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A Study of the Staining Techniques of Secondary Fibers for the Qualitative Analysis of Contaminants

Susan M. Soltis
Western Michigan University

A STUDY OF THE STAINING TECHNIQUES OF SECONDARY FIBERS FOR THE QUALITATIVE ANALYSIS OF CONTAMINANTS

By:

Susan M. Soltis

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ABSTRACT

Identification of the contaminants present in secondary fibers would aid in the reduction of the stickies problem common to the secondary fiber industry. Presently, identification and quantitative analysis of stickies is difficult. The staining characteristics of the hotmelts and latexes were studied and used to complete a flow chart analysis of a sample to determine the type and quantity of contaminants. Dupont #4 and Superlitefast Brilliant Blue dyes were shown to stain the hotmelts. Analysis of a sample through the flow chart developed for this thesis would determine the type and quantity of contaminants present. These results may be used to better determine the effectiveness of contaminant removal by different cleaning methods used in the recycle industry.

Keywords: Hotmelts, latexes, adhesives, recycle, cleaning methods

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INTRODUCTION

A major problem common to all secondary fiber mills is stickies. These are mainly latices and plastics which contribute to visual and printing problems of the final sheet. By identifying the stickies present, their removal from recyclable materials may be better evaluated. A standard method for qualitatively analyzing the contaminants has not yet been established due to discrepancies among the present methods. Reduction of these variables will aid the industry in better evaluating the stickies problem. The aim of this thesis is to study various staining techniques and to recommend a more standardized procedure for the identification of the contaminants.

THEORETICAL BACKGROUND

Problems Caused by Stickies

The most troublesome contaminants today falls under the general classification of stickies. These materials stick to mill equipment and to surfaces of the finished paper products. These substances; primarily hotmelts, and latex adhesives; are sticky under normal mill conditions where the pulping temperature is above 150 degrees Farenheit. This causes a variety of problems within a paper operation. First, they build up in mill white water systems. They then agglomerate in piping, breaking off in chunks which cause web breaks, spots, and holes in the sheet. These agglomerates will also plug screens, paper and cylinder machine wires, and felts, thereby reducing mill capacity by increased downtime for clean-up. Press roll and dryer accumulation causes sheet picking and sticking. Many mills mount "doctor blades" to attempt to scrape the stickies from the dryers. The spots in the paper become "shiners" once calendered. The stickies in the final wound roll can cause adjacent layers of the paper to adhere if left in storage for months. Although removal of these contaminants seems like the logical solution to these problems, it is not that easy. Becuase they have a specific gravity near that of water and fibers ($.95 \pm .1$), they cannot be floated out with the use of the floatation/deinking equipment. They are too small to be screened out and too large to be washed out.(1) Dispersion will not remove them, even with prolonged agitaiton. Finally, solvent removal, although effective, is far too costly to be considered in most mill

budgets. With the increased use of recycled paper, the paper industry is going to have to find a feasible method of this contaminant removal.

Advantages of Adhesives

Although sticky contaminants are hard to remove and cause many problems, they have many advantages which has caused an increased use of these materials. They can bond a variety of surfaces, have a fast speed of a strong bond formation and have a small space requirement for on-line use. Because they dry by cooling, there is no absorption into the substrate. Due to its increased viscosity upon cooling, they are good for porous surfaces. They form a water and sometimes a grease resistant layer depending on the polymers used in the chemical formulation of the adhesive. Due to these advantages, more companies are using hotmelts and latex adhesives in their products such as frozen food packaging, book bindings, and pressure sensitive adhesive labels.

Composition of Stickies

Although the term stickies is used to refer to a general classification of adhesives, there are two main groups to be considered, hotmelts and latex adhesives. Within these groups there are basic polymers which make up the base formulations for each type of adhesive.

Hotmelts

Hotmelts are 100 percent solid formations of thermoplastic material. As stated earlier, they solidify upon cooling, and soften at normal mill pulping temperatures between 150 to 250 degrees Farenheit. They are applied between 285 to 430 degrees Farenheit as a spreadable liquid.(2) Desired properties include wettability, water resistance, tack, viscosity,

and heat stability. These properties are achieved by mixing adhesives with modifiers or copolymers. Common types of hotmelt adhesives include polyvinyl acetate, polyamides, polystyrenes, and polyethylene.(3) Polyvinyl acetate was the first used. It is made by reacting acetylene and acetic acid in the presence of a catalyst. The catalyst is an organic peroxide which initiates polymerization by a free radical mechanism thus yielding additional reactions. Polyvinyl acetate is resistant to ink attack, improves ink transfer, and has fair flexibility and strength. It is used mainly for book binding and frozen food packaging.(4) Polyamides are made by dimerizing fatty acids and reacting them with diamines. Due to their linear properties, they have good oil resistance and strength with flexibility. They are used to bond aluminum foil paper, and for packaging food packages. Polystyrene is formed by the catalytic dehydrogenation of ethyl benzene. It has good resistance to salts, organic acids, and lower alcohols. It is a brittle to fair adhesive. Polyethylenes are not very common since they have poor adhesion, but they have good wettability and are used for coating of grease and water resistant materials.(3) The bonds formed by these adhesives are mainly physical but with the right modifier, chemical bonding can result.

