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The Effect of Shear on Asa Sized Paper

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THE EFFECT OF SHEAR ON ASA SIZED PAPER

By

Phillip G. Purpura

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

Western Michigan University
Kalamazoo, Michigan

Dr. Richard B. Valley, Advisor

ABSTRACT

Alkaline papermaking systems have many advantages over the conventional acid systems; however, they have disadvantages in that they do not produce a highly sized sheet of paper consistently. This study investigates how shear, at the point of addition of an Alkenyl Succinic Anhydride/starch emulsion to a cellulose system, effects the sizing properties of a sheet. It was found that at no agitation, the sizing was low and very non-uniform. This was probably due to a lack of mixing, and thus a lack of contact between the emulsion and the fiber. The sizing increased as the mixing and contact between the emulsion and the fiber increased. However, with extremely high agitation, no apparent increase or decrease in sizing was observed. This could be due to either a flattening of the curve towards the top of the peak prior to decline, or possibly a constant trend that would remain and not decline.

KEYWORDS

Alkaline papermaking, Alkenyl Succinic Anhydride (ASA), Internal Sizing, Shear, Emulsion.

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THE EFFECT OF SHEAR ON ASA SIZED PAPER

INTRODUCTION

Alkaline sizing systems are becoming more popular within the paper industry. These alkaline systems have many advantages over the conventional acid systems; however, they have disadvantages in that they do not produce a highly sized sheet of paper consistently. Alkaline sizing systems utilize Alkenyl Succinic Anhydride (ASA) as the main sizing agent, and because of the limited research done on ASA, the critical variables have not been isolated. My senior thesis work is to thoroughly study one of these variables in order to partially reduce the large gap in the industry's knowledge. This study proposes to determine how shear, at the point of addition of an ASA-starch emulsion to a cellulose system, effects the sizing properties of a sheet. There are many mechanisms that determine the effectiveness of ASA including emulsification, retention, and shear forces. These mechanisms merit discussion and will be addressed in this paper. The first topic of discussion is the alkaline papermaking system; its advantages, and justification for conversion which will give credence to my work.

THEORETICAL

Alkaline Papermaking

Alkaline papermaking is not a new concept. The process has been recognized for many years. However, it has only recently become more appealing to the papermaking industry due to advances in techniques and materials. Synthetic sizes and polymer additives have improved to the point where they have made alkaline papermaking an excellent system. A review of the published literature indicates that there are a number of potential advantages associated with this alkaline system. (1,2) The potential advantages are as follows:

- REDUCED ENERGY USAGE

The use of alkaline systems can lower the producers' energy costs by virtue of the fact that the ASA system is a more completely closed system which allows for retained heat and energy in the stock. Also, since alkaline sheets are generally stronger, they require less refining to develop equivalent strength. Additionally, alkaline sheets will allow for more filler loading which will reduce drying energy requirements. These are important advantages since power usage reduction is now an important issue in an energy-conscious industry.

- REDUCED CORROSION
- IMPROVED MACHINE CLEANLINESS

Acid systems have always been corrosive, and they cause extensive wear on papermaking equipment. Also, as white water systems close-up, a build up of corrosive salts from the process can accelerate these problems. The use of an alkaline system, pH 7 or above, can reduce this corrosive action and extend the life of piping and equipment. This will in turn reduce maintenance costs and improve machine life. Slimacide and defoamer usage is lowered. Machine felts have longer life and clean-ups are easier.

- INCREASED SYSTEM CLOSE-UPS
- EASIER EFFLUENT TREATMENT
- REDUCED FRESH WATER REQUIREMENTS

As previously mentioned, the alkaline system does not build up corrosive salts, alumina, or other contaminants which make white water close-ups more difficult. White water in an alkaline system can be reused in many areas of the mill with far fewer problems. With more reuse of white water, there is less effluent to be put into a wastewater treatment plant, and the mill experiences lower fresh water requirements. The effluent to be treated is already at a neutral or alkaline pH, and contains less troublesome contaminants which reduces both the cost of treatment and pH adjustments.

- INCREASED DRY STRENGTH
- INCREASED FILLER LOADING
- INCREASED SECONDARY FIBER USE
- INCREASED HARDWOOD USE

Alkaline papers are stronger than non-alkaline sheets. This is due, in part, to the increased strength of individual fibers at alkaline pHs. Also, alkaline sizing agents tend to interfere with the fiber bonding process less than do conventional rosin-alum sizing systems. (1) This increased fiber strength can lead to a reduction in refining, and the replacement of a strong fiber with a weaker fiber and sheet fillers. Weaker fibers, such as secondary fiber, can be utilized to reduce costs and improve sheet formation as well as feel. With alkaline systems, calcium carbonate can be used which will provide improved sheet properties (over clay filled sheets). With additional sheet loading, the cost of materials can be reduced which will increase a company's profitability.

- INCREASED SHEET PERMANENCE

Since alum is the major component that degrades paper, and alkaline papers retain only a minimal amount of alum in a sheet, they tend to age less rapidly than acid papers--making them more permanent. An acidic sheet tends to degrade at an accelerated rate over time because the acids formed increase the sheet's acidity which in turn accelerates the degradation process. Alkaline papers will also buffer the system. Increased longevity and permanence are highly desirable factors in papers used for

documents, books, and other pH sensitive applications.

These are only some of the examples of improvement with alkaline papermaking that are cited in the literature. The degree of improvement is specific to individual mill operations. These advantages are associated with the alkaline papermaking system; however, the use of Alkenyl Succinic Anhydride as a sizing agent in these systems has its own advantages.

Alkenyl Succinic Anhydride

Alkenyl succinic anhydride is a high molecular weight compound containing an unsaturated carbon side chain, which gives it its hydrophobic characteristics. This structure, and its bonding ability with cellulose, gives Alkenyl Succinic Anhydride its own advantages in addition to those connected with alkaline papermaking. In a report prepared by Brink and Gasper of National Starch (3), these advantages are highlighted. They include:

- a chemical bond is formed with cellulose
- most grades of pulp can be used
- most normal fillers can be used
- more hardwoods can be utilized
- machine runability is improved
- product first quality is improved
- first pass retention is higher

- dry efficiency is improved
- coater runability is better
- machine wash-ups are easier
- machine clothing life is extended
- pH is more stable
- temperature of operation is higher

With these many advantages, ASA and alkaline papermaking becomes a much more desirable system, and further research to control its application variables becomes essential.

ASA Mechanism

ASA is a sizing agent and, like all sizing agents, it has hydrophilic and hydrophobic groups. ASA is non-ionic to anionic and must therefore receive a charge to be retained in the sheet. The proper charge for particle retention is slightly negative. ASA is emulsifed with cationic starch to give it the necessary charge for retention on the fiber. The starch surrounds the ASA and protects it from hydrolysis until it has an opportunity to bond with the cellulose fiber. The ASA-starch colloid weakly associates with cellulose until the paper reaches the drier section. Here, the paper temperature elevates and a bond is formed. ASA is very reactive in the dryer section and forms a chemical bond with the cellulose chain. (Appendix VIII) Once the ASA is set onto the sheet, it repells water by having its

hydrophobic side chain exposed. This gives it its high degree of sizing. In order for this mechanism to work properly, a good first pass retention is essential. The emulsion is not very stable and will hydrolize and become ineffective if it is not retained first. Storage of the ASA-starch emulsion destroys its effectiveness because of hydrolization.

Sizing

Sizing refers to the resistance to liquid wetting, penetration, and absorption in paper and paperboard. This resistance is the result of a chemical additive known as a sizing agent. (4) Cellulose is very hydrophilic. Due to their high surface energies, cellulose fibers are easily wetted--acting much like sponges. The sizing agent works to create a hydrophobic layer on the fibers in order to retard surface wetting.

Liquid penetration into a sheet occurs by three processes. There is lateral movement on the surface of paper commonly called feathering. This action can be accelerated by capillary action of surface fibers. Also, liquid can move transversely or penetrate through the paper. Capillary action of pores or spaces between fibers draws the liquid into the sheet. Liquid can also travel through the cellulose material itself penetrating into the fibers and spreading along their surface. The degree of liquid movement in a sheet is seen in the contact angle formed between the

impinging liquid and the cellulose fiber surface. (Appendix IX) As the figure demonstrates, well sized sheets generally have a contact angle greater than 90 degrees. In order for a sizing agent to provide this high contact angle, it must provide a relatively low surface energy coating so that high energy liquids will form a high contact angle with the surface and retard the liquid movement.

