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The Effects of Molecular Weight of Cationic Retention Aids and Turbulence on the Retention of Organic Fines

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THE EFFECTS OF MOLECULAR WEIGHT OF CATIONIC RETENTION AIDS AND TURBULENCE ON THE RETENTION OF ORGANIC FINES

by

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ABSTRACT

The retention of organic fines is of major importance to the paper industry. Fines have been found to increase Mullen and tensile strengths. Increased retention, though, may adversely affect drainage. Fines also adversely affect the environment when not retained in the sheet. They increase the turbidity of receiving waters and otherwise affect the bottom fauna because of settling. In receiving waters they are a BOD problem, as well as a source of color.

The dynamic drainage jar was used to test the retention of fines involving only colloidal forces. Three degrees of turbulence were used, 500 rpm, 1000 rpm, and 1500 rpm. This range of turbulence is believed to cover the range of turbulence of commercial paper machines. Five cationic retention aids of similar chemical structure with varying molecular weight were used in five separate runs. A run without any retention aid gave the base for this study.

Retention appears to be linear with respect to turbulence. Retention dropped off, being close to the blank run, at 1500 rpm. The corresponding turbulence of a paper machine must be known to select the best retention aid. The length of time the retention aid is in contact with the stock solution may also affect retention. The higher molecular weight retention aid usually, but not always, has the greater retention at any given turbulence.

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INTRODUCTION

The interest in economy and water pollution have put increasing importance on retaining materials on the forming wire of paper machines. It was not until about twenty years ago that these two factors became important. Because of this, little work prior to the mid 1950's was concerned with the mechanisms of retention. As the cost of papermaking increased, the traditional system was beginning to be looked at critically. The loss of a substantial amount of material through the wire was literally pouring money down the drain. This also had secondary effects. If any recirculation was used in the system, a build-up of unretained material would occur. This would slow down grade changes because equilibrium must be reached. Since some of these unretained materials are organic in nature, they exert a biochemical oxygen demand (BOD) on the receiving water. These organic materials must be treated to comply with state and federal pollution standards. This treatment is costly.

The ultimate goal would be to operate paper machines at 100% retention (0% loss through the wire). It would have many economic advantages. There would not be any wasted material being sent to the sewer. There would not be any build-up of materials in the system so equilibrium is reached almost immediately and grade changes can be made quickly. But as the paper industry nears this goal, other adverse effects occur such as a decrease in the drainage rate.

THEORETICAL DISCUSSION

DEFINITION OF FINES

Fines have been defined qualitatively and quantitatively. The following are some of the ways fines have been defined. Han and Chang (1) define fines as fragmentary materials produced in conventional pulps mostly by mechanical treatment but partially by chemical treatment. Fines from softwood are mainly debris from fibrous materials. Fines from hardwood are largely non-fibrous materials such as ray cells and vessel segments. Because fines vary in shape and size as well as their morphological origins, they should be regarded with discrimination in regard to their behavior. Kibblewhite (2) states that fines material includes fiber remnants, fibrillar elements, linings of pit cavities, and granular debris detached from fiber surfaces. Richardson (3) uses an experimental approach to define fines as any fiber which would pass through a 150 or 200 mesh screen. For this thesis organic fines will be defined as the portion of material, inherent only from the pulp used, that will pass through a certain size mesh screen. This definition is a combination of the aforementioned definitions that excludes added materials such as fillers.

PURPOSES FOR RETENTION

Cost and Paper Properties

A number of investigations have given reasons for increased retention. These reasons cover the range from improvement in paper properties to the decrease in cost. Frankle and Sheridan

(4) experimented with one-pass retention and retention aids. They decided increased retention decreased material costs and gave better formation and drainage. The most profitable way to obtain the desired one-pass retention should be used. For material losses, the cost of the retention aid must be less than the cost of the extra material that is now retained. The cost difference should be minimized, not just the cost of the lost material.

