

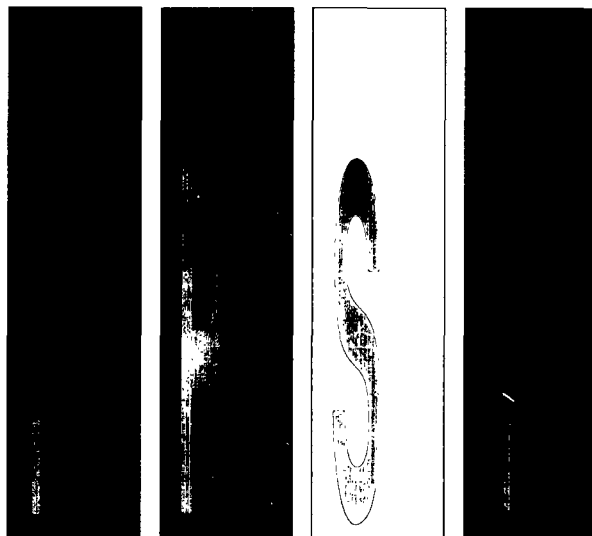


*Institute of Paper Science  
and Technology*

**ANNUAL RESEARCH REVIEW**

**SURFACE AND COLLOID SCIENCE**

March 24-25, 1992



*Atlanta, Georgia*

# INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

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**INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY**

**Atlanta, Georgia**

**ANNUAL RESEARCH REVIEW**

**SURFACE AND COLLOID SCIENCE**

**MARCH 24-25, 1992**



March 13, 1992

TO: MEMBERS OF THE SURFACE AND COLLOID SCIENCE PROJECT  
ADVISORY COMMITTEE

Enclosed is the status report covering the work to be presented at our March 24 meeting. Also attached is an agenda for the meeting.

We are looking forward to discussing our work with you and to hear your comments and suggestions.

If you have any questions, please call me at (404) 853-9720.

Sincerely,

Robert A. Stratton

Enclosure

RAS/sj

*Institute of Paper Science and Technology, Inc.*

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SURFACE AND COLLOID SCIENCE  
PROJECT ADVISORY COMMITTEE MEETING

March 24-25, 1992  
Wyndham Hotel  
Atlanta, Georgia

AGENDA  
March 24, 1992

TECHNICAL PROGRAM REVIEW

1:00	Welcome, Introductions, Antitrust Statement	Stratton/Lavery
1:15	Project Reviews	
	Project 3681--Utilization of Recycle Fiber	Stratton
	Part One: Removal of Contaminants by Flotation	
2:15	Coffee Break	
2:30	Project 3681--Utilization of Recycled Fiber	
	Part Two: Fiber Properties and Effect of Ozone Treatment	Ellis
3:30	Thesis Student Research	Friese
4:00	Project 3681 - Utilization of Recycled Fibers	
	Part Three: Improved Performance of Recycled Fibers	Waterhouse
5:00	Adjournment	



PROJECT ADVISORY COMMITTEE MEETING

AGENDA

March 25, 1992  
Wyndham Hotel  
Atlanta, Georgia

8:00 Coffee and Rolls

8:15 Committee Discussions

11:00 Adjournment

UTILIZATION RECYCLED FIBER

PART ONE: REMOVAL OF CONTAMINANTS BY FLOTATION  
PART TWO: FIBER PROPERTIES AND EFFECT OF OZONE TREATMENT  
PART THREE: IMPROVED PERFORMANCE OF RECYCLED FINES

STATUS REPORT

FOR

PROJECT 3681

TO THE  
SURFACE AND COLLOID SCIENCE  
PROJECT ADVISORY COMMITTEE

March 24, 1992  
Institute of Paper Science and Technology  
Atlanta, Georgia

## Technical Program Review Report

Project Title: Utilization of Recycled Fibers  
Project Staff: R.E. Ellis, R.A. Stratton  
Budget (FY 91-92): \$480,000  
Reporting Period: Jan., 91-Jan., 92  
Division: EPM  
Project Code: RECYC  
Project No.: 3681

**OBJECTIVE:** To develop the technological understanding necessary for a significant expansion in the use of recycled fiber from secondary sources. Examine the practical limitations on the expanded use of secondary fibers in various paper grades.

**SUMMARY:****Part One: REMOVAL OF CONTAMINANTS BY FLOTATION.**

This work began with a re-analysis of experiments done in 1980 on the flotation of hot melts and pressure sensitive adhesives. We showed that the efficiency of contaminant removal was a function of the strength of the attachment of the contaminant to the air bubble. This strength in turn depends on the surface tension of the liquor and the surface energy of the contaminant. An important finding was that the latter was a function of the contact time with the liquor. Several suggestions were made for this time dependence but not proved.

Beginning with a student research project (Cheryl Keller), the work was extended to a laser-printed toner as the contaminant. Although the results were incomplete, the same trends were found for the toner as for the hot melts: surface energy of the toner increased with time of contact with the deinking liquor, and removal during flotation increased with (calculated) bubble/toner attachment force. efficiency was strongly dependent on toner particle size with poor removal for particles greater than 150  $\mu\text{m}$  in size.

Various parts of this work were presented at the annual meeting of the American Institute of Chemical Engineers, the annual meeting of the American Filtration Society, and the 1st Research Forum on Recycling.

Further examination of the time dependence of the surface energy of the contaminant reveals that the effect does not depend on the contaminant composition but only on the deinking liquor properties. The evidence strongly

suggests that the effect is a result of the diffusion of the surfactant to the surface where its adsorption modifies the contaminant's surface energy.

A survey has been completed on the composition of electrostatic and laser-printing toners in use today. A total of 154 toners marketed by 11 major manufacturers and distributors were classified according to the composition of their matrix material and their pigment. This information will be valuable for subsequent phases of this project.

A program was developed to ascertain the critical factors necessary to achieve excellent flotation efficiency for electrostatic and laser-printed toners as found in mixed office waste. To carry out this research, we have designed a laboratory flotation cell which will allow control and measurement of bubble size and other crucial parameters.

## Part One: REMOVAL OF CONTAMINANTS BY FLOTATION.

### INTRODUCTION

Until recently most in the United States was carried out by washing alone. This was because the primary recycled fiber source was newsprint, and newsprint ink can be efficiently removed by washing because of its small particle size. With the decreasing amount of landfill space available the trend is toward recycling other fiber streams including mixed office waste. This material, containing electrostatic toners and laser-printed inks, results in much larger ink particles upon repulping. Typically, these range from 10 to 500  $\mu\text{m}$  in size and are not effectively removed either by washing or centrifugal cleaning. Flotation can be used to efficiently separate such particles from the fiber. It is also effective for removing stickies such as hot melts and pressure sensitive adhesives. Most new capacity being installed today in the U. S. includes one or more flotation steps. To effectively use this process, we must understand how flotation works.

### REVIEW OF PAST PROJECT ACTIVITY

The principle of flotation is that separation can be achieved by the selective attachment of air bubbles to one of the components in the slurry. In we want the bubbles to attach to the ink particles and not to the pulp fines or fibers. This is achieved in one of two ways:

- a) by taking advantage of the difference in surface energies between the contaminant and the pulp components, or
- b) by adding a chemical which will selectively adsorb on the contaminant to lower its surface energy and thereby magnify its difference from the pulp.

The process of bubble attachment is shown in Fig. 1. The contaminant is shown as a flat plate which is a good representation of the shape of repulped toner ink particles. In order for the air bubble to attach to the particle it must displace the liquid already there. This requires (1) that the contact angle  $q$  (defined in Fig. 1) be greater than zero. The contact angle in turn is determined by Young's equation

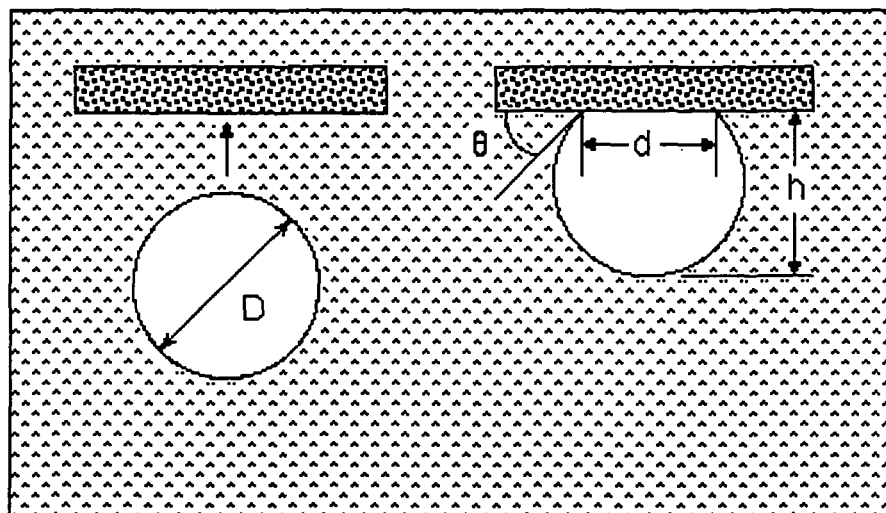


Fig. 1. Schematic representation of bubble attachment to a sticky. (1)

$$\cos \theta = (\gamma_{SV} - \gamma_{SL}) / \gamma_{LV} \quad (1)$$

where  $\gamma_{LV}$  is the surface tension of the liquid (the liquor),  $\gamma_{SV}$  is the surface energy of the contaminant, and  $\gamma_{SL}$  is the interfacial tension between the liquor and the contaminant.

Once the bubble is attached to the contaminant, it must maintain the attachment as it carries the latter to the surface of the flotation cell for skimming. During the ascent the bubble/contaminant pair is subjected to agitation and buoyancy forces which could separate the two. The force of attachment  $F_a$  is given (1) by

$$F_a = \pi d \gamma_{LV} \sin \theta \quad (2)$$

where  $d$  is the diameter of the circle as shown in Fig. 1. This can be rewritten (1) in terms of the bubble diameter before attachment  $D$ , which is more readily measured.

$$F_a \approx \pi D \gamma_{LV} \sin^2 \theta \quad (3)$$

The approximation is good to within 10% for contact angles of  $60^\circ$  or less; the exact expression is given elsewhere (1). The contact angle can be related to the surface energy of the contaminant through the Girifalco-Good-Fowkes-Young equation

$$\cos \theta = -1 + 2(\gamma_s^d \gamma_L^d)^{1/2} / \gamma_{LV} \quad (4)$$

where  $\gamma_s^d$  and  $\gamma_L^d$  are the dispersion components of the surface energies of the solid contaminant and the liquor, respectively (2). The value for the solid is approximately equal to  $\gamma_{SV}$  when the solid is hydrophobic.

Eqs. 3 and 4 predict that the attachment force will be a function of the bubble size, the liquor surface tension, and the surface energy of the contaminant. (For eq. 3 to hold the bubble attachment diameter  $d$  must be less than the size of the particle. If this is not the case, a more complex treatment (3) is necessary.) The relationship is shown graphically in Fig. 2 where the attachment force (calculated using the exact expression given in (1)) is plotted against the liquor surface tension at a constant bubble size  $D$  of  $300 \mu\text{m}$  and two different levels of contaminant surface energy. The lower energy material would correspond to a polyethylene-type material while the other would be much more polar. The negative value for the latter at a liquor surface tension of  $30 \text{ dyne/cm}$  indicates that the bubble would not be able to attach in this system. In general the attachment force increases with increasing liquor

