Comparison of Macrostickies Measurement Methods

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ABSTRACT

Pulp containing PSA was prepared in the laboratory and blended with sticky-free pulp in four different proportions. The four pulps were then dewatered and shipped to four laboratories for the evaluation of macro stickies in terms of mm^2/kg. Also, five pulp samples from specific locations in a deinking mill were dewatered and shipped to the same four laboratories.

Methods used by these laboratories include:
1. Black ink method
2. INGEDE method
3. Enzyme digestion method
4. Blue dye method

Details of these methods are presented in Appendices 1 to 4. All laboratories used slotted screens to separate macrostickies and other contaminants from the pulp. Screen slot size varied from 80 mm to 150 mm. In addition, researchers at FPL made handsheets directly from pulp samples without a screening step. Hydrophilic black ink, hydrophobic blue dye, or carbon black was used to improve contrast between contaminants and fibers. INGEDE method used alumina powder to distinguish between tacky and non-tacky contaminants. Interfering fiber bundles were removed by enzyme digestion by researchers at the CTP. All methods employed image analysis to determine the average number and size of contaminants. The details of the image analysis methods were not standardized.

In view of the significant differences in the methods used to measure the concentration of macrostickies, it is not surprising to see considerable variations in actual values of stickies area reported by the participating groups. However, we were surprised to see excellent linear correlation among all methods for both laboratory as well as mill samples. As a result, we can conclude that any one of the methods seems to be suitable for monitoring stickies content but one cannot compare actual values from different methods as they may vary significantly.

KEYWORDS

Adhesives, Contaminants, Enzyme, INGEDE, Macrostickies, Measurement methods, Microstickies, Recovered Papers, Stickies.

INTRODUCTION

One of the problems in paper recycling is due to stickies - adhesives and binders used in coating, printing converting applications, labels and envelopes. The problem is compounded due to the variety of stickies in terms of chemical composition and viscoelastic behavior (1). Furthermore, our inability to quantify them hinders the progress in paper recycling. Many quantification methods have been proposed but we do not yet have a rapid, reliable and reproducible method for measuring the concentration of stickies. Our objective is to compare some of the methods used for the quantification of macrostickies.

We prepared synthetic stickies pulp in laboratory and shipped to four participating laboratories for analysis. We also took samples from a deinking mill and shipped to the same four participating laboratories for analysis. The sample preparation procedure is described below while details of the measurement methods are given in the appendices.

LABORATORY SAMPLES

Avery 8162 Ink Jet Labels (34 mm X 102 mm) were applied to virgin bleached kraft market pulp. Pulp sheets with labels were stored for at least 24 hours followed by shredding by hand and pulping. A high consistency laboratory pulper with helical rotor was used to generate sticky pulp. Pulping was carried out at 14% consistency at room temperature - about 20 °C to 25 °C. The procedure was repeated but this time without labels to generate sticky-free pulp. Four pulp samples were prepared by mixing various proportions of sticky-containing pulp and sticky-free pulp as shown in Table 1.

We also tried to apply hot melt to virgin pulp but it did not adhere very well. Hot melt containing sheets were pulped together with sheets containing PSA labels. However, the hot melt material came out in the form of large "plastic" pieces. This material was therefore ignored in our analysis.

After mixing pulp samples in the proportions shown in Table 1, the samples were dewatered and stored in a cold room prior to shipping. All participants were asked to dilute each pulp sample to less than 1% consistency prior to withdrawing the necessary amount for stickies measurement. All participants were requested to make macrostickies measurements in triplicate.

Table 1. **Laboratory sticky pulp samples**

Sample	Weight % Sticky Pulp Sample 1	Weight % Sticky-Free Pulp
1-Lab	100	0
2-Lab	75	25
3-Lab	50	50
4-Lab	25	75

MILL SAMPLES

Five pulp samples were collected from an office paper deinking plant in Duluth. A block flow diagram of Stora Enso DRPM (Duluth Recycled Pulp Mill) is the same as that published earlier (2) and is shown in Figure 1 together with sample points. Note that four of the five pulp samples were taken before dispersion while only one pulp sample was taken after dispersion due to a relatively insignificant variation in macrostickies content after that point. Samples are designated as shown in Table 2. Samples were dewatered and shipped to all participants. As in the analysis of the laboratory samples, participants were advised to dilute each pulp sample to less than 1% consistency prior to withdrawing the necessary amount for stickies measurement. All participants were requested to make macrostickies measurements in triplicate.