Martin and Alexander (5) showed fines increase tensile strength. They believed either fines or possibly a water-soluble or colloidally dispersed portion of the pulp enhance the bonding between fibers. Thode and Ingmanson (6) found fines to be responsible for a major portion of the z-tensile strength of the sheet. This property resists "picking" out of individual fibers from the surface of the paper. In this case fines did not affect regular tensile but may reduce burst because of reduced stretch. The fines portion did contribute to high folding resistance. Even though fines do increase some strength properties, the surface area of fines is less useful than the surface area of whole fibers in the production of any strength property. As stated it would be uneconomical to discard the fines. Richardson (3) found fines to increase Mullen and tensile. The smaller the particle size of the fines, the greater was the strength improvement. The fines can contribute to the formation of a stronger fiber-to-fiber bond. Kibblewhite (2) found the entanglement of fibrillar fines to increase frictional forces between fibers which must improve wet web strength and extensibility. Fiber remnants have little effect on drainage or wet web behavior.

Kibblewhite (7) suggests fines retention can be used to give more flexibility to attain the properties required for the sheet. If a certain property can only be attained with more beating, then there will be more fines in the system. This is because beating produces fines. Williams (8) states that maximum retention of fines must be achieved with a minimum of chemical build-up. Optimum retention is of economic importance for the recovery of materials cost, improvement of paper properties, and water reuse. Han and Chang (1) found that the retention of fines is important also for the retention of fillers. Pigment particles may tend to be bound to fines more than to fibers for the same mass. In such a case, the retention of pigment is governed largely by that of the fines. Any means of promoting fines flocculation will enhance pigment retention in the sheet.

Environment

The following statements involve the effect of unretained fines on the water environment and reasons for retaining as much of the fines as possible. Britt (9) states that the loss of fines to effluent water from a mill constitutes a major problem in the paper industry. Williams and Swanson (10) state that fines are one of the major sources of organic stream pollution in paper mill effluent. Martin-Lof and his associates (11) found fines to increase the amount of suspended material in the waste stream. They increase the turbidity of receiving waters and otherwise affect the bottom fauna because of settling. In receiving waters they are a BOD problem, as well as a source of color. The problems of fines will be intensified if closed systems for the papermaking process are used. Britt (12) found when one-pass retention is low, it often happens that the concentration of fines in the headbox is many times the concentration of fines in the paper. This causes the following problems. On startup the first paper through will not be in equilibrium; poor drainage and felt performance; and high load on savealls and the recovery system. Tifft (13) found that selection of the best retention aid or combination of retention aids is a major factor in control of white water system losses.

ADVERSE EFFECTS OF RETENTION

Kibblewhite (2) stated fibrillated fibers and associated fines increase fiber hydrodynamic specific volumes and therefore increase the incidence of interfiber contact in pulp suspensions. This affects formation. Under suitable conditions, this can be expected to initiate floc formation which lowers both wet web and paper tensile properties. Britt (9) found that high retention may give undesirable side effects such as poor formation, inefficient utilization of pigment, and drainage problems. Richardson (3) believes that while the fines did add something to the strength of a sheet of paper, the adverse effect on drainage more than offsets any benefit. Wrist (14) also found that high amounts of fines may cause filtration problems.

