TESTING PROTOCOL

PEER REVIEWED

Image analysis protocol in evaluation of pressure sensitive adhesives

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ABSTRACT

This paper discusses use of the U.S. Forest Products Laboratory's image analysis protocol to evaluate effectiveness of various containment removal processes used in a pilot plant trial. The protocol itself is outlined, and its use is described. Description of the trial plan and objectives show how the testing protocol related to evaluation of the trial results.

Application:

Results from a testing protocol can help adhesive manufacturers design pressure sensitive adhesives that will perform well in pulping operations.

THE U.S. FOREST PRODUCTS LABoratory in Madison, WI, contracted with the authors' company in March 1997 to undertake a pilot study of the recyclability of postage stamp laminate material. This study was part of the U.S. Postal Service's efforts to develop environmentally benign pressure sensitive adhesives for postage stamps. The trial ran in the Beloit Pulping Group Research and Development Center in Pittsfield, MA, on May 9, 1997. The trial primarily used commercially sized equipment installed in the pilot plant recycled fiber system. Trials followed a system design and testing protocol developed and provided by the Forest Products Laboratory. The objective of the trial was to study the removal of pressure sensitive adhesive (PSA) in the various contaminant removal operations currently found in today's typical mixed office waste (MOW) deinking systems. The IAP-1 image analysis protocol was the primary means to evaluate the trial results.

EQUIPMENT AND PROCEDURE

Design of the trial plan and procedure followed as closely as possible the testing protocol provided by the U.S. Forest Products Laboratory. The material for the trial was a blend of 95% clean, 20 lb basis weight, Xerographic copy paper and 5% unprinted, standard stamp laminate adhered to part of the copy paper. The adhesive provided as a component of the stamp laminate was an aqueous acrylate. The intent of the trial was to simulate a post consumer waste stream. It contained no silicone release paper. The system design was part of the protocol. It consisted of high consistency pulping, coarse and fine screening (0.30 mm and .015 mm slots, respectively), forward cleaning, through-flow cleaning, flotation, washing, and dispersion as **Fig. 1** shows. The trial ran in batch mode rather than continuously to minimize the quality of material necessary to conduct the trials.

Each piece of equipment for the trials ran in its "normal" operating mode as if installed in a continuously operating commercial deinking system. Inlet, accepts, and rejects samples were collected around each unit operation for testing.

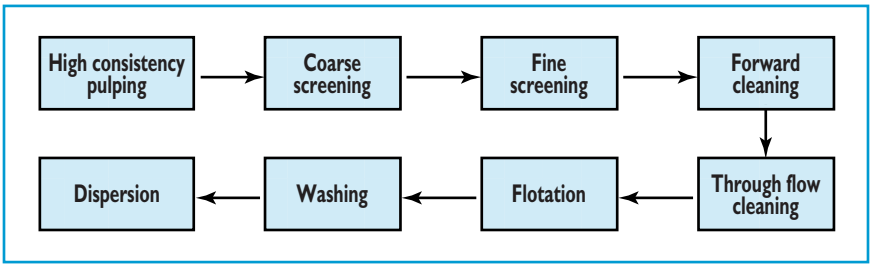
All sample testing except image analysis used TAPPI Test Methods. Image analysis used the Optomax Speck Check, 600 DPI scanner system following IAP-1 developed and provided by the U.S. Forest Products Laboratory. The appendix gives the complete image analysis testing protocol for testing the samples.

Twenty, 1 g handsheets from each sample were made according to TAPPI T205 sp-95, "Forming handsheets for physical tests of pulp." The handsheets are dyed using a solution of Morplas Blue dye and heptane, hung in a fume hood, and air dried for 24 h. This provides handsheets with a light blue color in the fiber and PSA with a dark blue color. The dry handsheets were then passed through three successive heptane washing baths and dried again. This process washes the dye from the fiber matrix (the fiber does not retain the dye) leaving stickies as a dark blue color. Ten dyed handsheets were scanned using the Optomax system using settings outlined in IAP-1.

