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THE EFFECT OF DRYING CONDITIONS
ON ASA SIZED PAPER

By:

Melanie K. Blanchard

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

Western Michigan University
Kalamazoo, Michigan
April, 1984

ABSTRACT

The object of this study is to determine the roles of the drying variable and solids content of the sheet in the ASA sizing mechanism. Handsheets were produced according to the Western Michigan University Standard Handsheet Procedure for Alkenyl Succinic Anhydride (ASA) sized sheets. A control value of 346 seconds was set by drying sheets in a forced draft oven at 221°F (105°C) for 60 minutes. Additional handsheets were then dried on a hot plate at one of the following temperatures: 100-120°F, 200-210°F, 317°F or 475°F, and they experienced vastly reduced sizing levels. Sheets were also fed into the drying stage at varied solids levels. Tests indicated the dependency of the resulting sizing level on drying temperature and rate of water removal. Also indicated by the results was the necessity of cationic starch as a retention aid. The data showed conclusively that ASA sized handsheets are two-sided and that the higher degree of sizing is obtained on the wire side. Also, the level of water resistance imparted to a sheet is dependent on the solids content of the sheet going in to the drying stage. Handsheets of higher solids content result in better sized sheets.

Keywords: Internal Sizing, Alkenyl Succinic Anhydride, Drying Temperature, Web Moisture, Alkaline Papermaking.

TABLE OF CONTENTS

	Page
INTRODUCTION	1
Alkaline Papermaking	1
Advantages and Disadvantages of ASA	2
Sizing and Sizing Agent Defined	3
The Mechanism of ASA Sizing	5
Other System Components	7
Effect of Drying Conditions on Rosin/Alum Sizing	8
STATEMENT OF PROBLEM	10
EXPERIMENTAL APPROACH	11
EXPERIMENTAL	12
Materials	13
Pulp	13
Sizing Agent	13
Cationic Starch	13
Sizing Emulsion	13
Handsheet Preparation	14
Drying and Conditioning	15
Evaluation	16
PREPARATION AND DISCUSSION OF RESULTS	17
Temperature and Rate of Water Removal	17
Solids Content	17
Two-Sidedness	25
Cationic Starch Addition	25
SUMMARY OF RESULTS	28
CONCLUSIONS	29
SUGGESTIONS FOR FURTHER WORK	30
LITERATURE CITED	31
APPENDIX A	32
APPENDIX B	35
APPENDIX C	36
APPENDIX D	38
APPENDIX E	45

INTRODUCTION

Many believe that dryer temperatures and dwell time affect the efficiency of alkaline sized paper when alkenyl succinic anhydride (ASA) is used as the internal sizing agent. Therefore, the object of this study is to evaluate the role of drying variables in affecting ASA sizing performance. This is of interest due to a need for sizing agents compatible with the alkaline papermaking system which is presently increasing in popularity. Alkenyl succinic anhydride sizing is being used in a limited number of mills which are working with alkaline systems (Appleton Paper's Roaring Spring Mill is one). A few other mills have run trials with ASA but discontinued its use, finding it difficult to control. These mills reverted back to their previous fatty-acid resin, wax or alkyl ketene dimer sizing methods. It is possible that mills have even reverted back to an acid system so as to use a rosin and alum size.

ALKALINE PAPERMAKING

In order to understand the demand for an effective alkaline sizing agent, one must know the advantages of alkaline papermaking and hence, the reasons for conversion. They are as follows (1,2,3,):

- eliminates acid corrosion
- increases color and strength performance
- increases dry strength
- produces paper which looks, feels and performs better than its acid counterpart
- allows for increased use of secondary fiber
- allows for increased use of hardwood fiber
- allows for the use of calcium carbonate as a filler and reduced titanium dioxide requirements
- reduces cost due to greater filler loading
- aids in white water close up
- improves papermachine cleanliness
- reduces energy usage
- increases pulp mill yield

The significance of the above benefits depends upon geographical location, grade structure, and prevailing market conditions. However, in order to enjoy the benefits of an alkaline system, the following disadvantages must be overcome (4):

- converting to alkaline system must be carried out throughout the entire mill; all machines, all water collection systems, all instrumentation and control loops, etc.
- sticking to press rolls is increased resulting in more wet web breaks
- retraining of entire staff
- crumbing on press rolls
- build up on the foils and suction box covers
- build up in the headbox
- varying retention
- sizing emulsion instability
- low to zero sizing levels

Many of the above disadvantages can be eliminated when familiarity with new equipment and with new instrumentation is attained. One must learn to live with the other disadvantages. It can be seen, however, that when the disadvantages of alkaline systems are overcome the advantages add up to increased savings when compared to an acid system.

ADVANTAGES AND DISADVANTAGES OF ASA

To improve the low sizing levels obtained with an alkaline system chemicals compatible with the high pH and which added water repellency to cellulosic fibers were studied. One of the chemicals was ASA and following are the reasons why it has potential as an alkaline sizing agent (5):

- includes benefits obtained with alkaline sizing
- forms a chemical bond with cellulose
- allows for the use of most normal fillers
- improves machine runnability by keeping parts cleaner
- improves product first quality
- gives a higher first pass retention
- improves drying efficiency
- allows for better coater runnability
- gives pH stability
- allows for operation at higher temperature (150°-250°C) (6)

The question which must now be answered is this: if ASA imparts all of these advantages to the papermaking process, why is it not enjoying more widespread use? The answer includes the disadvantages inherent in an alkaline system as well as lack of control of the sizing level obtained.

In order to further understand ASA sizing, the terms sizing and sizing agent must be defined.

SIZING AND SIZING AGENT DEFINED

Sizing is the process by which a chemical additive provides paper and paperboard with the resistance to liquid wetting, penetration and absorption (7). The chemical which reduces this interfiber and intrafiber penetration is the sizing agent. Aqueous liquid penetration of the surface of paper is accomplished by three mechanisms. First, there is lateral movement which is accelerated and enhanced by capillary action induced by fibers lying close together, oriented parallel to each other. Second, there is transverse movement through the paper in which the liquid is drawn into the sheet by the capillary action of spaces or pores between the fibers. Third, the liquid can travel through the structure along paths through the cellulosic material itself.

The rate of flow of a liquid through a capillary tube is represented by the Washburn equation (8).

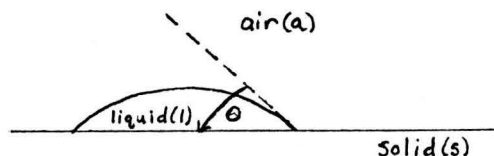
$$\text{Time rate of penetration} = \frac{dl}{dt} = \frac{v}{n} \cdot \frac{r}{l} \cdot \cos \theta$$

where v is the surface tension of the penetrating liquid, n is the viscosity of the penetrating liquid, r is the radius of the capillary, l is the length of the capillary filled with the penetrating liquid and θ is the contact

angle between the liquid and the capillary wall. Surface tension is defined as the force per unit length on the surface that opposes the expansion of the surface area.

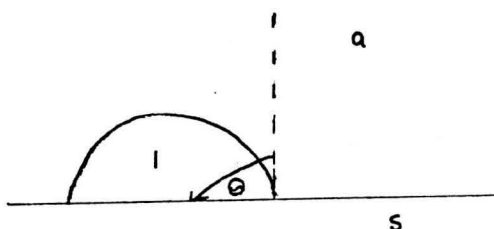
The governing factor for the extent of liquid penetration and spreading is the contact angle formed between the liquid and the paper (cellulose fiber) surfaces. See the following figure.

I



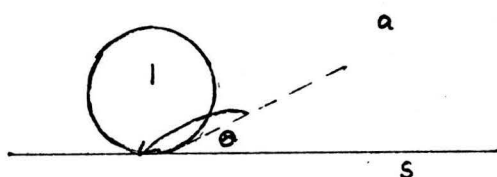
$$\theta \ll 90^\circ$$

II



$$\theta = 90^\circ$$

III



$$\theta \gg 90^\circ$$

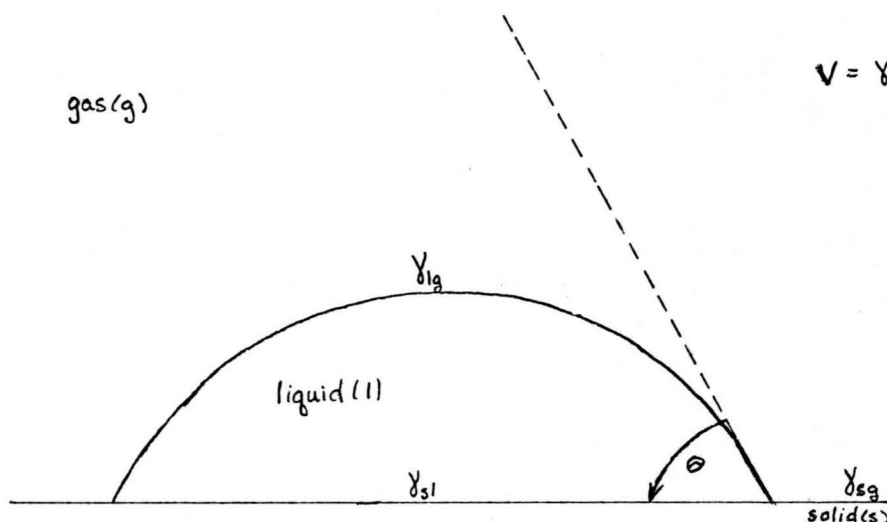
Contact Angle

In case I, there is extensive wetting and spreading and a strong tendency to penetrate the surface. In case II, there is limited wetting and spreading with no tendency to penetrate the surface. Finally, in case III, the most desirable condition for good sizing, there is very little wetting or spreading

and the tendency is to retract, not to penetrate. Another equation used in the determination of the contact angle between two surfaces is:

$$\cos \theta = \frac{V_{sg} - V_{sl}}{V_{lg}}$$

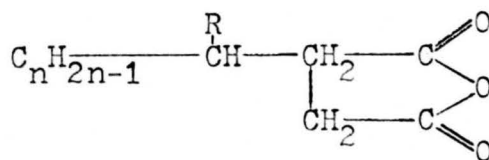
The variables are defined in the following diagram:



The purpose of a sizing agent is to lend the fiber surfaces a coating of relatively low surface energy so that high energy (aqueous) liquids will form a large contact angle when in conjunction and will not spread or wet the surfaces. Penetration is also slowed down due to the limited spreading and wetting.