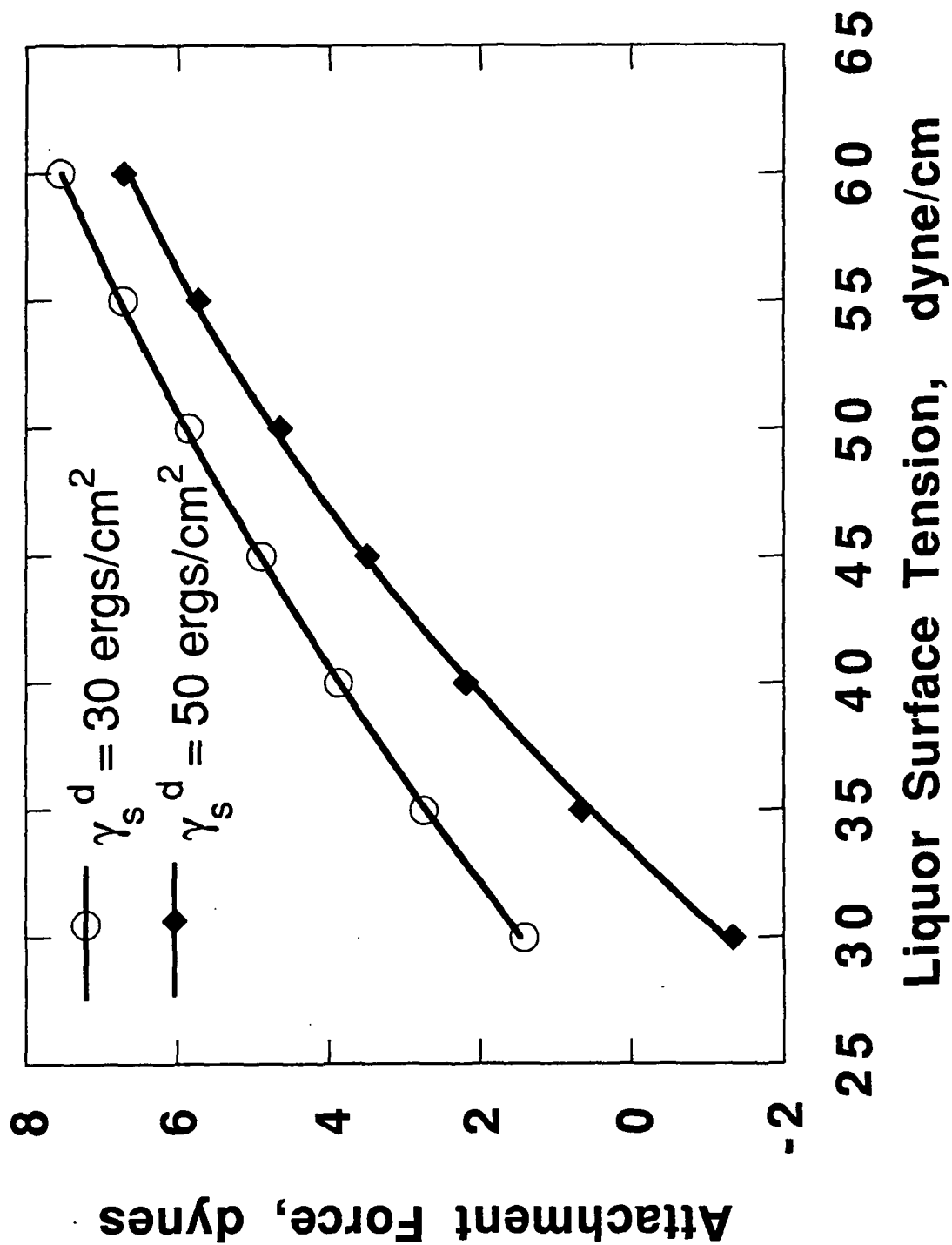


Fig. 2. Theoretical dependence of bubble attachment force on deinking liquor surface tension at two levels of contaminant surface energy using the exact expression. Bubble diameter is 300  $\mu\text{m}$ .



surface tension. The advantage of lowering the contaminant surface energy as much as possible to maintain a sizeable difference between it and that of the cellulosic materials is obvious.

The parameters that we can potentially control in the mill are the liquor surface tension and the contaminant surface energy. Chemical additives (called "collectors") have been developed in the mineral processing industry to preferentially adsorb onto one of the minerals present and thereby lower its surface energy and provide preferential bubble attachment. Whether an additive with broad applicability to a variety of contaminants can be produced for the paper industry is not yet known.

A further complicating factor has recently been found (4). The surface energy of the contaminant, when in contact with the liquor, increases with time. The changes occur in times of the order of a few minutes to tens of minutes before leveling out to an apparently constant value. These time frames are the same as those found in typical pulpers where the deinking chemicals are usually added. The direction of the change is disadvantageous, since it causes the contaminant to become more polar or more like the pulp. This will likely affect the efficiency of the separation. It also reduces the force of attachment between the contaminant and the air bubble as shown in Fig. 2. Perhaps chemicals can be developed to minimize or eliminate these changes.

#### DISCUSSION OF 1991 RESULTS

The capability of eq. 3 to correlate flotation data was tested using results obtained on a series of hot melts (5) and on recycled laser-printed paper (6).

Three hot melts with rather different (initial, i.e. before contact with the deinking liquor) surface energies were used. The surface energies, calculated using eq. 4 from contact angle measurements of methylene iodide on films of the hot melts, ranged from 27.1 to 50.7 ergs/cm<sup>2</sup>. Flotation (deinking) liquors were comprised of sodium silicate, a nonionic surfactant to provide stable bubbles, and caustic to adjust the pH to 10 or 12. The amount of surfactant was varied to give different liquor surface tensions. Contact angle measurements as a function of time for drops of the liquor on films of the hot melts provided information on the change of surface energy with time (4).

A transparent plastic pneumatic flotation cell developed at the Institute of Paper Chemistry (5) was used to assess the floatability of the hot melt particles. A flotation time of one minute was used throughout the experiments. Surfactant

concentration, pH, and time of contact of the hot melts with the deinking liquors were varied. Bubble size also varied with surfactant concentration. Contaminant removal was assessed gravimetrically.

Laser-printed paper, using a single source of alkaline-made paper and toner, was repulped in a Lamort laboratory pulper at 10.5% consistency (6). The consistency was then lowered to 0.8% and flotation was carried out in the same unit using a combination of a nonionic surfactant and oleic acid at various pH levels from 8 to 12. Different levels of surfactant were used to vary the liquor surface tension. Because the cell is constructed of metal, it was not possible to determine the bubble size. Flotation was carried out for 10 minutes, and standard TAPPI handsheets were prepared from the stock before and after flotation. A COCAP Dot-Counter 2.0 was used to determine the total area of ink particles in the range 50 to 100  $\mu\text{m}$  in these sheets. Comparison of the results before and after flotation provided a measure of efficiency (6).

The change in contact angle (surface energy) of the toner in contact with the deinking liquors was also assessed. Films of the toner were prepared by melting the toner particles on a glass slide in an oven until they formed a continuous smooth film. Representative results are shown in Fig. 3. Little, if any, change occurs when distilled water is used. When surfactant is present, however, changes comparable to those found for the hot melts (4) are noted. Possible explanations for the changes have been discussed (4).

The flotation efficiency for the hot melts is shown in Fig. 4 (4) plotted against the calculated value for the attachment force. The latter is from eq. 3 using the measured values of the bubble size  $D$ , the liquor surface tension, and the contact angle of the liquor on the hot melt at the corresponding length of contact time. The data are seen to fit a smooth curve which suggests that the various parameters are being weighted in the proper way by eq. 3. The sharp change over a narrow range of attachment forces suggests that this parameter is crucial to good flotation. The percent removal at a given attachment force is not unique but depends on the particular flotation cell geometry, the severity of agitation in the cell, the duration of the flotation, and the relative particle size to bubble size. A dependence of efficiency on the ratio of the two sizes has been suggested (7) and likely depends on the degree of agitation (hydrodynamic shearing) in the cell.

The results for the flotation from the laser-printed paper are shown in Fig. 5. Because no information on bubble size is available, we use a reduced attachment force,  $F_a/D$ , as the variable for correlation. The data from Fig. 4 are also shown plotted against the reduced force. Although the trend remains, it is apparent that

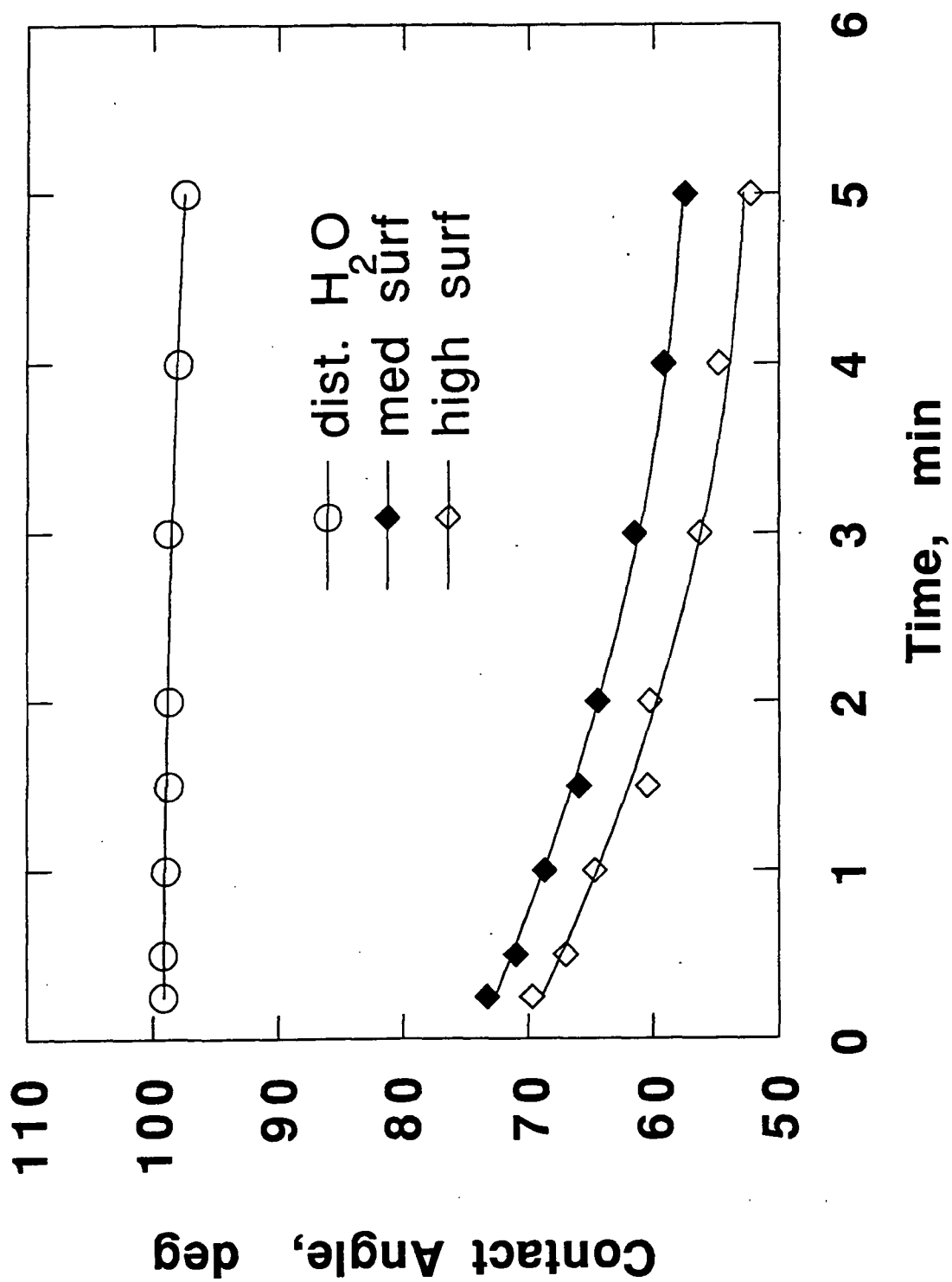


Fig. 3. Time dependence of the contact angle of various liquors on a toner.

