

Determination of Post-it® note adhesive particle size in handsheets and filter paper by image analysis

FRED GUSTAFSON AND JOAQUIN DELGADO

THE EFFECT OF ADHESIVES ON paper recycling are well documented. Adhesives produce “stickies” which can build up on deinking equipment and on the wire of the paper machine (1–3). Stickies can cause holes or clear spots in recycled paper. For this reason, office waste recycling programs typically exclude adhesive tapes, self stick labels, and envelopes with plastic windows from the recycling stream.

Post-it® notes are coated with a discontinuous particulate adhesive that is repulpable compared to many adhesive coated products. Office waste containing Post-it® notes is being successfully recycled into tissue and bond paper products (4). In order to quantitatively determine the fate of the adhesive during recycling, 3M conducted pilot scale repulping studies at the Black Clawson Technical Center. Although there are many good laboratory-scale repulpability test methods that emulate the repulping process (1–2, 5–7), pilot-scale experiments should be more representative of full-scale paper recycling.

During the repulping experiment at each unit operation in the deinking and screening process, the input pulp, accepts stream, and reject stream were saved for analysis. Handsheets were made from the accepts pulps and reject stream material was collected on filter paper. Image analysis was used to determine the adhesive particle size distribution in handsheets and filter

paper samples. Adhesive mass balance was determined by solvent extraction.

Image analysis has been used in the paper industry to determine residual ink in recycled paper (8–11). Those studies considered reflected light errors, calibration standards, particle size distribution, repeatability of results, pixel size errors, etc. Much of this research applies equally well to the determination of adhesive particles in paper. Klungness and co-workers used image analysis to measure adhesive particles in handsheets (11). To achieve contrast between the adhesive and the paper, Klungness stained the handsheet with a water-soluble ink. Hydrophobic adhesive particles were not wetted by the ink and appeared as bright spots on a dark background to the image analyzer. Chou used a dye to stain adhesive particles in handsheets for microscopic analysis (2).

We report on a new way to determine adhesive particles in handsheets and filter paper, which should be useful for many hydrophobic adhesives. The adhesive was stained purple with a combination of heptane-soluble red and blue dyes which preferentially stained the adhesive but not the paper. Dyed handsheets were then immersed in a thin layer of a perfluoro-octane solvent to increase light transmission through the paper. This non-toxic fluorochemical did not swell the fibers like water or glycerin does nor did it desorb the dye from the

ABSTRACT

Pilot scale repulpability trials of Post-it® notes were conducted at the Black Clawson Co. Image analysis was used to measure the adhesive particle size distribution in handsheets and in filter paper samples made from the outputs of the unit operations during repulping. The paper was stained with an adhesive selective purple dye mixture and immersed in a perfluoro-octane index matching solvent prior to analysis. Image analysis measured the number and volume particle size distributions, number average diameter, median diameter, Sauter average diameter, volume average diameter, and counts per unit area. Projected area percent was also measured by image analysis and compared with solvent extraction for determination of total adhesive content.

Application:

This study presents a useful method for measuring the size distribution of adhesive particles in handsheets from adhesive repulpability studies.

adhesive. The dye was not soluble in the perfluoro-octane. The same solvent was used for multiple samples because the handsheets did not contaminate the solvent. Perfluoro-octanes are sufficiently volatile that, following analysis, paper samples were dried in air and stored with no apparent changes in the adhesive particles or the flatness of the sheet.

Handsheets were illuminated with transmitted light so that adhesive particles on the bottom, center, and top of the handsheet could be detected by the image analyzer. The location of adhesive on filter paper was the top surface and reflected light could be used. Only particles on the top of the sample were visible in reflected light. Calibration of the image analyzer was accomplished by using a commercially available screen tint consisting of small uni-

form black dots on a clear film. The threshold level of the image analyzer was set so that the film read a pre-determined value of the area per dot.

