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Recovery of Fillers from Paper Mill High Ash Sludge by Froth Flotation

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"RECOVERY OF FILLERS
FROM PAPER MILL HIGH ASH SLUDGE
BY FROTH FLOTATION"

by

Prakasha Chandra Misra

Submitted To The Faculty
of the Department of Paper Science
and Engineering in Partial Fulfillment
of the Degree of Bachelor of Science

Western Michigan University
Kalamazoo, Michigan
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Prakasha Chandra Misra

ABSTRACT

The purpose of this project was to recover filler materials (most of which are clay) from the paper mill primary clarifier high and sludge utilizing froth flotation.

Initial attempts to determine the dosage of a water soluble cationic surfactant EHDA-Br did not prove successful. It was discarded, and a partially water soluble cationic surfactant was taken up next. With a dosage of 0.75% (based on suspended solids) and an air flow rate of 1.5 - 1.75 l/min. it was possible to increase the ash content over 68% (minimum 66.5%, maximum 69.5%) in the foam fraction after single stage flotation, compared to 51.67% in the original sludge. A maximum filler recovery of 92.8% was also achieved. To achieve these results trials were made over all the pH values from 2 to 12. The ash content (curve) of the sludge was found to be (practically) insensitive to pH variation. When the sludge was treated in a ball mill, with ion exchange resin and sodium hexametaphosphate it was found to be sensitive to pH variation. Only ball milling increased the ash content to 71.6% (filler recovery 91.22%) at pH 3. The treated sludge was of course floated at three pH values 3, 6.5 and 9, and not over all pH values.

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INTRODUCTION

The general method of flotation is of great importance to the mining industry where the desired mineral is separated from the "gangue" by flotation. Usually a cationic or anionic collector is used to promote contact and attachment between the particle to be floated and the air bubble. Cationic collectors are mainly used for flotation of silica and silicate minerals, and also for some metallic and nonmetallic oxides. Some of the cationic collectors are amines of fatty acids, resin acids and their acid salts, and condensate of fatty acids with polyalkylamines. By the adsorption of the surfactant the surface free energy is lowered and the particles of the ore are carried upward and held in the froth by virtue of their being attached to an air bubble depending upon the relative wettability of the surfaces.

The filler materials in the paper mill primary clarifier sludge, being mostly clay, belong to the family of the silicate minerals.

It is the objective of this present study to attempt recovery (by fractionation) of the filler materials from the high ash primary clarifier sludge by froth flotation using a cationic collector, of the fatty amine type.

FLOTATION PROCESSES

Flotation is a process for separating finely divided solids from each other. Commonly, it is applied to the concentration of metallic ores, the cleaning of solid fuels and the beneficiation of nonmetallic minerals. It can also be applied to the separation of suspended solids from liquids or nonmineral solids from each other.

The ore to be floated is first crushed until the dissimilar solids are detached from each other or the mineral is liberated from the rock matrix. This step is called liberation or severance.

In flotation the separation takes place in water in which the solid particles are suspended. When it is used as a process of concentration, the portion that contains the valuable (or desired) substances is called the "concentrates" and the other portion which contains the worthless (or undesirable) substances is called the "tailing". The tailings are usually discarded.

In almost all flotation processes levitation in water of particles heavier than water is used as a means of concentration of the desired product. If some particles are retained in an oil layer or at the interface between an oil layer and a water layer then the process is spoken of as "bulk-oil flotation". If the particles are retained at a free water surface as a layer "one particle deep" the process is called "skin flotation". If the particles are retained in a layer of foam several inches thick the process is called "froth flotation" (1). Of the three flotation processes only the froth flotation has survived the test of time and the term flotation is now used universally to describe froth flotation.

TABLE 1*
IMPORTANT STEPS IN THE EVOLUTION OF FLOTATION

<u>Approximate Date</u>	<u>Contributor</u>	<u>Contribution</u>
1491	Mansur	Use of differences in wettability of minerals by oils and water to beneficiate ultramarine and/or azurite.
1731	Petit	Air-solid adhesion useful to buoy submerged solids.
1860	Haynes	Bulk-oil process.
1877	Bessel	Boiling process for graphite
1885	Bessel	Chemical-generation gas process for graphite.
1886	Everson	Acidulated pulps desirable.
1902	Froment, Potter & Delprat	Gas as a buoyant medium for sulfide ores.
1905	Schwarz	Sodium sulfide to recover oxidized basemetal minerals.
1906	Sulman, Pickard & Ballot	Reduced amount of oil; introduction of gas by violent agitation.
1909	Greenway, Sulman & Higgins	Soluble frothing agents.
1913	Bradford	Sulfur dioxide to depress sphalerite.
1913	Bradford	Copper sulfate as activator for sphalerite.
1921	Perkins & Sayre	Specific organic collectors
1921	-	Alkaline circuits

* Source: A.M. Gaudin, "Flotation" Second Edition, McGraw-Hill Book Company, New York, 1957, p. 6.

1922	Sheridan & Griswold	Cyanides to depress sphalerite and pyrite.
1924	Sulman & Edser	Soaps for flotation of oxides.
1925	Keller	Xanthates as collectors.
1929	Gaudin	pH control
1929	Jeanprost	Flotation of highly soluble salines
1933	Nessler	Flotation separation of water-soluble chemical salt mixtures
1934	Chapman & Littleford	Agglomeration
1934	-	Alkyl sulfates as collectors
1935	-	Cationic collectors

Froth Flotation in Recent Years

The period 1930 to 1960 saw the application of flotation processes to nonsulfide ores. The wide spread application of fatty acid collectors, the introduction of cationic collectors and the use of pH controls, depressants, activators, dispersants and conditioning techniques became the key factors in the economic production of nonsulfide mineral ores, such as phosphates, cement rocks, crude salines, fluorites and barites. However, to date limited success has been achieved in separating silicates from each other. Progress in this direction is made difficult because of the chemical similarities and complexities among the silicates.

The most recent achievement is the application of cationic flotation reagents, the amines and quarternary compounds, for the flotation of acidic minerals such as quartz, feldspar, mica and kaolinite. These reagents were long known to have excellent collector properties but successful application was not achieved until suitable depressants were obtained.

The cationic collector dodcyl amine was used as early as 1935 by the Valley Forge Cement Company to float mica and talc from argillaceous limestone (2).

In 1936, the U.S. Bureau of mines floated five feldspar by lauryl amine hydrochlori and fluorine-bearing acids or salts in acid circuit. By 1946 the first commercial plant to recover feldspar by this method was already in operation.

In 1935 separation of halite and sylvite minerals by flotation in a saturate brine was established for potash production.

Recent developments of hydroxylated water - soluble frothers along with different types of collectors have achieved greater selectivity for nonmetallic minerals as a whole, and have expanded the application of flotation to such areas as the cleaning of coal and the separation of miscellaneous solids from each other or as a means of removing small quantities of suspended solids from large volumes of aqueous solutions.

Ultraflotation

Standard flotation methods are ineffective in that they are limited to minerals with a particle size greater than 30 microns. This particle size limitation presents problems with products normally in the 1-3 micron size. However, with the introduction of the "Ultraflotation" process, (19) effective removal of fine particle size impurities was achieved. In this process a finely grounded carrier mineral (e.g. CaCO_3) is added along with the standard flotation surfactants. The carrier particles present a larger surface area for fine particles. The fine particles attach themselves to these carrier particles and are then floated out in the froth. The froth is removed from the surface and discarded. The beneficiation of crude kaolin to Ultra White 90, a product with TAPPI Brightness (%) 90-92, is achieved through "Ultraflotation".

Microflotation*

Microflotation, a foam separation process introduced recently, has proven successful particularly in the separation of bacteria, algae and of organic colour from water. It requires the addition of a collector and a frother. But unlike conventional flotation processes produces, under optimum conditions, a thin and relatively dry foam on which the colloids are collected. It requires a very low gas flow rate and fine bubbles.

* By E.A. Cassell, A.J. Rubin, H.B. LaFever, and E. Matijevic, Associated with Clarkson College of Technology, Potsdam, New York are Dr. Cassell associate professor in the Civil Engineering Department; Dr. Matijevic, professor in the Department of Chemistry; and Mr. LaFever, senior student in the Civil Engineering Department. Dr. Rubin is associate professor in the Water Resources Center of Ohio State University, Columbus, Ohio. This article is based on a paper presented at the 23rd Purdue Industrial Waste Conference, held May 7-9, 1968 at Purdue University, Lafayette, Indiana.

RECOVERY OF FILLERS-BACKGROUND AND LITERATURE SURVEY

-General-

The paper mill clarifier sludge contains valuable filler materials along with cellulose fibers and other undesirable materials rendering the sludge unsuitable for papermaking purposes. If the filler materials could be removed, and the recovered product made acceptable to the paper mill then a substantial amount of money would be saved in the purchase of fresh filler materials, land filling, and waste disposal.

All methods applied until the present time involve complete removal of the suspended solids present. And no attempt has been made to remove any particular fraction. Flotation type of save-alls have been successfully used to remove solids from white-water. The process has also been extended to other industries where the applications include, removal of gluten from starch (3) sewage and industrial treatment (4, 5) cleaning of peas, wheat and coal (6) and many other processes in which "discriminating particles are made to work for the benefit of man " (7).

Unfortunately there is no published literature available indicating any application of differential flotation for the recovery of filler materials from "Paper Mill High Ash Sludge". The ash content of this clarifier sludge ranges approximately from 50 to 65% at a consistency of about 6-10%. The filler materials are mostly kaolin (clay) and titanium dioxide. The percentage of titanium dioxide being very small compared to the percentage of clay.

The present study attempts to recover the filler materials from the papermill primary clarifier sludge using froth flotation.

The system is a complicated one due to the presence of coagulants, electrolytes, different varieties and species of fibers, colouring materials, slimes and various grades of filler materials. Hence, before delving into the process variables and other discussions it is necessary to know about clay, cellulose fiber and the suspension of clay-fiber. The ion exchange capacities of clay (kaolin) and fiber, have also to be understood.

- Clay (Kaolinite) -

The structural formula of kaolin is $(OH)_8 Si_4 Al_4 O_{10}$ and the theoretical composition in oxides is:

SiO_2 - 46.54%

Al_2O_3 - 39.50%

H_2O - 13.96%

The clay particles are hexagonal in shape and the structure is well established (8, 9). The kaolinite crystal is composed of alternate layers of alumina and silica bound together through oxygen, and hydroxyl valence linkages. The silica to hydroxyl is a secondary valence bond. This is the weakest point in the kaolinite structure and forms a natural cleavage plane. It is argued that fracture along this plane is responsible for the plate like structure of the clay particles. But these fractures do not give rise to the negative charges present in the clay particles. Fractures occur in a direction vertical to the cleavage plane breaking the primary

valence bonds. This results in the creation of unsaturated active areas on the edges of the clay plates.

Origin of the Electrical Charge

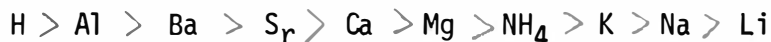
There are basically three ways whereby the kaolin particles can acquire a charge:

1. Substitution within the crystal lattice,
2. Creation of active (charged) sites due to unsaturation from broken bonds,
3. Absorption of ions onto the nonactive areas of the particle surface.

Ion Exchange and Sorption

Grim (10) has discussed the ion-exchange and sorption of clay minerals. Asdel (11) has shown that when a particle is placed in water hydroxyl ions are preferentially adsorbed, adjacent to the positively active areas (due to fracture of primary bonds). These hydroxyl ions carry water molecules with them which are adsorbed to the kaolinite particles and surround it completely. The net charge of this clay-water particle is negative. This negative charge attracts positive ions from the surrounding.

The clay minerals absorb certain anions and cations (the edges being amphoteric) and hold them in an exchangeable state. Unfortunately more information is available regarding cation exchange than anion exchange. The "Hofmeister Series" (12).



gives the order in which the cations are taken up (or adsorbed) readily by a clay particle in suspension. The cation exchange capacity of kaolinite is 3-15 milliequivalents per 100 g.