MECHANISM OF RETENTION WITHOUT RETENTION AIDS

Physical

Two general and overlapping mechanisms for retention of fines without the use of retention aids involve physical and chemical interactions. The physical aspect will be discussed first. One of the first papers written about retention was by Haslam and Steele (15) in 1936. They stated that the physical aspect could be divided into filtration and entrapment. Filtration is the process of removing particles larger than the pore openings during sheet formation. Entrapment is the physical collection of particles in the fiber lumens or in the fibril structure on the fiber surface. The main interest in retention came approximately twenty years later and has continued on up to the present. For filtration Abrams (16) found that the openings of a wire in a paper machine must be covered with at least a thin network of fibers before fines can be retained. Williams (8) also found these two mechanisms for retention but he used the word sieving instead of filtration. Han and Chang (1) grouped this section under the term of hydrodynamic forces. Hydrodynamic forces involve the motion of particles, their collisions with one another, and their interception by fibers. Brownian motion or diffusion causes the collisions of small particles (less than 0.1u) at low flow velocities. Larger particles (less than 1u) are retained by direct interception and at high velocities by inertial impaction. They also state that retention of fines in fiber mats is a dynamic phenomenon because motion and force are involved. It is also a statistical phenomenon because of the large number of particles involved. Estridge (17) agrees with the preceding by stating that fines retention is based on probability accounting for retention by surface adsorption as well as by direct mechanical interception. Britt (9) found that fines can be retained by mechanical entrapment because of hydromechanical forces. These forces need not involve colloidal forces of attraction between the fines and the forming web. This mechanical retention is affected by many conditions such as sheet weight, furnish composition, machine speed, and forming section design. Fines are retained better from paper stock containing very long fibers. Tifft (13) agrees with the others when he states that many factors affect wire retention including basis weight, machine speed, and furnish characteristics. In his experiment a six percentage gain in retention of filler was obtained for every ten pound increase in basis weight in the range studied. He also found that longer fibers increase retention. Han (18) also worked with filtration and entrapment. He states that small particles are subjected to Brownian motion when suspended in a fluid. This will cause collisions, but whether or not the particles attach to each other depends on the forces operating at the site of collision. The greater the number of fines, the greater the number of fines that will be retained by the fiber mat. Retention of small particles in fiber mats is dependent on the particle, fiber and fluid properties, the flow pattern in the porous structures, and the ionic conditions in the suspension. Work by Underhay (19) has shown that because of filtration, the top surface of the paper will have a greater proportion of short and fine material and a finer and closer structure. There will be a greater loss of fines in dilute stocks which indicates statistics is important in retention of fines. Parker and Mih (20) also found fines to be retained to a greater extent near the felt side of the paper. Because of differences in static and dynamic conditions this may not be true for handsheets.

Colloidal Forces

The chemical aspect is usually termed colloidal forces. Again Haslam and Steele (15) were working on this decades before others. The colloidal force they found to be important in retention was coflocculation. Coflocculation is the interaction of the interfacial forces of the particles which control ordinary colloidal flocculation. Williams (8) found coflocculation to be the dominant colloidal force for the retention of fines. A secondary colloidal force is coagulation. Fines have a negatively charged surface surrounded by a positive counter ion layer that produces repulsion. If the repulsive forces are strong enough, the particles (fines and fillers) will not coagulate upon collision. If the forces are not strong enough, the particles will coagulate due to London-Van der Waal's forces. Retention of fines is a surface charge controlled phenomenon. McKenzie (21) states three factors affecting coflocculation. The first one is the collision process controlled mainly by the extent of agitation and by the concentration and size of the particles. The second is aggregate formation. The third is the overall strength of the aggregate. Britt (9) also found the colloidal forces of attraction between the fines and the forming mat or particles of the mat to be important for retention. He states that retention, uniformity of formation, and electrokinetic charge are related.

Han and Chang (1) did extensive work on colloidal forces. They report the following. Cellulosic fibers have very weak negative electrostatic charges in pure water. Organic fines may have the same forces. These charges are insufficient to keep them apart from each other or from fibers on collision. If the fiber system is contaminated, some ions tend to adsorb preferentially onto the surfaces of hydrophobic particles. This produces a diffuse layer of opposite ions surrounding the charged particles by virtue of coulombic forces. Certain molecules adsorbed on the particles may also cause repulsive forces as with ions. In the absence of repulsive forces the attachment of any two particles is dependent on the net result of hydrodynamic, colloidal, and molecular forces at the site of collision. These are functions of the distance of separation. Britt (22) found that colloidal attractive forces are affected by a variety of chemical factors usually related to the presence of non-fibrous additives in the furnish.