Sizing agents have both hydrophilic and hydrophobic parts. The hydrophilic part will bond with the cellulose leaving the hydrophobic part pointing outward. The outward projections form a hydrophobic surface which retards the liquid movement in the sheet. (Appendix IX) In an ASA system, the long carbon side chain acts as the hydrophobic part providing sizing in the sheet after drying.

The mechanism for sizing in a rosin/alum system has been studied in detail (4). Dr. Richard Valley suggests that part of this mechanism (the precipitate retention by fiber), also applies to the ASA/starch system used in alkaline papermaking. (9) This mechanism, also known as the electrostatic mechanism, states that the presence of aluminum salts on size particle surfaces gives the particles a positive charge in water. Cellulose has a negative charge at its surface. Since the cellulose and the size particles are oppositely charged, retention of the size on the cellulose surface will occur by simple electrostatic attraction. (4) Once the sheet has been sized, adequate liquid repulsion

should be provided. In order to measure this liquid repulsion, a standard testing procedure should be used.

Sizing Measurement

There are several methods of measuring penetration of liquids into a sheet of paper. These methods are categorized by the type of method used to measure the end point of the penetration of the liquid. (5) One type measures the conductivity of paper as a conducting fluid (such as salt containing water), penetrates through the sheet. The major drawback with this method is its sensitivity to pinholes and to formation problems. Liquid can penetrate through a pinhole quickly before the bulk of the liquid penetrates yielding inaccurate results. This is especially critical in handsheets where formation is not always ideal. The second method is the use of reflectance to measure the penetration of liquid. The major weakness of these tests is the type of fluid used. In the Hercules Size Tester (HST), a low surface tension fluid containing formic acid is used. This fluid tends to enhance the penetration through the pores of the paper. There is evidence that the size-fiber bond strength is a factor in determining the rate of penetration of HST ink through the paper. This has been attributed to the formic acid in the HST ink.

The Hercules Size Tester is the most commonly used size tester.

Because of its ease of operation, the Hercules Size Tester will be used in this experiment to determine the size of the final sheets.

Shear and Retention

In order for ASA to do its job, it must first reach the fiber and remain there until it reaches the driers for setting. The mechanisms involved in the retention of polymer colloids have been discussed in work done by Stratton and Sikora. (7,8) This particle retention can be explained through two major theories of flocculation by polymers. The first theory is termed the bridging theory. (Appendix X) The bridging theory states that the polymer adsorbs on a particle to produce long loops and tails. When this particle comes close to another particle, the segments of extended loops and tails attach to the new particle forming a physical bridge. These bridges span the electrical double layer of particles that would normally keep them from bonding together.

The second theory is the electrostatic patch theory. (Appendix X) In patch theory, the polymer adsorbs onto the particle in a flat configuration instead of a looping-tail manner. The adsorption of the high charge density polymer provides the adsorption region with a positive charge. This local positive charge attracts negative particles giving the particles contact for secondary

bonding.

Both of the above mentioned mechanisms are shear sensitive. It has been shown that turbulence is beneficial and even necessary to provide the even distribution of polymer, and the movement of fiber, filler and fines leading to their mutual collision and aggregation (8). An excess of agitation can, however, break this network down giving poor retention. Though work has been done on the use of cationic polymers to aid in retention of fiber fines and filler and the systems reaction to shear, no available work has been done on the effects of shear in an ASA system. Just exactly what is the point where necessary agitation turns into excessive shear? This experiment proposes to answer that question.

EXPERIMENTAL APPROACH

In order to study only one variable at a time, a standard method for making paper with ASA sizing must be used. In my thesis work, the method was produced, and is explained in detail in the text of this report. My objective was to study the effect of shear, at the point of addition of an ASA-starch emulsion to a cellulose system, on sizing properties of the sheet. The procedure developed has provisions for the following: water water needs, stock make-up, chemical make-up, starch make-up, preparation of the sizing agent/nonionic surfactant emulsion,

sheet preparation, preparation of the ASA-starch emulsion, addition of sizing emulsion to stock, and sheet making procedure.

The addition of the sizing emulsion to the stock was varied with the use of a Variac and tachometer attached to the mixer blades. To find the mixing speeds, some preliminary lab work was done. The key to this experiment's success, and also its major problem, was to keep all other variables constant. That is: stock temperature, pH, drying time, etc., must be carefully controlled. After the sheets were formed, sizing was tested using the Hercules Size Tester. The results will be reported in this paper; however, my assumption was that the results should yield optimum shear rates and energies to achieve optimal sizing performance.

EXPERIMENTAL PROCEDURE

Apparatus Construction

This project deals with three separate theses--all vitally linked together in an effort to generate valuable 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 approaches--each according to the primary mechanical device involved. These devices are: the starch cooker, the emulsifier, the holding tank, and the handsheet former. (Appendix I) 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 run the experimental procedure. There was no 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 test 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 Phillip G. Purpura and Steven S. Riegsecker. Prior to its construction, the only method of preparing this emulsion known to us was a procedure in which activated ASA and cationic starch were mixed in a batch-wise, low shear method. It is our belief 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 lob-pump. (Appendix II)

The starch was pumped from the holding tank, through a newly designed glass venturi which was made by heating a hollow glass rod, and then stretching it to produce a restricted central orifice--the size and shape most closely resembling an "ideal" venturi. A Masterflex pump (a lobe-pump giving a plug flow) was used to pump the starch from the holding tank through the new 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 new 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.

The ASA had to be added to the starch flow prior to the mixture's entering the venturi. (Appendix III) Also, a continuous flow of ASA, at a pre-determined rate, was required. In order to provide this continuous flow, a "power syringe" with its discharging opening at the center of the pipeline leading into the venturi was constructed. This allowed the ASA to be added to the starch at a point directly preceeding the area where maximum shear would be generated. The point of addition needed to be as close as possible to the venturi opening in order to limit the time that the ASA was in contact with the starch prior to emulsification. A control for the rate of addition was fashioned by mounting a microscope slide directly above the syringe plunger. The guide

translated the circular rotating motion of the drill to the downward motion of the plunger. Ath 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 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. However, 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 tank, 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 shear conditions. The adaptions consisted of four baffles and a high shear mixer mounted in the water holding cylinder directly above a wire screen. (Appencix IV) The purpose for using the baffles was to eliminate any vortices that might be formed as shear was applied through the mixer. 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 as 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 30 ml 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. Weigh out 116.8 pounds of de-ionized water and charge the cooker.

4. Weigh out 2.5 pounds of CATO 15 and add to the starch cooker.

5. Turn on the agitator.

6. Open the steam valve and allow the temperature to reach 180 degrees F. Close the steam valve. The temperature will drift above 190 degrees 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 5 gallon container until it reached the temperature specifications required.

FIBRAN Preparation

1. Weigh 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 10.58 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 solution.

(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. A high flow rate will cause an excessive back pressure diluting the FIBRAN.

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 is realized, increase the starch slurry flow 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

which is set at the same temperature as the starch to be 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 in the tubing.

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 particles. In this way, an average particle size was determined.

Photographic Quality Check

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

2. Prepare a slide by placing 2 to 3 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 1-2 microns with an average size of approximately one micron.

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 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, to reduce the formation of vortices caused by the mixer. (Appendix IV)

   b. Place a propeller shaft mixer in the cylinder to provide variable shear.

   c. Add 0.3 cc ASA emulsion and 0.5 cc 1.6% alum solution to 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 degrees 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.

Shear Level

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

Six sheets per condition were made with four HST tests per sheet. This provided a total of 24 tests per condition.

Handsheet Testing Procedure

1. Condition for 24 hours in the paper-testing laboratory at 50% relative humidity and 72 degrees 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 HSTs 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. Testing variables were minimized by noting the serial numbers of each machine on each quarter tested.

