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THE EFFECT OF SHEAR AND ALUM ADDITION ON  
ALKENYL SUCCINIC ANHYDRIDE RETENTION

By:

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

Western Michigan University  
Kalamazoo, Michigan

June, 1984

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## ABSTRACT

Alkenyl succinic anhydride is an effective sizing agent involved in alkaline papermaking. There are many factors in the alkaline papermaking system which effect the retention of the sizing agent. This study reviews factors which influence sizing and the effects of shear and alum addition on the retention of ASA. The Dynamic Drainage Jar was employed to subject four levels of shear on the system and Hercules size determinations were conducted as a measure of retention. Alum is necessary in the system to provide proper retention of the ASA. Retention of ASA with alum present was found to increase with increasing shear until a point of 1000 RPM was reached. After this point, retention decreased sharply.

Keywords: Alkenyl Succinic Anhydride, Aluminum Sulfate, Retention, Shear, Sizing.

## INTRODUCTION

The purpose of this study is to investigate the effect of shear and alum addition on the retention of alkenyl succinic anhydride used as an alkaline sizing agent. Its chemistry and mechanisms of sizing will be studied as factors which influence its sizing and retention.

## LITERATURE

Alkenyl succinic anhydride (ASA) is an important part of alkaline papermaking. It is added to an alkaline papermaking system to control the penetration of aqueous liquids. It is therefore necessary to understand the chemistry and mechanisms which are involved. The following literature search deals with ASA and factors which may influence its sizing and retention.

### Chemistry of Cellulose Reactive Sizes

Cellulose reactive sizes are non-ionic organic compounds which are emulsified with dispersants and stabilizers prior to use as a sizing agent. There are two types of cellulose reactive sizes being used in alkaline papermaking. These are alkenyl succinic anhydrides and alkyl ketene dimers. This study is primarily concerned with alkenyl succinic anhydride.

Sizing occurs by the formation of a covalent bond between the size and the cellulose. Since these compounds are non-ionic, hydrophobic, organic compounds, they are insoluble in water. In order for the covalent bond to form, the compound must be emulsified to make it compatible with the aqueous papermaking system.(1)

A cellulose reactive size is composed of a hydrophobic and a hydrophilic group. The hydrophobic group provides water repellency when properly oriented away from the cellulose fiber. It provides sizing by raising the contact angle of the surface of the cellulose and helps determine the emulsion stability.(1,2) The reactive hydrophilic group is the most important component of the reactive size and determines the rate of reaction with water and cellulose.(2)

ASA size is a petroleum derived compound composed of a hydrophobic group attached to a hydrophillic group, as shown in Figure 1. As stated earlier, the hydrophobic end of the molecule provides water repellency, and the reactive hydrophillic group establishes the rate of reaction with the cellulose.

The mechanism of sizing involved with ASA consists of the hydrophillic group reacting with the hydroxyl groups of the cellulose fibers and forming a strong covalent bond.<sup>(2)</sup> This reaction is shown in Figure 2.

Dumas <sup>(1)</sup> found that ASA develops sizing quickly and the emulsion reacts quickly with water to form a hydrolyzate. This hydrolyzate interferes with sizing. He also found that a cationic material such as starch is required for proper size retention.

In the ASA sizing system, retention of the size particle occurs in the wet end. The retention continues into the dryer section where the orientation of the size molecules and the reaction mechanism occurs. The total sizing development occurs before the size press.<sup>(2)</sup>

### Factors Influencing Sizing

Penetration of aqueous liquids can occur by interfiber or intrafiber penetration. Interfiber penetration is fluid penetration through the pores or spaces between the paper fibers and intrafiber penetration occurs through the fibers themselves.<sup>(2)</sup>

Internal sizing consists of depositing a water repellent material on the fiber which results in a water resistant paper. The fibers and the size are anionically charged and repel each other. Therefore, in most cases, a cationic material such as alum is needed to bring them together.<sup>(3)</sup> As mentioned earlier, cationic starch is often used in an alkaline system.

