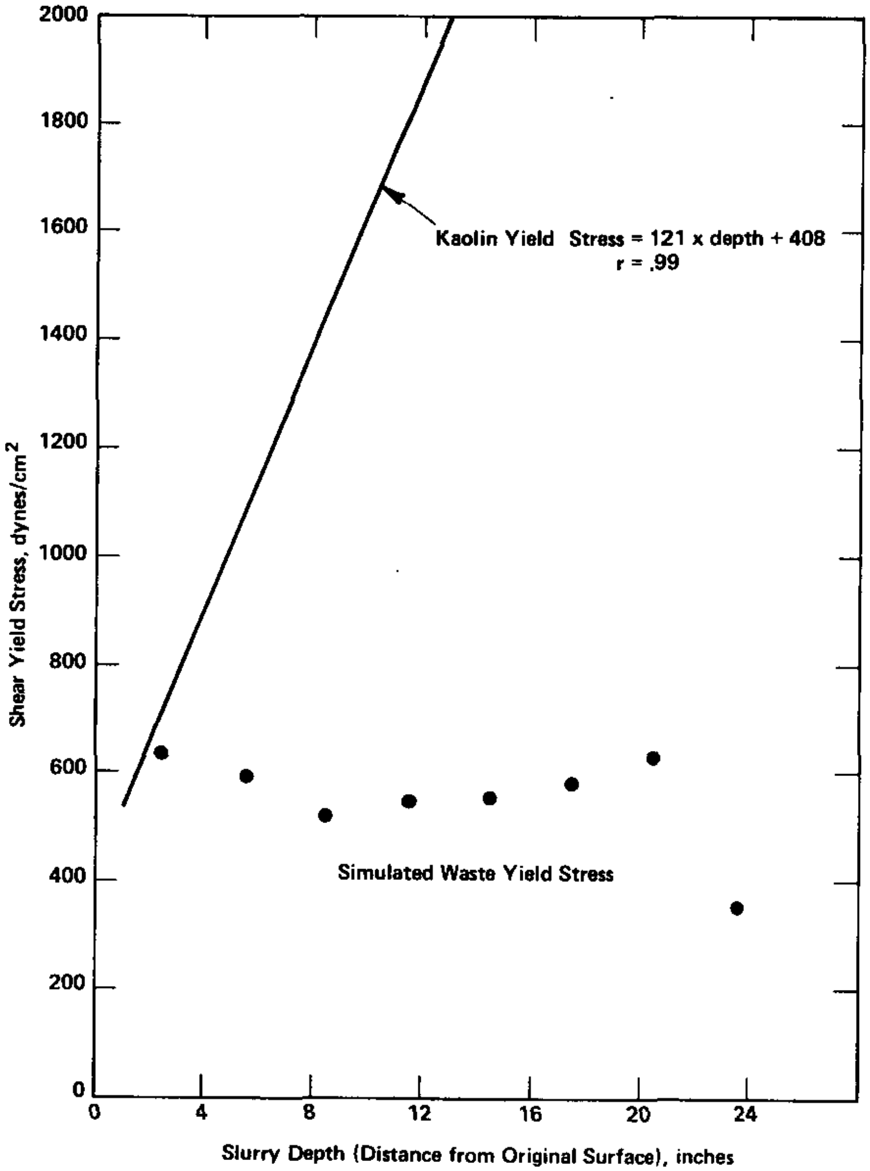


FIGURE 14. Simulated Waste Settled 1 year Compared with Kaolin Settled 5-1/2 Months

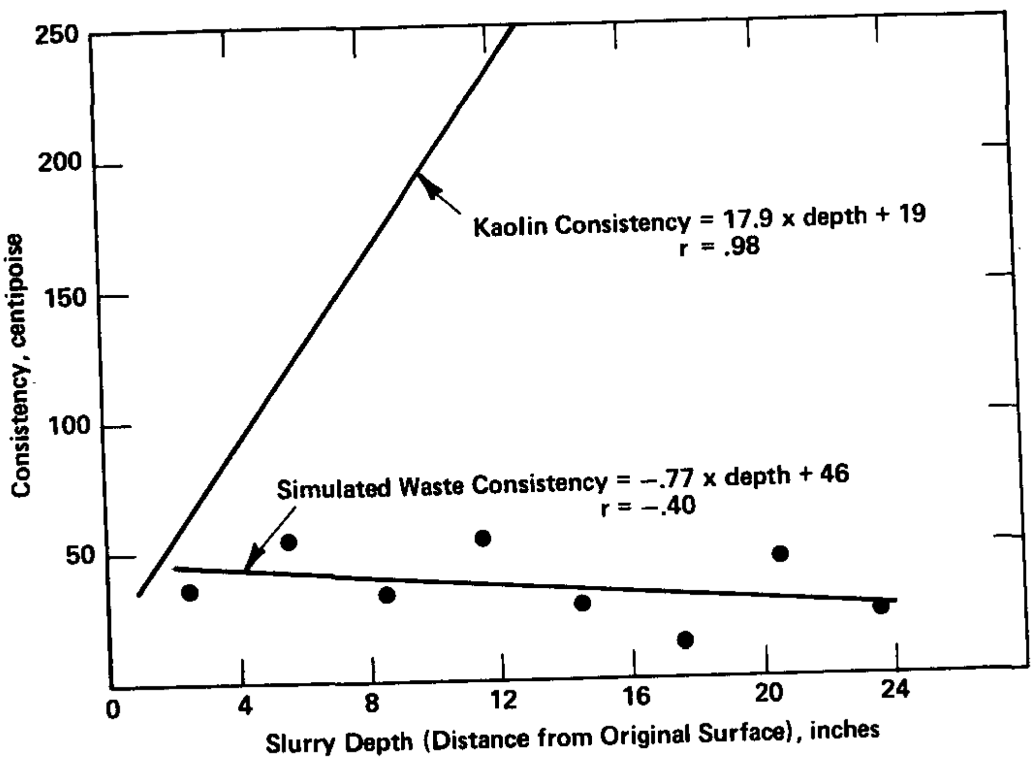


FIGURE 15. Simulated Waste Settled 1 Year Compared with Kaolin Settled 5-1/2 Months