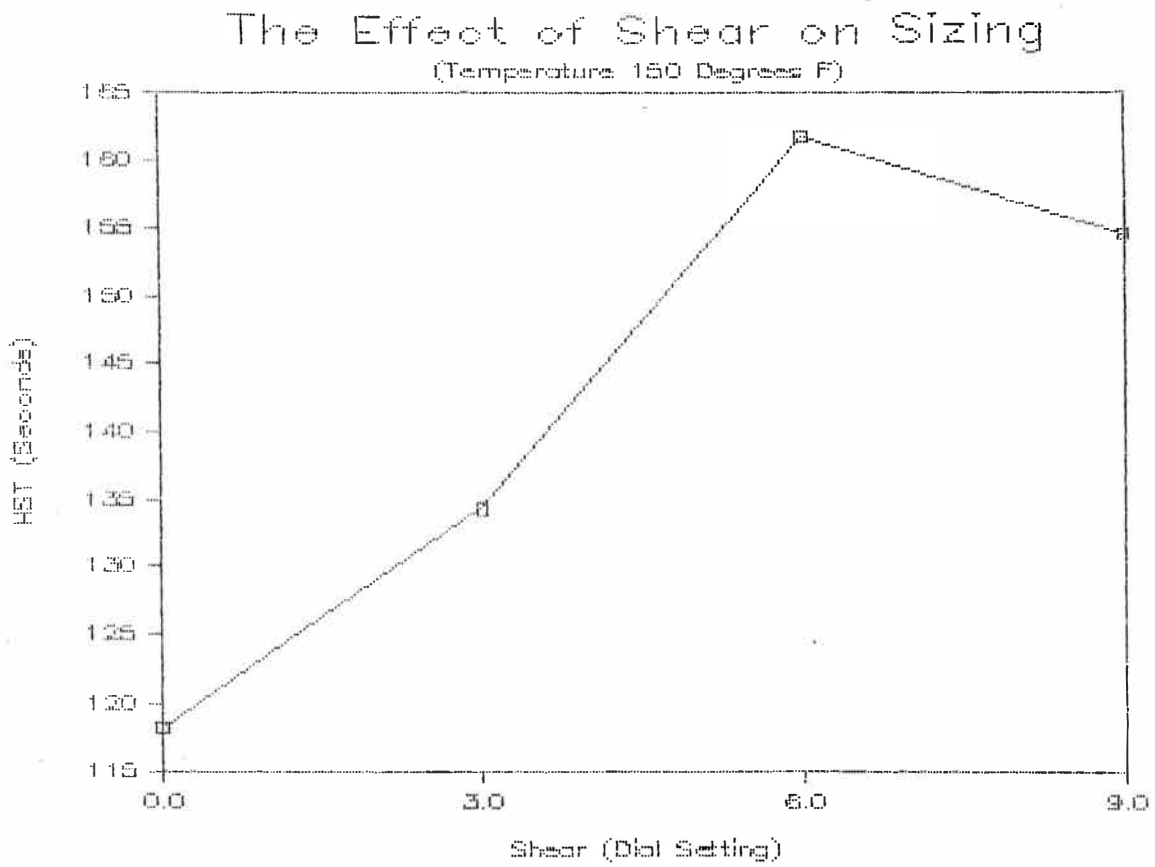
RESULTS PRESENTATION

In the experimental procedure, the amount of shear induced in the stock as the ASA/starch emulsion was added, ranged from no shear or mixing to the maximum shear generated by a laboratory mixer. The exact amount of shear generated by the mixer is difficult to measure; however, by varying the shear over a range, general

trends relating to the effects of increasing and decreasing shear can be shown.

The full range of shear was generated under four different emulsion temperature conditions. The report prepared by Steven S. Riegseker relates the starch temperature used for emulsification to the quality of the final ASA/starch emulsion.

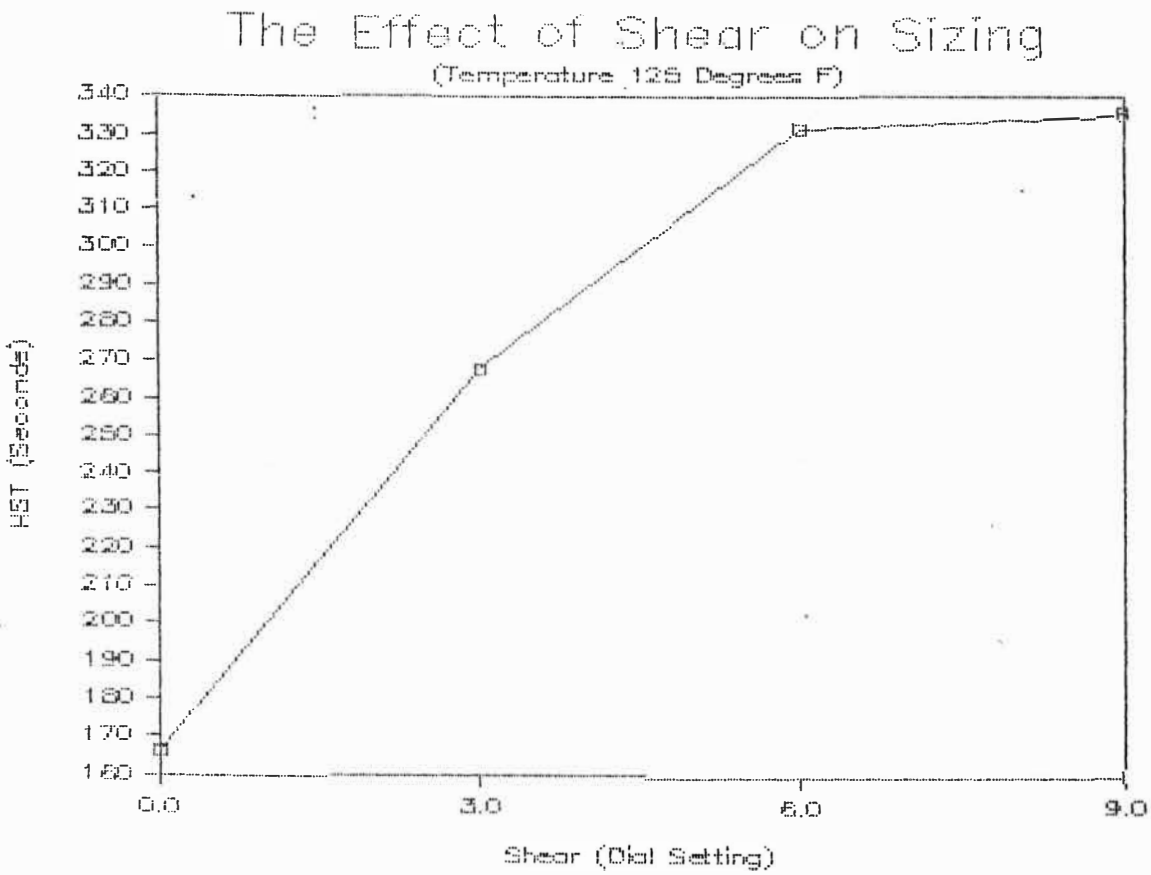
The first set of shear data was collected when the ASA/starch emulsion was 150 degrees F. A photograph of the emulsion (Appendix XIV) was used to measure the average particle size and range. The average particle of the 150 degrees emulsion was 10 microns with a range from 5 to 40 microns.



Temperature of emulsion (degrees F)	Shear setting	Average HST (Seconds)	Standard deviations
150	0	118.3	84.6
150	3	134.4	87.1
150	6	161.7	53.8
150	9	154.6	59.6

The shape of the graph at this temperature indicates that the level of sizing increases as shear increases; however, it would be difficult to draw further conclusions since the standard deviations associated with the graph are extremely high.