Paper Machine

Britt (9) found retention to be greatly affected by turbulence or hydralic shear existing during sheet formation. Frankle and Sheridan (4) found three main machine factors that influence retention. They are the machine itself, headbox chemistry, and the type of paper being produced. The mechanical action of the machine, specifically the drainage elements, affect retention. In the absence of chemical additives, retention can only be changed by major equipment changes. Estridge (17) gives more details about the effect of the drainage elements. On a paper machine, the forming mat is disrupted by water being driven back through the wire from the table roll. This incurs a greater chance for more fines to be lost. Paper machine loss is greater than handsheet loss because of this and the dynamic forces involved with the paper machine. Both Tifft (13) and Smith (23) found a decrease in retention as the speed of the paper machine is increased.

RETENTION AID MECHANISMS

Surface Charge

Britt (9) states that dispersions of pulp in water show a negative zeta potential of considerable magnitude. He found the same factors that reduce zeta potential promote flocculation and retention. Electrical potential may be the predominant factor in retention but it is not necessary to neutralize the original negative charge of the system in order for flocculation or retention to occur. One model for this phenomenon is given by the following. The negative charge on the surface attracts and adsorbs cationic polymers. These polymers, even though they have not neutralized the negative charge, are still capable of forming strong connecting bridges between nearby surfaces. This means charge neutralization is not directly related to retention of fines. The maximum retention for cationic polymers was found to be reached before the isoelectric point (zero zeta potential). Williams (8) found coflocculation to be increased by the collapse or compression of the electric double layers of the particles.

This phenomenon is called coagulation. Trivalent cations are used to reduce the repulsion by compressing the diffuse layer of counter ions by increasing the concentration of counter ions. The Schulze-Hardy rule states that the concentration of counter ion required to eliminate a net repulsion and achieve rapid coagulation is proportional to the inverse sixth power of its valence. Thus it only takes 0.0014 times the concentration of trivalent cations compared to monovalent cations to achieve the same coagulation. Alum is a common retention aid in this area. Alum has a disadvantage in that it is limited to a certain pH range. Britt (12) uses a term, soft floc, for this type of retention. A soft floc is one that can be broken up under turbulence and will reform once the turbulence is decreased. Britt and Unbehend (22) believe the soft floc is caused by electrokinetic forces without the formation of bridging by long chain polymers.

Bridging

The second method of retention is termed bridging. Others call it flocculation. Bridging is the development of polymer bridges between particles. It is achieved by the adsorption of a polymer molecule in solution onto the surfaces of two or more particles, thus bridging them into a network. This is sensitive to polymer concentration. Too low of a concentration will form few bridges. Too much polymer causes a stable dispersion. This is important because unretained polymer that is recycled may increase the concentration to the detriment of retention. These bridges can be broken by severe agitation. Britt (9) has found that bridging between particles appears to be necessary for good retention under dynamic conditions. Goossens and Luner (24) have divided bridging into two groups. The first type of bridging occurs when two particles are connected by the ends of a polymer chain. The second type involves bridging through the loops and tails of the polymer.

Retention Aids

The following is a discussion of some of the main types of retention aids. Britt (9) states that short chained cationics, cationic starch, alum, and all anionics give soft flocculation. High molecular weight anionic polymers have given good results. The mechanism for anionic polymers is not well understood. Two possible mechanisms have been discussed previously, charge attraction and bridging. Anionic polymers give better formation than cationic polymers. In the case of retention aids, two systems having the same zeta potential may be flocculated to different extents. Williams (8) states that polymers must be suited to the system. Anionic polymers require alum to reduce or perhaps to locally reduce the usual negative charge of the particle surfaces in order to adsorb. This limits anionic polymers to the alum pH range. Cationic polymers can be used over a greater pH range. Nonionic polymers can be used with an electrolyte.

DYNAMIC DRAINAGE JAR

Because the dynamic drainage jar will be used for this thesis, it is important to know something about it. The CF dynamic drainage jar was developed at State University of New York, College of Environmental Science and Forestry, in 1973.