THE MECHANISM OF ASA SIZING

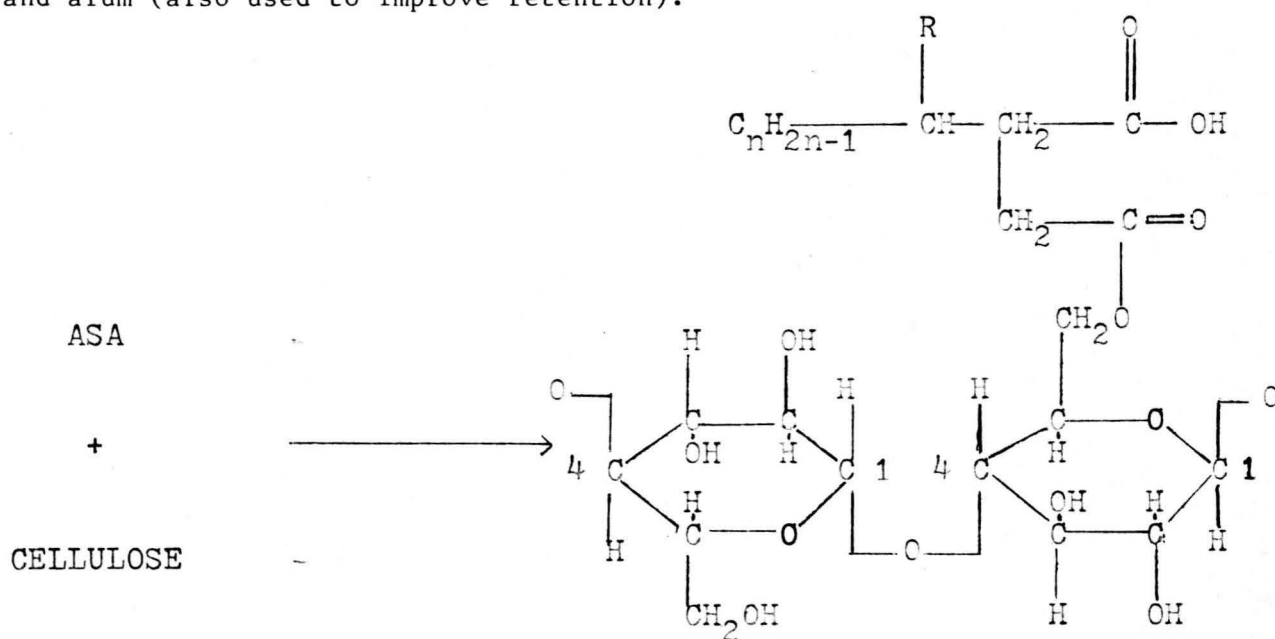
ASA is an alkyl succinic anhydride molecule with an unsaturated side group. This unsaturated group is hydrophobic which makes the molecule a semi-oil base resin.



Alkenyl Succinic Anhydride

The alkenyl group generally has a molecular weight range of 300 - 1000 grams per mole (9). The R group can be replaced by other groups such as potassium ion or other metal ions in order to impart desired characteristics to the size. When in a fiber slurry, ASA and the hydroxyl groups on cellulose and hemicelluloses rapidly undergo an esterification reaction producing a stable chemical linkage. The molecules are oriented so that the hydrophobic side chain is away from the fiber. (See figure.) This enables it to impart water resistance to the fiber. The retained particulates then melt during the drying process causing extensive spreading of the sizing agent over the fiber surfaces creating a monomolecular layer. A strongly bonded hydrophobic coating is the result.

Major disadvantages of the ASA are its rapid hydrolyzation and its instability to shear once it is attached to the fiber's surfaces. Hydrolyzation is not a problem if the sizing emulsion is used immediately after preparation. To control retention problems due to shear, the sizing agent must be added to low shear zones as well as be accompanied by a retention aid and alum (also used to improve retention).



OTHER SYSTEM COMPONENTS

Retention Aid This compound is a necessary part of the system because it increases the retention of ASA. Tests carried out by Meyer and Associates (11) produced data which showed systems using cationic starches (196 seconds) as having a greater sizing efficiency over systems employing amphoteric starches (170 seconds). This is typical of that found in the literature.

The mechanism of cationic retention involves the transfer of a positive charge to the sizing agent to improve adherence to the anionic fiber surface. Other reasons for the use of a cationic retention aid include its low cost and its benefit to the dry strength of the paper.

Alum Meyer (12) performed tests to see if any other trivalent ion would have the same effect on the ASA sizing system as aluminum. Results were negative, thus mills are still forced to have aluminum in their systems. According to further tests performed without this hydrated aluminum sulphate salt, acceptable sizing levels cannot be developed with ASA sizing material.

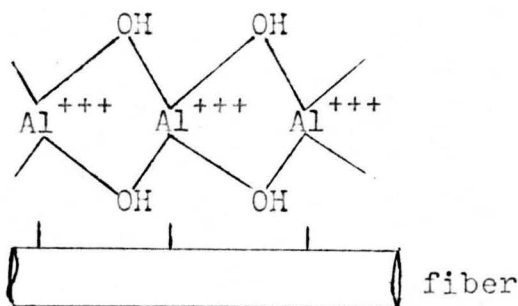
<u>Alum Addition</u> <u>(lbs./ton fiber)</u>	<u>IP Sizing*</u> <u>(Seconds)</u>
0	1
2	10
4	21
8	90

* IP Sizing uses the Hercules Size Tester (HST) at an end point reflectance of 60%. Test Ink #2 is used.

This indicates that alum aids in retaining ASA on the fibers.

The proposed mechanism is as follows (13): aluminum sulfate hydrolyzes in aqueous solution. Under alkaline conditions, the aluminum ion is present

as the polynuclear complex $[Al(OH)_3]_n OH^-$. ASA then reacts with the exposed hydroxyl groups.



Aluminum Complex With Fiber

EFFECT OF DRYING CONDITIONS ON ROSIN/ALUM SIZING

There are many theories which propose "ideal" drying conditions for paper sized with rosin (15). However, most agree that two stages are necessary and that the temperature in the first stage be between $60^{\circ}C$ and $90^{\circ}C$. Also, it is agreed that the moisture content of the paper during the various drying stages is critical.

In order to determine the best set of drying conditions for rosin sized paper, Ivanov and Markhonin carried out several experiments (16). In a test of single stage drying at a rate of addition of rosin of 0.75% by weight of fiber, the degree of sizing achieved decreased noticeably at drying temperatures above $90^{\circ}C$.

"A lowering of the degree of sizing at the higher temperature of single stage drying, apparently takes place because of the detrimental effect of the high temperature of cylinders in the early stages of drying, when the water contained in the web causes a migration of the particles of size together with the liquid and vapour; this apparently leads to a coarsening of the particles, and their non-uniform distribution in the paper.

In addition, the increased temperatures at the beginning of the drying, increases the porosity of the paper through the action of water vapour, which disrupts the structure of the web, and therefore lowers the degree of sizing. These experiments demonstrate in a convincing fashion the need for adopting, at the least, two drying stages for sized papers, so as to remove the excess water from the web gently, and at a low temperature." (17)

With the above conclusions, two stage drying tests were carried out. Paper was pressed to an initial dry content of 33-35% and tests were run with a first stage temperature of 70°C and a second stage temperature of 120°C . The dry contents were varied between 50% and 80%. The experiment revealed that the maximum degree of sizing was achieved at a dry content of 65% between the stages. A second set of experiments designed to determine the optimum temperature for the first stage of drying varied this temperature between 55°C and 90°C , with a 65% dry content between stages and a second stage temperature of 102°C - 105°C . The maximum degree of sizing was observed when the temperature of the first stage drying cylinders was 60°C - 65°C and an intermediate percent solids of 65. Varying second stage temperatures from 80°C - 140°C showed that a high temperature in the second stage of drying aids sizing. The optimum temperature was 110°C - 120°C with no detrimental effect if a further increase in drying temperature to 140°C occurred. It was also found that drying the paper to a final solids content of 96% to 98% did not reduce the sizing level (18). Experiments using three stages of drying did not show any significant change in sizing efficiency (19).

STATEMENT OF PROBLEM

As was stated earlier, the sizing of paper with ASA is difficult to control. Constant levels of sizing elude the papermaker. It is desirable to learn the contributions of each papermaking variable on the degree of sizing obtained when ASA is used in order to achieve constant levels of water repellency at the desired conditions. The study based on this literature review will look at drying variables. The first step will be to set up a control in which a constant degree of sizing is obtained according to the Hercules Size Test (HST). A second goal will be to decide if paper sized with ASA has two-sidedness. An attempt will be made to determine the effect on sizing of varying the solids content of the sheet before drying and of altering the drying process and temperature. The results of the experiments will hopefully aid in the understanding of the ASA sizing system.

EXPERIMENTAL APPROACH

The objective of this research project will be to determine the roles of the drying variable and solids content of the sheet in the ASA sizing mechanism. It will be accomplished by sized handsheet production, drying and evaluation.

Since the literature revealed the vast number of variables associated with the ASA system it has been decided to carry out the study using handsheets rather than making a paper machine run. This will reduce the number of uncontrolled variables and allow for the variance of the drying variables only.

Handsheets will be produced according to the standard Western Michigan University Handsheet Procedure for ASA Sized Sheets.(20) They will be dried on hot plates at various temperatures until bone dry. The time needed to reach a zero moisture content will be measured. The rate of drying will be calculated and should vary from run to run since the solids content of the sheets will vary after pressing. The papers will be conditioned to TAPPI Standard T-420 and evaluated using the Hercules Size Test. Figures will be drawn in an attempt to find correlations between the amount of sizing obtained and the drying condition.

The study should show whether in fact the drying stage is a variable in the ASA sizing of handsheets. The project is also expected to show the variation in sizing due to changes in the drying temperature and the solids content of the sheet going into drying. Also, the results should indicate the tendency of the paper to be two-sided with respect to sizing.

EXPERIMENTAL

In order to make the results from this study meaningful, it was necessary to control all variables of the system except those related to the drying of the sheet. Early work involved studying the size emulsion. This distribution of the ASA in the cationic starch was examined using a high powered microscope and the stability of the emulsion was evaluated using a Hercules High Shear Viscometer.

After the emulsion stability was established, it was necessary to establish a sizing control value to which all other levels of sizing could be compared. This was obtained by producing all handsheets by the standard procedure and drying them in a forced draft oven at 221°F (105°C) for 60 minutes. Several trials were required at this point due to water problems. Handsheets were produced and dried only to emerge from the oven yellow. It was assumed that this was due to the iron present in the water. A test was performed by passing this water through a filter paper and measuring the brightness loss. Once this filter had been dried its brightness was three points lower than its original value. Flushing the lines and allowing the water to stand at least 24 hours was ineffective in reducing the amount of iron in the water. Therefore, two ion exchange columns and one softener were installed in the water line. This resulted in a decrease in iron content of the water and results from the filter paper test showed no adverse effect in the brightness of the filter. This deionized water was used for all further trials.

With the control value established other handsheets were produced, pressed in the British Sheet Mold Press and dried on hot plates. The time to dry was measured and the rate of water removal was calculated. All handsheets

were then conditioned according to TAPPI Standard T-420 and tested using the HST.