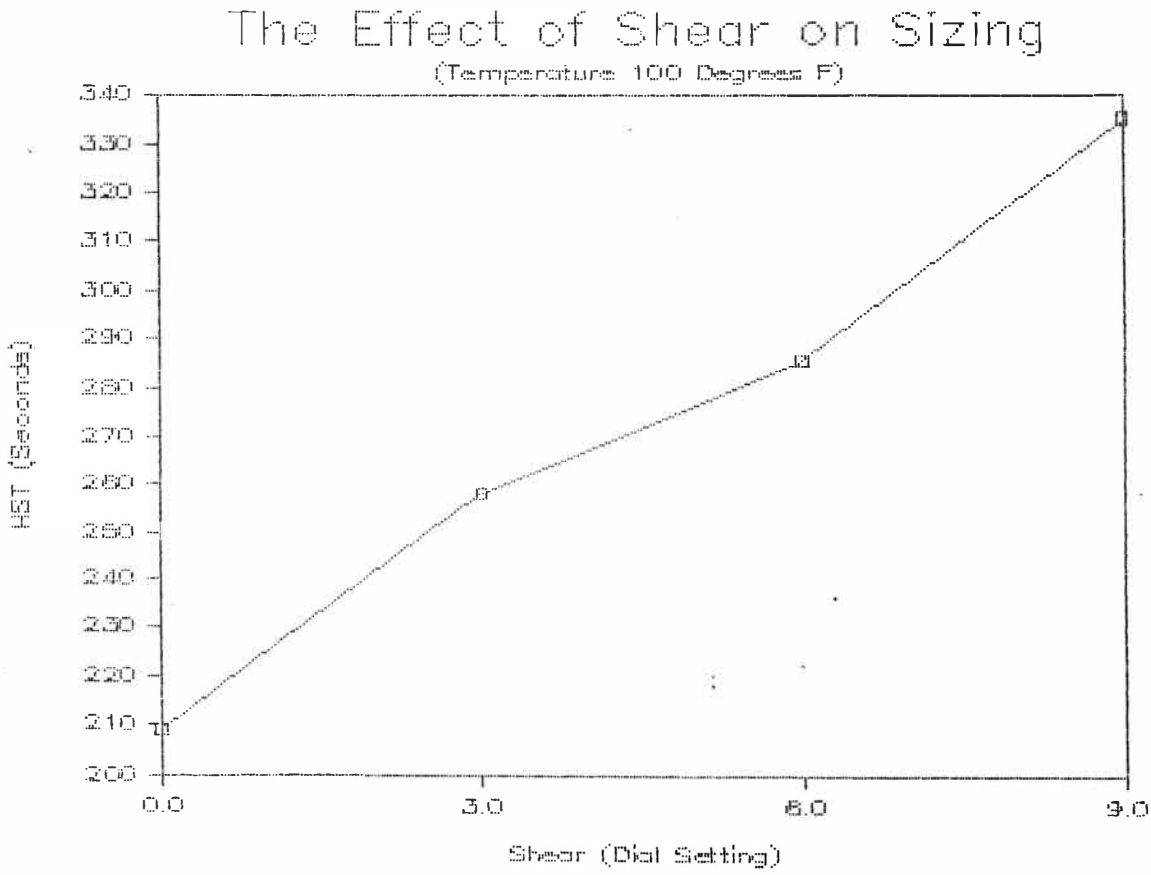
The second set of shear data was collected at an ASA/starch emulsion temperature of 125 degrees F. The emulsion photograph indicates an average particle size of 2 microns and a range from 1 to 5 microns.



Temperature of emulsion (degrees F)	Shear setting	Average HST (seconds)	Standard deviations
125	0	166.3	117.7
125	3	267.7	67.1
125	6	331.7	100.1
125	9	336.1	108.9

This graph indicates that there is an increase in sizing as the level of shear is increased.

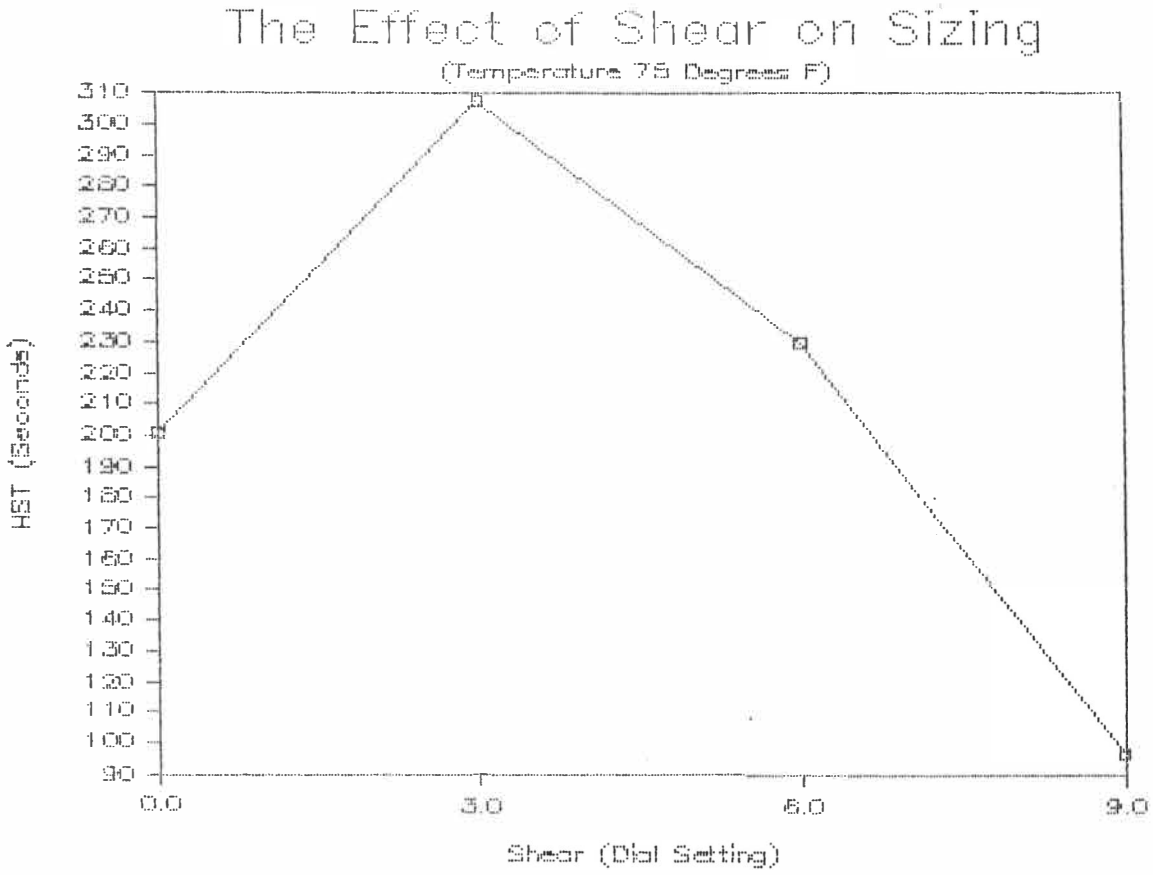
The third set of shear data was collected when the starch/ASA emulsion temperature was 100 degrees F. A photograph of the emulsion (Appendix XIII) shows that the average particle size of the emulsion is 1 micron with a range from 0.5 to 3 microns.



Temperature of emulsion (degrees F)	Shear setting	Average HST (seconds)	Standard deviations
100	0	209.2	217.4
100	3	257.9	142.8
100	6	285.8	68.6
100	9	335.5	177.8

This graph indicates an almost linear increase in sizing as the shear setting is increased. Again, further analysis is difficult due to the high standard deviations.

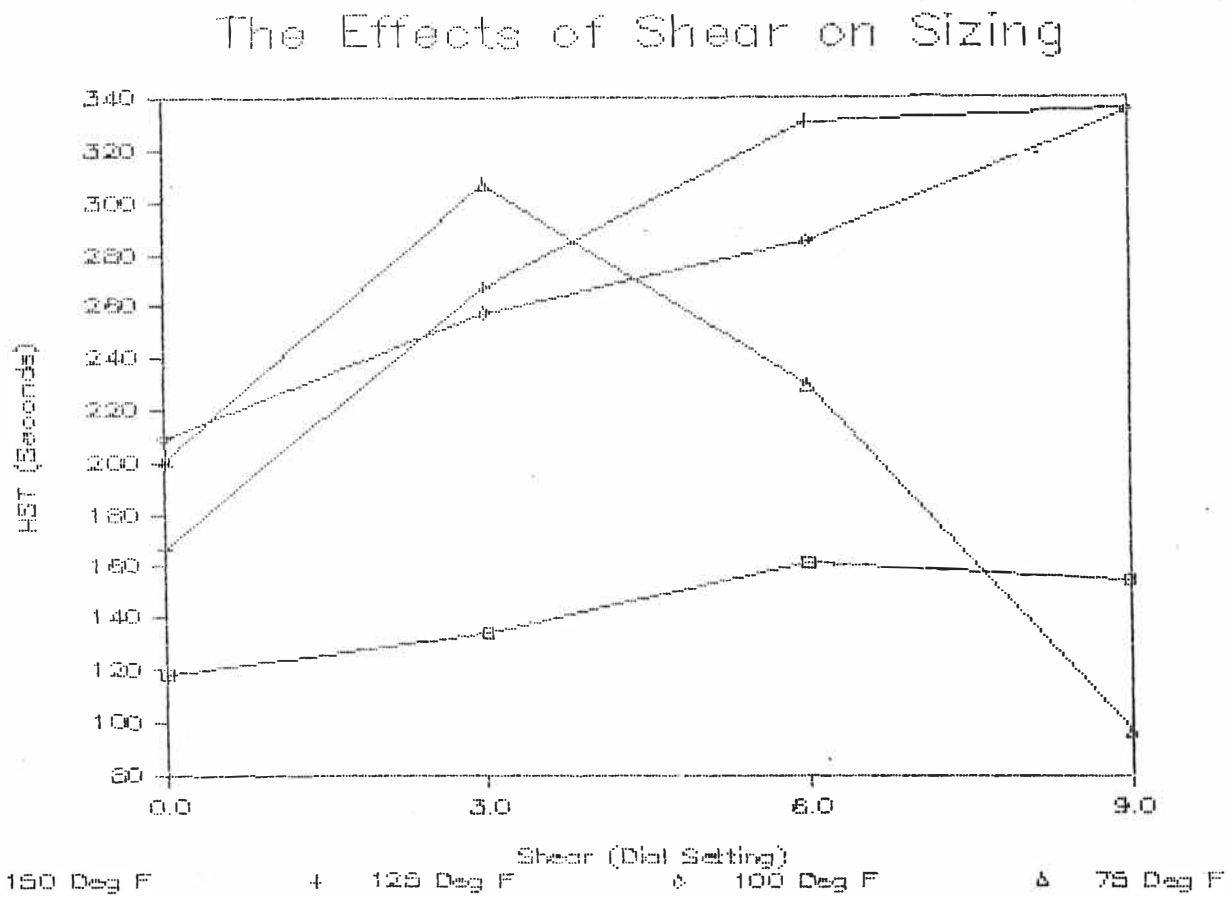
The fourth set of data was collected at an emulsion temperature of 75 degrees F. The emulsion photograph shows the average particle size to be 3 microns with a range from 2 to 8 microns.



