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## The Use of Electrocoagulation as a Flocculating Aide for Deinking

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THE USE OF ELECTROCOAGULATION  
AS A FLOCCULATING AIDE  
FOR DEINKING

by

James L. Schram

A Thesis submitted  
in partial fulfillment of  
the course requirements for  
The Bachelor of Science Degree

Western Michigan University

Kalamazoo, Michigan

December, 1981

## ABSTRACT

The effects of electrocoagulation as a flocculating aide for deinking was evaluated using a "homemade" electrode unit. The unit was inserted into a laboratory-size Voith-Morden forced-air flotation cell. The process of electrocoagulation involves two steps:

1. Reducing the zeta potential of the system to 0.0 mV by pH adjustment.
2. The passing of current through the stock suspension which causes the electrolysis of the water, and thus hydrogen and oxygen gas bubbles are produced. These gas bubbles collect ink particles to form solid-gas composites which can be removed more easily using flotation.

The zero zeta potential eliminates the naturally occurring negative charges on the particles, and thus promotes better flocculation.

The process was evaluated on the forced-air flotation process using brightness and microscope analysis. A relationship of deinking efficiency and shive analysis was sought, but did not prove informative. The results of the microscopic analysis and brightness measurements of the electrocoagulated stock illustrated the process had a detrimental effect on forced-air flotation system. It was determined, with further investigation, that the acid pH of the system was what was causing the detrimental effects. This suggested a more in depth look at the effects of pH on deinking; however, time was not available. The process could not be carried out under alkaline conditions due to the plating of ink on the electrodes and the degradation of the aluminum under alkaline conditions.

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## INTRODUCTION

This thesis is dedicated to the research and experimentation needed to justify the use of electrocoagulation as a flocculating aide for deinking.

Electrocoagulation involves the passing of an electric current through the water containing suspended solids, emulsified liquids, or dissolved solids. The main purpose of the current is to cause the electrolysis of the water present, thus producing hydrogen and oxygen gas bubbles. These micro-bubbles will collect the agglomerated ink particles to form gas-solid composites. These gas-solid composites will in turn allow for easier flotation of the material being separated. A prerequisite of the electrocoagulation process is the adjustment of the zeta potential of the material being separated to approximately 0.0 volts  $\pm$  5.0 mV. The reasons for adjusting the zeta potential is twofold:

- 1) Eliminate the naturally occurring repelling forces between particles such that the particles will form agglomerates more easily.
- 2) Eliminate the naturally occurring negative charge on the particles so they do not electroplate to the positive electrodes.

To determine the feasibility of using electrocoagulation as a flocculating aide for deinking, a model electrode unit will be constructed which can be inserted into a laboratory-size Voith-Morden forced air flotation cell. Test runs will be conducted to analyze its capabilities of performing as a deinking aide.

To determine how the electrocoagulation process performs as a flocculating aide, the author will compare electrocoagulation as a flocculating aide with the use of chemicals as a flocculating aide.

Test runs will be made using each of the preceding flocculating aides on pulped printed ledger. To remove the flocculated ink agglomerates, a Voith-Morden forced air flotation cell will be used. The deinked stock will be made into several handsheets and by using visual analysis, G. E. Brightness, and a shive analyzer, the stock can be analyzed to determine the effectiveness of each process.

In constructing the electrode unit, several test runs will be made to determine the most effective unit parameters. The most efficient stock consistency, electrode spacing, and stock retention time are the three parameters that must be found to produce an effective process.

## BACKGROUND

There is an ever-growing need for clean and recycled fiber in the pulp and paper industry. With raw materials dwindling away and the price of virgin pulp on an upswing, an alternative fiber resource must be found. A possible alternative fiber resource is recycled waste paper. A main problem with using recycled paper is the removal of undesirable contaminants such as inks, plastics, metals, stickies, hot melts, etc. There are several processes being utilized to remove these contraries, however, none of them are 100% effective and there is much room for improvement.

Ink removal has always been a main concern in recycling waste since there is no efficient means of removing the ink from a system. In recent years, a method known as forced air flotation has been developed and has shown great promise in the field of deinking. The process, however, is not 100% effective and it is the intention of the author to work with this type of deinking in conjunction with a newer process of contaminant removal known as electrolytic separation. The particular electrolytic process to be worked with is a proprietary operation of the Dravo Corporation. Its trade name is Lectro Clear Separation Process. This particular process has been used commercially in many fields of industry and these are a few applications:

- Acid Mine Drainage
- Algae Removal
- Broiler (Chicken Plant) Wastes
- Candy Manufacturing Wastes
- Clay and Minerals Processing Wastes
- Coal Preparation and Coal Washing Wastes
- Deinking Wastes
- Edible Oil Refining Wastes
- Heavy Metal Removal
- Industrial Laundry Wastes
- Meat Packing Wastes

- Paper Mill Wastes
- Petroleum and Petrochemical Wastes
- Tank Car and Truck Washing Wastes
- Textile Dyeing and Printing Wastes

Although deinking is listed as an application, it has been found by the author that there has been no fundamental work done with deinking using the Lectro Clear process. It is the purpose of this thesis to do such work.

The process of deinking by flotation, whether it be by conventional forced air flotation or by the Lectro Clear process, can be divided into three separate stages. The stages are the same for both processes, but it is the method by which these stages are accomplished that differ from one to the other. The particular stages of deinking are:

- 1) The ink must be detached from the fiber in the shortest time possible with a maximum detachment. It is very important that this be carried out with a minimal detrimental effect on the fiber.
- 2) The separated ink particles must be coagulated into flocs which can be removed with ease.
- 3) The ink must finally be removed from the pulp slurry by floating the ink to the surface and then skimming the ink from the surface. This step must be done with a minimum removal of fiber.

Ink is not readily removed from the fiber using water and mechanical action and thus alkaline is used to promote detachment. A common chemical used to promote detachment is caustic soda. Heat and mechanical action are also used to aid the detachment process. It should be noted that both the conventional flotation process and the Lectro Clear process use similar methods to disperse the ink into solution.

The next step requires the flocculation of the dispersed ink into flocs that can be removed by flotation. In the conventional flotation process, flocculation is mainly accomplished by chemical means. A flo-



tation agent, which is usually added at the repulper, is used to create flocculation. The flotation agent consists of two long molecular chains. One part is a hydrophobic group (water-hating) which reacts with the water hardness to form precipitated calcium salts. These precipitated flakes act as collectors which collect ink particles and dispersed air bubbles. These solid-gas composites are then capable of floating to the surface. The second molecular group is a hydrophilic group (water-loving) which is used to reduce the surface tension of the solution. A reduced surface tension produces a capability for the ink and dispersed air to form an agglomerate more easily.

The primary principle of electrocoagulation is that current is introduced into a pulp stock and water slurry which contains ink particles. The current causes the electrolysis of the water present and oxygen and hydrogen gas bubbles are produced at the electrodes. Hydrogen gas is produced at the negative electrodes, the cathodes, and oxygen gas is produced at the positive electrodes, the anodes. This production of microbubbles causes agitation of the stock slurry and these bubbles also collect ink particles to form gas-solid composites which can be removed by flotation methods more easily. The agitation of the stock slurry increases the chances of the ink particles to collide with one another and form flocs of ink particles. These flocs of ink are desirable because they have a better chance of attaching to a gas bubble for flotation. A prerequisite of the electrocoagulation process is the adjustment of the stock slurries zeta potential to approximately 0.00 mV. The purpose of a 0.00 mV zeta potential is to eliminate the naturally occurring negative charges on the ink parti-

cles. The reason for eliminating the negative charges on the ink particles is twofold:

- 1) Since all the particles are the same charge, they will tend to repel one another and flocs of ink will not form. By eliminating the charges and neutralizing the system of ink, the ink will attract to one another on collision and flocs of ink can form.
- 2) Since most particles in a solution are negatively charged and there are both positive and negative electrodes in the coagulation cell, the ink particles will electroplate to the positive electrodes. By eliminating the charges on the ink particles, they will not electroplate to either electrode.

The zeta potential of a solution can be adjusted by adjusting the solutions pH. It has been found experimentally that a pH of 4.0 will produce a zeta potential of approximately 0.0 mV. A zero zeta potential means a minimum surface tension and thus gas bubble-particulate composites can form easily. It is in the author's opinion that if electrocoagulation is used in parallel with a flotation agent, the efficiency of either conventional flotation or the Lectro Clear process will be increased.

Once the contaminants are flocculated and stable solid-gas composites are formed, they must be removed from the solution. In conventional forced air flotation, air is forced into the solution. As this air rises to the surface, it collects the contaminants on its ascent. Once at the surface, the froth is skimmed from the surface and removed. In using the electroflotation method of floating the ink, current is introduced to the solution by way of electrodes. This current in turn produces oxygen and hydrogen gas bubbles which float to the surface carrying with it the agglomerated ink particles. This floating froth is then removed from the surface by skimming the surface.

