THE EFFECT OF RELEASE LINER MATERIALS ON ADHESIVE CONTAMINANTS, PAPER RECYCLING AND RECYCLED PAPER PROPERTIES

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ABSTRACT

Release liner waste material is found in post-consumer waste streams and is also a significant component of the pre-consumer waste stream generated in the manufacturing of adhesive products. To date, very little has been reported pertaining to the behavior of release liner in paper recycling. In this study, the effect of the release liner material on the behavior of adhesive contaminants during laboratory pulping was investigated by dyeing the adhesives and the release liner with a blue dye and performing image analysis. The presence of release liner in the furnish caused a decrease in the size and an increase in the number of adhesive particles after pulping. The recyclability of the release liner was evaluated by processing a wastepaper furnish containing release liner and copy paper (no adhesive) through a laboratory pulping, screening, centrifugal cleaning and flotation processes. The release liner was removed with a laboratory screen and a laboratory centrifugal cleaner both with high efficiency but was not removed with a laboratory flotation device. The presence of residual release liner particles in recycled paper decreased the strength and printing properties of the recycled paper significantly.

INTRODUCTION

Release liner waste material is found in post-consumer waste streams and is also a significant component of the pre-consumer waste stream generated in the manufacturing of adhesive products. To date, very little has been reported in the literature pertaining to the behavior of release liner in paper recycling. Potential methods to deal with the release liner are incineration [1] and in the case of pre-consumer waste refurbishing and re-using the release liner rolls [2]. In general, release liner materials are considered to not be repulpable or recyclable with standard paper recycling technology [3]. However, a German patent has been awarded for a process utilizing pulping in the presence of a salt of monophosphoric acid esters to enhance the recycling of silicone-coated papers [4].

Adhesive contaminants are a serious problem in the paper recycling and paper making processes. Residual adhesive contaminants can deposit on equipment or cause defects in the final paper product. It is known that adhesive particles interact with other components in the wastepaper pulp and this changes the adhesive's properties [5,6,7]. Extensive work in this area has been performed at NCSU [6,7]. For instance, it was found for an acrylate pressure sensitive adhesive, PSA, applied to unsized paper that screening with a Pulmac Master Screen using 0.006 inch slots was able to remove 100% of the PSA from the pulp. However, when the same adhesive was applied to a rosin-alum sized, starch containing paper, screening was able to remove *only 95%* of the adhesive. Further, for the adhesive applied to the sized, starch containing paper, the PSA particles after pulping were significantly smaller and less tacky than the adhesive on the unsized paper. Later work showed that several components found in recycling systems can alter the size of adhesive materials after pulping [7]. In other work at NCSU, it was found that a very small addition of poly(vinyl alcohol) to a pulp containing PSA particles reduced the deposition of the PSA on a papermachine wire (PIRA Deposition Tester) by 97% relative to the untreated adhesive. These examples clearly demonstrate that when evaluating PSA products for recyclability, it is imperative to understand how the PSA contaminants interact with other species in the wastepaper furnish. Accordingly, it is necessary to understand how release liner Components, such as the release coating, affect the behavior of adhesives.

It has been observed in pilot plant trials at the USDA-Forest Service Forest Products, Laboratory that in some cases the presence of release liner material in the pulper causes the PSA to form smaller particles than when no release liner is present. This has serious implications due to the fact that screening, arguably the best method to remove PSA particles, is most effective in removing large particles. It is, therefore, of interest to better understand how release liner materials at pre- and post- consumer levels affect the behavior of adhesive particles. This paper further explores the efficiency of screening, flotation and cleaning on the removal of release liner particles and the effect that residual unremoved release liner particles have on the properties of recycled paper.

EXPERIMENTAL

Materials

The pressure sensitive adhesive and the release liner used in this study (sample #34252) were supplied by the Forest Product Laboratory. This sample was part of the Environmentally Benign Pressure Sensitive Adhesives for Postal Applications project [8]. The composition of the PSA was not reported. A pre-dyed acrylate-based pressure sensitive adhesive received from Avery Dennison Inc. was also used in this study as an adhesive with known composition. The Avery Dennison adhesive film was supplied between two release liners (no facestock present). The copy paper used in the study was Georgia-Pacific, Ardor Bond, A Premium #4 Writing Paper, White, 75g/m^2.