MATERIALS

For exact figures, see Appendix B.

Pulp The stock used in handsheet production was an equal blend of bleached kraft hardwood and bleached kraft softwood. Dry lap was desintegrated in a Morden Slush Maker and refined in a Valley Beater to a Canadian Standard Freeness of 350 to 400 ml at a consistency of 1.57% according to TAPPI Standard T200 os-70. When not in use, the stock was stored at 40°F and not allowed to age past two weeks. There was no biocide added to the pulp slurry.

Sizing Agent The ASA used in all trials required emulsification with a non-ionic surfactant. Ninety-three percent ASA and 7% IGEPAL 606 (surfactant) were mixed on a lab shaker for 20 minutes. A rest period of 10 minutes was sufficient to remove air bubbles from the emulsion. Everytime the cap was taken off the jar of the ASA/surfactant emulsion, the jar was purged with nitrogen gas before replacement. This allowed the emulsion to be kept and used for seven days.

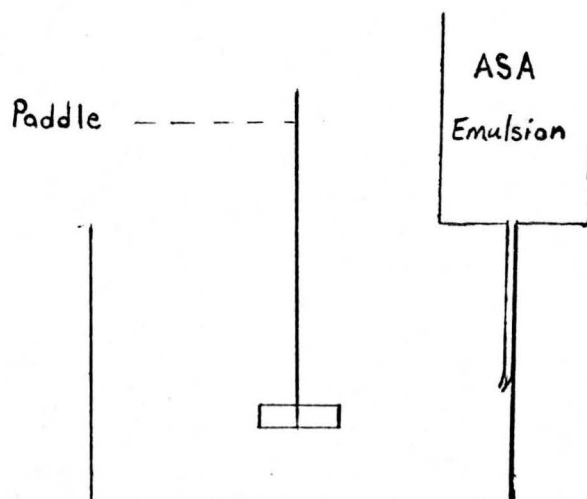
Cationic Starch Cool, deionized water was the dispersing medium for 6.01 grams of cationic starch. This was then heated to 190°F and held there in a double boiler for 30 minutes. This master batch of stock was allowed to cool to 90 to 100°F before being diluted with hot, deionized water to the original 0.8% solids level. The life time of this mixture was two hours when kept between 90 and 100°F with mild mixing. It was later learned that the starch mixture needed to be rediluted to the original solids level before being used the second time.

Sizing Emulsion The ASA/surfactant emulsion was then added to the

cationic starch over a period of 45 seconds in an emulsification tower stirred with a three pronged propeller. At the end of the two minutes the pH was adjusted to 7.6. The lifetime of the resultant sizing emulsion was considered to be 30 minutes.

HANDSHEET PREPARATION

In beginning handsheet production, stock to make ten handsheets of 1.2 grams O.D. weight each was diluted to 0.5% consistency. With mixing, 0.7% alum based on dry fiber was added to the stock and mixed further for ten minutes. A solution made up of deionized water and the remaining alum (0.3% based on dry fiber) was then prepared for final dilution of the stock. In order to control the shearing action of the mixing, the addition of the sizing emulsion to the above mentioned stock was carried out in the following manner. With the constant mixing of a paddle at a speed of two to three, the emulsion was added dropwise into the stock tank in a controlled way. A representation of the addition equipment is given below. The sized stock was poured through a funnel into the tank which held the dilution water reducing the exposure of the stock/ASA to shear. A constant state of stock motion was obtained by slow mixing with a paddle mixer.



After recirculating one liter of stock through the funnel and into the holding tank, two liters of the 0.06% consistency stock was poured through a funnel into the British Sheet Mold. After mixing with the standard British Mold Mixer the mold was drained and the sheet was couched off onto a TAPPI Standard blotter and finally onto a metal plate. The first sheet thus produced was used to check compliance with the desired 1.2 grams weight. Necessary adjustments were made and six more handsheets were produced. They were stacked plate down in the sheet press with two TAPPI Standard blotters between each sheet. The pressure was raised to 50 psi over a period of one minute and held constant for five minutes. After releasing the pressure, the sheets were restacked, plate up, with one blotter between each sheet and two on the top and the bottom of the stack. The original bottom sheet was again on the bottom of the pile. A period of one minute was again taken to obtain a pressure of 50 psi before being held constant for $1\frac{1}{2}$ minutes. Air tight plastic bags were then used to store the handsheets in until the time to dry (usually less than three hours).

Tests were performed to determine the solids contents of the sheets after pressing. Differences in weights before and after drying showed that the solids content of a handsheet depended on its position in the stack of six while being pressed. The solids contents for sheets one through six were found to be 69.6%, 65.3%, 62.6%, 60.5%, 59.9%, and 56.3% on the average.

DRYING AND CONDITIONING

For the establishment of the control value, the handsheets were dried in contact with the metal plates in a force draft oven at 221°F (105°C) for 60 minutes. Sheets from all other runs were removed from the metal plates immediately before drying. In the removal process the handsheets tended to

stick to the plates and consistently left a fiber residue on the plate. Cleaning the plates with different detergents was to no avail therefore, since the sticking seemed to be uniform for all sheets, it was ignored. (The literature search revealed that sticking to press rolls was a disadvantage of an alkaline sizing system).

Handsheets were then dried to a zero moisture content in one of four ways. Four drying temperatures and two different hot plates were used for this part of the procedure. A pyrometer was used to measure the temperature for each run. The two lower ranges (100-120°F and 200-210°F) were obtained with a 120 volt hotplate set on low and plugged into a 120 volt Variac. The dial on the Variac was set at 27 and 43 for the respective ranges. The two higher temperatures (317°F and 475°F) were obtained using a 440 volt hotplate set on low and medium respectively. The time to dry was measured for each sheet using a stop clock.

A final set of handsheets was allowed to air dry at 70°F in contact with the metal plates.

After reaching zero percent moisture the handsheets were conditioned for at least 24 hours at 73°F and 50% relative humidity.

EVALUATION

Each handsheet was weighed, cut in quarters and tested on the HST with the blotter side of the sheet up. The testing instrument was set and calibrated to an 80% reflectance level and Hercules 1% acidity ink was used.

The final step in the study was to evaluate the moisture content of the handsheets after conditioning so that the dry weights of the sheets could be calculated. It was found to be 5.18% using ASA-sized sheets. The data generated from the test enabled the calculation of the rates of water removal in pounds per hour per square foot for each handsheet.

PRESENTATION AND DISCUSSION OF RESULTS

The results from all tests were gathered, analyzed statistically and graphed to yield that which follows.

TEMPERATURE AND RATE OF WATER REMOVAL

Figure 1 shows that for each solids content going into the drying stage, the lower the temperature, the longer the period of time needed to reach 100% solids. Figures 2 and 3 show a decline in the sizing level of the sheets as the temperature in the drying stage was increased. However, there was a sudden high increase at 475°F. In Figure 3, the values for the vertical axis were calculated by dividing the level of sizing in seconds from the individual runs by the level of sizing in seconds from the control run. These figures suggest that there is a decrease in ASA sizing with an increase in temperature or rate of removal of water as shown in Figure 4 until a high temperature is attained. When a curve shows a minimum like this it often indicates competing reactions. The theory developed at this point is that the competing "reactions" are actually the competing mechanisms of sizing developed due to temperature and sizing developed due to rate of water removal. It is proposed that the amount of sizing obtained is dependent on the rate of water removal at low temperatures but not at high temperatures.

SOLIDS CONTENT

Figure 5 demonstrates the variability of sizing due to the solids content of the sheet before drying. Figure 6 shows much more clearly the association between solids and the level of sizing obtained. It is apparent that the sizing achieved is dependent on the percent solids of the sheet with values increasing as the solids content of the sheet increase.

