DEPOSITION ANALYSIS OF PRESSURE SENSITIVE ADHESIVES

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ABSTRACT

In this study, a set of pulps containing different PSA products that were recycled at the Forest Products Laboratory (FPL) pilot plant were analyzed for deposition using the PIRA deposition device at North Carolina State University. The work is part of the United States Postal Service Environmentally Benign Pressure Sensitive Adhesive effort. The deposition device consists of a counter-rotating paper machine wire immersed in a low consistency pulp suspension. The contrast between the deposited PSA and the paper machine wire was increased by selectively dyeing the PSA deposited on the wires. Image analysis was performed on the wires to provide a value of the overall ppm, average size, and size distribution of deposits on the wire. The same pulps were dyed with an adhesive specific dye to increase the contrast between adhesive and fiber and image analysis was performed on handsheets at the FPL. The deposition results were found to correlate in general with image analysis results of the same pulp.

INTRODUCTION

The measurement of stickies in pulp is a key issue in paper recycling [1]. There are many different measurement techniques but none have been accepted as a standard method. In this paper two of these techniques will be correlated.

The concentration of stickies material in a pulp can be measured by selectively dyeing the adhesive and then performing image analysis on handsheets made from the pulp. This method is especially convenient when no other dark contaminant exists in the pulp. This can be the case in model labotatory or pilot plant experiments in which the system components are controlled. However, for industrial samples containing dark contaminants that are not stickies, image analysis must be performed of the same pulp with and without dyeing. If the undyed adhesive is not in contrast with the pulp fibers, and if the dye selectively darkens the adhesive only, then the difference in image analysis results between dyed and undyed samples provides a measure of the concentration of stickies.

This image analysis method depends on the assumption that sticky material will be selectively dyed. This assumption should be verified for any study in which it is invoked. It is possible using this method that some sticky materials will not be dyed and some non-sticky materials will be dyed. To check this assumption, manual inspection of the dyed and undyed contaminants with a stereo microscope and a probing pick can be performed. Deformation of the contaminants with a pick reveals using a microscope if the contaminant is tacky, soft, and elastic. This method can be tedious.

Another method that could be used to check the above mentioned assumption is to expose the pulp containing suspected sticky particles to a deposition surface and analyze the deposition surface for adhering contaminants. This technique ensures that all material counted in image analysis is sticky (with respect to the deposition surface). A drawback with the deposition technique is that the experiment is more complex and time consuming than simple image analysis of the dyed pulp. Also, the results are very sensitive to the deposition conditions so that great care must be taken in keeping the experimental conditions the same amongst tests with different samples.

A deposition tester that has been reported in the literature as useful has been developed by PIRA [2-4]. The apparatus consists of counter-rotating a paper machine wire in a volume of low consistency pulp at a controlled temperature. In one report [2] using this apparatus it was found that pulping of pressure sensitive adhesive (PSA) containing paper at high pH (relative to neutral or low pH) produced more sticky particles that subsequently deposited. The subsequent pH condition during deposition testing did not have a significant effect on amount of deposition. In the same report, the addition of calcium chloride to give a water hardness of 30 ppm was found to decrease the deposition by about 70%. It was also found that a paraffin solvent based cleaner could increase deposition significantly under certain conditions. The same researchers reported [3] that when the deposition tester was used at a woody newsprint mill that the tester showed no deposits even though a large amount of stickies were known to be present. At a wood-flee tissue mill, stickie deposition was found to correlate with contamination levels. In a recent report [4], it was shown that the deposition of an acrylic pressure sensitive adhesive was significantly greater at 50°C than at 25°C.

In a study of secondary stickies with a device similar to the PIRA deposition tester, it was found that a maximum in deposition of a hydrodispersible pressure sensitive adhesive occurred when a theoretical amount of cationic polymer was added to the neutralize anionic charge in the water phase of the pulp [5].

In this report, several pulps containing pressure sensitive adhesive materials that were processed in a pilot plant at the Forest Products Laboratory (FPL) as part of the United States Postal Service Environmentally Benign Pressure Sensitive Adhesive effort [6] were tested using a PIRA deposition device at North Carolina State University (NCSU), The results from the deposition device were correlated with image analysis results of dyed handsheets made from the same pulp at FPL. The objective of the work was to determine if the handsheet image analysis results correlate with deposition measurements.

EXPERIMENTAL

Materials

Several pulps were supplied to NCSU by the FPL to be tested for deposition. The samples were copy paper containing different experimental pressure sensitive adhesives for evaluation in the United States Postal Service Environmentally Benign Pressure Sensitive Adhesive research. Some samples contained the release liner of the PSA while other samples did not. The samples had been processed in a pilot plant using high consistency pulping, pressure screening, forward and reverie cleaning, washing and flotation. The samples arrived at NCSU at approximately 40% consistency in sealed plastic bags. The samples were refrigerated until use.

