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THE EFFECT OF TEMPERATURE ON THE EMULSIFICATION OF ALKENYL SUCCINIC ANHYDRIDE

By:

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

Western Michigan University
Kalamazoo, Michigan

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ABSTRACT

The objective of this study is to look at the effect of starch temperature on the emulsification of ASA. Historical information was limited, but it was discoverd that an emulsion particle size range of 1-2 microns would give optimum size results.

A continuous emulsification apparatus was constructed using a glass venturi and power syringe to emulsify the starch and ASA. The basic conclusions drawn from the experiment were that the starch temperature did have an effect on the final sizing characteristics of the sheets. It was also possible to correlate a visual inspection of the emulsions with the final test results. A starch temperature range of 100-125°F was successful in producing a stable, uniformly distributed ASA-starch emulsion.

Keywords: Alkaline Papermaking, Alkenyl Succinic Anhydride (ASA), Internal Sizing, Temperature, Emulsion.

TABLE OF CONTENTS

INTRODUCTION

The purpose of this study is to investigate the affect of temperature on the sizing propertis of Alkenyl Succinnic Anhydride (ASA). Past experiences have shown that ASA in an alkaline system is sensitive to the ASA-starch emulsion, pH, temperature and shear in the system. A venturi-type emulsifier will be used to standardize the emulsification. Standardizing the addition rates of cationic starch and activated Fibran will allow the temperature of the starch slurry to be varied to determine the effect of temperature on the sizing characteristics of ASA. Fibran 68 is the name brand for the ASA used in this experiment. It is marketed by National Starch and Chemical Corporation.

ANALYSIS OF LITERATURE

History

Alkaline papermaking is not new to the paper industry. Some of the first papermaking systems ever developed were in the neutral to alkaline pH range. Recently, there has been renewed interest in alkaline papermaking due to the development of synthetic internal sizing agents. But problems with inconsistent production performances has checked the progress of these petroleum based synthetic sizes (1).

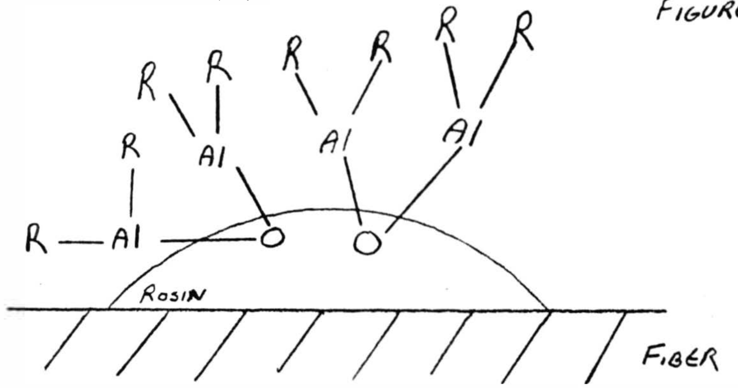
Internal sizing in an acid system (rosin-alum) has met with wide spread acceptance in recent history due to its compatability to relatively inexpensive and dynamically stable systems. Problems that have occured in acid systems include corrosion, residual alum buildup and decreased strength properties (1).

Ideally, internal sizing in an acid system consists of depositing a water repellent material (rosin) on the fiber so that when the fibers are formed into a mat and dried, the resulting paper will be water resistant. Since both the fibers and rosin are anionically charged and mutually repellent, a cationic material (usually alum) is needed to bring them together. The method is simply to mix the size thoroughly with the stock and then precipitate it with alum (2).

The anchoring of ASA, a cellulose reactive size, is provided by the covalent bond formed between the size and the cellulose. In order to form this bond, these nonionic, hydrophobic organic compounds which are not soluble in water, must be made compatible with the aqueous papermaking system. This is done by emulsifying them using dispersants as necessary. Utilization of cationic retention aides give the emulsified particles a net positive charge which provides a retention mechanism similar to that of the rosin size precipitate (3). However, the attraction forces usually are weaker because the emulsion particles are relatively large and because the cationic retention aid is not necessarily firmly attached to the particle surface. Once the particle is retained on the cellulose fiber surface, it must be distributed. This occurs only during the wet pressing and drying operations on the papermachine. The cellulose reactive sizes spread over the fiber down to molecular cellulose. This very effective monomolecular coverage is one reason for the high efficiency of synthetic sizes. Finally, after retention and distribution occur, the reaction with cellulose occurs and sizing is obtained (1).

According to Schwalbe (4), the aluminum ion plays a key roll in tying up the resinate to the surface of the fiber. Figure #1 allows the rosin-alum agglomerate attached to a fiber (4).

FIGURE #1



Renewed interest in alkaline sizing is primarily due to the development of petroleum based synthetic sizes. There are several similar types of synthetic sizes available on the market. This study will deal solely with ASA. Figure #2 shows the chemical formulation of ASA as purchased from a supplier (5). The $C_n H_{2n-1}$ group is an alkyl chain varying in length depending on the supplier.

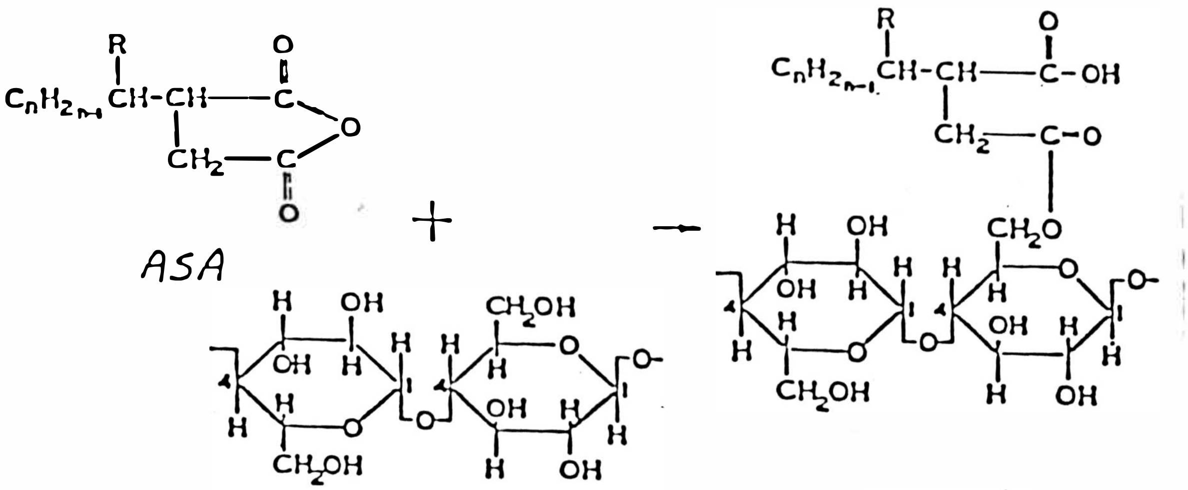


Figure #2

Following is a list of the major advantages and disadvantages of acid and alkaline internal sizing systems as outlined by Maher (6).

Advantages of Acid Internal Sizing

1. Proven performance record.
2. Relatively inexpensive compared to alkaline sizing.

Disadvantages of Acid Internal Sizing

1. Acid corrosion problems due to the low pH of the system.
2. Problems from redisual alum build-up in the white water.
3. Decreased strength properties when compared with those found in alkaline systems.

Advantages of Alkaline Internal Sizing

1. Improved strength properties with out sacrificing quality.
2. Calcium Carbonate may be used as a filler.
3. Reduced energy usage (primarily in drying the sheet).
4. Reduced requirements for fresh water.
5. An alkaline sheet is more permanent than an acid sheet (strength and color).

Disadvantages of Alkaline Internal Sizing

1. Inconsistent performance record because of sensitivity of the emulsion to temperature and shear.

Measuring the Degree of Sizing

The permeation of water through paper follows three pathways: a) through the paper via the open spaces n the web structure (interfiber penetration), b) through the paper via fiber-to-fiber contact (intrafiber penetration), and c) via capillary action (7).

The basic properties of paper that effect both the permeation of paper by water and also the pathway by which the water penetrates the paper are: a) the hydrophibicity of the fibers, b) the pores in the sheet structure, c) the surface of the paper, and d) the sheet structure itself (7).

An article discussing the various test methods available for measuring the sizing in paper by Gess states (7), "The parameters that dictate the degree of sizing required in papers has not changed, but papermakers have managed to increase the number of tests available to more than 50".