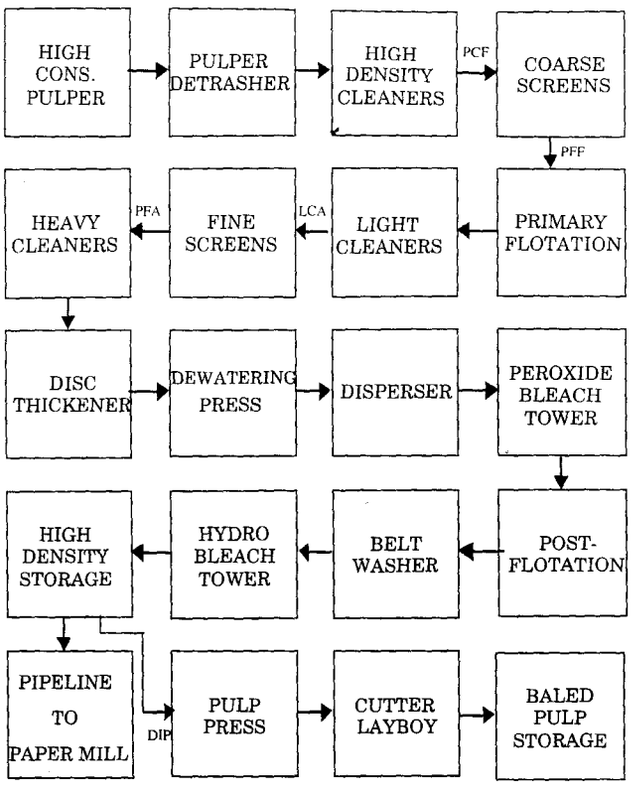

Figure 1. **Block flow diagram for Stora Enso Duluth Recycled Pulp Mill, Duluth, MN.** (See Table 2 for the description of sample points identified as PCF, PFF, LCA, PFA and DIP.)

Table 2. **Samples collected at Duluth Recycled Pulp Mill**

Mill Samples	Description
PCF	Primary coarse Screen Feed
FFF	Primary Flotation Feed
LCA	Light Cleaner Accepts
PFA	Primary Fine Screen Accepts
DIP	Deinked Pulp

MACROSTICKIES MEASUREMENT METHODS

Four methods, described in detail in Appendices 1 to 4, were used to measure the concentration of macrostickies in pulp samples. In the first method, Black Ink Method (Appendix I), Pulmac 150 µm (0.006") slotted screen rejects are collected on a white filter paper and dried. The filter paper is then dyed with black hydrophilic ink. Cellulose fibers appear black while hydrophobic stickies particles appear light colored in a black background.

The first few steps of the INGEDE method (Appendix 2) are same as the above Black Ink Method, namely, collect rejects on a white filter paper and dye them with water soluble black ink. To distinguish between sticky and non-sticky particles white alumina powder is sprinkled on the

filter paper. It is then covered with a heavy plate and dried in an oven to fix alumina powder to stickies. Excess alumina powder is then lightly brushed off and white specks against black background are measured.

In the CTP Enzyme Method (Appendix 3), 25 g of pulp is digested with cellulases for 12 hours. The pulp is then screened using an 80µm slotted screen plate. The screen rejects are collected on a filter paper, dried and non-tacky contaminants are brushed off. Black carbon powder is sprinkled over the filter paper to enhance the contrast between stickies and the background.

Hydrophobic Morplas blue dye is used by researchers at FPL (Appendix 4) to distinguish stickies from a white fiber background. They measure stickies on 150 µm (0.006") slotted Pulmac screen rejects as well as directly on handsheets without the screening step.

The team at the Stora Enso Duluth mill modified the INGEDE method by using pressurized air to blow off excess alumina powder followed by light brushing. Image analysis measurements were taken both before and after the brushing step. They used 17 g to 25 g pulp samples in a Pulmac 100 µm (0.004") slotted screening process.

RESULTS AND DISCUSSION

Three sets of data were used to compare several macrostickies analysis methods. The first two sets were generated by using the various methods to measure the macrostickies levels in the four laboratory-generated pulps. The area and number of stickies for these pulps are shown in Tables 3 and 4. A third set of measurements was on five pulps samples taken from process streams in the recycling mill. This data set is shown in Table 5. Results are also displayed in Figure 2 for laboratory samples and Figure 3 for mill samples. Methods are designated as follows:

A: Stora Enso modified INGEDE Method with air and brush (used only with mill samples).
B: Stora Enso modified INGEDE Method with air (used only with mill samples).
C: CTP Enzyme Method.
D: INGEDE Method.
E: Black Ink Method.
F: Blue Dye Method (using Pulmac Screen).
G: Blue Dye Method (without using Pulmac Screen).

The sticky area data, shown in Table 3 and Figure 2, were analyzed using linear regression, t-testing, and F-testing. The pulp samples were generated using mixtures of differing proportions of sticky-containing and clean pulp. Because the sample variance introduced during the production of the samples was likely very small, an assumption was made that all the observed variance was due to the analysis methods. The goals of the statistical tests were to (1) determine if the results from the various methods of analysis were correlated with the theoretical proportions and (2) determine if a linear model could be used to relate the results of one analysis method to the results of another.