Temperature of emulsion (degrees F)	Shear setting	Average HST (seconds)	Standard deviations
75	0	200.6	110.6
75	3	307.5	49.4
75	6	229.9	57.2
75	9	96.4	33.4

The general trend in this graph is a decrease in sizing as shear is increased. Firm observations about the initial increase point are difficult to support.

A composite graph containing each of the separate curves was made in order to compare the curves.



The graph shows that the 150 degrees F curve produces sizing levels below those of the 125, 100, and 75 degree curves. In addition, the 125 and 100 degree curves resemble each other in shape and magnitude. The 75 and 150 degree curves both give low sizing at high shear.

DISCUSSION OF RESULTS

In order to discuss the results obtained in this experiment, it was necessary to determine the validity of the collected data. While evaluating the four different emulsions produced, it became apparent that not all of the emulsions were of the same quality, nor should they all be considered equally in the analysis. The emulsions produced at 150 and 75 degrees F had a large average particle size and an even larger range. This demonstrates that the emulsions produced at these temperatures are not of sufficient quality to afford uniform sizing--even if the shear conditions are not varied.

The two emulsions produced at 100 and 125 degrees F have a much more uniform particle distribution, and are excellent emulsions. Therefore, the discussion will be focused around these two temperature emulsions.

The two emulsions are physically very similar; therefore, it was not surprising that they produced curves and data that were very similar.

In my original hypothesis, I speculated that the curve of the final results would resemble a bell shaped distribution. That is; the sizing would be low with no agitation, peak at optimum shear, and decline past this optimum point. As a result of experimentation, it appears that the sizing at no agitation

(shear 0) is low and very non-uniform. This is probably due to a lack of mixing, and thus a lack of contact between the emulsion and the fiber. As would be expected, the sizing increases as the mixing and contact between the emulsion and the fiber increases (shear 3 and 6). At shear level 9, which is an extremely high agitation, no apparent increase or decrease in sizing (from shear level 6) was observed. This observation could be either a flattening of the curve towards the top of the peak prior to decline, or it could possibly be a constant trend that would remain and not decline. It was not possible to obtain a shear level higher than 9 in the laboratory; therefore, the results of higher shear could not be observed.

Since it was not possible to measure the shear at each of the different levels, it is not known if the shears above level 9 are significant or if they are higher than any shear generated on a fourdrinier machine.

CONCLUSIONS

1. The emulsions produced at starch temperature 150 and 75 degrees F were not of sufficient quality to be used in the analysis of the data.

2. At shear 0, low sizing results were due to the lack of contact between the emulsion and the fiber. In addition,

there was a high degree of non-uniformity.

3. At shear levels 3 and 6, there was improved sizing due to increased contact.

4. No increase in shear was observed past the shear level 6. This indicates that either a) no further sizing was possible or b) there was insufficient shear to increase or decrease sizing.

RECOMMENDATIONS

The original intent of this study was to find an optimum shear level which could be recommend along with the best point of addition for an ASA/starch emulsion. The findings of this study suggest that when a high quality emulsion is used, a higher shear produces higher sizing results. Therefore, it would be adviseable to add the emulsion at a point prior to a high shear condition--such as directly before the fan pump.

As a follow-up to this work, it is recommended that shears higher than the level 9 be investigated, or an attempt should be made to provide proof that shear level 9 is higher than any shear generated in the wet end of a paper machine.

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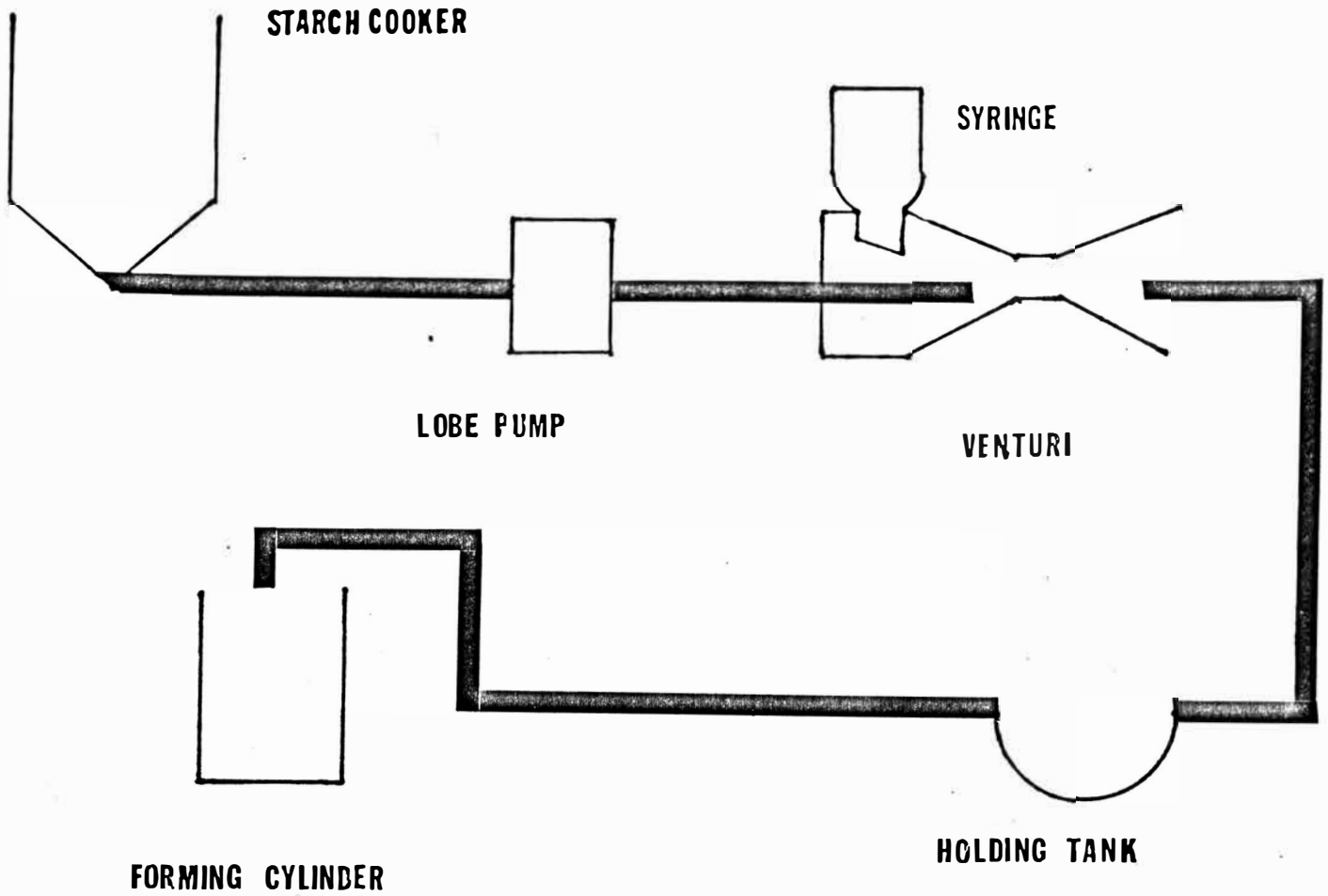
Appendix XII.....Emulsion - 100 Degrees