In order to understand the relationship between observed deposition results and content of stickies material in a pulp, a series of model pulps with known amounts of adhesives was created. The pulps were made using softwood market pulp and an actylate based pressure sensitive adhesive film alone. No release liner or adhesive paper stock was included in the pulp. In the preparation of the separate samples, weighed amounts of the PSA from 0.0625% to 1% by weight of OD fiber was pressed by hand between the two sheets of the market pulp. The paper-PSA composite was pulped in a Tappi British Disintegrator for five minutes at 3000 rpm, 3% consistency, 50°C and neutral pH.

Deposition Procedure

The deposition tester included a stainless steel holding frame that held a paper machine wire (supplied by Weavexx, CFM-354). The wire was of polyester type and was different on the top and bottom. The frame and wire counter-rotated at 1 Hz in a pulp stock for 30 minutes. The pulp stock was contained in a stainless steel beaker that was placed in a constant temperature water bath to maintain the pulp stock at 50°C. The pulp stock had a volume of 8000 ml and a consistency of 0.5%.

After the deposition procedure, the wire was removed and rinsed gently with cool tap water. The wires were then air dried and weighed. The wires were then treated with a solution containing Drew Blue Stickie Dye to enhance the contrast of the adhesive with the paper machine wire. Initially, 100 ml of a solution of 1 part Drew Blue Stickie Dye and 9 parts ethanol were added to 1500 ml of de-ionized water. The paper machine wire was immersed in this mixture at 65°C for 15-30 minutes. The adhesive picked up the blue dye while the paper machine wire remained white. After dyeing, the wires were air-dried. The wires were then placed in a sealed plastic bag and image analysis was performed. Image analysis was performed using the Apogee Spec*Scan Image Analyzer and a Hewlett Packard ScanJet 4C flatbed scanner operating at 400 DPI using 256 gray scale values (GSV). The same 1.5 by 4 inch rectangular area of the wire was scanned for all samples. A threshold for contaminant detection was set at 135 GSV. The number of deposits, the average deposit size, the area of wire covered by deposits in PPM, and the size distribution of deposits were obtained.

RESULTS AND DISCUSSION

Deposition Results for Pulps with Known PSA Concentration

The number of deposits and the ppm of deposits for the pulp samples with known mass fraction of acrylate PSA are shown in Figures 1 and 2, respectively. The relationship between the number of deposits and mass fraction of PSA is linear with a correlation coefficient (R^2) greater than 0.8. The same was found for PPM vs. mass fraction of PSA.

The test at 2500 ppm mass fraction of PSA (i.e., 0.25% mass fraction) was performed four separate times to determine the variability of the test results. The average (AVG) number of deposits was 390, the standard deviation (SD) was 170, and the relative error, calculated as 100%*SD/AVG, was 45%. The average area covered by deposits in PPM was 6970, the standard deviation was 2770, and the relative error was 40%.

These results show that the variability of the deposition test is large. This variability is expected, as the final result is a function of several steps (weighing out adhesive and paper, disintegration and transfer, deposition, dyeing, image analysis). Also, it was observed that the deposits distubute themselves somewhat randomly on the paper machine wire. However, it was observed that more deposits were found toward the outside of the frame and where the edge of the frame contacted the wire. Due to the observed edge effects (and colored wire markings from the manufacturer on parts of the wire) only a portion of the wire was used for image analysis. The somewhat random distribution of particles inside and outside of the analyzed area contributed to the variability of the results. The variability of the results should be kept in mind when comparing different samples.

Deposition Results for Pulps with Experimental PSA

Pulps supplied to NCSU containing the experimental PSA products were evaluated using the deposition tester. These pulps had all been processed using the same extensive pilot plant recycling procedure. At the FPL, handsheets had been made of the product pulp after dyeing and image analysis performed. The deposition results were plotted vs. the FPL image analysis results of handsheets.

The number of deposits and the ppm of deposits in the deposition test vs. the handsheet image analysis results from the FPL are shown in Figures 3 and 4, respectively. The results show that the FPL image analysis results correlate well with the deposition results. Keeping in mind the variability of the deposition tests as shown with the pulp containing the known 0.2% acrylate PSA, the scatter of the data is appropriate. The results in Figures 3 and 4 support the assumption that the selective dyeing procedure used by the FPL is able to identify stickie material in the pulps. (It should be noted for Figure 4 that it is not expected that the deposition PPM should be equal to the image analysis determined PPM in the corresponding hartdsheets. The two values are related but should not necessarily be equal.)