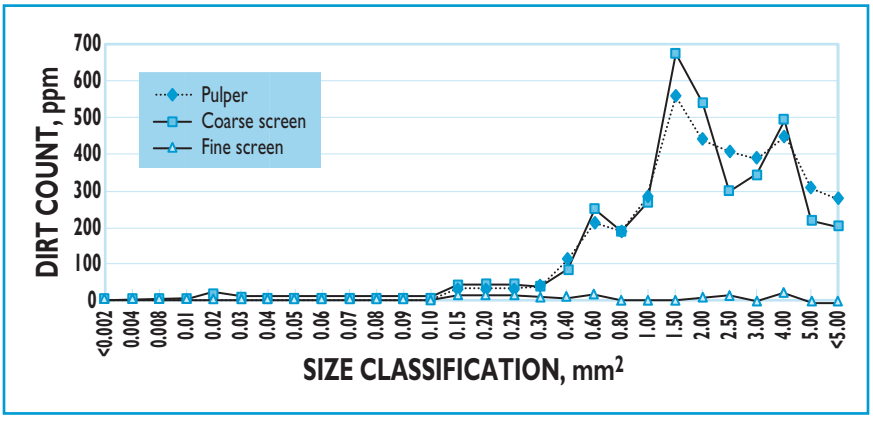
Each sample collected was also tested using the Valley Flat Screen equipped with a .015 mm slotted screen plate to provide comparison with the image analysis procedure.

DISCUSSION

The object of this work was to provide verification of a pilot plant trial protocol developed by the U.S. Forest Products Laboratory and Springborn Testing and Research as part of the U.S. Postal Services' efforts to develop an environmentally benign pressure sensitive adhesive for postal products. Since this work used an adhesive currently used for postage stamps, it also provides data for testing alternative adhesives in the laboratory to determine their relative recyclability. The IAP-1 is part of the pilot plant trial protocol, but it may also have use for other applications in the recycled fiber industry.



1. System block diagram



2. Particle size distributions for the pulper, coarse, and fine samples

The technique for dying the stickies is simple. It allows the adhesive in the samples to be dyed. This provides good contrast to the fiber and allows use of image analysis to measure adhesive levels in various samples. The procedure for treating the handsheets is exacting and contains several steps, but it does give a uniform contrast between the white fiber and the blue-dyed adhesive material regardless of stickie size. This allows accurate measurement of the size and surface area of the stickies in each sample using image analysis. The image analysis system provides a measure of the number and area of particles broken down into twenty eight bin sizes from smaller than 0.002 mm^2 to more than 5.0 mm^2. This is very useful as a research and product development tool as well as quality assurance. The process allows counting a significantly greater number of samples and larger samples for each evaluation. This increases the statistical accuracy and validity of the results.

Each unit operation in a contaminant removal (recycling) system is designed to remove materials with different physical characteristics. They primarily include size and shape, density, and surface charge. For the U.S. Postal Service's project, the particle size distribution is important because it provides clues why one adhesive type may be easy to remove compared with another adhesive that may be very difficult to remove. In this trial, the adhesive remained as large particle sizes greater than 0.3 mm^2. Contaminants in this size range are often easy to remove in screening operations. The trial results confirm this is true with a combined screening efficiency (coarse and fine slots) of 95.5%.

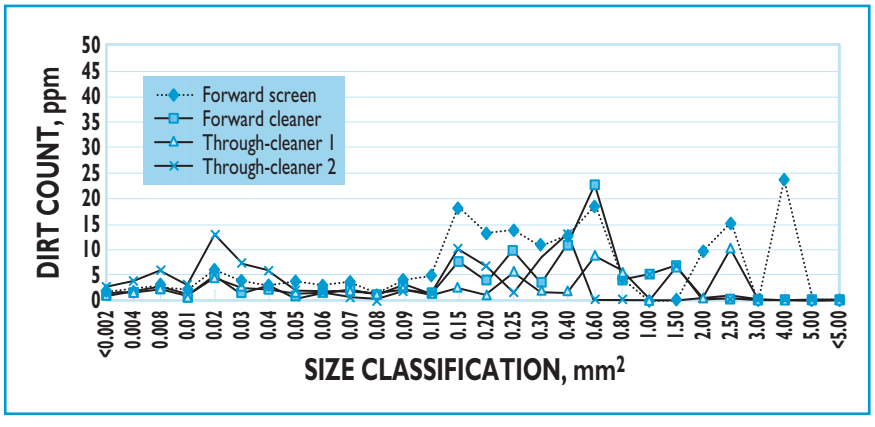
Figure 2 compares the analyses for the pulper and coarse and fine screen accepts samples. It shows the effect of the screening operations on the contaminant concentration in each size range. The coarse screen shows little removal of PSA even in the larger size classifications due to the two dimensional aspects of the particles. This adhesive released very easily from the fiber in the pulping process due to the large surface areas and postage stamp construction that uses a water soluble underliner beneath the pressure sensitive adhesive. The containment particle geometry made removal in the coarse screening operation difficult and ineffective.