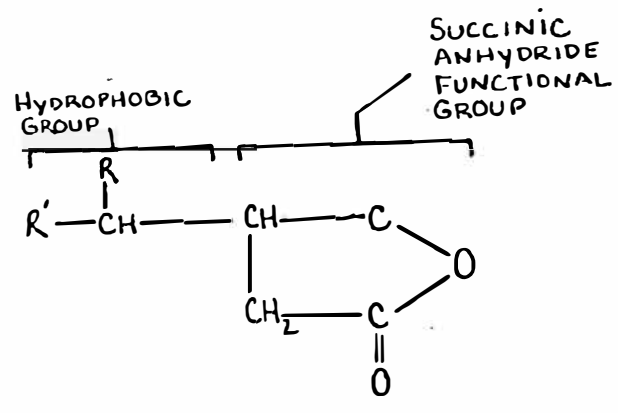


Figure 1 Structure of ASA Size

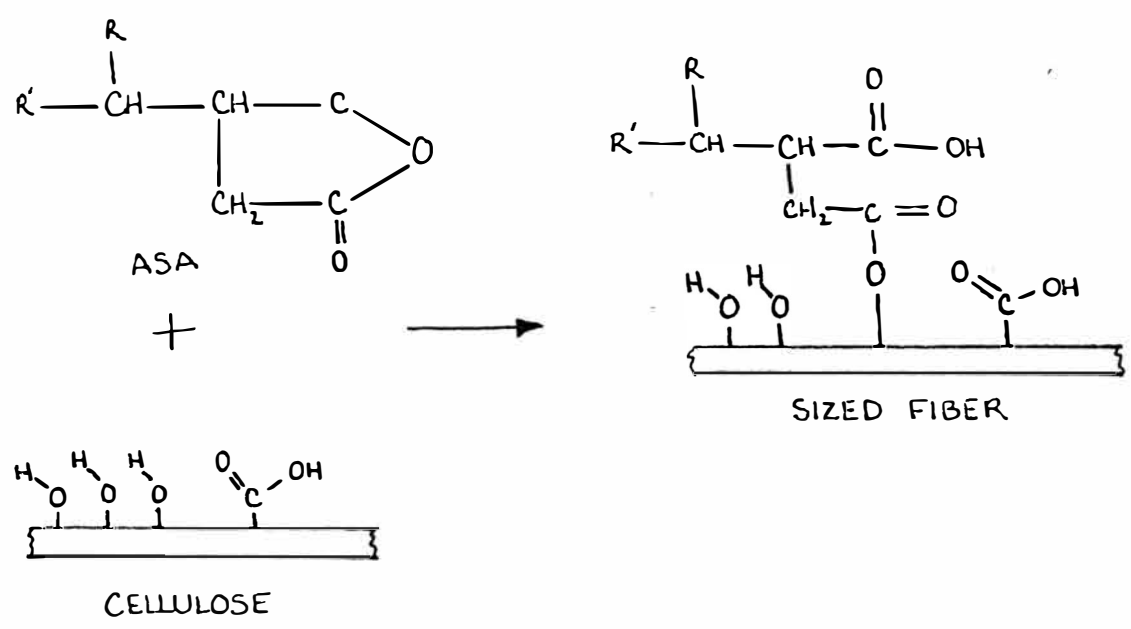


Figure 2 Reaction of ASA with Cellulose



To obtain the best efficiency of the sizing agent, it should be retained on the long fibers which are the easiest to retain in the system. This is made difficult because of the fines which are present. These are the most difficult to retain and also have the greatest affinity for the cationic size.(1)

Poor sizing can be attributed to factors such as poor retention or ineffective sizing. Poor retention can occur if the fibers are not receptive enough to the size. The larger the surface area, the better the retention. pH is an important factor influencing the retention of the size. If the pH is wrong, retention aids will fail. Along with pH, the sequence and timing of the addition of the size, retention aid, and other chemicals is critical. Meaker (3) suggests that a common cause of ineffective sizing when synthetic sizes are used, is poor orientation. This can be related to poor retention or improper drying.

One of the main factors which causes a loss is retention on the paper machine is shear.(4,5) This loss of retention may be related to poor sizing due to poor sizing retention. Abson (4) studied the relationship of the dependence of sizing on hydrodynamic shear (rosin size) and found that retention is improved initially with increasing shear, and subsequently declined at higher shear. Abson's study dealt with a rosin system and although alkaline size has been studied extensively, the relationship between alkaline size retention and shear does not appear to have been studied systematically. No research has been found to explain what happens to an ASA molecule when is is subjected to shear.

### Retention

The retention of fines and other colloidal particles by the cellulose

fiber is one of the most complex physiochemical problems associated with papermaking. Because of this and the possibility that a loss of fines may result in a loss in sizing, a better understanding of how fines are retained is in order.

Walkush and Williams (6) suggest three different mechanisms of retention: 1) filtration, the process of removing particles larger than the pore openings during sheet formation; 2) entrapment, the physical collection of particles in the fiber lumens or in the fibril structure on the fiber surface; and 3) coflocculation, the interaction of the interfacial forces of the particles which control ordinary colloidal flocculation.

Using the double layer model developed by Stratton & Swanson (7), the interaction of the suspended colloidal particles can be explained by Figure 3. The charge on a particle (usually negative) attracts ions of opposite charge. The next layer, known as the Stern layer, contains immobilized counterions. The Gouey layer is formed by the remainder of the counterions. The surrounding liquid is bound to the particle for a definite thickness and an electric potential exists at the plane of shear between the bound liquid and the fill liquid. By using electrokinetic methods, this double layer can be measured. The electrokinetic charge on the particle as it moves through a fluid is a negative charge, diminished in part by the positive ions in the inner layer. This charge is known as the zeta potential.(7) The net negative charge could be a repulsive charge. In addition to this, all particles have an attractive electrostatic charge known as Van Der Waal's forces.

To improve retention one must reduce the zeta potential to zero. This means that the charges have been neutralized. A means of accomplishing this end is to add trivalent cations to the system. These ions interact with the negatively charged particles, and because they possess a stronger charge,