Britt and Unbehend (22) state that the dynamic drainage jar determines the relative tendency of the fines fraction to pass through the screen with the fluid phase or to remain adsorbed as part of the solid phase. The total fines portion must be known. Britt (9) believes any testing device of this nature must simulate sheet formation conditions. Sheet formation takes place under conditions of both turbulence and high rates of hydraulic shear. Turbulence must be included because colloidal behavior can be different under static and dynamic conditions. The dynamic drainage jar produces a screened filtrate of white water from a stock sample at controlled degrees of turbulence. The range of turbulence from 500 to 1500 rpm is believed to cover the range of turbulence of commercial paper machines. Williams and Swanson (10) also found retention to be sensitive to agitation. Britt (9) found that testing under turbulence is not as precise as testing under static conditions. The dynamic drainage jar only studies the effect of colloidal factors, not hydromechanical factors since a mat is not permitted to form. The effect of turbulence can be studied as an independent variable. Frankle and Sheridan (4) have found the dynamic drainage jar to correlate with machine retention. Britt (12) found that a system of pulp, filler, and distilled water above 1000 rpm will give essentially 0% retention of fines and filler, and essentially 100% retention of true fiber.

STATEMENT OF PURPOSE

Several researchers have done work on the colloidal forces involved during retention. Few of these researchers, though, have used the dynamic drainage jar as the testing device. Commercial cationic retention aids of varying molecular weight will be used in the dynamic drainage jar. Thus, only the colloidal forces involved will be examined. The retention of organic fines will be run at different degrees of turbulence believed to cover the range of most commercial paper machines. The pulp used will be of commercial quality. The absence of filler differs with work done by others. Filler will be left out to reduce the number of variables affecting the retention of the organic fines. Statistically sound results will be obtained by repetition of the runs and use of the t-distribution.

EXPERIMENTAL PROCEDURE

PULP

The pulp used was a 50-50 mixture of bleached hardwood kraft and bleached softwood kraft. The softwood was put through a Wiley mill twice using the fine screen. The hardwood was put through a Wiley mill once using the fine screen. The pulp was refined in a Valley beater to 350 Canadian Standard Freeness. The desired amount of fines was 20% to 30% of the total pulp.

FINES FRACTIONATION

The procedure for fines fractionation is obtained from The CF Dynamic Drainage Jar for Paper Stock Testing Information Manual. The following is a brief description of the procedure. A 500 ml stock sample at 0.1% consistency is placed in the drainage jar at 750 rpm. It is allowed to drain. It is then washed four times with 500 ml of wash water containing equal parts of TAMAL 850, tripolyphosphate, and sodium carbonate, each at 0.05% concentration. The residue of fiber on the screen is transferred to weighed filter paper, dried, and weighed. The total weight of the sample minus the weight of the fiber is the weight of the fines. The percent fines is the weight of the fines divided by the total weight of the sample multiplied by 100. The screen used contained 14.5% open area from 76.2 micron diameter holes.

RETENTION AIDS

The following table lists the retention aids used, their structure and molecular weight, and the suggested loading rate along with the rate actually used. The loading rates are based on the number of pounds of active retention aid per ton of dry pulp. The retention aids were prepared according to the manufacturer's directions.

TABLE 1

Retention Aid	Manufacturer	Structure
Delfloc 50	Hercules	Polyamide
Reten 304	Hercules	Polyamide
Cartaretin F-8	Sandoz	Polyamide
Separan CP-7	Dow Chemical	Polyacrylamide
Percol 292	Allied Colloids	Polyacrylamide

Retention Aid	Mol. Weight	Sug. L.R.	Applied
Delfloc 50	$<$ 100,000	0.2-2.0	1
Reten 304	$\sim$ 100,000	0.5-1.5	1
Cartaretin F-8	$\sim$ 250,000	4 - 12	8
Separan CP-7	$\sim$ 1,000,000	0.2-0.75	0.5
Percol 292	$>$ 1,000,000	0.1-0.5	0.3