Latex Adhesives

A latex is synthetic resin emulsion made of a dispersion of very small water insoluble particles held in aqueous suspension by a balance of surface active agents.(5) There are three main polymers which are styrene butadiene, polyvinyl acetate, and acrylic latex. Styrene butadiene consists of two monomers, styrene and butadiene, in a water suspension.

They are carboxylated which means a carboxyl group is added for stability that is mainly controlled by the ratio of styrene to butadiene. Styrene is a hard thermoplastic, while butadiene is a soft, flexible, elastic polymer. Therefore, this ratio also affects the strength properties of the monomer. Polyvinyl acetate was discussed earlier in the hotmelt section. Acrylic latex is a monomer of synthetic resin based on esters of acrylic and methacrylic acid. These are easily copolymerized with each other and other monomers. The low acrylic esters are soft, the medium are tacky, and the high esters are waxy. They have excellent shear stability, low odor, and are resistant to light, yellowing, heat, and chemical degradation.(5,6)

The uses for hotmelts and latex adhesives are continuing to grow and their impact on recycling is becoming a major concern. By identification of these contaminants in the recyclable materials, better removal methods can hopefully be found.

Present Identification Methods

In developing this identification method, many obstacles arise such as no common formula for hotmelt adhesives since each supplier has their own formulation, and only a small amount of written materials are available. Two basic test methods are presently being used and both have strong disadvantages. These test methods are described in the Appendix. Doshi, Dyer, and Kruegar (7) recommend fluorescent speck counting as a potentailly attractive method for the quantification of stickies. The disadvantage of this technique is "all that fluoresces does not necessarily represent stickies, and all stickies do not necessarily always fluoresce."

Due to this limitation, fluorscent speck counting will not be considered in the scope of this thesis. The "peel" test, developed by Walmsley (8), uses a pulsating screen apparatus. Stock is filtered through the screen, with the residue being washed off and filtered using a Buchner funnel. A second filter is then placed on top of the pair and dried under slight pressure. When cool, the two filters are pulled apart and sticky particles are defined as those adhering to both papers. This method has the limitation of not being able to distinguish between stickies, thermoplastics, and fibrous specks.

Smith (9) discusses various fiber identification techniques currently in use. Information concerning microscopic appearance, specific gravity, melting point, and solubility of the contaminants in a dye solution of fiber indicator is made available. He also lists colors obtained by treating different synthetic fibers in a dye solution of fiber indicator. By finding a stain for these contaminants, one would be able to use this as an identification method for contaminants.

PROCEDURE

Research began with the staining of known contaminants of two samples of hotmelts, one from Coat-it Corporation, and the other from R.R. Donnelly. This was done to obtain a control sample to determine which dye would be most effective. It was found that Crompton and Knowles' Superlitefast Brilliant Blue, and Pylam Products' Dupont #4 dyes have the most reproducible results. The actual procedure for the making of the hotmelt handsheets, dye solutions, and staining of the sheets can be found in the Appendix. The other dyes tested were C-Stain, Ciby Geiby's Solophenyl Blue dye and Atlantic's Resin Fast Blue dye. These are all water soluble dyes. The Superlitefast Brilliant Blue dye stained the blotter fibers blue and the hotmelts milky white. The Dupont #4 dye stained the blotter fibers green and the hotmelts yellow.

The bulk of the experimental research was carried out using pulp samples from the indicated points in Figure 1 from James River Corporation, paper machine #3. Samples were taken on two separate days to attempt to get a representative sample for analysis. This machine was chosen because it makes filler board which contains the most contaminants. British handsheets were then made according to the procedure outlined in TAPPI Standards.(10) These handsheets were analyzed using the flow chart in Figure 2. All test procedures can be found in the Appendix, unless otherwise indicated. The pH was determined since ASA flakes found in alkaline paper are potential stickies in the system. Oxygen ash tests were run to determine the filler

content. The Hercules Size test was used to find the amount of sizing, if any.(11) The Wet Strength Resin test indicated the presence of urea or melamine formaldehyde. The dirt count determined the amount and type of contaminants. This is a subjective test and therefore has low reproducibility. The polyvinyl acetate test indicated its presence whereas the styrene butadiene test indicated not only its presence, but that of polyvinyl acetate also. The hotmelt staining was used to indicate the degree of success of the earlier research. The stained sheets were compared with both the control sheets and unstained sheets to attempt to reduce the subjectivity of this test. All tests were performed by methods similar to those used in actual mill conditions to best simulate mill results.