Procedures

Adhesive pre-dyeing procedure.
In some cases, pre-dyeing of the adhesive was performed prior to pulping. The pre-dyeing was according to the USPS Protocols for Recycling Evaluation of New Generation of Pressure Sensitive Adhesives [9]. The procedure follows. The adhesive release liner laminate was cut into 7.5” x 7.5” sheets and the release liner was removed. The adhesive face stock with adhesive side up was mounted on two sheets of 8” x 8” blotter paper, In a well-vented fume hood, the adhesive was flooded with about 15 mL of dye solution (0.067% Morplas Blue 1003 heptane solution) by applying it evenly across the surface of the adhesive. The dye solution was swirled so that an even blue color was developed on the adhesive as the solvent evaporated. An effort was made to minimize the amount of dye going over the edges of the adhesive and staining the face paper. The dyed adhesive/face stock was left in a hood overnight, and allowed to air dry overnight.

Handsheet post-dyeing procedure.
In some cases, handsheets were formed from undyed samples that contained adhesive and then dyed. The handsheets were dyed with 0.067 % Morplas Blue 1003 dye heptane solution at room temperature. Individual handsheets were swirled manually for 10 seconds in the dye solution and dried overnight. The next day the handsheets were rinsed consecutively by swirling in two baths of heptane to remove the dye stained on the handsheets. A 500 mL bath of the dye solution and two 500 mL baths of the heptane (approximately) were used for about 40 handsheets. The stickies and the release liner particles retained the blue color after rinsing. The handsheets were dried in a hood for 1 ~ 2 h.

Pulping.
Pre-dyed adhesive was evenly applied on the copy paper by hand. Paper, liner, and pre-dyed adhesive facestock were cut into 6 mm width strips with a paper shredder. The mixture, total weight 450 g, was soaked in 3300 mL of D.I. H_2O (12% consistency) at 40°C for 10 minutes and then poured into an Adirondack 450H High Consistency Laboratory Pulper equipped with a water jacket for temperature control. The furnish was pulped at 40 °C for 30 minutes at 350 RPM for each case. No pH adjustment was performed (pH = 8.2 before and after pulping). Five handsheets were produced for each sample using TAPPI standard method T 205 om-88.

Recycling of release liner and copy paper furnish.
Experiments were performed with 25% release liner from sample #34252 and 75% copy paper (no adhesive present) to (a) investigate the removal efficiency of the release liner using laboratory recycling procedures and to (b) determine the effect of the release liner on the properties of the recycled paper. The 25% release liner and 75% copy paper mixture was shredded and pulped in the same way as described above in the pulping section. The resulting pulp was screened with a Pulmac Master Screen with a 0.006 inch slotted barrier. A sample of 22.5 OD grams of the screen accepts were floated in a Wemco Laboratory Flotation Cell, 0.75% consistency, 3000 mL, room temperature, with 3 drops (0.08 g) DI 600 surfactant for 5 minutes. The foam was continuously scraped off the surface of the flotation vessel manually. Handsheets were made of the pulp stock after pulping, screening and flotation. The procedure was repeated in its entirety for copy paper alone (without release liner) and handsheets made for comparison. The handsheets were tested using TAPPI Standard methods. Also, some of the handsheets

were dyed according to the handsheet dyeing procedure appearing above and image analysis was performed on them to determine the removal efficiency of the screening and flotation processes.

The removal efficiency of release liner particles using centrifugal cleaners was also investigated using a continuos, laboratory centrifugal cleaner. The cleaner used, Dorr Oliver, Doxie Type P Cyclone, is two inches in diameter, 6.25 inches in height and has 0.5 inch openings for the accepts and rejects ports. A pulped (as above) sample of 25% release liner from sample #34252 and 75% copy paper (no adhesive present) was diluted to 0.14% and fed to the cleaner at a pressure of 30 psig. The accepts and rejects pressure were 10 psig and 0 psig, respectively.

Image analysis.