## EXPERIMENTATION

### Construction of the Electrocoagulation System

It was the original intent of the author to design, engineer, and construct an electrocoagulation cell. However, due to the unavailability of the materials needed, the high material costs, and the presence of time constraints, an alternative mechanism was required to perform the experimental electrocoagulation operations. Since the main problem encountered was the construction of the cell itself, a new design was made that eliminated the cell. The new design utilized the available laboratory-size Voith-Morden forced air flotation unit in such a way that an electrode unit could be inserted into the flotation unit.

The new mechanism was much less expensive, required less construction time, and was more convenient for laboratory operations. The main difference with the new design compared to the original design was the original was designed for continuous operation and the new design was designed for batch operations.

The electrode insert was constructed using 32 feet of  $\frac{1}{4}$ -inch diameter aluminum rod, one 8-inch by  $\frac{1}{2}$ -inch by 60-inch pine board, and 20 feet of 14-gauge copper wire. The aluminum rod was cut into 34 11-inch pieces, which were used as the electrodes for the insert. Aluminum was chosen because of its low cost, good conductance and its resistance to degradation in an acid system. Both ends of each electrode were drilled to accommodate a 6- by  $\frac{3}{8}$ -inch self-tapping sheet metal screw. These screws were used to fasten a wire to each electrode so the electrodes could be connected together. The pine board was cut into six pieces, 6 inches wide by  $9\frac{1}{4}$  inches long, which acted as three sets

of end plates for the coagulation unit. Each set of end plates was drilled to accommodate the electrodes and hold them in place. A typical illustration of these end plates is shown in Figure 1. Each set of end plates accommodated 24 electrodes or 12 electrode pairs. There are three tiers of electrodes with a one-inch spacing between tiers. Each tier was offset from the adjacent tier to give maximum coverage by the electrodes. The electrode pairs are separated by one inch on either side. Each set of end plates designates the spacing between the anode and cathode of each electrode pair. The following electrode spacings were dictated by the end plates: (1)  $1/4$  of an inch, (2)  $1/8$  of an inch, (3)  $1/16$  of an inch. Wood was chosen as the end plate material because of its low cost, good insulating ability, and ease of handling. An illustration of the electrode insert is shown in Figure 2.

The wiring of the electrode unit was very simple. An electrode from each electrode pair was connected together in series to form one long electrode that weaved back and forth throughout the electrocoagulation unit. A wire was then connected from the last electrode of the series to the positive terminal of the battery. This series of electrodes acted as the anode of the system. The other electrode of each electrode pair was connected together to form a series of electrodes which weaved back and forth parallel to the anode. A wire was connected from the first electrode of the series to the negative terminal of the battery. This series of electrodes acted as the cathode of the system. It should be noted that the battery terminals were connected at opposite ends of the electrode unit. This allowed for an equal current through each electrode. Figure 2 illustrates the wiring diagram.

FIGURE 1  
(Electrode unit)

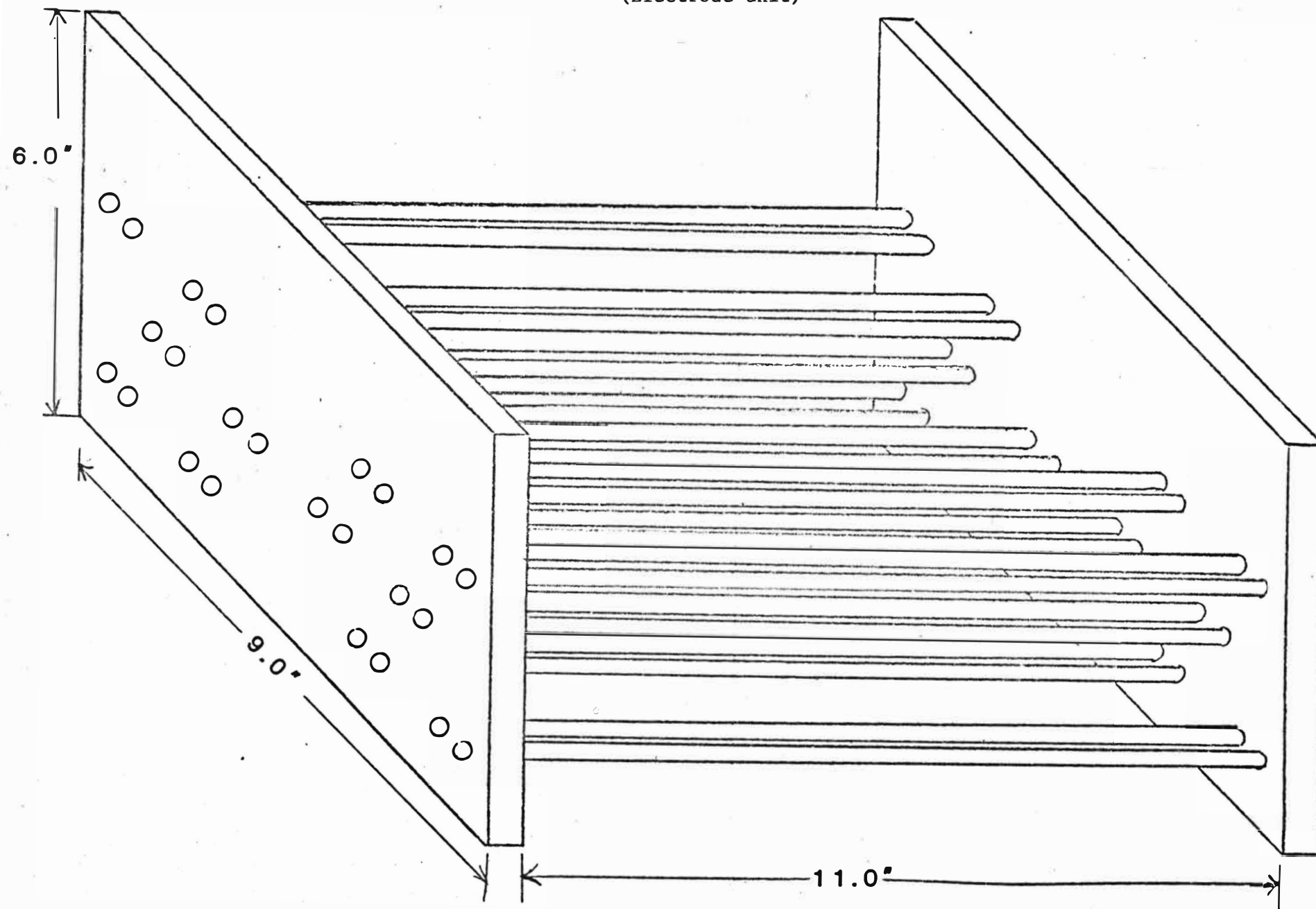
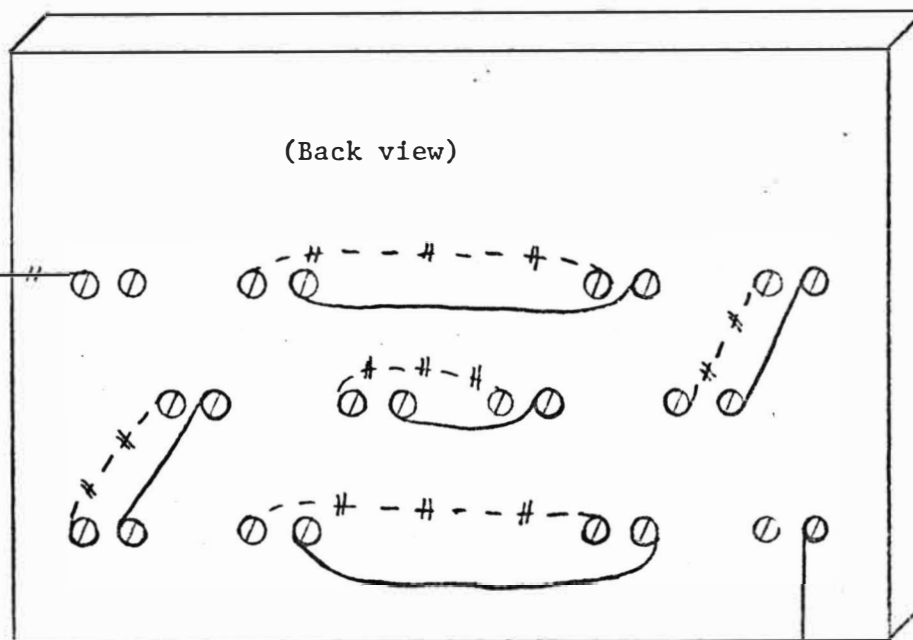
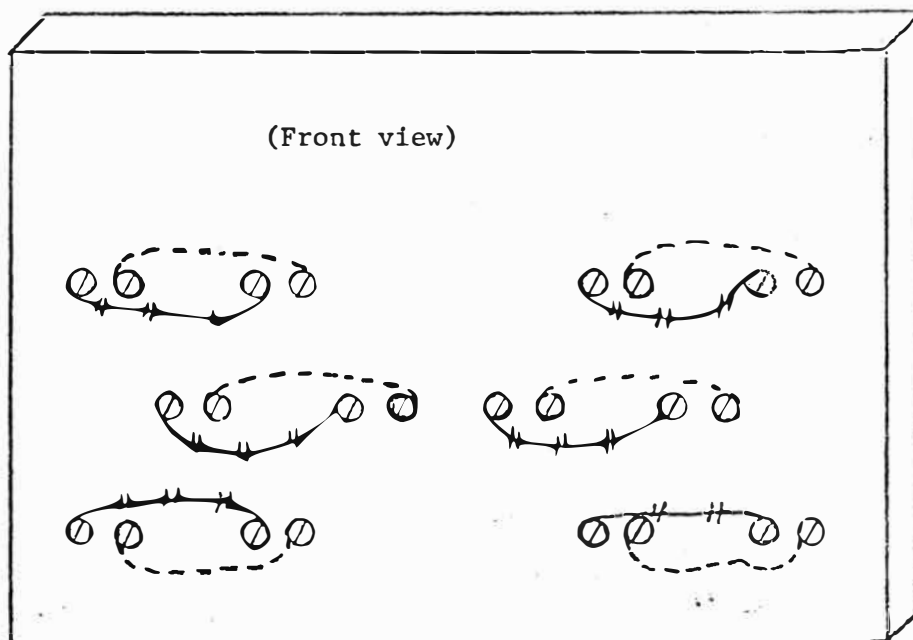