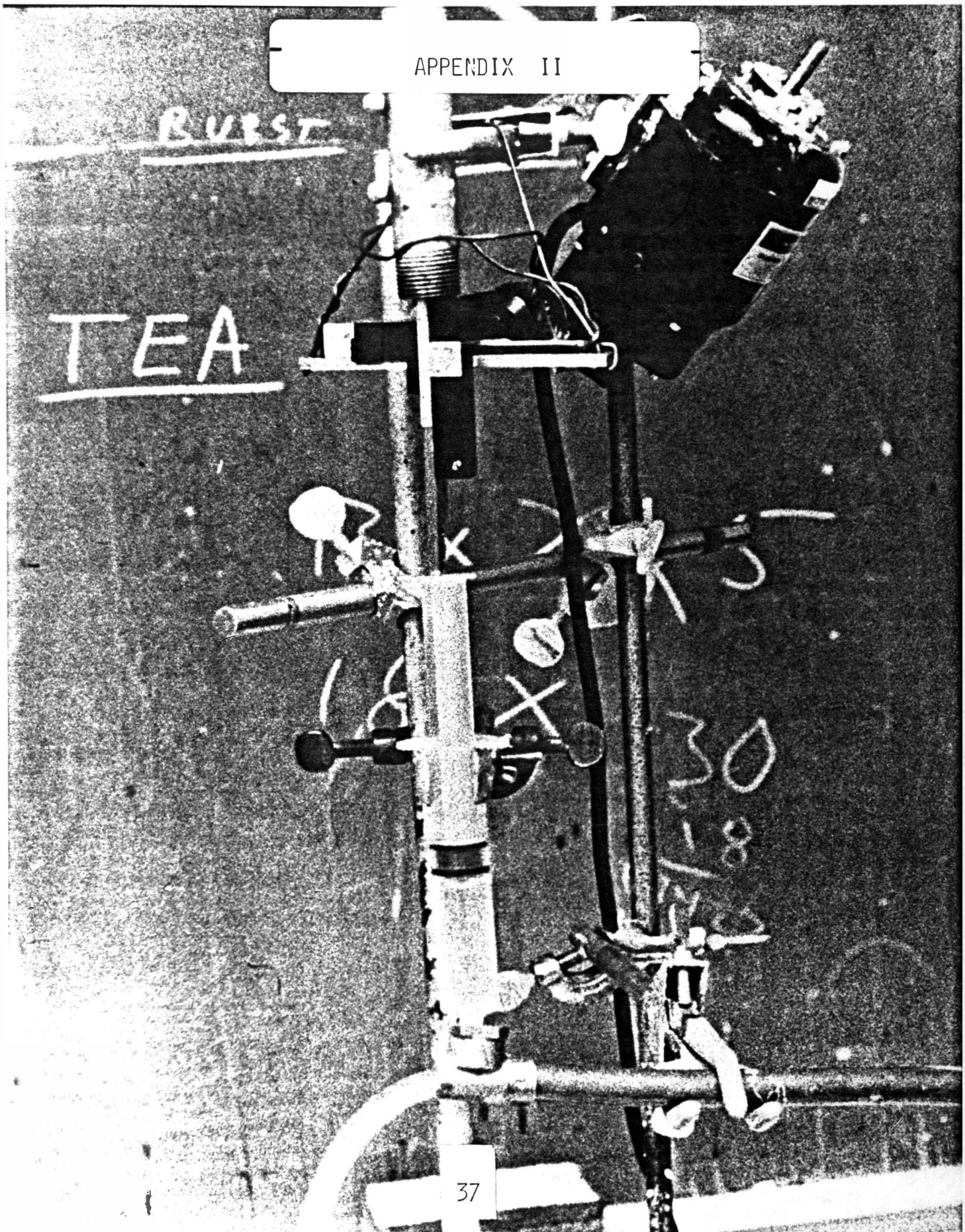
Appendix XIII.....Emulsion - 125 Degrees