The deposit counts for size ranges with equivalent diameter (D) of D<80microns, 80<D<225 and D>225 are plotted in Figures 5, 6, and 7 vs. the FPL image analysis results, respectively. For all three size ranges, the number of deposits increases with the FPL image analysis PPM results.

The data in this report includes all of the tested commercial PSA samples to date. More samples will be evaluated in the same reamer and included in a future report.

CONCLUSIONS

In this study, pulps containing experimental pressure sensitive adhesive materials were examined using two methods to determine sticky content. A correlation was found between image analysis results of dyed handsheets and deposition tests using a PIRA deposition tester. The correlation indicates that the image analysis of dyed handsheets is selectively dyeing sticky material and is an appropriate method for measuring sticky content.

REFERENCES

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6. Proceedings of the Environmentally Benign Pressure-Sensitive Adhesives for Postal Applications, July 7-9,1997.

Fig. 1. Number of Deposits vs. Mass Fraction of PSA in Pulp

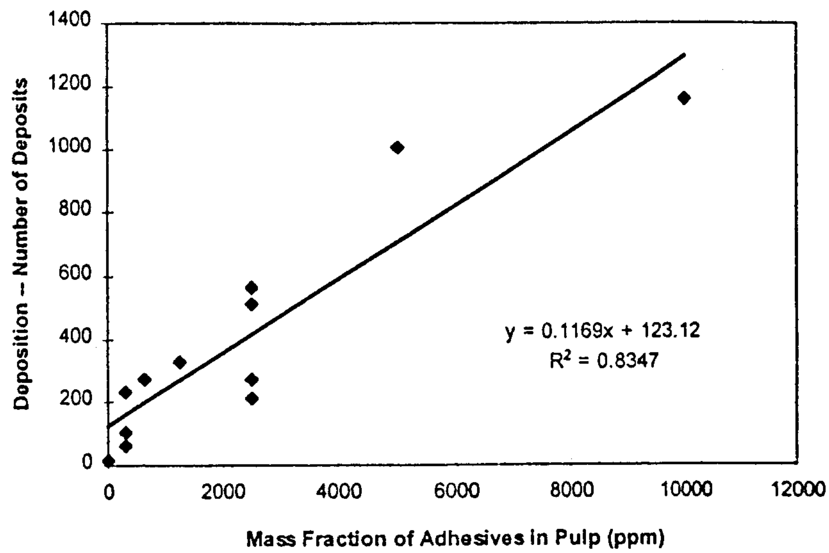
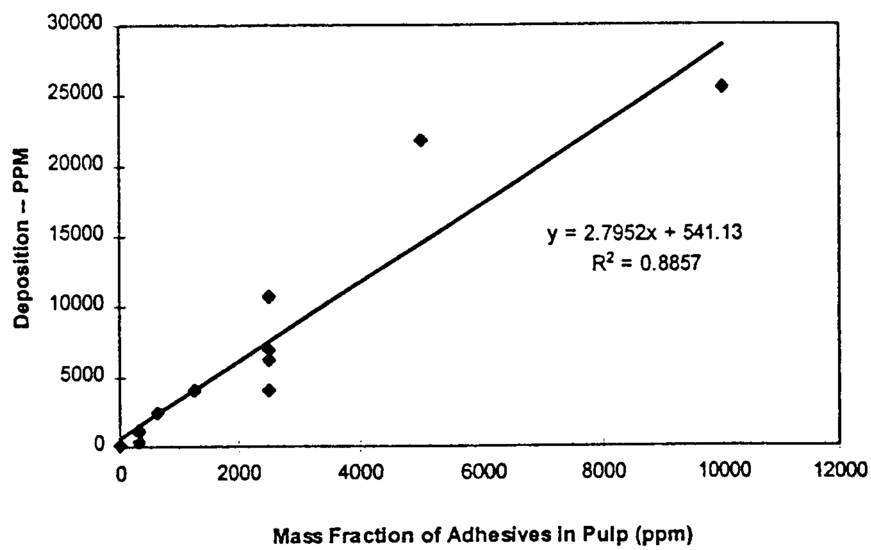