FIGURE 2  
(Wiring diagram)



An automobile battery was used as the power supply which produced 12 volts D.C. across its terminals at approximately 60 amperes.

The only preliminary experimentation that involved the electrode unit was that needed to determine the spacing between anode and cathode. To determine the most efficient spacing, the bubble quantity and quality was looked at for three different spacings. The three different sets of end plates dictated the spacing. The different electrode setups were run in clear water so the bubble formation and quantity could be observed. The  $1/16$  of an inch spacing was looked at first. There were several problems with the wires connecting the electrodes, which kept coming in contact with each other and thus shorting out the circuit. Once the circuit was completed properly, the bubble formation was very good and bubble size was very small at the electrodes, but formed larger bubbles at the surface. The  $1/8$  of an inch spacing produced no noticeable difference in bubble quality and quantity. The same quality and quantity of bubbles was produced using a  $1/4$  of an inch spacing. However, with the  $1/4$  of an inch spacing, the problem with the end wires short-circuiting was eliminated. For this reason, a  $1/4$  of an inch spacing was chosen.

There were two things noticed while doing this preliminary work. It was noticed that the retention time of two minutes, which was thought to be an efficient time from literature studies, would not be sufficient. A time of approximately 20 minutes seemed more reasonable. It was also noticed that a device would be needed for agitation since the formation of bubbles was not rapid enough to cause sufficient agitation.

### Electrocoagulation and Flotation Experimentation

Once the electrocoagulation unit was constructed and tested for operating difficulties, the preliminary experimentation could be conducted.

The first operation of the experimentation was the pulping of the stock which was used for the trial runs. The stock was printed ledger donated by a Western Michigan University Recycling Pilot Plant client. The type of printing was unknown; however, all of the stock had the same type of printing. There were three different pulpings made, all of them conducted with the same equipment under the same conditions. Two of the pulpings were done using the printed ledger which contained ink, while one pulping was done using border and margin clippings of the ledger so that no ink was present.

The stocks were pulped in a Voith-Morden slush maker at 160° F and 5.0% consistency. Prior to pulping, 3.0% of caustic soda was added to the pulping water to aid fiber and ink disassociation. During the first pulping, samples of the slurry were taken from the pulping material every five minutes. The samples were diluted to a very low consistency and agitated in a viewing beaker. The stock was observed for bundles of fibers and ink. It was observed that after 20 minutes of pulping, a totally defibered stock slurry existed. A 20-minute pulping time was then used for all of the pulpings. A pH of the pulp was taken for reference purpose and found to be approximately 11.0.

After the stock was pulped, two one-liter samples of the inked and uninked pulp were taken so handsheets could be made and a shive analysis done. G. E. Brightness and a microscopic analysis of the handsheets were done. The results of the visual analysis, the brightness



measurements, and the shive analysis gave results of the two extremes-- a totally inked stock and a totally uninked stock. These results were the standards to which the different coagulation and flotation operations could be compared.

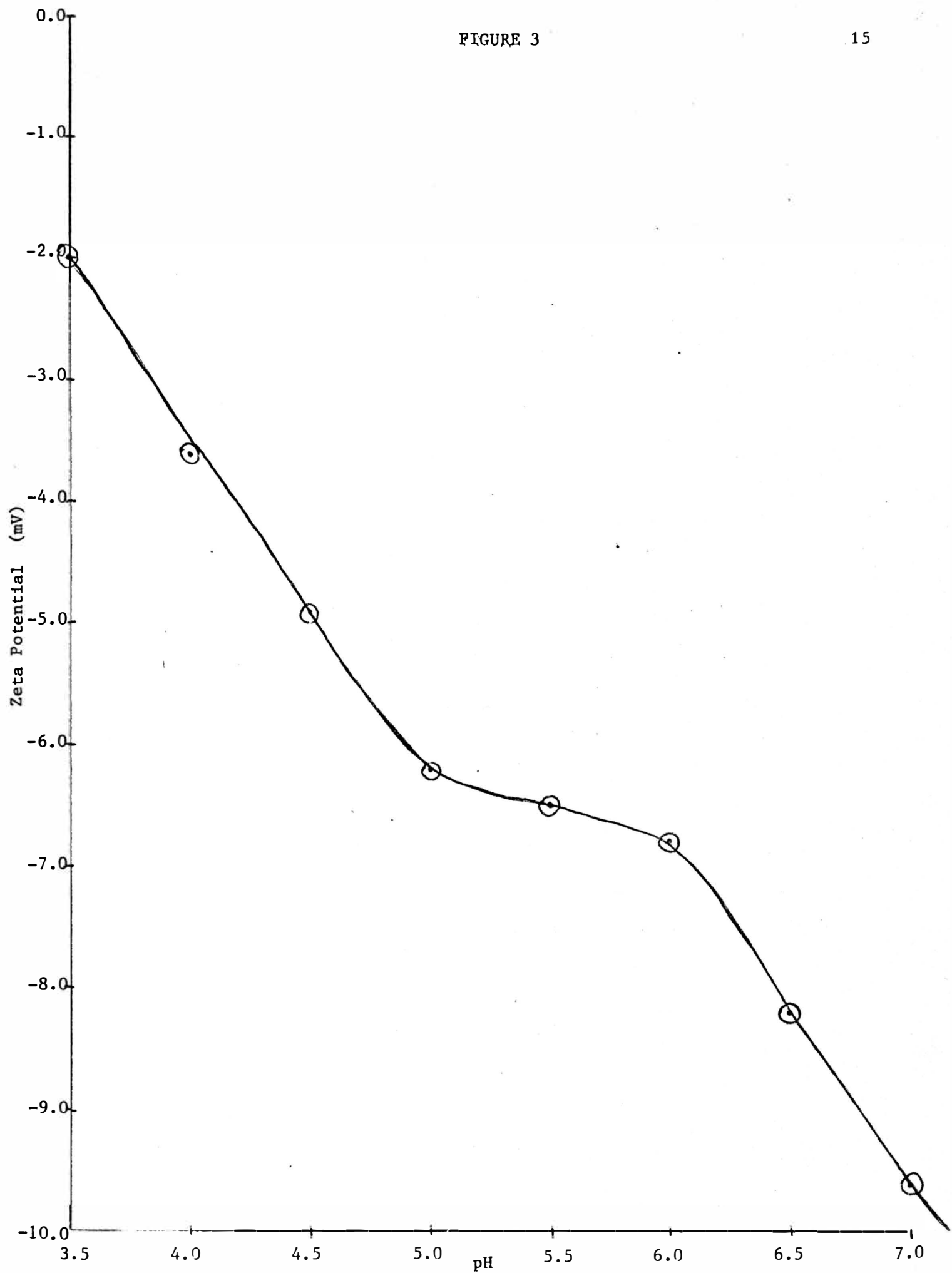
The next step was determining the correct pH for the pulp slurry that would give approximately a 0.00 mV zeta potential. For the electrocoagulation process, a  $0.00 \text{ mV} \pm 5.0 \text{ mV}$  zeta potential is required. A Laser Zee Meter was used to determine the zeta potential. Since the object of this thesis is concerned mainly with coagulating ink particles, it would be ideal to find a 0.0 mV zeta potential of the ink particles only. However, since the stock system contains ink, fibers, clays, and other additives which cannot be separated from one another totally, this was impossible. It should also be noted that the Zee Meter cannot differentiate between the particles present, and since the particles may have different size charges on them, an average zeta potential of the whole system was taken.