FIGURE 1

Machine Flows

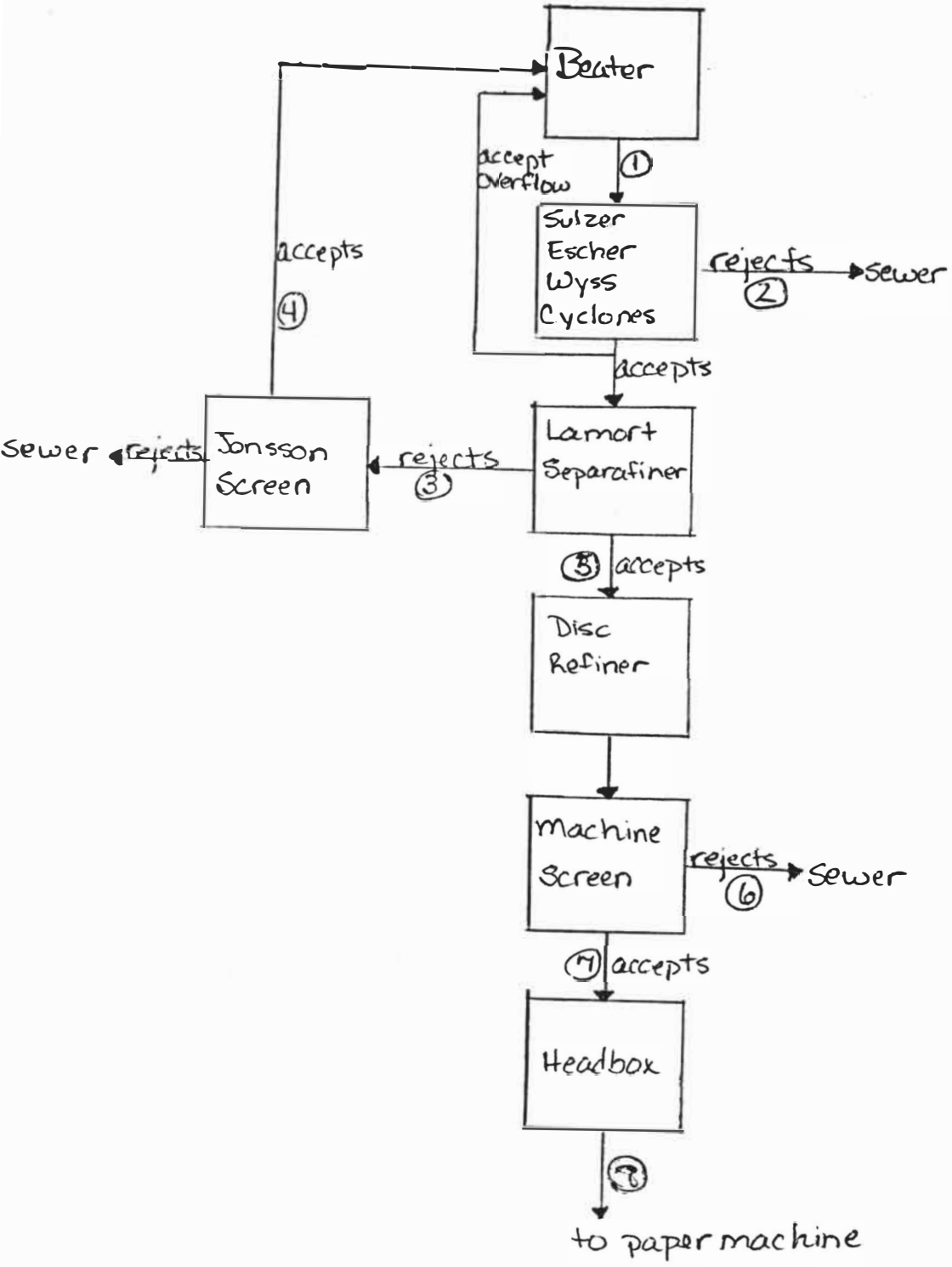


FIGURE 2

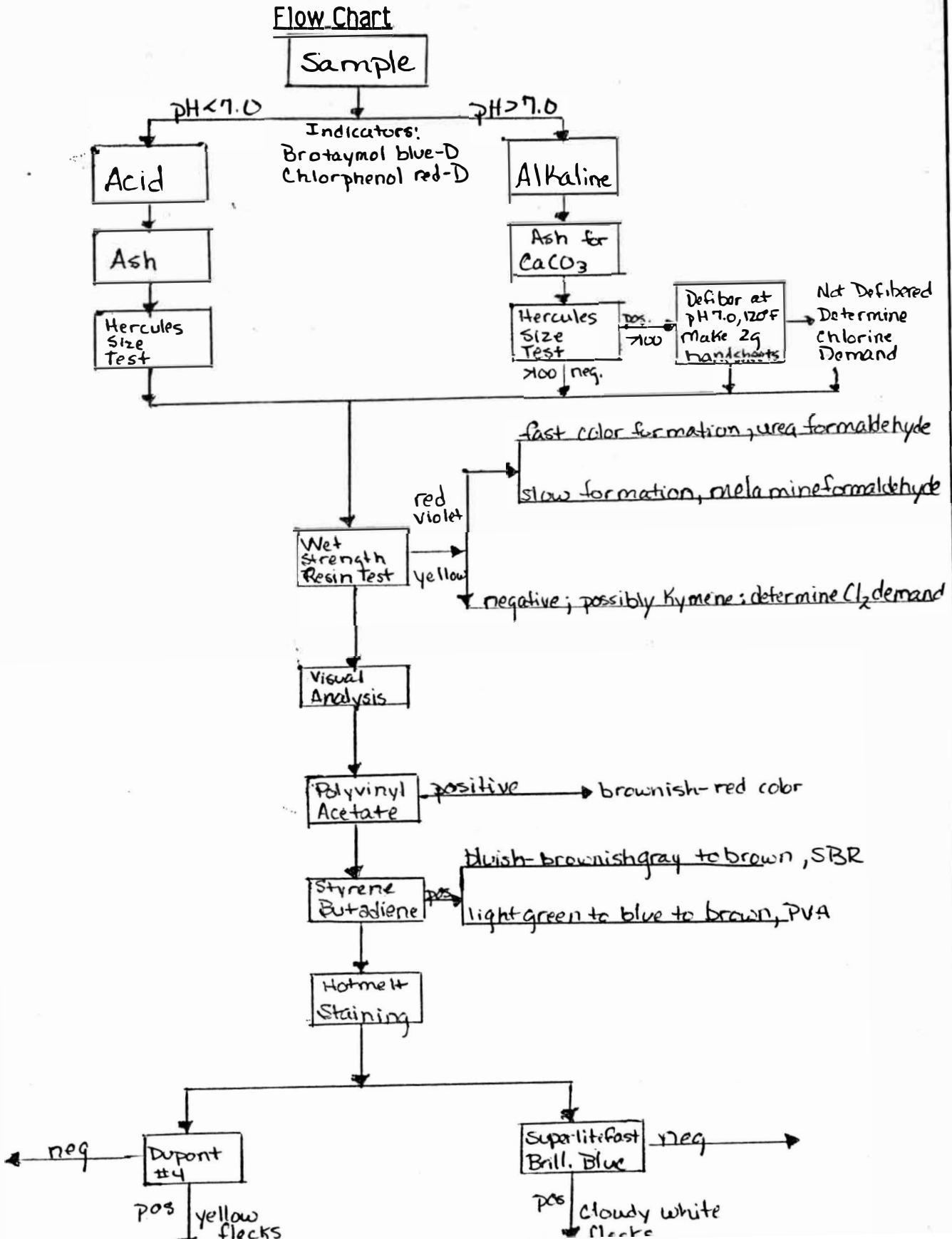

Flow Chart
Sample
pH<7.0
pH>7.0
Indicators:
Brotaymol blue-D
Chlorphenol red-D
Acid
Alkaline
Ash
Ash for $CaCO_3$
Hercules Size Test
Hercules Size Test
pos.
>100
Defiber at pH 7.0, 120°F
Make 2g handsheets
Not Defibered
Determine Chlorine Demand
>100
neg.
Wet Strength Resin Test
red violet
yellow
fast color formation, urea formaldehyde
slow formation, melamineformaldehyde
negative; possibly Kymene; determine Cl_2 demand
Visual Analysis
Polyvinyl Acetate
positive
brownish-red color
Styrene Butadiene
pos
bluish-brownishgray to brown, SBR
light green to blue to brown, PVA
Hotmelt Staining
neg
Dupont #4
pos
yellow flecks
Superlitefast Brill. Blue
neg
pos
cloudy white flecks

TABLE 1. RESULTS

	pH	Ash (%)	HST (sec)	Wet Strength	PVA	SBR	Dupont #4	Superlitefast Brilliant Blue	Dirt Count (#/64 in2)
Beater									
Day 1	6.0	3.5	0.9	neg	pos	neg	pos	pos	428
Day 2	6.2	4.9	2.1	neg	pos	neg	pos	pos	568
Escher Wyss Lt. Rejects									
Day 1	6.2	7.7	1.5	neg	neg	neg	neg	neg	312
Day 2	6.0	10.0	2.1	neg	pos	neg	neg	neg	321
Lamort Rejects									