The water exclusion of kaolin with depth may explain why long beaches were formed in kaolin sludge during pump tests. The kaolin in the beach may be much more viscous and difficult to slurry than surface sludge. The water retention of simulated sludge may indicate that real sludge will show less tendency to form long beaches. The beaches left in the Tank 16 demonstration were apparently shorter than the beaches left in the kaolin tests. Simulated sludges which do not become more viscous with depth would presumably be removed more readily from the tank bottom than kaolin sludges which become more viscous. Experience in the SRL Semiworks half tank showed that the lower layer of the sludge that was much more difficult to remove than that formed during two weeks of settling. A half tank is a semicircular pump-test tank with the same diameter as a waste tank but half the circumference, Figure 16.

Beach Formation in Sludge

The shape of the beach formed during pump tests in the mockup tanks was determined by lowering a 10-centimeter-diameter disk attached to a pole into the kaolin. The disk was lowered until a thickness of kaolin that was viscous enough to support it was reached. Repeat measurements were made along a line at several locations sufficient to define the shape of the bowl created by slurrying (Figures 17 and 18). The formed beach rose at an angle of about 15 degrees (Figure 19). Although the tendency of kaolin to increase in viscosity with depth may partially account for the beach, a similar beach might be expected to form in real waste. This might be expected because the liquid exiting the pump nozzle forms a conical jet with maximum force at the center and minimum force at the edges where the nozzle effluent interacts with the stagnant or returning slurry. The outer part of the cone strikes the tank bottom, and the flow is redirected there. The redirected flow moves along the tank bottom, and this flow interferes with the impingement of nozzle effluent moving at a narrower angle. The scouring action of this stream is lost or reduced, permitting kaolin sludge near the tank bottom to remain unslurried. The deflected stream grows in size with distance so that the further from the pump, the greater is the interference with the impinging conical flow and the thicker is the beach.

A suggested method for offsetting the jet deflection on the tank bottom is to incrementally raise the Bingham™ pump so that the jet stream strikes progressively further from the pump extending the bottom scouring action to greater distances. This proposal was tried using a laboratory scale mockup of a Bingham™ pump in a large beaker (Figure 20). The mock pump was tried at two levels, 1/4 inch and 1-3/4 inches from the bottom in a 2-inch-deep kaolin sludge. The slurried kaolin was distinguished from the kaolin sludge by measuring the temperature of the kaolin with a thermocouple. Kaolin that had passed through the pump, which was driving