The Apogee Spec*Scan system was used to perform image analysis on handsheets using a HP ScanJet 4C flat bed scanner at 600 DPI. Image analysis was made on the 5 handsheets, front and back, 6-inch round, 0.275 m^2 total scanned area, using 0.02 mm^2 as the smallest speck counted. A value of 80% of the average gray scale value was used as the threshold to detect specks.

Printing performance evaluation.

The printing performance of paper was evaluated by printing a line using an HP DeskJet 712C printer with black ink. The width of the line was set to 3 points on Microsoft Word 97. The printed lines were examined under a microscope and analyzed using ImagePro software. The procedure was as follows. An image of the line was acquired under 5x magnification and two parallel measurement lines were drawn through the edges of the printed line. The parallel lines were placed at a location that was roughly covered by 50% ink and 50% uncovered surface. The distance between the two parallel lines represented the width of the printed line. The widths of lines printed on copy paper, the recycled copy paper and recycled paper consisting of 75% copy paper and 25% release liner were compared. In another method to evaluate the printing performance, a measured linear distance (S) of 1.5 mm was arbitrarily chosen on one of the parallel lines. Then a trace at 10x magnification along the contour of the dark areas of the printed edge was performed and the length of the trace line (T) was determined. The ratio of TIS provided a measure of the roughness of the edge of the printed line, higher values of T/S indicated poorer printing performance.

RESULTS AND DISCUSSION

Interactions of Release Liner and Adhesive Particles in the Pulping of Model Post-consumer Furnishes

In pilot plant experiments at the USDA-Forest Service Forest Products Laboratory it had been observed that the presence of release caused the adhesive particles to break down in the pulper into smaller particles relative to the case in which no release liner was present. However, these observations were made after inspecting dyed handsheets containing both adhesive and release liner particles. As will be shown later in this paper, release liner particles adsorb dye and are detected in image analysis, as are adhesive particles, complicating the image analysis results.

Thus, in order to verify whether or not the presence of release liner causes the adhesive particles to be smaller, pre-*dyed* adhesive (2.5%) was pulped with and without the presence of (undyed) release liner (10%) with the balance of the stock being copy paper. This ratio of furnish was intended to be a model for post-consumer recovered paper. It is acknowledged that the levels of adhesive and release liner are higher than typical, however, these levels were chosen to magnify the effects that could be observed.

In this case, image analysis on the resulting handsheets only detected the pre-dyed adhesive particles, not the undyed release liner particles. The results show that the number of particles after pulping increases, the average size of the particles decreases and the median size of the particles decreases in the presence of release liner **(Table 1),** This is the case for both the adhesive sample #34252 and for the acrylate-based Avery Dennison adhesive. The practical implication of this size reduction on industrial screening efficiency of the adhesives is expected to be negative.

Also shown in **Table 1** are data from handsheets containing the same amount of adhesive sample #34252 (2.5%) and release liner (1 0%) and copy paper (87.5%) but without pre-dyeing of the adhesive. In this case, handsheets of the pulped mixture were dyed, allowing both adhesive and release liner to adsorb the dye. The large effect that this procedure has on the results is noteworthy, causing the number of specks to increase, the average particle size to decrease, and the median particle size to decrease. These changes are due to the release liner being broken down into smaller particles than the adhesive, adsorbing the dye, and being recognized by the image analysis system as contaminant. This phenomenon demonstrates a weakness of the dyeing procedure, i.e., the inability of the procedure to distinguish between stickies and other dye adsorbing contaminants.

lnteractions of Release Liner and Adhesive Particles in the Pulping of Model Pre-consumer Furnishes

It was of interest to investigate the interactions of adhesive and release liner at higher levels in the pulping furnish, such as might be found in "pre-consumer" waste. Three types of furnishes were processed: case (I) 25% adhesive/25% release liner/50% copy paper, case (2) 25% adhesive/75% copy paper and case (3) 25% release liner/75% copy paper. After pulping, the stock was either screened using a Pulmac Masterscreen or subjected to a deposition tester. The handsheets or paper machine wires from the deposition test were then dyed and image analysis performed (deposition data not shown here).