Image analysis was useful for determining the particle size of adhesive in paper samples but not for measuring the total amount of adhesive in the sample. The image analyzer calculated the projected area percent covered by adhesive particles in the paper; however, solvent extraction of pulps was found to be a better measure of total adhesive content. The solvent extraction test is also presented here.

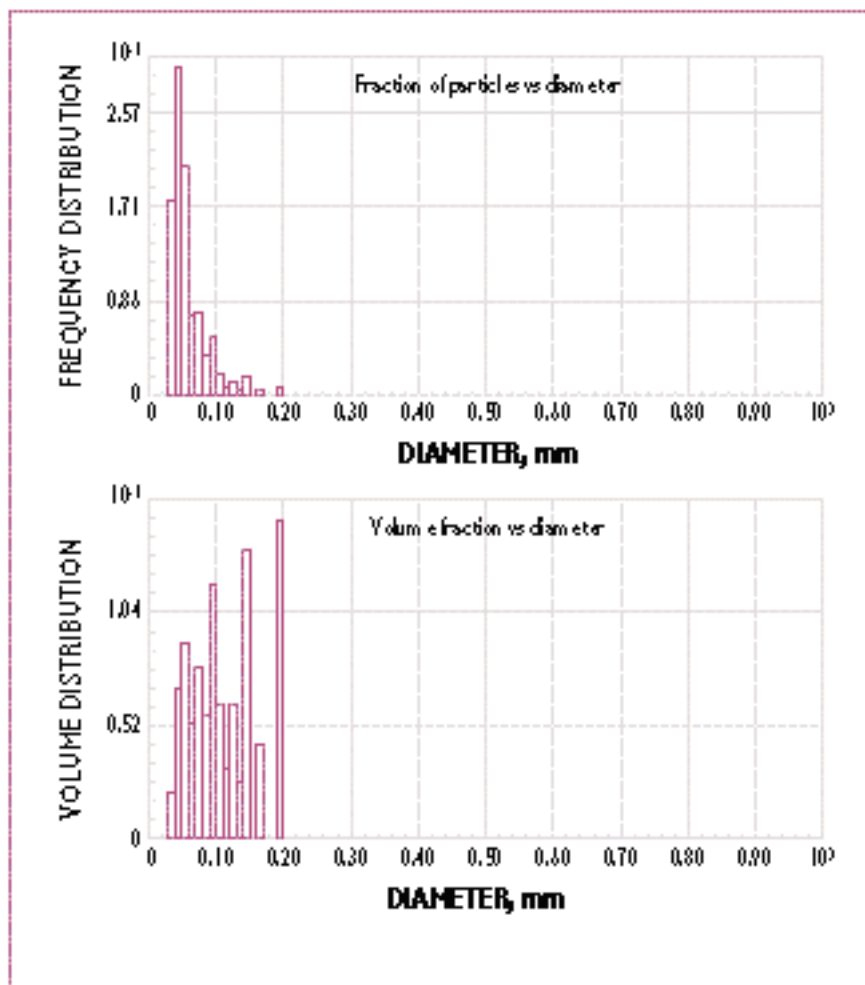
RESULTS AND DISCUSSION

Repulping experiment

The pilot-scale repulping experiment consisted of pulping 1500 lb of paper followed by centrifugal cleaning, screening, washing, thickening, and flotation unit operations. A repulping sequence was also performed with screening, forward and reverse centrifugal cleaning, and washing in series. Each unit operation had an input pulp, an accept pulp, and a reject stream which were retained for analysis. Hand-sheets were made from the accepts streams of each unit operation. Reject streams had low fiber content and were filtered through filter paper to collect the adhesive. Input, accepts, and reject pulps at each stage of the operation were extracted with solvent to determine the adhesive mass balance. Hand-sheets and filter paper samples were analyzed for adhesive particle size distribution.