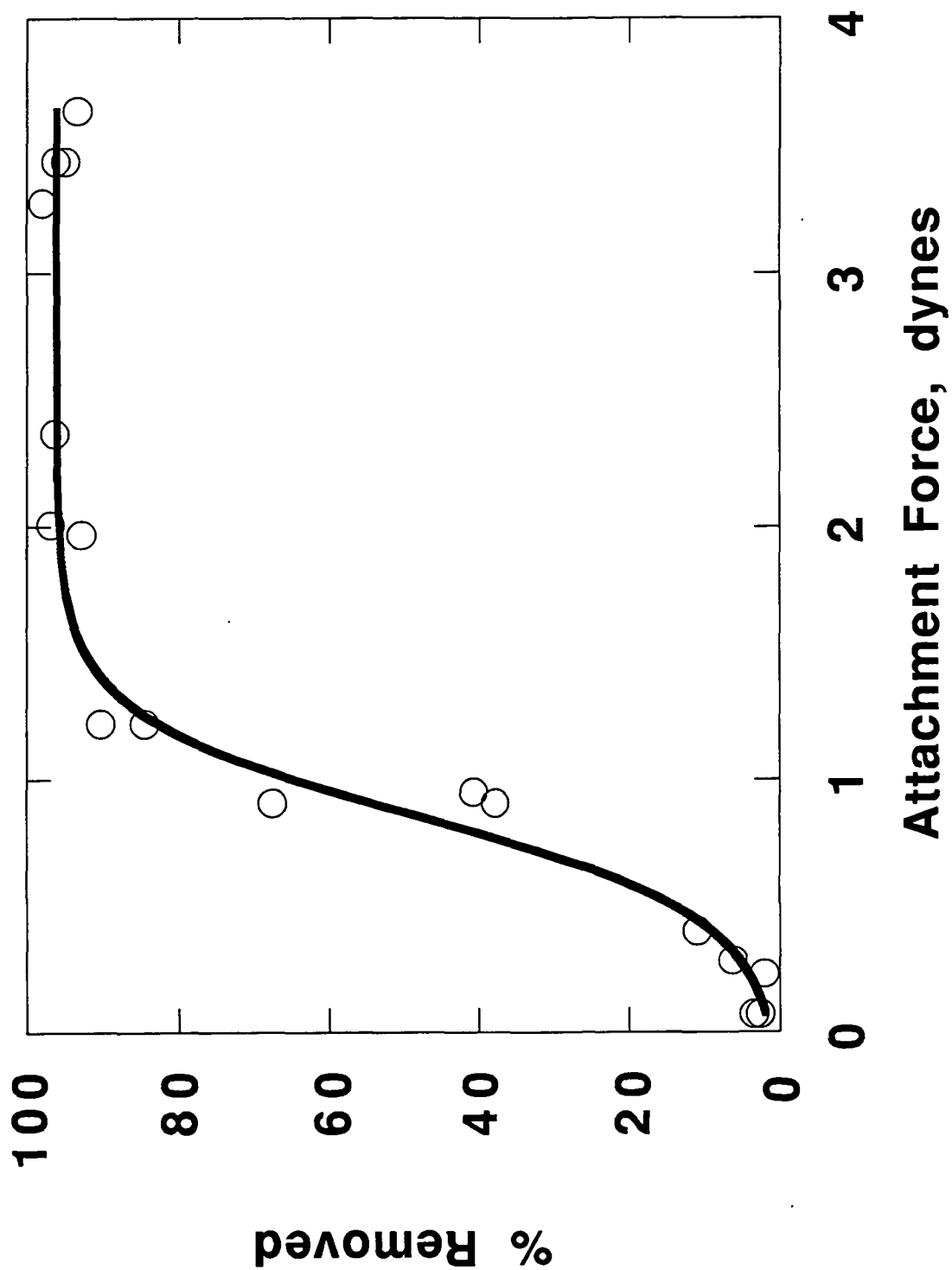


Fig. 4. Flotation efficiency of hot melts as a function of calculated attachment force.  
(4)

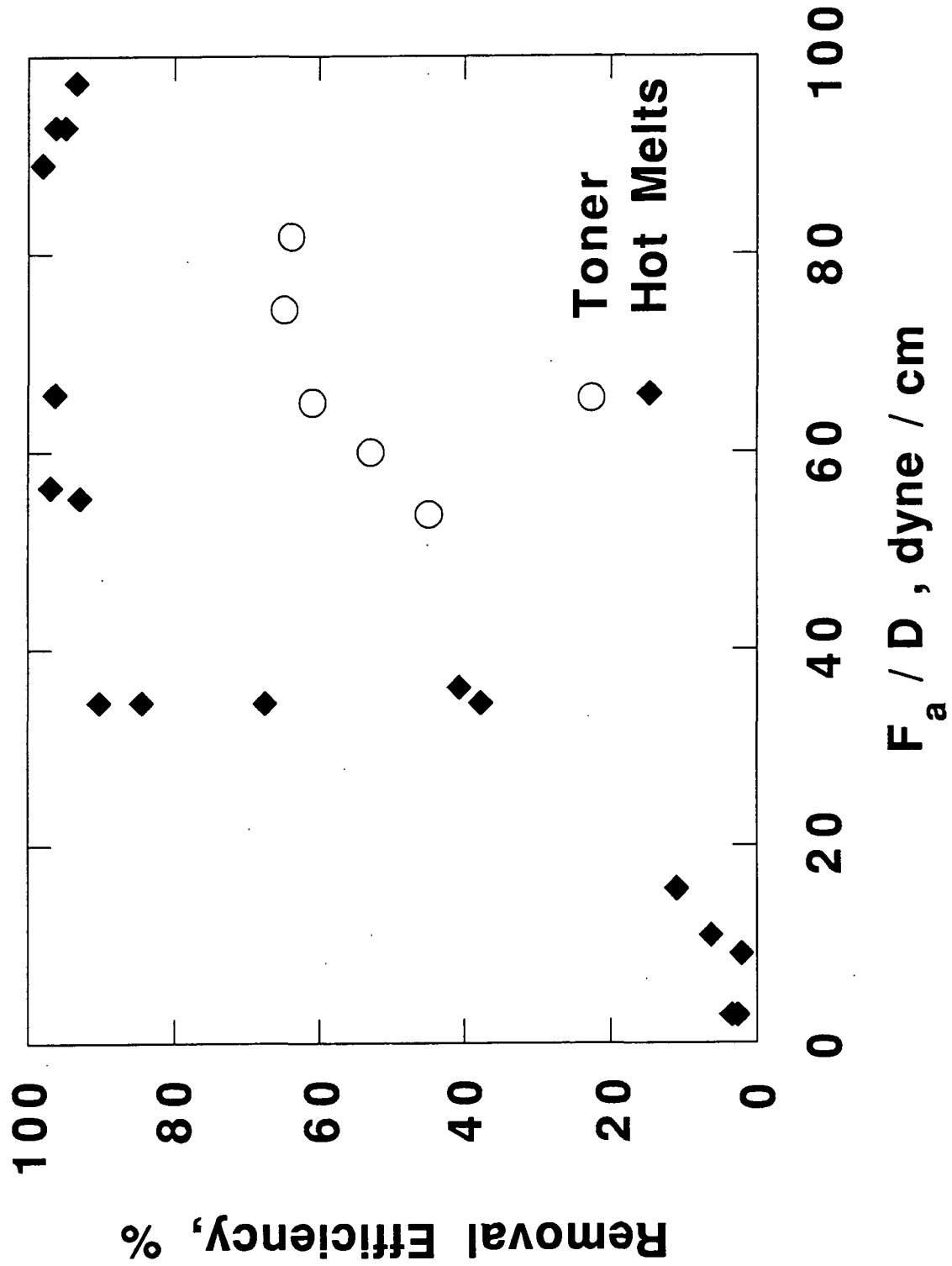


Fig. 5. Comparison of flotation efficiencies of laser-printing toner and hot melts as a function of reduced attachment force. (4)

the inclusion of the bubble size results in a smoother curve for the hot melt data. The results for the toner appear to be leveling off at a much lower value of efficiency than was found for the hot melts. This is likely due to the rather different agitation conditions in the two flotation cells. The addition levels of surfactant and collector were not optimized at this stage and likely contribute to the inferior flotation performance. Removal efficiencies were even poorer for larger toner particles ( $> 150 \mu\text{m}$ ). This suggests that either a) greater attrition of the ink particles may be necessary or b) the flotation chemistry needs to be improved, or c) a better match between bubble size and particle size is needed. Clearly, a great deal of additional work is needed to better understand the contributions of the several variables to efficient flotation of contaminants.

One of the explanations (4) for the change in contact angle (and therefore contaminant surface energy) with time was a slow adsorption of surfactant from the liquor onto the contaminant. Initially, we suggested this was not a likely explanation because of the small size of the surfactant molecule and hence its large diffusion coefficient. A time for adsorption equilibrium on the order of seconds rather than minutes was suggested (4). Upon further consideration of the low concentration of the surfactant, we re-evaluated this explanation. If we assume that the change in surface energy is directly proportional to the amount of surfactant adsorbed, we can use the equation for diffusion from a semi-infinite sink (8) to produce the following equation.

$$\gamma_s(t) = \gamma_s(0) + (d\gamma_s/dc_A)(2c_0)(D/\pi)^{1/2} \sqrt{t} \quad (5)$$

where  $\gamma_s(t)$  and  $\gamma_s(0)$  are the surface energies of the contaminant after contact times with the liquor of  $t$  and zero, respectively,  $c_A$ ,  $c_0$ , and  $D$  are the amount adsorbed, the initial concentration, and the diffusion coefficient of the surfactant. This equation predicts that the contaminant's surface energy should increase linearly with the square root of contact time. Data for three hot melts and a polyethylene-coated kraft paper are shown in Fig. 6. A linear regression was performed on the results for sample HMA. Linear regressions for the other samples were done by forcing their slopes to be identical to that of HMA. Reasonable fits are found in all cases. The contaminants have very different compositions and initial surface energies, yet show the same changes with time. This suggests that the time dependence has nothing to do with the contaminant, but only reflects the surfactant's concentration (the same for all samples here) and diffusion coefficient. Using a reasonable value for the diffusion coefficient, we can calculate a value for the slope from the coefficient of  $\sqrt{t}$  in equation (5) which is in fair agreement with the experimental slope in Fig. 6. Additional studies are in progress to validate these findings.

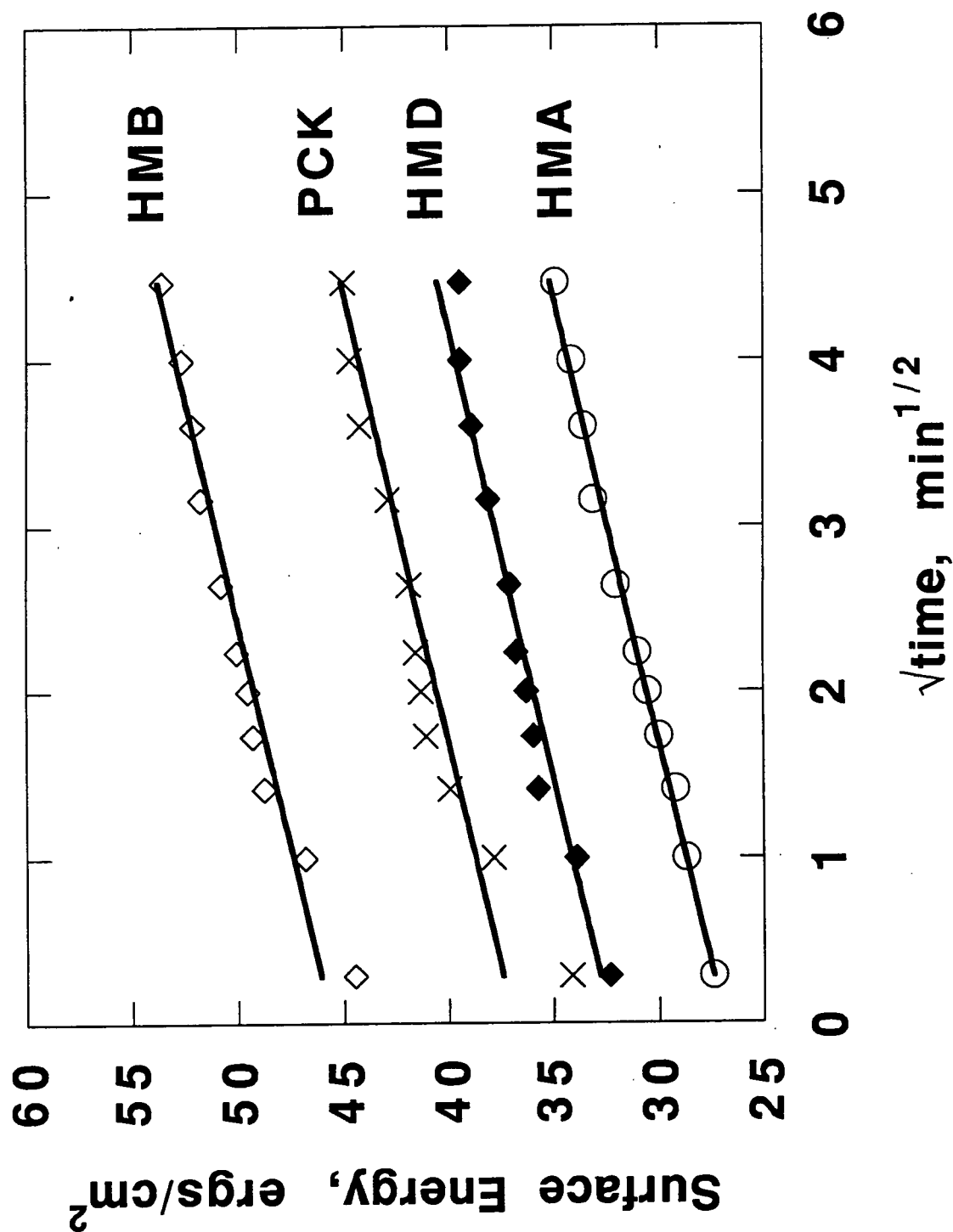


Fig. 6. Variation of surface energy with the square root of contact time with deinking liquor for three hot melts and a polyethylene-coated kraft (PCK).

In preparation for an expanded study of mixed office waste, we have completed a survey of the most widely used electrostatic and laser-printing toners. The following table represents the survey results of 11 major toner manufacturers and distributors. Each toner manufacturer contributed material safety data sheets (MSDS) for each of the 154 toners tabulated below. The table integrates data from all manufacturers and summarizes the basic chemical compositions of all toners.