Table 3. **Average sticky area (sq. mm/kg) analysis results for four laboratory-generated samples using five methods.**

Sample	% of Sample 1	Analysis Method				
		C	D	E	F	G
1-Lab	100	8,350	27,266	18,041	9,063	28,682
2-Lab	75	6,835	25,455	11,634	6,989	11,614
3-Lab	50	4,450	13,679	6,894	5,316	5,199
4-Lab	25	1.795	5,475	3,164	1.775	776

Table **4. Average sticky count (counts/kg) analysis results for four laboratory-generated samples using four methods.**

Sample	% of Sample 1	Analysis Method				
		C	D	E	F	G
1-Lab	100	No	20,611	19,582	24,458	22,143
2-Lab	75	Count	13,727	8,137	9,500	17,000
3-Lab	50	Data	9,676	16,418	9,375	8,889
4-Lab	25	Available	5,743	10,268	4,083	6,042

Table **5. Average sticky area (sq. mm/kg) analysis results for five mill samples using seven methods.**

Mill Samples	Analysis Method						
	A	B	C	D	E	F	G
DIP	21	122	575	281	222	47	74
PFA	200	1,114	1,870	1,130	400	227	571
LCA	800	2,859	3,580	5,712	938	1,477	1,693
PFF	1,707	5,862	4,815	7,809	1,515	3,632	3,694
PCF	2,806	6,715	6,660	7,907	1,339	4,234	2,816

Table 6 shows the results of several statistical calculations. The simplest linear model that could be used to describe the data is:

Stickies Concentration, sq. mm/kg = m * (% Sticky Pulp)

A first question is to determine if a more complex model is justified by the data. If one assumes that the addition of an offset is a reasonable

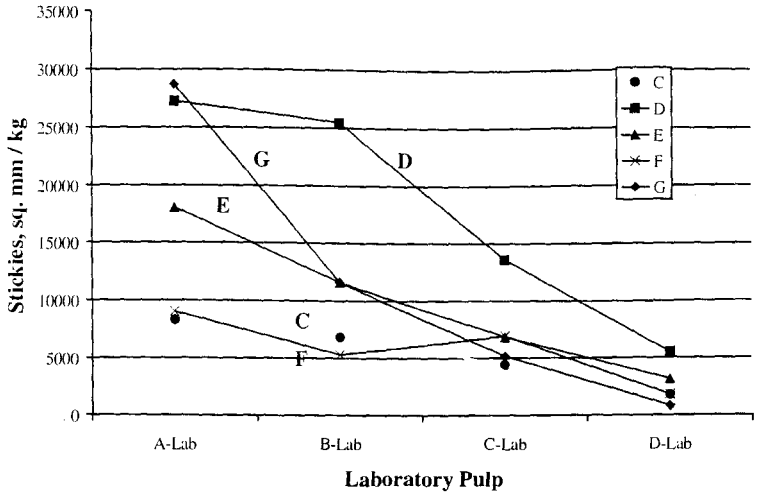

Figure 2. **Comparison of macrostickies measurement methods, laboratory samples.** C: CTP Enzyme Method; D: INGEDE Method; E: Black Ink Method; F: Blue Dye Method (using Pulmac Screen); G: Blue Dye Method (without using Pulmac Screen).

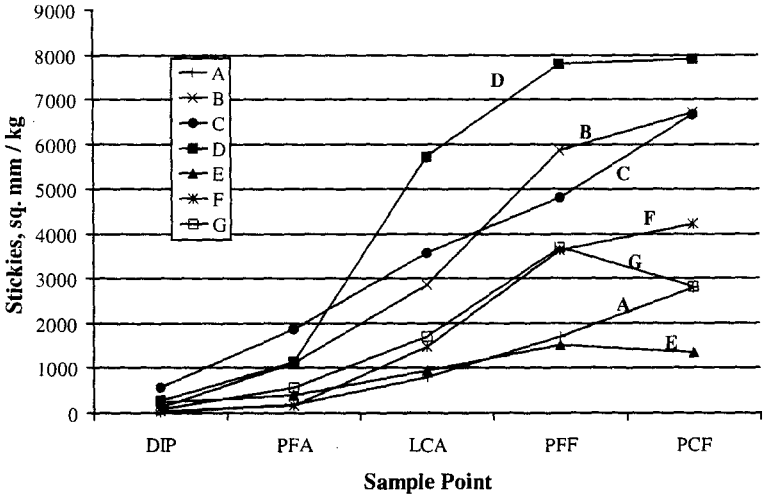

Figure 3. **Comparison of macrostickies measurement methods, mill samples.** A: Stora Enso modified INGEDE Method with air and brush; B: Stora Enso modified INGEDE Method with air; C: CTP Enzyme Method; D: INGEDE Method; E: Black Ink Method; F: Blue Dye Method (using Pulmac Screen); G: Blue Dye Method (without using Pulmac Screen).

model, then one can use a t-test to determine if the calculated offset is significantly different from zero. Table 6 shows the probability that the calculated offset for each analysis method is different from zero. Although the data for method E indicate that an offset may be required, the other data do not support nonzero offsets, and thus the simple model is likely justified.

The results of a linear regression assuming a zero offset, i.e., a line that passes through the origin, are also shown in Table 6. An F-test shows that the results for methods C to F fit the model to a high level of significance, $p < 0.6\%$. The results from method G are less correlated to the theoretical proportions, $p = 6.7\%$. The regression results can also be used to estimate the standard error in the estimate for the various methods. In Table 6, the standard error in the estimate is expressed as a percentage of the results for pulp sample number 1.