Image analysis

Contrast between adhesive and paper fibers was achieved by staining the handsheets with a mixture of Solvent Blue 36 and Solvent Red 27 dyes. Purple particles absorbed more light than red or blue ones. The adhesive was selectively stained purple, but the paper fibers were only



1. Number average and volume average diameter of PSA particles in a handsheet. No. of particles counted = 248. No. average diam. = 59.4 μm . Sauter diam. (D_{32}) = 98.71 μm . Vol. average diam. = 110.92 μm . % Area of the handsheet containing PSA = 0.05.

lightly colored because the dyes are hydrophobic and have a limited affinity for paper.

To improve contrast between adhesive and fibers, the dyed handsheet was placed in a Petri dish and covered with a few milliliters of FC-75, a 3M perfluoro-octane liquid. The refractive index of the liquid was 1.276. This inert liquid reduced light scattering between fibers by raising the index of refraction of the medium between them from 1.0 to 1.276. FC-75 did not dissolve the dye nor did it disturb the handsheet in any apparent way. Without this index matching fluid, it was impossible to

get adequate contrast between dark areas in the paper and small adhesive particles. Image analysis was performed in the transmission mode because adhesive particles on the top, middle, and bottom of the sheet need to be detected.

Limitations of the test

TAPPI handsheets have a nominal weight of 1.2 g for a sheet diameter of 160 mm. The handsheets analyzed in transmission must weigh less than about 3.5 g/160 mm diameter to assure adequate transmission of light through the sheet. The image analyzer has a high and low light level feature which signals the operator

Data	Up-diam	Down-diam
1	92.26	82.09
2	78.10	84.05
3	85.57	101.09
4	89.89	73.05
5	89.17	80.93

	Level	N	Mean
Up-diam	5	86.998	5.522
Down-diam	5	84.242	10.305

Pooled STDEV = 8.267
p value = 0.612

I. Analysis of same handsheet in up and down position. Average diameter in μm .

when the amount of light reaching the TV camera is in the linear response region of the CCD camera. Handsheet formation must be uniform because the image analyzer will consider dark fiber flocs to be adhesive particles. Handsheet weight should also be the same for each sheet. The filter paper samples in this study weighed more than 3.5 g/160 mm diameter and had to be analyzed in reflected light.

A blank TAPPI handsheet was prepared, dyed, and imaged. Some fiber crossings and locally high concentrations of dye were dark enough to be imaged as adhesive particles. To reduce the imaged area of a blank handsheet, adhesive particles with diameters of less than 35 μm had to be rejected in software by an area reject feature.

Calibration

All image analytical methods require calibration. A millimeter ruler was used to determine the true imaged area of the sample. This value was then programmed into a multiplier function so that the image analyzer read length and area directly in micrometer and square micrometer units. An internal calibration function in the image analyzer was used to set all of the pixels in the TV camera to the same voltage after blank samples were properly illuminated.

The calibration standard for particle detection was a photographic film screen tint with 49 μm diameter black dots arranged in a 5% screen pattern. A small square of the film was placed directly on top of a blank TAPPI handsheet immersed in the fluorocarbon on the stage of the image analyzer. When the threshold level of the image analyzer was set so that the calibration standard measured 49.0 μm dots, adhesive particles in the handsheet did not appear completely imaged. This was because the dots were opaque but the dyed adhesive particles were translucent and transmitted some light. The adhesive image better matched the actual adhesive particles when the film dots measured 53 μm in diameter. Therefore, the calibration standard did not have an absolute value but only a constant value relative to the sample.

Data analysis

In each field of view of the sample, each particle was individually numbered and its area was determined in square micrometers. After each particle in the field was counted, a zero was entered in the data table. The total number of zeros in the data table was the number of fields analyzed. The data were stored in an ASCII file. An external PASCAL program read the data from the file and calculated the number of particles

counted, using the method of moments of distribution, the number average particle diameter, the Sauter average diameter (ratio of the 3rd moment of the distribution to the 2nd moment), the volume average diameter, and the projected area percent of the handsheet covered by particles. It also printed a histogram of the number and volume particle size distributions as shown in Fig. 1.