Day 1	6.2	8.9	0.9	neg	neg	neg	pos	pos	277
Day 2	6.0	6.5	0.6	neg	neg	neg	neg	neg	339
Jonsson Accepts									
Day 1	6.0	2.7	2.0	neg	pos	neg	pos	pos	318
Day 2	6.2	9.6	1.2	neg	pos	neg	pos	pos	363
Lamort Accepts									
Day 1	6.4	5.9	1.0	neg	pos	neg	pos	pos	291
Day 2	6.0	7.9	1.1	neg	pos	neg	pos	neg	251
Machine Screen Rejects									
Day 1	6.0	4.9	0.9	neg	pos	neg	neg	neg	351
Day 2	6.2	5.8	1.2	neg	pos	pos	pos	pos	272
Machine Screen Accepts									
Day 1	6.2	10.1	2.1	neg	pos	neg	pos	pos	233
Day 2	6.2	11.4	1.6	neg	pos	pos	pos	pos	221
Headbox									
Day 1	6.2	6.6	4.4	neg	neg	pos	pos	pos	69
Day 2	6.2	7.3	1.2	neg	neg	pos	pos	pos	119

DISCUSSION

This section will divided into two parts. The first part will analyze the data from Table 1 by examining the results of each test. The second part will analyze the data according to the sample point to explain the relationships between the machinery.

Analysis by Test

The pH of the system was relatively constant at approximately 6.2 for both days of sampling and therefore the alkaline branch of the flow chart was eliminated. The rest of the analysis was completed using the acid flow line.