The pH of the medium has a great effect on the charge of the clay particles. The negative charge grows and positive charge decreases with rising pH as a result of "increasing ionization of the acid groups and decreasing proton addition to the basic groups. The charge goes in the opposite direction with decreasing pH". The effect of pH is obvious since the H^+ and OH^- are the potential determining ions for the clays.

Surface Charge and Zeta Potential

The sign and magnitude of surface charge influence greatly the adsorption characteristics of mineral surfaces. Froth flotation depends on the nature of solid-liquid and solid-liquid-gas interfaces. Hence, it is not surprising that zeta-potential relate to the flotation behaviour of many minerals. The works of Iwasaki, Cooke and Colombo (13) explains the close correlation of zeta potential with flotation behaviour. "In the flotation of oxide minerals (to which clay belongs), where the collectors must be adsorbed by the surfaces, anionic collectors should be effective on positive surfaces, and cationic collectors on negative surfaces". At zero point of charge (isoelectric point) little collector will be adsorbed. Hence, flotation recovery will be practically nil. An elaborate discussion of the relationship between flotation and zeta potential is also given by

Alpan and Fuerstenau (14) and by deBruyn and Agar (15). The influence of electrokinetic charge on inorganic paper fillers on various processes of paper production is also discussed by Huber and Weigel (16, 17).

Zeta-Potential and the Metal Ions

In sewage and papermill effluents, there is an appreciable amount of lyophilic materials present. These lyophilic colloids arise from the metal ions (besides others) added to the system during papermaking process. For better flocculation, and hence clarification, coagulants such as alum, starch and other polyelectrolytes are added to the effluent which lower markedly the zeta potential of the suspended particles (colloidal form) effecting easy removal of the solids. The metal ions present (mostly Al^{3+} , Ca^{2+} , Mg^{2+}) occupy the exchangeable sites of the clay (kaolinite) particles preventing further adsorptions of cations. Hence, for efficient adsorption of surfactant cations on the clay surface (for flotation) the metal ions have to be removed and the charged sites on the clay surface regenerated.

In the present study attempt will be made to regenerate the exchangeable sites of clay and optimize the surface charge of the clay particles. Riddick (18) has discussed the role of zeta potential in coagulation involving hydrous oxides. Black and Hannah (19) have discussed the effect of alum on the zeta potential of different forms of clays in their study of turbidity removal by coagulation with aluminium sulfate.

Ion Adsorption and Double Layer

Gaudin (20) has discussed the double layer surrounding a colloid particle and the effects of ion concentration in the diffuse layer. Since electrokinetic phenomena involve electrical effects at the interfaces, hence, the adsorption of ions effect the nature and the diffusion of the double layer which in turn affect the flotation of mineral particles. For the present study it is to be mentioned that, as a note of caution; high concentrations of any ion will cause the Stern layer to become "crowded" and will lower both the Stern and zeta-potentials. This includes the surfactant ions also since they function as counter ions in the electrical double layer at the solid-liquid interface.

Contact Angles

The air bubble usually does not adhere to a clean mineral surface. However, if a suitable reagent is added the mineral acquires a hydrophobic surface and the air bubble attaches quite readily. Contact angle is a good measure of the work of adhesion at the solid-air interface. A contact angle of 0° between mineral and water means nonfloatability. Conversely, a contact angle of 180° represents wetting of mineral by air to the exclusion of water. But no solid is known to give a contact angle (air-water) larger than 110° . However, in flotation plants adhesion of air bubble to the solid occurs even when the contact angle is less than 90° .

An elaborate discussion is given by Gaudin (21).

-Fiber-

The papermaking fibers are negatively charged to varying degrees. The cause of charge development varies from one pulp type to the other. For bleached sulphites and krafts it is due to the anion-active water solubles, whereas for bleached sulfite due to uronic acid group content. Another part of the negative charge of the cellulose is due to adsorbed hydroxyl and sulfate ions. Due to this surface charge the cellulose fibers also give rise to electrokinetic phenomena, which play significant roles on papermaking processes (22, 23, 24, 25).

Like clay the zeta-potential of most fibers increase with increase of pH. This similarity in the nature of charge and effect of pH complicates the fractionation of filler and fiber, from the clarifier sludge, by differential froth flotation.

Millet et al(25) have measured the ion-exchange capacity of some wood pulps. Calculations based on their results and the surface areas measurements of cellulose show that the charge density of clay (me/m^2) is appreciably higher than that of cellulose fibers, Appendix-5.

The flocculated fiber in the clarifier has almost zero (or close to zero) zeta-potential.

DEVELOPMENT OF THE PROBLEM

The objectives of the present study is to achieve selective fractionation of the filler from primary clarifier high ash sludge utilizing froth flotation.

During the summer of 1972 the author carried on certain investigations in this line. A model synthetic sludge consisting of 70% clay and 30% papermaking fiber with other additives (starch, rosin size, alum) was prepared to simulate a paper mill high ash sludge. This model sludge was floated using a cationic surfactant. The recovered clay had a purity over 90%. But when the same conditions were applied to the real system at a selected pH value (flotation was found to be pH dependent and highest purity was obtained at about pH 10.5 - 11.0) little success was achieved. The reasons were thought to be the following:

1. The optimum₁ pH value (10.5-11.0) for the model system not being the same for the real system.
2. Improper dispersion or insufficient dispersion for the highly flocculated clarifier sludge (due to the coagulants present). Too much dispersion will reduce the particle size below 30 rendering the flotation ineffective.
3. Surface contamination (slime, mud, etc.) and blocking of the clay surface. Hence, all ion exchangeable sites not being available.

4. Presence of various metal ions
(Al^{3+} , Ca^{2+} , Mg^{2+}) and reversal
of (or reduction) ZP.

It was believed that if these surface contaminants could be removed by mechanical means (dispersion, ball milling) and the metal ions removed (by chemical treatment, chelation, ion-exchange resins) then the ion exchange sites would be regenerated.

A dispersant (sodium silicate or sodium hexametaphosphate) would then help to disperse the system by increasing the ZP, which could be optimized by suitable methods.

Based on the above assumptions the following subjects are approached.

Choice of the Collector

Alpan and Fuerstenau (26) in the "Principles of Nonmetallic Mineral Flotation" suggested both cationic and anionic collector for the flotation of nonmetallic oxide minerals. Iwasaki et al (13) in their study used 10^{-3} M solutions of dodecyl ammonium chloride, sodium dodecyl sulphate or sodium dodecyl sulfonate. On the other hand Crandal and Grieves (27) used EHDA-Br (Ethylhexadecyl-dimethylammonium bromide) which was a cationic surfactant (quaternary ammonium salt). In the present study it was decided to use the above mentioned EHDA-Br. This surfactant is water soluble and gives a stable foam. Therefore, a separate frother is not required.

In case trials with EHDA-Br does not prove successful it was also decided to try Kemamine-Q-6502-C which is partially water soluble (soluble in 2-propanol) and also does not need a frother.

Effect of Ball Milling

Grinding has definite effects on both structure and properties of the clay minerals. The investigations of the different workers have been described by Grim (28). Relatively short periods of grinding increased the cation-exchange capacity and the solubility of the alumina and silica in neutral salt solutions and dilute acids and bases.

Replacement of Cations of Clay by Ion Exchange Resins

The experiments of Lewis (29) show that the cations of a particular clay can be exchanged to NH_4^+ , Ca^{++} , Mg^{++} and H^+ forms by passing the clay suspension through columns of sulfonic acid type ion-exchange resins at various concentrations and flow rates. Since the clarifier sludge contains various metal ions which interfere in flotation, it is believed that treatment with ion-exchange resins can regenerate the exchangeable sites on the clay surface and hence achieve better recovery by flotation.

Treatment With Na_2CO_3 (or NaHCO_3), Na_2SiO_4 , and Polymetaphosphates

Chemical pretreatment can have substantial influence on the cation-exchange capacity of the clay minerals in general. Jackson (30) found this to be especially true for allophane (amorphous clay mineral with varying composition). Pretreatment of his samples with mild alkaline solution of sodium carbonate caused a relatively high capacity where as pretreatment with sodium acetate buffered at pH 3.5 caused a relatively low capacity. Black and Hannah (19) pretreated their clay samples with NaHCO_3 solution (to give a concentration of 50 ppm) and then allowed the suspension to come in contact with the ion-exchange resin columns. Addition of sodium carbonate to a clay suspension raises the zeta-potential of

clay by replacing the Ca^{++} present with Na^+ . This "precipitation and replacement mechanism" as Worrall calls it (31), is the basis of many deflocculating agents used in the industry.

There is another class of deflocculants, such as sodium silicate and sodium hexametaphosphate which function rather differently. They are powerful deflocculants, quite effective at quite small additions and raises the zeta potential of the system to a very high value. This is an elegant way of achieving deflocculation by reversing the positive-edge charge and creating a well developed negative-edge double layer. The positive-edge to negative-face attraction is thus eliminated and an edge-to-face repulsion is created. Van Olphen has given an excellent discussion of the subject of what he calls as the "peptization" of clay suspensions (32). The Polyphosphates complex or sequester some of the polyvalent metal ions which assist in the formation and precipitation of sticky deposits.

It is therefore possible that polyanions when adsorbed onto the surface of the clay particles increase the number of exchange (or exchangeable) sites and hence, the charge density.

EXPERIMENTAL SECTION

- Apparatus -

Flotation Cell

The schematic diagram of the set-up used, along with the flotation cell, is shown in Figure 1. The set-up consists of a flotation cell, (with a 6.1 cm, 10 μ stainless steel gas dispersion tube and a circulating pump), a rotameter with traps, a pressure chamber with distilled water to bubble air through the water, and a compressed-air cylinder with pressure gauge and other fittings. The cell has a drain tube. The cell is also connected to a make-up-water-feed-bottle to maintain the liquid at a desired level in the flotation cell. The capacity of the cell is 2.1 liters. But always a sample of 2.0 l was used. At the top, the cell is provided with outlets to collect the foam fraction.

Other Equipment

Among the other equipment were a solid state Waring blender, a Gyra-therm II magnetic stirrer-hot plate, a Beckman pH meter, filtration set-up, muffle furnace hot-air oven, hot plate for drying hand sheets, a triple-beam dial-o-gram balance, a semimicro analytical balance and a suitable timer to keep time in seconds. Besides the above a Ball Mill was also used.