A further test of the variation in the data was obtained by analyzing replicate values. Although duplicate or triplicate samples are likely insufficient to adequately determine the expected variation, the coefficient of variation (COV), standard deviation/average, was calculated. The COV for methods F and G were obtained from 6 and 16 independent replicates, respectively. The two measures of variation suggest that method C has the least variance <8%, while the other methods exhibit variances that are likely less than 20%.. The results of the regression calculations suggest that, to a high level of certainty, the results from methods C to F can be used to predict any of the other results. The results from method G are less correlated.

***Table 6*. Results of statistical calculations for the data shown in Table 3.**

	Method				
Analysis	C	D	E	F	G
Probablility of nonzero offset (t-test)	22%	31%	98%	9%	80%
Slope (m)	26.2	291.0	165.4	92.8	214.3
Correlation coefficient (R^2)	0.958	0.811	0.928	0.972	0.725
F-statistic of the correlation	158	47	142	104	8
Probability correlation due to random variation	0.1%	0.6%	0.1%	0.2%	6.7%
Percent standard error of estimate	6%	15%	10%	6%	30%
Average coefficient of variation (COV) (SD/Ave.)	8%	27%	14%	17%	12%
Factor relative to method F	0.93	3.13	1.78	1.00	2.31

Because a simple linear model with no offset was found to be adequate to describe the relationships, a simple multiplicative factor relates results of the various methods. The factors relating results are also shown in Table 6. For example, the results from method D appear to be 3.13 times higher than those from method F. The fact that these calculated factors are different from 1.0 was investigated further. Table 4 shows the particle counts expressed as counts/kg. Statistical calculations similar to those shown in Table 6 were done for the count data.

Inspection of factor values for the count data shown in Table 7, shows that, in contrast to the area data, the count data appear to give the same absolute value. This observation suggests that all the methods are identifying essentially the same number of particles. Furthermore, the differences observed for the absolute values of the area data are likely due to differences in apparent size of individual particles. Note that although the results from method E agree with this conclusion, the fact that the probability of the correlation was due to random variation was 44% suggests that one should interpret the count data from this method with caution.

To further compare the results of the methods five mill samples were analyzed using seven different methods. Since there are no theoretical sticky values for these pulps all comparisons must be relative to the results of the other methods. Analysis of the lab-generated samples indicated that method E required an offsets to correlate the data. The mill data indicated that methods C and E required offsets. This suggests that the other contaminants in the mill samples may have an effect on the results for these

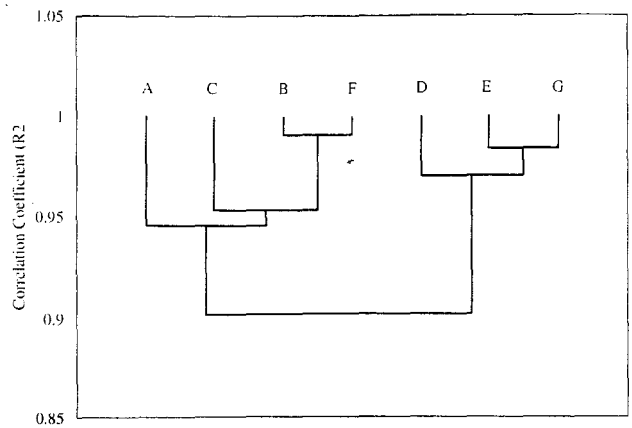

Figure 4. Dendrogram indicating the relationships between the various methods.

methods. The correlation coefficients for the two-parameter linear regressions between the various methods are shown in Table 8.

To explore the relationships between these results, a cluster analysis using the correlation coefficients as a metric was conducted. The results of this analysis are represented by the dendrogram shown in Figure 4. In the dendrogram, the lengths of the vertical lines indicate the level of correlation between methods. Short lines indicate high correlation. Inspection of Figure 4 shows that the methods seem to break into two groups. Furthermore, methods B and F give results that are closely correlated, and methods E and G give results that are closely correlated.

Because the dendrogram indicates that method F is a member of the largest group, the results using this method were

Table 7. **Results of statistical calculations for the data shown in Table 4.**

	Method				
Analysis	C	D	E	F	G
Probability of nonzero offset (t-test)		57%	83%	70%	61%
Slope (m)		198.3	194.5	198.9	217.9
Correlation coefficient (R^2)		0.979	0.366	0.778	0.970
F-statistic of the correlation		137	1	10	97
Probability correlation due to random variation		0.1%	43.6%	4.8%	0.2%
Standard error of estimate		5%	32%	21%	6%
Factor relative to method F		1.00	0.98	1.00	1.10