TIME AT TEMPERATURE

Figure 1

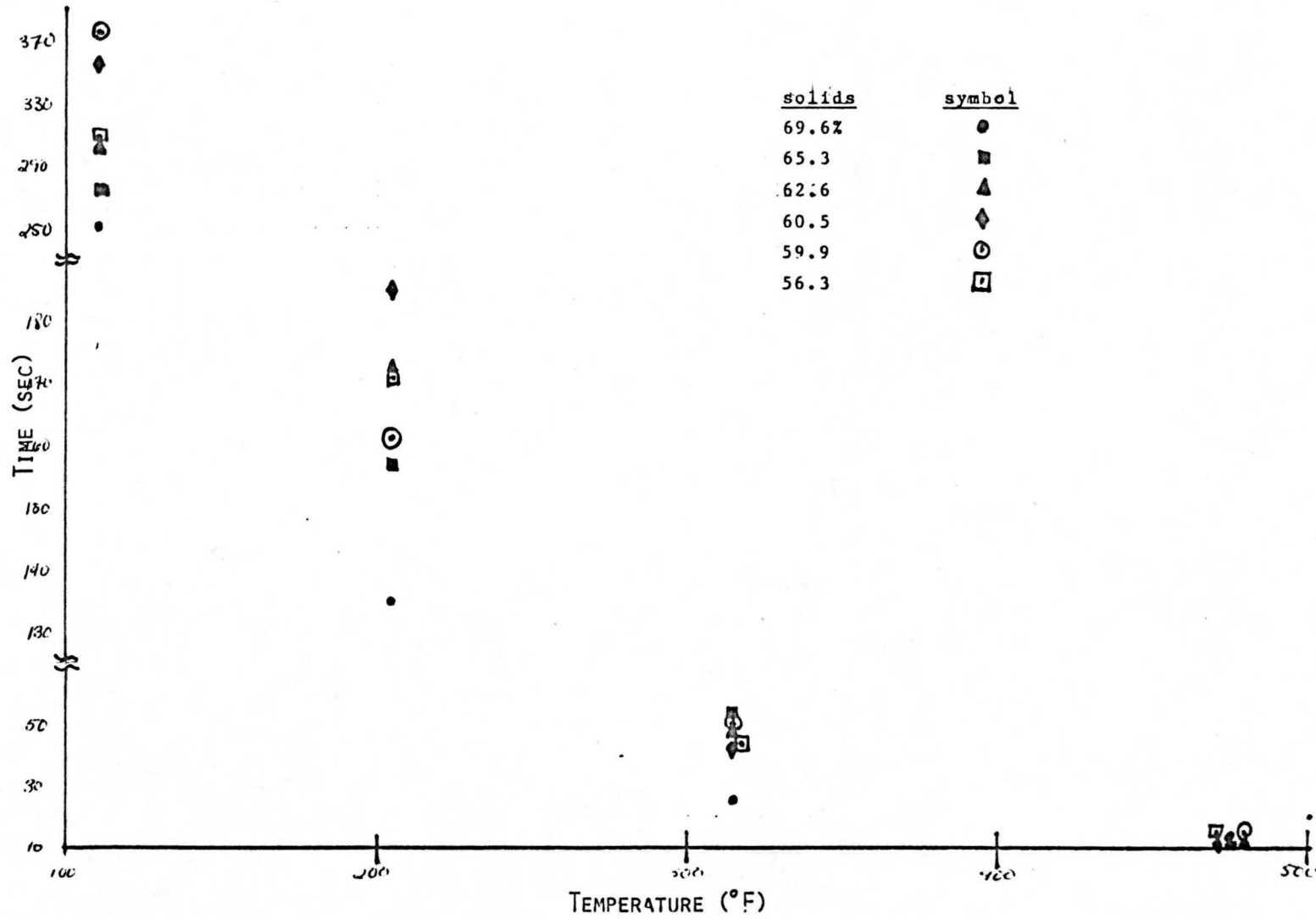


Figure 2

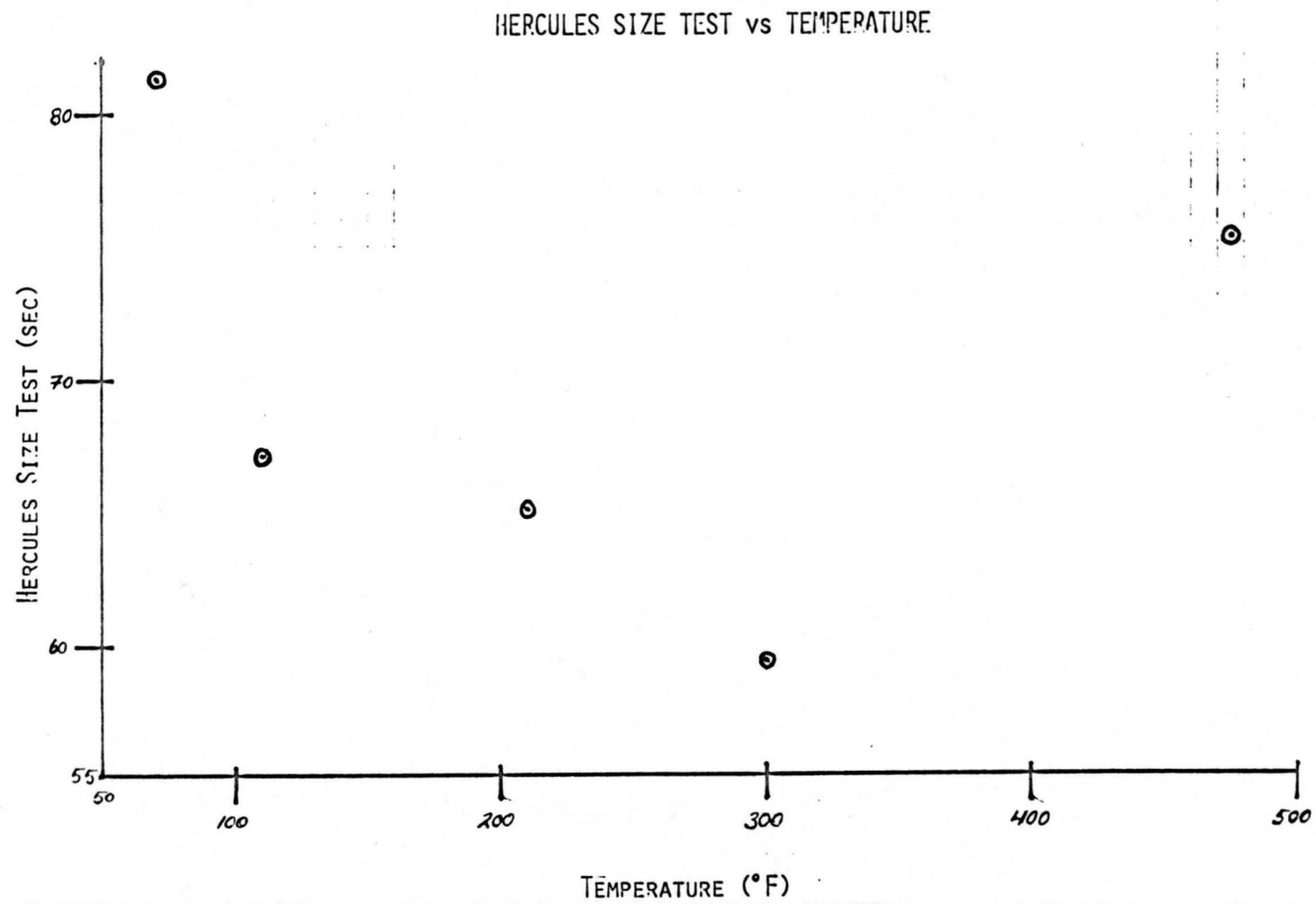


Figure 3

NORMALIZED HERCULES SIZE TEST vs TEMPERATURE

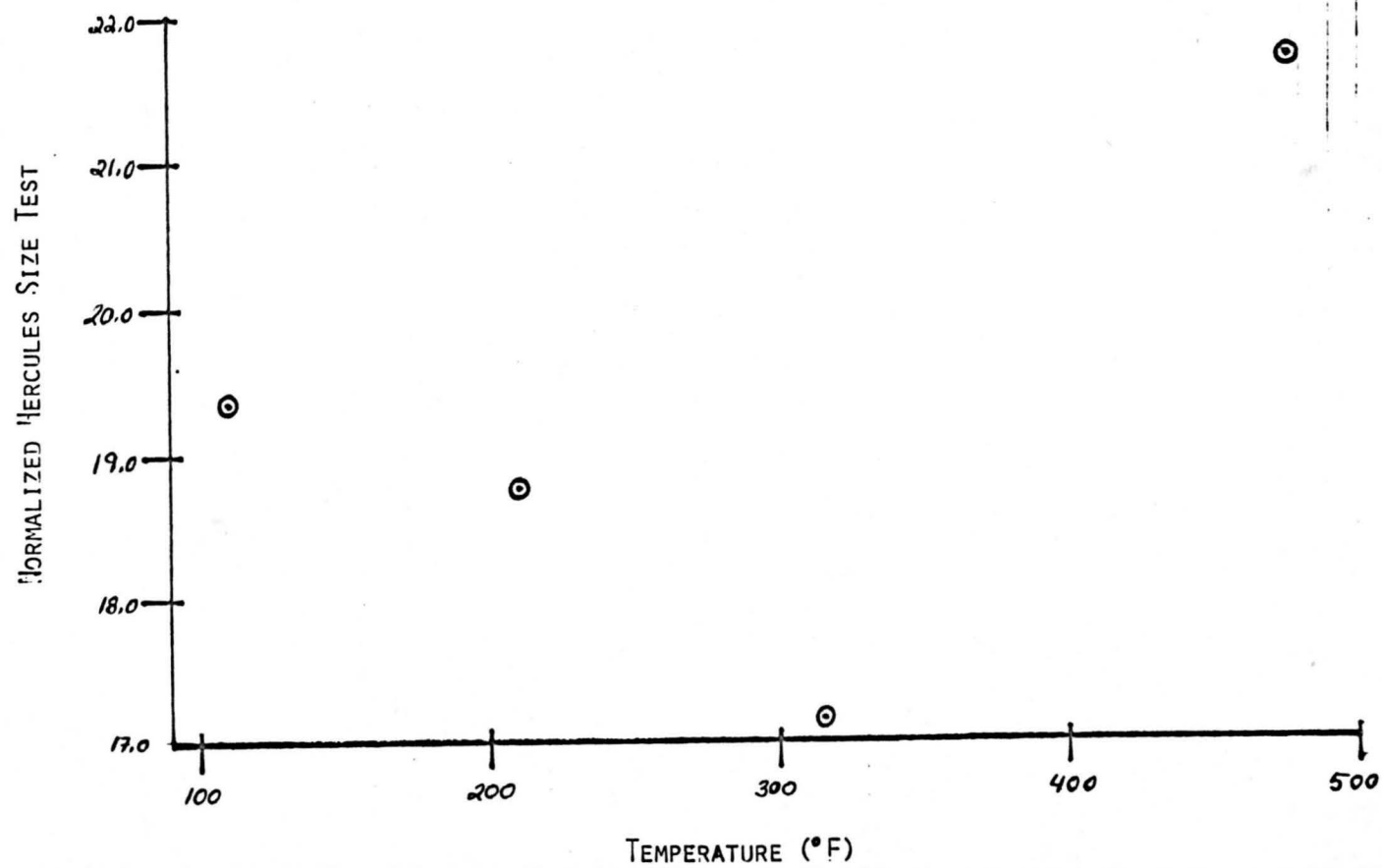


Table I

HST vs TEMPERATURE

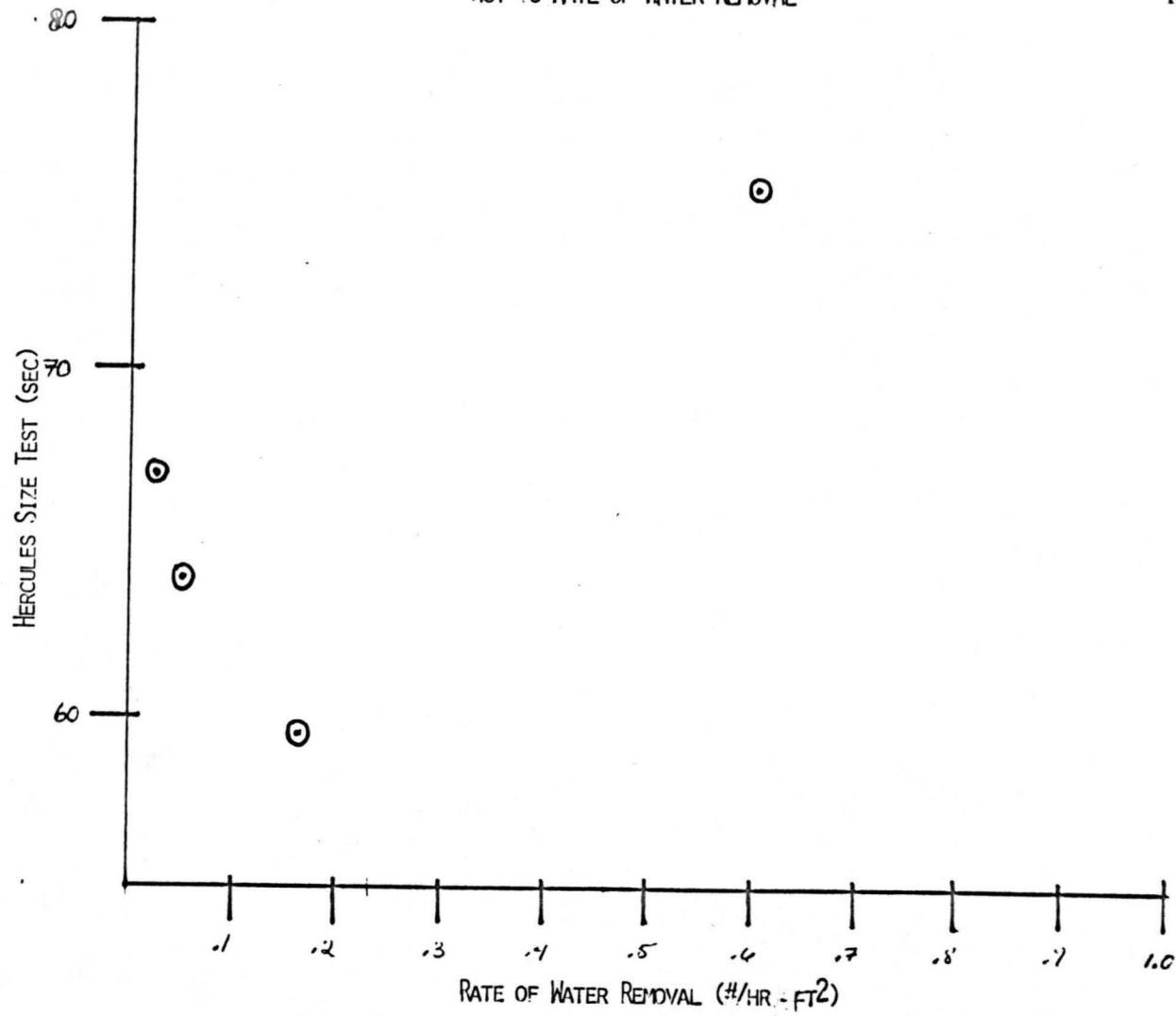
	<u>T</u>	<u>HST</u>	<u>% OF CONTROL</u>
Control	221°F	346.6s	100
	100-120	67.1	19.36
	200-210	65.1	18.78
	317	59.4	17.14
	475	75.2	21.70
Air Dry	70	81.3	23.46