- Procedure -

Preparation of the Sample

The consistency, ash content, pH and the brightness (Elrepho) of the sludge was first determined. Assuming approximately 15% loss due to

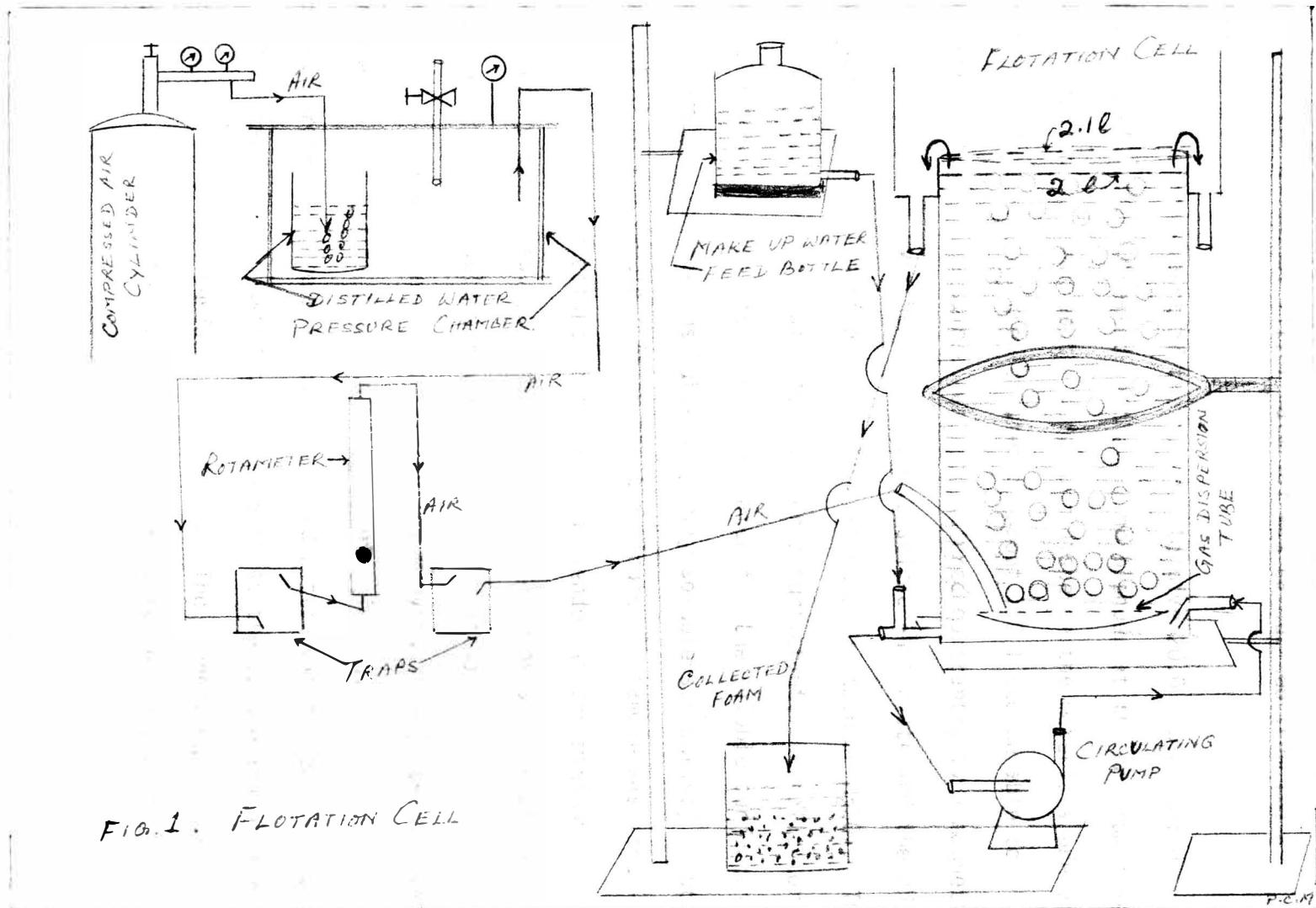


FIG. 1. FLOTATION CELL

ignition the amount of filler material was calculated from the ash content.

The required amount of the sludge, to give 20.0 g b.d. solid, was weighed out and treated in the Waring blender at the maximum speed for 5 minutes to break up the lumps and disperse the materials present in the sludge. It was then diluted to about 1.0 l (to be able to measure the pH). The sample was kept stirred constantly by a magnetic spin-bar over the Gyratherm-II magnetic stirrer-hot plate and the pH was adjusted to the desired value by a solution of NaOH or H₂SO₄. The required amount of the collector (cationic surfactant, the collector solution was prepared with distilled water for the soluble surfactant and 2- propanal for the insoluble or partly soluble surfactant) solution was added to the sample. After about a minute the sample was conditioned with the surfactant in the Waring blender at the minimum speed for exactly 5 minutes. During conditioning 4 drops of pine oil was added when an insoluble surfactant was used. It was then transferred to a 2.5 l flask where it was diluted (by previously adjusted tap water to the desired pH) to 2.0 l under constant stirring. The pH was adjusted carefully. The make-up water feed bottle was filled with about 1.5 l of tap water adjusted to the same pH as the sample and blended with one drop of the surfactant solution. The connecting rubber tube was pinch-clamped. The sample was next transferred to the flotation cell

(drain closed) and the circulating pump started at a minimum speed (with a rheostat) so as to prevent the sample from settling. Opening the gas cylinder air was then allowed to bubble through the distilled water and flow through the rotameter. The gas flow was adjusted to 1.75 l/min but maintained between 1.5 - 1.75 l/min. Once the gas flow became steady it was then connected to the dispersion tube and the timer started. When the foam in the cell began to overflow the pinch-clamp on the connecting tube of the make-up water feed bottle was opened and adjusting the flow of the make-up water by suitable means the level of the liquid in the flotation cell was maintained constant, at 2 l mark. The foam fraction was collected. Air flow was continued until foaming ceased. Then disconnecting the dispersion tube from the air the time was recorded and the gas valve closed. By draining the contents of the flotation cell to a suitable container the tailing fraction was also collected.

Analysis of the Collected Fractions

After flotation the pH of the collected fractions was adjusted to the neutral value (the sludge pH was between 7.1 and 7.3), and allowed to settle. Next the fractions were filtered through Whatman #2, 11.0 cm qualitative filter paper (previously weighed) using a Buchner funnel. The filter papers were dried over the hot plate and in the drying oven at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for two hours. The two fractions were weighed. The dry weight, excluding the filter paper, gave the amount of suspended solid

(of course approximately) in each fraction. The total of the two gave the amount of the sludge floated.

The ash content in each fraction was determined (at $1595 \pm 25^\circ\text{F}$ for four hours). From the ash content the (approximate) filler content was computed, assuming 15% loss due to ignition.

The solid content, ash content, filler content, percent of solid recovered and amount of filler recovered of each fraction was computed and tabulated. For final discussions only the values of the foam fractions were reported and discussed.

The brightness of each fraction was also measured and reported.

Other evaluations such as TiO_2 content and abrasiveness were not done for lack of proper set-up and sufficient time.

- Dosage Study -

One of the variables in the flotation trials is the dosage of the collector surfactant, and the frother. The frother chosen for the insoluble surfactant was pine oil, but for partly soluble and completely soluble surfactant no frother was necessary since they produced sufficient froth.

The soluble surfactant was taken at first. At the neutral pH with air flow rate of 1.75 l/min the sludge was floated with 0.1%, 0.25%, 0.5%, 0.75%, and 1.0% collector dosage based on total suspended solid (i.e. 20 g), (collector EHDA-Br, Ethylhexadecyldimethyl ammonium bromide) with no frother. The details of the trials are given in Appendix-I.

The analysis of the foam fractions (tabulated in Table II and shown in Figure 2, Chapter VI) showed practically no increase in the ash content in the foam fraction compared to the original sludge; shown in the broken line. It was then decided to study the partially soluble surfactant Kemamine-Q-6502-C*. The details of the trials are given in Appendix-I B. The analysis of the foam fractions (tabulated in Table III and shown in Figure 3, Chapter VI) showed an increase in the ash content in the foam fraction after the flotation; minimum 15.36% and maximum 18.10%. Solid and filler recoveries were also good. From Figure 3, ~~A~~ dosage of 0.75% was chosen for all other trials at the same air flow rate and with this partially water soluble cationic surfactant Kemamine-Q-6502-C. No further trials were made with EHDA-Br.

* A Humco product

- Variation of pH -

With a dosage of 0.75% and flow rate of 1.75 l/min the required amount of sludge (20 g) was next floated (single stage) at each pH values from 2 to 12. The preparation of the sample was exactly the same as described before. The pH was adjusted by NaOH and H₂SO₄. The samples were floated and the respective fractions were analyzed for ash and solid content. From these two values other values were computed.

- Ball Milling -

The barrel of the ball mill was approximately half filled with a known weight of the sludge (in this experiment suitable for 6 trials) and the 10/16" dia standard spheres were then dropped in the barrel. About 1 1/2" to 2" of sludge stood above the level of the spheres. The barrel was then closed and rotated at the speed of the equipment (only one speed) for 10 hours. The sludge was treated at neutral pH and at the same original consistency. At the end of 10 hours the barrel was emptied to a suitable container, the spheres picked up, washed and separated. Required amount of the ball milled sample was floated as usual at the desired pH values, and the collected fractions analysed.

- Treatment With Ion Exchange Resins -

About 50 g of Amberlite - 120 (H⁺ form, manufactured by Dow Chemical Company) were filled in 10 nylon bags and with the ends properly closed were

dropped in a container containing 4 l of the sludge at about 2.0% (1.5 - 2.0) consistency (pH about 4.5). The sludge was under moderate mixing. The container was kept cooled by placing in running cold water. The mixing was good enough for the liquid to come in proper contact with the resin beads without breaking the bags. The sludge remained in contact with the ion-exchange resins for 48 hours, after which the nylon bags were removed, and the bags washed carefully free of filler and fiber into the sludge. Removing the calculated amount of sludge to give 20 g, the sample was treated with the surfactant and floated at the desired pH values in the proper manner.

- Treatment With Sodiumhexametaphosphate -

In this case, the sludge was first adjusted to the required pH value to which sodium hexametaphosphate 0.3% based on the amount of filler was added. It was then dispersed in the Waring blender at full speed for 5 minutes. From there on the rest of the flotation procedures were as usual.

- Others -

For the purpose of comparison, the sludge was floated with an insoluble cationic surfactant, Kemamine-Q-1902-C* at pH 10.5 after treating in the ball mill, with ion-exchange resin and sodium hexametaphosphate, in the similar manner as described for the partially soluble surfactant. A control trial was also made at this pH value. In these trials 3-4 drops of pine oil were used for frothing.

* Also Humco product

- PRESENTATION AND DISCUSSION OF
EXPERIMENTAL RESULTS -

- The Results -

The results of the dosage studies to EHDA-Br, and Kemamine-Q-6502-C are given in Appendix I. The results of the foam fractions are given in Table II (for EHDA-Br) and Table III (for Kemamine-Q-6502-C) and are drawn in Figures 2 and 3, respectively.

For reasons mentioned in the experimental section no further trials (except the dosage study) were made with EHDA-Br.

Table IV presents the results of analysis of the foam fractions, after single stage flotation, with Kemamine-Q-6502-C at all pH values from 2 to 12. Details of these trials are given in Appendix 2. The data of Table IV (excluding increase in brightness and ash content) are plotted in Figure 4 .

The flotation results of the pretreated sludge (ball milling, ion exchange resin and sodium hexametaphosphate) at pH 3, 6.5, and 9 are tabulated (foam fraction only) in Table V along with the values of the untreated sludge. These three pH values were selected from Figure 4, the three peaks of the percent filler recovered curve.. Details of the trials after ball milling, treatment with ion exchange resin and sodium hexametaphosphate are given in Appendix 3. The effects of ball milling, ion exchange resin treatment and treatment with sodium hexametaphosphate (data from Table V except increase in ash content and brightness) are shown in Figure 5, Figure 6, and Figure 7 respectively. For the purpose

TABLE II

Analysis of the foam fractions after flotation with water soluble cationic surfactant EHDA-Br at various dosages

<u>Collector Dosage % Based on S.S.</u>	<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>
0.1	Foam	8.32	44.30	52.12	39.8	36.17
0.25	Foam	8.70	50.85	59.82	49.1	51.2
0.5	Foam	13.20	48.48	57.04	69.10	68.65
0.75	Foam	14.70	47.5	55.9	75.0	74.5
1.0	Foam	14.61	45.5	53.5	76.0	73.5

Legend:

Dosage:	Varied
pH:	7
Consistency of the sludge:	1.81%
Ash in the sludge:	48.8 %
Filler in the sludge:*	57.41
Brightness of the sludge (% elrepho);	70.9
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	Varied 10-14 min.