Figures 1 and Figure 2 show scanning electron microscope (Hitachi S3200N SEM) pictures of adhesive # 34252 with release liner (case 1) and without release liner (case 2). Particles of the release liner are observed to attach to the surface of the adhesive particle in **Figure 1.** The adhesive particles with release liner attached is shown to be flatter or two-dimensional than the adhesive particle without release liner attached in **Figure 2,** which appear to be' "rolled-up" and more three dimensional. From inspection of many samples, the attachment of release liner particles to adhesive in the "pre-consumer" experiments was found to be common.

The presence .of the release liner in the pulper at "pre-consumer" levels has a significant effect on the final characteristics of the adhesive. **Table 2** displays the PPM, number of spots and average particle size of the adhesives and release liner in the handsheets. The adhesive in this case was not pre-dyed; handsheets were post-dyed after their formalion. By subtracting the PPM and number of specks in cases 2 and 3 from case I, the difference indicates the effect that the release liner has on the adhesive when they are pulped together. If this difference is equal to zero, then no interaction exists. It is shown in **Table 2** that the effect of pulping the adhesive in the presence of the release liner increases the adhesive PPM by 2 1,750 and the number of specks by 1,800 relative to what would be expected if there were no interaction during between adhesive and release liner. Thus, the release liner interacts with the adhesive, generating more adhesive particles. This is in agreement with the results of the pre-dyed experiments that show increases in the number of adhesive particles present after pulping due to the release liner presence, **Table I.**

Further, two other adhesive constructions, samples #34255 and #34286, at the same pre-consumer furnish ratios showed similar behavior (data not shown) to that in **Table 2** for sample #34255. This demonstrates that the phenomenon is not unique to a single type of adhesive construction.

In related work [7] it was found that materials that detackify the adhesives (such as talc) enhance adhesive breakage in the laboratory pulper. It was hypothesized that the detackified adhesive particles remain extended and susceptible to breakage in the high-shear fields of the pulper. Adhesive particles that are not detackified self-adhere and fold, forming a more spherical shape that is harder to break. The representative images in **Figures 1 and 2** illustrate this hypothesis. It is suggested that the release liner particles coat the adhesive making it less tacky and more extended, such as observed in **Figure 1.** This extended state eventually leads to more breakage. In **Figure 2,** in the absence of release liner, the adhesive particles are observed to be folded and thus less susceptible to breakage.

Laboratory Recyclability Study of Release Liner Material

Laboratory recycling experiments were used to indicate the removal efficiency of the release liner material during the screening, flotation, and cleaning processes. A sample of 25% release liner from sample #34252 and 75% copy paper (no adhesive) was pulped as described in the experimental section, screened and floated. The image analysis results of dyed handsheets are shown in **Table 3.** The pulp stock PPM values decreased from 5,740 to about 140 PPM after screening, indicating an effectiveness of the screen to remove the release liner particles. Flotation, however, was not effective at removing the residual release liner particles in the screen accepts, resulting in a negligible decrease in PPM, **Table 3.** This inefficiency may be due to a number of reasons including inappropriate particle size, attachment of fibers to the release liner coating, a high density of the release liner particles, or a non-conducive contact angle with air. The particle size of the screen accepts is approximately 0.060 mm^2 or an equivalent diameter of 140 microns, which is, in general, considered too large for flotation. Also, by weighing and measuring the dimensions of the release liner paper, it was found that the density was 1.13 g/cm^3, significantly higher than the copy paper used in this study, 0.72 g/cm^3. Thus, the attachment of the release liner to the air bubble might be disrupted by gravitational or inertial forces. By microscopic inspection, it was observed that the release liner particles contained fibers. It is known that the attachment of fibers to toner significantly decreases the flotability of the toner particles [10].

It was also observed that the release liner particles sank in water. This observation, coupled with the density of the release liner product being equal to 1.13 g/cm^3, suggested that centrifugal cleaning would be able to remove the release liner contaminant particles from the pulp. A pulped 25% release liner #34252 and 75% copy paper furnish at 0.14% consistency was processed through a laboratory centrifugal cleaner. The PPM decreased from 5740 in the feed to 195 in the accepts **(Table 3).** The cleanliness efficiency (= 100% * $[PPM_f - PPM_a]/PPM_f$) was determined to be 97%, where a and f indicate accepts and feed, respectively. The reject efficiency (=100% • $[PPM_f \bullet Flow_f \bullet \%K_f - PPM_a \bullet Flow_a \bullet \%K_a]/PPM_f \bullet Flow_f \bullet \%K_f$) was determined to be 99%, where flow and %K indicate the volumetric flowrate and consistency, respectively. The solids yield of this laboratory process was 44%, a rather low yield for industrial operations. Despite the yield, these results indicate that the release liner particles are readily rejected in centrifugal cleaning operations.