Two-sided handsheets

Adhesive particles in a handsheet should be randomly distributed throughout the sheet. To test the theory that the image analyzer detects all particles in a handsheet, one was analyzed five times and then turned upside down and analyzed five times again. Table I shows the analysis of variance of the data as calculated in the Minitab statistical program. The mean, standard deviation, and 95% confidence interval of each data column is presented in the table. The p-value for the difference between the two data sets was 0.612, indicating a high probability that the two sides were not different.

Precision

Repeatability calculations for number average diameter and area percent coverage are presented in Table II (performed according to TAPPI Test Method T 1206). Each handsheet was analyzed twice each to get the quoted precision. Repeatability for number average particle diameter was 10.2 μm with a grand average of 88.9 μm . This means that two different handsheets whose number average diameters differ by more than 10.2 μm are considered different at the 95% confidence level.

In addition to particle diameter, the image analyzer also determined the projected area percent coverage of the adhesive in the sheet. The projected area percent was typically in the range of 0.05% to 1% and was greatly influenced by a few large particles. Particles with diameters larger than the thickness of the handsheet

Handsheet number: Label up or down:	Sheet 1 Up	Sheet 2 Down	Sheet 3 Up	Sheet 3 Down
Number of particles counted in the handsheet	511	845	174	167
Test 1 (diameter in micrometers)	98.37	85.67	92.26	82.09
Test 2	101.98	86.15	78.1	84.05
Test 3	98.4	82.93	85.57	101.09
Test 4	99.29	87.85	89.89	73.05
Test 5	93.76	87.93	89.17	80.93
Average = $\bar{x}$	98.36	86.11	87.00	84.24
Grand average = $\bar{x}$ bar bar	88.93			
Std. dev. = s	2.96	2.04	5.52	10.30
Degrees of freedom	4.00	4.00	4.00	4.00
Pooled standard deviation	6.12			
No. tests required for test result = n	2	2	2	2
Std. dev. test result = $SDTR = s/\sqrt{n}$	2.10	1.44	3.90	7.29
Repeatability, $r = 2.77*SDTR$	5.80	4.00	10.82	20.18
Average repeatability = r bar	10.20 μm			
Handsheet number: Label up or down:	Sheet 1 Up	Sheet 2 Down	Sheet 3 Up	Sheet 3 Down
Number of particles counted in the handsheet	511	845	174	167
Test 1 (area % containing adhesive)	0.62	0.67	0.12	0.08
Test 2	0.66	0.88	0.08	0.08
Test 3	0.57	0.71	0.09	0.17
Test 4	0.59	0.71	0.09	0.07
Test 5	0.53	0.74	0.12	0.1
Average = $\bar{x}$ bar	0.59	0.74	0.10	0.10
Grand average = $\bar{x}$ bar bar	0.38			
Std. dev. = s	0.05	0.08	0.02	0.04
Degrees of freedom	4.00	4.00	4.00	4.00
Pooled standard deviation	0.05			
No. tests required for test result = n	2	2	2	2
Std. dev. test result = $SDTR = s/\sqrt{n}$	0.03	0.06	0.01	0.03
Repeatability, $r = 2.77*SDTR$	0.10	0.16	0.04	0.08
Average repeatability = r bar	0.09 area %			
Percent repeatability = $r*100/\bar{x}$ bar	16.25	21.40	36.64	79.56
Avg. percent repeatability	38.46			

II. Precision of handsheet particle size analysis. Number average diameter, $\bar{m}$, and area percent coverage.

were apparently flattened by the action of making the sheet. Precision of determining area percent was also poor. Samples with low percent area coverage were not distinguishable. Area percent coverage was not considered as reliable as solvent extraction for determining the total amount of adhesive in a handsheet. The precision of the data is influenced by counting statistics of

specks, which are described by a Poisson distribution (12).