Table 8. **Cross-correlation matrix for the mill data shown in Table 5.**

Correlation	A	B	C	D	E	F	G
A	1.000	0.937	0.950	0.796	0.768	0.946	0.711
B	0.937	1.000	0.954	0.920	0.933	0.991	0.902
C	0.950	0.954	1.000	0.896	0.850	0.926	0.777
D	0.796	0.920	0.896	1.000	0.970	0.897	0.913
E	0.768	0.933	0.850	0.970	1.000	0.913	0.984
F	0.946	0.991	0.926	0.897	0.913	1.000	0.886
G	0.711	0.902	0.777	0.913	0.984	0.886	1.000

chosen to represent the "true" value for the various mill pulps. Statistical calculations were conducted to test the correlation between the results from method F and the other results. Repeating these tests with other choices of "true" values showed that the conclusions are not sensitive to this choice. Because the results from method F likely exhibit as much random variation as results from the other methods, a modified linear regression was used that is based on the assumption of equal variance for both the dependent and independent variables. This is in contrast to the "standard" linear regression that assumes all the variation to the dependent variable. The results of these calculations are shown in Table 9.

Inspection of Table 9 indicates that the results of all the methods are highly correlated with the results from method F. The probability that this conclusion is due to random variation is very small, $p<0.8\%$. When the slopes in Table 9 are compared to the factors relative to method F shown in Table 6, one can conclude that the results from the mill samples appear to be more similar to each other than the laboratory-generated samples. This observation may be due to the fact that the average particle sizes were smaller for the mill samples, and thus the processing method does not have as large an impact on their apparent size. The COV was also calculated for the mill data. As with the laboratory samples Method C exhibited the lowest variation, and the other method gave values that ranged between 12 and 26%.

The slope values shown in Table 9 can be used to scale the results from each to a common value. This allows one to assess the variation of the values observed. Figure 5 shows a plot of these scaled values on a logarithmic scale. Note that the variation among the scaled values increases as the magnitude decreases. Calculation of the coefficient of variation (COV) shows that for the PFF and PCF samples the standard deviation was 13% of the average, but for the DIP sample the COV was 275%. Specifically, for the DIP sample methods E and C appear to be indicating a significant stickies loading that the other methods are not finding.

Significant variations in the results for DIP seen in Figure 3 can be attributed in part to sample size in relation to relatively low concentration of stickies in this sample compared to others. When sample size is small and stickies concentration is low repeatability error may increase. At the same time, differences in different methods (slot size, image analysis resolution, etc.) may be magnified.

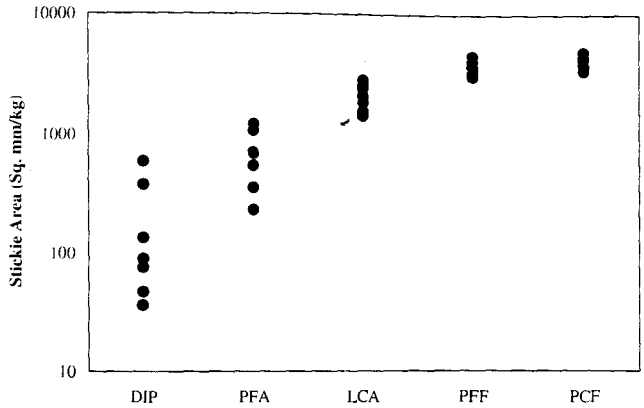

Figure 5. **Plot of the scaled values for the mill samples that shows increased variation as sticky area values decrease.**

CONCLUSIONS

A comparison of the macrostickies analysis methods indicates that all of them are highly correlated with each other. Unfortunately, there appears to be some diversity in the apparent area exhibited by the macrostickies particles as they are processed using the various methods. When particles are small, as with the mill samples, the variation in absolute value is reduced. Clearly, for purposes of process control or troubleshooting in a mill setting, any of the methods will prove useful.

LITERATURE CITED

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2. Dyer, J.M. New deinked market pulp mills: A survey, Part I. Progress in Paper Recycling, 6(1): 12-16 (Nov. 1996).

Table 9. **Results of statistical calculations for the data shown in Table 5.**

	Method						
	A	B	C	D	E	F	G
Slope (m)	0.63	1.52	1.25	1.91	0.29	1.00	0.78
Correlation coeffient (R^2)	0.864	0.964	0.912	0.887	0.800	1.000	0.886
F-statistic	82	349	83	62	52	n/a	23
Probability due to random variation	0.1%	0.0%	0.1%	0.1%	0.2%	n/a	0.8%
Average coefficient of variation	26%	19%	4%	12%	23%	17%	12%

Appendix 1. Black Ink Method (Analysis Method E)

The consistency of each of the samples was measured upon receipt. Each sample was then diluted to a 1% consistency with tap water in a 10-gallon plastic bucket. After dilution the sample was mixed using a motorized stirrer (Lightnin Model: LI U08F) at 300 RPM for 30 minutes to remove fiber bundles. All large pieces of plastic that floated on the surface of the slurry were removed. Each sample was screened, using a Pulmac Masterscreen with 150 µm (0.006") slots, 3 times (17 OD grams per screen) for an individual test. The rejects of each screen were captured on a 20.5 cm white filter paper.