These 50 test methods fall into three basic categories: those methods that measure the penetration of aqueous fluids through paper, those that measure characteristics of the surface of the paper and correlate this with sizing, and those that measure a property of paper related to the degree of sizing to that sheet (i.e. curl, ink feathering, etc.) (7).

The Hercules Size Test (HST) measures the level of sizing by measuring the reflectance of the sheet that is being penetrated by an acid ink. The endpoint for the test is the amount of time (seconds) required for the ink to deplete the reflectance to 85% of its original value.

According to Gess (7), the greatest weakness of the reflectance method is the low surface tension of the inks used. This low surface tension tends to enhance penetration via the pores of the paper. In poorly formed or highly directional papers, one can see how the effect of pore penetration could cause a lowering of sizing test results.

The testing for this study will be done on handsheets prepared in the British sheet mold. Since directionality of the fibers is minimal in the British sheet mold and the formation is good, it is assumed that the effect of pore penetration by the ink will be minimized.

The reasons for selecting the Hercules Size Test as the size test for this study include a) widespread acceptance in the paper industry, and b) recommendation by Dr. Raymond Janes (8).

STATEMENT OF PROBLEM

The problem to be investigated in this study is the effect of varied cationic starch temperature on the sizing characteristics of Fibran.

The results of this experiment will be as measured by the Hercules Size Test. The data will be tabulated and plotted with the HST results in seconds vs. temperature. It is hoped that there will be correlatable data to show the effect of starch slurry temperature on the sizing characteristics of ASA.

EXPERIMENTAL PROCEDURE

Apparatus Construction

This project deals with three separate theses linked together in an effort to generate information regarding the sizing of alkaline paper with Alkenyl Succinic Anhydride. The first phase of the laboratory procedure was to construct an apparatus by which ASA and cationic starch could be emulsified. Additionally, it was felt that an important goal of the project was to develop methods of operation that could be easily scaled up to industrial production requirements; therefore, it was important to develop a process that would be continuous. The apparatus that was constructed will be detailed in four different sections -- each according to the primary mechanical device involved. These devices are: the starch cooker, the emulsifier, the holding tank, and the handsheet former (Appendix 1). Each of these devices provided a separate function, and can be scaled to continuous industrial machine production.

The Starch Cooker

Western Michigan University's pilot plant starch cooker was used to cook National Starch's CATO 15 brand cationic starch. There wasn't any modification of the cooker itself. De-ionized, pH controlled water and cationic starch were used to produce the starch slurry. The exact running conditions and quantities of the slurry will be included in the text of this report.

The Emulsifier

The emulsifier used in this experimental procedure was a continuous ASA-Starch emulsifier developed by Brian J. Dufour with the assistance of Steven S. Riegsecker and Phillip G. Purpura. Prior to its construction, the only method of preparing this emulsion was a procedure in which activated ASA and cationic starch were mixed in a batch-wise, low shear method. It is believed that this new emulsifier was the key to the successful development of a continuous process. The apparatus developed consisted of a glass venturi, a power syringe, and a lobe-pump (Appendix II).

The starch was pumped from the holding tank through the newly designed glass venturi which was made by heating a hollow glass rod, and stretching it to produce the restricted central orifice -- the size and shape most closely resembling an "ideal" venturi. A Masterflex pump (a lobe-pump producing a plug flow) was used to pump the starch from the holding tank through the venturi. It was assumed that the back-pressure created by the restricted orifice on the venturi allowed the pump to run in a continuous flow fashion. The purpose of using the venturi was to provide an area within the pipeline whereby high velocity and shear could produce the degree of mixing required for a good ASA/starch emulsion.

A pre-determined, continuous flow of activated ASA is needed for addition to the starch prior to the mixture entering the venturi (Appendix III). In order to provide this continuous flow, a "power syringe" was constructed with its discharging opening at the center of the pipeline. This allowed the activated ASA to be added to the starch at a point directly proceeding the area where maximum shear would be generated. A control for the rate of addition was fashioned by mounting a microscope slide guide directly above the syringe plunger. The guide translated the circular rotating motion of the drill to the downward motion of the plunger. At the same time, the guide acted as a positive displacement pump providing a constant flow against the back-pressure of the starch. Both the starch pump and the drill attached to the slide guide were connected to variable speed transformers to allow for calibration of the apparatus to the desired addition rates.

The Holding Tank

The holding tank was a portion of the experimental apparatus that would not necessarily be required in a machine scale trial. At an actual industrial site, the prepared emulsion could be added directly to the pulp slurry as it advances to the paper machine. In this experiment it was necessary to make handsheets one at a time. Therefore, a holding tank for the emulsion was required. A constant temperature water bath was used to maintain a predetermined temperature of the emulsion in the small container holding tank. There was no aggitation in the holding container, and the maximum holding time was set at 15 minutes.

The Handsheet Former

After several trials and modifications, the final handsheet former apparatus used in this experiment was a British handsheet mold with adaptions to facilitate high shear conditions. The adaptions consisted of four baffles and a high shear mixer mounted in the water holding cylinder directly above the wire screen (Appendix IV). The purpose for using the baffles was to eliminate any vortices that might be formed as shear was applied through the cylinder. The mixer was attached to the mold as well as to a variable speed transformer. These connections permitted variations in mixer speed without changing either mixers or mixer blades. The blade used was a four blade propeller.

The next section details the operation of the apparatus, and the exact quantities of materials used.

EXPERIMENTAL DESIGN

This procedure describes a method for preparing a laboratory size sample of FIBRAN emulsion. FIBRAN is the National Starch product name given to Alkenyl Succinic Anhydride.

The FIBRAN emulsion is prepared by metering a mixture of FIBRAN and an activator into a stream of starch as it passes through a venturi orifice. The high shear developed by this starch flow is sufficient to produce a fine particle size emulsion ready for on-machine use.

The emulsion was produced in this experiment by using a newly constructed miniature venturi system. The system consisted of a Masterflex lobe pump, a 30ml power syringe, a glass venturi, and two holding vessels.

Starch Preparation

1. Cook starch at 2% solids in Pilot Plant starch cooker. National Starch's CATO 15 brand starch was used.
2. Open steam valve to remove any condensate build-up in the steam lines.
3. Close the drain valve. Weight out 116.8 pounds of de-ionized water and charge the cooker.
4. Weight out 2.5 pounds of CATO 15 and charge the starch cooker.
5. Turn on the agitator.
6. Open the steam valve and allow the temperature to reach 180°F. Close the steam valve. The temperature will drift above 190°F which is sufficient to cook the starch.
7. After 30 minutes of cooking time, the starch will be ready for use.

In this experimental procedure, the fully cooked starch was allowed to cool in a five gallon container until it reached the temprature specifications required.

FIBRAN Preparation

1. Weight out 200 grams of liquid ASA. (ASA = FIBRAN).
2. Add 7% activator by weight and mix for 15 minutes.
   200 grams x .07 = 14 grams Igepal CO. 630
3. The FIBRAN is now ready for emulsification.

Apparatus Calibration

The apparatus was calibrated to meet the addition rate goals of 3.5 pounds per ton ASA at 93% solids and 8.75 pounds per ton starch at 2% solids. To minimize flow variations and pulses through the venturi, the Masterflex lobe pump was maximized at 1230 ml per minute. At this flow rate of starch, it was calculated that activated ASA must be added at the rate of 10.6 grams per minute (Appendix V).

Emulsifier Operating Procedure

1. Add the activated ASA to the power syringe. Fill the syringe beyond the porthole to ensure that there is no air gap between the syringe plunger and the ASA mixture (Appendix III).
2. Obtain approximately one gallon of starch slurry.
3. Turn on the lobe pump to a low flow rate -- one that will not dilute the ASA in the power syringe. High starch flow rates will cause an excessive back pressure, diluting the FIBRAN in the power syringe.
4. Allow the starch slurry to completely fill the tubing lines, ensuring that there are no air pockets present.
5. As soon as the starch slurry fills the tubing lines, turn on the transformer for the power syringe. This will start the flow of the FIBRAN.
6. After the flow of ASA into the starch slurry has started, increase the slurry starch to the pre-calibrated setting.
7. When both flows are at the pre-determined rates, allow 15 seconds for them to reach steady state.

8. Once steady state has been reached, the emulsion discharging from the venturi should be saved to make the handsheets.

9. The prepared emulsion should then be placed in a water bath set at the same temperature as the starch used for the emulsification.