Effect of Release Liner on the Properties of Recycled Pulp

The properties of handsheets made from the recycled 25% release liner and 75% copy paper (no adhesive) after screening and flotation were evaluated **(Table 4).** It was known from the experiments described above that this pulp had a measured contamination level of 140 PPM of release' liner particles. These particles are not visible unless dyed. Handsheets from 100% recycled copy paper after screening and flotation were also evaluated as a baseline **(Table 4).** It was determined that the recycled paper containing release liner had 12% lower tensile strength index, 7% lower tear index, and 38% lower burst strength than the recycled copy paper.

The recycled paper with release liner had a rougher surface than the recycled copy paper. For example, the slide angle increased by 9% and the Parker Print Surface Roughness increased by 6% to 10%, depending on the testing pressure. A wax pick test indicated that the surface strength of the paper was reduced significantly. This, along with the lower strength properties, suggests that fiber-fiber bonding was disturbed by the presence of the release liner material. This decrease in fiber-fiber bonding is also reflected in a lower density and higher air permeability. The decreased strength, increased roughness and decreased fiber-fiber bonding are expected to produce paper with poorer printing performance.

To evaluate the printing performance, image analysis was performed on ink jet printed lines on the recycled release liner containing paper, the recycled copy paper, and the copy paper as received **(Table 5).** The measured width of the line printed is larger for the recycled release liner containing paper than for the recycled copy paper or the copy paper as received. This is due to the ink spreading away from the intended printed surface area more for the recycled release liner containing paper. Further, the measured roughness of the edge of the printed lines (T/S) was also greater for the recycled release liner paper (due to increased wicking) than the recycled copy paper and the copy paper. It is likely that the decreased density, increased porosity, decreased surface smoothness, and decreased surface strength of the recycled paper containing release liner reduces the printing performance.

Industrial Implications of Research

- It has been shown that release liner material can interact with adhesives, resulting in smaller particle sizes in a laboratory pulper. It is suggested that mills with a lack of line screening or other methods to deal with stickies be sensitive to the amount of release liner in the incoming furnish.
- Residual release liner particles in recycled pulp can be responsible for degraded strength and printing properties. These release liner particles should be tracked just like any other contaminant.
- Mills receiving release liner material can use screening and cleaning but not flotation as effective removal methods.

- The release liner particles are white and not visible in bright recycled pulps. A simple dyeing method can be used to enhance the contrast between the background and the release liner particles.
- The dyeing procedure stains both adhesive and release liner, Caution must be exerted in using the dyeing technique to distinguish adhesive contaminants in systems that may contain release liner.

CONCLUSIONS

The presence of release liner causes a decrease in the size and an increase in the number of adhesive particles in pulped models for both pre- and post consumer wastes. The release liner particles in white pulp or paper can not be detected by conventional image analysis methods but can be dyed with a common dyeing agent used to also detect adhesives. The release liner could be removed with a laboratory screen and a laboratory centrifugal cleaner with high efficiency but could not be removed with a laboratory flotation device, The presence of the release liner particles in recycled paper decreased the strength and printing properties of the recycled paper.