HST vs PERCENT SOLIDS

<u>SHEET</u>	<u>% SOLIDS</u>	<u>CONTROL</u>	<u>100-120°F</u>	<u>200-210°F</u>	<u>317°F</u>	<u>475°F</u>	<u>Mean</u>
1	69.6	347.3s	60.7s	73.1s	68.1s	75.8s	125.0s
2	65.3	333.8	61.5	62.4	65.6	96.3	123.9
3	62.6	352.6	57.0	61.8	57.0	87.4	123.2
4	60.5	335.0	69.1	63.0	55.4	59.1	116.3
5	59.9	369.3	84.9	66.5	52.7	70.1	128.7
6	56.3	347.8	69.1	63.6	57.7	62.6	120.2

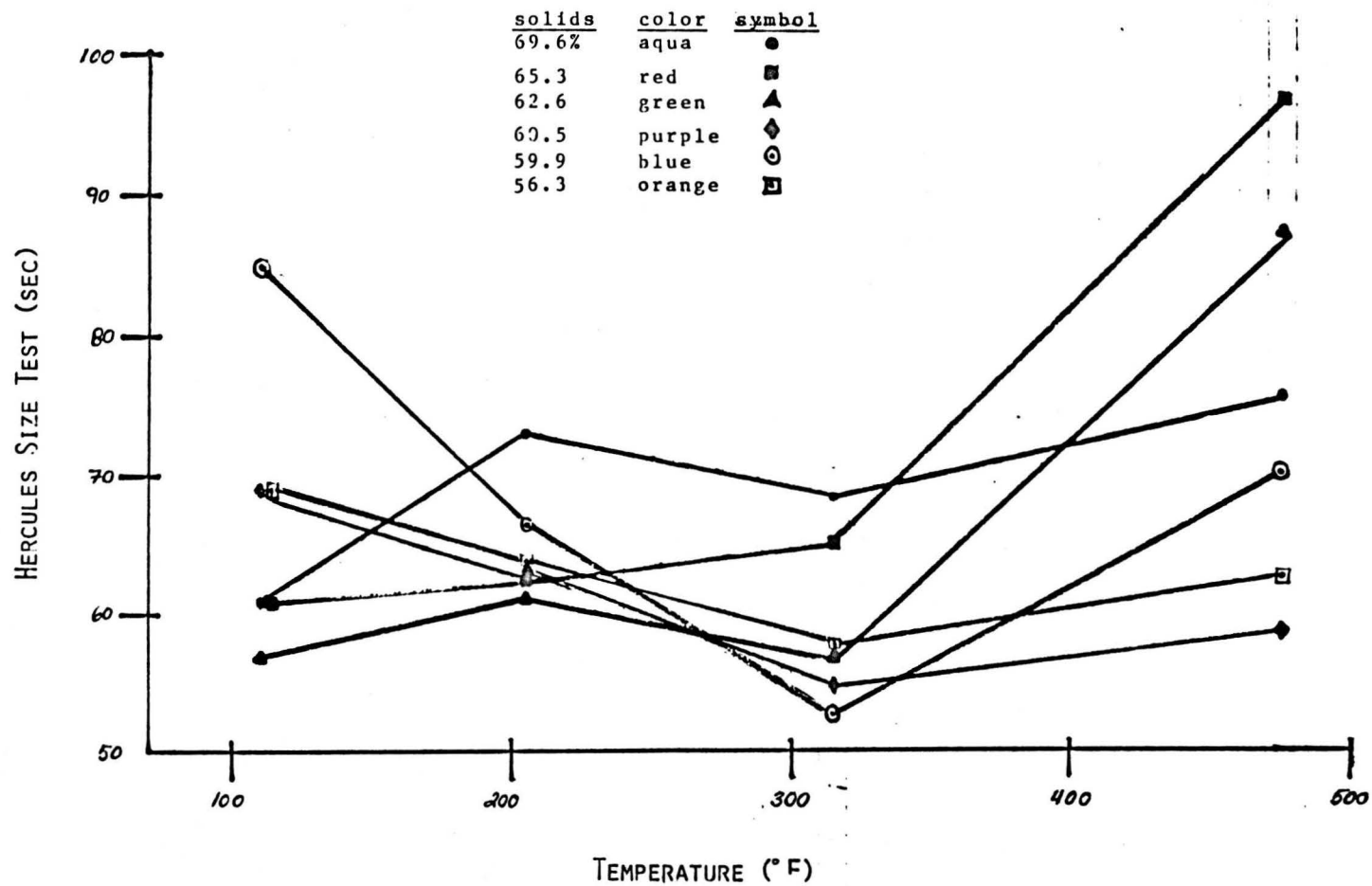
HST vs RATE OF WATER REMOVAL

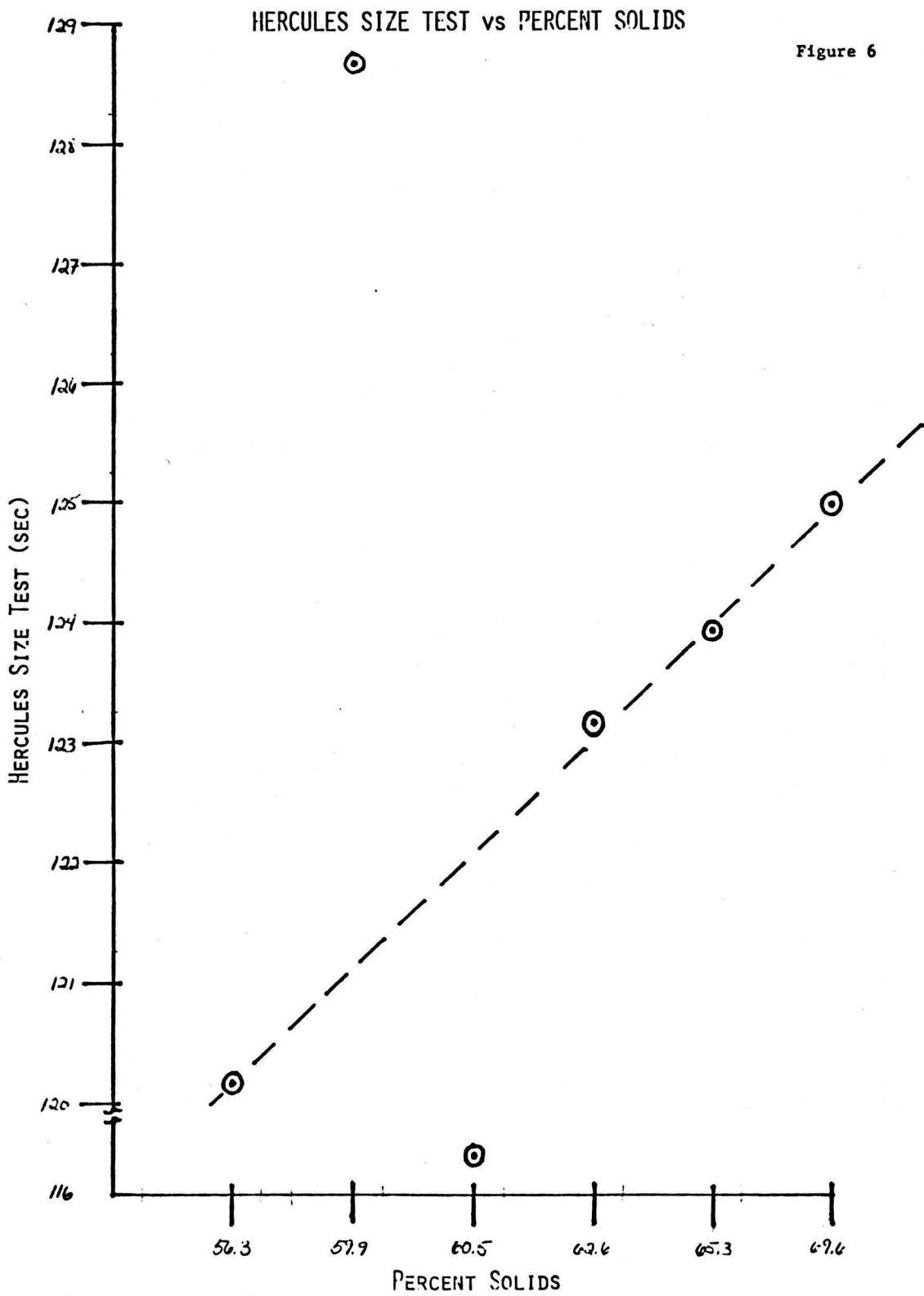
Figure 4



HERCULES SIZE TEST vs PERCENT SOLIDS

Figure 5





TWO-SIDEDNESS

It can be seen from the data in Table II which was generated by the air dry run that ASA-sized handsheets have two-sidedness. The side of the handsheet which dried in contact with the metal plate experienced higher sizing levels than the blotter side four out of five times. This variation from side to side may be due to the difference in pore structure or the difference in fines content. The side which dried in contact with the smooth plate had a smoother surface and probably a smaller average pore radius. The reduction of this radius would increase the contact angle between fiber surfaces and the liquid and thereby decrease penetration. Hence, an increase in sizing level. In handsheet production, fines tend to get caught on the forming wire and therefore increase in concentration on the bottom half of the sheet. These high surface area fines hold a large percentage of the ASA, starch and alum in the system and therefore give better sizing on the bottom side or the plate side of the sheet.