The first step in acquiring a sample for the Zee Meter was to separate as much of the ink from the other components of the system as possible. This was accomplished using conventional forced air flotation. The Voith-Morden flotation cell was set up and 15 liters of inked stock at 0.80% consistency was added to it. A chemical flocculating aide named Triton X-100, a product of Rohm and Haas Company, was added to the system. In the author's previous experience in working with the flotation unit, it was found that 0.1% of Triton X-100 was sufficient for good foam quantity and formation. The flotation device was run for 15 minutes. During this time, the froth was skimmed from the surface and collected in a beaker. The froth contained a majority

of ink particles along with some fiber and additives. The collected froth was diluted and the foam was allowed to break down. This solution was passed through a 100 mesh screen, which removed a majority of the fibers present. This sample was then used for the zeta potential tests. The sample was divided up into eight 100 ml samples and their pH was adjusted using 10% sulfuric acid. Each sample was adjusted to a different pH, ranging from 7.0 to 3.5 at intervals of 0.5. Three zeta potential measurements were obtained for each sample. The three measurements for each pH were averaged and the results were plotted against pH. The graph shown in Figure 3 shows that a zeta potential of 0.0 mV was never obtained with the pH's that were used. It was noticed that the systems zeta potential did not vary much at the different pH's. By using the graph and what was found in literature, the author decided a pH of 4.0 would be sufficient to give good experimental electrocoagulation results.

The deinked stock, from which the froth was taken for the zeta potential measurements, provided one of the stocks needed for experimental analysis. This stock was used to analyze a chemical flocculating aide and how it assisted deinking. A one-liter sample of the stock was used for shive analysis and the remaining stock was made into TAPPI Standard handsheets. G. E. Brightness measurements were obtained from the sheets along with a microscopic analysis. The results of these tests were used to analyze how the conventional flotation process performed as a deinking process. They were also used as another standard to which the electrocoagulation process could be compared. The results of the testing will be discussed in the Results Presentation.

FIGURE 3



Once all of the preliminary experimentation was completed, the actual electrocoagulation experimentation was conducted. The first step in finding an efficient electrocoagulation operation was to find an operating consistency. It was the original intent of the author to experiment with consistencies of 0.5%, 1.0%, 1.5%, and 2.0%. However, it was found that with the electrode insert in place, the higher consistencies could not be used. At the consistencies of 1.5% and 2.0%, it was observed that the gas bubbles became trapped beneath the surface. At these higher consistencies, there were also many dead spots in which stock became trapped and even violent agitation would not eliminate the dead spots. The electrode unit was operated at 1.0% and 0.5% consistencies, and it was found that there were more bubbles produced at the lower consistency; however, at 1.0% consistency there was good bubble formation. Another factor that had to be taken into account was that the higher the consistency, the more ink that would be present. The more ink that was present in the system, the easier it would be to analyze. The final consistency that was used was a compromise between 0.5% and 1.0%. The consistency chosen was 0.8%, which was also the consistency that the forced air flotation unit was operated at.

The operating retention time was very difficult to obtain. In the original experimental outline, retention times of 3 minutes, 2.5 minutes, 2.0 minutes, and 1.5 minutes were going to be used, and the results produced could be analyzed to determine the correct retention time. However, due to the limitations of the electrode unit and the power source, the retention time of 20 minutes was found to be more reasonable. The 20-minute retention time was obtained by filling the

flotation cell with 0.8% consistency stock and 0.1% of Triton X-100. The electrode insert was connected to the battery and the surface of the pulp slurry was observed for bubble formation. After five minutes, bubbles started appearing at the surface; at ten minutes there was a generous amount of bubbles present; and at fifteen minutes there was a good head of foam present. It was determined at this time that a retention time of 20 minutes would be sufficient time for the electrocoagulation process to produce results that could be used to determine the use of electrocoagulation as a deinking aide.

After the operating consistency and retention time were determined, the electrocoagulation operations were conducted. Five 15-liter samples of inked stock were prepared at 0.8% consistency. Their pH was adjusted from their original pH of 11.0 to a pH of 4.0 using 50% sulfuric acid.

The first sample was placed in the flotation cell. The agitator was started and the coagulation unit was connected for 20 minutes. After the 20 minutes, the sample was removed from the cell and placed aside for further testing. There was no flotation used on this stock and no chemical flocculating aide was added. Some of the bubbles produced were skimmed from the surface and examined for ink content. It was found that there was very little ink present in the froth.

The second run was a repeat of the first with the exception that after 20 minutes of electrocoagulation, 0.1% of Triton X-100 was added and forced air flotation was run for 15 minutes. The foam produced was examined for ink content, and it was found that there was very little ink present. This was contrary to what the author believed would happen. A sample of the deinked stock was made into a handsheet and

the handsheet was examined under the microscope. It was observed that there was still a large quantity of ink present, even after deinking.

Run #3 was a repeat of Run #2. The same outcome was observed. While observing the handsheet from this run under the microscope, it was noticed that at times the ink particles seemed to be attached to the fiber. There was not a large quantity of this observed; however, it gave reason to speculate that the low pH was causing an attraction between the fiber and ink particles.

Run #4 was used to determine if an acid system was causing the ink not to float from the stock. An original sample of inked stock was added to the flotation cell at a consistency of 0.8% and a pH of 11.0. The electrocoagulation insert was connected and ran for approximately ten minutes when the bubbles seemed to quit appearing at the surface. When the stock was cleared away from the electrodes, it was noticed that the positive electrodes were covered with ink. The polarity of the electrodes was reversed in hope of releasing the ink from the electrodes. This was not the case and the electrodes had to be removed and cleaned.

Run #5 consisted of trying to float the ink off using electrocoagulation and Triton X-100, but no forced air flotation. A 15-liter sample of stock at 0.8% consistency and a pH of 4.0 was added to the flotation cell. The electrodes were connected to the battery and allowed to run for 20 minutes. The gas bubbles produced were much more stable with the flotation agent present, but there was very little ink in the froth.

Run #6, which became the last run, was another attempt to avoid using an acid system for the electrocoagulation operation. This time

the pH was adjusted to a neutral pH of 7.0. The electrocoagulation unit was run under the same conditions as Run #4. After the 20 minutes of electrocoagulation, 0.1% of Triton X-100 was added and forced air flotation was conducted for 15 minutes. It was again noticed, after the deinked stock was removed from the cell, that the positive electrodes were covered with ink. It was also noticed that the froth was virtually ink free. At this time, the author decided to discontinue experimentation until the previous samples were analyzed.

## RESULTS PRESENTATION

There were three tests conducted on the samples to determine the efficiency of each operation. The three tests included G. E. Brightness, M/K Systems, Inc., Shive Analysis, and microscopic analysis. The use of TAPPI Dirt Count was contemplated; however, after analyzing the results of the three other tests, it was determined to be unnecessary.

Every sample was divided into two quantities. One-liter samples of every run were taken and a shive analysis was done. The stock remaining from each sample was made into TAPPI Standard handsheets, which were used for brightness measurements and visual analysis.

The following samples were taken so an analysis could be made:

Sample 1: Clean border stock with no ink present.

Sample 2: Pulped, printed ledger containing large quantities of ink.

Sample 3: Deinked stock using Triton X-100 as a flotation agent and forced air flotation.

Sample 4: Electrocoagulated stock with no flotation agent or flotation.

Sample 5: Electrocoagulated stock with forced air flotation and Triton X-100 added as a flocculating aide. The pH of the stock was 4.0.

Sample 6: Electrocoagulated stock with forced air flotation and Triton X-100 added as a flocculating aide. The pH of the stock was 7.0.

There were several other tests performed; however, an analysis of the above six samples gave enough information to conclude the effects of electrocoagulation as an aide for deinking.

### Microscopic Analysis

Each sample was examined under a microscope using a 10x power.



The following three characteristics were looked at:

- 1) ink particle size
- 2) quantity of ink present
- 3) the structure of the ink and fiber network

Sample 1: Clean border stock with no ink present.

Observed a very clean sheet with no ink present. There were some small dirt specks present, but nothing out of the ordinary.

Sample 2: Pulped printed ledger with large quantities of ink present.