Filter paper samples

In repulpability trials when there were not enough fibers in the sample to make a handsheet, the adhesive was collected on filter paper. Unlike a handsheet, all the adhesive particles were collected at the top surface of the filter paper. If the filter paper has uniform formation and its

weight is less than about 3.5 g per 160-mm diameter sheet, it can be analyzed in transmission. If the formation or light transmission of the filter paper is poor, it must be illuminated with reflected light.

Image analysis and calibration in reflected light

The light source for the reflected light measurement was a four-point

Filter paper number:	Filter 1	Filter 2	Filter 3
Number of particles counted on the filter paper	249	361	1203
Test 1 (diameter in micrometers)	61.68	57.74	64.43
Test 2	59.4	58.05	65.85
Test 3	62.41	58.16	64.15
Test 4	59.03	60.17	66.17
Test 5	62.08	58.69	65
Average = $\bar{x}$	60.92	58.56	65.12
Grand average = $\bar{x}$	61.53		
Std. deviation = s	1.58	0.96	0.88
Degrees of freedom	4.00	4.00	4.00
Pooled standard deviation	1.18		
No. tests required for test result = n	1	1	1
Std. dev. test result = $SDTR = s/\sqrt{n}$	1.58	0.96	0.88
Repeatability, $r = 2.77*SDTR$	4.39	2.66	2.43
Average repeatability = r	3.16 mm		
Area % microsphere coverage			
Filter paper number:	Filter 1	Filter 2	Filter 3
Number of particles counted on the filter paper	249	361	1203
Test 1 (area % containing adhesive)	0.07	0.09	0.53
Test 2	0.05	0.11	0.70
Test 3	0.08	0.10	0.63
Test 4	0.06	0.10	0.69
Test 5	0.08	0.12	0.63
Average = $\bar{x}$	0.07	0.10	0.64
Grand average = $\bar{x}$	0.27		
Std. deviation = s	0.01	0.01	0.07
Degrees of freedom	4.00	4.00	4.00
Pooled standard deviation	0.04		
No. tests required for test result = n	1	1	1
Std. dev. test result = $SDTR = s/\sqrt{n}$	0.01	0.01	0.07
Repeatability, $r = 2.77*SDTR$	0.04	0.03	0.19
Average repeatability = r	0.09 area %		
Percent repeatability = $r*100/\bar{x}$	53.11	30.37	29.48

III. Precision of filter paper particle size analysis. Number average particle diameter, μm , and area percent coverage.

focusing light attached to the bottom of the zoom lens. The lens was set to the 2X click stop. Filter paper samples were still placed in the bottom half of the large Petri dish, but no FC-75 was added and the top half of the dish was not used. Both the fluorocarbon and the top dish reflected too much light to the TV camera.

For calibration with reflected light, the same screen tint film calibration standard was placed on a square of bond paper inside a Petri dish. It could not be placed on the sample as before because, in the absence of the fluorocarbon, the filter paper was not flat enough. To set the value of the calibration standard relative to a real sample, a sample

was imaged and then the calibration standard was read with the sample removed. The reference value of the calibration standard was determined to be 1200–1300 sq. μm per particle to get proper imaging of the adhesive particles.