The fine slotted screening operation using 0.15 mm slots removed PSA in sizes greater than 0.1 mm^2 as in Fig. 1. This type of information is useful for an equipment supplier to select appropriate contaminant removal equipment or correct slot size for fine screening operations. The information also provides a comparison and projection of removal efficiencies for other adhesives that may have a different particle geometry or size distribution after pulping. Adhesive manufacturers can focus on the characteristics of a particular product that make it difficult to remove from the pulp slurry completely. For this adhesive, 45 ppm adhesive was 0.04–0.10 mm^2. These sizes are not removed with any high degree of efficiency by screening. This information allows the adhesive manufacturer to study ways of adjusting the formulation to decrease the number of smaller particles formed in the pulping process. This would make removal of the material through screening more efficient.

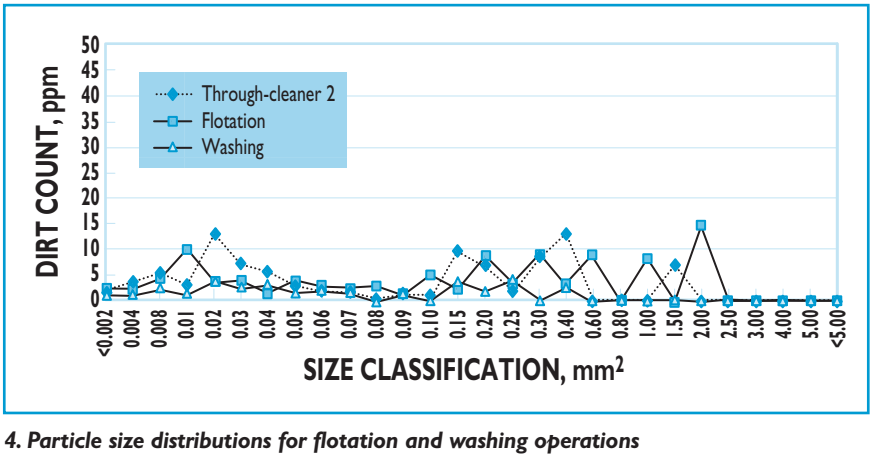
This same analysis with particle size distribution can be used for each operation in the trials. The unit operations following the screening were centrifugal cleaning, one pass of forward cleaning, and two passes of through- flow cleaning. The cleaning operations do not provide high removal efficiencies across the unit operations but do contribute to total system reduction as **Fig. 3** shows. The cleaners contributed significantly to removing many smaller, visible particles that the screening process did not remove.

	Image analysis scanner		0.015 mm Valley Flat Screen	
	TAPPI dirt count 0.04 mm2 and larger	Cumulitive reduction	% retained	Cumulitive reduction
Pulper	3594 ppm	-	1.040	-
Coarse screen	3613 ppm	0.0%	0.670	35.6%
Fine screen	161 ppm	95.5%	0.020	98.1%
Forward cleaning	85 ppm	97.6%	0.027	97.5%
Through-flow cleaner 1	52 ppm	98.6%	0.006	99.4%
Through-flow cleaner 2	54 ppm	98/5%	0.003	99.7%
Flotation	78 ppm	97.8%	0.045	95.8%
Washing	18 ppm	99.5%	0.020	98.1%

I. PSA (stickie) reduction



3. Particle size distribution for forward and through-flow cleaning operations



4. Particle size distributions for flotation and washing operations

The remaining two contaminant removal operations—flotation and washing—usually target removal of the smallest particle size contaminants. **Figure 4** shows the results from these operations. The operations reduced the TAPPI dirt count (0.04 mm^2 and above) to the reported 18 ppm.

The measuring technique provided a system removal profile for this adhesive type to compare other adhesives in the pilot plant to predict their ease or difficulty of removal in a commercial system.

The image analysis results in **Table I** were useful for evaluation of the trial results and comparison to bench scale and pilot plant testing at the U.S. Forest Products Laboratory and another pilot plant. The Valley Flat Screen analysis included in Table I provides a general confirmation of the image analysis results. The two techniques use different measurements. Image analysis uses an area measurement. The flat screen uses inclusion of a many contaminants with varying physical characteristics such as specific gravity, size, and shape. In this case, a reasonable correlation exists primarily due to the presence of only one contaminant in the furnish.