Observed large quantities of ink in the sheet, but noticed the ink was completely detached from the fiber. Most of the ink particles were very small and invisible to the naked eye. There were some large ink particles present, but not very many. The sheet appeared light grey to the eye.

Sample 3: Deinked stock using Triton X-100 as a flotation agent and forced air flotation.

Observed very little ink present; however, some ink was found in the sheet. The ink particle size was mostly small particles; however, there were some larger particles present. The sheet appeared white to the eye, but not as bright as the sheet with no ink.

Sample 4: Electrocoagulated stock with no flotation agent added and no forced air flotation. The pH of the stock was 4.0.

Observed large quantities of ink present and many more larger particles. It was noticed that at times the ink seemed to be attached to the fiber. There were still large quantities of minute size ink particles present. The sheet appeared light grey with a blue tint.

Sample 5: Electrocoagulated stock with forced air flotation and Triton X-100 as a flocculating aide. The pH of the stock was 4.0.

Observed large quantities of ink in the sheet. The particle size was larger than when no electrocoagulation was present. The particles appeared to be detached from the fiber. The sheet appeared greyish blue to the eye.

Sample 6: Electrocoagulated stock with forced air flotation and Triton X-100 added as a flocculating aide. The pH of the stock was 7.0.

Observed large quantities of ink present in the sheet. Noticed the particles were large and were detached from the fiber. Did

not notice any difference in particle size between Sample 5 and 6. The sheet appeared grey to the eye.

From what was observed under the microscope, it was noticed that the only operation that seemed to remove the ink was the forced air flotation with the chemical flocculating aide. Excellent deinking results were obtained using the forced air system. It was a surprise to the author to find that when electrocoagulation was used along with forced air flotation, very little ink was removed. It was speculated that under acid conditions, the ink was redepositing back on the fiber. This was not observed when the handsheets were viewed under the microscope. Test runs were done at pH's of 7.0 and 11.0 to see what effects electrocoagulation had on a neutral system or an alkaline system. The results for the experiments using stock at a pH of 7.0 were the same as those using a stock at a pH of 4.0. When the pH was increased to 11.0, the electrodes of the unit plated with ink so quickly that correct results could not be obtained.

It was noticed with the microscope that the ink particle size increased slightly under acid conditions due to a decreased zeta potential of the ink particles.

#### Brightness Measurements

G. E. Brightness measurements were performed on all of the samples, and the results are presented in a bar graph in Figure 4. The brightness readings ranged from 80.3% for a sheet with no ink present, down to a brightness of 77.1% for a sheet of pulped printed ledger. The big surprise was that whenever electrocoagulation was used, the brightness never got above 77.5%. When electrocoagulation was used along with forced air flotation and a flocculating aide, the brightness reached 77.4%, which was more than a 1.0% difference from the

FIGURE 4

Sample 1 - Clean border stock, no ink.

Sample 2 - Pulped printed ledger.

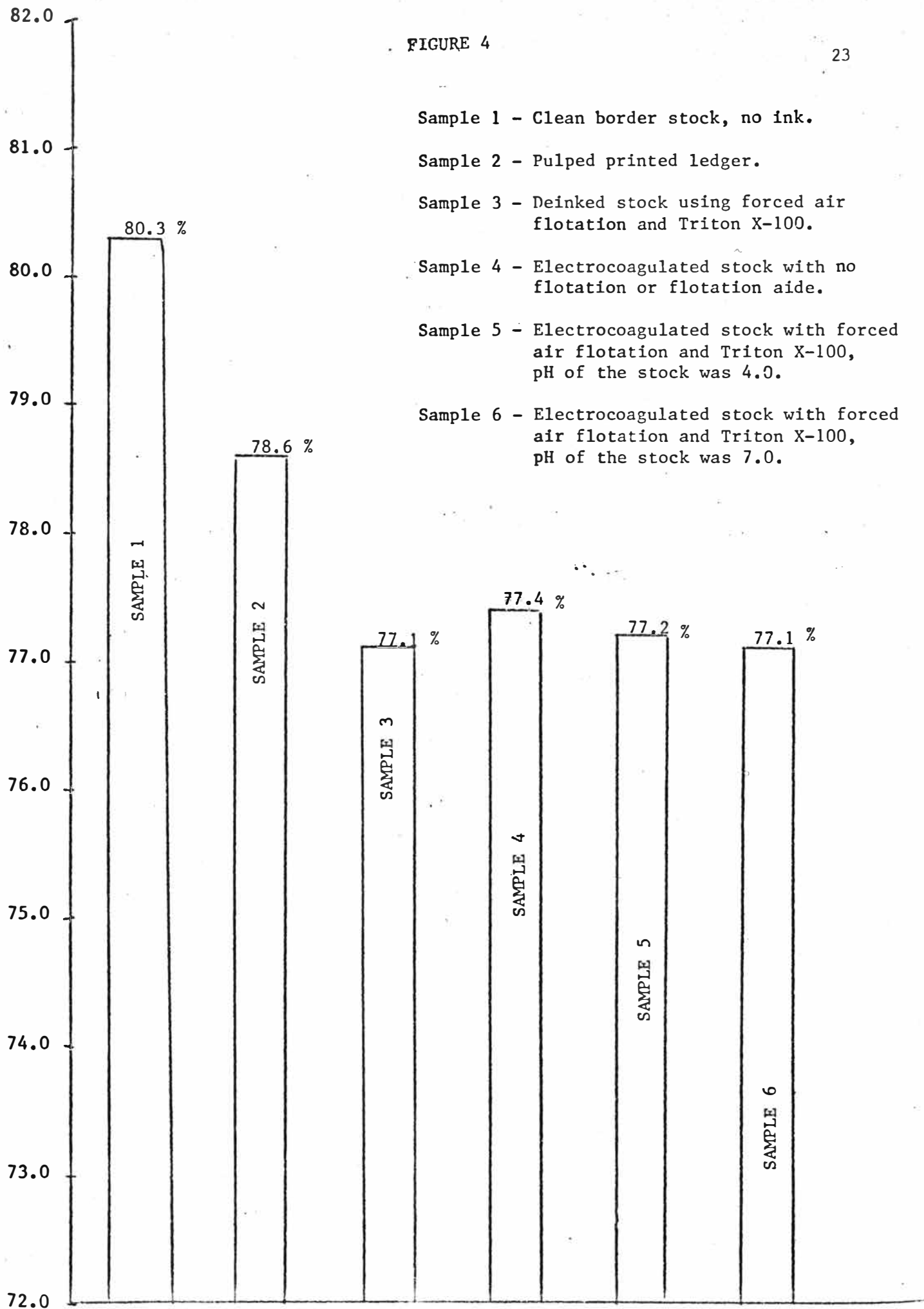
Sample 3 - Deinked stock using forced air flotation and Triton X-100.

Sample 4 - Electrocoagulated stock with no flotation or flotation aide.

Sample 5 - Electrocoagulated stock with forced air flotation and Triton X-100, pH of the stock was 4.0.

Sample 6 - Electrocoagulated stock with forced air flotation and Triton X-100, pH of the stock was 7.0.

G. E. Brightness (%)



brightness obtained using flotation without the electrocoagulation. This does not seem like a very large difference, but when the difference in brightness between an inked stock and an uninked stock is 3.0%, 1.0% is a large difference. It should be noted that when acid is added to the pulp, the pulp turned light blue. This did not show up in the brightness readings. The brightness obtained for the deinked stock using forced air flotation and Triton X-100 as a flotation agent was 78.6%, which is less than the stock with no ink but still good when compared to the stock with no deinking. There was no significant difference in the brightness readings obtained when the pH of the stock was changed from 4.0 to 7.0 to 11.0.

#### M/K Systems, Inc., Shive Analyzer

The operating principles and procedures for the shive analyzer can be found in the Operator Instructions Manual, which is located in Appendix A.