* Assuming approximately 15% loss due to ignition

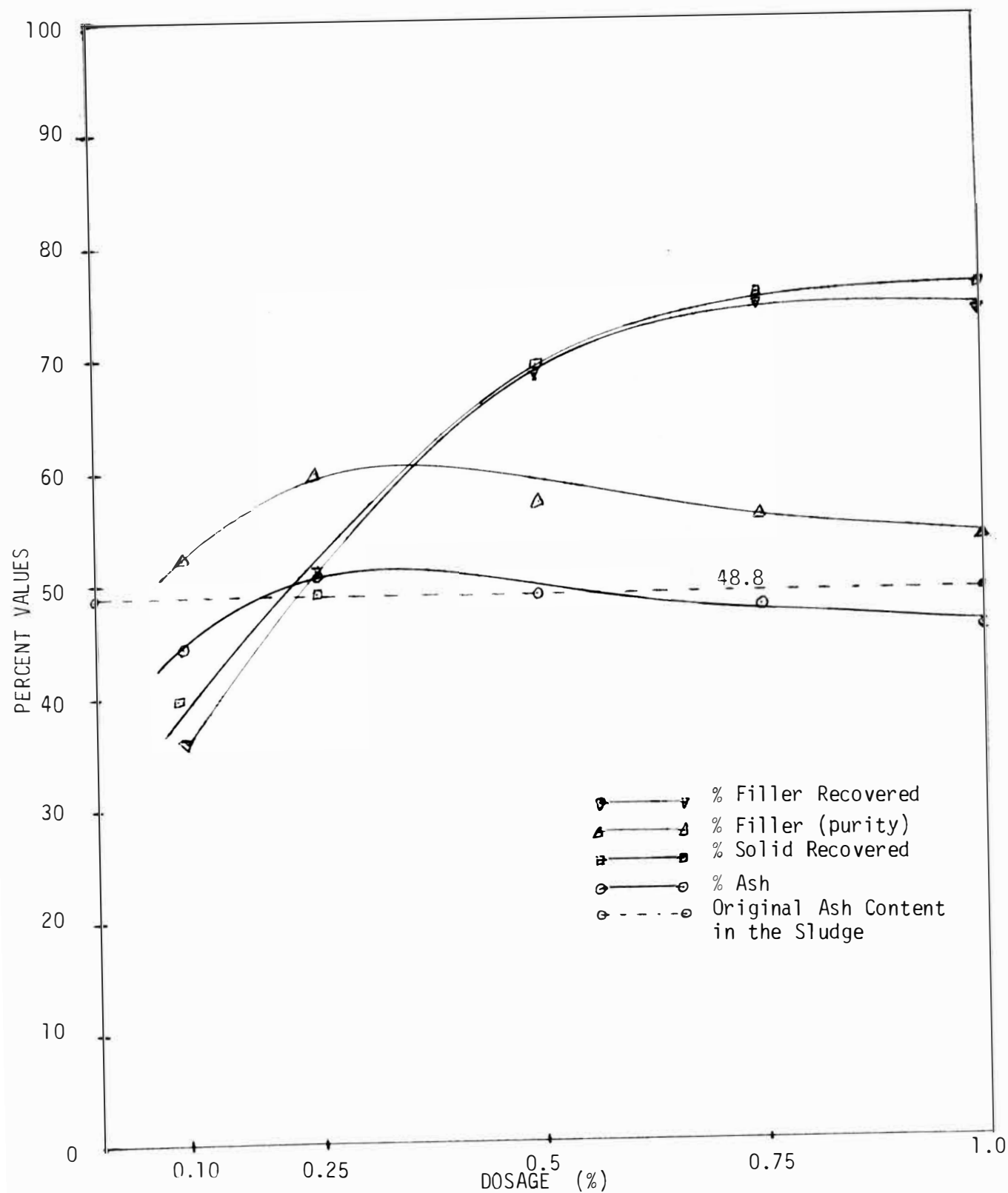


Figure 2 - Analysis of the foam fractions after flotation with water soluble cationic surfactant EHDA-Br at various dosages.

TABLE III

Analysis of the foam fraction after flotation with partially water soluble cationic surfactant Kemamine-Q-6502-C at various dosages.

<u>Collector Dosage % based on S.S.</u>	<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>
0.1	Foam	11.9	67.6	79.5	59.5	77.6
0.25	Foam	13.19	68.15	80.17	63.78	84.16
0.5	Foam	13.56	67.03	78.86	68.97	89.45
0.75	Foam	13.56	68.45	80.53	70.04	92.77
1.0	Foam	13.43	69.77	82.10	67.15	90.62

Legend:

Dosage:	Varied
pH:	7
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge*:	60.79%
Brightness of the sludge (% elrepho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	Varied 7-8 minutes

* Assuming approximately 15% loss due to ignition.

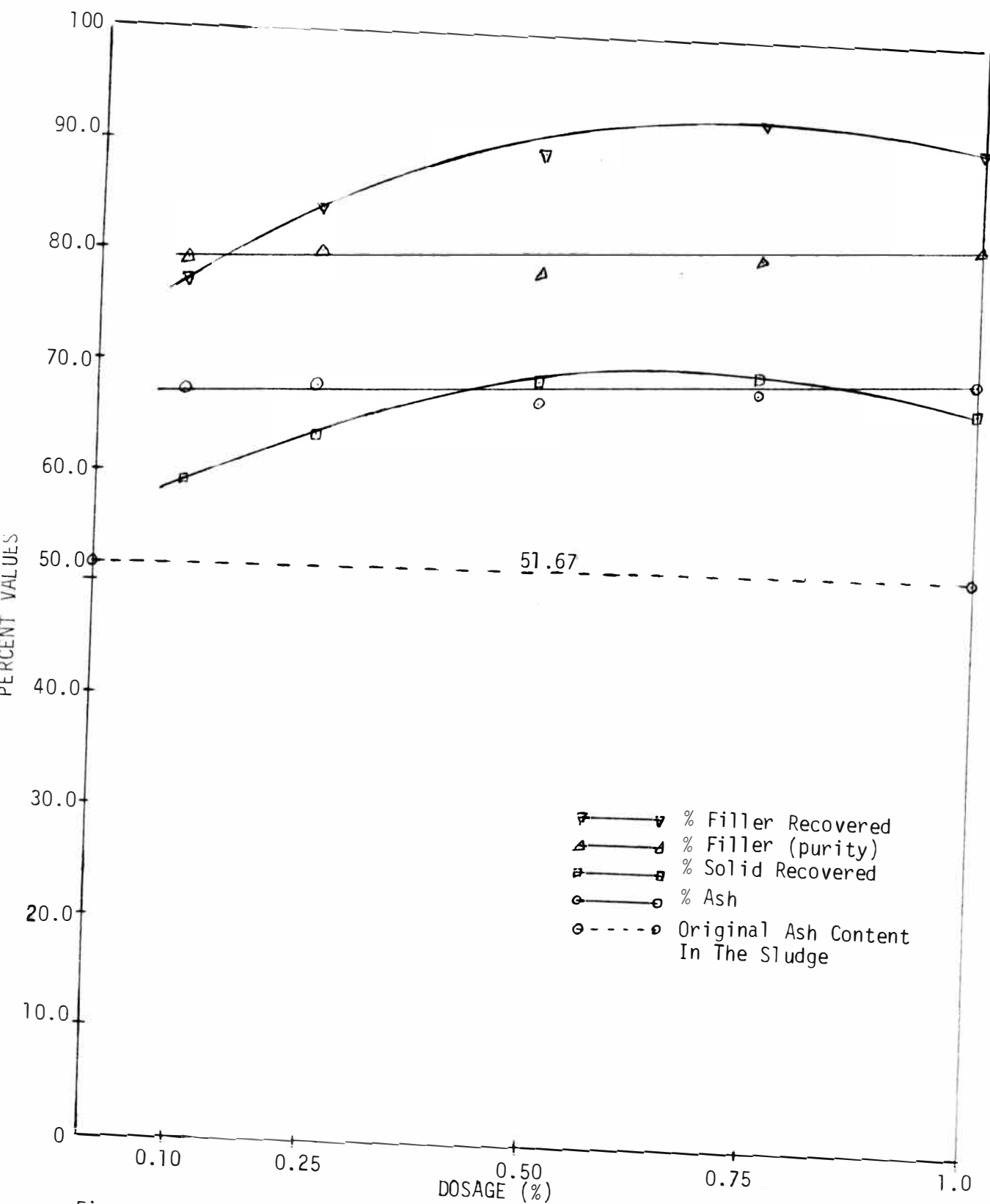


Figure 3 - Analysis of the Foam Fractions after flotation with partially water soluble cationic surfactant Kemamine-Q-6502-C at various dosages.

TABLE IV

Analysis of the foam fractions (after flotation) at various pH values

<u>pH</u>	<u>% Ash</u>	<u>% Filler* (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Increase in Ash Content</u>	<u>Brightness (% Elrepho)</u>
2	67.0	78.50	65.5	84.9	15.33	71.2
3	69.5	81.70	67.1	89.1	17.83	71.5
4	66.8	78.60	66.8	86.8	14.13	72.2
5	67.5	79.30	68.3	89.1	15.83	71.0
6	68.6	80.80	70.0	92.6	16.93	71.0
7	68.45	80.53	70.0	92.8	16.78	72.0
8	65.6	77.20	67.0	85.0	13.93	70.0
9	68.6	80.59	69.6	88.2	16.83	73.0
10	65.9	77.52	65.2	82.7	14.23	73.0
11	66.5	78.25	67.7	86.8	14.83	71.0
12	67.23	79.10	68.0	88.0	15.56	71.0

Legend:

Dosage:	0.75%
pH :	Varied from 2-12
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
*Filler in the sludge	60.79%
Brightness (% Elrepho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of flotation:	Varied, 5- 7 minutes

* Assuming 15% loss due to ignition, i.e. 85% ash \simeq 100% filler

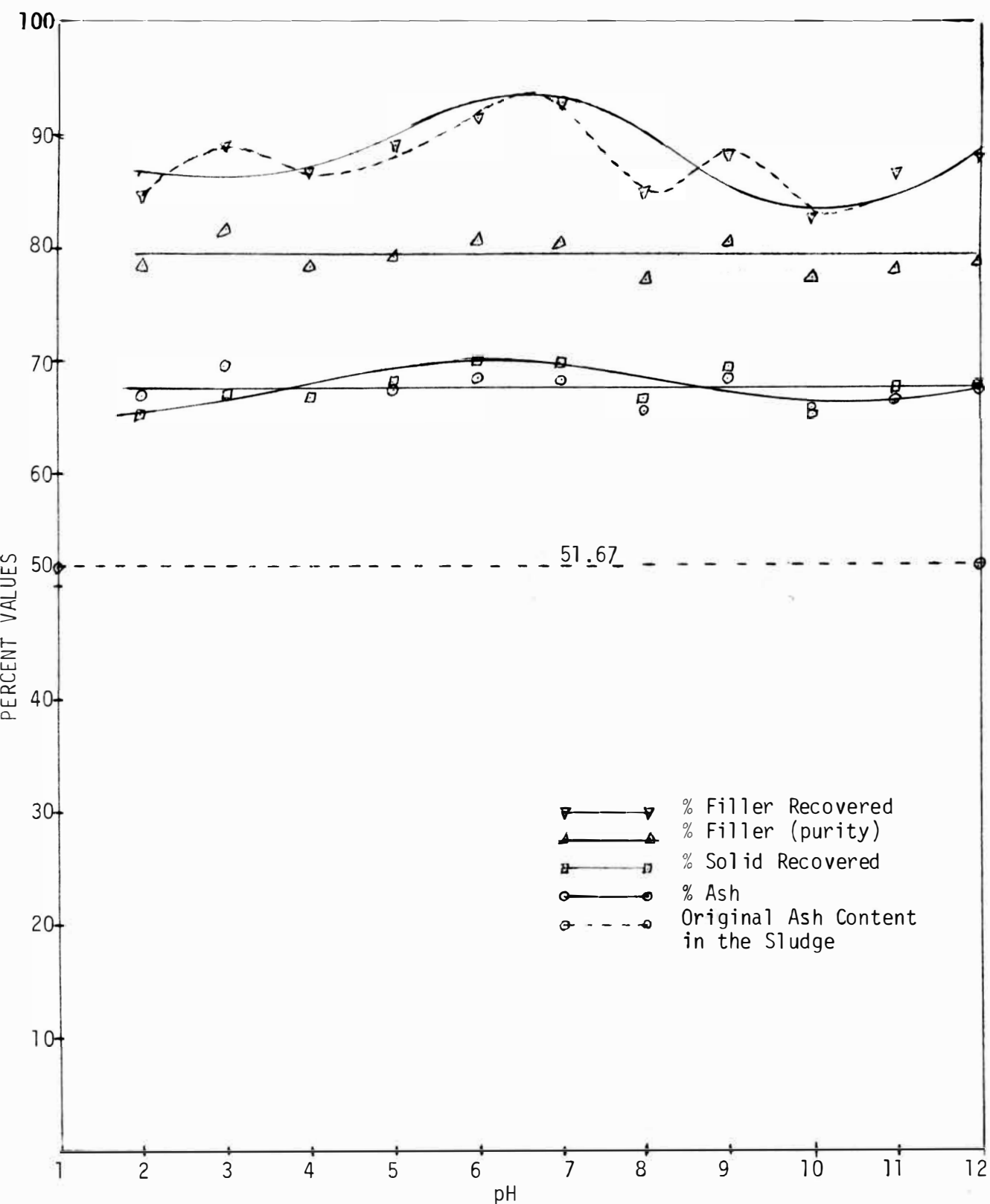


Figure 4 - Analysis of the foam fractions after flotation at various pH values with Kemamine-Q-6502-C.