10. The emulsifying apparatus should be kept running until all of the ASA is ejected from the syringe.

11. To clean the apparatus, flush hot water through all of the components until the system is void of entrained ASA and starch.

12. To prevent freeze-ups caused by the sticky nature of the FIBRAN, keep the apparatus full of water.

Emulsion Quality

It was thought that the emulsion quality could be checked by using a capillary tube viscosity procedure. Presumably, the capillary tube viscosities would determine the emulsion stability with respect to time. However, due to the low solids solutions used in the experimental emulsification, the control of 2% starch slurry and the sizing emulsion were too closely matched to determine any difference. Therefore, capillary tube viscosities were eliminated as a source for determining quality. As an alternative, a Reichert Binocular microscope was used to make a visual inspection of the emulsions.

Photographic Quality Check

1. Obtain a sample of freshly prepared emulsion from the apparatus.

2. Prepare a slide by placing two to three drops of emulsion directly on a clean glass slide, then cover it with a slide cover.

3. Photograph the emulsion at 12.5x magnification.

4. Using the same magnification as the emulsion photographs, photograph a graduated slide with a micron scale on it. This will provide a scale for comparison.

The quality of the emulsion is determined by the optimum colloidal particle size. From information obtained in the research aspect of this thesis, optimum particle size should range from one to two microns with an average size of approximately one micron (5).

Handsheet Make-Up

1. Make-up a Valley Beater following TAPPI method (T-200 os-70) using 180 grams OD bleached Kraft hardwood and 180 grams OD bleached Kraft softwood.
2. Refine the mixture to 450 csf following TAPPI method (T-227 os-58).
3. In a 55 gallon drum, dilute the stock to 1.2% consistency. Use de-ionized water obtained from a Calgon de-ionizing system.
4. Standardize the de-ionized water to pH 7.6 with a reagent grade Sodium Hydroxide.
5. Make British Handsheets following TAPPI standard (T-205 om-80) with alterations as follows:
    a. Mount four baffles in the cylinder above the sheet forming wire in the sheet mold. This will inhibit the formation of vortices caused by the mixer (Appendix IV).
    b. Place a propeller shaft mixer in the cylinder to facilitate the shear specifications.
    c. Add 0.3cc ASA emulsion and 0.5 cc 1.6% alum solution to the column (Appendix VI).
    d. The shear is controlled by using a variable speed transformer. Agitate at the prescribed shear for five seconds prior to draining.
    e. Dry the handsheets in a convection oven at 105°C for one hour -- to cure the FIBRAN.

Sampling Technique

In order to obtain statistically significant results, a pre-run trial was conducted to determine minimum sample size. It was found that 20 samples per condition were required (Appendix VII). The total number of conditions was 16; four different shear levels, and four different temperature levels. Six sheets per condition were made with four HST tests per sheet. This provided a total of 24 tests per condition.

		Shear Level				
		0	3	6	9	
Temperature (F)	150	A1	A2	A3	A4	
	125	B1	B2	B3	B4	
	100	C1	C2	C3	C4	
	75	D1	D2	D3	D4	n=24

Handsheet Testing Procedure

1. Condition for 24 hours in the paper testing laboratory at 50% relative humidity and 72°F.
2. Cut each sheet into four quarters.
3. Run a Hercules Size Test on each quarter following TAPPI method (T-530 pm-75).

Handsheet analysis and HST's were conducted in the paper testing room adjacent to the Pilot Plant paper machine. Two HST meters were set up in the testing room, and used throughout the entire period. Each handsheet was weighed and cut into quarters. Two of the quarters were tested on each machine. This procedure allowed two size tests to be run on each sheet by each of the HST meters.

RESULTS PRESENTATION

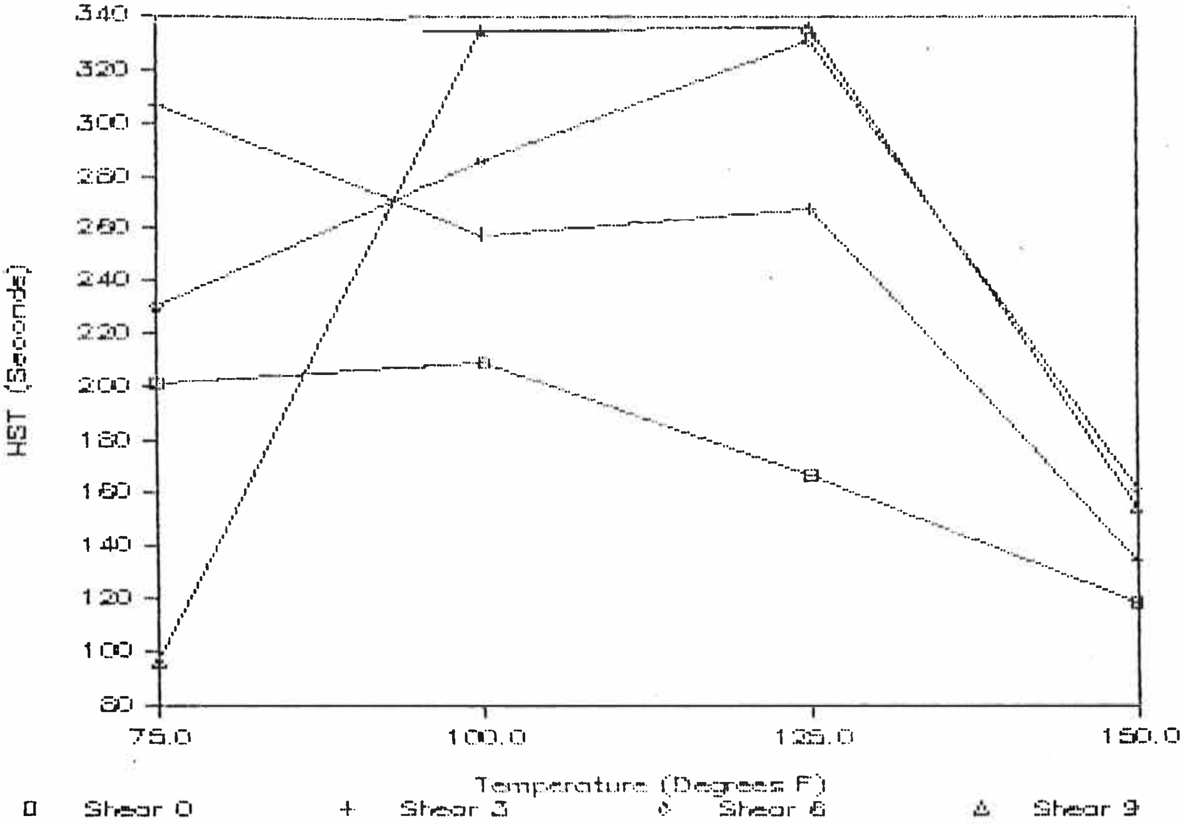
Temperature (F)	Shear Level 0	3	6	9
150	118.3 ±84.6	134.4 ±87.1	161.7 ±53.8	154.6 ±59.6
125	166.3 ±117.7	267.7 ±67.1	331.7 ±100.1	336.1 ±108.9
100	209.2 ±217.5	257.9 ±142.8	285.8 ±68.6	335.5 ±177.9
75	200.6 ±110.6	307.5 ±49.4	230.0 ±57.2	96.4 ±33.4