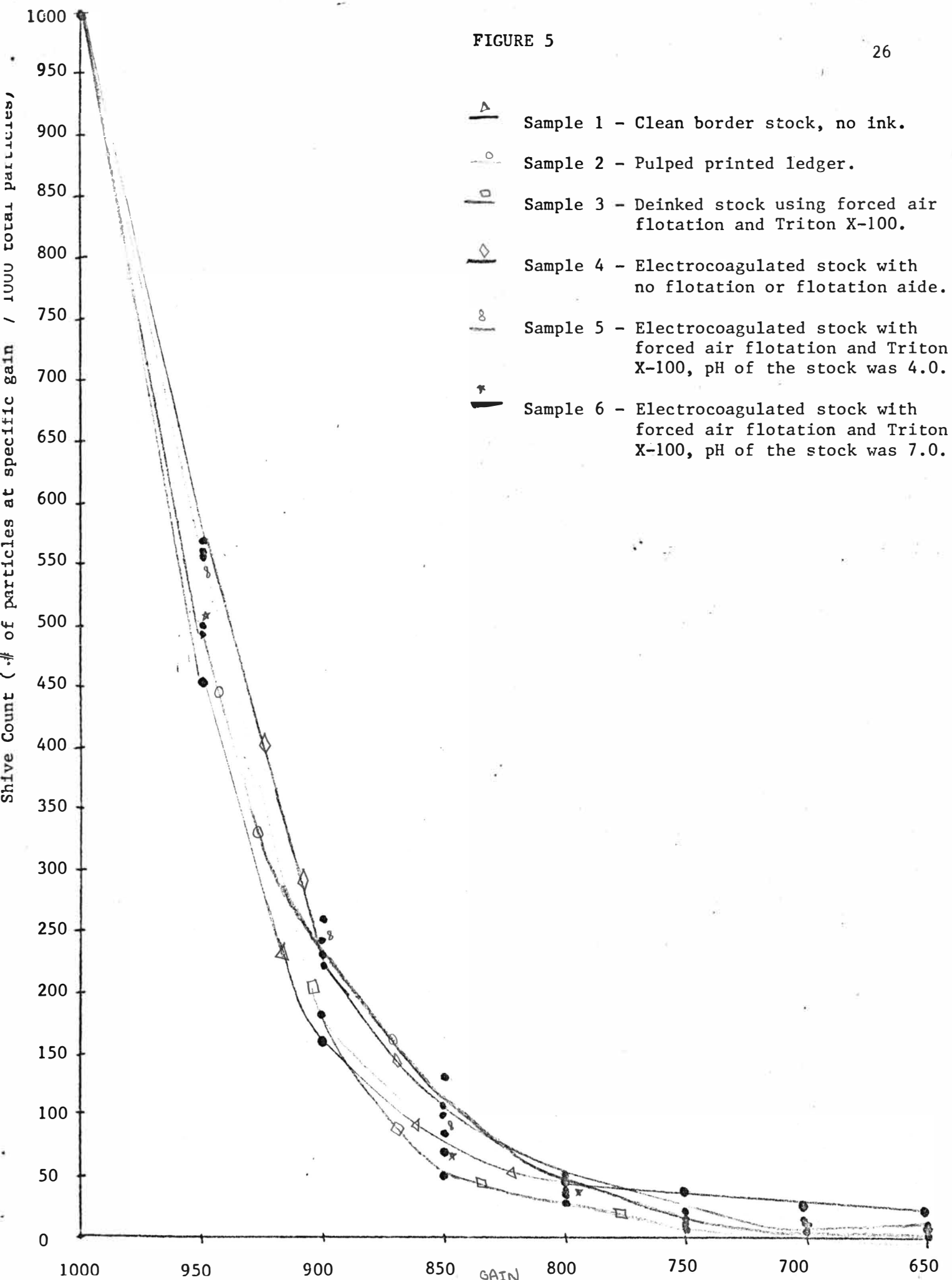
There was no defined procedure for determining how deinking efficiency and ink particle size related to the results obtained from the shive analyzer. It was impossible to set the analyzer to count a certain size ink particle because there was no standard to which the analyzer could be calibrated to.

The procedure for relating deinking with the shive analysis was to run shive analyses on the six samples. Each analysis would consist of eight different settings of the gain controller. The gain setting determines the size particle that is counted by the analyzer. The readings from the shive analyzer are read as number of particles for a certain gain setting per 1000 total particles. The readings obtained

for the six analyses were plotted against gain control and six curves were produced, as shown in Figure 5.

From the graph, the author arrived at the following relationships. The larger the gain setting, the smaller the particles that will be counted. At a maximum gain setting of 1000, the shive analyzer counts all of the particles and the same reading was obtained for all the samples. At a gain setting of 950, the stocks with ink and no flotation gave the highest readings. The readings for the stocks where some deinking was used were the same and fell between the readings obtained for the stocks with no deinking and the stock with no ink present. This was consistent with what was expected. At the 900 gain setting, the sample with no deinking or electrocoagulation and the sample with electrocoagulation and no flotation had the most particles of this size. The samples with forced air flotation and electrocoagulation gave a smaller reading than when no flotation was used, but gave a greater reading than the sample with flotation but no electrocoagulation. This suggests that the electrocoagulation process had a detrimental effect on the flotation process, which is supported by the microscopic analysis and the brightness readings. At a gain setting of 850, the sample of inked stock gave the highest reading. It was noticed that there was not much difference in readings between the samples where electrocoagulation was used. The readings for these samples were much greater than the reading for the sample of forced air flotation stock. This again suggests that electrocoagulation had a detrimental effect on forced air flotation. At the gain settings of 800, 750, 700, and 650, the readings were the same for all the stocks. This must suggest that there was little ink counted at this size since

FIGURE 5



the stocks with ink in them were the same as the readings for the stock with no ink present.

The author believes that the use of the shive analyzer to determine deinking efficiency and ink particle size was not very dependable. Mainly for the reason that there were too many different types of particles being counted and no way to determine what size particles were what. It was for that reason that all of the conclusions drawn from the shive analysis were assumptions backed by the microscopic analysis and brightness measurements.

## CONCLUSIONS

In analyzing the electrocoagulation process as a deinking aide, it was found that the process had a detrimental effect on forced-air flotation rather than a beneficial effect. It was found that when the system was under acid conditions, the ink would not float to the surface. It was assumed that at the lower zeta potentials the ink may have been attaching to the fibers. This was noticed somewhat with the microscopic analysis. The gas bubble formation was good, but they may not have been stable enough to collect the ink particles. When the coagulation process was used, the froth produced contained very little ink and the bubbles broke down faster than the froth produced using forced-air flotation.

It was attempted to electrocoagulate at higher pH's, but the electrodes kept plating with ink and a trial under alkaline conditions could not be completed. It was noticed that even a neutral pH of 7.0 caused the electrodes to plate with ink.

When determining the zeta potentials at different pH's, it was found that there was very little difference in zeta potential at the different pH's. However, when the pH was varied slightly for the electrocoagulation process, the electrodes plated quickly. A variation of pH from 4.0 to 7.0 caused a great amount of plating.

The reduced zeta potential did cause some flocculation which was noticed when the stock was viewed under the microscope. Particle size did not greatly increase; however, some flocculation was observed.



## SUGGESTIONS

The author believes that the use of electrocoagulation as a flocculating aide for deinking demonstrated very little promise. The "home-made" equipment used may have lead to some of the detrimental effects of the process; however, the results found for the prescribed conditions showed an overall detrimental effect.

From what was done during this thesis, the author believes that the only suggestion for further study of the electrocoagulation process would be a more in depth look at how pH affects deinking efficiency.

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OPERATING INSTRUCTIONS

for

M/K SYSTEMS INC. SHIVE ANALYZER

July, 1980

## INSTALLATION

The M/K Systems, Inc. Shive Analyzer consists of a Sample Handling System and an Electronics Console connected by a cable.

The Sample Handling System is comprised of a 2 liter sample tank, a variable speed peristaltic pump, and the Measuring Cell.

The Electronics Console consists of the logic circuitry, the instrument controls, and a printer.

The only precaution which must be exercised in locating the Shive Analyzer is that the 110 v., 60 Hz line current employed must be free of spikes and other distortions. If such a line is not available, a Solar transformer should be employed.

Agitation to the sample tank is provided by air bubbles entering from a hole in the bottom of the tank. The air is supplied through the 1/4" tygon tube with the quick-disconnect fitting. This fitting should be connected to a supply of low pressure air whose flow can be regulated.

Alternatively, gentle mechanical agitation can be used.

Note: If this air agitation is not employed, the quick-disconnect fitting must be kept at a height above the water level in the Sample Tank. Otherwise, the sample will leak out.

## PRINCIPLES OF OPERATION

Stock at a consistency in the 0.005 - 0.001% range (its exact value is immaterial) is pumped at 500 ml/min through a rectangular  $0.5 \times 10 \text{ mm}^2$  glass tube (see Figure 1). The long side of the

sample tube is illuminated uniformly by infra-red light transmitted to it by a glass fiber bundle the height of the cell and 0.5 mm in width. The light passing through the sample cell is collected by an identical fiber bundle and measured by a photocell. This optical system is so sensitive that it is able to detect all particles passing it which are down to small hardwood fibers in size.