The filter paper, between a metal plate on the heated bottom surface and a Teflon plate on the top, was dried using an Emerson Speed Dryer at 300°F for 5 minutes. The filter paper was then allowed to condition in the TAPPI conditioning room overnight before obtaining a reject weight (data not reported here). The filter papers were then dyed with Parker Quink Ink. Dyeing involved placing the filter paper on the surface of a shallow pool of the ink (screen rejects facing up) until the ink absorbed through the filter paper, typically about 5-10 seconds. The filter paper was placed on a single sheet of blotter paper (screen rejects exposed to air, filter paper in contact with the blotter paper beneath it) and allowed to dry overnight at ambient conditions.

A 20-cm diameter circle was analyzed for each of the three filter papers. The stickies appeared white whereas the background filter paper appeared black. Image analysis using the Apogee Specscan program was as follows:

Reverse Threshold
20 cm round sheets
256 grayscale
400 dpi resolution
Threshold setting, 23 manual
Minimum particle size detected 0.02 mm^2
Set of 3 filter pads per test round

** ** ** ** ** **

Appendix 2. INGEDE Method (Analysis Method D)

Purpose and scope of application
This INGEDE method is used to analyze macrostickies in undeinked or deinked pulp (DIP). It is based on a laboratory screening procedure, where the reject is prepared after screening in such a way that an image analysis of the adhesive impurities can take place.

Equipment
Macro stickies can be separated from recycled pulp suspensions using various laboratory sorting devices. Possible sorting devices are the Haindl classifier (ZM V/1.4/86), the Somerville tester (TAPPI UM 242) or the Pulmac Master-Screen. The use of narrow slots is recommended, e.g. 100µm.

An image analysis system comprising a flatbed scanner and a PC with a suitable control and analysis program is used for the measurements. The flatbed scanner should work according to the reflectance principle with a minimum resolution of 600 X 600 dpi. The software must be able to detect white particles on a black background. The overall procedure is depicted in Figure 1.

Sorting
Before sorting the samples were disintegrated for 60 s at 2.5 % in a standard disintegrator. The Haindl classifier was used for screening (ZM Vl1.4/86), but without connecting to the McNett unit. In order to guarantee problem-free sorting of 50 g of oven-dry deinked pulp, unlike ZM V/1.4/86, the screening conditions were set up as follows. The stroke frequency of the membrane was increased to 480 double strokes/minute (maximum stroke rate). The wash water flow was 10 liters per minute for the entire screening duration. After continuously adding pulp for 5 minutes, the deinked pulp continues to be screened for 5 minutes until screening was complete.

Remark 1: When using a plastic screen, mechanical stress can lead to material fatigue and destruction of the slotted plate. For this reason the use of a metal plate is recommended.
For a statistically sound statement about the macro sticky content, the screening of three individual samples, each containing 50-g of oven-dry material from one sample is recommended.

Remark 2: Increasing the number of tests leads to insignificant improvements to the precision of the measurements only.

Dewatering the reject
After screening in the Haindl classifier, all the reject is flushed from the slotted plate into a container using about a liter of water, and then dewatered in the sheet former (Rapid-Köthen model) using a moistened white paper filter above the sheet forming wire. When the reject sample and an additional liter of water are in the sheet former, the aeration is started before dewatering. After dewatering, the specimen that has been formed is placed onto a couching board with the bottom of the filter (reject-free side).

Drying
The topside of the specimen is then covered with the coated side of the silicone-coated sheet of release paper. Then the sample is dried for 10 minutes in the sheet dryer (Rapid-Köthen model) at 94 °C and a pressure of 95 kPa.

Sticky examination

After drying, the stickies are examined by utilizing their adhesive properties in order to provide the contrast to the specimen's background, which is required for image analysis. In order to achieve this, the silicone-coated release paper should be removed after drying. The dried specimen is then drawn through a submersion bath containing black water-based ink. so that the entire surface is covered. The dyed specimen is then laid with its bottom side on a piece of blotting paper (bleached sheet of cellulose), so that any excess ink is absorbed. Then the specimen is dried for another 10 minutes, the topside covered with the previously used silicone-coated release paper.

In order to avoid discoloration of the drying equipment, the specimen should be placed between two couching boards during drying.

Subsequently, the specimen is completely covered with a thick layer of white special fused alumina powder, the top and bottom sides are covered with couching board and it is then dried for 10 minutes in an oven at 105 °C. The specimen is loaded with a pressure of 950 Pa (6 kg metal plate, 28 cm diameter) to fix the powder on the tacky areas. The metal plate should be stored in the oven permanently to keep the high temperature. After the procedure is complete, the specimen should be removed from the oven. Excess, loose powder has to be removed with a soft cosmetic brush, without applying pressure, while holding the specimen in a vertical position.

After the stickies have been contrasted a visual inspection takes place. This serves to check whether all white hydrophobic impurities such as pieces of plastic film have been removed. In order to do this, the components to be eliminated should either be removed using tweezers or marked using a black permanent marker so that they are not detected during the subsequent image analysis. Since experience has shown that the proportion of these types of particles is small, this manual action only involves a minimum effort.

