

## **RCA IAP-2**

### **Determination of Adhesives in Paper Handsheets by Image Analysis**

#### **1. Scope**

- 1.1 This procedure is intended to quantitatively determine the residual adhesive content of paper handsheets generated by the RCA LRP and RCA MRP testing protocols.
- 1.2 During the laboratory and mill scale recycling protocols, paper handsheets are made after each unit operation. The adhesive in the handsheet is then stained with a blue dye. Handsheets with stained adhesive particles are scanned on a scanner. Image analysis on the scanned image is used to determine the size distribution of the detected adhesive particles, the average particle size, the total projected area of the adhesive in ppm, and the particle number in counts/m<sup>2</sup>.
- 1.3 This test method was qualified in the US Postal Service Benign Adhesive Program and found to be reliable.

#### **2. Referenced Documents**

- 2.1 US Postal Service Specification USPS-P-1238F for Benign Adhesive Stamps, Section 80, Image Analysis Protocol, US Postal Service, Washington, DC.
- 2.2 RCA LRP, Laboratory Testing Protocol For Paper Labels Coated With Recycling Compatible Pressure Sensitive Adhesives
- 2.3 RCA MRP, Mill Recycling Protocol
- 2.4 RCA standards are available on the TLMI web site, [www.tlmi.com](http://www.tlmi.com), Environmental Committee page.
- 2.5 TAPPI T 205, Forming Handsheets for Physical Tests of Pulp
- 2.6 TAPPI T 213, Dirt in Pulp

#### **3. Apparatus**

- 3.1 Scanner-based image analysis dirt counter system such as the Verity IA LLC system or the Optomax SpeckCheck 2.
- 3.2 AGFA Argus II scanner, Hewlett Packard scanner, or equivalent. The scanner must have a minimum resolution of 600 dpi.
- 3.3 Standard handsheet mold as described in TAPPI T 205.
- 3.4 Standard couch roll.
- 3.5 Standard blotting paper as described in TAPPI T 205.
- 3.6 Handsheet dryer with an operating temperature of 150 °C.
- 3.7 Plastic or metal tray large enough to hold a blotting paper.
- 3.8 Foam paintbrush
- 3.9 Rubber ink roller.
- 3.10 Laboratory timer.
- 3.11 Standard press as described in TAPPI T 205.

#### **4. Materials**

- 4.1 Solvent blue 36 dye. The dye is also known as Morplas Blue 1003 and can be obtained from Pylam Products Company Inc., 2175 East Cedar Street, Tempe, AZ 85281, (602) 929-0070.
- 4.2 N-heptane.

#### **5. Reagent**

- 5.1 Dye solution composed of 0.67 g of solvent blue 36 dye in 1 liter of n-heptane.

#### **6. Procedure**

- 6.1 Form handsheets according to TAPPI T 205, except end the procedure after couching. After couching, the second wet blotting paper is discarded and a third dry one is placed to protect the handsheet attached to the first blotting paper. Label each sheet with trial designation and sheet number.
- 6.2 Stack the handsheets and blotters, using care to align sheets in a uniform stack. Place the stack in a press and, over a period of 30 seconds, raise the pressure to 345 kPa (50 psig). Maintain this pressure for 3 minutes.

- 6.3 Carefully unstack the sheets and place each pair of blotter papers, with a handsheet between them, on the dryer. The intent is to leave the handsheet firmly attached to the couching blotting paper until it is dyed. Dry for 3 minutes at 150°C with gentle restraint. If the adhesive material is likely to melt at this drying temperature, the handsheets should be air-dried against the couch plate in air according to TAPPI T 205.
- 6.4 Place dry handsheet attached to blotter paper face down in tray. Stain the handsheet by applying the dye solution evenly to the back side of the blotter paper with the foam paintbrush. This allows the dye to uniformly penetrate the handsheet. A small rubber roller can be used to help saturate the sheet uniformly. Furthermore, as the dye solution passes through the blotting paper undissolved dye particles are filtered from the solution. Place the dyed blotter paper/handsheet in between two blotter papers and roll with a roller to remove excess dye solution. This step of the procedure should be conducted in a ventilation hood to avoid exposure to heptane vapors and gloves should be worn as both the dye and heptane are skin irritants.
- 6.5 Let the heptane evaporate from the blotting paper/handsheets without separating the handsheet from the blotter paper by hanging them with clips attached to the blotter paper in the ventilation hood. Typical drying times are 2-3 minutes.
- 6.6 With a gloved hand, gently peel the dyed handsheet from the blotting paper. Place the side that was toward the blotting paper on the glass of the flat bed scanner. Using a weight with a blotter paper between, hold the sheet flat on the scanner.
- 6.7 Use the image analysis software to quantify the number of particles on the sheet. To compensate for sheet-to-sheet variations in dye intensity, best results are obtained by using a threshold that is automatically set 20% below the mode of the sheet image point luminance value frequency distribution. Typical dyed sheets exhibit a mode luminance value of 220, so the threshold should be 176. Table I includes typical settings for the Optomax SpeckCheck 2 and Verity IA Systems.

Table 1. Image Analysis Parameters

Optomax Setting	Value	Verity Setting	Value
Detection Level	180	Contrast Setting	20%
Scan Resolution	600 dpi	Scanner Resolution	600 dpi
Type of Frame	Circle	Region of Interest	Circular
Circle Radius	2.800 inches	ROI Width	96%
Frame Y center	3.150 inches	Warm and Calibrate	Yes
Frame X center	3.075 inches	Minimum Size	0.02 mm <sup>2</sup>
Guard Region	5.0		
Size File	0.02 - 999 mm <sup>2</sup>		
Matrix Detection	Off		
Video Inverted	Off		
Brightness Tracking	Off		

## 7. Calculation

- 7.1 Using the IA software, determine the total area covered by the adhesive in PPM and the particle count per square meter for each handsheet.
- 7.2 If the image analyzer does not report average particle size, calculate average size by the following formula: Average particle size = (Area in ppm)/(counts/m<sup>2</sup>). It may be possible to program this function into the image analyzer so that it is calculated and displayed automatically.
- 7.3 Subsequent statistical analysis should be conducted to determine the average PPM value, upper and lower 95% confidence limits on the average, average particle size and 95% confidence limits on the size. Simple calculations assuming normal distributions are not likely valid when the particle counts are below 3 particles/handsheet. Calculations assuming a Poisson distribution are likely more valid. A Microsoft Excel spreadsheet is available on the TLMI web site that does the proper calculation assuming both a Normal and Poisson distribution. The spreadsheet is entitled “Statistical Analysis of Adhesives”.

## 8. Report

- 8.1 For each unit operation, report the average and confidence interval for PPM value, particle size in  $\text{mm}^2$ , and particle count in  $\text{counts/m}^2$ .
- 8.2 Report identity of the image analysis system , the instrument parameters, and number of sheet analyzed.