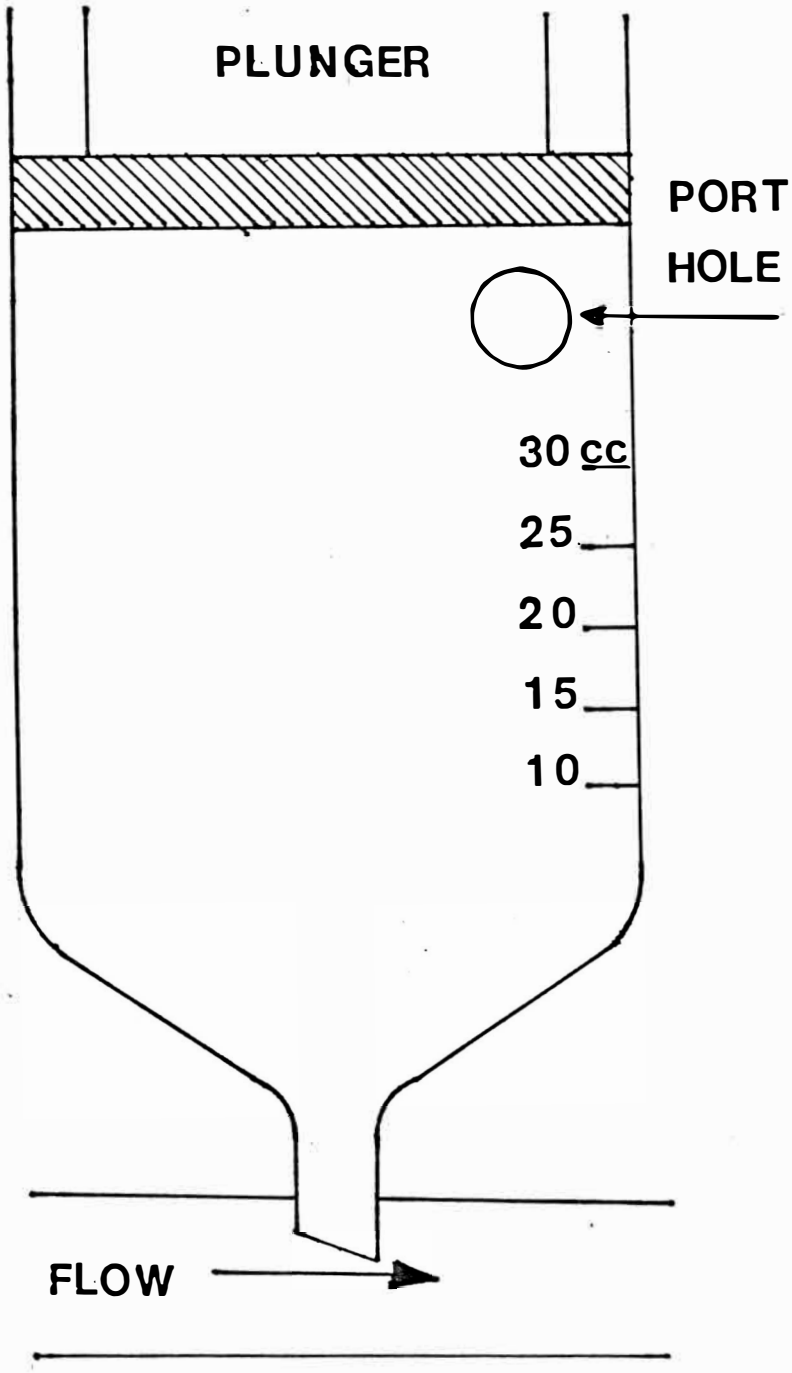
Appendix XIV.....Emulsion - 150 Degrees


STARCH COOKER
SYRINGE
LOBE PUMP
VENTURI
FORMING CYLINDER
HOLDING TANK

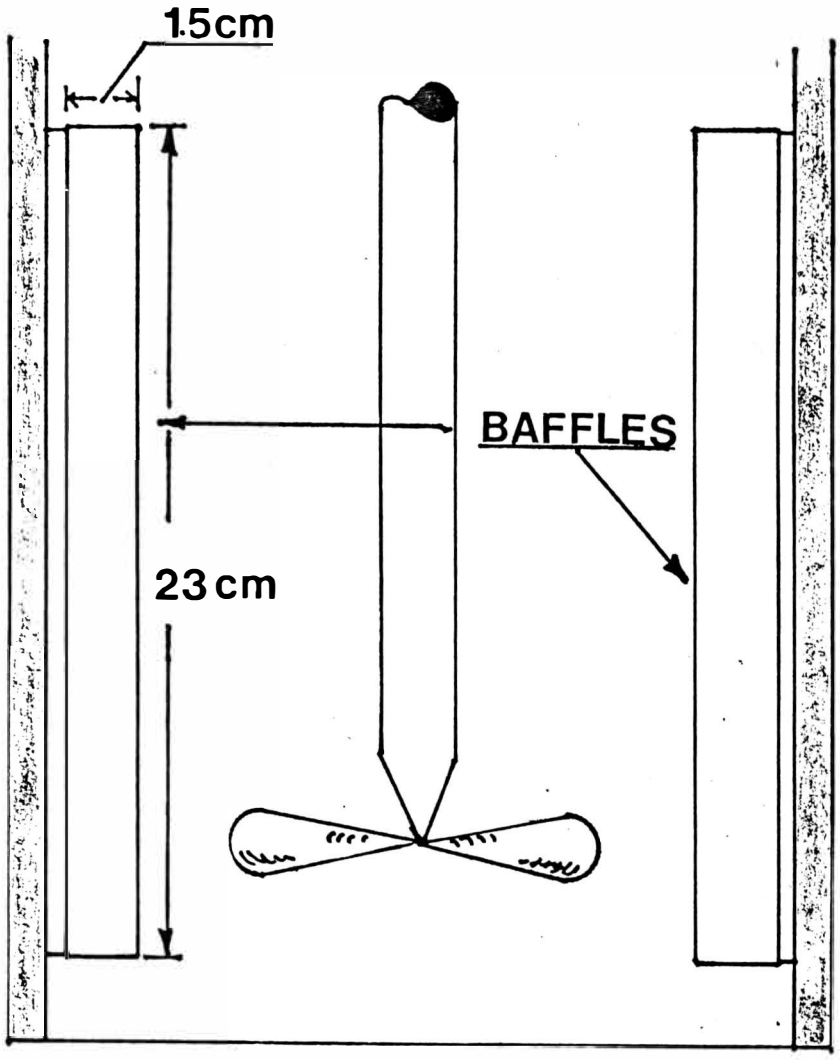
APPENDIX I

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

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

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

Appendix VI

Emulsion addition

2.26 mg FIBRAN + 26.3 mg CATO 15 = 29 mg $\approx$ .3 cc

Alum addition

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

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

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

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

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:

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

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.

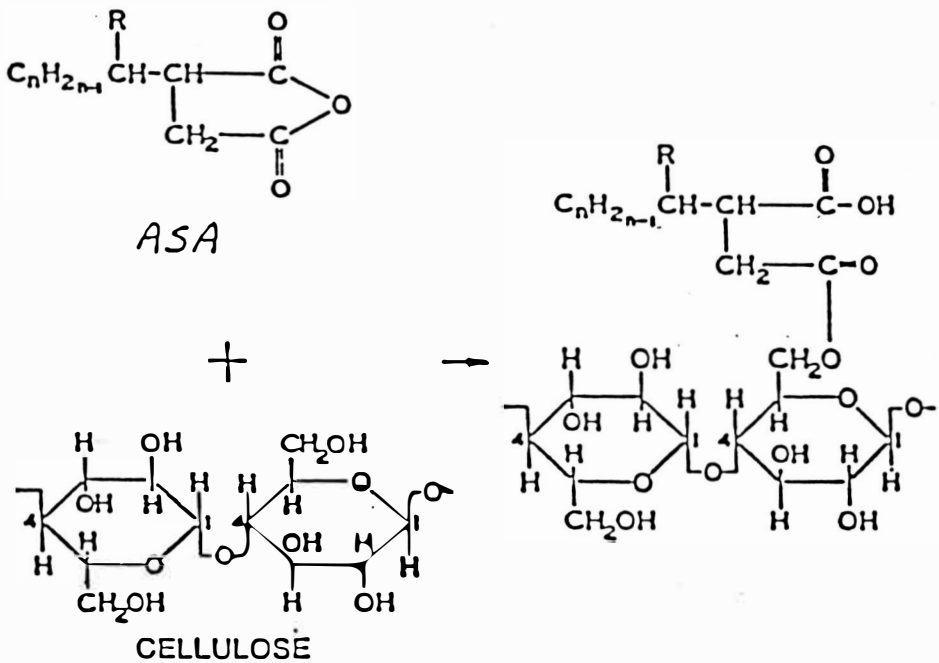
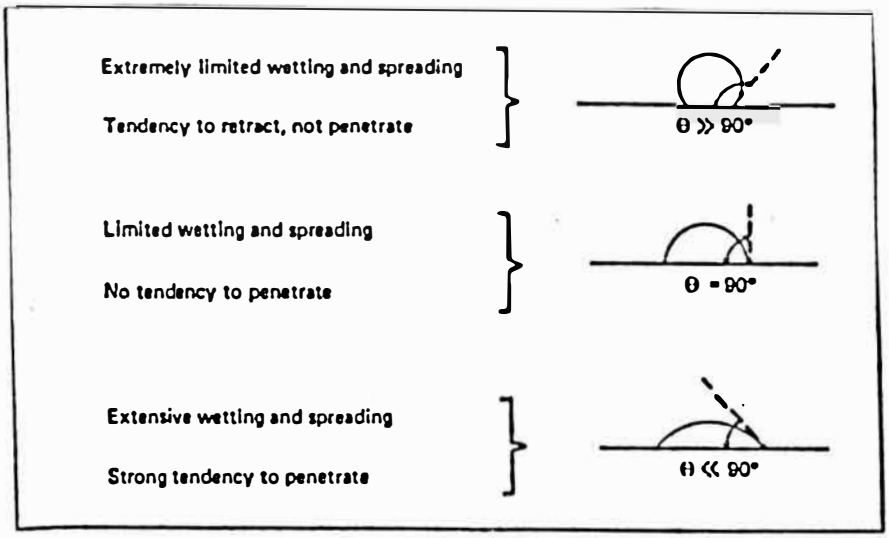
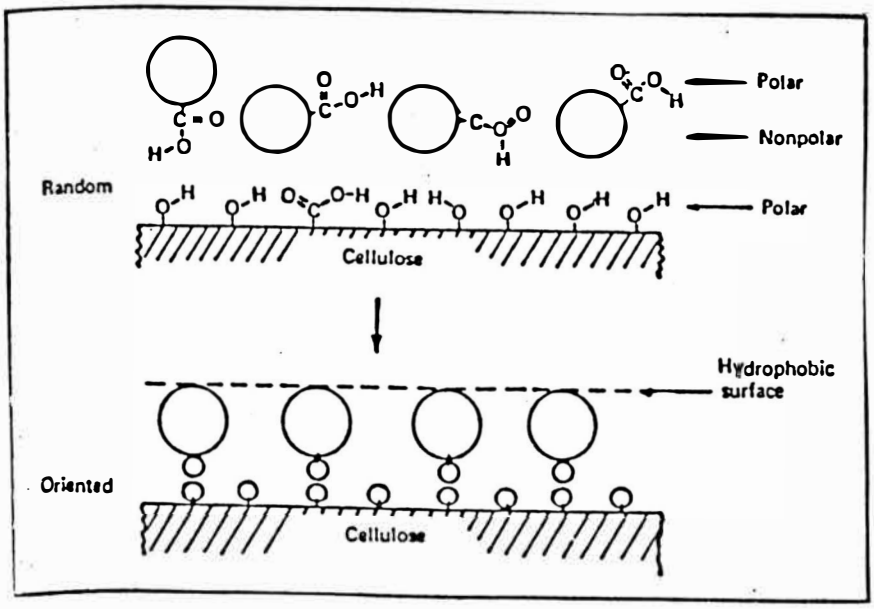
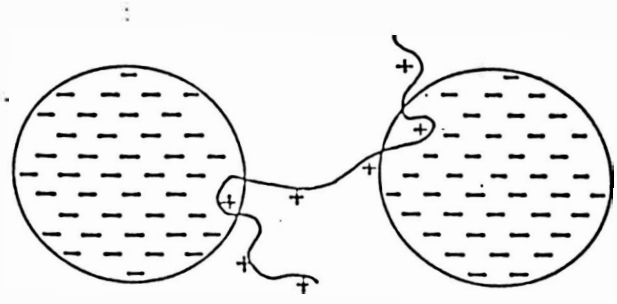


Fig 1—REACTION OF ASA WITH CELLULOSE


Extremely limited wetting and spreading
Tendency to retract, not penetrate
θ ≫ 90°
Limited wetting and spreading
No tendency to penetrate
θ = 90°
Extensive wetting and spreading
Strong tendency to penetrate
θ ≪ 90°