CATIONIC STARCH ADDITION

Table III which contains data from the 475°F trial indicates that the cationic starch addition rate is extremely important in determining the resultant sizing level. The only difference between the two runs was that the percent starch solids was higher in the second run. The master batch of starch had been kept at 90-100°F for 70 to 75 minutes and evaporation had occurred resulting in a greater solids concentration. Increasing the percent cationic starch in the system gave a tremendous increase in HST values. Therefore, starch made a major contribution to this ASA sizing mechanism.

Table II

TWO-SIDEDNESS DATA											
SHEET	1	2	3	4	5	6					
Weight	1.2510g	1.2510g	1.2414g	1.2475g	1.2371g	1.2280g					
	Used for demonstration	P	B	P	B	P	B	P	B	P	B
HST		96.4	78.0	106.8	80.7	114.5	86.6	91.2	73.2	77.1	83.2
		86.9	78.2	87.8	85.2	100.7	88.9	97.1	65.8	74.2	93.3
Mean		91.7	78.1	96.0	83.0	107.6	87.8	94.2	69.5	75.8	88.3

B= Blotter

P= Plate

Mean of blotter side (2-6) 81.3s
 Standard deviation 7.80
 Coeff. of variance 9.59

Mean of plate side (2-6) 93.1s
 Standard deviation 11.41
 Coeff. of variance 12.27

													Table III			
475°F	1		2		3		4		5		6		MEAN	S.D.	C. OF V.	
A																
% Solids	69.6		65.3		62.6		60.5		59.9		56.3		62.36	4.63	7.43	
Dry Time	15s		13s		13s		11s		12s		10s		12.3s	0.013	14.20	
Weight	1.2254g		1.2360g		1.2239g		1.2243g		1.1960g		1.1296g		1.2209g	0.013	1.09	
HST	55.8	88.1	79.3	79.8	66.3	90.4	59.9	50.3	56.9	80.5	64.1	50.8	75.9	18.65	24.57	
	84.2	75.1	111.7	114.4	100.3	92.1	---	67.2	58.2	84.7	53.6	81.1				
Mean	75.8		96.3		87.4		59.1		70.1		62.6					
Std. Dev.	14.4		19.37		14.76		8.48		14.57		14.04					
C. of V.	19.0		20.12		16.89		14.33		20.80		22.43					
B	1		2		3		4		5		6		MEAN	S.D.	C. OF V.	
% Solids	69.6		65.3		62.6		60.5		59.9		56.3		62.36	4.63	7.43	
Dry Time	13s		14s		14s		12s		12s		13s		13s	0.894	6.88	
Weight	1.2070g		1.2177g		1.1996g		1.2042g		1.1997g		1.1712g		1.1999g	0.016	1.30	
HST	188.1	155.9	97.1	158.3	152.3	146.3	143.8	---	119.9	157.6	103.5	153.6	155.8	28.82	18.50	
	187.2	136.3	----	150.5	162.0	168.6	173.3	135.0	192.8	139.5	214.5	191.2				
Mean	166.9		135.3		157.3		150.7		127.5		165.7					
Std. Dev.	25.29		33.31		9.93		20.10		27.76		48.47					
C. of V.	15.16		24.62		6.31		13.31		21.78		29.25					
A-B															Mean HST	75.2s
Mean Time	14s		13.5s		13.5s		11.5s		12s		11.5s			Std. Dev.	14.41	
Mean Wt.	1.2162g		1.2269g		1.2118g		1.2143g		1.1976g		1.1954g			C. of V.	19.16	
Mean HST																
Std. Dev.																
C. of V.																

SUMMARY OF RESULTS

Statistical analysis and graphing of data from all of the runs show several trends when comparing sizing levels to various drying conditions. After all tests were complete it became apparent that the overall best sizing levels were obtained by drying handsheets in the forced draft oven at 221°F (105°C) for 60 minutes.

Data indicates that the temperature and the rate of water removal effect the amount of sizing obtained in a sheet. The sizing level in the sheet decreases as the drying temperature increases to a certain point at which the HST values again increase. This suggests a competition between temperature during drying and rate of water removal during drying. Figures in the text demonstrate the variation of sizing level as the solids content of the sheets going into drying is changed. The general trend is to observe an increase in sizing level as these solids are increased.

Data from the 475°F trial reinforce the opinion that an ASA sizing system requires a retention aid to achieve a high degree of sizing. This is demonstrated by the increase in HST values at higher starch addition rates.

Finally, it is apparent that ASA does not give equal water resistance to both sides of a handsheet. Data indicate that the bottom side of a handsheet made on a British handsheet mold will give higher sizing values than the top side of the sheet. It is not known whether or not this is due to pore structure differences or fines migration or some combination of the two.

CONCLUSIONS

Given the results of this study, it is possible to conclude that drying is a variable in the ASA sizing mechanism. The temperature and type of drying will effect the amount of water resistance obtained. Also, the level of sizing obtained is dependent on the solids concentration of the handsheet going into the drying stage. The greater the percent solids, the more water resistance imparted to the sheet. Finally, British Mold handsheets made from pulp treated with ASA experience a sizing differential from side to side. The bottom side of the handsheet has the greater degree of sizing.

SUGGESTIONS FOR FURTHER WORK

This study could well be rounded out by repeating the procedure and testing the hot plate-dried handsheets for two sidedness. By elimination of plate contact during drying, it might be possible to determine how much of the two sidedness was due to pore structure. Also, a study which examined the effect of exposing the handsheets to heat for periods of time longer than those necessary to remove the water might prove useful. This might allow more insight into the effect of temperature alone on the degree of sizing obtained. Finally, it might be beneficial to vary the pressing and drying variables in a paper machine run using ASA to see how the sizing data compares to laboratory data.

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APPENDIX A

SYSTEM COMPONENTS

Standardized Water

1. water passed through one softener and two ion exchanger columns

Stock

1. 50% softwood, 50% hardwood
2. refined in Valley Beater to CSF of 350-400cc
3. consistency should be determined
4. stock should be stored at 40°F for no longer than two weeks

Chemicals

1. 0.1M HCl, 0.1M NaOH, 1% alum

Starch

1. National Cato 15 (793715)
2. should be made fresh every two - three hours
3. master batch: 700 grams wet, 0.8% solids,

$$\frac{700 (0.8/100)}{.9312*} = 6.01 \text{ grams starch}$$

$$700 - 6.01 = 693.99 \text{ grams cool standard H}_2\text{O}$$

$$*.9312 = \frac{\text{oven dry starch}}{\text{air dry starch}}$$
4. should be dispersed with mild mixing in a lab mixer
5. should be cooled in a double boiler for 30 minutes at 190°F with mild mixing
6. should be cooled to 90°F with addition to H₂O to make up for evaporation loss
7. should be kept on hot plate kept at 90-100°F with mild mixing until needed

APPENDIX A

(Cont.)

Sizing Agent/Nonionic Surfactant Emulsions

1. surfactant should be 7.0% of total emulsion weight
2. ASA should be 93% of total emulsion weight
3. above two should be mixed in a lab shaker for 20 minutes
4. emulsion should rest for 10 minutes to separate out air

ASA Starch Emulsion

1. starch should be diluted as follows:

$$V = \frac{1800y}{0.8}$$

where: V = volume of 0.8% solids Cato 15 (112.5 grams)

y = solids of Cato 15 desired (0.05%)

V will then be diluted to 1800 grams with H₂O at 100°F

2. starch add-on level should be calculated*

$$Z = \frac{1.2x}{y}$$

where: Z = volume of y% solids Cato 15/sheet (19.2 ml/sheet)

x = add-on level of Cato 15 (0.8%)

y = solids of diluted Cato 15 (0.05%)

*all calculations based on 1.2 grams OD weight handsheets

3. sheets per charge at above (x) add-on level should be calculated

$$S = \frac{1800}{z}$$

where: S = sheets per charge (93.75)

z = volume of y% solids starch/sheet (19.2 ml/sheet)

4. (Z) x 10 should be placed in the emulsifier for production of ten sheets (192.0 grams)

5. the ASA add-on level should be calculated as follows:

$$\frac{1.2 \times A}{B} = C$$

where: C = grams ASA/sheet (0.00258 grams/sheet)

A = add-on level (0.2%)

B = % ASA (93%)

APPENDIX A

(Cont.)

ASA Starch Emulsion (Cont.)

6. the ASA charge should be calculated

$$D = S \times C$$

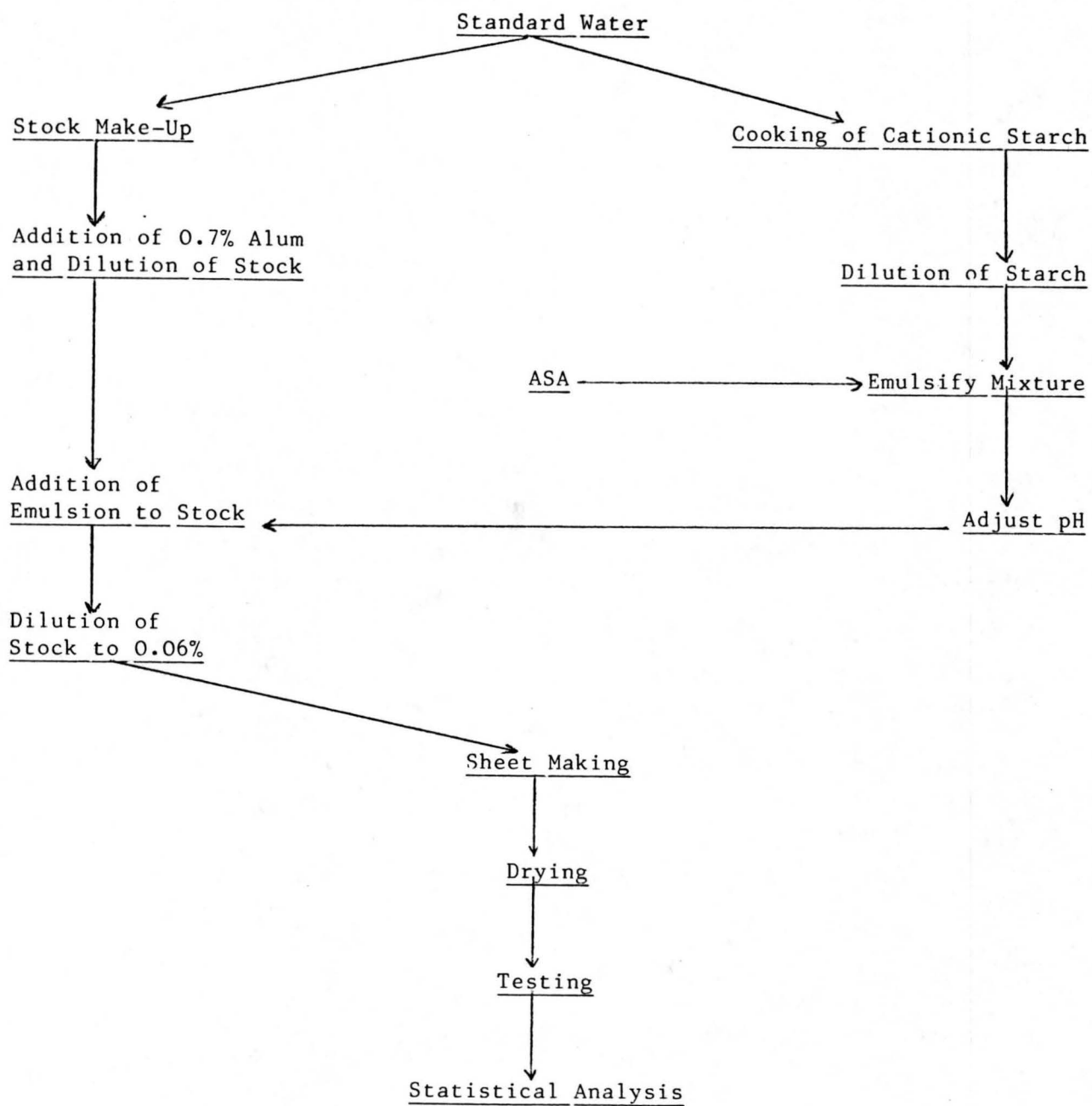
where: D = grams ASA/charge (0.242 grams/charge)
S = sheets/charge (93.75 sheets/charge)
C = grams ASA/sheet (0.00258 grams/sheet)