TABLE V

Analysis of the foam fractions (after flotation) of the untreated and treated sludge at pH 3, 6.5 and 9.

<u>Treatment</u>	<u>pH</u>	<u>%Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Increase In Ash Content</u>	<u>Brightness (% elrepho)</u>
No treatment (control)	3	69.5	81.70	67.1	89.1	17.83	71.5
	6.5	68.52	80.67	70.0	92.7	16.85	71.5
	9	68.6	80.59	69.6	88.2	16.85	73.0
Ball Milling (10 Hours)	3	71.6	84.2	65.84	91.22	19.93	71.0
	6.5	71.0	83.6	57.66	79.24	19.33	71.5
	9	67.0	78.8	65.44	84.86	15.33	71.6
Ion Exchange Resin (48 hours)	3	63.74	75.00	61.64	76.02	12.07	70.0
	6.5	59.89	70.47	59.68	69.17	8.22	71.0
	9	64.03	75.33	64.72	80.21	12.36	71.6
Sodium Hexameta-phosphate (0.3% based on filler)	3	69.76	82.07	63.09	85.20	18.09	71.6
	6.5	61.50	72.36	70.22	83.56	9.83	72.0
	9	69.18	81.38	65.96	88.27	17.51	71.8

Legend:

Dosage:	0.75%
pH:	Varied 3, 6.5 and 9
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge*:	60.79%
Brightness of the sludge (% elrepho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of flotation:	Varied, 4-7 min.

* Assuming 15% loss due to ignition, i.e. 85% ash \approx 100% filler

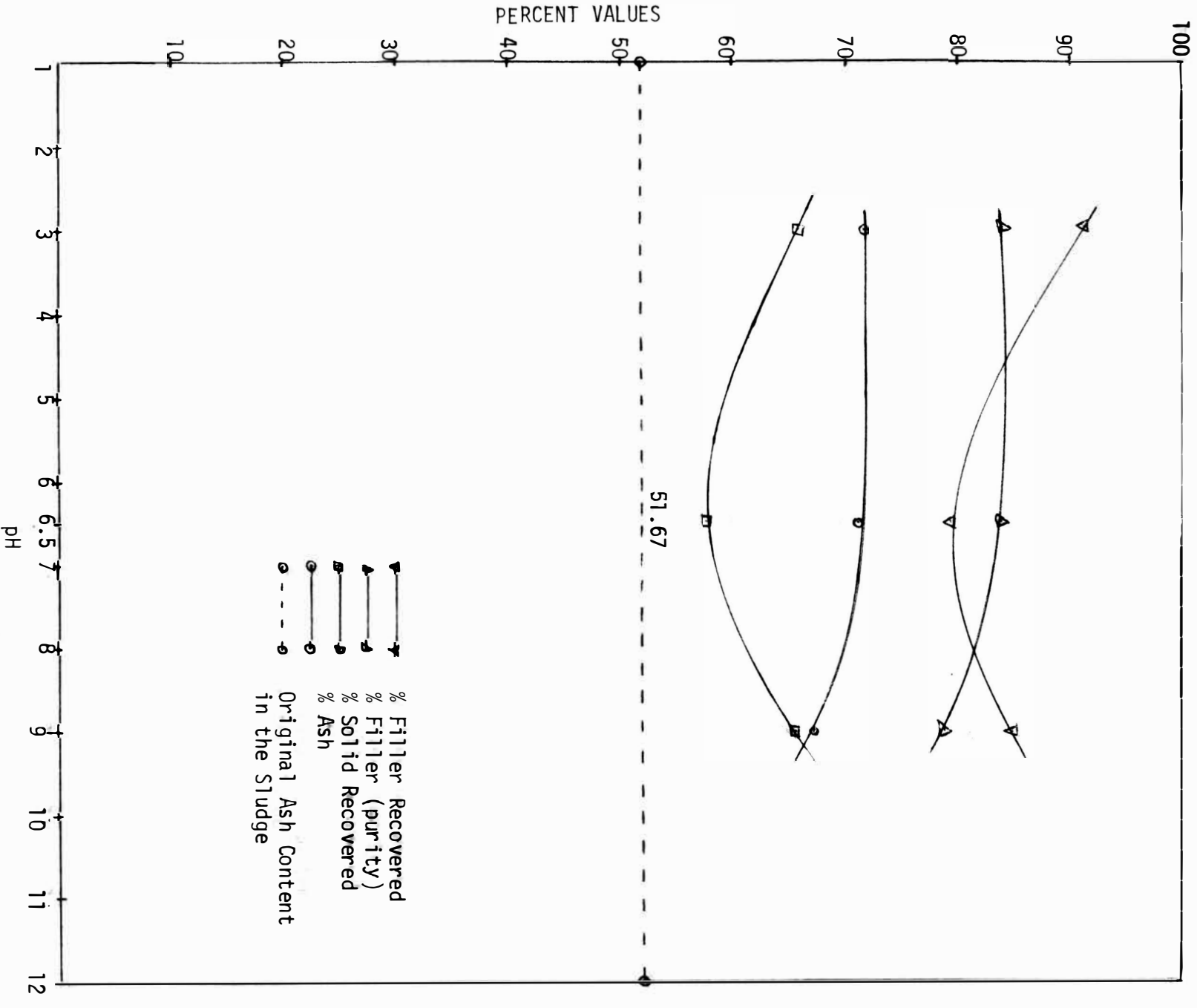


Figure 5 - Effect of Ball Milling at pH 3, 6.5 and 9 (analysis of the foam fractions only are shown)

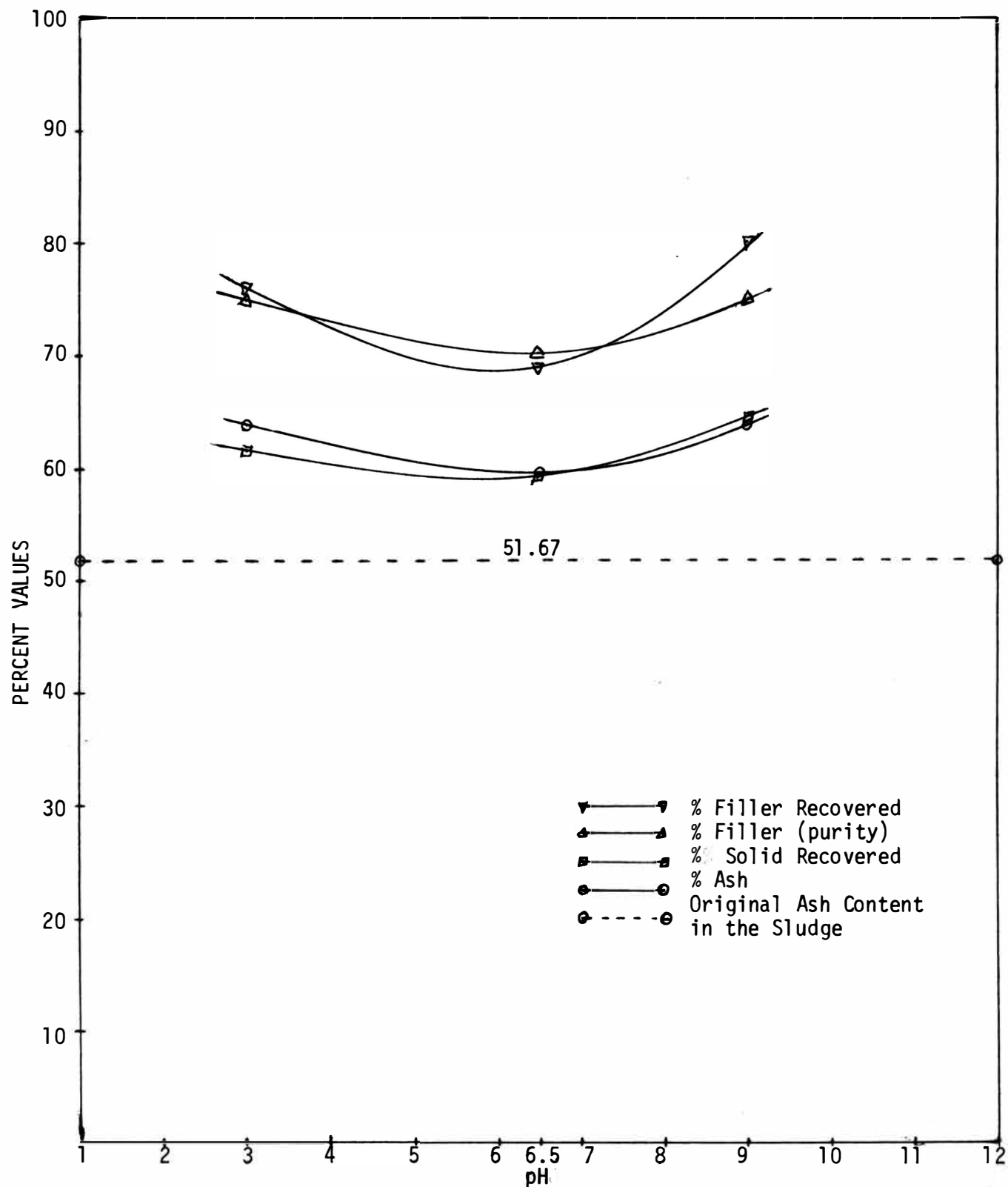


Figure 6 - Effect of Ion exchange resin treatment of pH 3, 6.5 and 9 (analysis of the foam fractions only are shown)