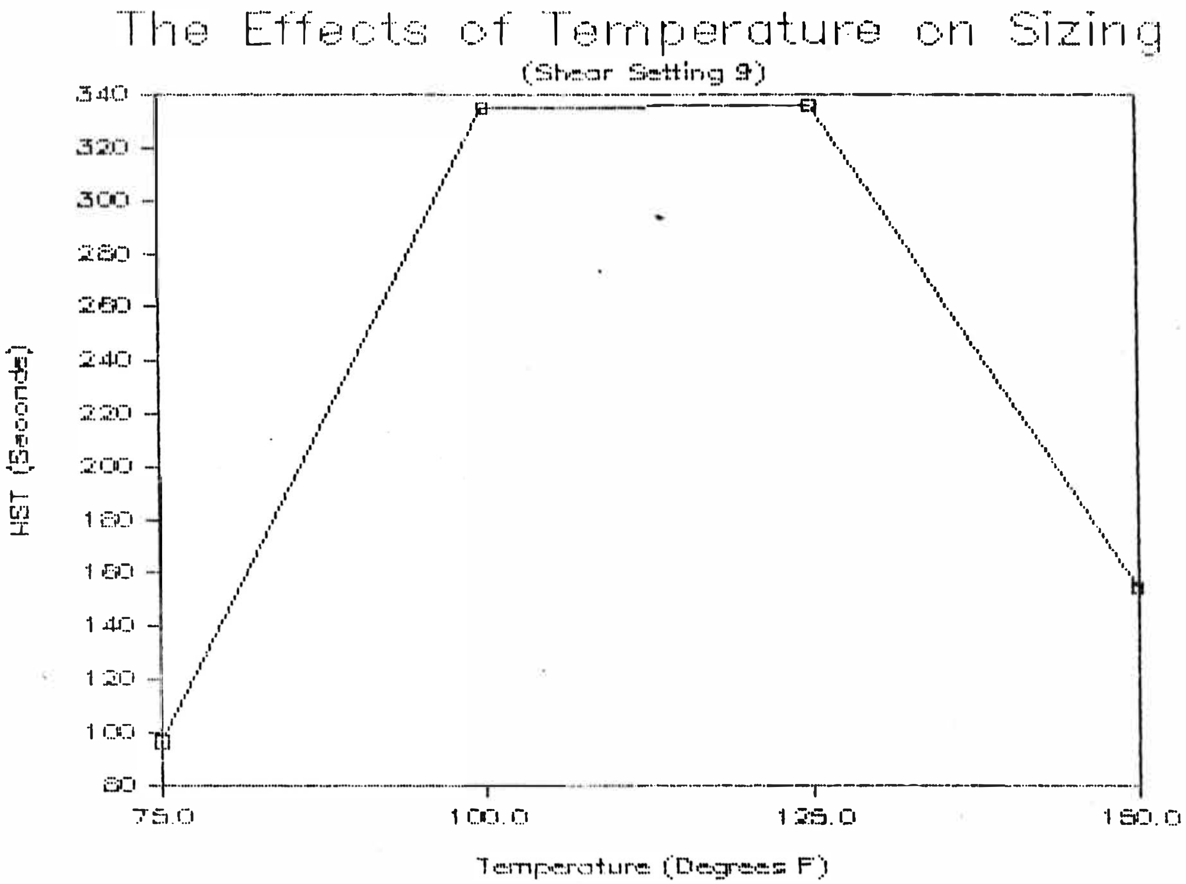
TABLE I

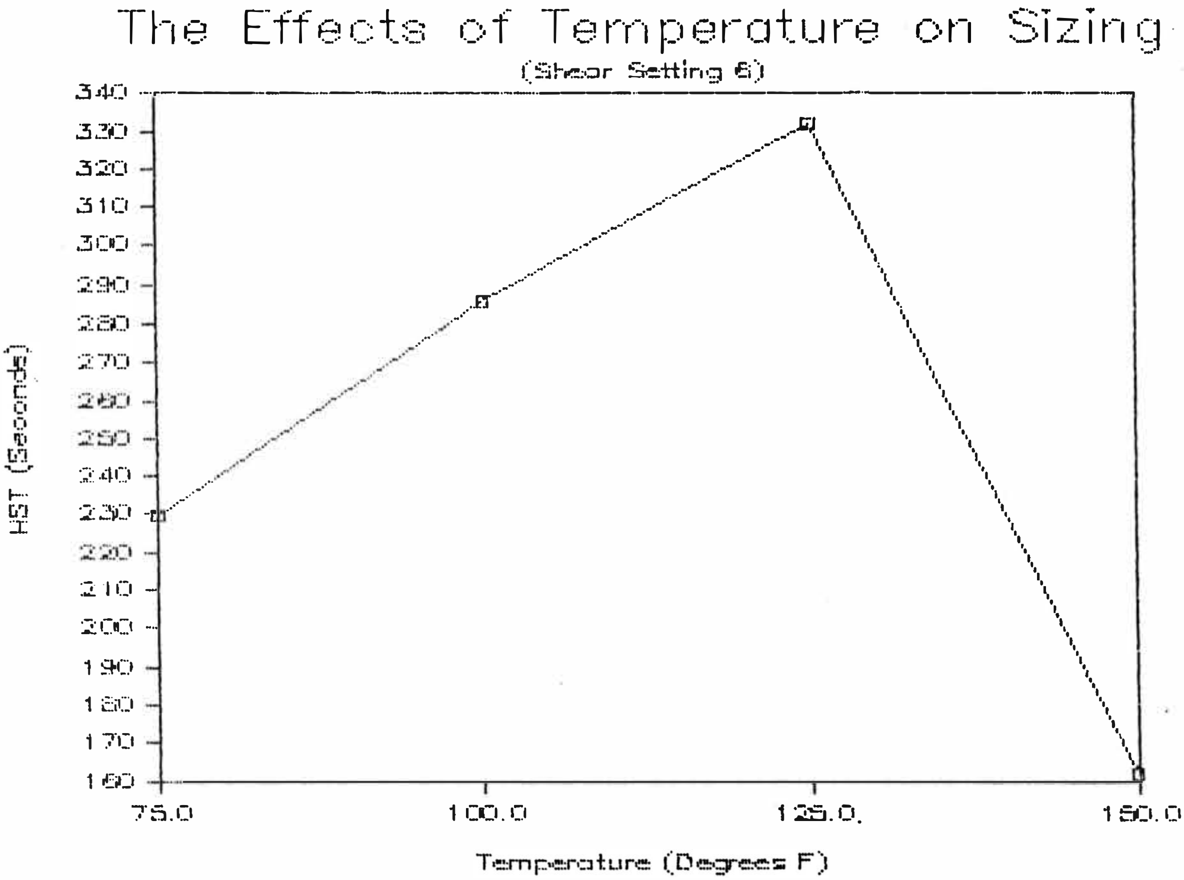
Table of Results

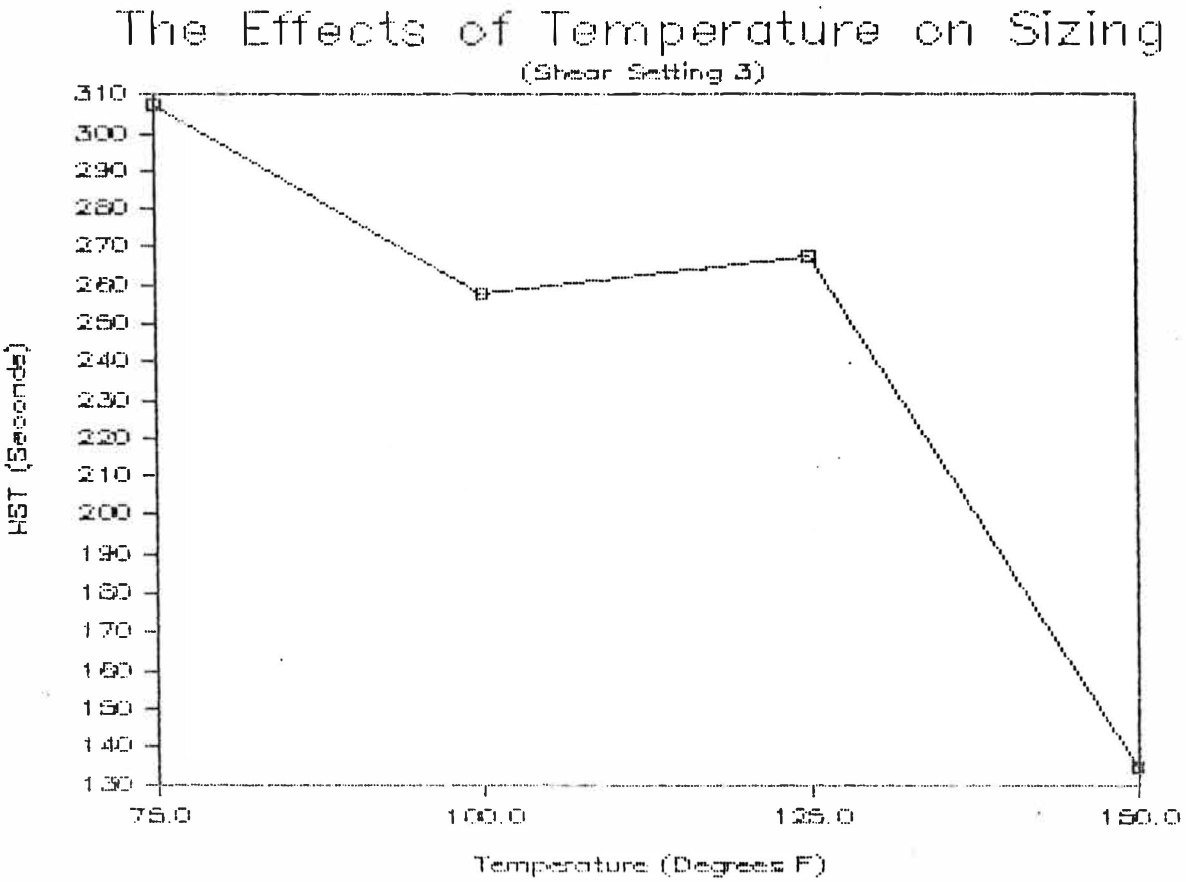
The data for Table I was tabulated using 20 of the 24 points in each condition. The four points farthest from the mean were discarded.