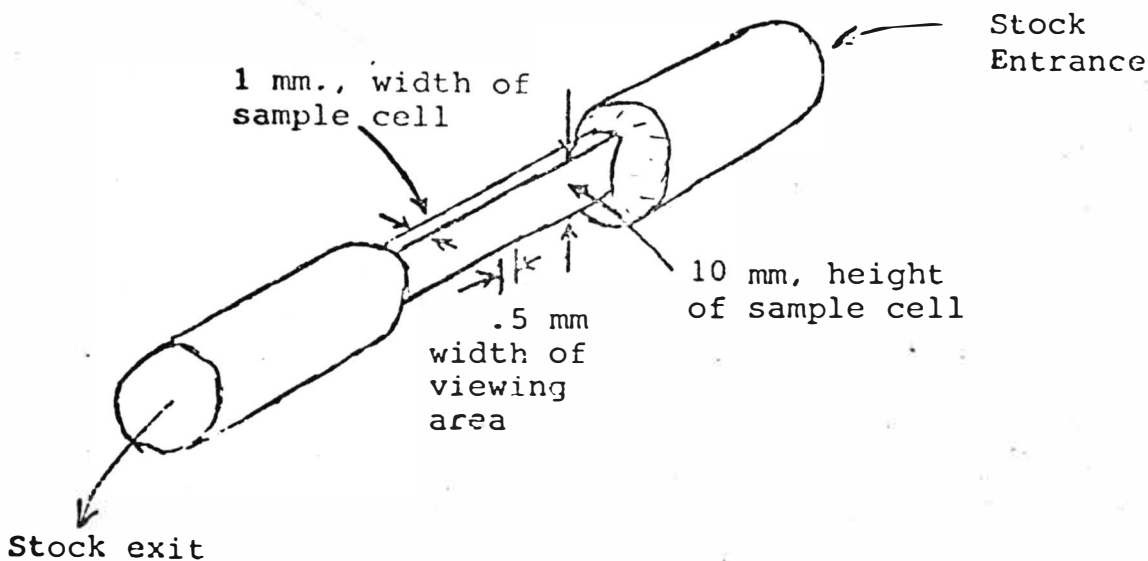


FIGURE 1  
SAMPLE CELL

In order to function as a Shive Analyzer, the instrument must be able to count the particles in a stock sample which are greater in size than individual fibers. The minimum size of the particles which triggers the counting circuit is determined by a Discriminator Pot on the front panel of the Console; the higher its setting, the smaller the particles counted, and vice versa. In the following discussion, this shive counting circuit is referred to as Circuit S.

It has been established using strobe light photography under a microscope that fibers and shives pass through the cell with their long axis parallel to the direction of flow through the cell (See Figure 2). Furthermore, because of the narrow dimensions of the cell, the flat face of the fibers and shives is oriented parallel to the face of the cell. Thus, what the instrument actually measures is the amount of light attenuated by the particles which is proportional to their cross-sectional area. By setting the Discriminator Pot to a position where the Circuit S does not count the fibers, the instrument can be used as a shive analyzer.

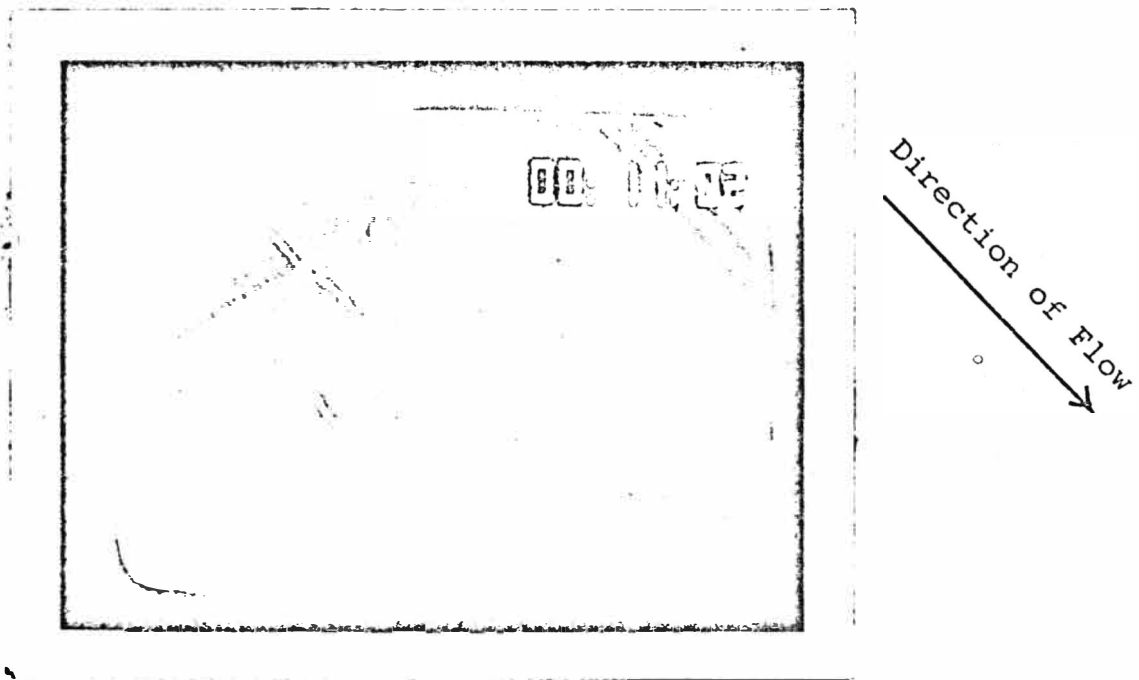


Figure 2

Simultaneous to counting the particles above a pre-specified size, the instrument also counts all of the particles, i.e., all the fibers and shives passing through the cell by a second circuit, re-

ferred to here as Circuit T. This circuit T acts as a control circuit for the instrument and compensates for variations in stock consistency. Thus, for example, when 1000 total particles have passed through the instrument, the Circuit T causes the printer of the instrument to print out the number of particles counted by circuit S, and simultaneously resets its count to zero.

Circuit T in fact acts as a consistency regulator for the instrument. As long as the stock consistency is sufficiently low so that the fibers and shives travel singly past the glass fiber bundles, the actual consistency of the stock is immaterial. For example, if the stock consistency drifts from 0.005% to 0.0007%, the only effect on the measurement is that Circuit T will trigger the printer sooner.

If the stock consistency is allowed to go too high, say to 0.0015% or above, multiple fibers often pass the viewing window and the instrument becomes consistency sensitive. Thus, it is important only to maintain the consistency below about 0.001%. (For very finely-divided pulps with low shive contents a slightly lower operating consistency range is required to eliminate the possibility of multiple particles passing through the viewing area at one time.)

#### OPERATING INSTRUCTIONS

Precaution: The Shive Analyzer must employ clean, bubble-free water; it is an exceptional "bubble counter". Deaerated, and if possible, deionized water should be used. Cold water which heats up

and precipitates air bubbles is unusable.

Before initiating a series of runs, two liters of the water in which the samples are to be dispersed is run through the instrument just like a sample to make certain that the water is free of contaminants, air bubbles, etc. which could be counted. This run is made with the following settings. (Air agitation is not required.)

The digital GAIN pot is turned to its furthest clockwise position, 1000, which reads 000. The selector switch is set to the AUTOMATIC position.

The black pump control switch on the back of the Sample Handling case is set to the speed position #5. Its direction switch (CW/CCW) is set to the position which causes the flow to discharge from the bottom of the Sample Tank and return via the port near its top. The operator can readily determine the direction of flow by placing a finger over the upper port and observing pressure or vacuum.

As long as the number of counts observed in several minutes is very low, say under 50 or so, than the water is sufficiently clean and deaerated. The run is terminated by switching off the pump.

An actual determination is made in a similar manner. A two-liter stock sample at about .001%, which is equivalent to 0.02g of fiber, is placed into the Sample Tank. The air agitation is initiated so that there is a small flow of large bubbles in the Sample Tank. The Selector Switch is set to AUTO, and the pump speed control to 5. The direction of flow should be from the bottom of the Sample



Tank through the Sample Cell and back into the top of the Sample Tank.

In many cases when a sample is first run, entrapped air will flow through the instrument and give a false reading. Such entrapment is especially common in the Sample Cell itself. One can readily determine whether or not there is air in the Sample Cell by removing its cover plate. If such air is observed, tilt the instrument slightly to free it up and pass it out of the line. DO NOT MECHANICALLY TAP THE GLASS SAMPLE CELL. IT IS FRAGILE AND NOT COVERED BY THE WARRANTY.

The first determination of a new sample should be made at a Discriminator Pot Setting of 1000 to make certain that the Circuits S & T are counting the same particles. The count of circuit S will rarely be exactly 1000, generally between 900 & 1000, and reproducible to within 25 counts. A count of  $1000 \pm 100$  of Circuit S at a Pot Setting of 1000 is considered satisfactory.

If the count should fall outside of this range, it can generally be brought back in by further dilution of the sample. If gross miscounts occur after significant dilution, the instrument has a problem, and M/K Systems, Inc. should be notified.