Each toner composition is predominantly a polymer matrix encapsulating a black pigment. For most toners the major component is the polymer matrix (approx. 70-90%), although some iron oxide-based toners are composed of only 30-50% polymer, and 50-70% iron oxide. Toner are classified according to the major polymer, the minor polymer, and finally, the pigment type. The inorganic components typically make up less than 5% of the overall toner composition, and vary among different toners within the same classification. For each classification, the total number of toners of a given composition are totalled, followed by appropriate reference numbers for each of the 154 toners listed below the table.

A wide variety of matrix materials (polymers) are currently being marketed. Samples representative of the major classes of toners will be used in subsequent studies.

#### CONCLUSIONS/RECOMMENDATIONS

1. Flotation efficiencies for sticky contaminants and electrostatic toners can be correlated using the concept of the force of attachment between the air bubble and the particle to be floated.
2. Increases in the surface energy of the contaminants occur with increasing contact time with the liquor. Results to date suggest this is caused by surfactant adsorption onto the contaminant and is independent of the latter's composition. Theory suggests the rate is determined by the concentration and diffusion coefficient of the surfactant.
3. Flotation efficiency of the laser-printing toner studied here was a strong function of the toner's particle size in the repulped stock. Particles in the size range 50-100  $\mu\text{m}$  were most efficiently removed with increasingly poorer results for larger particle classes.
4. A wide range of compositions of electrostatic and laser-printing toners are currently being marketed.



Commercial Toners Survey						
Major Polymer	Minor Polymers	Inorganics	Pigment	Density	No.	Ref.
Acrylic (Cont.)	Maleated Rosin Hydrogenated Rosin		CB	NA	1	137
	Styrene-allyl Alcohol	Amorphous Silica	CB	NA	1	138
	Styrene-allyl Alcohol Polyamide	Amorphous Silica	CB	NA	1	139
Styrene			CB/Organic	1.2	2	140-41
Paraffin	Ethylene Vinyl Acetate		Iron Oxide/CB	2.0	1	142
Styrene Methyl-methacrylate	Styrene Butylacrylate		CB	1.1-1.7	1	143
Polyethylene	Ethylene Vinyl Acetate	Fumed Silica	Iron Oxide/CB	2.0	3	144-46
Epoxy Resin		Fumed Silica Fluorochemical	Iron Oxide/CB	2.0-2.2	3	147-49
Vinyltoluene Acrylate			CB/Organic	NA	1	150
Vinyltoluene Butadiene		Amorphous Silica	CB	NA	1	151
CAS 65328-52-1*			CB/Organic	1.2	2	152-53
CAS 68240-06-2**			CB/Organic	1.1	1	154

\* 2-Propenoic acid, 2-methyl-, methyl ester, polymer with ethylbenzene 2-ethylhexyl 2-propenoate and 2-propenoic acid, compound with 2-(dimethylamino) ethanol;  $(C_5H_8O_2 \cdot C_{11}H_{20}O_2 = C_8H_8 \cdot C_3H_4O_2)_x \cdot xC_4H_{11}NO$

\*\* 2-Propenoic acid, 2-methyl-, 2-methylpropyl, polymer with ethylbenzene and 2-ethylhexyl 2-propenoate;  $(C_8H_{14}O_2 \cdot C_{11}H_{20}O_2 \cdot C_8H_8)_x$

Commercial Toners Survey					
Major Polymer	Minor Polymers	Inorganics	Pigment	Density	Ref.
Styrene Acrylate	Polypropylene	Amorphous Silica Zinc Stearate	Carbon Black (CB)	1.1-1.5	12 1-12
		Amorphous Silica Dioxide Copper Oxide	CB/Organic	1.1-1.5	27 13-39
		Silica	Iron Oxide /CB/Organic	1.3	1 40
		Amorphous Silica Silicon Carbide	Iron Oxide/Organic	NA	7 41-47
	Polyester Polypropylene	Amorphous Silica	CB/Organic	0.3-0.5	1 48
		Strontium Titanate	Iron Oxide	1.3-1.5	2 49-50
	Polypropylene Polyethylene Wax	Silica	Iron Oxide/Organic	1.4	1 51
	Fatty Acid Amide Maleated Rosin	Amorphous Silicon Dioxide	CB	0.9	5 52-56
	Maleated Rosin Polyvinyl Butyral		CB	0.9	2 57-58
	Polyvinyl Butyral		CB/Organic	NA	1 59
			CB	NA	2 60-61
		Salicylic Acid Chromium Amorphous Silica	Iron Oxide	1.4-1.7	4 62-65
		Amorphous Silica	Iron Oxide/Organic	NA	3 66-68
		Titanium Oxide Amorphous Silica Aluminum Oxide	CB/Organic	1.1-1.5	22 69-90

Commercial Toners Survey						
Major Polymer	Minor Polymers	Inorganics	Pigment	Density	No.	Ref.
Styrene Acrylate (Cont.)		Quaternary Ammonium Amorphous Silica	CB	1.0-1.1	13	91-103
		Amorphous Silica	CB/Organic	0.3-0.5	2	104-5
Styrene Butylacrylate	Polypropylene		CB	NA	5	106-10
		Stainless Steel Powder	Iron Oxide/CB	NA	1	111
Styrene Butadiene	Petroleum Resin Polypropylene		CB	1.1	1	112
		Steel Powder Quaternary Ammonium	Iron Oxide/CB/ Organic	NA	2	113-14
		Quaternary Ammonium	Iron Oxide/CB	NA	4	115-18
			CB	1.1	2	119-20
Polyester	Fatty Ethylene Diamide, Wax	Titanium Dioxide Amorphous Silica	CB	1.1	2	121-22
	Polyvinyl Butyral		CB	NA	1	123
	Polypropylene		CB	NA	2	124-25
		Salicylic Acid Cr Chelate	Organic	1.0-1.2	1	126
		Amorphous Silica Salicylic Acid Cr Chelate Zinc Stearate	CB	1.0-1.2	2	127-28
Acrylic		Salicylic Acid Cr Chelate	Iron Oxide	1.4-1.6	2	129-30
		Amorphous Silica	Iron Oxide/CB	NA	3	131-33
	Maleated Rosin	Amorphous Silicon Dioxide	CB	0.9	3	135-36

## Toner References

1. IBM 4216 Printer Toner
2. Kodak HX Toner
3. Lanier 416 Toner
4. Lanier 439 Toner
5. Lanier 6055 Black Toner
6. Lanier 6525/6540 Black Toner
7. Lanier 6523 Black Toner
8. Toshiba T-1710 Black Toner
9. Toshiba T-2510 Black Toner
10. Nashua Xerox XT-20 Dry Ink Plus
11. Nashua Xerox XT-25 Dry Ink Plus
12. Nashua Xerox XT-35 Dry Ink Plus
13. Mita DC-3555, 3585, 4555, 4585, 4585F, 7085, 8585, 1824ZS
14. Mita AC-6500
15. IBM 3835 Printer Toner
16. 3M Brand Type 965 Toner
17. Lanier Type 143/153/702 Toners
18. Lanier Type 541 Black Toner
19. Lanier T-64P(701) Toner
20. Lanier 6055 Black Toner
21. Lanier 6020 Black Toner
22. Lanier 143/153/702 Toners
23. Lanier 6242 Toner
24. Lanier 6517 Black Toner
25. Lanier 6450, 6550 Black Toner
26. Lanier 6230 Black Toner
27. Lanier 6360 Black Toner
28. Toshiba T-44P Black Toner
29. Toshiba T-58PN Black Toner
30. Toshiba T-61P, T-62P, T-64P, T-66P, T-80P, T-88P, T220P Black Toner
31. Toshiba T-68P Black Toner
32. Toshiba T-85P Black Toner
33. Toshiba T-5020 Black Toner
34. Nashua Mita MT-23 Toner
35. Nashua Mita Mit-131, Mit-161 Toner
36. Nashua Mita 8112, 8120, Toner
37. Nashua Sharp 750, 771, 781 Toner
38. Nashua Sharp 800, 811, 820, 850 Toner
39. Konica Royal Toner 115/122 and 130/130R
40. Lanier 6315 Black Toner
41. Nashua Canon CT-1 Toner
42. Nashua Canon CT-3 Toner
43. Nashua Canon CT-CX Toner
44. Nashua Canon CT-SX Toner
45. Nashua Ricoh NT-5, 10, 2050 Toner
46. Nashua Ricoh NT-4080 Toner
47. Konica Royal Toner 1200 MC
48. 3M Brand Type 148 Toner
49. Nashua Canon CT-7 Toner
50. Nashua Kodak KT-85 Monocomponent Toner
51. Lanier 6215 Black Toner

52. IBM 6670 Printer Toner
53. IBM Series III Mod 10/20 Toner
54. IBM Series III Mod 30/40/50/60/70/85 Toner
55. Kodak High Density Toner for Series III 30/40/50/60/70/85
56. Kodak High Yield Toner for Series III 10/20
57. IBM Page Printer 3800 Toner
58. Konica Royal 700, 702
59. Nashua Siemens ST-22 Toner
60. Nashua Xerox Type 4, 8, 9 Toner
61. Nashua Xerox XT-55 Dry Ink Plus
62. HP Toner 92282A
63. HP Toner 92285A
64. HP Toner 92295A
65. Kodak Monocomponent B Toner
66. Nashua Canon CT-2 Toner
67. Nashua KP-10 Toner
68. Nashua Xerox 1012/5012/5014
69. Mita Toner DC-111, 111C, 113, 114, 142RE, 211, 213RE, 313Z, 313ZD, 1001, 1205, 1255, 1415, 1435, 1855, 1605, 1656, 1685, 2055, 2105, 2254, 2255, 2285, 2555, 2585, 3255, 3285, 4055, 4085, 1842RE, 1824ZII, 1824Z
70. Mita Toner DC-152Z, 1655, 1785
71. Mita Toner DC-5555, 5585, 5585H
72. Mita Toner DC-3648
73. HP Toner 92182D
74. 3M Brand Quantimatic Toner Type 748F
75. Lanier Type 475 Toner Powder
76. Lanier Type 416 Toner Powder
77. Lanier Type 439 Black Toner
78. Lanier 6110/6112 Toner
79. Toshiba T-30P Black Toner
80. Toshiba T-41P Black Toner
81. Toshiba T-50P Black Toner
82. Toshiba T-56P Toner
83. Toshiba T-120P Black Toner
84. Toshiba D-44PA Developer Toner
85. Toshiba D-44PC Developer Toner
86. Nashua Ricoh NT-30, 40, 50, 60, 5010 Toner
87. Nashua Savin ST-5015, 5020, 7035, 7045 Toner
88. Nashua Toshiba TT-33 Toner
89. Nashua MT-27, MT-37, MT-41, MT-45, MT-47, MT-49 Toner
90. Nashua MT-T1 Toner
91. Xerox 8200/8700/8790/9200/9210/9400/9500/9700/9790/9900/9950 Dry Ink, 7080/7085 Dry Imager
92. Xerox 1075/1090/4050/4090/4650 Dry Ink, 1075/1090 Toner
93. Xerox 1040/1045/1048/1050/5042/5052 Dry Ink Plus
94. Lanier Type 552 Toner
95. Lanier Type 690 Secretary II Toner
96. Lanier 6113/6213/6413/6514 Black Toner
97. Ricoh FT 4000 Series Toner
98. Ricoh D-1210 Developer Toner
99. Nashua IBM IT-8, IT-38 Toner
100. Nashua Kodak KT-1 Toner
101. Nashua Kodak KT-2 Toner
102. Nashua Xerox XT-36 Type 2, 6, 10, 15 Toner