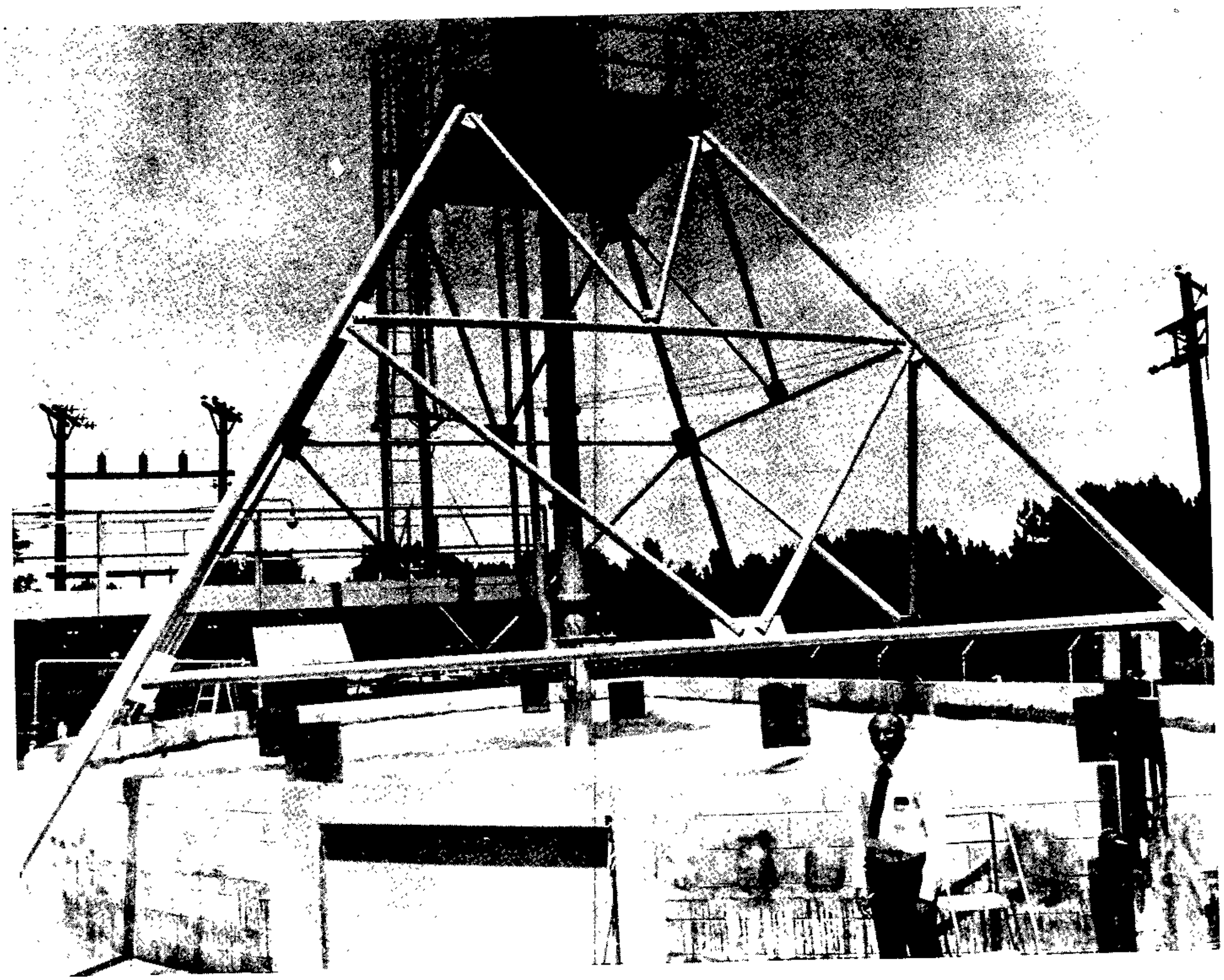
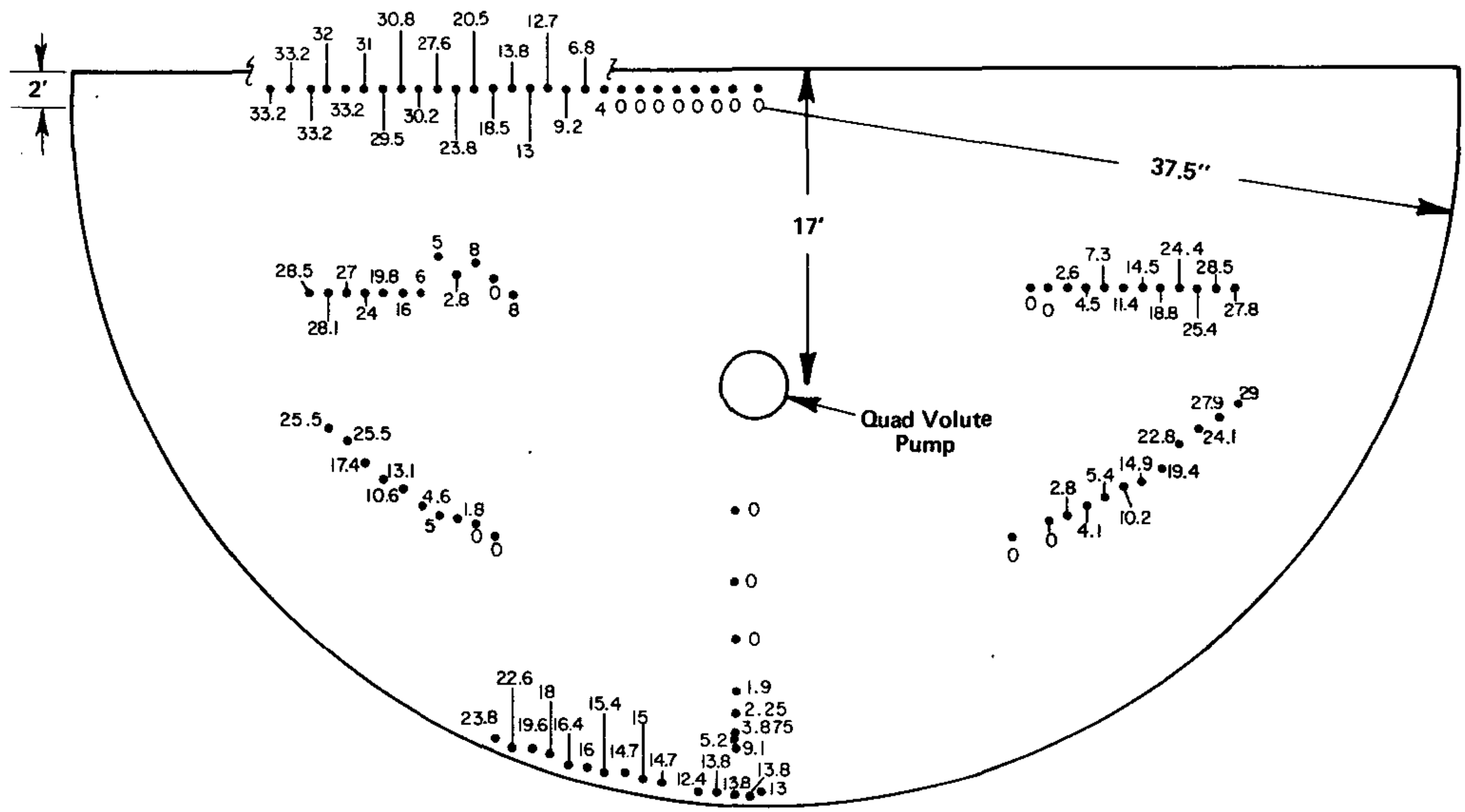


FIGURE 16. SRL Semiworks Tank, A Pump-Testing Tank Simulating the Diameter and Half of the Circumference of a Waste Tank



- Sounding Location

XX.X Kaolin Sludge Depth, inches
Scale: 1" = 10 ft
1/2 Mock Tank
1340-Minute Pump Operation
Wafer— 10-cm dia Stainless Steel