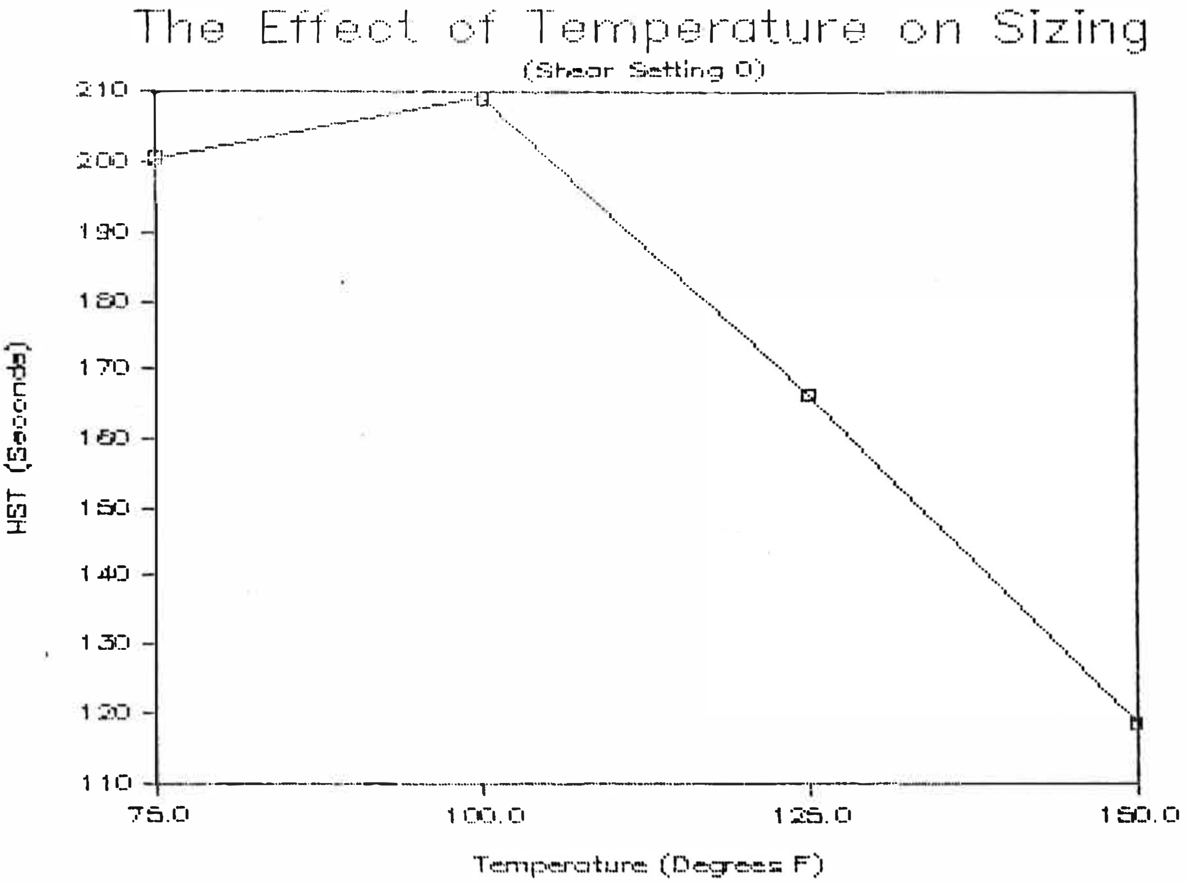
Following are individual graphs of starch temperature plotted against Hercules Size Test results. Following the temperature graphs are copies of actual photos taken of the emulsions at various temperatures. It is believed that key results to this experiment lie in correlation of the visual inspection of the photographs with the size test results, at the different temperatures.


The Effects of Temperature on Sizing


The Effects of Temperature on Sizing
(Shear Setting 9)
HST (Seconds)
340
320
300
280
260
240
220
200
180
160
140
120
100
80
75.0
100.0
125.0
150.0
Temperature (Degrees F)

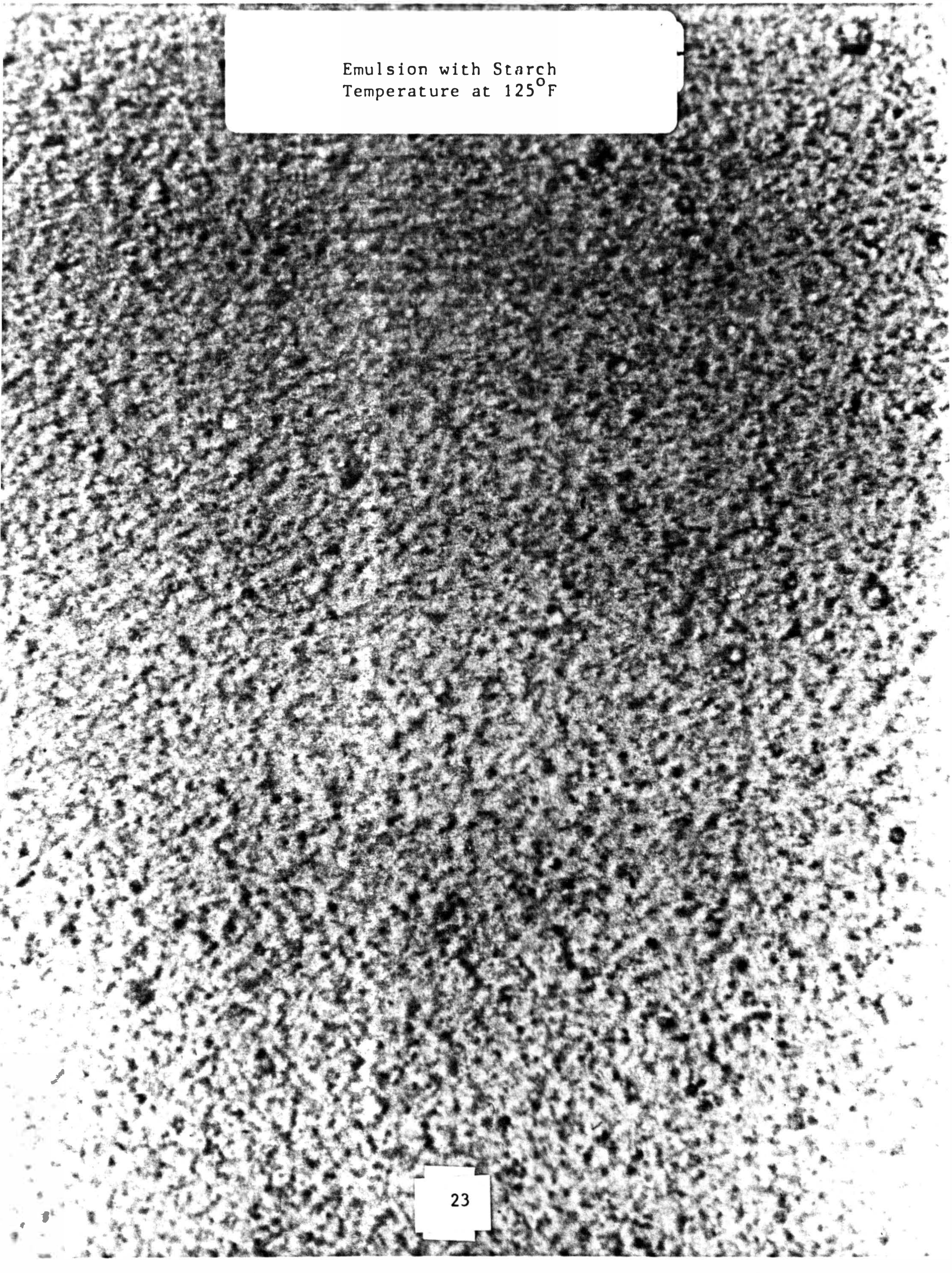

The Effects of Temperature on Sizing
(Shear Setting 6)
HST (Seconds)
340
330
320
310
300
290
280
270
260
250
240
230
220
210
200
190
180
170
160
75.0
100.0
125.0
150.0
Temperature (Degrees F)