103. Nashua Xerox XT-54 Type 17, 20, 24 Dry Ink
104. 3M Type 164 Toner
105. Konica Royal Toner 110
106. IBM 3827 Printer Toner
107. IBM Laser Printer High Yield Cartridge
108. Kodak Ektaprint L Toner
109. 3M Brand Type 207 Toner
110. Lanier Type 554 Toner
111. IBM 3828 Printer Toner
112. IBM 3900 Printer Toner
113. Xerox 1065/5046/5047/5065 Dry Ink
114. Xerox 5018/5028/5034 Black Dry Ink
115. Xerox 5090/DocuTech/4135 Dry Ink
116. Nashua Xerox XT-18 Toner
117. Nashua Xerox XT-5046 Dry Ink
118. Nashua Xerox XT-65 Dry Ink
119. IBM 3816 Printer Toner
120. IBM 3812 Printer Toner
121. Konica Royal Toner 145/145R, 3302MR, 4003ZMR/4290, 5003ZMR/5503ZMR, 4090, 5590, 6090/7090
122. Konica Royal Toner 1503ZMR, 1602MR/2502MR/1803ZMR/2803ZMR, 2203/3290, 1290, 1790/2590, 2290, 8010
123. Nashua Xerox XT-40 Type 12 Dry Imager
124. Nashua Royal RT-1803 Toner
125. Nashua Xerox XT-37 Dry Imager
126. Kodak Coloredge 1550 Black Toner
127. Kodak Coloredge 1525 Black Toner
128. Nashua Xerox XT-30 Dry Imager, Type 14, 18, 21 Toner
129. Kodak Monocomponent 90 Toner
130. Lanier 6070 Black Toner
131. 3M Brand Type 340 Micro-bond Imaging Powder
132. Lanier Type 885 Imaging Powder
133. Xerox XT-34 Magnetic, XT-44 Dry Imager
134. IBM Copier I Toner
135. IBM Copier II Toner
136. Nashua IBM IT-2 Toner
137. IBM 3825 Printer Toner
138. Nashua IBM IT-3, IT-66 Toner
139. IBM IT34 Toner
140. IBM 3820 Printer Toner
141. IBM Executive 102 Copier Toner
142. IBM Scanmaster I Monocomponent Toner
143. Kodak Ektaprint K Toner
144. Lanier Type 414 Imaging Powder
145. Lanier Type 412 and 413 Imaging Powder
146. Mita DC-900D, 800D, 600D, 500D, 17-Dry
147. Lanier "VCQ" Imaging Powder, Type 355
148. Lanier "VH3" Imaging Powder, Type 842
149. Lanier Type 884 Imaging Powder
150. Nashua RBC-1, IR, IV Toner
151. Nashua Xerox Type 5 Toner
152. Mita DC-121, 122, 131, 132, 133R, 161, 162, 232
153. Mita DC-181R, 191, 312RE, 412RE, 1824
154. Mita DC-513Z, 5055

## FUTURE ACTIVITY

The critical operational parameters for flotation will be determined using the bench scale research flotation cell currently under construction. These include bubble size, bubble/contaminant size ratio, chemicals, air flow rate, agitation intensity, and changes in contaminant surface energy with time. Based on the results of these studies, larger scale experiments will be conducted using a laboratory flotation unit (Lamort). This will provide us with enough deinked fiber to conduct handsheet studies on sheet physical and optical properties.

The changes in contaminant surface energy with time of contact with liquor will be carefully studied to validate the proposed mechanism. A better understanding of this process will allow us to predict the effects of different chemicals on the flotation process.

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PART THREE: IMPROVED PERFORMANCE OF RECYCLED FINES

STATUS REPORT

FOR

PROJECT 3681

March 24, 1992  
Institute of Paper Science and Technology  
Atlanta, Georgia



**TECHNICAL PROGRAM REVIEW REPORT**

**PROJECT TITLE:** UTILIZATION OF RECYCLED FIBERS - Improved Performance of Recycled Fines

**PROJECT STAFF:** J. Waterhouse, C. Esworthy

**BUDGET (Fy 91-92):** \$480,000

**REPORTING PERIOD:** July 91 - March 92

**DIVISION:** Engineering and Paper Materials

**PROJECT CODE:** RECYC

**PROJECT NO:** 3681

**OBJECTIVE:**

Determine the impact of the fines component of various recycled furnishes on selected paper properties including: mass density distribution, stiffness, strength, and pore size distribution.

**SUMMARY:**

A survey of the literature relevant to the performance of recycled pulps, particularly the role of recycled fines, has almost been completed. Although the impact of fines on paper properties has received considerable attention, less is known about the impact of recycling, contamination, and the behavior of fines from different pulp sources. Specific strategies for the separate treatment of fines does not appear to have received attention.

Formation, an important quality related factor, is expected to continue to play a role in our recycling research. A Neural network approach is being investigated for possible applications in this and other areas of papermaking, i.e., texture and pattern recognition applications. A Neural Network simulation has been conducted in relating visual uniformity to optical density measurements.

A progress report "Strength Improvement and Failure Mechanism - 2. Formation has been distributed to member companies.

A paper entitled "Effect of Some Papermaking Variables on Formation" has been prepared for publication, and is included in Appendix 1 of this report. Two papers entitled "The Failure Envelope of Paper When Subjected to Combined Out-of-Plane Stresses" and "Strength Development Through Refining and Wet Pressing" were presented at the International Paper Physics Meeting in Hawaii.

## 1. INTRODUCTION

"Strength Improvement and Failure Mechanisms" Project 3469 was officially closed out in July 1991 in order to re-direct resources toward a major new project embracing a number of areas of activity in recycling. The specific area of concern dealt with here is the effect of recycling on fines, their subsequent treatment, and contribution to paper and board properties. It is assumed initially, that in seeking ways to enhance the papermaking properties of recycled pulps, that the long fiber and fines fraction will benefit from separate and distinct treatments. By analogy, we often find that the separate treatment of softwood and hardwoods can enhance the papermaking properties of blends of these pulps.

## 2. REVIEW OF PAST PROJECT ACTIVITY (March 1991 - March 1992)

During the past year a second progress report "Strength Improvement and Failure Mechanism - 2. Formation" was completed and distributed to member companies. We plan to use the IPST Formation Tester, which has the capability of making both optical and mass density measurements in our recycling project.

A paper entitled "Effect of Some Papermaking Variables on Formation" has been prepared for publication, and is included in Appendix 1 of this report. Two papers entitled "The Failure Envelope of Paper When Subjected to Combined Out-of-Plane Stresses" and "Strength Development Through Refining and Wet Pressing" were presented at the International Paper Physics Meeting in Hawaii.

In the area of recycling, work to date has been mainly concerned with a review of the literature. In addition, Keiichi Omori a student is working on a related project concerned with measuring and characterizing the fines fraction performance of a number of recycled furnishes. We will also be concerned with the impact of fines level, type, and treatment on formation an important quality related paper property. In particular the relationship between relationship between optical and mass density will be examined since it is clear that the fines fraction of recycled pulps, i.e., their type and treatment, play an important role in determining optical properties.

The potential applications of neural networks, e.g. texture and pattern recognition in formation etc., has been briefly investigated. A neural network simulation involving an input of either optical or mass density measurements and an output of visual ranking is in progress. Other possible applications involve wet end characterization with respect to formation, the relationship between optical and mass density.

Other activity has included a research project (in co-operation with Dr. R. Stratton, IPST) funded by the National Science Foundation entitled the "The Role of Melamine Formaldehyde in Currency Papers". The major concern here is with the ultimate removal of melamine formaldehyde from currency paper, and its replacement by a suitable alternative. Our major concerns with this project is a better understanding of wet strength, and minimizing the stiffness loss associated with the end use performance of currency papers. A presentation entitled "Wet Strength Characteristics of Cellulose-Melamine-Formaldehyde Composites" was made at Cellulose '91 held in New Orleans December 1991.

Chapter 4 "The Mechanical Properties of Paper" by John F. Waterhouse in Volume 9 Pulp and Paper Manufacture Series a Joint TAPPI/CPPA Text Book Series will be published shortly.

Two papers "The Structural and Mechanical Properties of Handsheets containing Cell-Wall Materials from Three Green Algae" authored by R. Atalla, J. Hackney, and J. F. Waterhouse (oral presentation) and "Wet Strength Behavior of Paper" J.F. Waterhouse (poster presentation) have been accepted for the International Progress in Paper Physics Meeting to be held in Otaniemi, Finland June 8-11, 1992.

### 3. DISCUSSION OF 1991 RESULTS

#### 3.1 Improved Performance of Recycled Fines

The technical issues of recycling appear to fall into the following broad categories namely:

1. Contaminant removal (e.g. printing inks, stickies, and other foreign materials etc.)
2. Stock treatment and preparation to maximize the papermaking potential of recycled furnishes (including re-pulping, bleaching and other chemical and mechanical treatments).
3. Characterizing and determining the potential of recycled furnishes.
4. Improvement of current papermaking practices to enhance paper and board recyclability.

Our current recycling project concerned with improving the performance of recycled fines appears to fall into categories 2 and 3 given above.

The role of fines in papermaking has always been a somewhat controversial area, and even less is known about the impact of recycling. It is well known that fines have an adverse effect on the drainage resistance of a pulp, but can have a very positive benefit on some paper properties, e.g., the mechanical properties of paper. How these papermaking variables are affected by different recycling strategies has yet to be determined.

The main factors controlling the tensile strength of paper are shown in Figure 1. It is noted that for a given level of bonding, i.e., R.B.A. or apparent density, strength is controlled by interfiber bond strength, fiber strength, and fiber geometry. Densification by both combined wet pressing and refining can increase strength by increasing R.B.A. or apparent density. It is suggested that refining can lead to an increase in fiber strength and a reduction in stress concentration.

It has been shown that refining does not increase interfiber bond strength when measured on isolated bond pairs. However one of the major differences between isolated bond pairs and the bonds in a sheet of paper is that fines are present in the latter situation. Therefore, in paper, fines are not only expected to increase bonded area, but to reduce stress concentration as shown in Figure 1. Stress concentration will be governed by the size, shape, and bonding characteristics of the fines all of which may be altered during recycling.

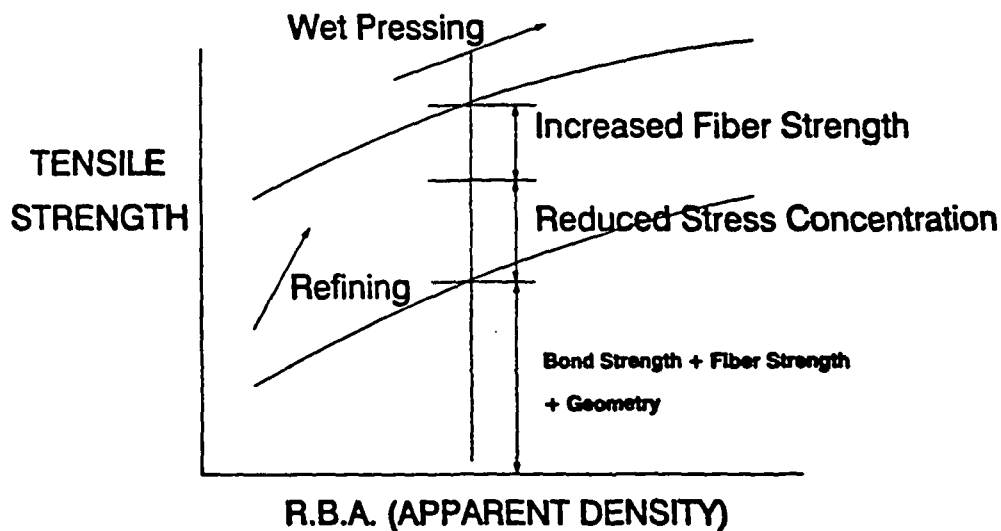


Figure 1. Variation of Tensile Strength with R.B.A. or Apparent Density

One of the major consequences of recycling is to produce hornification, which is a loss in swellability, water uptake, and surface area, of both the long and fines fraction of a pulp. Other adverse affects can include contamination by inks and other materials as well as "damage" to the long fiber fraction.

To better understand the role of fines, particularly with respect to recycling, we will, for the time being, focus our attention on the fines fraction of a recycled pulp, its separate treatment, and subsequent impact on papermaking and properties.

Fines have been defined as the short fiber fraction, slime, flour, fiber debris, and crill. Some researchers have described fines as the pulp fraction which passes through a 200 mesh screen, while others have used a 150 mesh screen. Htun and De Ruvo (1) characterized fines from a bleached kraft pulp according to size, morphology, chemical composition, swellability, physical structure, and mechanical properties. Using a Bauer McNett classifier they showed that fraction passing a 200 mesh screen had the most effect on mechanical properties. This fraction had a higher water swellability, and resulted in handsheets with a higher density and improved mechanical properties. In this case the chemical composition of the fines was similar to the long fiber fraction, however, the crystallinity was lower.

According to Mancebo and Krokoska (2), fines can be classified as being either primary or secondary. Primary fines are present in the pulp prior to refining, and secondary fines are produced during refining. Primary fines are identified as fragments of parenchyma cells, vessels, and the cell wall. Primary fines exhibit a higher lignin, ash, and extractive content than secondary fines or the whole pulp. According to the thorough review by Becher (4), it is generally agreed that primary fines contribute little to bonding.

Sandgren and Wahren (5) used the term "crill" instead of secondary fines. They found that the amount of crill increases approximately linearly with refining time, and has a large influence on the drainage properties of the pulp. With crill removal sheet density decreased resulting in an increase in tear and a decrease in tensile strength. It was stated that the loss in tensile strength attributed to a loss in density could be compensated for by an increase in wet pressing, although no evidence was provided to verify this supposition.