7. the mixture of ASA and starch should be emulsified

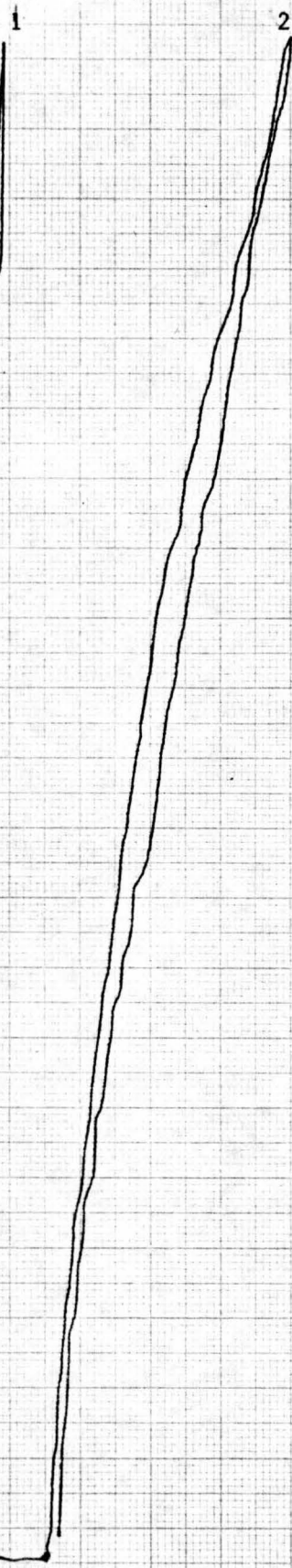
- A. weigh out D in a syringe (0.242 grams)
- B. add to vortex caused by emulsifier when on speed four over a period of 45 seconds
- C. turn to speed five after two minutes and take the pH
- D. adjust the pH to 7.6 with 0.1M NaOH and 0.1M HCl as needed

8. the emulsion should be used within 30 minutes

APPENDIX B
PROCESS DIAGRAM

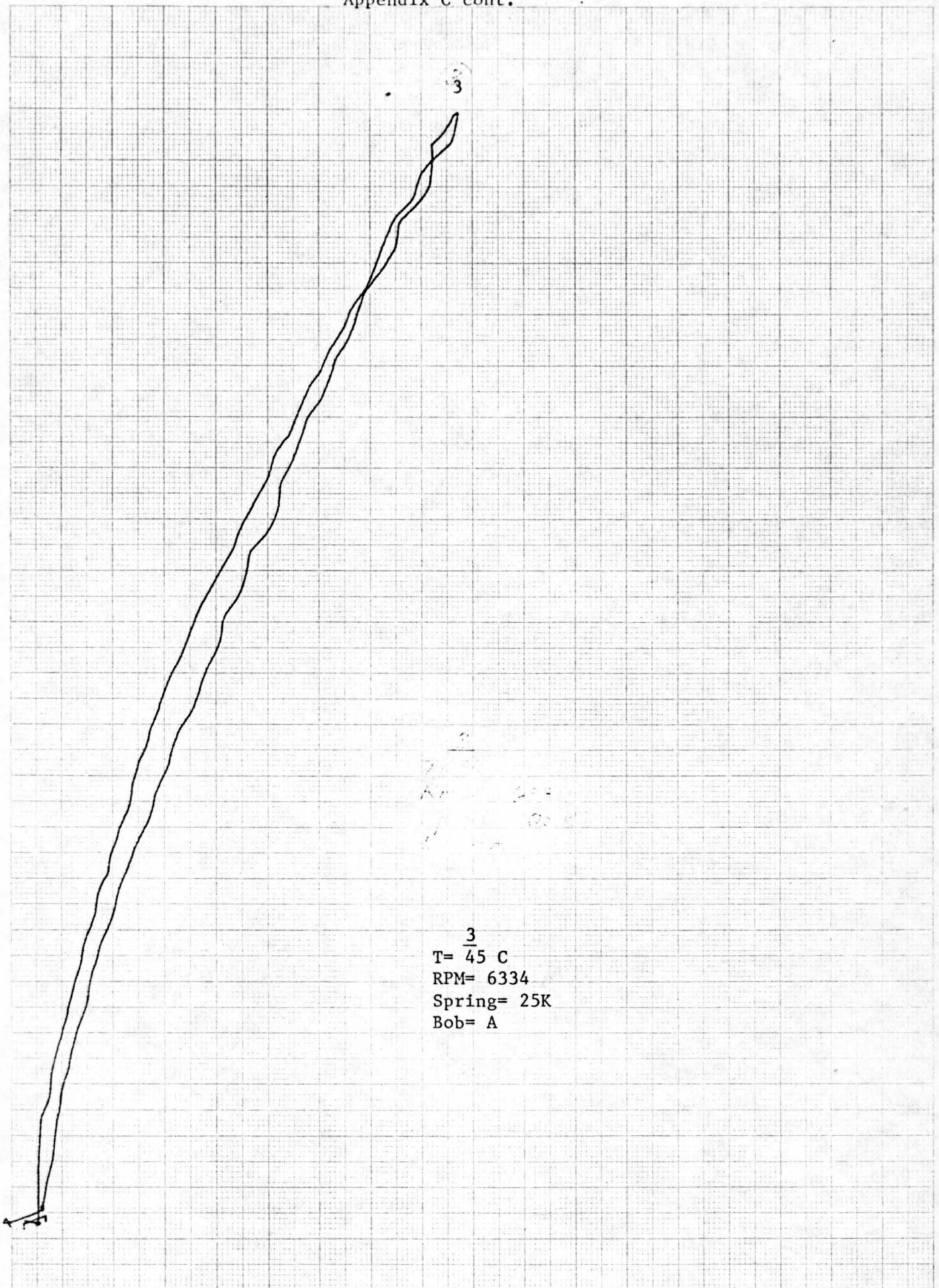


ASA/STARCH EMULSION RHEOGRAMS



$T = \frac{1}{45} \text{ C}$
RPM = 2200
Spring = 50K
Bob = A

$T = \frac{2}{45} \text{ C}$
RPM = 5290
Spring = 50K
Bob = A



Appendix D

HST vs RATE OF WATER REMOVAL

SHEET	100-120°F		200-210°F		317°F		475°F	
	HST	Rate	HST	Rate	HST	Rate	HST	Rate
1	60.7	.021	73.1	.040	68.1	.230	75.8	.342
2	61.5	.023	62.4	.041	65.6	.126	96.3	.483
3	57.0	.023	61.8	.042	57.0	.118	87.4	.538
4	69.1	.022	63.0	.042	55.4	.158	59.1	.695
5	84.9	.021	66.5	.049	52.7	.116	70.1	.693
6	69.1	.029	63.6	.053	57.7	.220	62.6	.906
Mean	67.1	.023	65.1	.045	59.4	.161	75.2	.601
Std. Dev.	10.76	.003	7.79	.005	6.06	.051	14.41	.194
C of V	16.05	12.9	11.96	11.78	10.20	32.03	19.16	32.31

Rate is in pounds of water per hour per square foot
HST is in seconds

C of V = coefficient of variance

105°C (221°F)- Control														Table IV		
A	1		2		3		4		5		6		MEAN	S.D.	C. OF V.	
% Solids	69.6		65.3		62.6		60.5		59.9		56.3		62.36	4.63	7.43	
Dry Time	1 hr		1 hr		1 hr		1 hr		1 hr		1 hr		1 hr	--	--	
Weight	1.3100g		1.0485g		1.0537g		1.0960g		1.1059g		1.1047g		1.0898g	0.032	2.94	
HST	295.8	263.9	216.6	265.4	228.4	338.4	278.8	303.9	357.1	308.8	411.4	317.6	304.0	46.76	15.38	
	271.3	315.8	230.3	---	330.2	280.7	308.6	307.9	338.1	345.1	370.6	307.7				
Mean	286.7		237.4		294.4		299.8		337.3		351.8					
Std. Dev.	23.71		25.17		50.86		14.15		20.54		48.37					
C. of V.	8.23		10.67		17.28		4.72		6.09		13.75					

B	1		2		3		4		5		6		MEAN	S.D.	C. OF V.
% Solids	69.6		65.3		62.6		60.5		59.9		56.3		62.36	4.63	7.43
Dry Time	1 hr		1 hr		1 hr		1 hr		1 hr		1 hr		1 hr	---	----
Weight	1.1639g		1.1731g		1.1386g		1.1588		1.1290g		1.1081g		1.1453g	0.024	2.14
HST	395.5	403.1	411.8	384.4	487.4	382.0	466.8	298.9	408.2	357.5	357.2	381.9	392.4	46.91	11.96
	454.7	379.9	423.0	405.2	371.7	401.8	----	380.1	430.4	408.9	356.7	279.1			
Mean	407.8		406.1		410.7		381.9		401.3		343.7				
Std. Dev.	32.68		16.22		52.62		84.0		30.93		44.66				
C. of V.	8.01		4.00		12.81		21.98		7.71		44.66				