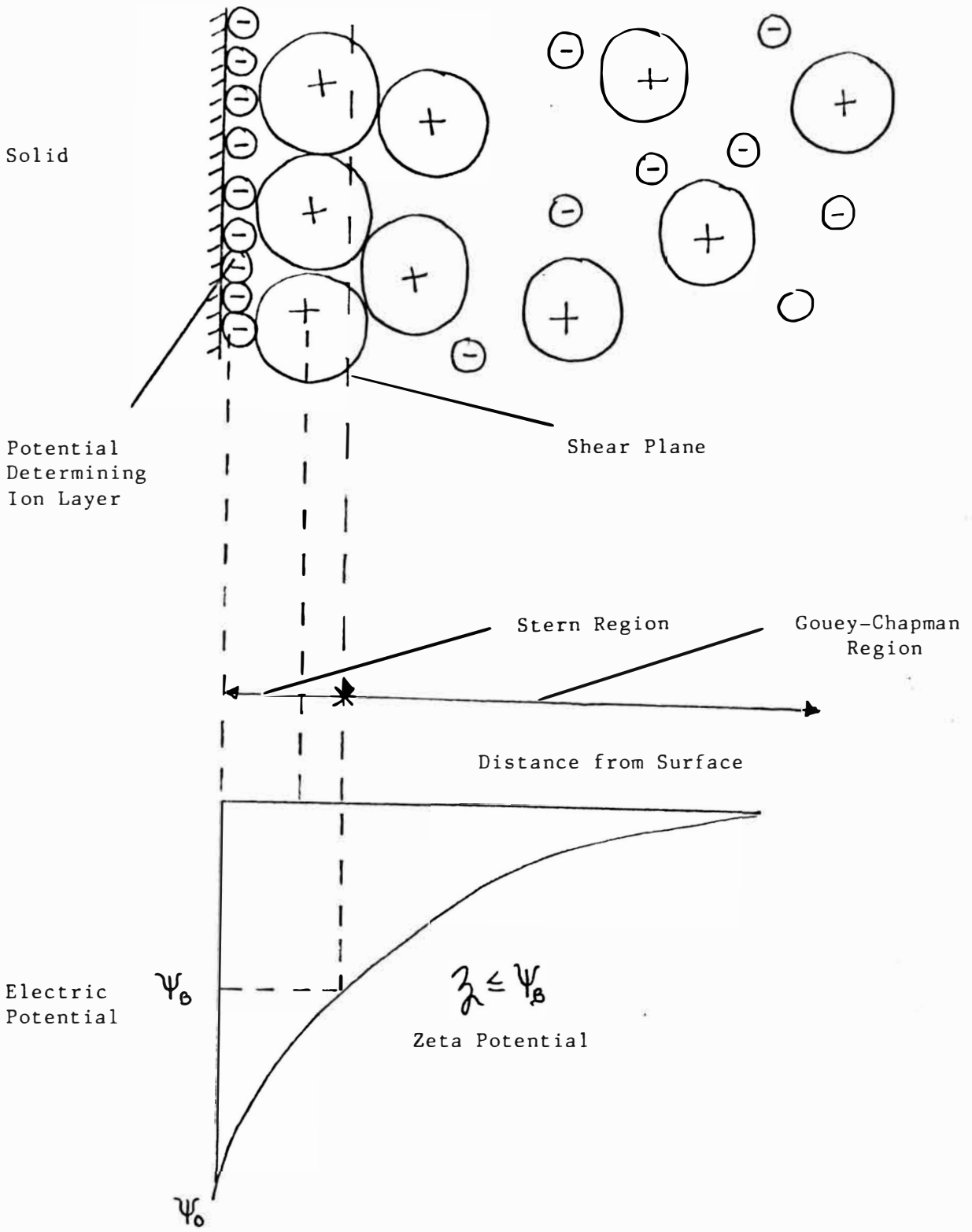


Figure 3

displace the monovalent cations. The effect of this is to reduce the net negative charge and thus lower the repulsive force. In this condition, the particles will not repulse each other and upon colliding, will bond together.(8)

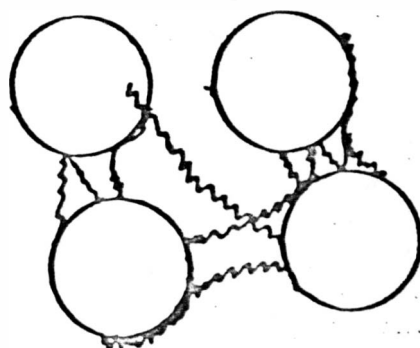
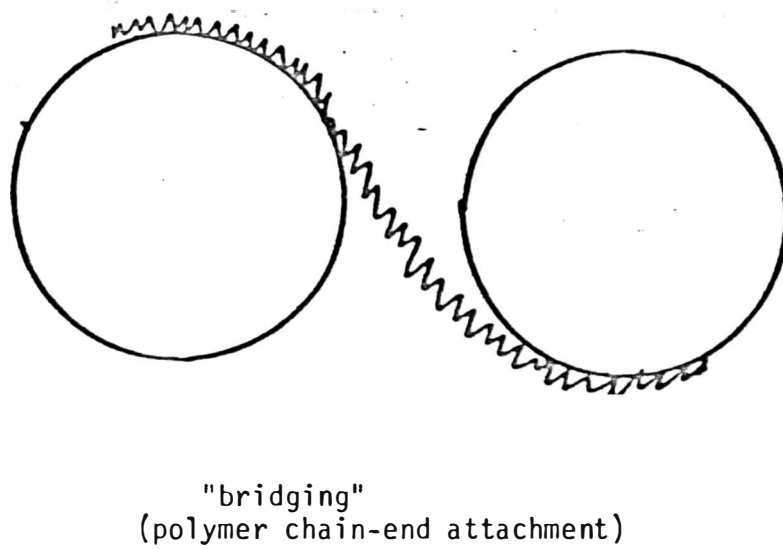
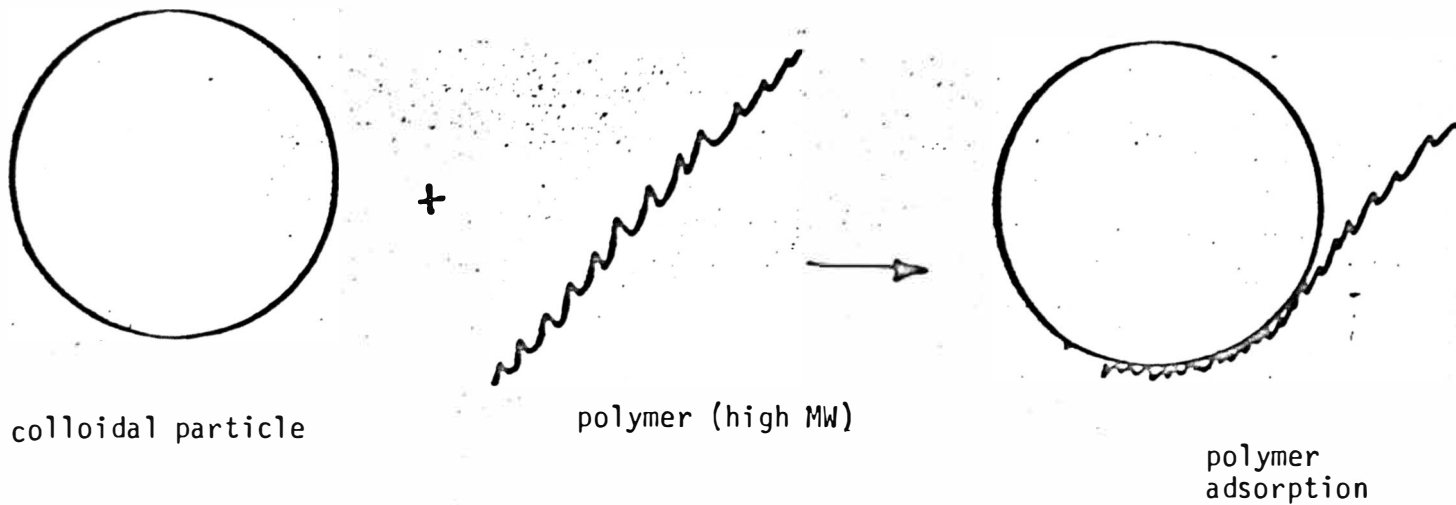
### Flocculation