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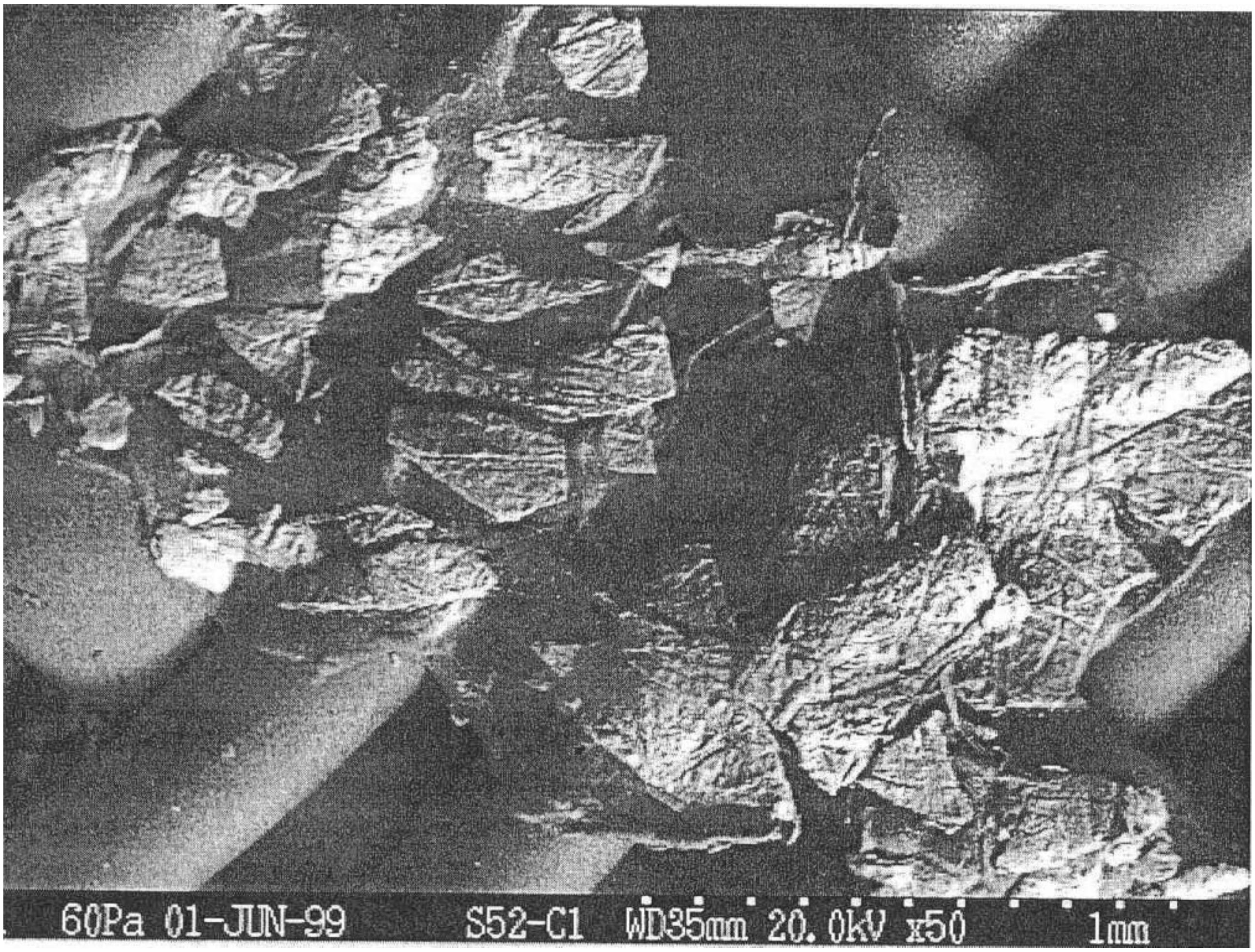

Figure 1. SEM picture of adhesive pulped in the presence of release liner (case 1).