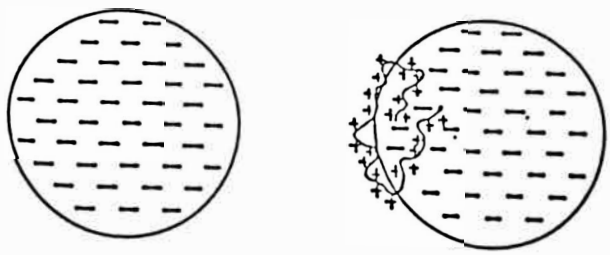

Polar
Nonpolar
Random
Polar
Cellulose
Hydrophobic surface
Oriented
Cellulose

APPENDIX X



a)

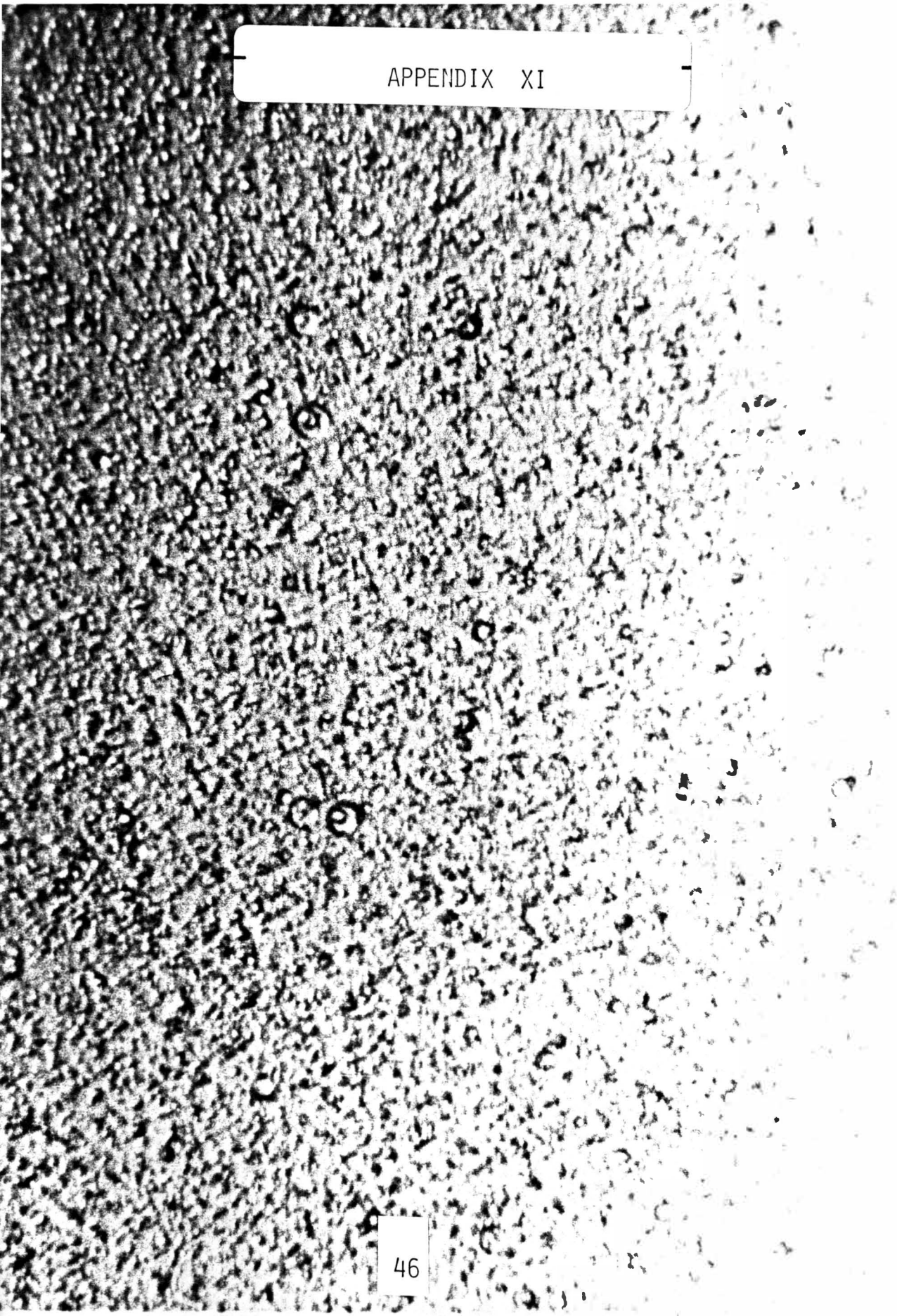
BRIDGING



b)

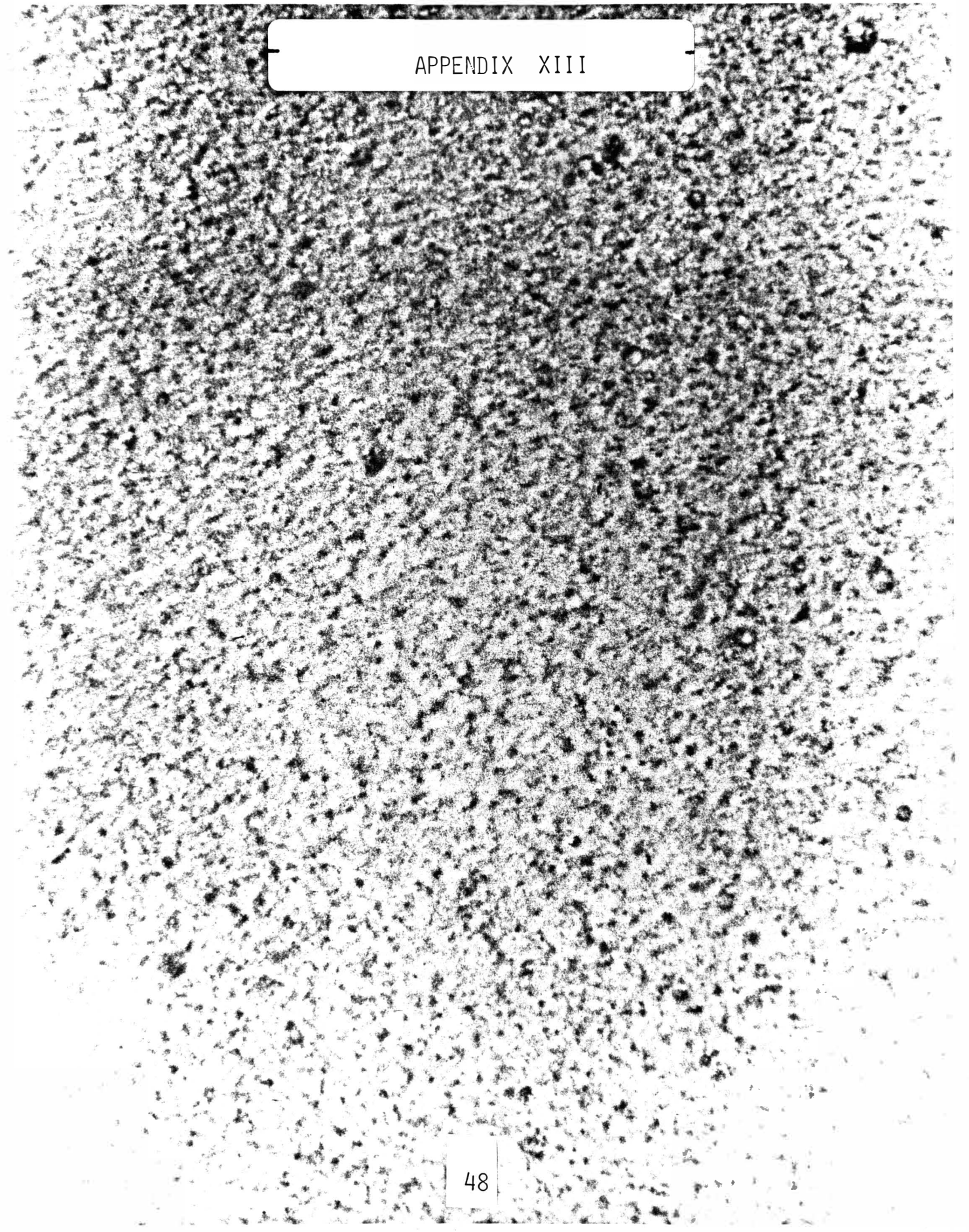
ELECTROSTATIC PATCH

APPENDIX XI



APPENDIX XII

APPENDIX XIII



APPENDIX XIV