Flocculation or aggregate formation in a cellulose system can occur via three basic mechanisms: 1) coagulation, 2) bridging by high molecular weight polymers, and 3) electrostatic patch formation.

Walkush and Williams (6) define the coagulation process as the aggregation of charged particles induced by the compression of the electrical double layer or by the reduction in surface charge. This method does not fall under the general description of flocculation, as bridging and patch formation do, although its end result is the same.

Flocculation by the bridging mechanism can occur for a wide variety of colloidal particles including the pulp fines. This mechanism consists of a connection between a long chained polymer and two or more particles (Figure 4). Davison (9) describes bridging as a large loosely structured floc of the fine particles, held together weakly by the adsorbed long - chain bridging molecules. These flocs were found to form very rapidly, were partially degraded by turbulent shear forces, and re-formed readily when turbulence ceased.

According to LaMer and Healy (10), two types of bridging exist: bridging through chain end attachment and bridging through loops and tails. In chain end attachment, one end of a polymer chain is adsorbed onto one particle and the other end attaches to yet another particle. With loops and tails, a long chain polymer will adsorb at various locations on the particle surface while



bridging through loops and tails

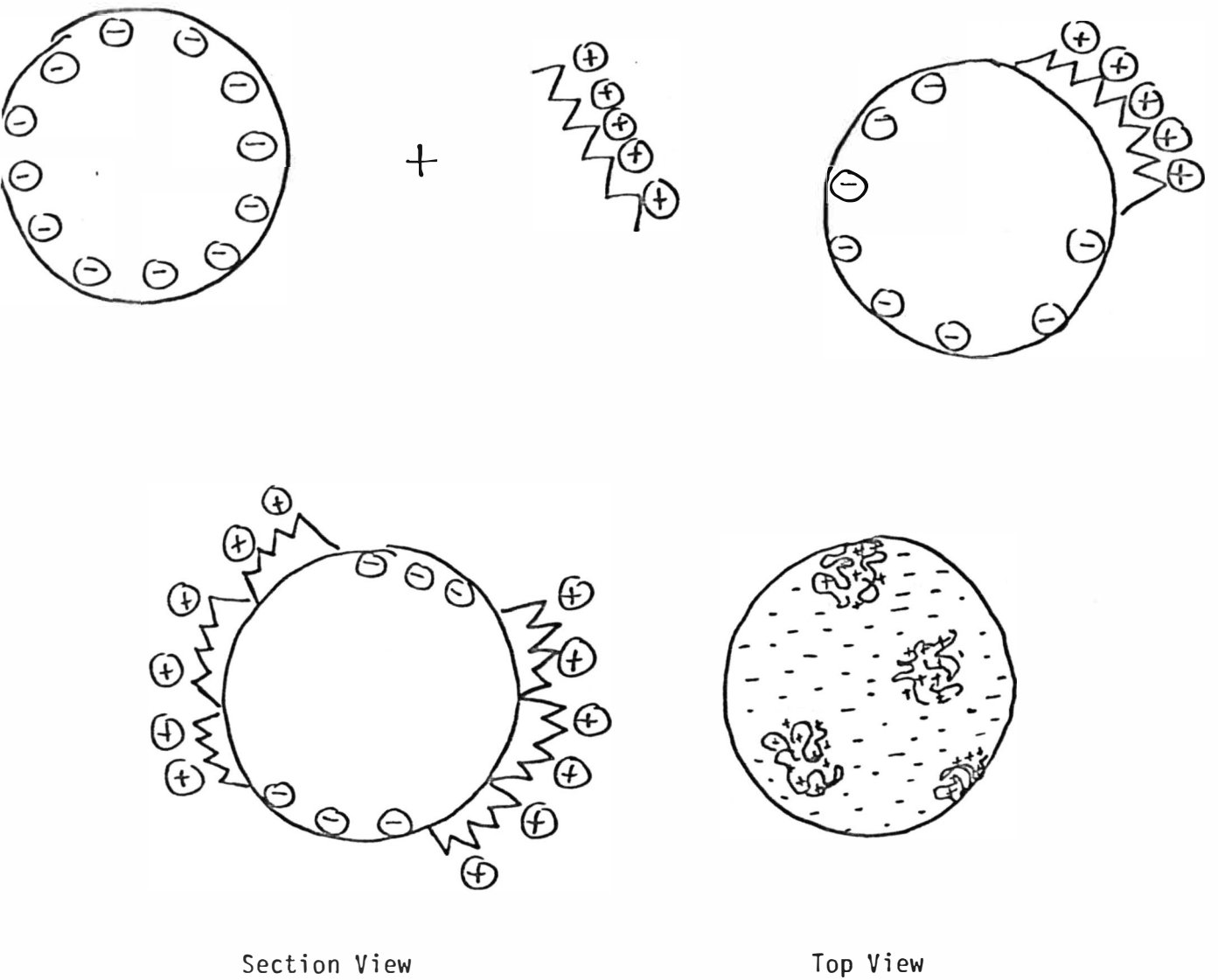
the unattached portions form bonds with other particles. Once an aggregate is formed, the strength of its internal attractive forces compared to the hydrodynamic shear forces it encounters in the medium, determine its stability.