Kibblewhite (6) examined the quality and quantity of fines prepared from *Pinus Radiata* kraft pulps. He concluded that the quality and quantity of fines strongly affected pulp freeness, but had little effect on paper strength.

Lobben (3) found that the fines from chemical pulps had a significant effect on strength properties depending on fiber type and the extent of refining. The effect of fines were

greater for a eucalypti kraft pulp than for a pine kraft pulp, with the effects being more pronounced when the long fiber fraction was unbeaten.

In a model study Terao et al (7) used a micro-fibrillated cellulose and a crystalline cellulose (Avicel) to study the impact of fines on the structure and properties of paper. The microfibrillated cellulose consisted of fibrils whose width was of the order of a few microns or less, whereas the Avicel consisted of particles in the size range of width 10-30  $\mu$ m and length 40-70  $\mu$ m. It was found that MFC increased sheet density and tensile strength while Avicel had the opposite affect. It would be interesting to know whether this result was simply a particle size effect or differences in structure, i.e., relative crystallinity.

Nanko and Ohsawa (8) investigated the structure of interfiber bonding using transmission, scanning electron, and scanning laser microscopes. The secondary fines of beaten pulps were found to reside in the spaces between fibers on the surface of the fiber. They termed the layer between the fibers a "bonding layer" which is made of secondary fines and external fibrils. It was conjectured that the bonding layer reduced stress concentration more than the S1 layer of the fiber.

There is general agreement that recycling causes a decrease in strength properties of paper, and it has been shown by a number of researchers that this decrease is related to a reduced bonding ability of the fibers termed "hornification". Hornification, i.e., loss of swelling and surface area, implies a loss of fiber flexibility. McKee (9) observed that the water retention value (WRV) decreases with recycling. Lundberg and DeRuvo found a direct relationship between WRV and tensile strength.

According to Mancebo and Kroska (2), the fines fraction of recycled pulp has a negative impact on strength since they are only acting as filler material. The fines supposedly become inert due to "hornification", and the effect is irreversible even with refining!

Szwarcstajn and Przysysz (14) also found that fines and fibers become hornified with recycling, and that strength properties decrease.

On the other hand, Hawes and Doshi (15) found that fines from recycled paper are effective in increasing paper strength. They examined the impact of primary and secondary fines from three pulp types, (a 50% yield northern softwood kraft, an 80% softwood-20% hardwood recycled kraft pulp, and a southern pine/Virginia pine TMP) on an unrefined and refined fines free recycled unbleached kraft bag paper.

The results of Hawes and Doshi (15) are shown in Figure 2. The level of fines added to the unrefined fines free pulp was 20% and the level added to the refined fines free pulp was around 8.7%. We see that a 20% fines addition to the unrefined fines free pulp results in an increase in densification and strength. The kraft and recycled fines are about equal in

performance while the TMP fines are much less effective. The recycled bag paper originally contained about 20% fines, but strength and density figures for this paper are not given. The influence of 8.7% fines addition on the fines free refined fiber, results in a reduction of sheet density, although there is a net increase in strength, at least for kraft and recycled fines addition. The reduction in sheet density is greatest for the TMP fines and the change in strength is not significant. The reason for the reduction in sheet density with the introduction of fines into refined pulp, as shown in Figure 1, is not immediately obvious. This finding was noted, but not commented upon by the above authors.

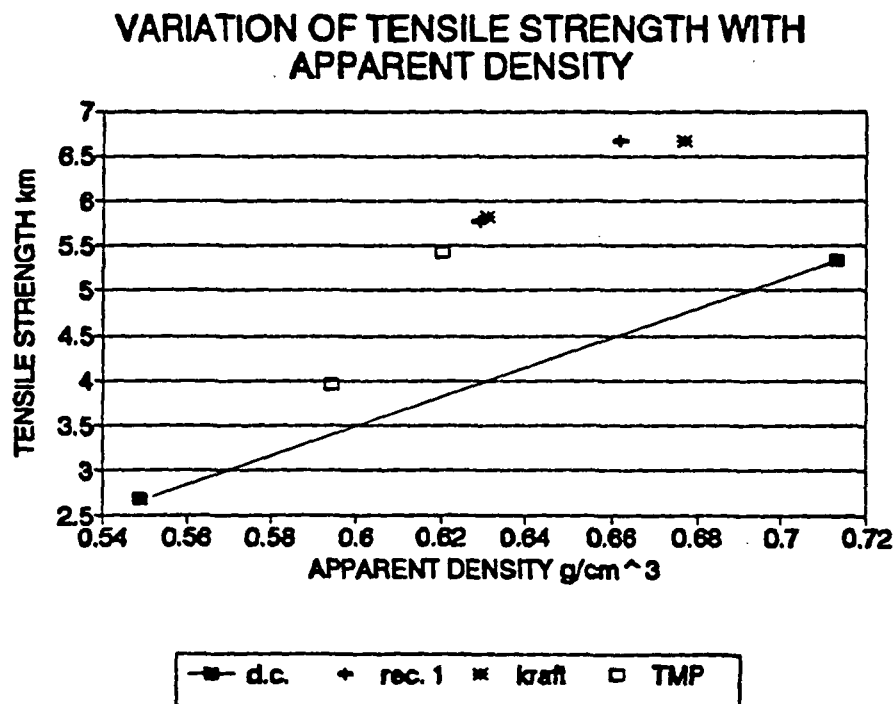


Figure 2. The Influence of Secondary Fines Addition Level and Type on Tensile Strength Development

According to Howard (11), there are four ways to recover the lost property potential of recycled pulps and include:

- a. Beating or refining
- b. Chemical treatment
- c. Blending with virgin pulp
- d. Fractionation

Bhat et al (12) used several techniques to enhance the strength of secondary fiber. They examined refining, high shear field refining (HSR) and or alkali treatment. It was found that a combination of alkali treatment followed by HSR was most effective, and in some instances the performance of the secondary fiber almost equalled that of the virgin pulp.

Ehrnrooth et al (13) examined the use of acetylation to reverse the effects of recycling. It was found that acetylation resulted in swelling and strength properties being comparable with those obtained using a never dried pulp.

Separate treatment of the long fiber and fines fraction of pulps does not yet appear to have received consideration, although it seems clear that the respective treatments required may be very different. Furthermore it does not appear that there have been any studies concerned with the effect of contaminants on fines performance, which is another important aspect of recycling.

It is therefore proposed that the main objectives of this project are as follows:

1. Establish methods for determining the critical properties of fines.
2. Determine the effect of virgin and recycled fines from a number of pulp sources on selected physical properties of paper.
3. Evaluate various strategies for fines treatment.

### 3.2 Neural Networks

An interest in Neural Networks arose out of our work in the area of formation, and the potential for such application in the area of texture or pattern recognition. It seems worthwhile to continue this topic in conjunction with our work in the area of recycling, and its impact on formation.



We begin with an introduction to neural network technology, after which possible areas of application are discussed.

Neural Networks are a type of parallel distributed processing. Parallel processors are sometimes thought of as several conventional (Von Neumann type) processors operating in tandem. However, parallel processing is fundamentally different than typical processing units in that information is stored not in memory cells, but in the relationship between memory cells.

This technology developed out of models used to understand how the human brain works. In the brain, the basic unit is the neuron. Each neuron is interconnected with other neurons via axons and dendrites. A neuron is only capable of sending a pulse or not (electrical analog of logical 1/0). The function of each neuron is to sum inputs sent to it from other neurons. Then, if that sum exceeds the neuron's internal threshold, it will send a pulse to the neurons on the "next level" of interconnections. The human brain contains on the order of  $10^{11}$  neurons, each with as many as 10,000 connections. For the purposes of our model we will organize the neurons into distinct levels. Each node on any one level is fully connected to all of the neurons on adjacent levels.

Now, each connection (axon) contains its own weight factor. So a pulse (value = 1) sent along a connection (weight = 0.5) will contribute 0.5 to the receiving neuron. The connection weight can be thought of as electrical conductivity (1/resistance) in that a weight of 1 would indicate complete connection and a value of zero no connection.

Let us concern ourselves with a typical neural network architecture, namely a three layer fully-interconnected network. Let  $a$  be the number of nodes on the bottom layer(A),  $b$  the number of nodes of the hidden layer(B), and  $c$  the number of nodes on the top layer(C). Then,

$$A = [ A(1), A(2), \dots, A(i), \dots, A(a) ]$$

$$B = [ B(1), B(2), \dots, B(j), \dots, B(b) ]$$

$$C = [ C(1), C(2), \dots, C(k), \dots, C(c) ].$$

Each node is connected to every node on the adjacent layer. Let  $W1$  be the matrix of connection weights between the bottom layer and the hidden layer, and  $W2$  be the matrix of connection weights between the hidden layer and the top layer. Then,

$$W1 = \begin{bmatrix} w(1,1) & w(1,2) & \dots & w(1,b) \\ w(2,1) & w(2,2) & \dots & w(2,b) \\ \vdots & \vdots & \ddots & \vdots \\ w(a,1) & w(a,2) & \dots & w(a,b) \end{bmatrix}$$

where  $w(i,j)$  is the connection between the  $i$ th node of the bottom layer and the  $j$ th node on the hidden layer.  $W2$  is defined in a similar manner between the  $j$ th node on the hidden layer and the  $k$ th node on the top layer. Later it will be important to remember that the connections are valid in both direction.

To begin to move toward applications, we identify the bottom layer as the input layer with  $A$  being the set (or vector) of our input values. The top layer is then the output layer (again a vector). It should be mentioned at this point that it is possible to simulate a neural network using conventional computing methods. The problem is approached from a linear algebra standpoint and hardware implementation is reserved for another time.

Now we come to the real beauty of neural networks. The system is not programmed to perform a specific function as conventional computers are. The system is trained by example to imitate the process. The learning process is achieved through a variety of paradigms. We will be concerned with the back error propagation method.

The first step in back error propagation is to seed the connection matrices with random numbers. For simplicity we restrict the seed range to be from -1 to +1. This is not a restriction of the value that connections might acquire through learning. Note that the connections are allowed to have negative values, thereby contributing negative inputs to the receiving node. This is vital to the convergence of the system to a useful state.

The network is then presented an input vector and is allowed to compute an output vector with its current set of connection weights. The output vector is compared to the correct output vector. An error vector of dimension  $c$  is computed that reflects the node by node difference in the system output and the correct output. The network is then operated "in reverse", propagating the error vector back through the system. This results in adjustments to the connection weights that are computed so that the same input vector will trigger an output that is closer to the correct answer.

The adjustments are made in small increments governed by the system's learning rate. The back error propagation is repeated until the system settles into a state such that the input vector gives the correct output vector.

In order to properly train the network, a sufficient number of samples must be used. These samples constitute the training set. A sufficient number means that the samples in the training set, should adequately represent the range of properties to be tested. For instance, a training set for formation should include samples with very good formation and samples with very poor formation, as well as several intermediate levels. Each member of the training set and its associated correct output vector are presented to the network until a set of connection weights are found that give the correct output vector for each input vector. This is no small task as we are trying to solve  $N$  matrix equations of the form

$$C(n) = W_2 W_1 A(n)$$

where  $n = 1 \dots N$ , the number of training samples. This problem quickly becomes intractable using conventional methods.

Once the system has been trained, the back error propagation is no longer used. The connection weights are fixed and only the forward pass (bottom  $\rightarrow$  hidden  $\rightarrow$  top) is performed. The network will operate properly for new samples whose characteristics fall within the limits set by the training set. The quality of the network then becomes the quality of the training set used.

Neural network technology is particularly useful when the equation governing a specific model are not known. For instance, when humans make a decision it is not always possible to quantify the factors contributing to that decision, i.e. when the decision tree is not known. Neural networks have already found application in feature detection, pattern recognition, and signal analysis. A recent paper by Cook et.al. (16) uses a neural network to predict control conditions in particle board manufacturing.

Let us now consider possible applications in paper science. One model will be explored in some detail, namely that of formation. There are several measures of formation, most of which are a quantitative measure of mass density or light transmittance readings over an area. Another measure used is the Visual Uniformity Ranking of paper, which may be quantitatively determined using pair comparison techniques. In these comparisons the judges are asked to rank the sample based on their individual definition of visual uniformity. A quantitative measure of formation that exactly relates to Visual Ranking has not been found. Therefore, assuming that there are other, and as yet not quantified, factors involved in the judges decision is reasonable. For this reason the ability of a neural network to imitate the subjective decision of the judges was investigated.