Image analysis

The prepared specimen is then analyzed using a scanner-based image analysis system. When selecting the measuring area, the preparation area should be used in order to analyze as many of the stickies, which were retained during screening as possible. If only smaller of the surface of the specimen can be measured, the largest possible measuring area should be selected.

When setting the class limits, the size of the slots in the slotted plate that was used for screening should be used as the lower limit (e.g. 100 µm). Smaller stickies cannot be expected because of the sticky surface increase that is associated with the drying process. All other classes can be varied at will. Only the final class may not have an upper limit, so that all stickies are recorded.

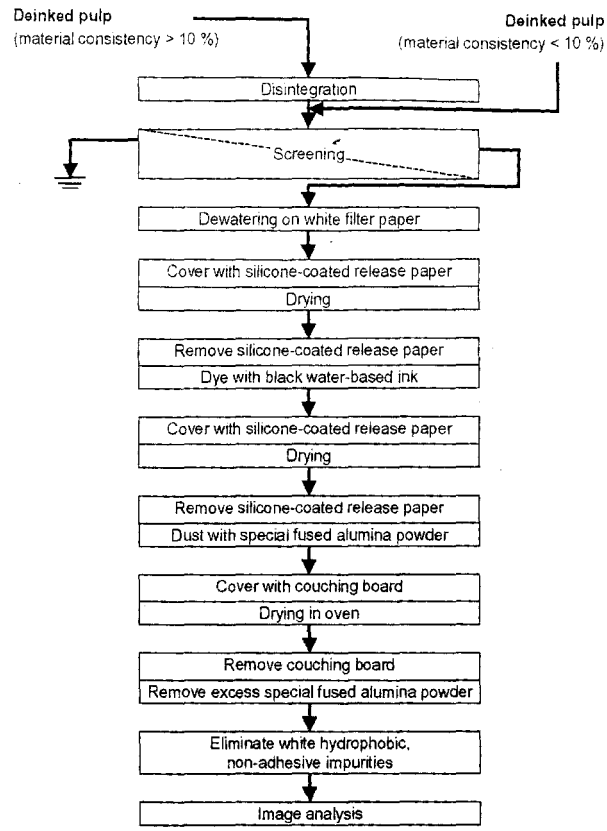

Fig. 1
Flow sheet of the method *IINGED PR 12/9*

Results are expressed as macro sticky area per kg according to the following equation:

$$\text{macro sticky area in } \frac{mm^2}{kg} = \frac{\text{sticky area in } \frac{mm^2}{m^2} \text{ x specimen area in } m^2}{\text{amount of material in kg}}$$

Sources

Reference was made to the following standards in this method:

ZM V/1.4/86: Simultaneous determination of shives and fiber fraction content (in German)

ISO 5263: Pulps - Laboratory wet disintegration

ISO 5269/2: Pulp - Preparation of laboratory sheets for physical testing - Part 2: Rapid-Köethen method

DIN 54 358-T01: Manufacture of laboratory sheets for physical testing - Rapid-Koethen method (in German)

TAPPI UM 242: Shive content of mechanical pulps (Somerville fractionator)

ISO 287: Paper and Board - Determination of moisture content - Oven drying method

Bibliography

Ackermann, C.; Putz, H.-J.; Göttsching, L.: INGEDE *Method for the Analysis of Macro Stickies in DIP*. Das Papier 51 (1997), no. 6, 271-282 (in German)

Ackermann, C.; Putz, H.-J.; Göttsching, L.: *Improved Macro Sticky Analysis for DIP based on Screening.* Progress in Paper Recycling 7 (1998), no. 3, 23-32

References

Special fused alumina powder: Obtained from Institut für Papierfabrikation, TU Daimstadt. Alexanderstrasse 8, D-64283 Darmstadt

Silicone-coated release paper: Herma, Adhesive Paper Department, Sales Department, Fabrikstrasse 16, D-70794 Filderstadt

Filter paper: Binzer & Munktell Filter GmbH, type Ederol No. 12

Ink: Pelikan No. 4001 or Parker Quink.

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Appendix 3. Enzyme Method (Analysis Method C)

In this method an enzymatic treatment of the pulp with cellulases is carried out to prevent the presence of large amount of fibrous materials in the screening rejects (which make it difficult to quantify the stickies). The enzyme treated pulp is then screened through a conventional laboratory screen. The rejects, collected on the plate of the screening device, are therefore nearly free of fibers making quantification of stickies and other contaminants relatively easy (please see reference for more data).

To quantify only the stickies portion in the rejects, the total contaminants are pressed against a filter paper after gentle heating. This step fixes only tacky contaminants, stickies, to the paper. A light brushing of the filter paper removes other contaminants.

Quantification of stickies may be carried out different ways: image analysis, weighing, manual counting. In the work reported in this paper only quantification with image analysis was performed after a coloration of the stickies particles using carbon black. The exact methodology is described below.