There exists three basic requirements for flocculation: 1) extended segments must be available for bridging, 2) these segments must be long enough and large enough so that stable flocs can be formed for bridging, and 3) free surface area must be available to act as bridging sites. A flocculated system can become and remain un-flocculated if bridging is prevented from occurring. This can happen when the surface of the particles become over-saturated or when the extended segments interfere with one another.(11)

The patch model deals with a partial neutralization of the particle. An attraction exists between the negative and positive sites on different particles (see Figure 5). Partial neutralization of the particles is brought about by the absorption of low molecular weight, high charge density cationic polymers, thus positive areas attract the unabsorbing negative areas, causing flocculation.(12)

Unbehend (13) proposed that there exist two different types of floc formation pertaining to retention. The first type is referred to as a "soft floc" and is described as any combination of fiber and fines (cellulosic and/or mineral) which shows an improvement in overall fines retention at relatively low turbulence. When exposed to high levels of turbulence and then allowed to reflocculate, the system returns to its original retention level. The second type, "hard flocs", is described as any stock system plus additive that exhibits good fines retention over a wide range of turbulence for brief periods. This will break down after subsequent exposure to high degrees of turbulence for longer times.

Fig. 5 Schematic of a charge patch model



Important factors which influence the effectiveness of flocculation and stability include polymer type, polymer concentration, electrolyte type, electrolyte concentration, fines addition level, agitation level, and pH. It is desirable to have good formation and retention. Therefore, the ultimate condition would be one with good fiber dispersion while maintaining good fines retention. The fine particles of the stock system contribute large surface areas and therefore require greater polymer concentrations to achieve maximum flocculation. They also require more polymer to reach redispersion.(14)

#### Measurement of Retention

Two methods of which are available for measuring retention are zeta potential control and the Dynamic Drainage Jar.

As mentioned earlier, the addition of trivalent cations to the system will reduce the net negative potential. Since the same influences that reduce the negative zeta potential also promote flocculation and retention, it is suggested by Britt (15) that there is a close relationship between zeta potential and flocculation. He also suggested that electrical potential may be the predominant influence in retention. Stratton and Swanson (7) also suggest that the zeta potential is a measure of the amount of an additive that is absorbed on surface which is retention related.

The Dynamic Drainage Jar was developed by Britt and Unbehend to measure the degree of colloidal retention between fibers and fines in a paper stock system. This measurement is independent of mechanical filtration variables that effect retention.(16)

#### Alum Addition

There are numerous additives that have the effect of increasing the



degree of flocculation of the paper stock. One of the most common additives used to achieve flocculation in paper stock systems is alum. Alum, which is chemically known as aluminum sulphate is the primary source of aluminum for most papermaking systems. The pH of a papermaking system using aluminum salts can range from slightly alkaline to acidic. However, most systems operate at pH 4.0 - 5.5 because better results are obtained in this range.(17) The accepted structure of the aluminum ion in solution is a hydrated form containing six molecules of water, or  $\text{Al}(\text{OH})_6^{+3}$ . The oxygen atoms are coordinated with the aluminum in the center of a three-dimensional structure. This hydrated ion is a weak acid and can ionize.(18) Thus, alum can affect the zeta potential in two ways. It can adsorb onto the surface as a specific ion to change the surface potential or it can act as a multivalent ion in solution to increase the ionic strength of the medium and thereby compress the double layer.(7) Arnson (17) has shown that the amount of aluminum absorbed on the cellulose is governed by the equilibria between the species  $\text{Al}^{+3}$ ,  $\text{Al}_8(\text{OH})_{20}^{+4}$ , and  $\text{Al}(\text{OH})_3$ . The relative amounts of each species depends on both total aluminum concentration and pH.

The main difference in the acid vs. alkaline systems relates to the role of alum in the rosin sizing process. In the acid process, the alum reacts with the rosin to form a size precipitate. The rosin-alum chemistry determines the retention, distribution, and anchoring functions of the size on the pulp. The chemical transformation generally takes place at the wet end of the paper machine.(1)

In alkaline systems, the size performs in a similar manner except the anchoring which occurs, is provided by the covalent bond which is formed between the size and the cellulose. Meaker (3) found that acceptable sizing levels can not be obtained in an alkaline system (using ASA) without alum. He

also determined that the effect of alum on sizing, levels off near 12 lb./ton and it should be added as early as possible in the blend sequence to produce optimum sizing levels.