RETENTION RUN PROCEDURE

The retention run procedure was slightly modified from the one given in <u>The CF Dynamic Drainage Jar for Paper Stock Testing Informational Manual</u>. The following were the steps of the modified procedure. Prepare the stock sample (about 500 ml at 0.3% consistency). Prepare the retention aid. Add the stock sample and stir at 300 rpm. Add the retention aid with syringe or pipette and wait 20 seconds (use a timer.) Turn the mixer up to the desired speed. Take the pinch clamp off and remove 100 ml. Replace the pinch clamp and dump the 100 ml back into the jar. Remove the pinch clamp and remove 50 ml. Do not replace the pinch clamp after the preceding step. Collect about 50 ml in a pre-weighed weighing jar. Weigh the jar. This will give the weight of the filtrate. Evaporate the filtrate to dryness. Let the jar cool. Weigh it. This will give the weight of the fines. The percent of the fines retained is calculated by the following equation. % retained = 100 - (100A)/(BCD), where A is the weight of the dried fines, B is the weight of the filtrate, C is the consistency of the stock sample, and D is the fines fraction of the stock sample.

PRESENTATION OF RESULTS

The amount of fines in the stock sample used was about 27.3% of the total pulp. The precision of this value is not important in showing relative trends as long as its true value stays constant. The following table gives the results of the retention runs. The 90% confidence limits are presented as the mean of the three runs and plus and minus half the confidence limit. Figure 1 on the following page presents the data from Table 2 in the form of a graph.

TABLE 2

	90% Confidence Limits		
Retention Run	500 rpm	1000 rpm	1500 rpm
No Retention Aid	45.8±3.2	38.6±3.0	32.8±1.9
Delfloc 50	69.5±2.7	51.0±2.4	33.8±0.7
Reten 304	68.7±4.3	52.9±6.7	35.6±3.3
Cartaretin F-8	64.7±1.6	48.4±7.0	28.6±0.8
Separan CP-7	72.9±3.7	51.6±5.5	38.1±3.6
Percol 292	84.2±1.8	60.7±5.4	35.6±2.3