The ash content of each sample was analyzed to determine the filler content. It was a good representation of the filler content except in the case of the Sulzer Escher Wyss rejects. Since these contained particulate matter such as rocks and staples which do not burn off in this test, the ash test results reflected the amount of material which would not burn, not the filler content. Otherwise, the ash content is as to be expected for each sample point.

There was relatively no sizing in the sheets which was to be expected since filler board does not need to be highly sized. Because all of the size tests were under five seconds, it is correct to assume there was not any sizing in the samples.

Since the Hercules Size test illustrated there was no sizing in the sheet, it is to be expected that the wet strength resin test would be negative. This test indicates that there is no urea or melamine

formaldehyde present.

Dirt counts were completed during the visual analysis to attempt to determine the efficiencies of the individual machines. Due to lack of data, actual efficiencies could not be calculated, but trends can be observed. As expected, the beater had the highest dirt counts since all contaminants are present here, at the start of the process. In general, the reject lines had higher dirt counts than the accept lines which is sensible, because the purpose of the machinery is to remove the contaminants. The headbox had the lowest dirt count and the system removed approximately 81 percent of the dirt and contaminants based on the beater and headbox dirt counts. The visual analysis in general showed a large amount of white coating flecks, and colored paperboard to stay within the system. Figure 3 illustrates a Dirt Count Range Graph, used to show the decrease in dirt count as the stock flows to the headbox and also to indicate ranges for the dirt counts of each sample point.

The next two tests can be combined since the styrene butadiene test is also a verification of the polyvinyl acetate test. The results did verify themselves since if polyvinyl acetate was indicated in the polyvinyl acetate test, it was also indicated in the styrene butadiene test. Styrene butadiene was less prevalent than polyvinyl acetate. Polyvinyl acetate is used in the makeup of both hotmelts and latexes.

As with the styrene butadiene and polyvinyl acetate tests, the dyes were used as verification of the results to reduce the subjectivity of the hotmelt test. It is possible for one of the samples from the first day to have hotmelts present and the second day sample to be negative, but the

credibility of the test method is questioned when the results from one day at one sample point do not agree. This only occurred once with the Lamort Accepts. This could be due to many causes such as not enough samples and subjectivity of the viewer.

Machinery

The beater was the dirtiest and had the highest contaminant level of all the samples. This is because this is where the system begins and all contaminants identified later in the system, enter the system here.

The Sulzer Escher Wyss Cyclones are forward cleaners used to remove high density particles from the system. This is done first to remove contaminants which may harm the machinery down the line. These are large cylinders, with one per machine. Since this cleaner is used to remove heavy particles, the reject sample contained articles such as heavy-duty staples, rocks, and pieces of metal. This was damaging to the British handsheet wire and therefore samples were not made. The light weight rejects, taken from the top of the reject trough, were analyzed. These also contained a small amount of heavy particles such as rocks. A sample was unable to be collected from the accept line due to inaccessibility of a sample point.

The Lamort Separafiner is designed to combine deflaking and course screening in one continous operation, removing contaminants such as plastics, styrafoam, shives, rubber, adhesives, and glue.(Figure 4) Light contaminants are to be separated instead of being reduced and passed through the system. From the results of later machinery, it is evident

that the separafiner is not 100 percent efficient. The accepts from this go to the disc refiner and machine screens. The rejects go to the Jonsson screen.

The Jonsson screen is used to remove large reject particles from the system.(Figure 5) This is evident by the type of material rejected, such as bandaids, tape, brown paper towel, candy wrappers, sponge, styrafoam, string, and more. The rejects did not contain the adhesives which they were combined with, for example, the tape and bandaid had no adhesive backing attached. These rejects were not in slurry form, but in pieces, therefore this sample could not be run through the analysis. The accepts from the Jonsson screen are recirculated back to the beater to reclaim as much good fiber as possible. This sample did contain adhesives as evidenced by the positive polyvinyl acetate, styrene butadiene, and hotmelt tests.

The machine screens are designed to remove as many of the remaining contaminants as possible before the stock flows to the headbox. Although the dirt counts are relatively close, it does show that contaminants are being removed. Both accept and reject flows contain polyvinyl acetate, styrene butadiene, and hotmelts, thereby indicating that these are not being separated out. The rejects from this are sewered, and the accepts are sent to the headbox.

The headbox flow was obtained from sampling overflow of the wire. It was shown to contain hotmelts, and styrene butadiene, but the polyvinyl acetate disappeared and the dirt count dropped. It is not directly known as to where these contaminants went.