## CONCLUSIONS

Alkenyl succinic anhydride can be used effectively in alkaline papermaking as a sizing agent. It is used in an emulsified form and the addition of a cationic polymer has been found to give size retention. ASA is composed of a hydrophobic and hydrophilic group. The hydrophobic end of the molecule provides water repellency and the reactive hydrophilic group establishes the rate of reaction. The mechanism of sizing involved consists of the hydrophilic group of the ASA reacting with the hydroxyl groups of the cellulose to form a covalent bond.

The major factor causing a loss in retention may be shear. Other factors, such as pH, timing, addition of chemicals, and orientation also affect the retention of the size. In order to fully understand how the ASA is retained, it is important to understand flocculation and the mechanisms such as bridging and patch formation which are involved.

Methods of measuring retention consist of zeta potential and the use of the Dynamic Drainage Jar.

Such additives as alum play an important role in the retention of ASA size. Without the alum, sufficient sizing levels can not be obtained.

## STATEMENT OF THESIS

The mechanisms and variables involved in the retention of alkenyl succinic anhydride used as an alkaline sizing agent have been presented in the previous literature search. It is the intention of this study to investigate the effect of shear on the retention of ASA, with and without the addition of alum. To accomplish this objective, the Dynamic Drainage Jar will be utilized to subject a standard furnish containing ASA size to four different levels of shear. Hercules size determinations will be conducted on handsheets made from the contents of the jar. The Hercules size tests will determine the amount of ASA retained.

## EXPERIMENTAL DESIGN

### Objectives

This work has two primary goals. These are to experimentally establish a relationship between the effect of shear on the retention of ASA and to determine if and how the addition of alum effects the retention of ASA.

### Furnish

In carrying out the experimentation necessary to accomplish the objectives, the following furnish was used:

50% bleached hardwood kraft

50% bleached softwood kraft

The furnish was made in accordance with TAPPI T-200 os-70 and refined to approximately 400 CSF.

### ASA Emulsion

The procedure utilized in the preparation of the ASA emulsion was one developed by Osorno.<sup>(19)</sup> Deionized water was used throughout the experimental work. The chemicals involved in the procedure were 0.1M HCl, 0.1M NaOH, and 1.0% alum. Cationic starch (National Cato 15) served as a retention aid for the sizing agent. The procedure involved the use of Chevron ASA and a nonionic surfactant (IGIPAL - 630). A detailed procedure including calculations can be found in the Appendix.

### Sheet Formation and Retention Analysis

500 ml of the master stock slurry, adjusted to approximately 0.3%

consistency, was placed in the Dynamic Drainage Jar. A standard procedure developed for the Dynamic Drainage Jar was followed.(20) Deviations from this procedure included addition times and the incorporation of handsheet making.

After the sample was added to the jar, 15 seconds was allowed for equilibrium to be reached. Alum was then added and the mixture was mixed for 15 seconds. The required amount of ASA was then added. The pulp, alum, and ASA was mixed 15 more seconds and 100ml was drained off. If alum was not used, the ASA was added after 15 seconds, mixed an additional 15 seconds, and drained.

The remaining sample in the jar (approximately 400ml) was divided into four samples of 100ml each. Each sample was filtered over a 7cm funnel with low vacuum on a Noble and Wood screen, cut to size. Each sheet was then pressed between a metal plate and TAPPI blotters and dried for one hour at 105°C on the metal plate. The samples were conditioned at standard temperature and relative humidity.

#### Sizing Evaluation

The sizing evaluation consisted of Hercules size determinations. Six tests were done at each condition. Handsheets from three jars at each of the following conditions were tested:

0% Alum, .25% ASA at 500, 750, 1000, and 1500 rpm

1% Alum, .25% ASA at 500, 750, 1000, and 1500 rpm

0% Alum, .40% ASA at 500, 750, 1000, and 1500 rpm

1% Alum, .40% ASA at 500, 750, 1000, and 1500 rpm

A list of runs can be found in the Appendix.

Data calculations were done using W.M.U. Dec-10 system, which calculated the mean standard deviation and coefficient of variance (see the Appendix).

## RESULTS AND DISCUSSION

The results from this study will be discussed and presented on the basis of alum addition and shear effects. Using the retention data found in the Appendix, two four-point graphs were prepared (see Figures 6 and 7).

### Alum Addition

The effect of alum addition on retention of ASA was investigated by comparing Hercules size determinations at different shear levels of a system containing no alum and one containing 1% alum. The alum was added prior to ASA addition. The data can be depicted graphically by plotting retention (expressed as HST) vs. RPM (see Figures 6 and 7).

It was found that the presence of alum resulted in much higher sizing levels in both the .4% and .25% ASA system. This corresponds with studies conducted by Gaspar, Brink and Meyer.(2,3)

### Shear

Figures 6 and 7 can also be employed in the discussion of the effect of shear on ASA retention. It can be deduced from these graphs that the retention increases with increasing shear to a certain point (1000 rpm) and then sharply decreases. This relationship was found to exist with both .25% and .4% ASA addition levels. A probable explanation of this relationship may be the breaking of the covalent bond between the cellulose and ASA molecules. A certain amount of shear may be required to break this bond causing a sharp decrease in retention of the ASA.