CONCLUSIONS

IAP-1 provided an easy method to enhance the contrast between the PSA in these trials and the fibers. This allows effective use of image analysis as a method to determine particle size distributions and measure removal of adhesive from a pulp suspension. Determination of the most effective contaminant removal unit operations and the particle size ranges they can remove are also possible. This standardized testing procedure can be very useful to those involved in the U.S. Postal Service's program to develop an environmentally benign PSA for postal use. It should also be helpful to those involved in the recycling industry who want to develop improved equipment and removal processes for these materials. **TJ**

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APPENDIX

Image analysis protocol

These procedures evaluate the contaminant present in samples from the Forest Products Laboratory pilot scale recycling trials and Specialized Technology Resources laboratory recycling studies. The trials are for recyclability of environmentally benign postage stamp materials.

Post stained handsheets

I. Equipment and materials

1. Optomax Speck Check Dirt Counter System with a HP 4C 600 dpi color scanner or equivalent
2. Weighted scanner block, 200 cm x 200 cm
3. Camera lens wipes
4. Glass cleaner
5. Morplas Blue 1003 dye
6. Heptane, 95%
7. Balance weighing to the nearest 0.01 g
8. Forceps, filter
9. Spatula and laboratory spoon
10. Plastic photography tray, 600 cm x 600 cm
11. Paper wiping towels
12. Metal paint pan
13. Paint roller, 230 cm mohair with 5 cm nap
14. Pyrex crystallizing dish, 190 mm x 100 mm
15. Pyrex beaker, 2000 mL
16. Pyrex beaker 100 mL
17. Funnel, glass short stem
18. Rubber gloves
19. Small binder clips
20. String

II. Staining

1. Weigh out 0.67 grams of Morplas Blue 1003 dye.
2. Mix with 1 L of heptane. (All staining should occur in a chemical fume hood while wearing appropriate safety equipment.)
3. Filter the Morplas blue and heptane solution to remove any undissolved dye particles.
4. Pour 250 mL of Morplas Blue and heptane solution into the pan and allow the roller to absorb the Morplas Blue and heptane solution completely.
5. Place the handsheet to be stained into the clear plastic photography tray. (Wire side of the handsheet should face the photo tray.)
6. Take the paint roller and make one pass over the handsheet starting from the bottom of the sheet and working toward the top. (Stain the felt side of the handsheet.) Wipe any excess dye solution from the handsheet with a paper towel.
7. Pick up the handsheet with filter forceps and allow to dry. (Run a piece of string across the fume hood and attach small binder clips to the string. Hang handsheets by the clips.)
8. Continue staining handsheets until all sheets are stained.

III. Image analysis

1. Randomly select ten dyed handsheets from the set to be analyzed by image analysis.
2. Turn on the Optomax unit. Allow the machine to warm up for 1 h.
3. Clean scanner glass with cleaner and paper towel. Wipe scanner glass with camera lens wipe to remove any particles or lint material.
4. Do ten scans with the lid closed to warm up the scanner bulb.
5. Open lid. Place stained handsheet (wire side down) on upper right hand corner of scanner bed. Place weighted scanner block carefully onto the handsheet. (Take care that the handsheet stays in position.)
6. Scan dyed handsheet.
7. Remove handsheet from scanner bed. Examine scanner glass for adhesive residue, clean glass with glass cleaner, and wipe with camera lens wipe.
8. Place next dyed handsheet onto scanner bed repeating steps 4–5 until ten handsheets are scanned.
9. Print scanner output.

IV. Wash

1. Pour 500 mL of heptane into three 190 mm x 100 mm crystallizing dishes. (All washing of handsheets should use a chemical fume hood while wearing appropriate safety equipment.)
2. Place dyed handsheet into the first crystallizing dish. Swirl handsheet in the heptane with filter forceps.
3. Remove and place handsheet into the second crystallizing dish. Swirl handsheet in the heptane.
4. Remove and place handsheet into third crystallizing dish. Swirl handsheet until all traces of Morplas Blue dye are removed from handsheet. (PSA will retain the blue dye.)
5. Remove handsheet and hang to dry.
6. Repeat steps 2–5 until all handsheets of the set are complete.
7. Scan all handsheets of the set by image analysis procedure.

(Before washing the sheets with heptane, determine whether the adhesive will retain the blue color after staining. Try washing a sample of the handsheet in heptane. If dye is removed from the adhesive, leave the handsheets until the dye remains with the adhesive. This process can take anywhere up to three weeks.)

Settings for Optomax Speck Check Image Analysis System

Detection level: 100 for Morplas Blue dyed sheets and 140 for post washed sheets
Video mode: Normal
Shade compensation: Off
Scan resolution: 600
Display resolution: x 5
Window X start: 0.0
Window Y start: 0.0
Window X size: 6.0
Window Y size: 6.0
Type of frame: Circle
Circle frame radius: 2.8
Size file in use: 2–5
Brightness pre-scan: Off