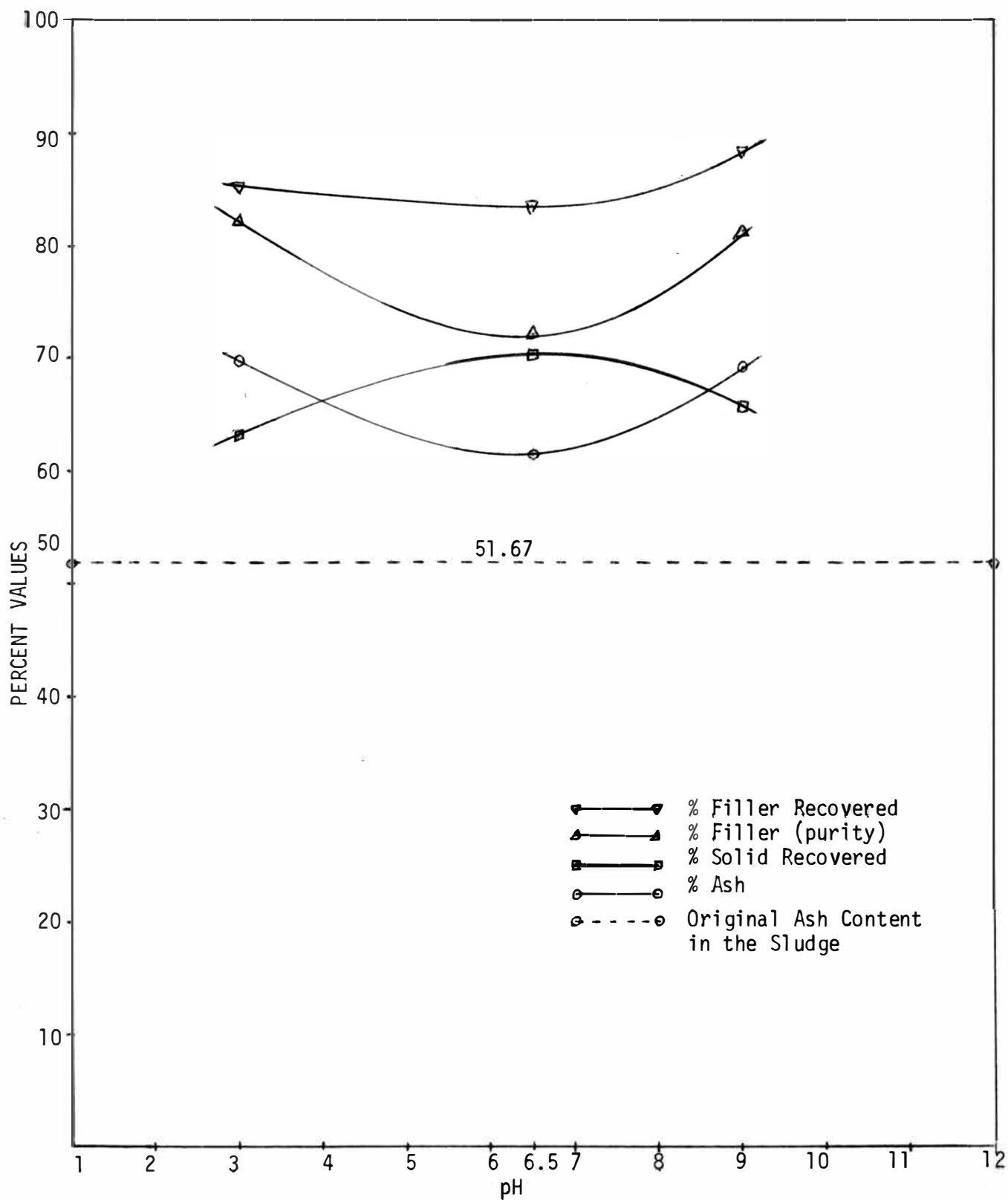


Figure 7 - Effect of Sodium Hexametaphosphate at pH 3, 6.5 and 9 (analysis of the foam fractions only are shown)

of comparison, the ash content and the percent of filler recovered for the foam fractions of untreated and treated sludge, (after flotation at pH 3, 6.5, 9) are tabulated in Table VI and shown in Figure 8. These values are taken from Table V.

The results of the flotation trials with the insoluble surfactant (only the ash content, percent solid recovered and percent filler recovered of the fractions) are tabulated in Table VIII along with the control. Details of the trials are given in Appendix 4.

TABLE VI

Comparison of ash content and percent filler recovered of the treated and untreated sludge (only the foam fractions after flotation).

<u>Treatment</u>	<u>pH</u>	<u>% Ash</u>	<u>% Filler Recovered</u>
No treatment (control)	3	69.5	89.1
	6.5	68.52	92.7
	9	68.6	88.2
Ball Milling (10 hours)	3	71.6	91.22
	6.5	71.0	79.24
	9	67.0	84.86
Ion Exchange Resin (48 hours)	3	63.74	76.02
	6.5	59.89	69.17
	9	64.03	80.21
Sodium Hexameta- phosphate (0.3% based on filler)	3	69.76	85.20
	6.5	61.50	83.56
	9	69.18	88.27

Legend:

Dosage:	0.75%
pH:	Varied, 3, 6.5 and 9
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge*:	60.79%
Brightness of the sludge (Elrepho)	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of flotation:	Varied, 5-7 minutes

* Assuming 15% loss due to ignition, i.e. 85% ash \approx 100% filler

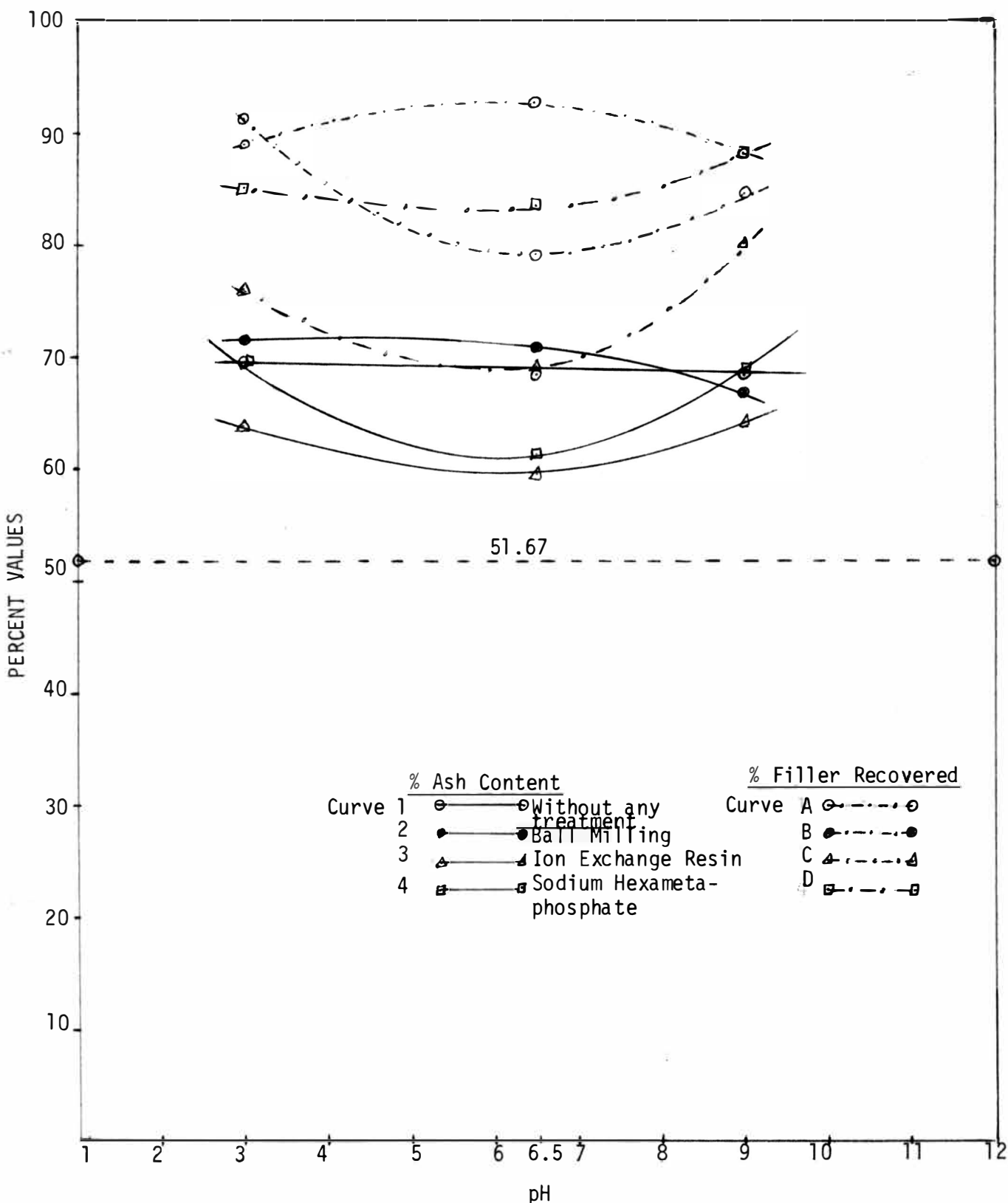


Figure 8 - Comparison of ash content and percent filler recovered of the treated and untreated sludge at pH 3, 6.5 and 9 (analysis of the foam fractions only are shown)

TABLE VII

Flotation results (foam fraction only) of the treated and untreated sludge after flotation with the insoluble cationic surfactant Kemamine-Q-1902-C.

<u>Treatment</u>	<u>pH</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
No treat- ment (control)	10.5	67.0	78.82	64.28	83.35	66.0
Ball Milling (10 hours)	10.5	61.46	72.31	57.53	68.83	67.0
Ion Ex- change Resin (48 hours)	10.5	64.85	76.30	70.25	86.0	67.0
Sodium hexameta- phosphate (0.3% based on filler)	10.5	61.65	72.53	74.23	88.54	66.6

Legend:

Dosage:	1.0%
pH:	10.5
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness of the sludge (% elrepho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	Varied 5-7 min.

- Discussion of the Results -

Dosage Study

The dosage studies for the two cationic surfactants are included in the experimental section. It can be seen from Figure 2 that the water soluble surfactant EHDA-Br practically had no effect in the recovery of the fillers, though the slope of the percent ash curve (Figure 2) varied with the dosage. The slopes of the percent filler recovered curve and percent solid recovered curve are steep until a dosage of 0.5% is reached after which they begin to rise slowly and finally flatten out after a dosage of 0.75% is reached. On the other-hand the partly water soluble surfactant Kemamine-Q (Kemamine-Q-6502-C by Humco Company, soluble in 2-propanol) behaved differently. The percent ash curve Figure 3 exhibited very little slope, hardly sensitive to the dosage of the surfactant. The ash content of the foam fraction was higher than the ash content of the original sludge though. The rise of the other two curves (% filler recovered and % solid recovered) are somewhat less steep until the dosage 0.75% is reached afterwhich they also flatten out.

The quaternary ammonium salt, EHDA-Br is almost completely dissociated in solution. Only a slight amount of hydrolysis takes place to form quaternary ammonium bases. The ionic species are $C_{16}H_{33}C_2H_5(CH_3)_2N^+$ and Br^- (19). At neutral pH both cellulose and clay particles carry negative charges. However the fibers and clay particles, of the primary

clarifier sludge are contaminated by various amounts of divalent and trivalent metal ions (e.g. Ca^{++} , Al^{+++}) and other organic materials which are absorbed on the clay and cellulose surfaces. These materials tend to reduce the charge densities on the clay and cellulose both. To determine the amount of surfactant necessary (from the dissociation constant) it is therefore necessary to measure the potential of the system and that of the individual components i.e. clay and fiber. When too large a concentration of the surfactant is present in water it decreases the bubble-particulate contact angle producing a competition between the non-absorbed surfactant and the particulate for the bubble surface. Hence the foam separation of the particulates decreases.

The organic cation of a quaternary ammonium salt tend to replace the inorganic cations originally present in the clay surfaces, often in amounts exceeding the cation exchange capacity of the clay. This is probably followed by an indiscriminate adsorption on any negatively charged species present, i.e. cellulose. The overall effect will therefore be a reduction in the ash content (due to lower proportion of clay). This fact is evident from the solid contents (or the percent solid recovered) of the foam fractions. It was 39.8% at 0.1% dosage and increased to 49.1% at 0.25% dosage, 69.1% at 0.5% dosage, 75.0% at 0.75% dosage and 76.0% at 1.0% dosage. Based on this reasoning better filler recovery should then be observed at 0.1% and 0.25% dosages. But a decrease in the ash content

(below 48.8%, the ash content of the sludge) is observed at 0.1% dosage and about 50% ash content is observed at 0.25% dosage. At 0.1% dosage it is interesting to note that the percent solid recovered (also percent filler recovered) curve lies below the percent ash curve and all the three curves intersect each other (or meet) at 0.25% dosage after which the percent solid recovery (also percent filler recovered) increases sharply. That means the fiber content in the foam fraction increases.