A-B

Mean Time	1 hr	1 hr	1 hr	1 hr	1 hr	1 hr							
Mean Wt.	1.1470g	1.1108g	1.0962g	1.1274g	1.1175g	1.1064g							
Mean HST	347.3	333.8	352.6	335.0	369.3	347.8							
Std. Dev.	69.92	92.04	78.49	66.16	41.69	43.32							
C. of V.	20.14	27.57	22.26	19.75	11.36	12.46							

Mean HST 346.60s

Std. Dev. 57.35

C. of V. 16.55

100-120°F													
A	1		2		3		4		5		6		Table V MEAN S.D. C. OF V.
% Solids	69.6		65.3		62.6		60.5		59.9		56.3		62.36 4.63 7.43
Dry Time	252s		271s		312s		361s		453s		290s		323s 73.89 22.86
Weight	1.2215g		1.2277g		1.2177g		1.2201g		1.2109g		1.1868g		1.2141g 0.014 1.19
HST	76.6	72.2	60.1	64.8	52.9	72.3	56.0	62.7	84.3	85.9	63.9	82.9	68.2 11.68 17.14
	54.0	61.5	52.2	53.0	63.6	61.8	63.2	72.7	70.6	95.6	74.9	77.9	
Mean	66.1		57.5		62.7		63.7		84.1		74.9		
Std. Dev.	10.25		6.01		7.95		6.87		10.29		8.04		
C. of V.	15.51		10.44		12.70		10.79		12.23		10.74		

B	1		2		3		4		5		6		MEAN S.D. C. OF V.
% Solids	69.6		65.3		62.6		60.5		59.9		56.3		62.36 4.63 7.43
Dry Time	250s		280s		290s		353s		303s		328s		300.7s 36.33 12.08
Weight	1.2793g		1.2460g		1.2561g		1.2586g		1.2387g		1.2172g		1.2493g 0.021 1.67
HST	54.3	62.7	65.4	62.8	56.1	58.5	58.6	73.5	53.7	87.0	72.9	64.7	66.0 15.70 23.80
	52.1	52.5	68.6	65.5	46.7	44.1	91.8	74.1	99.0	103.2	53.9	62.0	
Mean	55.4		65.4		51.4		74.5		85.7		63.4		
Std. Dev.	4.96		2.43		7.02		13.58		22.43		7.83		
C. of V.	8.95		3.71		13.67		18.23		26.16		12.36		

A-B													Mean HST 67.1s
Mean Time	251s		275.5s		301s		357s		378s		309s		Std. Dev. 10.76
Mean Wt.	1.2540g		1.2369g		1.2369g		1.2394g		1.2248g		1.2020g		C. of V. 16.05
Mean HST	60.7		61.5		57.0		69.1		84.9		69.1		
Std. Dev.	9.39		5.97		9.20		11.53		16.18		9.59		
C. of V.	15.45		9.71		16.15		16.69		19.05		13.87		

200-210°F							Table VI		
A	1	2	3	4	5	6	MEAN	S.D.	C. OF V.
% Solids	69.6	65.3	62.6	60.5	59.9	56.3	62.36	4.63	7.43
Dry Time	185s	165s	170s	189s	149s	166s	162.3s	18.53	11.41
Weight	1.2783g	1.2590g	1.2553g	1.2464g	1.2489g	1.2368g	1.2541g	0.014	1.13
HST	66.2 69.6 68.5 58.8	54.9 56.5 50.3 65.8	45.3 61.0 53.2 60.6	51.1 50.0 55.3 62.3	70.0 60.1 52.4 58.2	62.8 58.6 60.5 66.6	59.10	6.71	11.36
Mean	65.8	56.9	55.0	54.7	60.2	62.1			
Std. Dev.	4.86	6.50	7.41	5.57	7.32	3.44			
C. of V.	7.39	11.44	13.47	10.19	12.17	5.54			

B	1	2	3	4	5	6	MEAN	S.D.	C. OF V.
% Solids	69.6	65.3	62.6	60.5	59.9	56.3	62.36	4.63	7.43
Dry Time	135s	150s	165s	182s	173s	177s	163.7s	17.95	10.97
Weight	1.2776g	1.2826g	1.2816g	1.2637g	1.2534g	1.2542g	1.2698g	0.013	1.06
HST	75.1 72.7 81.4 92.7	72.2 67.6 69.7 62.0	76.4 66.1 65.0 66.6	69.6 60.6 69.1 86.1	74.8 70.6 68.0 78.1	69.7 62.0 62.5 66.0	71.0	7.8	10.99
Mean	80.5	67.9	68.5	71.3	72.9	65.1			
Std. Dev.	8.94	4.34	5.29	10.69	4.47	3.57			
C. of V.	11.11	6.40	7.72	15.00	6.13	5.49			

A-B							Mean HST	65.1s
Mean Time	135s	157.5s	172.5s	185.5s	161s	171.5s	Std. Dev.	7.79
Mean Wt.	1.2780g	1.2708g	1.2685g	1.2551g	1.2512g	1.2455g	C. of V.	11.96
Mean HST	73.1	62.4	61.8	63.0	66.5	63.6		
Std. Dev.	10.3	7.80	9.36	11.86	8.81	3.61		
C. of V.	14.09	12.50	15.15	18.84	13.24	5.67		

317°F							Table VII		
A	1	2	3	4	5	6	MEAN	S.D.	C. OF V.
% Solids	69.6	65.3	62.6	60.5	59.9	56.3	62.36	4.63	7.43
Dry Time	30s	55s	40s	40s	35s	30s	38.3s	9.31	24.29
Weight	1.2563g	1.2301g	1.2309g	1.2319g	1.2148g	1.2162g	1.1937g	0.015	1.22
HST	179.7 202.7 204.4 198.0	215.6 205.9 202.8 152.1	168.3 147.0 210.5 191.4	161.2 197.4 159.7 205.8	163.1 159.6 167.7 136.6	136.3 163.9 178.5 183.0	178.8	24.29	13.59
Mean	196.2	194.1	179.3	181.0	156.8	165.5			
Std. Dev.	11.33	28.53	27.59	13.45	13.84	20.92			
C. of V.	5.77	14.70	15.39	7.43	8.83	12.64			

B	1	2	3	4	5	6	MEAN	S.D.	C. OF V.
% Solids	69.6	65.3	62.6	60.5	59.9	56.3	62.36	4.63	7.43
Dry Time	22s	49s	55s	47s	65s	40s	46.3s	14.58	31.47
Weight	1.2078g	1.2143g	1.1882g	1.1888g	1.1804g	1.1827g	1.1937g	0.014	1.22
HST	64.2 58.4 68.5 81.1	65.6 75.3 65.1 56.4	47.0 69.9 57.2 53.7	49.4 61.1 70.9 40.2	53.1 46.1 56.5 55.2	66.2 58.7 56.7 49.3	59.4	9.81	16.52
Mean	68.1	65.6	57.0	55.4	52.7	57.7			
Std. Dev.	9.63	7.72	9.61	13.41	4.63	6.95			
C. of V.	14.16	11.77	16.88	24.21	8.79	13.04			

A-B

Mean Time	26s	52s	47.5s	43.5s	50s	43s	Mean HST	59.4s
Mean Wt.	1.2321g	1.2222g	1.2096g	1.2104g	1.1976g	1.1995g	Std. Dev.	6.06
Mean HST							C. of V.	10.20
Std. Dev.								
C. of V.								

												Table VIII			
475°F A	1		2		3		4		5		6		MEAN	S.D.	C. OF V.
% Solids	69.6		65.3		62.6		60.5		59.9		56.3		62.36	4.63	7.43
Dry Time	15s		13s		13s		11s		12s		10s		12.3s	0.013	14.20
Weight	1.2254g		1.2360g		1.2239g		1.2243g		1.1960g		1.1296g		1.2209g	0.013	1.09
HST	55.8	88.1	79.3	79.8	66.3	90.4	59.9	50.3	56.9	80.5	64.1	50.8	75.9	18.65	24.57
	84.2	75.1	111.7	114.4	100.3	92.1	---	67.2	58.2	84.7	53.6	81.1			
Mean	75.8		96.3		87.4		59.1		70.1		62.6				
Std. Dev.	14.4		19.37		14.76		8.48		14.57		14.04				
C. of V.	19.0		20.12		16.89		14.33		20.80		22.43				

B	1		2		3		4		5		6		MEAN	S.D.	C. OF V.
% Solids	69.6		65.3		62.6		60.5		59.9		56.3		62.36	4.63	7.43
Dry Time	13s		14s		14s		12s		12s		13s		13s	0.894	6.88
Weight	1.2070g		1.2177g		1.1996g		1.2042g		1.1997g		1.1712g		1.1999g	0.016	1.30
HST	188.1	155.9	97.1	158.3	152.3	146.3	143.8	---	119.9	157.6	103.5	153.6	155.8	28.82	18.50
	187.2	136.3	----	150.5	162.0	168.6	173.3	135.0	192.8	139.5	214.5	191.2			
Mean	166.9		135.3		157.3		150.7		127.5		165.7				
Std. Dev.	25.29		33.31		9.93		20.10		27.76		48.47				
C. of V.	15.16		24.62		6.31		13.31		21.78		29.25				

A-B							Mean HST	75.2s
Mean Time	14s	13.5s	13.5s	11.5s	12s	11.5s	Std. Dev.	14.41
Mean Wt.	1.2162g	1.2269g	1.2118g	1.2143g	1.1976g	1.1954g	C. of V.	19.16
Mean HST								
Std. Dev.								
C. of V.								

Appendix D cont.

MOISTURE CONTENT DETERMINATION

SAMPLE	A.D. WEIGHT	O.D. WEIGHT	A.D.-O.D.	% MOISTURE
1	1.2628g	1.1940g	0.0688	5.4%
2	0.3282	0.3110	0.0172	5.24
3	0.3368	0.3206	0.0162	4.81
4	0.3154	0.2992	0.0162	5.14
5	0.3189	0.3020	0.0169	5.30
<u>Average</u>				<u>5.18%</u>

APPENDIX E

CALCULATION FOR RATE OF WATER REMOVAL

$$(A) (0.948) = B$$

$$\frac{(O.D. \text{ wt.})}{S} = Y$$

$$Y - B = W$$

$$\left(\frac{W}{(T) \cdot (H)} \right) \left(\frac{1\#}{454 \text{ grams}} \right) \left(\frac{12 \text{ inches}}{1 \text{ foot}} \right)^2 \left(\frac{3600 \text{ seconds}}{1 \text{ hour}} \right) = R$$

A = air dry weight of handsheet

B = oven dry weight of handsheet

S = percent solids of handsheet

Y = weight of sheet after pressing

W = weight of water removed during drying

T = time to dry in seconds

H = area of handsheet in square inches

R = rate of water removal in pounds/(hour x foot²)