Data analysis and precision

Data analysis is the same as previously described. The repeatability

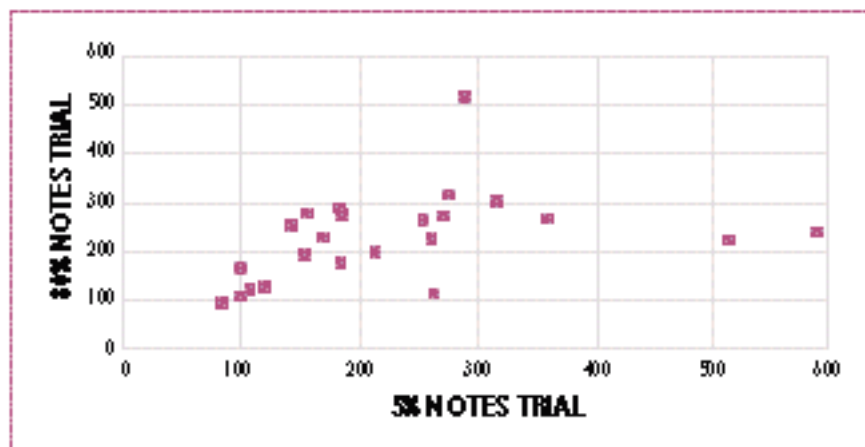
data for reflection is presented in Table III. Repeatability of number average particle diameter was 3.16 μm ; repeatability of percent area coverage remained at 0.09% absolute. As with the handsheets, area percent coverage had low precision and was not used to estimate the total amount of adhesive on the filter paper.

Particle size results

Mixed office waste should contain less than 1% Post-it® notes (3). In the pilot repulping trials, 5% and 80% Post-it® notes were repulped and analyzed for average particle size. With the particle size analysis, the effect of adhesive concentration can be determined. Figure 2 is a plot of the average particle diameter from each operation in 80% and 5% Post-it® notes. There is much scatter at the larger particle sizes, but at smaller sizes there appears to be a good correlation between the 5% and 80% Post-it® notes runs. At higher particle size, the correlation breaks down. This could be due to an actual concentration effect or to the influence of larger particles on the average particle size.

ADHESIVE EXTRACTION

Alkanes would be ideal solvents for extracting adhesives from paper because there is very little alkane-soluble material in paper. The heptane extraction blank of our paper was less than 0.03 mg/g. The heptane solubles were below the detection limit of the analysis. Post-it® adhesive alone was extracted from water with heptane with a recovery greater than 90%. However, when Post-it® notes were pulped, recovery of the adhesive from the pulp with heptane was low, ranging from 12 to 44%. Apparently, heptane did not separate the adhesive from the paper in a pulp. Methyl isobutyl ketone (MIBK) was tried as an extraction solvent. With MIBK, recov-



2. Average particle diameter (μm) comparison between 5% and 80% Post-it notes furnishes

ery of adhesive from pulps was greater than 90%. Ammonium sulfate was found to increase the recovery of the adhesive, and about 1 g was added to each extraction slurry. Presumably the ammonium sulfate increases the ionic strength of the aqueous phase forcing more of the organic material into the solvent phase.

Blank value calculation

The furnish for one of the trials at Black Clawson consisted of 80% Post-it® notes, 12% Boise Cascade Offset paper, and 8% Sharp Copy Bond paper. Each of these papers was extracted to determine the MIBK extractables. The overall blank value when adjusting for the percentages of paper was 2.9 mg/g dry paper. The standard deviation of the blank value for each paper was 0.2 mg/g (variance = 0.04). For three papers making up the furnish, the total variance was therefore $3 \times 0.12 = 0.36$ and the standard deviation was 0.35 mg/g.

Adhesive loss during centrifugation

Centrifugation was used to remove surfactant from the pulps. Since the density of Post-it® adhesive is less than 1.0, any free adhesive in the pulp would be expected to rise to the top of the centrifuge tube. The adhesive would then be discarded with the supernatant liquid. For several pulp samples, the supernatant

was decanted through filter paper to determine if any free adhesive was being rejected. The filter paper was stained with a solution of Solvent Blue 36 dye in heptane. Fewer than 10 adhesive particles were observed on any of the filter papers, meaning almost none of the adhesive was being lost. The adhesive was probably still attached to the paper fibers in the pulp or it was trapped in the fiber mat at the bottom of the centrifuge tube.