When a surfactant is absorbed on the clay surface it renders the surface hydrophobic there by reducing the surface tension of water (changing from hydrophilic to hydrophobic) facilitating bubble attachment and finally separation. It is therefore probable that certain degree of hydrophobicity in the nature of the surfactant itself is necessary to achieve certain degrees of foam fractionation. Of course no pine oil was used with EHDA-Br. However, there are not sufficient evidences (in the present study) to support this assumption except that a higher percentage of ash content was obtained with the partially soluble surfactant (Kemamine-Q-6502-C) which is oily (probably coconut oil).

Considering all the above factors involved it is therefore not possible to explain the exact reasons of the inability of the soluble surfactant EHDA-Br to achieve any fractionation in a system such as the primary clarifier sludge.

Flotation With The Selected Cationic Surfactant At Various pH Values (2-12)

Table IV and Figure 4 show that the ash content increased by single stage flotation. The least increase is 13.93% at pH 8 and the maximum increase is 17.83% at pH 3. The ash content of the foam fraction remained insensitive to the effect of pH throughout. So also the filler content of the foam fraction. The percent solid recovered is slightly curved with a peak at about pH 6.3. Following the same pattern one could draw a similar curve for percent filler recovered with a peak at about pH 6.6 and two local minima at about pH 3.3 and pH 10. But that would exclude too many points. Moreover good results are also obtained at pH 3 and pH 9. So a curve with three peaks (at pH 3, 6.5 and 9) is drawn.

The ash content remained insensitive to the change in pH. Since the ZP potential of the system was not measured, it is therefore not possible to state the effect of the magnitude of this potential in this system. In spite of that, there should have been some curvature, had the filler materials been sensitive to pH variation (refer summer work). To say it differently, the foreign materials present (contaminants, metal ions, etc.) reduced the sensitivity of the fillers to pH variation. The peaks in the percent filler recovered curve are due to the increase in solid recovery both clay and fiber. The adsorption therefore can be considered mostly physical for the fillers than electrokinetic. If this assumption is true then removal of these contaminants would increase the sensitivity of the

system to pH variation. Metal ions (divalent or trivalent) will affect clay more than cellulose. In cellulose the main source of charge being due to the adsorption of hydroxyl ions and ionization of weakly acidic groups, the charge of cellulose should increase with increase in pH and decrease to almost zero when the solution is strongly acidic, say around pH 2. There are evidences that clay bears a small negative charge below pH 3.5, though literatures disagree in this respect (16, 17, 19). This explains why equally good fractionation (or increase in ash content) is obtained at pH 3. Beyond this no proper explanation can be given for the peculiar behaviour of the system to pH variation.

Effect of Ball Milling

Ball Milling removes surface coating of slimes (desliming), and reduces the particle size of the filler particles. This way it generates more exchange sites and increases the surface area. It also reduces the fiber length and decreases fiber freeness. It increases the cationic demand of the fibers. The system becomes thereby slightly more sensitive to pH changes. At higher pH the system becomes dispersed and therefore filler recovery decreases. Again all values being maximum at pH 3 show that a flocculated filler with a negative charge less than -10 mv of ZP is necessary for better filler recovery. At this pH value of 3 fiber has very little negative charge. So most of the surfactants are absorbed on the filler surfaces increasing the filler recovery. In this system excess of alum is also present, otherwise the negative ZP could exceed -10 mv and reduce

the effect of recovery as the system would remain somewhat dispersed. The effect of Ball Milling is shown in Figure 5. Of course, the general trend can not be deduced from three points only.

Treatment With Ion Exchange Resin

Most of the metal ions present were removed by the ion exchange resins, rendering the system to a system of almost pure and cellulose fiber. The general trend is impossible to predict from three points only, Figure 6. Again from the diagrams given by Black and Hannah it is evident at pH 3, 6.5 and 9 the negative ZP will be greater than -13 mv and hence rendering the system dispersed and flotation ineffective. The value of the negative ZP being maximum at pH 6.5 (among pH 3, 6.5 and 9) there is comparatively less dispersion at pH 3 and pH 9 than at pH 6.5. Depending upon this assumption it is then obvious that percent filler recovered curve and percent solid recovered curve should follow this trend. And this is actually the case.

Treatment of the sludge with ion exchange resin made the sludge (filler and fiber) sensitive to pH effect which was not observed in the case of the untreated sludge. Again with three points on the curve it is difficult to assess the trend and cause of the trend.

Effect of Sodium Hexametaphosphate

In this study 0.3% sodium hexametaphosphate based on filler was used. No further study was made to determine the various amount of hexametaphosphate necessary to achieve the maximum sequestering effect and

increase in ZP. From Figure 7, with three points it is difficult to predict the general trend. However, it is well known that this level of hexametaphosphate addition can increase the ZP to almost twice the value over a wide pH range. Since fiber and other metal ions are present, with this level of addition it is impossible to state exactly how the hexametaphosphate affected the system. From Figure 7 it can be seen that it increased the sensitivity of the system to pH variation similar to ion exchange resin treatment. The minimum ash content was at pH 6.5. The values at pH 3 and 9 are almost equal to those of the untreated sludge. The percent solid recovered is maximum at pH 6.5 though the percent filler recovered is least at this pH. It is therefore evident that the hexametaphosphate rendered both substrates more electro-negative but with filler exceeding cellulose. This dispersed the filler but solid recovered increased due to the increase in negative charge of the cellulose and probably increased adsorption of the surfactant on cellulose. At low pH, i.e. at pH 3 cellulose has a very small negative charge, so has clay. So the recovery was higher. On the otherhand at pH 9 due to the presence of excess hydroxyl ions the double layer was compacted, adversely affecting the dispersion. Hence recovery was also higher than that of pH 6.5. This is of course an assumption since the potential of the system was not measured. At this pH therefore more surfactant was adsorbed on clay surface and not on cellulose thereby reducing the solid recovery.

In all the three cases the surfactant dosage necessary after treatment was not evaluated. Hence the effect of surfactant concentration (probably less) might be a contributing factor in all these cases.

Brightness

In most cases a higher brightness for the foam and tailings were obtained. In certain instances as high as 75 brightness were obtained compared to 71 of the original sludge. None of the fractions were however bleached to observe further increase in the brightness.

The Insoluble Surfactant Kemamine-Q-1902-C

Since the effects of ball milling, ion exchange resin, and sodium hexametaphosphate were not studied at different pH values, but only at pH 10.5, nothing definite can be stated as to the results obtained except that lower ash content values were obtained in all the three case compared to the control. Slightly higher percentage of filler was recovered in case of sodium hexametaphosphate followed by ion exchange resin.

SUMMARY AND CONCLUSIONS

1. With single stage flotation and using a partially water soluble cationic surfactant (Kemamin-Q-6502-C) it was possible to increase the ash content of the foam fraction by about 17-18 percent and recover about 90-92 percent of the filler materials. But the ash content curve remained practically insensitive to pH variation.
2. Treatment of the sludge by ball mill, ion exchange resin and sodium hexametaphosphate rendered the system more sensitive to pH variation.
3. Considering the ash content, solid recovery and filler recovery, all three together, best results were obtained by ball milling and floating the sludge at pH 3. In this case the ash content was raised to 71.5% compared to the original ash content of the sludge 51.67%. About 91% of the filler was recovered. At this pH value without ball milling the ash content of the foam fraction was 69.5% (2% less) with a filler recovery of 89.1%. However, when the pH was raised to 9 lower results were obtained by ball milling compared to the sludge which was not treated in the ball mill.
4. Ion exchange resin treatment lowered the results at pH 3, 6.5 and 9 compared to the results of the untreated sludge. Among the above three pH values lowest results were obtained for pH 6.5 probably due to dispersion effect.

5. Compared to the untreated sludge, the values for the sludge treated with sodium hexametaphosphate were lower only at pH 6.5. The values at pH 3 and 9 were almost the same as that of the untreated sludge.

No improvement was obtained at this level of sodium hexametaphosphate addition.

6. Removal of metal ions by ion exchange resin or sequestering by hexametaphosphates render the system more sensitive to pH variation. In certain cases probably cause dispersion and hence reducing effective fractionation.
7. To know the exact effect of the three treatments (ball milling, ion exchange resin and sodium hexametaphosphate) the system should be studied at all pH values and not just at selected pH values.
8. Particle size is important for the froth flotation to be effective.
9. It is essential that the potential (ZP) of the system (and the components if possible) be measured by some suitable and convenient means (e.g. mass transfer cell).
10. At 10.5 the flotation results were better for the partially soluble surfactant than that of the totally insoluble surfactant Kemamine-Q-1902-C.

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APPENDIX I

Dosage Study

A - Flotation (single stage) with a water soluble cationic surfactant

EHDA-Br (Ethylhexadecyldimethyl ammonium bromide).

TABLE A-I

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	8.32	44.30	52.12	39.8	36.17	70.8
Tailings	12.56	52.26	61.48	60.0	62/2	68.2
Total	20.88			99.8	98.37	

Legend:

Dosage:	0.1%
pH:	7
Consistency of the sludge:	1.81%
Ash in the sludge:	48.8 %
Filler in the sludge:	57.41%
Brightness of the sludge (% elrepho)	70.9
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	10 minutes

TABLE A-II

<u>Fraction</u>	<u>Amount of</u> <u>S.S. g</u>	<u>% Ash</u>	<u>% Filler</u> <u>(purity)</u>	<u>% Solid</u> <u>Recovered</u>	<u>% Filler</u> <u>Recovered</u>	<u>Brightness</u> <u>(% Elrepho)</u>
Foam	8.70	50.85	59.82	49.1	51.2	71.5
Tailings	9.00	47.67	56.10	50.0	57.6	68.3
Total	17.70			99.1	98.6	

Legend:

Dosage: 0.25%
 pH: 7
 Consistency of the sludge: 1.81%
 Ash in the sludge: 48.8 %
 Filler material in the sludge: 57.41%
 Brightness of the sludge (% elrepho) 70.9
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of Flotation: 12 minutes

TABLE A-III

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.20	48.48	57.04	69.10	68.65	69.0
Tailings	5.90	48.59	57.16	30.0	30.10	68.8
Total	19.10			99.10	98.75	

Legend:

Dosage: 0.5%
 pH: 7
 Consistency of the sludge: 1.81%
 Ash in the sludge: 48.8 %
 Filler material in the sludge: 57.41%
 Brightness of the sludge (% elrepho): 70.9
 Air Flow Rate 1.5-1.75 l/min.
 Duration of Flotation: 13 minutes

TABLE A-IV

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% El repho)</u>
Foam	14.70	47.5	55.9	75.0	74.5	70.0
Tailings	4.9	49.0	57.6	25.0	25.0	68.6
Total	19.60			100.0	99.5	

Legend :

Dosage: 0.75%
 pH: 7
 Consistency of the sludge: 1.81%
 Ash in the sludge: 48.8 %
 Filler in the sludge: 57.41%
 Brightness of the sludge (% elrepho): 70.9
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of Flotation: 13 minutes

TABLE A-V

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	14.61	45.5	53.5	76.0	73.5	70.5
Tailings	4.59	51.0	60.0	23.9	25.5	69.0
Total	19.20			99.9	99.0	

Legend:

Dosa ge: 1.0%
 pH: 7
 Consistency of the sludge: 1.81%
 Ash in the sludge: 48.8 %
 Filler in the sludge: 57.41%
 Brightness of the sludge (% elrepho): 70.9
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of Flotation: 14 minutes

B. Flotation (single stage) with a partially water soluble cationic surfactant, Kemanine - Q - 6502 - C*

TABLE B-I

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	11.90	67.6	79.5	59.5	77.6	73.0
Tailings	8.00	25.9	32.0	40.2	20.1	72.8
Total	19.9			99.7	97.7	

Legend:

Dosage:	0.1%
pH:	7
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness of the sludge (% elrepho):	71.0
Air Flow Rate:	1.50 - 1.75 l/min.
Duration of Flotation:	7 minutes

* Product of Humco

TABLE B-II

<u>Fraction</u>	<u>Amount of</u> <u>S.S.(g)</u>	<u>% Ash</u>	<u>% Filler</u> <u>(purity)</u>	<u>% Solid</u> <u>Recovered</u>	<u>% Filler</u> <u>Recovered</u>	<u>Brightness</u> <u>(% Elrepho)</u>
Foam	13.19	68.15	80.17	63.78	84.16	73.6
Tailings	7.49	20.48	24.09	36.0	14.14	73.2
Total	20.68		104.26	99.78	98.30	

Legend:

Dosage:	0.25%
pH:	7
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness of the sludge (% el repho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	8 minutes

TABLE B-III

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.56	67.03	78.86	68.97	89.45	73.2
Tailings	6.10	13.04	15.34	31.00	10.00	72.4
Total	19.66		94.20	99.97	99.45	

Legend:

Dosage:	0.5 %
pH:	7
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness of the sludge (% elrepho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	8 minutes

TABLE B-IV

<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.56	68.45	80.53	70.04	92.77	72.0
Tailings	5.80	6.96	8.19	29.0	6.0	73.8
Total	19.66			99.04	98.77	

Legend:

Dosage: 0.75%
 pH: 7
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness of the sludge (% elrepho): 71.0
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of Flotation: 8 minutes

TABLE B-V

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.43	69.77	82.10	67.15	90.62	73.0
Tailings	6.57	13.05	15.25	32.00	8.18	73.0
Total	20.00		97.35	99.15	98.80	

Legend:

Dosage:	1.0%
pH:	7
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness of the sludge (% elrepho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	8 minutes

APPENDIX 2

Flotation of primary clarifier sludge with a cationic surfactant* at various pH values.