FIGURE 17. Slurrying Test

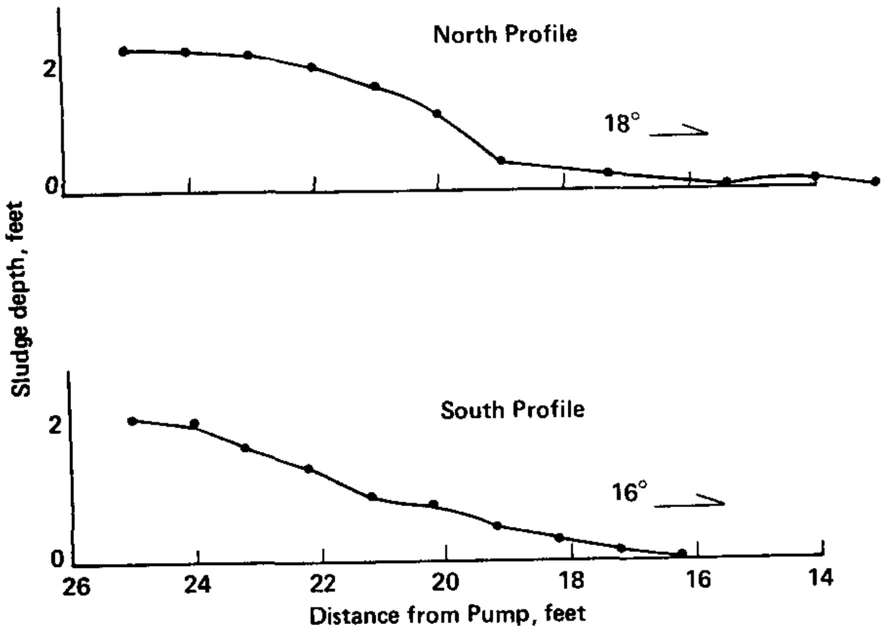


FIGURE 18. Profile of Beach After Slurrying

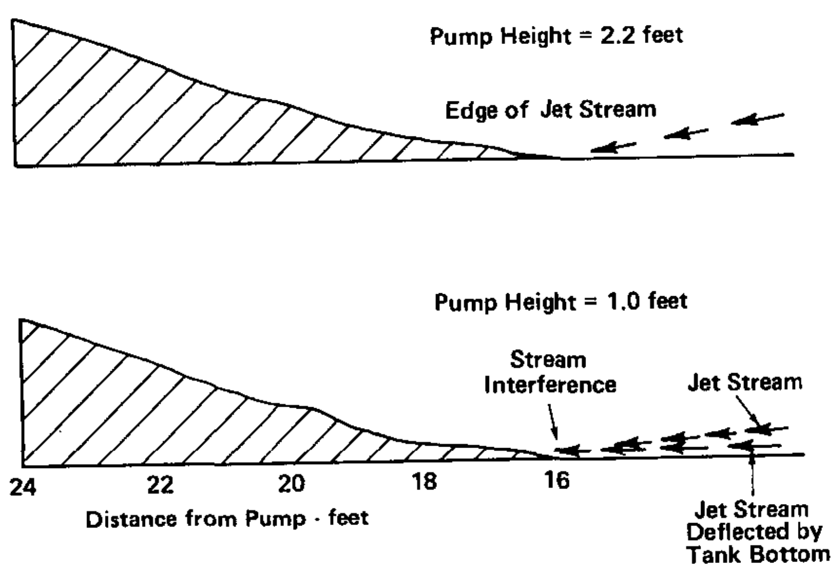


FIGURE 19. Incremental Raising of the Bingham Pump to Remove the Beach*

*The pump jet stream begins to strike the tank bottom about five feet from the pump when the pump nozzle is located one foot above the tank bottom. Striking the bottom between five and sixteen feet, the jet stream loses some of its energy and rides along the tank bottom. At the edge of the beach, the incoming jet stream collides with the deflected jet stream, thus reducing the scouring action. Raising the pump above two feet causes the extreme of the jet to strike the edge of the beach so that there is no deflected stream to interfere.