Detection limit

The MIBK extraction blank of the paper was 2.9 mg/g pulp and the standard deviation of the blank was 0.35 mg/g. If the detection limit of the adhesive extraction is considered to be the blank plus three standard deviations, the value would be 3.95 mg/g or about 0.4%. The weight of adhesive on a Post-it® note is only about 1%. For the purpose of gravimetric analysis, a pilot repulping trial furnish of 80% Post-it® notes was chosen for analysis. This furnish yielded pulps with adhesive concentrations at twice the detection limit of gravimetric analysis or about 0.8%.

Repeatability of the extraction test

At the first stage of the Black Clawson pilot study, the furnish was repulped in a Hydrapulper at 12% consistency. The resulting pulp was diluted several different ways as input to seven unit operations or

Operation	Adhesive conc., mg adh./g pulp	Run number
X-clone	10.3	1
	13	2
U-clone	6.8	3
	11.4	4
Vertiscreen	5.8	5
	9	6
	10.2	7
	7.3	8
Hydradenser	10.6	9
	7.7	10
PSL	9.7	11
	9.7	12
Flotator	10.4	13
	5.5	14
DNT washer	14.3	15
	13	16
Statistics	Value	
Mean	9.67 mg/g pulp	
Std. error	0.64mg/g pulp	
Std. dev.	2.57 mg/g pulp	
Range	8.8 mg/g pulp	
Rel. std. dev.	0.26 No units	

IV. Repeatability of extraction tests. Analysis of initial feed streams Raw data

sequences. Each of these input pulps as well as the Hydrapulper output was analyzed in duplicate for adhesive content by solvent extraction. The data are presented in Table IV. The mean value of the 16 determinations was 9.7 mg adhesive/gram pulp. Standard deviation was 2.57 mg/g pulp. All samples were analyzed in duplicate so that the standard deviation of the mean would be $2.57/(2)^{1/2} = 1.82$ mg/g pulp. Therefore the relative standard deviation (RSD) of the test = $1.82/9.7 = 0.18$ or 18%. The RSD of the test can be reduced by analyzing each sample more times.

MATERIALS AND EXPERIMENTAL PROCEDURES

Instrumental

The image analyzer was a Model 3000 unit from Image Technology Corp. The transmitted light source for handsheets was a Volpi 5-cm diameter focusing fiber optic ring light connected to a Volpi Intralux 5000 light source powered by a Hewlett Packard 20V dc power supply. A four-point focusing fiber optic

light guide attached to the camera lens was used to illuminate filter paper samples in reflected light. A COHU black and white CCD television camera was attached to an extender tube. At the other end of the tube was an Optem Z65 zoom lens set to the 2X click stop. A simple manual transmission stage held the camera, lens, and fiber optic ring light. The image analyzer was operated in the Shape Mode in which the area of each particle in the field of view was measured and displayed. The true area viewed by the TV camera was $5.0 \text{ mm} \times 4.2 \text{ mm} = 21 \text{ mm}^2$. Since the active area of the CCD camera was $6 \times 7 \text{ mm}$, the overall system magnification was 1.4X. The image analyzer had the following properties: pixel resolution = $9.84 \text{ } \mu\text{m}/\text{pixel}$; diameter of smallest speck analyzed = $34 \text{ } \mu\text{m}$ due to an area reject setting; number of gray scale levels full scale = 256; and number of gray levels used in setting the detection threshold = 125.

Materials

Solvent Blue 36 dye and Solvent Red 27 dye were dissolved in AR grade

heptane at concentrations of 1 mg/mL for the blue dye and 0.5 mg/mL for the red dye. A 3M perfluoro-octane solvent (FC-75) was used as an index matching liquid. Other 3M perfluoro-octanes such as FC-77 or FC-104 can also be used. The calibration standard was a commercially available Bychrome Screen Tint (95% screen) in a 100 lines per inch pattern. This means that 5% of the area on the film was covered with $49 \text{ } \mu\text{m}$ diameter black circles and the rest of the area was clear.