GRAPH OF PERCENT RETENTION OF FINES VS TURBULENCE

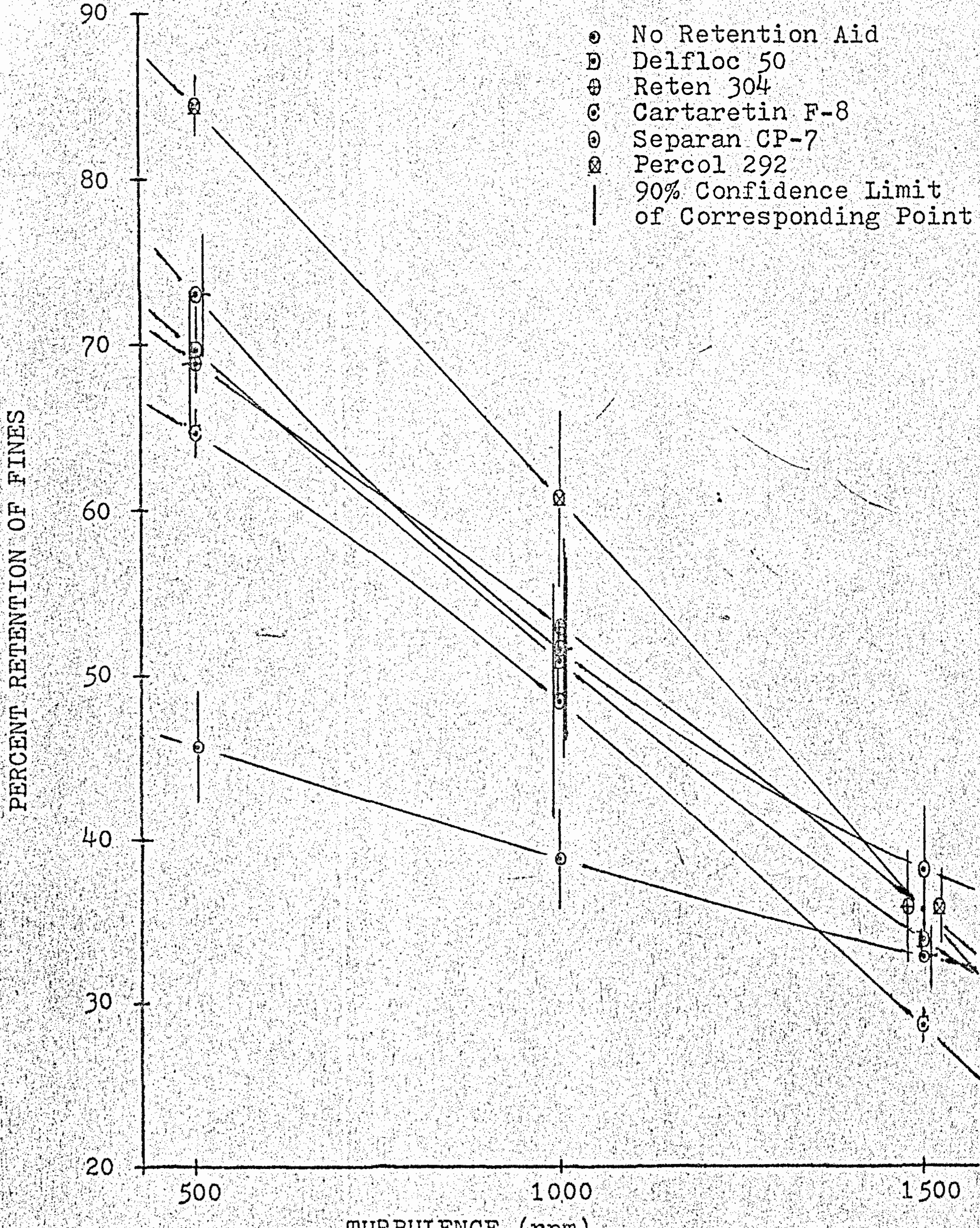

FIGURE 1

DISCUSSION OF RESULTS

The retention of fines in the run with no retention aid is dependent on both physical and colloidal forces. The physical forces involved are hydrodynamic forces including the motion of the fines, their collisions, and their interception or entrapment with fibers. Filtration is excluded because a mat is not allowed to form. The main colloidal force involved is coflocculation. It deals with the interaction of the interfacial forces and surface adsorption. All of these mentioned forces are important. They decreased slightly with increased turbulence (see Figure 1) and form the base for retention with retention aids. This base appears to be linear with respect to turbulence.

Retention using cationic retention aids involves two more methods, bridging and reduction of surface charge. Bridging is the stronger of the two and is more important at medium to high turbulence. Reduction of surface charge is more important at low turbulence. It is hard to distinguish what fraction of the retention is due to either method at low turbulence. Retention in each case is almost linear with respect to turbulence. The manner in which the retention aid was added may have caused the shape of the graph. The retention aid was in contact with the stock exactly 20 seconds before the mixer was set at the desired speed. This length of time may not have been long enough for bridging to occur to its fullest extent. The higher molecular weight retention aid usually, but not always, has the greater retention at any given turbulence. The surface charge on the lower molecular weight retention aids appears to be the deciding factor at low turbulence. The three polyamide retention aids have different molecular weights, yet the retention was opposite of what was expected due to bridging. The molecular shape, and not the molecular weight, may be the dominant factor at higher turbulence. At very high turbulence, the highest molecular weight retention aids gave better retention, although it was only slightly higher than the run without any retention aid.

Considering this data, retention aids may not be economical at high turbulence levels. In the long run it may be less expensive to make equipment changes to reduce the turbulence than to add retention aids. The corresponding turbulence must be known for a particular paper machine to use the data. This is because different retention aids are best at different turbulence. Cost must also be considered. Turbulence is definitely an important factor to be considered when using retention aids.

CONCLUSIONS

Retention appears to be linear with respect to turbulence. The corresponding turbulence of a paper machine must be known to select the best retention aid. The surface charges of the retention aid is important for retention at low turbulence. The size, shape, and chemical structure of the retention aid is important for retention at greater turbulence, depending on which aspect will give the stronger bridges.

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