The Effects of Temperature on Sizing
(Shear Setting 3)
HST (Seconds)
310
300
290
280
270
260
250
240
230
220
210
200
190
180
170
160
150
140
130
75.0
100.0
125.0
150.0
Temperature (Degrees F)

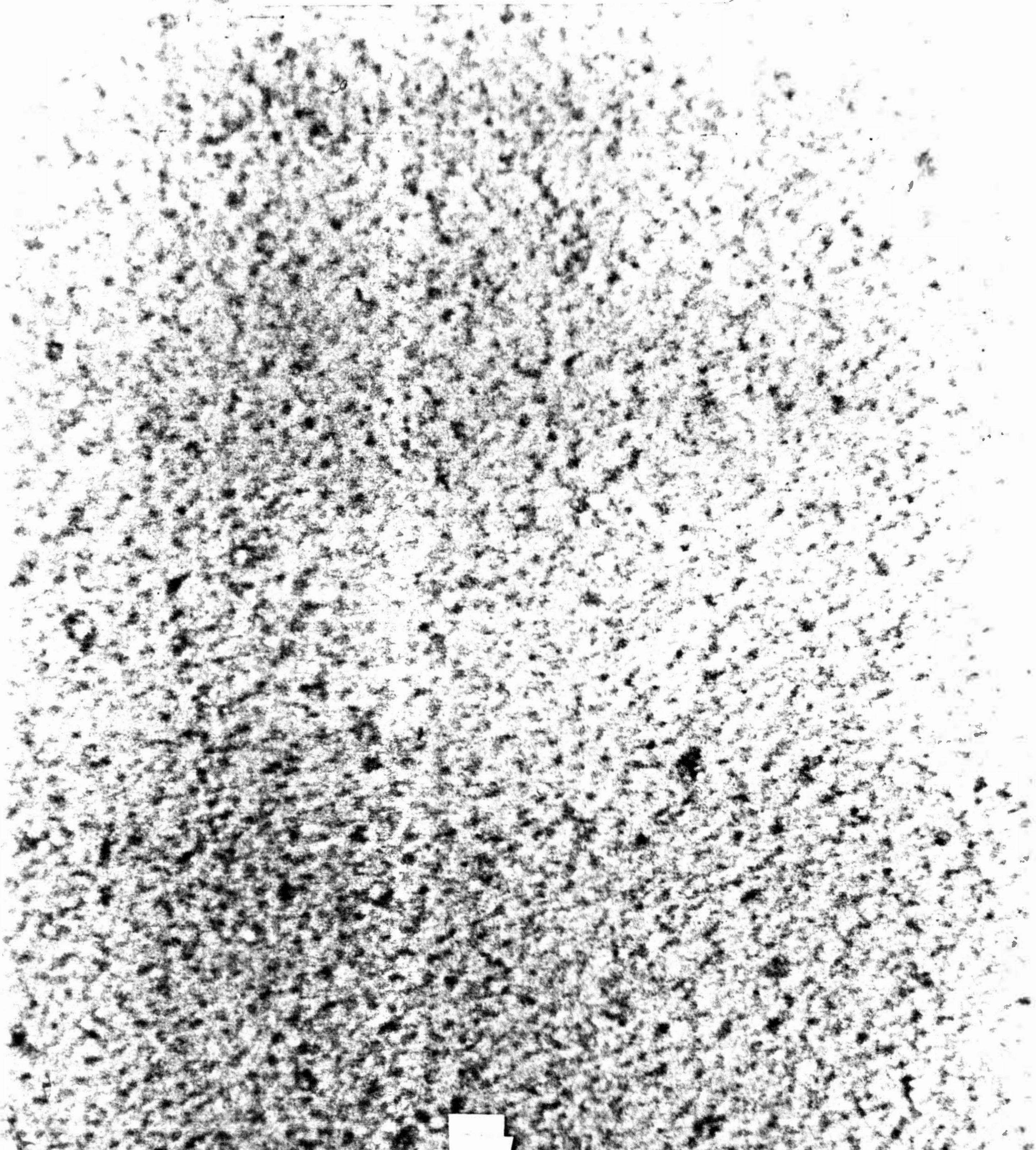

The Effect of Temperature on Sizing
(Shear Setting 0)
HST (Seconds)
210
200
190
180
170
160
150
140
130
120
110
75.0
100.0
125.0
150.0
Temperature (Degrees F)

Emulsion with Starch
Temperature at 150°F

Emulsion with Starch
Temperature at 125°F



Emulsion with Starch
Temperature at $100^{\circ}F$



Emulsion with Starch
Temperature at 75°F

DISCUSSION OF RESULTS

Table I shows the emulsion made with 150°F starch giving the lowest overall sizing results. The particle size distribution of the 150°F run ranged 5-40 microns, well above the 1-2 micron range suggested by National Starch Corporation.

Two possible conclusions can be drawn from the results of the 150°F run. One possibility is that emulsion particles of this size do not retain well in the sheet forming cylinder due to the inability of the secondary forces (cationic charge) of the starch to hold the particle in the fiber mat. The other possibility is that the high temperature caused an accelerated hydrolyzation of the FIBRAN leading to the low size test results.

The size test results of the 125°F and 100°F runs show a significant increase over the 150°F run. The photographs of these two emulsions show a more uniform particle size distribution ranging from 1-5 microns.

This information is supportive of the hypothesis that the secondary forces between the cationic starch and fiber have a greater interaction with smaller emulsion particle size. The fiber is able to more closely hold the smaller particles giving a stronger mechanism for retention.

The results obtained from the run using the maximum amount of shear in the forming cylinder (shear 9) shows the best supportive evidence for the effect of starch temperature on the sizing results. Not only do the results show an optimum temperature range between 125°-150°F, but the test results at shear 9 are higher than the results obtained from the lower shear rates. This indicates that the more uniform particle size distributions are more stable in high shear conditions. This is supportive evidence that smaller emulsion particle size allows more interaction between particle and fiber giving better retention of the emulsion in the sheet.

The results of the 75°F run are difficult to interpret because of the standard deviations found in the data. Analysis of the average results shows that they fall somewhere between the 150°F run and the 100°-125°F range. Industrial application of starch at 75°F would be difficult due to the tendency of starch to gel and skin at this temperature.

CONCLUSIONS

The basic conclusion of this study is that the temperature of the cationic starch slurry, prior to emulsification, does have an effect on the final sizing characteristics of the sheet. The data and the pictures of the emulsions support the hypothesis that small, uniform particle size distribution is essential in making a stable FIRBAN emulsion. The starch temperature range of 100°-125°F was found to give this uniform particle size distribution.

These results may have significant industrial applications, especially where a jet cooker is employed to cook the cationic starch. A jet cooker allows large quantities of starch to be cooked quickly and continuously, but at elevated temperatures. If the retention time in the holding tank is not sufficient to allow the starch to cool, the starch may enter the emulsifier at temperatures significantly greater than 125°F. A possible solution to this problem may be to cook at higher solids and use dilution water to cool the starch prior to emulsification.