A neural network has been simulated at the Institute of Paper Science and Technology on the RISC/6000 minicomputer. This program uses as input the 6400 points of the 80mm X 80mm light transmittance matrix obtained from the IPST Formation Tester. The hidden layer contains 100 nodes and the output layer contains 25. For software

considerations the 25 nodes of the output layer are related to the 1-25 (25 best) scale of Visual Ranking used by the judges. A Visual Ranking of say 12.5 is rounded to 13 and is represented by the vector

$$13 = [00000000000010000000000000].$$

The training set consists of offset samples. Six levels of formation are considered with two samples from each level. The Visual Ranking of these two samples is similar, but not necessarily equal. The 80 X 80 matrix is converted to a 6400 point vector. This vector is inverted for each of the twelve samples giving twelve additional members in the training set (24 total). The Visual Ranking for the "inverted" samples is set equal to its non-inverted counterpart. The inversion of the data points is equivalent to a physical inversion of the test sample, i.e. formation should be independent of physical orientation.

The results from this program are not yet available. Processing time is estimated at 35 hours on the RISC/6000. A preliminary network has been simulated and tested successfully. Sixty-four formation vectors (dimension 100) were taken from a typical offset 80 x 80 matrix. These vectors were assigned an arbitrary Visual Ranking. The purpose was to verify that the three-layer neural net program, and the back-propagation were working properly. The results were excellent. After the network was trained, each member of the training set was presented again. The neural network gave the correct answer for all sixty-four samples. The network was trained for 10000 iterations on each sample. The network probably converged to a solution well before the 10000th iteration. A simple test can be added to the program to determine if the network meets convergence criterion.

The long processing times involved in the previous example encourage us to look for ways to optimize the network. One way to do this is to reduce the number of inputs on the bottom layer. Certainly it is improbable that a human judge is concerned with light transmittance at a 1 mm<sup>2</sup> resolution. Preprocessing the raw data would reduce the number of nodes required on the bottom layer. Several types of preprocessing are being considered. Co-occurrence matrices (17) have been used to characterize formation in terms of textural properties. This type of analysis is based on the Spatial Grey Level Dependence Method. Haralick (18) has proposed a set 14 fourteen textural features that can be deduced from second-order statistics of the co-occurrence matrices. Most researchers (19-25) only consider the five most important ones, namely energy, contrast, correlation, entropy, and homogeneity. Utilizing this method would reduce the number of input nodes to 14 (or 5).

A second type of preprocessing considers the relationship between increasing areas of the sheet. A quantitative measure of this data might be any of the first-order statistics, such as the mean, standard deviation or coefficient of variation (%CV). This method is informally called the boxcar method. An 80 x 80 matrix might be processed in the following manner. First the %CV of all 6400 data points is calculated. This is the input for the first node of

the bottom layer. Next, the data is averaged together in 2 x 2 boxcars to give a 78 x 78 dimension matrix. The %CV is calculated and is used for the second input. This is repeated as many times as desired within the size limits of the original matrix. An 80 X 80 data matrix might give seven values for input to the bottom layer.

Another way to reduce the size of the network is to optimize the hidden layer. The nodes of the hidden layer self-organize to detect distinct features in the bottom layer. By looking at the connection weights between each node of the hidden layer and all of the nodes of the bottom layer, we can see which "features" are most important. Let's say that energy, entropy and contrast are being used for the input layer. For example, by examining the value in W1, we might learn that energy and contrast are very important while entropy is negligible. Also, we can see which, if any, of the hidden layer are detecting the same feature and are, therefore, redundant.

These optimization will significantly reduce the number of calculations, and greatly increase the learning time. Once the system is trained, i.e. once the values for W1 and W2 are determined, only one pass through the network is required for each input. The values of W1 and W2 are static and back error propagation is not required. Herein, lies another important advantage of neural network. The network can be hard-wired by converting the connection weights into electrical resistances. If so desired, preprocessing can be performed by a microcomputer or dedicated board, thus supplying input to a neural network circuit of reasonable dimensions. Input signals are then processed in parallel, significantly decreasing calculation time.

In conclusion, the basic theory of neural networks has been presented. An application in the area of paper formation has been investigated and preliminary results are excellent. A full-scale implementation is currently being tested. Neural networks provide us with a useful tool that despite appearances is surprisingly simple. Neural nets will find application in areas where relationships are too complex to quantify or where the decision tree is unclear and/or unknown.

#### 4. CONCLUSIONS AND RECOMMENDATIONS

A survey of the literature relevant to the performance of recycled pulps, particularly the role of recycled fines, has almost been completed. It seems reasonable to assume initially, that in seeking ways to enhance the papermaking properties of recycled pulps that the long fiber and fines fraction will benefit from separate and distinct treatments. Although the impact of fines on paper properties has received considerable attention, less is known about the impact of recycling, contamination, and the behavior of fines from different pulp sources. Specific strategies for the separate treatment of fines does not yet appear to have received attention.

## 5. FUTURE ACTIVITY

1. Establish methods for fines separation and characterization.
2. Determination of fines content a) before and after paper manufacture of selected paper grades, b) selected recycled furnishes and of c) production versus laboratory made papers.
3. Impact of fines (virgin fines, once dried-untreated, once-dried treated) on selected paper properties in combination with the following furnishes:
  - a) Fines free virgin fibers at a low and high level of refining
  - b) Fines free virgin curled and microcompressed fibers

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## APPENDIX 1.

### EFFECT OF PAPERMAKING VARIABLES ON FORMATION

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#### INTRODUCTION

The purpose of this contribution is to report on the impact of some papermaking variables on formation, we have recently examined using a formation tester which has the capability of making both optical and mass density measurements.

With the recent emphasis on improving the quality of paper and board products there has been a resurgence of interest in formation. Formation to many still remains a confused area. This is in part due to the fact that there is no universally agreed on definition of formation, although the coefficient of variation of mass density, i.e.,  $CV(W)$  has recently been proposed by Dodson (1) as a universal measure of formation. Furthermore, every maker of either off-line or on-line formation sensors has there own index or measure of formation.

Problems also arise when we try to directly relate formation measurements to the converting and end use performance of paper, an area which is still in its infancy. These relationships can be quite complex, but it has to be realized that in addition to formation other variables may be involved. For example we can show that sheet strength is very much dependent on formation, but there have been very few systematic studies (Seth (2)) involving other variables controlling strength, e.g., fiber geometry and strength, interfiber bonding, and grammage.

We have already mentioned measures or indexes of formation, and of course it would be nice to keep life simple with one universally agreed upon measure of formation, i.e.  $CV(W)$  as suggested by Dodson (1). This author believes that we can go a long way with this definition, and need to more thoroughly explore its possibilities. However, as Cresson (3) and others have pointed out, papers having identical  $CV(W)$ 's can have very different texture or pattern features, e.g. floc size distributions, to use the jargon of image analysis. Clearly, it would be nice to fingerprint a piece of paper with respect to its manufacturing process, i.e., twin wire, high turbulence headbox etc. and texture or pattern recognition techniques might be a realistic approach to this goal. Nevertheless, we have still to demonstrate how these additional measures of formation might be related to converting and end use performance of paper, e.g. coating and printing.

The early work of Corte and Dodson, and Herdman (4,5), and more recently Dodson (1), has shown that the coefficient of variation of mass density  $CV(W)$  of an ideal random network of fibers is related to a fiber length parameter  $k$ , fiber coarseness  $C$ , and average sheet grammage  $W$  as follows:

$$CV(W) = \sigma(W)/W = (k \times C/W)^{1/2} \quad (1)$$

It is perhaps surprising that equation (1) has not yet been completely verified. However, this is not a trivial matter since it is difficult to maintain constant forming conditions while changing the parameters contained in equation (1). In recent work Seth (2) has demonstrated that  $CV(W)$  is proportional to the squareroot of fiber coarseness, i.e., the weight/unit length of fiber. However, this result was based on light transmission measurements.

One useful feature of equation (1) is that it can be used to compare formation measurements made on sheets of different grammage or basis weight. In practice, sheet basis weight is increased by increasing headbox consistency, but this usually has a negative impact on sheet formation. However, equation (1) indicates that formation should improve with increasing basis weight.

In a further paper Dodson and Fekih (5) have modified equation (1) to account for fiber orientation. They have predicted that formation will deteriorate, i.e.,  $CV(W)$  will increase with increasing fiber orientation. This result appears to be true for Fourdrinier made papers as demonstrated by the work of and Svensson and Osterberg (7). This result seems counter intuitive, since one might expect to get more uniform coverage with increased fiber orientation.

Dodson (8) has also shown that network consolidation during forming and (wet pressing?) should lead to improved formation. Using his approach it can be shown that  $CV(W)_T$  as a function of consolidation is given by

$$CV(W)_T = ((CV(W))^2 + (1/1-e)(lw/x^2))^{1/2} \quad (2)$$

where  $e$  is the porosity,  $l$ ,  $w$  fiber length and width, and  $x^2$  is the aperture area. In the results which follow we will briefly examine the effects of refining, forming consistency, fiber orientation and wet pressing with reference to equation (1).

## EXPERIMENTAL

Formation measurements have been made on handsheets, formed either on a Noble and Woods Sheet Former, British handsheet mold, or on the Formette Dynamique, using the IPST Formation Tester, briefly described below.

The Formette dynamique gives a unique, but high level of formation depending on the furnish used. It supposedly comes closest to the ideal of forming a random network of fibers. It also has the possibility to form handsheets with a non-random fiber orientation.

After formation the sheets are wet pressed and dried under full restraint using the IPST press and dryer combination. With the sheets made on a British sheet mold TAPPI recommended procedures were followed.

The IPST Formation Tester is shown in Figure 1. It has the capability of making light transmission and reflectance measurements in the wavelength range of 400 - 700 nm, as well as beta particle absorption measurements. An aperture of 1 mm x 1 mm is used for both light transmission and beta measurements.

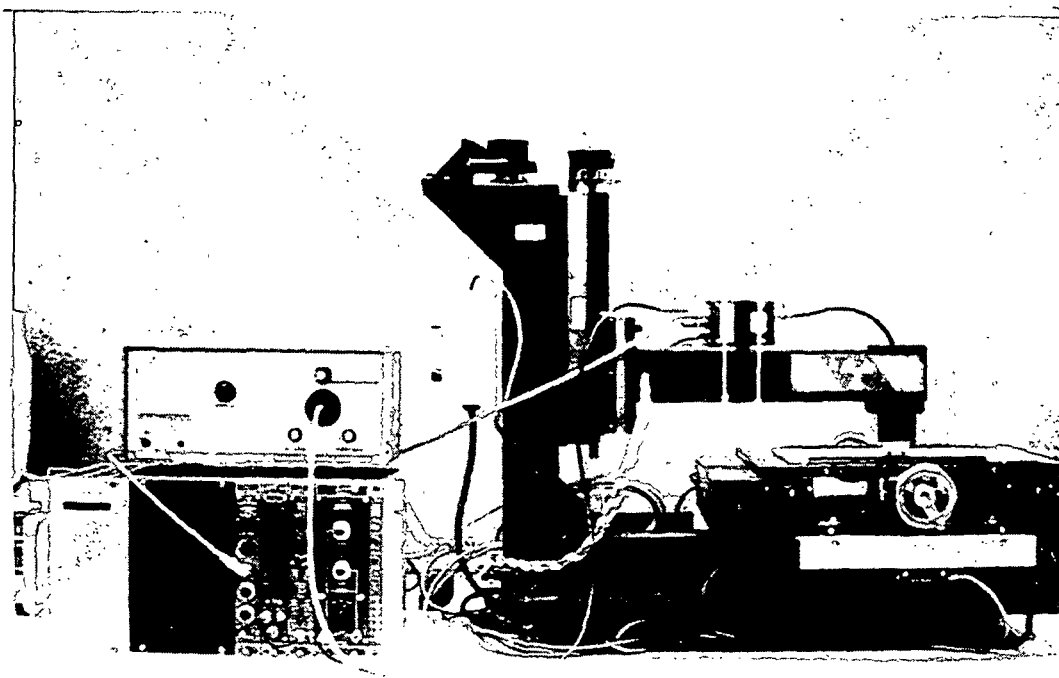


Figure 1. IPST Formation Tester

Formation samples of various sizes can be used, and are held in place on the x-y table by magnetic clamps. User friendly software has been developed for the x-y table to transport the sample, so that formation measurements can be made over the area selected.

The incident illumination is a 100-watt quartz halogen lamp transmitted through a fiber optic cable, and a bifurcated cable is used when reflectance measurements are made. The beta emitter used is a 50 mCi promethium source mounted on a 5 mm diameter aluminum disc with a 2 micron silver window (obtained from Amersham International).

Optical measurements can be made continuously, however, when mass density measurements are being made the discontinuous mode is used with delay times varying from 1 to 30 secs depending on the average grammage.