Fig. 2. Deposition PPM vs. Mass Fraction of PSA in Pulp



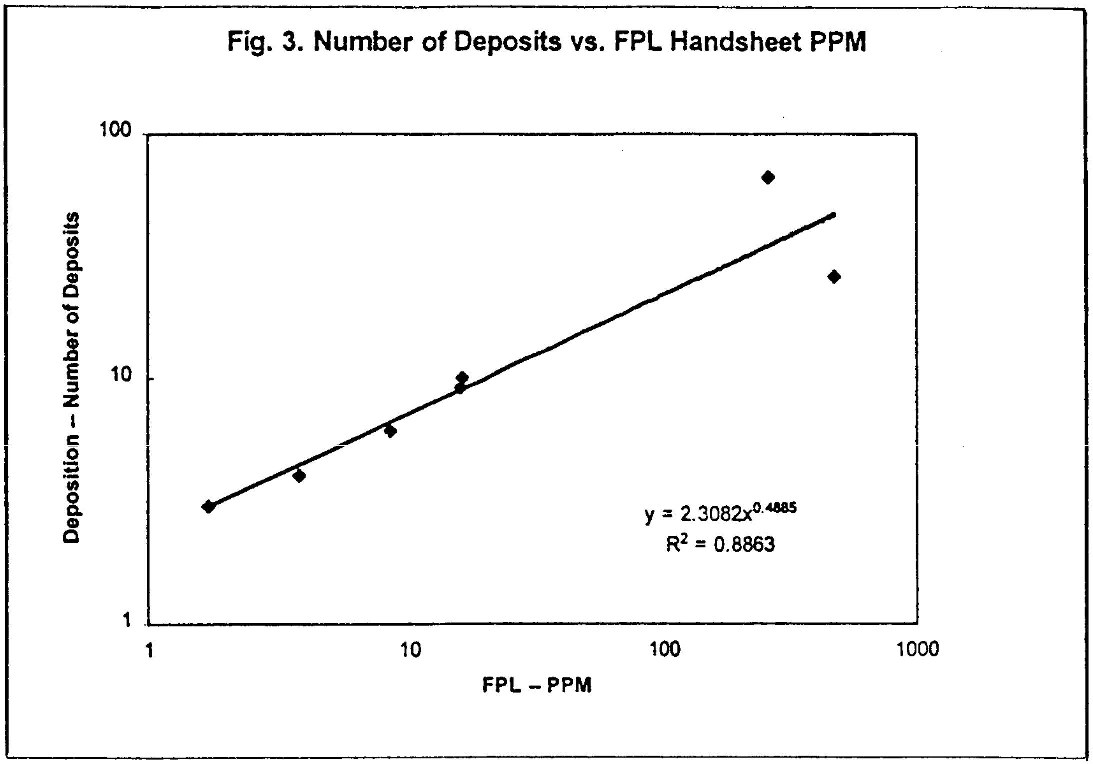


Fig. 3. Number of Deposits vs. FPL Handsheet PPM
100
10
1
Deposition -- Number of Deposits
y = 2.3082x^0.4885
R² = 0.8863
1
10
100
1000
FPL - PPM