FIGURE 3

DIRT COUNT RANGE GRAPH

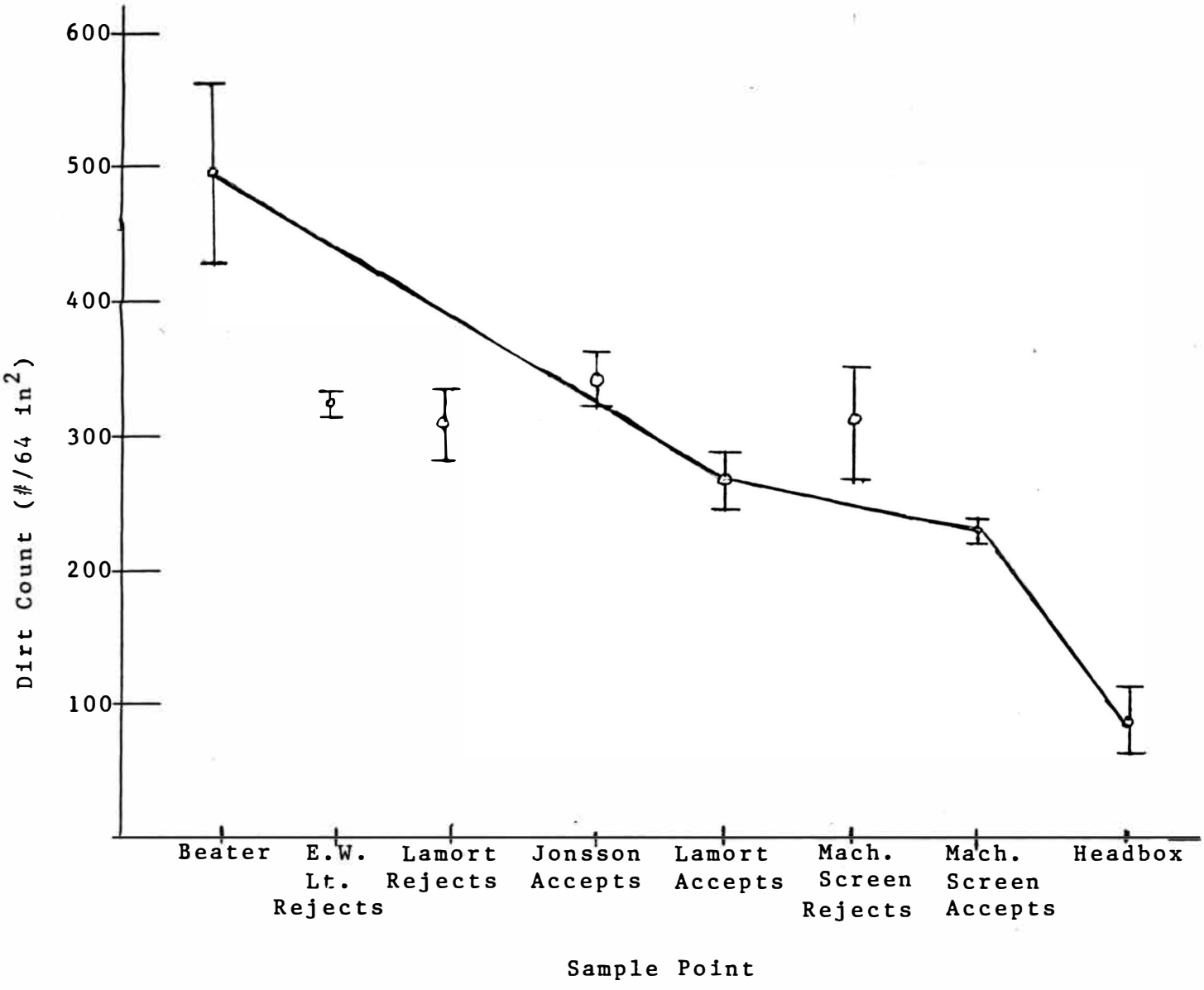
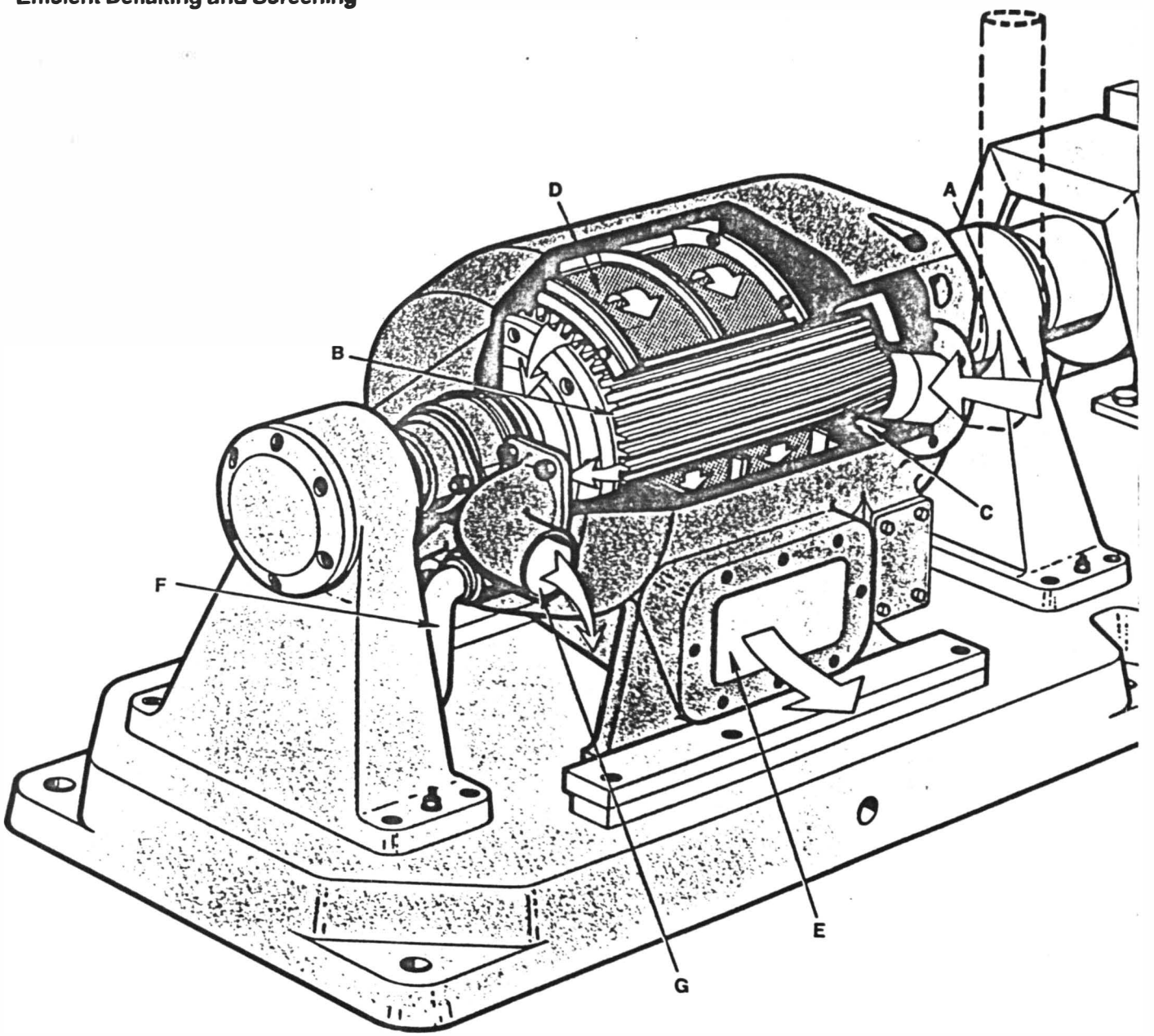