Once the Circuit S has been verified at a Pot setting of 1000 in a couple of runs, the Pot setting should be reduced to the value at which a Shive Count is made. This is generally in the 800 range.

The determination of the Pot setting for detecting shives is somewhat arbitrary because the definition of a shive is arbitrary.

This is because the number of fibers in a fiber bundle will range from 2 up to dozens. A couple of techniques for selecting the proper Pot setting are discussed below.

After every half a dozen runs or so, the direction of flow through the instrument should be reversed for a couple of runs by resetting the CW/CCW switch. This will clean out any particles which might have been entrapped on the inlet side of the Sample Tube. Also, if counting should virtually stop in the middle of a determination, the chances are the Sample Tube is plugged. In this case, stop the run, reverse the direction of flow to clean out the Sample Tube, and restart the determination.

Note: DO NOT REVERSE THE DIRECTION OF FLOW WITH THE PUMP RUNNING.

The Console has several additional controls. The Reset button resets the count of Circuit S to zero. The MANual position deactivates the Circuit T, and the instrument will count until the Reset button is pushed in. Finally, the Printer itself has two controls. Pressing Print will print the particular number in memory, and Feed will advance the paper.

Some instruments have a Selector Switch setting of Classify. This function is not built into the Shive Analyzer as its circuitry is not yet fully developed.

At the end of a determination, the sample is removed from the Sample Tank by means of the valve next to the speed controller of the pump.

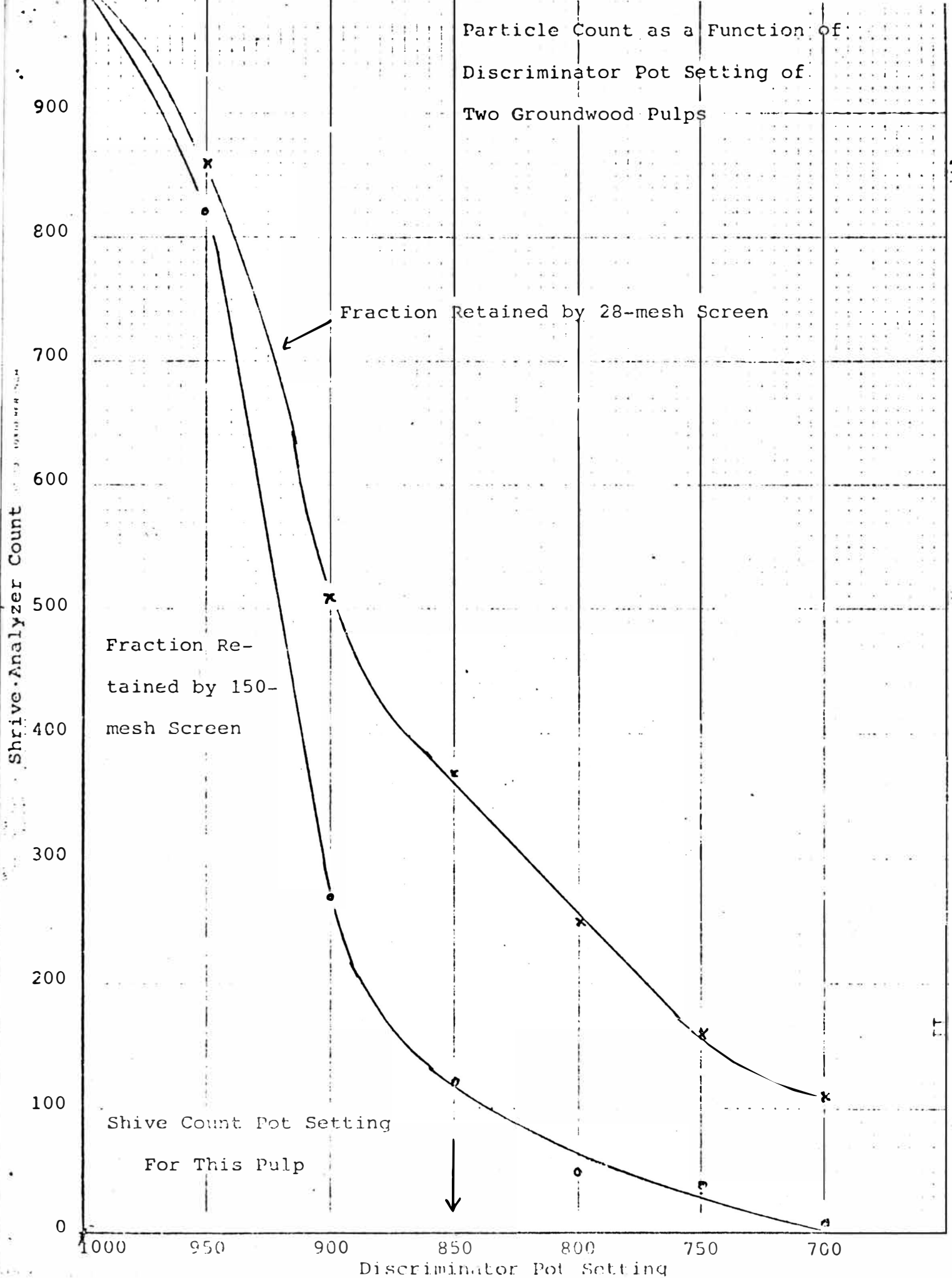
#### DETERMINING THE DISCRIMINATOR POT

##### SETTING FOR SHIVE COUNTING

The most direct means for determining the proper Shive Count Pot Setting for a particular stock is to run it through the instrument before and after screening at several pot settings such as 1000, 950, 900, ... 700, 650, or even lower. The count of the screened sample will drop off much more rapidly than that of the unscreened sample. This is because the count (per 1000 total particles) at a given Pot setting represents all of the particles greater in size than a specific value. Thus, a highly refined, well-screened stock comprised almost exclusively of small individual fibers will produce a very low count even at a high Pot setting because it contains so few larger particles, i.e., fiber bundles. An unrefined unscreened sample of the same stock will still show a large count at a high setting because of its fiber bundles and shives. So the proper Discriminator Pot Setting for counting shives is the one at which most of the fine particles are no longer seen.

This approach is illustrated in Figure 3 which shows the Shive Analyzer counts at several Pot settings of two Bauer McNett fractions of a groundwood furnish. The upper curve is of the coarse fraction retained by the 28-mesh screen while the lower is of the fine fraction which passed through the 100-mesh screen and was retained by the 150-

Particle Count as a Function of  
Discriminator Pot Setting of  
Two Groundwood Pulps



mesh screen.

At a pot setting of 875, the instrument sees only 20% of the particles in the fine 150 mesh stock. At this setting, however, it counts almost half (45%) of the particles in the coarse 28-mesh samples. At a pot setting of 850, 12% vs. 37% of the two samples are seen respectively. So we would use a Pot Setting of 850 for determining Shive Counts on groundwood with the instrument by which these particular measurements were made. (This is not the setting for groundwood for this particular instrument; we leave that judgment up to the user.)

We recognize that the above procedure is imprecise and somewhat arbitrary, but the same holds true for all Shive Analyzers. The instruments based on screen plates hold back most particles greater than an arbitrary size, i.e., the width of the cut of the plate. But this separation process is not uniform either; rather, it is probabilistic.

One means of absolutely calibrating the Shive Analyzer would be to pass through it particles of uniform shape and dimension, i.e., artificially cut shives. But here again, the selection of the dimensions would be arbitrary. What is the exact size shive which causes a specific problem? Again, an unknown. So until the relationship between shive size and shive-induced problems are better defined, precise calibration of Shive Analyzers is of limited value.

## AS A CLASSIFIER

The ability of the Shive Analyzer's optical system to "see" small fibers and fiber debris makes it potentially usable as an optical fiber classifier. In this case, the electronics would have a number of equally displaced counting circuits, all of whose counts would be printed out at the end of a determination. In other words, the instrument would have the built-in capability to print out the data for each of the curves shown in Figure 3 in a single determination. Preliminary development work of these circuits has been carried out.

The Shive Analyzer can of course be used now as a manually operated classifier. The two curves of Figure 3 contain the data of such a classification. A fiber classification is obtained by plotting the "differential counts" as a function of "class size". A differential count is the difference in the number of particles counted at two Pot settings which in turn define a size class; see Figure 4.

In Figure 4, the last "Residual" class represents the sum of the counts made at 650, 600, 550, etc. The fine 150-mesh fraction had no residual count, the coarse 28-mesh fraction, 11%. So if one were certain from a microscopical examination of the 150-mesh pulp that it contained no shives, a Pot Setting of 700 could be used to define shives. Most likely, they would be mini-shives.

# FIBER CLASSIFICATION BY SHIVE ANALYZER

Frequency - 50%

KIMBLE & ESSER CO.