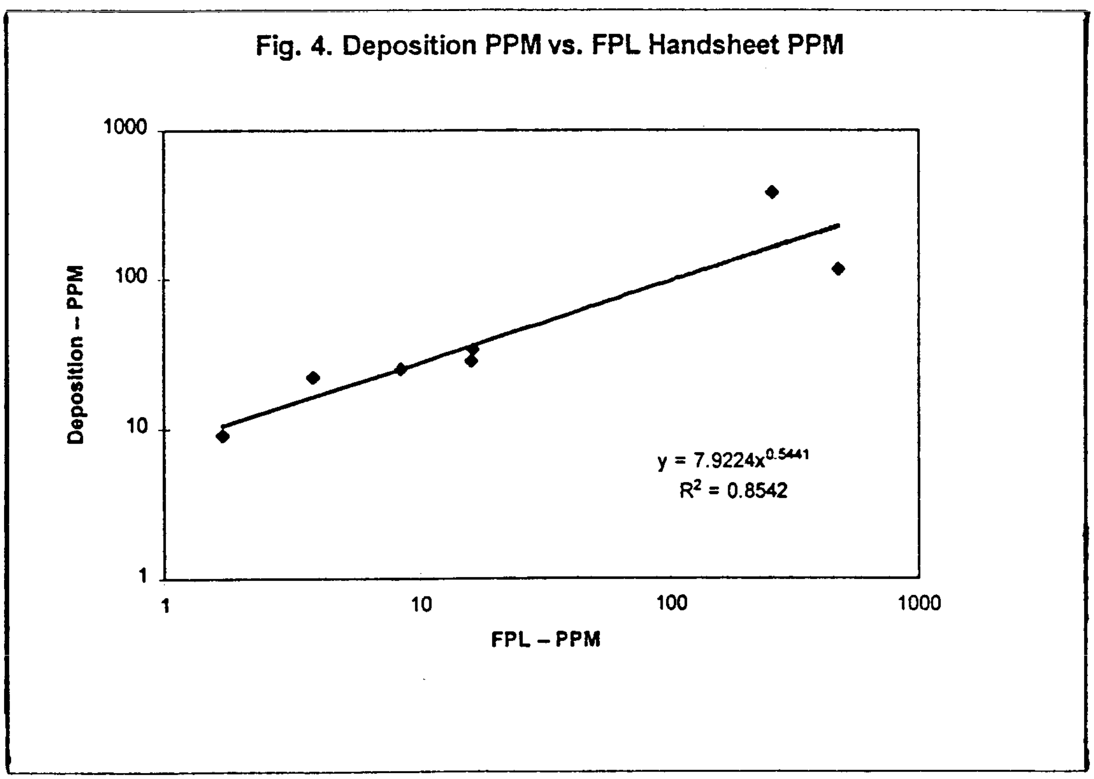


Fig. 4. Deposition PPM vs. FPL Handsheet PPM
1000
100
10
1
Deposition -- PPM
y = 7.9224x^0.5441
R² = 0.8542
1
10
100
1000
FPL - PPM

Fig. 5. Number of Deposits D<80 microns vs. FPL Handsheet PPM

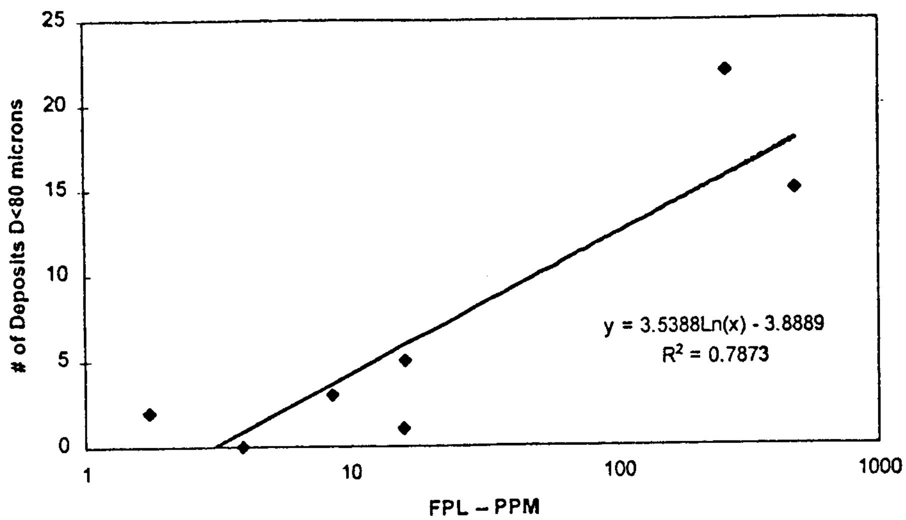


Fig. 6. Number of Deposits 80<D<225 microns vs. FPL Handsheet PPM

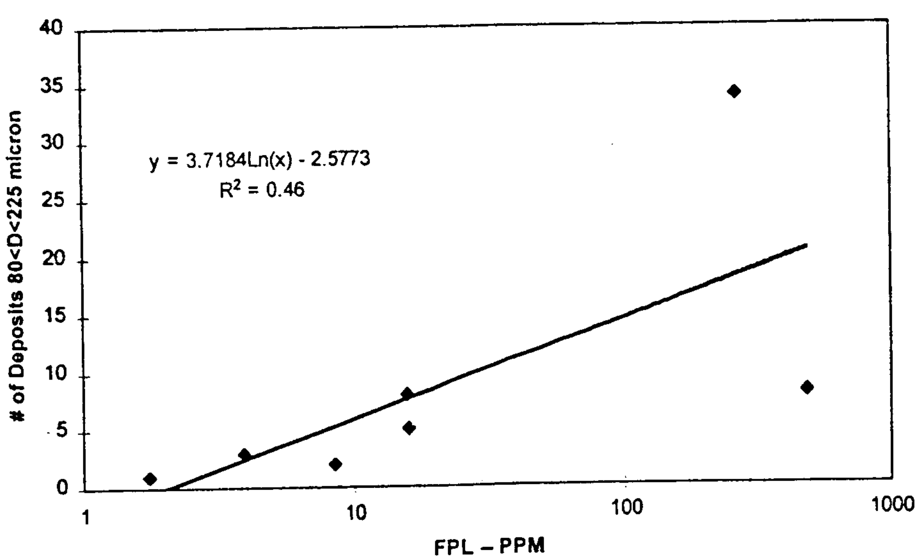


Fig. 7. Number of Deposits D>225 microns vs. FPL Handsheet PPM