## CONCLUSIONS AND RECOMMENDATIONS FOR FURTHER RESEARCH

On the basis of the data collected, the following conclusions were drawn:

1. The addition of alum is needed for proper retention of alkenyl succinic anhydride.
2. The retention of alkenyl succinic anhydride (with 1% alum) increases with increasing shear until a breaking point of 1000 rpm is reached. After this point, retention is lost.

Further experimentation research is recommended in the following areas:

1. Retention studies employing the Dynamic Drainage Jar should be conducted with various alum addition levels to explore the amount needed for optimal sizing.
2. Addition sequence of alum and ASA emulsion should be studied to investigate which sequence provides the best sizing retention.
3. Zeta potential measurements could be used to verify assumptions concerning the retention mechanisms involved with alkenyl succinic anhydride.
4. Studies should be conducted on the mechanisms involved which cause the sudden decrease in retention at 1000 rpm.



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## APPENDIX

### ASA EMULSION PREPARATION

#### Water Needs

The water used throughout the experimental should be kept constant. Deionized water is recommended.

#### Stock Make-Up

The pulp used should be made following TAPPI T-200 os-70. The amount of soft and hardwood used along with the final freeness of the stock (TAPPI T-277 m-58) is of one's own choice.

#### Chemical Make-Up

The chemicals that will be used in the procedure are: 0.1 M HCl, 0.1 M NaOH, and 1.0% alum. All solutions should be made up with standard water as follows:

1. 0.1 M HCl in a 500ml volumetric flask, place 4.17ml of concentrated HCl and dilute to line with standard water.
2. 0.1 M NaOH in a 500ml volumetric flask, place 2.0g dry NaOH and dilute to line with standard water.
3. 1.0% alum in a 1000ml volumetric flask, place 10g dry alum and dilute to line with standard water.

#### Starch Make-Up

In this procedure a cationic starch, i.e. National Cato 15 is used as a retention aid for the sizing agent. The starch should be made fresh every two to three hours during the work day as follows:

1. The master batch of starch to be made up will be 700g wet weight

and 1.5% solids, so the amount of starch needed was calculated as follows:

$$700\text{g} (1.5/100)/.932 = 11.3\text{g starch}$$

where 700g is the batch size, 1.5% solids of the starch dispersion, and .9312 is the ratio of oven dry starch to air dry weight starch. Therefore, 700g total - 11.3g starch = 688.7g standard water.

2. The 11.3g of Cato 15 should then be dispersed by adding it to the 688.7g cool water while mixing mildly with a lab mixer.
3. The starch should then be cooked by double boiler for 30 minutes at 190°F while mixing mildly.
4. The dispersion should then be allowed to cook to about 90°F. "water should then be added to make-up for evaporation loss during cooking."
5. At this point, the Cato 15 is ready to use. It should be placed on a hot plate and be kept at 90-100°F with mild mixing until it is needed.

#### Preparation of the Sizing Agent/Nonionic Surfactant Emulsion

1. Tare the emulsification container on an analytical balance. The nonionic surfactant is to comprise of 7.0% of the total emulsion weight.

x = emulsion weight

y = amount of nonionic surfactant

z = amount of sizing agent

$$(X) \times 0.07 = y$$

$$x - y = z$$

2. Add the ASA (sizing agent) to make-up 93% of the total weight. "Add nitrogen gas to purge ASA container before reclosing."
3. Place the container in lab shaker for 20 minutes to emulsify the system.

The emulsion is now ready to use. "Each time a container is opened it must be flushed with nitrogen."

#### Preparation of the ASA/Starch Emulsion

1. Dilute the Cato 15 down to the desired solids as follows:

- A. Calculate volume of Cato 15 at 1.5% needed.

$$V = 1800y/1.5$$

V = Volume of 1.5% solids Cato 15

y = solids of Cato 15 desired

Take this volume "V" and dilute it to a total of 1800g with standard water at 100°F.

- B. Calculation of starch add-on level\*

$$z = 1.2x/y$$

z = volume of y% solids Cato 15/sheet

x = add-on level of Cato 15

y = solids of diluted Cato 15

- C. Calculation of sheets/starch charge the emulsifier must have 1800g starch in it so one must calculate the sheets per charge at your add-on level to calculate the amount of ASA to add to the starch.

$$S = 1800/z$$

S = sheets per charge

z = volume of y% solids starch/sheet

- D. Place this diluted Cato 15 in the emulsifier.

2. Calculation of ASA add-on level\*

$$1.2 \times A/B = C$$

C = g ASA/sheet

A = add-on level

B = % ASA

3. Calculation of ASA charge

$$D = S \times C$$

D = g ASA/charge

S = sheets/charge

C = g ASA/sheet

4. Emulsification of ASA/starch mixture of ASA.

- A. Weight the amount "D" out in a syringe. "Remembering to flush ASA container with nitrogen."  
 B. Turn emulsifier on at speed seven.

- C. Add ASA to starch vortex while syringe is clamped in place, over a period of 45 seconds. After two minutes of mixing turn the emulsifier down to speed five, and read the pH of the emulsion. Use the 0.1 M HCl or 0.1 M NaOH to adjust the pH to 7.6.
- D. The emulsion is now ready to use and must be added to the prepared stock within a half an hour.

\*All calculations are based on 1.2g OD sheets.