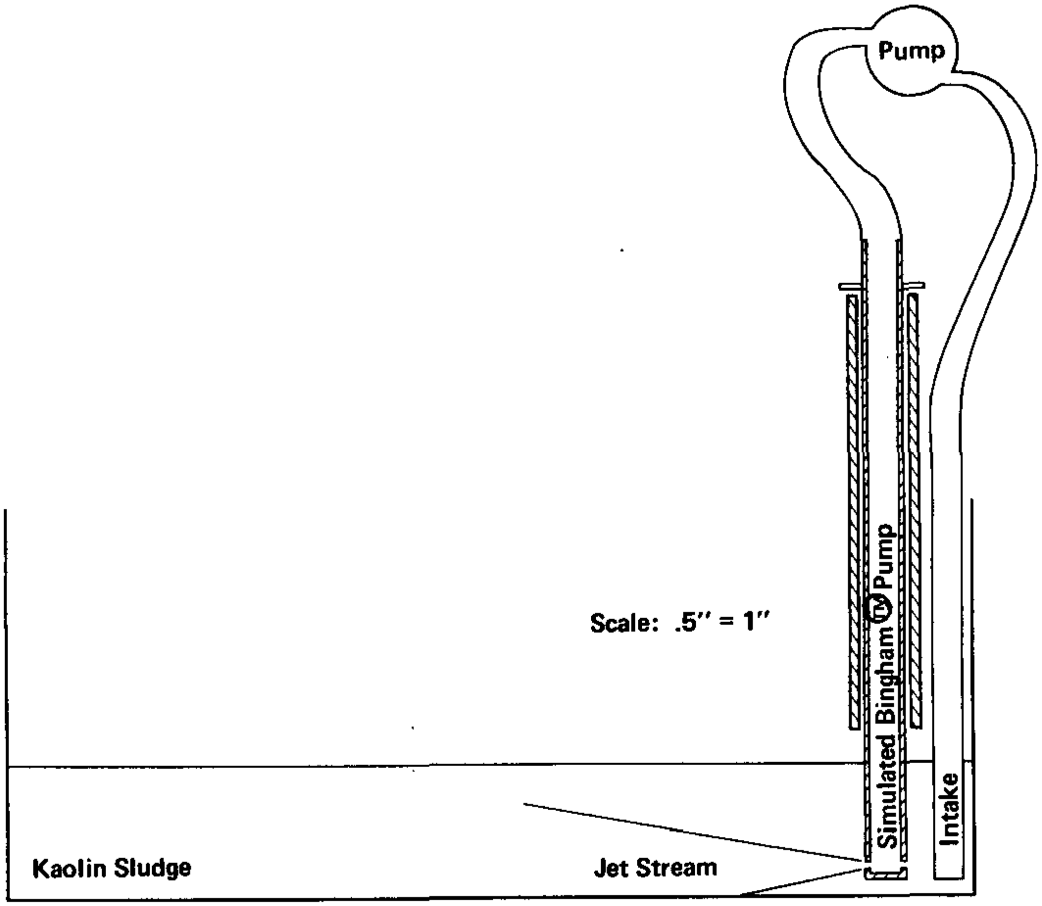


FIGURE 20. Laboratory Apparatus for Determining the Effect of Pump Position on Cleaning Radius

the mock pump, was heated by friction and by the warm pump. It was not possible to distinguish vertical temperature layering. However, the heated slurry extended nine inches from the pump when the pump was in the upper position, compared with six inches in the lower position.

Dilution

Although it would be expected that kaolin slurries of differing concentration would settle to equal concentration sludges, tests showed that the more concentrated a parent slurry, the more concentrated the daughter sludge that settled from it (Table 4).

This tendency of sludges to parallel their parent slurries may explain the existence of a very viscous sludge (yield stress - 3600 dynes/cm^2) left in an area of the half mockup tank after all slurriable sludge was removed. This viscous sludge was located in the position where kaolin had been added to the tank during slurry makeup. At first, the kaolin had been added to the tank with a screw conveyor, which dispersed the kaolin in water so sufficiently that it was swept into the pump and circulated at low concentration. Later, the conveyor broke, and the kaolin was added with a clam shell which dropped 50-lb sacks of kaolin at a time into the water. This kaolin apparently sank before it could be moved into the pump to form a dilute slurry. It probably formed a concentrated slurry which settled to a concentrated viscous sludge. Precautions should be taken during tank slurry makeup to ensure that kaolin is charged in small quantities in a place where it will soon be drawn into the pump and mixed to a dilute slurry.

Because supernate and water may be used to dilute sludge prior to intertank transfer, and water may be used to remove salts and to dilute prior to transfer to the DWPF, the effect of dilution with both liquids was determined. Also, HLC studies of real sludge can

TABLE 4

Concentration of Sludges Formed from Various Concentration Slurries

Concentration - wt % Kaolin	
Parent Slurry	Daughter Sludge
15	24.4
20	28.5
25	29.0
30	31.2

be facilitated if water rather than supernate is used in rheological evaluation. If supernate dilution were necessary for the HLC, the rheological properties of the diluted slurry might vary depending upon the source of the supernate. The rheological effects of artificial supernate (Table 5) and water were shown to be equivalent (Figure 21). Although an equal volume of simulated supernate reduces yield stress more than water does, the results are comparable. Water dilution results in a conservative estimate of pumpability. The presence of salts in the diluent has little effect on the rheological properties.

Temperature

The rheological properties of both kaolin and simulated waste were shown to be independent of temperature below 50°C (Figures 22 and 23). Temperature independence is important because the Bingham™ pump mockup tests and the waste tank must be operated at ambient temperature. In addition, analysis is facilitated because the viscometer temperature control bath can be omitted from the space-limited HLC setup and because the temperature will vary during stirring in real waste tanks.

pH

The pH of kaolin mixtures was measured during concentration vs. rheology experiments. Kaolin forms an acid slurry, which obviously changes pH with dilution as shown in Figure 24. The pH does not affect the rheology.

TABLE 5

Supernate Composition

	Wt %
Water	84.538
$NaNO_3$	7.053
$NaNO_2$	2.863
$NaAlO_2$	1.546
NaOH	1.131
Na_2CO_3	1.200
Na_2SO_4	1.607
NaCl	0.0485
NaF	0.0032
Na[HgO(OH)]	0.00968