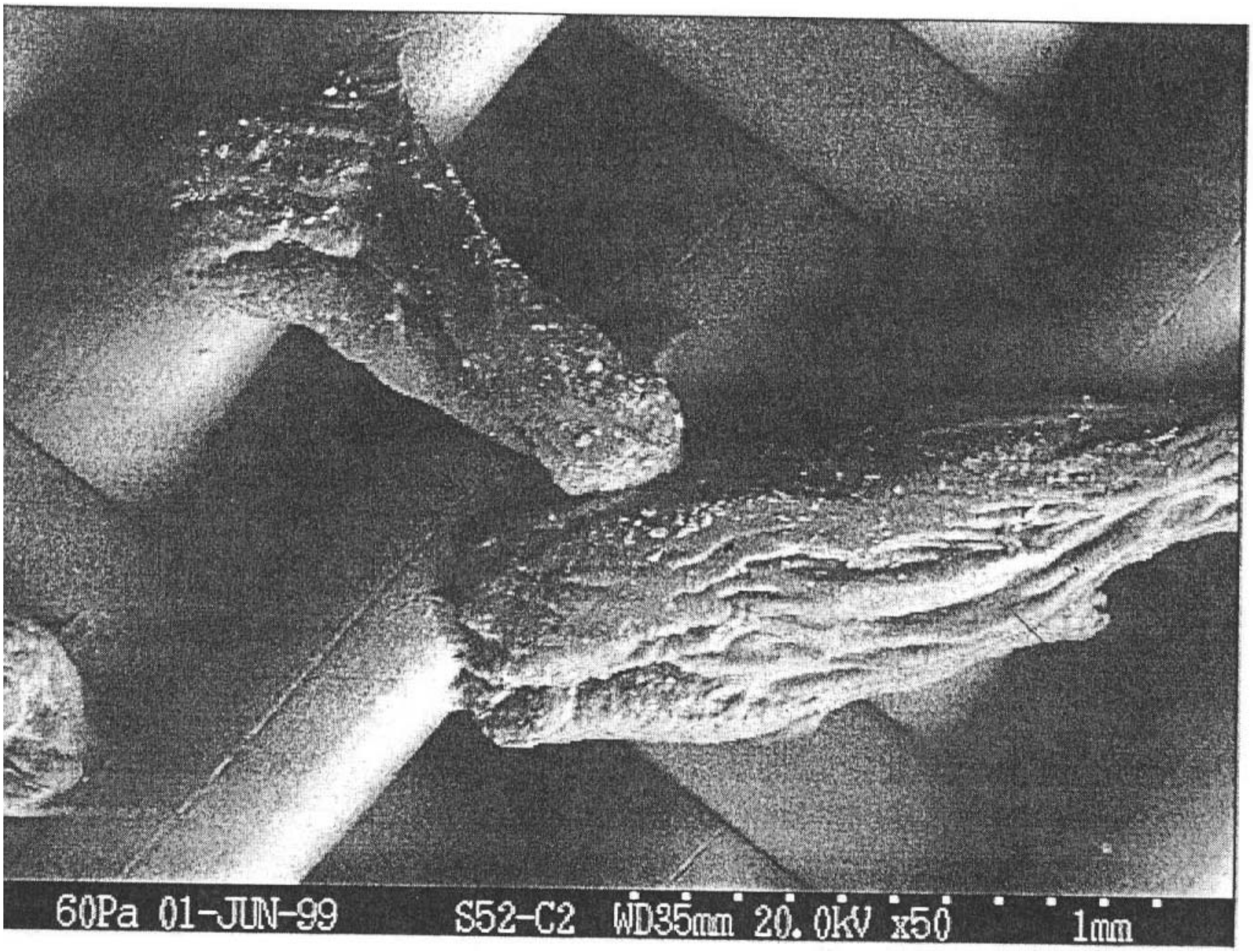

Figure 2. SEM picture of adhesive pulped without release liner present (case 2).

Table 1. Image Analysis of Pre-dyed Samples (Post-Consumer Model)

Sample	Handsheets		
	# of specks	Avg. size mm^2	Median. size m^2
2.5% Adhesive #34252 **97.5% Copy Paper**	416 437 Avg = 425	1.28 1.36 Avg = 1.32	0.87 0.99 Avg = 0.93
2.5% Adhesive #34252 **10% Release Liner #34252** **87.5% Copy Paper**	472 515 Avg = 494	1.05 1.00 Avg = 1.02	0.60 0.51 Avg = 0.56
2.5% Avery Denison Adhesive **97.5% Copy Paper**	227	1.32	0.09
2.5% Avery Denison Adhesive **10% Release Liner #34252** **87.5% Copy Paper**	293	1.17	0.08
2.5% Adhesive #34252 **10% Release Liner #34252** **87.5% Copy Paper** ***Handsheets containing release liner dyed**	1,557 1,662 Avg = 1,610	0.62 0.52 Avg = 0.57	0.17 0.13 Avg = 0.15