TABLE I

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.01	67.0	78.50	65.5	84.9	71.2
Tailings	6.88	20.4	24.0	34.0	14.0	74.5
Total	19.89			99.5	98.9	

Legend:

Dosage:	0.75%
pH:	2
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrepho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	5 minutes

* Kemanine - Q - 6502-C, a Humco product

TABLE II

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.50	69.5	81.70	67.1	89.1	71.5
Tailings	6.56	12.82	15.1	32.1	9.6	75.0
Total	20.06			99.2	98.7	

Legend:

Dosage: 0.75%
 pH: 3
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.57%
 Filler in the sludge: 60.79%
 Brightness in the sludge (% elrepho): 71.0 %
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of flotation: 5 minutes

TABLE III

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.51	66.8	78.60	66.8	86.8	72.2
Tailings	6.70	14.66	17.25	33.0	12.1	75.0
Total	20.21			99.8	98.9	

Legend:

Dosage: 0.75%
 pH: 4
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness in the sludge (% elrepho): 71.0%
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of flotation: 5 minutes

TABLE IV

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.87	67.5	79.3	68.3	89.1	71.0
Tailings	6.42	14.0	16.5	31.1	9.6	68.0
Total	20.29			99.4	98.7	

Legend:

Dosage: 0.75%
 pH: 5
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness of the sludge (% Elrepho): 71.0
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of Flotation: 5 minutes

TABLE V

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.93	68.6	80.8	70.0	92.6	71.0
Tailings	6.01	10.6	12.4	29.4	6.2	71.5
Total	19.94			99.4	98.8	

Legend:

Dosage: 0.75%
 pH: 6
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness in the sludge (% elrepho): 71.0 %
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of Flotation: 6 minutes

TABLE VI

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.56	68.45	80.53	70.0	92.8	72.0
Tailings	5.80	6.96	8.19	29.6	6.0	73.8
Total	19.36			99.6	98.8	

Legend:

Dosage:	0.75%
pH:	7
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrepho):	71.0%
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of flotation:	7 minutes

TABLE VII

<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.10	65.6	77.2	67.0	85.0	70.0
Tailings	6.48	19.2	22.6	32.45	13.2	73.3
Total	19.58			99.45	98.2	

Legend:

Dosage:	0.75%
pH:	8
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrepho):	71.0%
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	6 minutes

TABLE VIII

<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	14.0	68.60	80.59	69.6	88.2	73.0
Tailings	7.0	17.24	20.21	30.0	10.1	69.4
Total	21.0			99.6	98.3	

Legend:

Dosage: 0.75%
 pH: 9
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness of the sludge (% elrepho): 71.0
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of flotation: 7 min.

TABLE IX

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.10	65.9	77.52	65.2	82.7	73.0
Tailings	7.04	21.12	24.85	34.6	16.2	74.5
Total	20.14			99.8	98.9	

Legend:

Dosage: 0.75%
 pH: 10
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness in the sludge (% elrepho): 71.0%
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of Flotation: 6 minutes

TABLE X

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.62	66.5	78.25	67.7	86.8	71.0
Tailings	6.55	18.12	21.36	32.0	12.1	75.0
Total	20.16			99.7	98.9	

Legend:

Dosage:	0.75%
pH:	11
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrepho):	71.0 %
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	7 minutes

TABLE XI

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.80	67.23	79.10	68.0	88.0	71.0
Tailings	6.50	13.67	16.12	31.6	10.1	73.0
Total	20.30			99.6	98.1	

Legend:

Dosage:	0.75%
pH:	12
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrehpo):	71.0 %
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of flotation:	5 minutes

APPENDIX - 3

A. Effect of Ball Milling

TABLE A I

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.32	71.6	84.2	65.84	91.22	71.0
Tailings	6.91	10.72	12.62	33.00	7.18	71.0
Total	20.23			98.84	98.40	

Legend:

Dosage:	0.75%
pH:	3
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrepho):	71.0 %
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of Flotation:	5 minutes

TABLE A II

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	11.62	71.0	83.58	57.66	79.24	71.5
Tailings	8.53	25.78	30.33	42.0	20.0	71.5
Total	20.15			99.66	99.24	

Legend:

Dosage: 0.75%
 pH: 6.5
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness in the sludge (% elrepho): 71.0 %
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of flotation: 6.5 minutes

TABLE A III

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% elrepho)</u>
Foam	13.26	67.0	78.8	65.44	84.86	71.6
Tailings	7.00	23.3	27.5	34.00	14.0	71.3
Total	20.26			99.44	98.86	

Legend:

Dosage: 0.75%
 pH: 9
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness in the sludge (% elrepho): 71.0 %
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of flotation: 5 minutes

B. Effect of Ion Exchange Resin

TABLE B I

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	11.80	63.74	75.0	61.64	76.02	70.0
Tailings	7.34	24.33	28.6	37.04	22.0	70.5
Total	19.14			98.68	98.02	

Legend:

Dosage:	0.75%
pH:	3
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrepho):	71.0 %
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of flotation:	4.5 minutes

TABLE B II

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	11.72	59.89	70.47	59.68	69.17	71.0
Tailings	7.91	33.36	39.20	40.0	29.0	70.0
Total	19.63			99.68	98.17	

Legend:

Dosage: 0.75%
 pH: 6.5
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness of the sludge (% elrepho): 71.0 %
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of flotation: 6 minutes

TABLE B III

<u>Fraction</u>	<u>Amount of S. S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	12.04	64.03	75.33	64.72	80.21	71.6
Tailings	6.55	18.91	22.22	35.0	17.0	70.0
Total	18.59			99.72	97.21	

Legend:

Dosage: 0.75%
 pH: 9
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness in the sludge (% elrepho): 71.0 %
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of flotation: 5 minutes

C. Effect of Sodium Hexametaphosphate

TABLE C I

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.04	69.76	82.07	63.09	85.20	71.6
Tailings	7.61	22.34	26.30	36.0	13.40	71.5
Total	20.65			99.09	98.60	

Legend:

Dosage:	0.75%
pH:	3
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrepho):	71.0 %
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of flotation:	6.5 minutes

TABLE C II

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	14.43	61.50	72.36	70.22	83.56	72.0
Tailings	6.14	28.25	33.1	29.20	15.0	72.0
Total	20.57			99.42	98.56	

Legend:

Dosage: 0.75%
 pH: 6.5
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness in the sludge (% elrepho): 71.0 %
 Air Flow Rate 1.5 - 1.75 l/min.
 Duration of flotation: 5 minutes

TABLE C III

<u>Fraction</u>	<u>Amount of S.S.(g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.56	69.18	81.38	65.96	88.27	71.0
Tailings	7.0	17.8	20.95	33.14	10.33	72.8
Total	20.56			99.10	98.40	

Legend:

Dosage:	0.75%
pH:	9
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness in the sludge (% elrepho):	71.0 %
Air Flow Rate:	1.5 - 1.75 l/min.
Duration of flotation:	6 minutes

APPENDIX 4

Flotation with insoluble cationic surfactant Kemamine-Q-1902-C at pH 10.5

TABLE I
Untreated Sludge (Control)

<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	12.6	67.0	78.82	64.28	83.35	66.0
Tailings	7.0	20.0	23.53	35.7	16.0	67.0
Total	19.6			99.98	99.35	

Legend:

Dosage:	1.0%
pH:	10.5
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness of the sludge (% elrepho):	71.0
Air Flow Rate	1.5 - 1.75 l/min.
Duration of flotation:	6.5 min.

TABLE II
Effect of Ball Milling

<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	10.84	61.46	72.31	57.63	68.43	67.0
Tailings	8.0	38.52	45.32	42.37	31.60	66.8
Total	18.84			100	100.03	

Legend:

Dosage:	1.0%
pH:	10.5
Consistency of the sludge:	5.29%
Ash in the sludge:	51.67%
Filler in the sludge:	60.79%
Brightness of the sludge (% Elrepho):	71.0
Air Flow Rate	1.5 - 1.75 l/min.
Duration of flotation:	5 min.

TABLE III

Effect of Ion Exchange Resin

<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	14.05	64.85	76.30	70.25	86.0	67.0
Tailings	6.45	20.7	24.35	29.75	4.00	67.5
Total	20.50			100.00	100.00	

Legend:

Dosage: 1.0%
 pH: 10.5
 Consistency of the sludge: 5.29%
 Ash in the sludge: 51.67%
 Filler in the sludge: 60.79%
 Brightness of the sludge (% elrepho): 71.0
 Air Flow Rate: 1.5 - 1.75 l/min.
 Duration of flotation: 6 min.

TABLE IV
Effect of Sodium Hexametaphosphate

<u>Fraction</u>	<u>Amount of S.S. (g)</u>	<u>% Ash</u>	<u>% Filler (purity)</u>	<u>% Solid Recovered</u>	<u>% Filler Recovered</u>	<u>Brightness (% Elrepho)</u>
Foam	13.40	61.65	72.53	74.23	88.54	66.6
Tailings	4.65	19.62	23.10	25.77	11.46	67.0
Total	18.05			100.00	100.00	

Legend:

Dosage:	1.0%
pH:	10.5
Consistency of the sludge:	5.29%
Ash in the sludge:	51.61%
Filler in the sludge:	60.79%
Brightness of the sludge (% Elrepho):	71.0
Air Flow Rate:	1.5 - 1.75 l/min
Duration of flotation:	6.5 min.

APPENDIX - 5

An approximate calculation of charge densities:

	<u>CLAY (KAOLINITE)</u>	<u>CELLULOSE</u>
Specific Surface Area: (B.E.T. Method)	$22 \frac{\text{m}^2}{\text{g}}$	$130* \frac{\text{m}^2}{\text{g}}$ (a typical value)
ion exchange capacity:	$9 \frac{\text{me}}{100\text{g}} (\text{NaOH})$	$\frac{6 \text{ m.m.}}{100\text{g}} (\text{Ca}^{++})$
Charge Density:	$9 \frac{\text{me}}{2,200 \text{ m}^2}$	$12 \frac{\text{me}}{13,000 \text{ m}^2}$
i.e.	$4.09 \times 10^{-3} \frac{\text{me}}{\text{m}^2}$	$0.92 \times 10^{-3} \frac{\text{me}}{\text{m}^2}$

Hence the charge density on clay is higher than cellulose fiber

* Source: Merchant, M.V., "A Study of Water Swollen Cellulose Fibers Which Have Been Liquid Exchanged and Dried From Hydrocarbons", Tappi, Vol. 40, No. 9, (1957), p 773.