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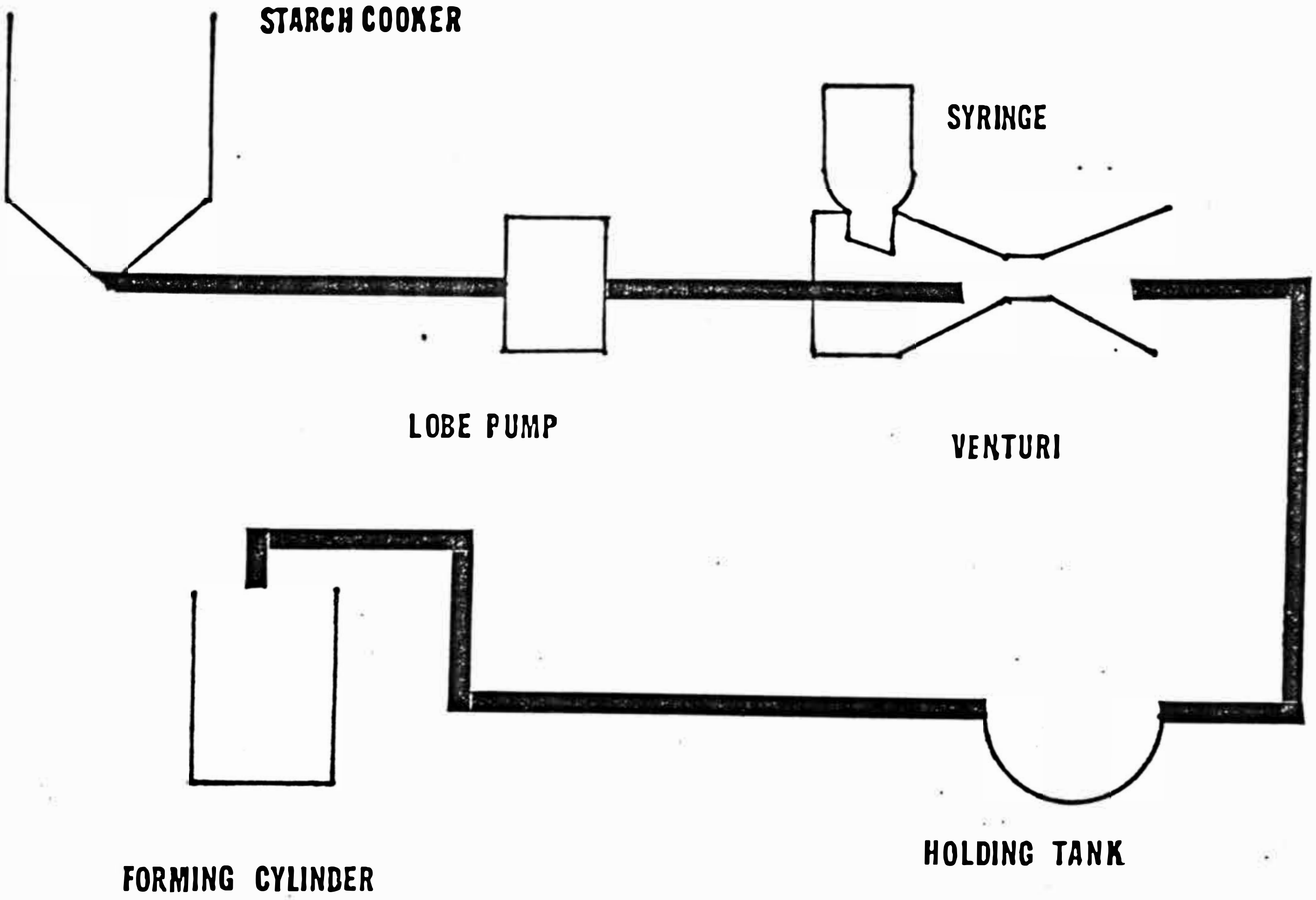
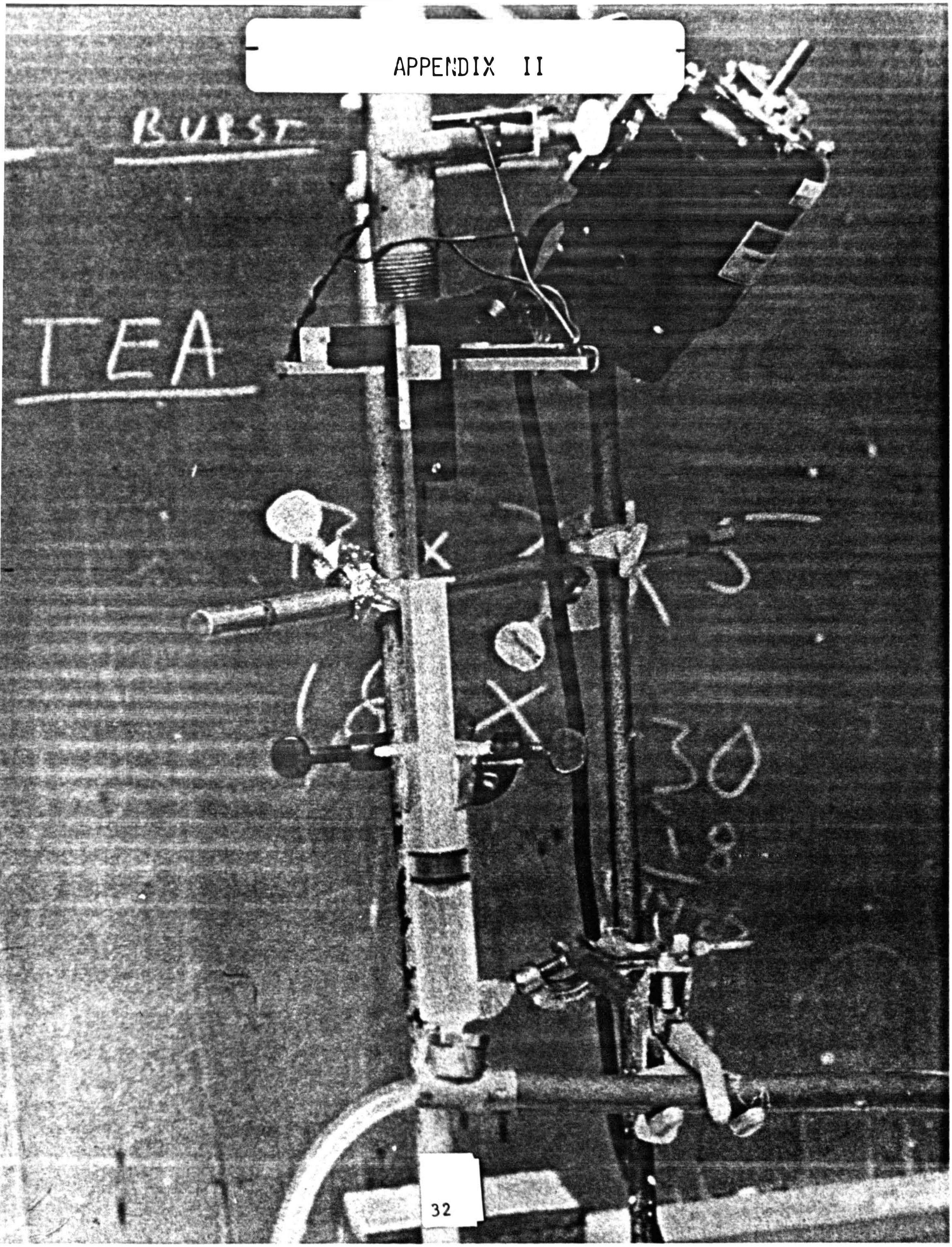
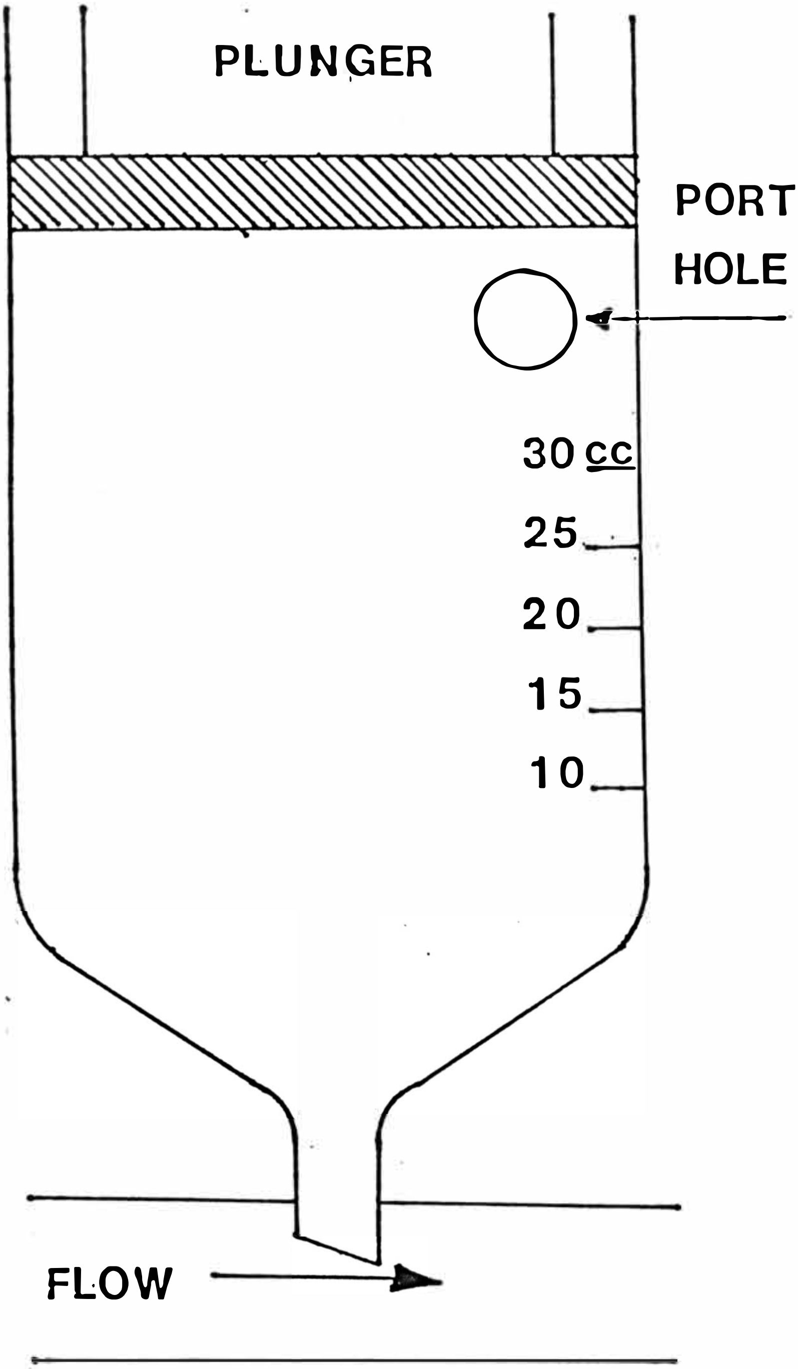
TABLE OF APPENDICES