FIGURE 4

Lamort Separafiner

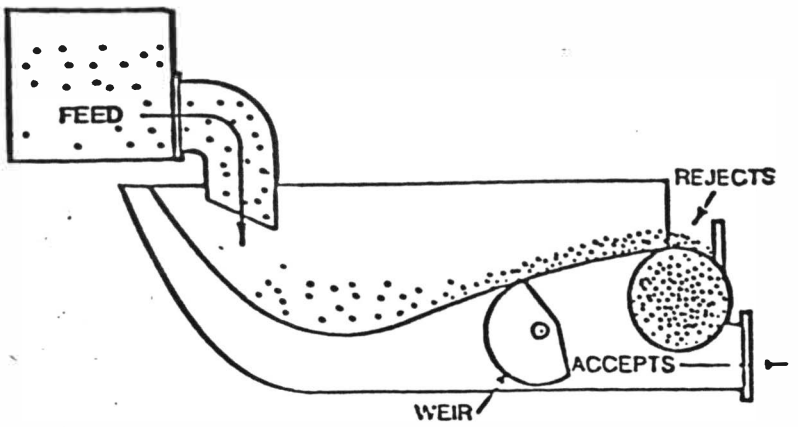
Efficient Deflaking and Screening



A. Stock from cleaner enters Separafiner.
B. Grooved stainless steel rotor disintegrates, deflakes stock.
C. Stainless steel stator contains grooves to aid deflaking.
D. Polished stainless steel screen plate contains counter-bored perforations, .157 inch (4 mm) in diameter for efficient screening.
E. Accepted stock is passed to adjustable level box mounted on Separafiner.
F. White water inlet for diluting and controlling rejects.
G. Reject outlet.

FIGURE 5

Jonsson Screen



How It Works

Stock flows into the head end of the screen where it is subjected to intense, controlled vibration while suspended over the screen plate. The plate is contoured to form a pool extending approximately 80% of the screen plate length providing more available screen plate area than comparable screens. The pool depth is controlled an adjustable weir. Throughout this section, accepts in the form of good clean fibers are assisted through the perforations by the vibratory motion while rejects are rapidly transported to the discharge end.

Rejects are in contact with the screen only at the final 20% of its length where it emerges from the pool to form a horizontal beach. At this point, a Bird Aqua-Purge Shower is used to scour off remaining good fibers that may adhere to knots or other rejects. Passage across this section is brief with little time for rejects to bounce around and break up into screenable particles.

CONCLUSIONS

(1) Hotmelts can be stained and thereby identified.

(2) A sample analysis, such as the one used here, would be effective in a mill situation.

(3) The analysis of selected sample points would lead to efficiency reports being possible on the various equipment.

RECOMMENDATIONS

(1) The dirt count range graph can be used to monitor the dirt counts over a period of time, thereby determining a range of operational dirt counts.

(2) If one of the sample points should deviated from this range, the sample analysis could be run to determine the types of contaminants.

(3) Constant monitoring of the system would lead to a clearer understanding of the true efficiency of the equipment.

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APPENDIX

Fluorescent Speck Counting

Purpose: To determine the presence of stickies.

Procedure: Filter papers from both sides of the handsheet are removed and examined under ultraviolet light. The number and size of fluorescent spots are estimated using the TAPPI Dirt Estimation Chart. A 6-W UV lamp emitting 365-nm wavelength light was used in the study.