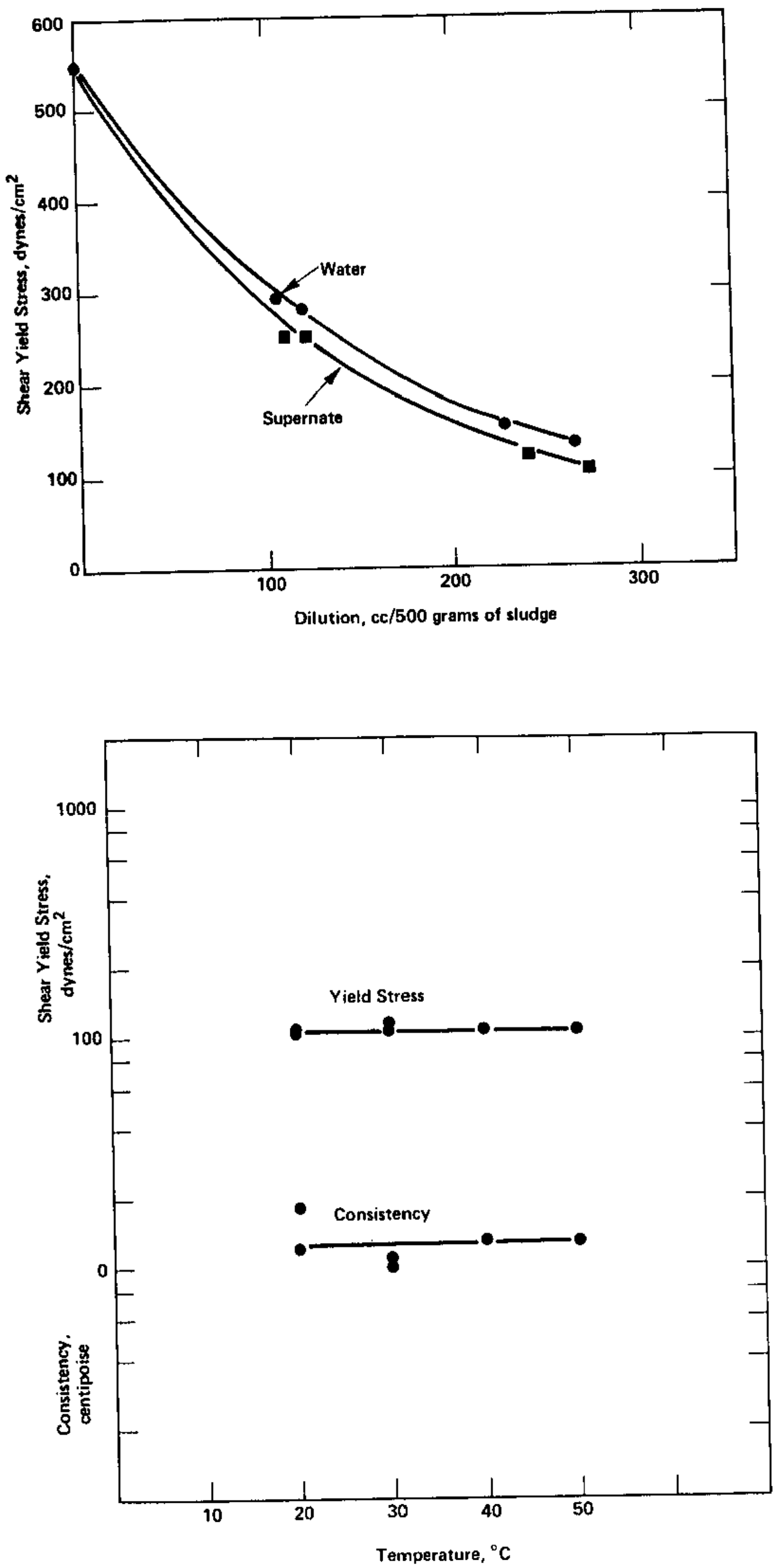


FIGURE 22. Temperature Independence of Rheological Properties of Kaolin

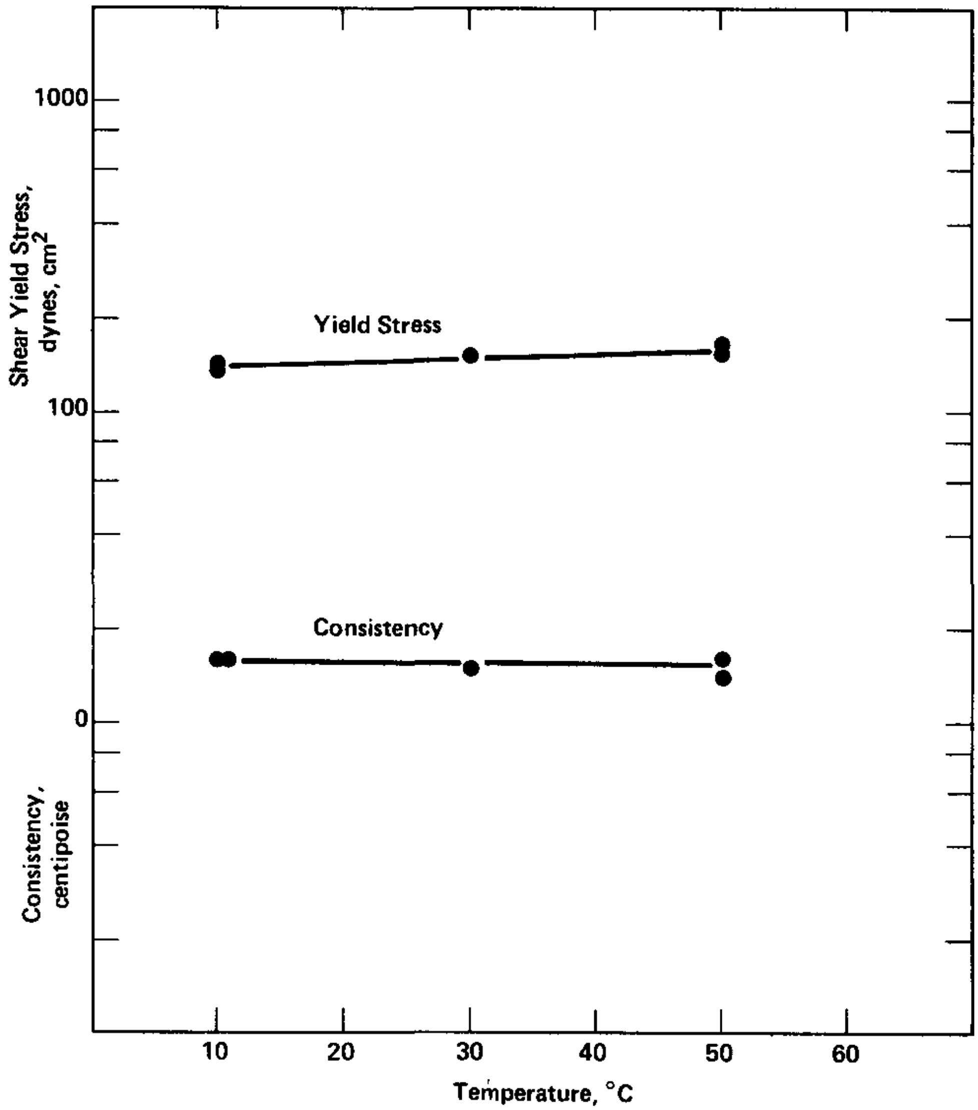


FIGURE 23. Temperature Independence of Rheological Properties of Simulated Sludge

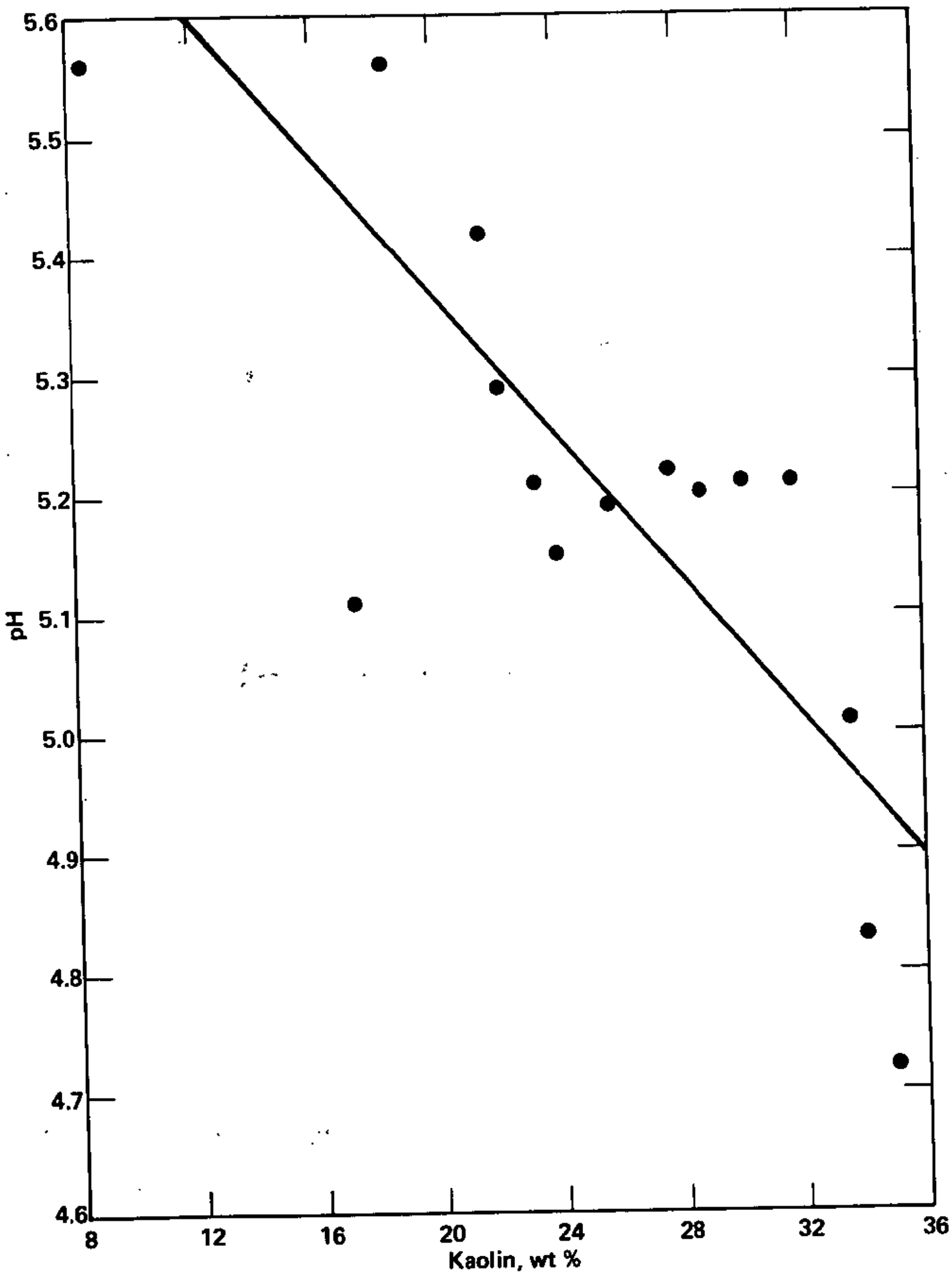


FIGURE 24. Effect of Kaolin Concentration on Acidity

Radiographic Analysis

An alternate analytical technique was developed for determining the concentration, and hence from Figure 5, the rheology of kaolin-water slurry. Slurry of 15 to 30 wt % kaolin in 5 wt % increments was placed in one-inch glass tubes and was radiographed. These concentrations and presumably closer concentrations can be distinguished. No practical application was made of this technique because space was not available in the HLC for autoradiographic analysis, which presumably would allow comparable analysis. The method could be useful in determining concentration and rheological changes during settling.