Sample Calculations for 0.4% ASA, 1% Starch

- Consistency of Stock: 0.29%  
 $.29/100 \times X/500$   
 1.45g OD fiber in 500ml
- Sizing Agent/Nonionic Surfactant Emulsion  
 wt. of surfactant = 1.41  
 wt. of ASA = 20.000  
 % ASA =  $20.000/21.41 = 93.4\%$
- Volume of Cato 15 Calculations  
 $V = 1800(.05)/1.5 = 60g$  to dilute to 1800g
- Calculation of Starch Add-On Level  
 $(1.45)(1)/.05 = 29.0g/sheet$   
 $S = 1800/29.0 = 62.1$  sheets/charge
- Calculation of ASA Charge  
 $.0058/.934 \times 62.1 = .386g$  ASA

## DATA SHEET

0.25% ASA, 0.0% Alum

RPM	HST (Sec.)	Std. Dev.	% Variance
500	496	59.1	11.9
750	400	80.2	20.0
1000	334	52.6	15.7
1500	160	28.2	17.6

0.25% ASA, 1.0% Alum

RPM	HST (Sec.)	Std. Dev.	% Variance
500	144	36.0	25.0
750	478	117.8	24.6
1000	920	134	14.5
1500	78	15.9	20.4

0.40% ASA, 0.0% Alum

RPM	HST (Sec.)	Std. Dev.	% Variance
500	675	109.5	16.2
750	888.	122.2	13.8
1000	704	110.7	15.7
1500	679	90.8	13.4

0.40% ASA, 1.0% Alum

RPM	HST (Sec.)	Std. Dev.	% Variance
500	1170	219.2	18.7
750	1512	183.3	12.1
1000	2318	312.9	13.5
1500	1377	184.3	13.4

Figure 6 Retention of 0.25% ASA vs. Shear

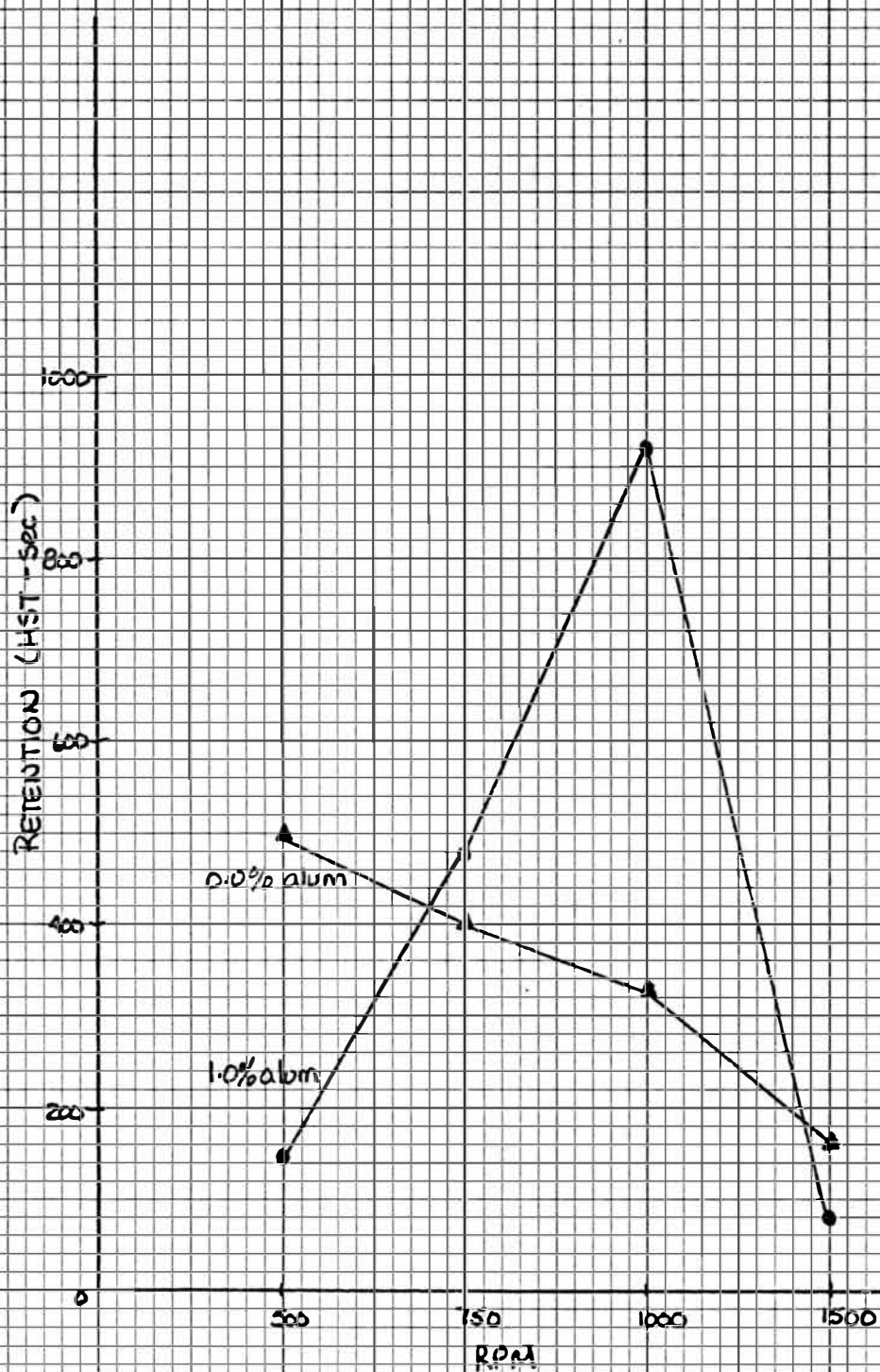




Figure 7 Retention of 0.4% ASA vs: Shear