A- Enzymatic treatment

- 25 g of pulp (consistency 12 to 15 %) is introduced in a two liter Erlenmeyer flask.
- 700 ml of acetate buffer 0.5 M, pH 4.6 is added.
- 1.25 g (5 %) of cellulases are added (Cellulases mixture Onozuka R 10 - from Yakult Pharmaceutical, Japan).
- The vessel is then placed in a bath equipped with a thermal regulation set up at 37°C and the pulp is stirred with a magnetic stirrer.
- The digestion takes place during 12 hours.

B- Laboratory screening

- The slurry obtained after digestion is directly poured into the laboratory screening device
- At CTP a Somerville apparatus equipped with an 80 µm slotted plate is used. The duration of the screening phase is 20 minutes.

C- Collection of the contaminants

- After screening, the contaminants on the plate are recovered in a large quantity of water in order to avoid the agglomeration of stickies.
- A previously weighed filter paper is placed on the wire of the Rapid-Kothen sheet former.
- The suspension with stickies is poured into the chamber of the handsheet apparatus.
- The agitation system of the sheet former is started, stopped (after a short agitation time) and the water is removed (vacuum).
- The filter is recovered and dried at low temperature (40° C) on a blotting paper.

At this step all large sized contaminants (stickies and non-stickies) are recovered.

D- Stickies fixation

The goal of this step is to separate the stickies (potentially tacky contaminant) from the other non-tacky contaminants.
- The dried filter is placed in an oven at 105°C during 15 minutes (this heating enables to make tacky stickies which are not tacky at room temperature).
- The filter is then pressed against a silicon paper (using a 13-kg roll).
- The silicon paper is then removed and the filter is gently brushed (using a paintbrush) in order to remove the non-tacky contaminants (shives, plastics, pieces of aluminum foil, etc.).

E- Stickies quantification

- The stickies surface is assessed by image analysis after coloration of the stickies particles using carbon black.

REFERENCE

1. Delagoutte,T. and Laurent A.: "Modified method for the quantification of primary Stickies in a recycled pulp" Progress in Paper Recycling, vol 10(4), 14-19 (August 2001).

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Appendix 4. Blue Dye Method (Analysis Methods F & C)

Contaminants, (stickies. caused by adhesives or wood resins, and shives, debris, coarse fibers not suitable for papermaking), which are significantly larger than the diameter of a pulp fiber must be screened out of dilute slurry of pulp fibers before quality recycled paper can be made. A Pulmac Masterscreen can be used to quantify the contaminants that can be removed by typical screening operations.

Sampling

The sampling will ensure a representative random sample has been taken. The sample size will vary, depending upon the level of contaminants present. The test specimen required for screening may be as small as 2 g., or as large as 100 g. Special care must be taken in the sampling of pulp slurries, due to the heterogeneous contaminant distribution in the pulp slurry.

For properly sampling pulp slurries, low consistency and proper agitation are required to minimize the impact of non-uniformity. The multiple test repeatability of contaminant retention from the same source pulp should be ± 5%.

Screening

Standard operation conditions during screening includes: specimen preparation, choice of the appropriate screen slit size 150 mm (0.006 in.), and the desired control water cycle of the Pulmac Masterscreen. After the pulp is processed, the retained contaminants are discharged to a collection basket and collected on a filter paper. The contaminants in the collection basket can be flushed out, filtered, dried and weighed to determine the contaminant content as a percent of the original pulp by weight. Contaminants collected by the AutoFilter can be evaluated subjectively or prepared for an evaluation by optical microscopy or automated image analysis. The sticky counts can be evaluated by first dyeing the sample with Morplas blue dye and then scanning the contaminants on the filter paper with the scanner and automated image analysis.

Hydrophobic contaminant identification method

Reagent

Dye solution 0.67 g of (2.1. solvent blue 36 in 1 liter of n-heptane. The dye is also known as Morplas Blue 1003 and can be obtained from Pylam Products Company lnc., 2175 East Cedar Street, Tempe, AZ 85281, (602) 929-0070

Procedure

I. 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.

2. Place the two blotter papers, with the handsheet between them, on the dryer. The intent is to leave the handsheet firmly attached to the blotting paper until it is dyed. Dry for 3 minutes at 160°C with gentle restraint.

3. Dye the handsheet by applying the dye solution to the backside of the blotter that has a handsheet attached. This allows the dye to uniformly penetrate the handsheet. Furthermore, as the dye solution passes through the blotting paper dye undissolved particles are filtered from the solution. Typically, dying is done by placing the blotting paper/handsheet with the handsheet side down on another blotting paper in a tray, and then painting the blotting paper with a foam brush that has been dipped in the dye solution. This step of the procedure should be carried-out in a ventilation hood to avoid exposure to heptane vapors. Since the dye is a mild sensitizer, heptane-tolerant gloves are also required.

4. 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.

5. 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 white surface, hold the sheet flat on the scanner.

6. Use the color 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 picture point luminance value frequency distribution. The software system developed by Verity IA LLC was used in this study. The scanner used was an AGFA Arcus II. ■