Staining of handsheets and filter paper

Dyed handsheets or filter paper samples were first cut to fit inside the larger dish of $150 \times 20 \text{ mm}$ glass Petri dish set. In a hood, paper was dipped into a solution of purple dye in a plastic tray. Alternatively, the paper was sprayed with dye solution on both sides so that the entire surface was covered with dye. The sheet was dried in a hood.

Calibration for handsheets

A blank TAPPI handsheet was placed inside the Petri dish set and a few milliliters of the perfluoro-octane was added to immerse the handsheet. The light level was adjusted so that the proper amount of light was getting to the TV camera. Then the calibration function of the image analyzer was run to set all the pixels in the camera to the same voltage level. A 1-inch square of the calibration standard was placed on top of the paper in the solvent. Then the threshold level of the image analyzer was adjusted and the area per dot was measured until the average of all the dots was 2200–2300 square μm . Calibration was verified with each new handsheet analyzed in case the caliper or formation of the handsheet required recalibration.

Image analysis procedure for handsheets

Handsheets were placed in the larger half of the Petri dish set. About 5 mL of FC-75 were added to the dish

KEYWORDS

Adhesive papers, dyeing, extraction tests, filter papers, hand sheets, image analysis, particle size distribution, pressure sensitive adhesives, reclaimed fibers, recycling, repulping, staining, test methods.

to wet the handsheet. The smaller dish was then inverted on top of the handsheet so that glass was touching both sides of the handsheet. After focusing the camera on adhesive particles, the analysis began. At least 50 different areas in the same sample were analyzed. The Petri dish was moved by hand about 1 cm between analyses of single fields in a pattern which covered most of the sheet.

Calibration for filter paper

A square of white bond paper was placed in the bottom of the larger half of the Petri dish set. The proper light level was set and the calibration function was run to set all the pixels to the same voltage. The photographic film calibration standard was placed on top of the bond paper. After focusing on the black dots, the light level was adjusted. The threshold value for detection of an image was changed until the average area of the dots was 1200–1300 square μm .

Image analysis procedure for filter paper

Filter papers were cut to fit the Petri dish and placed into the larger half of the dish. Since the adhesive was already on the top surface of the filter paper, it was not necessary to add fluorocarbon to the dish. At least 50 different areas on the filter paper were analyzed. Care was taken to focus the camera between fields

because some of the filter paper filters were wavy.

Adhesive extraction

One gram of ammonium sulfate was added to a 40-mL pulp sample. It was then centrifuged at 5000 rpm for 10 min and decanted to eliminate as much surfactant as possible. Water was added to return the sample to 40 mL. Then 40 mL of AR grade MIBK was added to the pulp. After shaking the mixture overnight on a laboratory shaker, as much MIBK as possible was decanted into a separatory funnel. Then a disposable pipette was used to add the rest of the MIBK layer to the separatory funnel. Care was taken to get all of the MIBK layer.

The MIBK was then back extracted with water and the water was totally drained. The MIBK was evaporated in a solvent oven at 110°C, leaving adhesive and paper extractables. The jar was cooled in a desiccator and weighed to determine total residue. A blank value for the paper extractables was determined and was subtracted from the adhesive residue weight to get an estimate of the adhesive content of the pulp samples. All jars used for the final samples were dried in a desiccator prior to weighing. It was necessary to get an accurate weight on the jars.

CONCLUSION

A new method has been described for determining the particle size distribution of adhesives in paper by image analysis. This method can detect and count adhesive particles at levels far below the gravimetric detection limit by selectively staining the adhesive but not the paper. The repeatability of the average particle size measurement was adequate for

adhesive research, but the repeatability of the projected area percent of the adhesive was not. The method was applied to Post-it® adhesive, but it may have wider applicability. TJ

Gustafson and Delgado are, respectively, senior research specialist, Commercial Office Supply Division, and manager, Adhesive Technologies Center, 3M Co., 3M Center, St. Paul, MN 55144-0001.

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