Table 2. Image Analysis of Post Dyed Samples (Pre-consumer Model)

Pulped Furnishes		PPM	# of specks	Avg. size mm^2
25%A/25%RL/50%C[a)]	Case 1	55,720	7,000	1.10
25%A/75%C	Case 2	28,230	2,271	1.71
25%RL/75%C	Case 3	5,740	2,934	0.269
	Case 1-2-3	21,750	1,795	–

a) A = adhesive, RL = release liner, and C = copy paper

Table 3. Recycling Results of Furnish Containing 25% Release Liner and 75% Copy Paper

	PPM	# of specks	Avg. size mm^2
After pulping	5,740	2,930	0.269
Screen accepts	139 146 Avg = 142	340 335 Avg = 338	0.056 0.060 Avg = 0.058
Screen/Flotation accepts	136 132 Avg = 134	286 304 Avg = 295	0.065 0.060 0.062
Centrifugal cleaner accepts	195	297	0.090

Table 4. Physical Properties of Recycled Paper

Sample	Recycled Copy Paper	Recycled Copy Paper (75%) and Release Liner (25%)
Density	0.536 +/- 0.024 g/cm^3	0.501 +/- 0.018 g/cm^3
Basis Weight	58.6 +/−2.6 g/m^2	61.1 +/- 2.2 g/m^2
Thickness	4.3 +/- 0.1 mil (inch x 10^{-3})	4.8 +/- 0.1 mil (inch x 10^{-3})
Tensile Strength	Strength, 1.99 ± 0.10KN/m Breaking length, 3.46 ± 0.17Km Strength Index, 33.9 ± 1.7 N*m/g	Strength, 1.83 ± 0.15KN/m Breaking length, 3.06 ± 0.18Km Strength Index, 30.0 ± 1.4N*m/g
Tear Strength	Tear Index, 2.88 ± 0.25mN*m^2/g	Tear Index, 2.68 ± 0.20mN*m^2/g
Burst Strength	Wireside, 2.32 ± 0.12Kpa* m^2/g Topside, 2.35 ± 0.12Kpa*m^2/g	Wireside, 1.48 ± 0.04Kpa* m^2/g Topside, 1.41 ± 0.05Kpa*m^2/g
Wax Pick	Wireside, 9 Topside, 6	Wireside, 7 Topside 3
Gurley Air Permeability (Second)	3.6 +/−0.3	1.5 +/- 0.2
Sheffield Precionaire Test	Wireside, 279 ± 4 Topside, 349 ± 3	Wireside, 311 ± 4 Topside, 360 ± 4
Slide Angle	22 +/- 1	24 +/- 1
Parker Print Surface (PPS) Test Roughness (micro meter)	Wireside 0.5 Mpa 6.93 ± 0.10 1.0 Mpa 6.68 ± 0.04 2.0 Mpa 6.20 ± 0.13 Topside 0.5 Mpa 10.39 ± 0.14 1.0 Mpa 8.80 ± 0.20 2.0 Mpa 7.47 ± 0.09	Wireside 0.5 Mpa 7.46 ± 0.14 1.0 Mpa 7.12 ± 0.20 2.0 Mpa 6.75 ± 0.09 Topside 0.5 Mpa 10.58 ± 0.19 1.0 Mpa 8.99 ± 0.08 2.0 Mpa 7.65 ± 0.20

± indicates 95% confidence intervals of testing results

Table 5. InkJet Printing Results of Recycled Paper

	Recycled Copy Paper		Recycled Copy Paper (75%) and Release Liner (25%)		Copy Paper	
Width, mm	Wireside,	1.26 ± 0.01	Wireside,	1.30 ± 0.01	Wireside,	1.11 ± 0.01
	Topside,	1.28 ± 0.01	Topside,	1.29 ± 0.01	Topside,	1.10 ± 0.01
Ratio of T/S line	Wireside,	1.51 ± 0.07	Wireside,	2.27 ± 0.20	Wireside,	1.29 ± 0.06
	Topside,	1.85 ± 0.24	Topside,	2.48 ± 0.18	Topside,	1.32 ± 0.04

± indicates 95% confidence intervals of testing results.

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