Rheology by Multiple Disk Test

A technique was developed for crudely estimating the yield stress of a sludge without removing the sludge from its immediate environment. A series of stainless steel disks were set on top of the sludge in succession, the largest first. The last one to sink a maximum of 0.16 cm (1/16") into the sludge was recorded and plotted against the sludge yield stress. As seen in Figure 25, the data provide a rough estimation of the yield stress. The greater than 10-cm data points represent sludges where the largest disk (10-cm diameter) sank more than 0.16 cm.

RESULTS

Below are the most significant results of the sludge rheology program:

Kaolin and chemically simulated waste are both Bingham plastics which perform rheologically similar (rheograms can be made almost identical by adjusting kaolin concentration).

The corresponding rheological compositions of kaolin and chemical simulant can be determined from linear equations of the logarithms of their yield stresses.

Kaolin attains shear stability in less than 32 hours of pump-operation shear rate, 1600 sec^{-1} in the full-scale mockup waste tank or in three hours in a Waring™ blender. Thereafter, its rheological properties will change only with changes in water content or the addition of selected chemicals.

Stagnant kaolin sludge becomes more viscous with time due to water exclusion for periods up to two months.

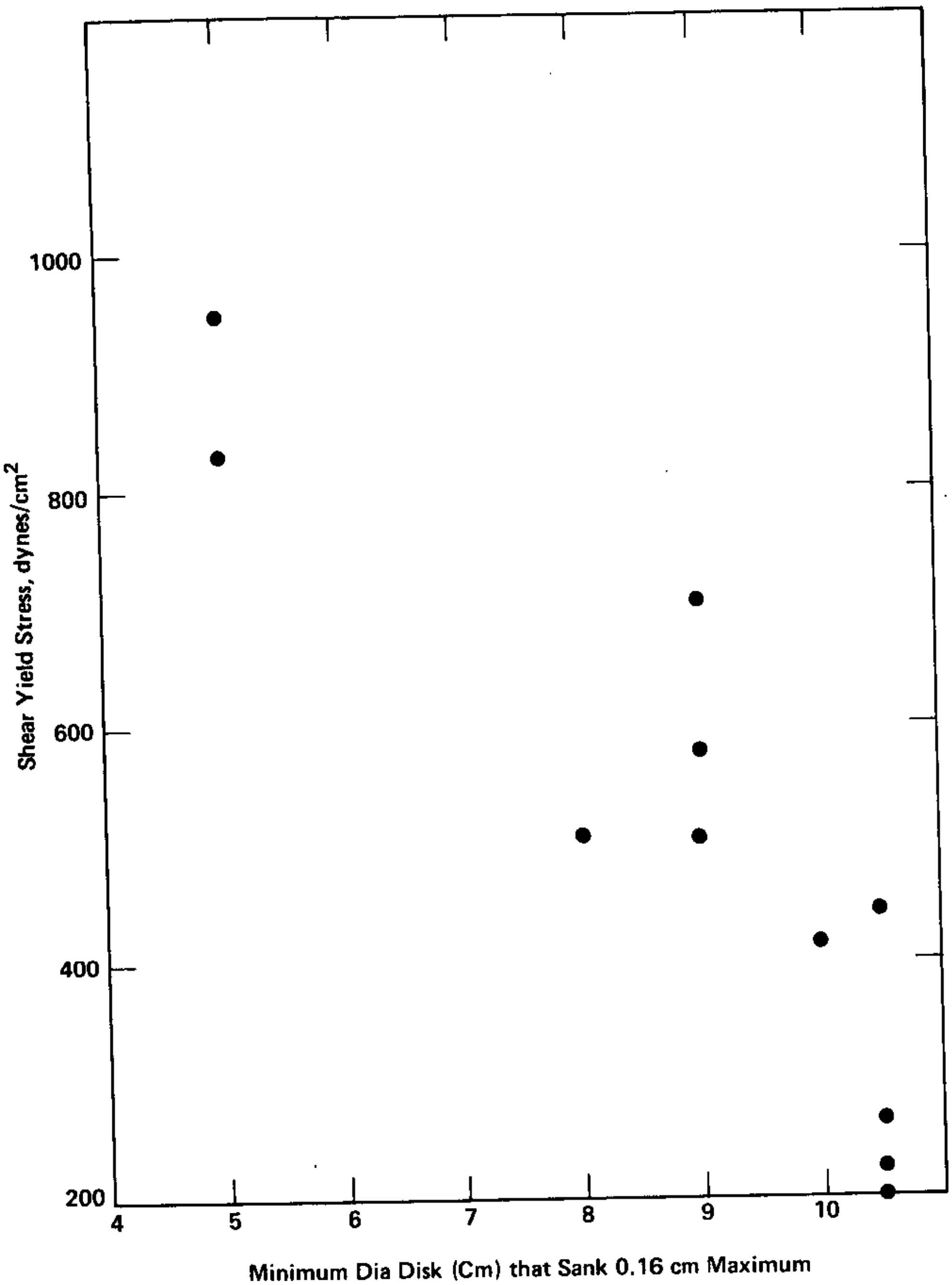


FIGURE 25. Prediction of Yield Stress Using Stainless Steel Disk

Kaolin becomes more viscous with depth, due to water exclusion. The effect on tank tests can be minimized by stirring all of the kaolin in the test area every two or three weeks.

Chemically simulated sludge does not become more viscous with depth; it does not exclude water with depth.

Dilution of simulant with equal volumes of water or simulated supernate has the same rheological effect. Typical salt in the supernate does not affect the rheological change.

The rheology of kaolin and simulated waste is unaffected by temperature in the range of 10 to 50°C.

A 15° beach-like incline is created during slurry tests in kaolin. Reduction of this beach by incrementally raising the pumps is promising. Equipment modification would be required.

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