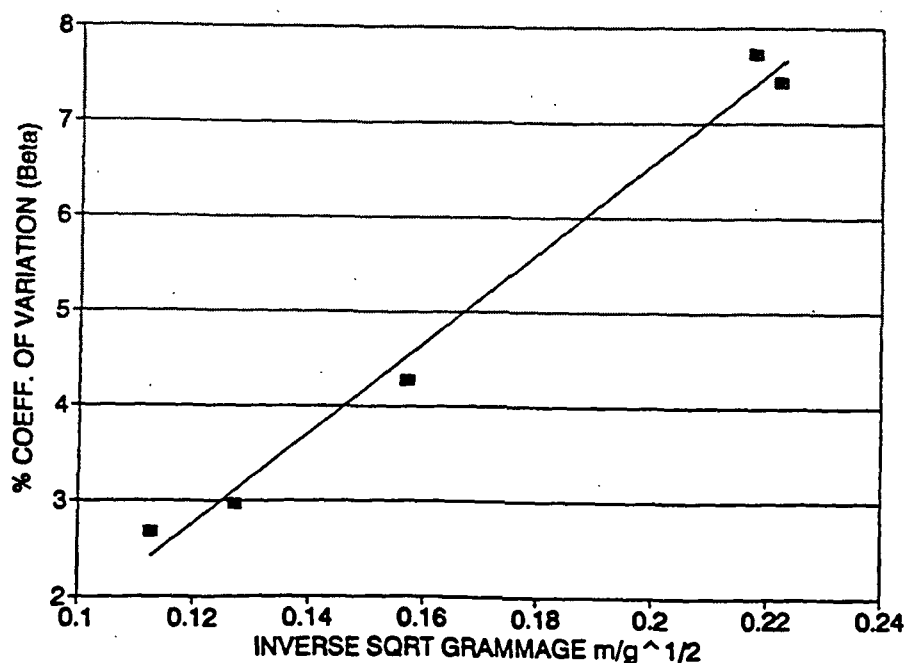
The objective in software development was to have a user-friendly system with a variety of options for formation measurement and information presentation, including CV(W), CV(T), FFT, Spatial Gray Level Dependence Method.

## RESULTS AND DISCUSSION

The coefficient of variation of mass density CV(W) with inverse square root grammage, i.e.,  $1/(W)^{1/2}$  is shown in Figure 2 for sheets made on the Formette Dynamique at a constant forming consistency of 0.3%. We see that there is good agreement with the prediction of equation (1). Figure 3 shows that as formation deteriorates, i.e.,

CV(W) increases, tensile strength decreases. When forming handsheets at constant grammage on the Formette Dynamique, we have found tensile strength to be invariant with forming consistency over the range of 0.1% to 2.0% (9).

Figure 2. Variation of Specific Tensile Strength with Coefficient of Mass Density CV(W)



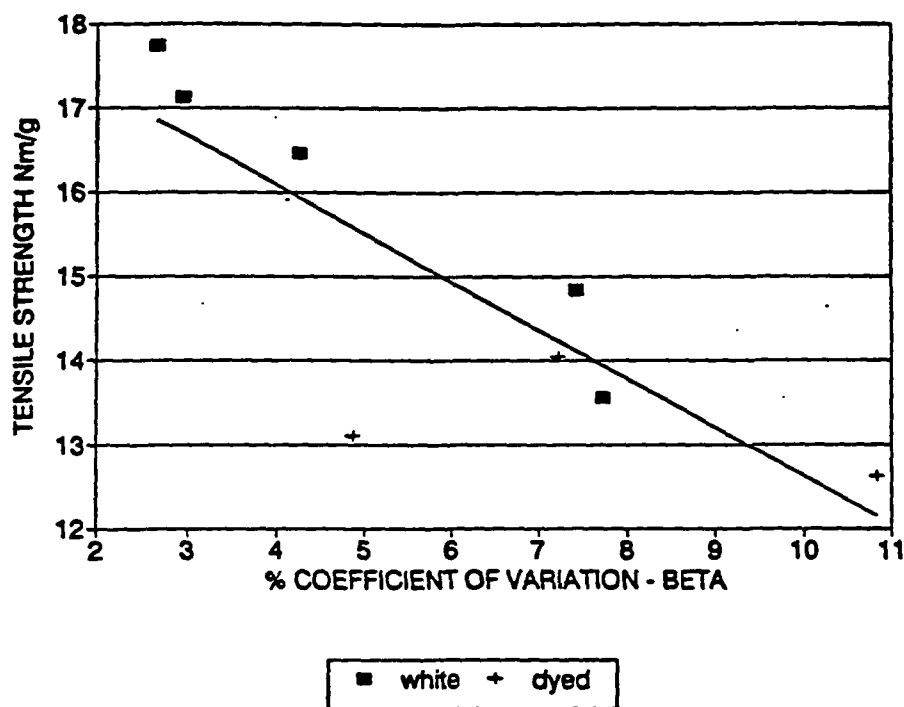


Figure 3. Variation of the Coefficient of Variation of Mass Density  $CV(W)$  with Inverse Square Root Grammage

The effects of furnish and refining on formation for a bleached eucalypti kraft and bleached northern softwood pulp are summarized in Tables I. The predicted values of  $CV(W)_{pred}$  given in the final columns are calculated using equation (1) and measured values of coarseness and fiber length. The latter was used to estimate the value of the fiber length factor  $k$  (5). In these experiments very little change in fiber coarseness and length was found with PFI refining.

Table I. Effects of Furnish and Refining on Formation

## BLEACHED EUCALYPT KRAFT

PFI Refining REVS	CSF ml	CV(T) %	CV(W) %	CV(W) <sub>pred</sub> %
0	580	2.79	2.58	2.05
500	476	2.87	2.60	1.98
1500	341	3.38	3.15	2.23
3000	134	3.24	2.66	2.15
5000	79	4.48	3.23	2.27

## BLEACHED NORTHERN SOFTWOOD KRAFT

PFI Refining REVS	CSF ml	CV(T) %	CV(W) %	CV(W) <sub>pred</sub> %
0	623	5.38	5.43	3.85
2500	406	5.49	2.60?	3.70
5000	224	7.78	5.14	3.64
7500	84	10.21	4.95	3.70
10000	41	25.27	15.63	3.61

As one would expect, on the basis of fiber length and coarseness, the hardwood sheets show better formation than the softwood sheets. It is interesting to note, that at the highest level of refining, the formation shows significant deterioration, particularly for the softwood handsheets. This is attributed to the longer drainage time which occurs at low freeness levels, i.e. allowing more time for flocculation to occur. It was found with this work, which was part of a class assignment in paper physics at IPST, that tensile strength also fell at the highest level of refining. There are a number of instances in the literature concerned with pulp evaluations where the tensile strength falls off at high levels of refining, and clearly formation could be responsible for this.

We note that even at the higher levels of freeness measured values of formation are higher than the predicted values. This is presumed to reflect a deviation from an ideal random network of fibers. We also see that as formation deteriorates, the difference between optical and mass density measurements increases.

The effects of different forming conditions are illustrated in Table II. In the first part of Table II the effects of delay time before sheet formation, addition of a cationic wet strength agent (kymene 557H), and forming consistency on elastic properties and their variability is shown. As expected, increases in these variables leads to a deterioration in formation. Interestingly, the average values of in-plane elastic properties are maintained, while there is an improvement in the out-of-plane elastic constant. However, there is a significant increase in the variability of those measurements, particularly for the out-of-plane elastic constant.

Also shown in Table II are results for Formette handsheets made using the same furnish for two levels of fiber orientation  $R (=E_{md}/E_{cd})$ . The Formette made handsheets are slightly better than the best formed Noble and Woods handsheets, but now we note a small improvement in formation with increased delay time. In the case of the Formette, the delay time is the time between network formation on the forming fabric and the initiation of drainage from the network. The improvement in formation may be due to increased fines retention, since there is a slight increase in grammage with the increase in delay time.

We note that there is no adverse effect of fiber orientation on sheet formation, and if anything formation is improved slightly with increasing fiber orientation, at least as judged by the light transmission measurements, i.e.,  $CV(T)$ . There is also a lower variability in both in-plane and out-of-plane elastic properties when the Formette handsheets are compared with the Noble and Woods handsheets. On the basis of these results, it appears that fiber orientation does not have an inherently adverse affect on formation. However, it is clear that in creating a non-random fiber orientation on the paper machine, i.e., by changing the difference in jet to wire speed, that this does have an adverse affect on formation.

Table II. Characterization of Handsheets having Various Levels of Formation

## NOBLE &amp; WOODS HANDSHEETS

CONSIST. %	t <sub>b</sub> secs	PAE %	GRAMMAGE g/m <sup>2</sup>	DENSITY g/cm <sup>3</sup>	CV(T)	CV(W)	V <sup>2</sup> (km/s) <sup>2</sup>	CV(V <sup>2</sup> ) %	V <sub>z</sub> <sup>2</sup> (km/s) <sup>2</sup>	CV(V <sub>z</sub> <sup>2</sup> ) %
0.025	0	0	59.2	0.738	8.88	6.94	8.36	5.1	0.145	8.9
0.025	0	0.6	58.3	0.765	16.93	11.14	8.64	5.2	0.141	9.9
0.025	5	0	61.1	0.795	17.82	12.51	8.28	4.4	0.159	15.0
0.025	5	0.6	67.9	0.800	22.00	15.61	8.74	4.9	0.161	20.6
0.250	0	0	61.3	0.741	29.14	18.09	8.05	9.6	0.167	22.6
0.500	0	0	65.0	0.799	70.4	30.78	7.74	12.4	0.167	31.5

## FORMETTE HANDSHEETS

CONSIST. %	t <sub>b</sub> secs	R	GRAMMAGE g/m <sup>2</sup>	DENSITY g/cm <sup>3</sup>	CV(T)	CV(W)	V <sup>2</sup> (km/s) <sup>2</sup>	CV(V <sup>2</sup> ) %	V <sub>z</sub> <sup>2</sup> (km/s) <sup>2</sup>	CV(V <sub>z</sub> <sup>2</sup> ) %
0.400	0	1.12	67.4	0.773	5.61	6.78	8.79	1.8	0.188	4.4
0.400	5	1.15	70.2	0.769	5.53	6.54	9.05	2.0	0.184	2.1
0.400	0	3.46	68.1	0.830	5.34	6.70	8.13	2.9	0.195	5.3
0.400	5	3.22	72.0	0.808	5.34	6.25	8.13	2.5	0.185	3.4

t<sub>b</sub> dwell time in headbox or delay time before drainage on FormetteR = (Vx<sup>2</sup>/Vy<sup>2</sup>)V<sup>2</sup> geometric mean in-plane elastic constantV<sub>z</sub><sup>2</sup> out-of-plane elastic constant or modulus

PAE Kymene 557



The impact of refining and wet pressing on the strength properties of paper was recently examined by Bither and Waterhouse (10). In addition we have examined the effects of network consolidation by refining and wet pressing on formation, and the results are summarized in Table III. The formation of the handsheets, which were made on the Formette Dynamique, is improved by refining and wet pressing. At a constant refining level sheet densification by wet pressing implies a reduction in void volume, and therefore an improvement in formation might be expected. The limit of course would be the densification of the sheet to a non-voidal continuum with a  $CV(W)=0$ .

Table III. The Effects of Refining and Wet Pressing on Formation

CSF ml	Wet-Pressing psig	Density g/cm <sup>3</sup>	CV(T) %	CV(W) %
-	0	0.300	6.86	5.21
741	40	0.810	5.72	4.72
155	0	0.791	4.49	4.00
157	40	0.880	5.38	3.70
270 Cl.	0	0.740	4.42	3.86
270 Cl.	40	0.874	5.36	3.67

We also note, that there is a small improvement in formation at constant density or void volume. This implies that the average pore size and its distribution must be different, and we have actually found this to be the case (10). Our formation results seem to be consistent with this idea. The reader might expect a similar trend to appear in the results given in Table I, i.e., as refining is increased formation should improve. In discussing Table I, we have already noted that no significant change in average fiber length or coarseness was observed, however, it is believed that the increased drainage time may be responsible for negating the above effect.

## CONCLUSIONS

The influence of a number of papermaking variables on sheet formation has been examined. It has been shown that the coefficient of variation of mass

density  $CV(W)$  is inversely proportional to the inverse square root of grammage as predicted by theory.

The formation of TAPPI handsheets can deteriorate at low freeness levels, due to an increase in drainage time, and may be responsible for the drop off in tensile strength, sometimes found in pulp beating evaluations.

Flocculation effects produced by a delay in drainage, cationic strength additives, and increased forming consistency, was found to have an adverse affect on formation, and although the mean values of in-plane and out-of-plane elastic constants were not changed, their variability increased significantly.

It was found, with handsheets formed on the Formette Dynamique (close to ideal forming conditions), that an increase in fiber orientation did not have an adverse affect on formation. Using the same forming method it was also found that formation improved through densification by refining and wet pressing.

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