STARCH COOKER
SYRINGE
LOBE PUMP
VENTURI
FORMING CYLINDER
HOLDING TANK

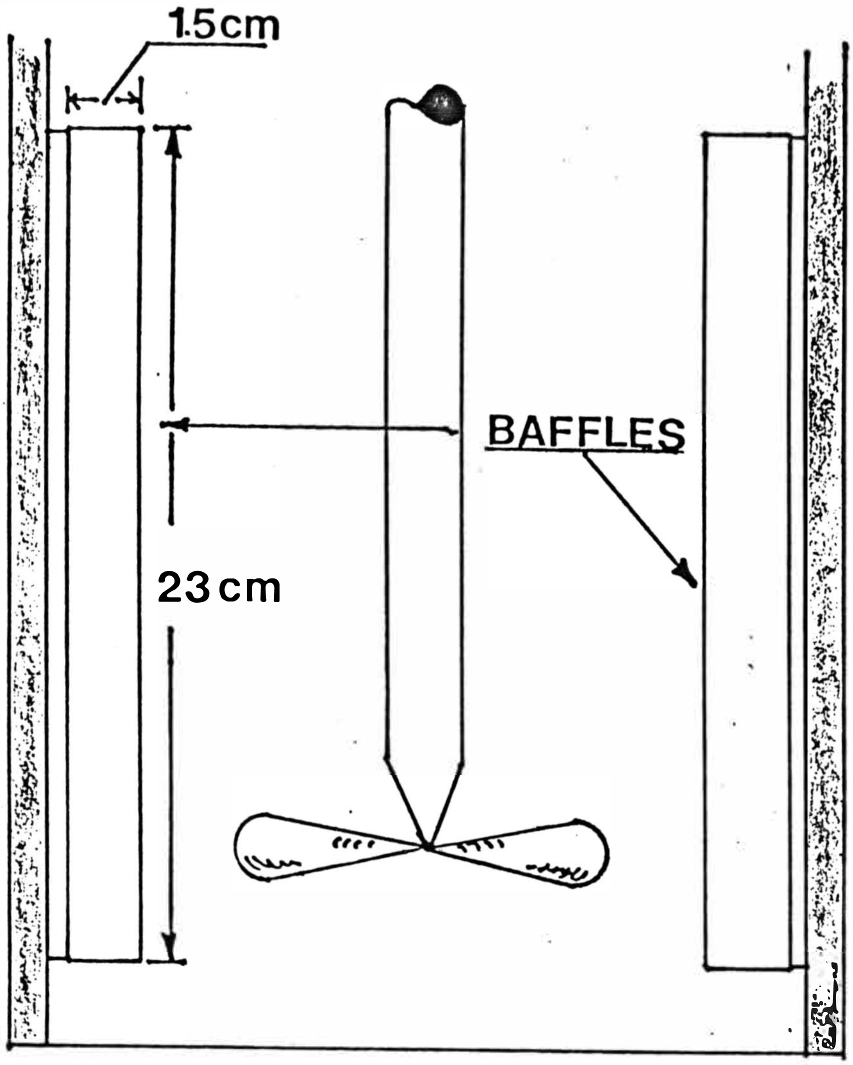
APPENDIX I

APPENDIX II





APPENDIX III



FORMING CYLINDER

APPENDIX IV

Appendix V

Addition Rate Goals

3.50# FIBRAN/Ton @ 93% Solids
8.75# CATO 15/Ton @ 2% Solids

$$\frac{3.50\#\ \text{FIBRAN}}{2000\#\ \text{O.D. FIBER}} \times 100 = .175\%\ \text{FIBRAN}$$

$$\frac{8.75\#\ \text{CATO 15}}{2000\#\ \text{O.D. FIBER}} \times 100 = .438\%\ \text{CATO 15}$$

$$\frac{1.20\text{g O.D. FIBER} \times .175\%\ \text{FIBRAN}}{0.93\%\ \text{Solids}} = 2.26\ \text{mg/Sheet}$$

$$\frac{1.20\text{g O.D. FIBER} \times .438\%\ \text{CATO 15}}{22.0\%\ \text{Solids}} = 26.3\ \text{mg/Sheet}$$

FIBRAN Addition Rate

$$\frac{1230\text{g CATO 15}}{1\ \text{Minute}} \times \frac{1\ \text{Sheets}}{.263\text{g CATO 15}} = \overset{4676.8}{\cancel{4685.7}}\ \frac{\text{Sheets}}{\text{Minute}}$$

$$\frac{\overset{4676.8}{\cancel{46685.7}}\ \text{Sheets}}{1\ \text{Minute}} \times \frac{2.26\text{mg FIBRAN}}{\text{Sheets}} = \frac{\overset{10.57}{\cancel{10.58}}\text{g FIBRAN}}{1\ \text{Minute}}$$

Appendix VI

Emulsion addition

2.26 mg FIBRAN + 26.3 mg CATO 15 = 29 mg ≈ .3 cc

Alum addition

Addition rate goal 14# Alum/200# O.D. FIBER = .7%

$$\frac{1.2\text{g O.D. FIBER}}{1\text{ Sheet}} \text{ X } .7\%\text{ Alum} = \frac{8.4\text{ mg Alum}}{1\text{ Sheet}}$$

$$(1.6\%\text{ Alum}) \times (Ycc) = 8.4\text{ mg Alum}$$

$$\frac{Ycc = .53\text{ cc of }1.6\%\text{ Alum}}{1\text{ Sheet}}$$

Appendix VII

ESTIMATING THE MEAN

ESTIMATING THE MINIMUM REPLICATIONS REGQUIRED FOR A SPECIFIC ERROR OF MEAN AT A CERTAIN LEVEL OF CONFIDENCE

If the average value of a series of observations is used as an estimate of the mean, we can be (1-a)100% confident that the error will be less than a specified amount e when the sample size is at least:

$$n=(Z_{a/2} \text{ x s.d. } / \text{ e})^2$$

where, n = the mimimum number of observations

$Z_{a/2}$ = the Z value above which we find an area of a/2 in the distribution curve

s.d. = the standard deviation of the recorded measurements

e = The error of mean. How close we want our observations' average to approximate the true mean value.

For this study, $Z_{a/2}$ = 01.96

s.d. = 71.30

e = 30.80

Plugging these numbers into the above equation results in the minimum replications required (n) to be at least 21.