"Peel" Test for Sticky Contaminants

Purpose: To determine the presence of stickies.

Procedure: Stock containing 100 grams O.D. fiber is added slowly to a Valley pulsating screen apparatus. Screening is continued until all loose fiber has been removed. The residue on the screen is washed off with water into a beaker. The suspension is then filtered onto filter papers using a Buchner funnel, sufficient papers being used so that the individual particles are well separated on the paper. A second filter paper is then placed on top and the "sandwhich" dried under slight pressure. When cool each pair is carefully pulled apart and sticky particles can be identified as being those adhering to both papers and showing stretch and elasticity.

pH Test

Purpose: To determine the pH of the sample.

Procedure: Place 0.025 grams of sample into the Hercules pH tester vial. Add 7 ml of distilled water. Add 7 drops of indicator. Compare the color of the sample vial to that of the control to determine the pH. Indicators used were Bromtaymol blue-D (pH range of 6.0 to 7.6), and Chlorphenol red-D (pH range of 5.2 to 6.8).

Oxygen Ash Test

Purpose: To determine the ash content.

Procedure: Enough sample was added to the tared metal crucible to fill, but not pack it. A wick of ashless filter paper was placed in the crucible, lit , and the entire apparatus placed inside the oxygen filled jar until it stopped burning. The crucible was reweighed and percent ash calculated by dividing the weight of the crucible after burning by the weight of the crucible before burning and multiplying by 100.

Visual Analysis

Purpose: To visually count and identify as many contaminants as possible.

Procedure: Eight, 1 inch by 1 inch, areas were examined using a linentester to enlarge the view. Specks greater than one-sixteenth of an inch were counted and indentified.

Wet Strength Resin Test

Purpose: To determine the presence of urea or melamine formaldehyde.

Procedure: Place the sample to be tested in a watchglass. Put 4 drops of Reagent A on the sample. Wait 30 seconds, then place 1 drop of Reagent B on the sample.

A red-violet color with fast color formation indicates urea formaldehyde, slow color formation indicates melamine formaldehyde. If the solution stays yellow, formaldehyde is not present.

Reagent A: Mix 1.34 grams of Phenol hydrazine hydrachloride with 50 grams of Sulfuric acid prepared at 41.7 grams of concentrated acid and 8.3 grams distilled water. Dilute the entire solution to 100 grams.

Reagent B: Mix 10 grams of Ferric chloride ($FeCl_3 \cdot 6H_2O$), with enough distilled water to make 100 grams of solution.

Polyvinyl Acetate Test

Purpose: To indicate the presence of polyvinyl acetate.

Procedure: Place the sample in a watchglass. Add 3 drops of the iodine solution to the sample.

A brownish-red color indicates the presence of polyvinyl acetate.

Iodine Solution: 1.06 grams of Iodine dissolved in 100 grams water.

Styrene Butadiene Test

Purpose: To indicate the presence of either styrene butadiene or polyvinyl acetate.

Procedure: Place the sample in a watchglass. Add 3 drops of dilute sulfuric acid (50% solution). Add 4 to 5 drops of acetic anhydride to the acid.

A bluish or brownish gray changing to brown indicates styrene butadiene.

A light green to blue to brown indicates polyvinyl acetate.

No color change indicates neither is present.

Hotmelt Staining Test

Purpose: To indicate the presence of hotmelts in the sheet.

Procedure: Place the sample in a watchglass. Soak the sample for 30 seconds with 100 degrees Celcius distilled water. Pour off the excess water. Add enough dye solution to cover the sample in the watchglass. Soak for one minute and rinse with room temperature distilled water. Place the sample between 2 blotter papers and press out excess water with hands. The sample was then viewed through a linentester to observe the presence of any hotmelts. The dyed sample was compared to an undyed sample and a control sample to increase the accuracy of the findings.

Dupont #4 dye produced yellow flecks of hotmelts.

Superlitefast Brilliant Blue dye produced milky white flecks of hotmelts.

Dye Solution: Mix 1 gram of dye into 100 grams of distilled water at 100 degrees Celcius.

Hotmelt Handsheets

Purpose: To determine if hotmelts can be stained.

Procedure: Heat hotmelts (5 grams) to 350 degrees Farenheit. Using a drawdown board and course Meyer rod, place a thin coating of hotmelt of a blotter paper and then place another blotter paper on top. Dry overnight, then place one sandwhich into the Waring Blender for 2.5 minutes with 2 liters of water at 35 degrees Celcius. Next, make handsheets using the Noble and Wood apparatus. Press the sheet and leave to air dry overnight. Place the sample in a watchglass, and soak in 100 degree Celcius distilled water for 30 seconds. Pour off the excess water and pour on the dye solution. Soak for one minute, then rinse with distilled water. Blot dry.

Dyes used were Dupont #4, Superlitefast Brilliant Blue, C-Stain, Solophenyl Blue, and Resin Fast Blue. These are all water soluble dyes. Dupont #4 produced yellow hotmelts, Superlitefast Brilliant Blue produced milky white hotmelts. The others gave unacceptable results.

Dye Solution: Mix 1 gram of dye into 100 grams of distilled water at 100 degrees Celcius.