A new stickies test method — statistically sound and user friendly

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ABSTRACT

There is no standardized global method for measuring stickies content in recycled market pulp. This paper proposes a test method that is statistically rigorous and easy to use. Statistical requirements for choosing the correct sample size are explained, and the test procedure is discussed in detail. The use of a specially designed coated paper is an essential aspect of the proposed method. Design specifications for this specialty coated paper are described. The concept of stickies area density is introduced as a useful means of quantifying stickies content. Graphing the stickies area density over the stickies size classes provides an excellent tool for fine-tuning operations to improve stickies removal efficiency across the recycle process.

Application:

A proposed test method for measuring stickies content in recycled pulps ensures that only tacky contaminants exposed to temperature and/or pressure are measured within statistically sound confidence intervals.

STICKIES ARE CONTAMINANTS THAT become tacky when subjected to pressure and/or temperature. These contaminants are introduced into the papermaking process via recovered paper, coated broke, virgin pulp (pitch), and papermaking additives.

The stickies content in a recycling process and in recycled market pulp is an important quality parameter, ranking among such other quality parameters as brightness, color, freeness, ash count, and dirt count. Stickies increase paper machine downtime, impair runnability, create problems in the converting operation, and reduce the visual quality of the finished sheet of paper or board.

North American and European recycle mills specify stickies counts for unbleached recycled pulp (<500 mm^2/kg oven-dry (o.d.) fiber) and for bleached recycled pulp (<100 mm^2/kg o.d. fiber). The highest quality demands are established for recycled pulp used in coated grades. Some sources believe that the stickies count must be below 15 mm^2/kg o.d. fiber to ensure trouble-free coating with a bill blade *(1)*. Unfortunately, there is no standardized test method for quantifying stickies, which renders meaningless these cited quality thresholds for stickies.

In this paper, we propose a new method for quantifying macrostickies content in pulp. Macrostickies are materials that are large enough to be retained on a 0.15-mm (0.006 in.) or a 0.10-mm (0.004 in.) laboratory flat screen. The microstickies that pass through the flat screen plate with the filtrate are not measured. **Figure 1** summarizes the major steps in the proposed method.

Table I lists the various test methods that are being used to quantify macrostickies. Unfortunately, different methods yield different results for the same recycled pulp. Any test method for quantifying macrostickies must fulfill the following three requirements:

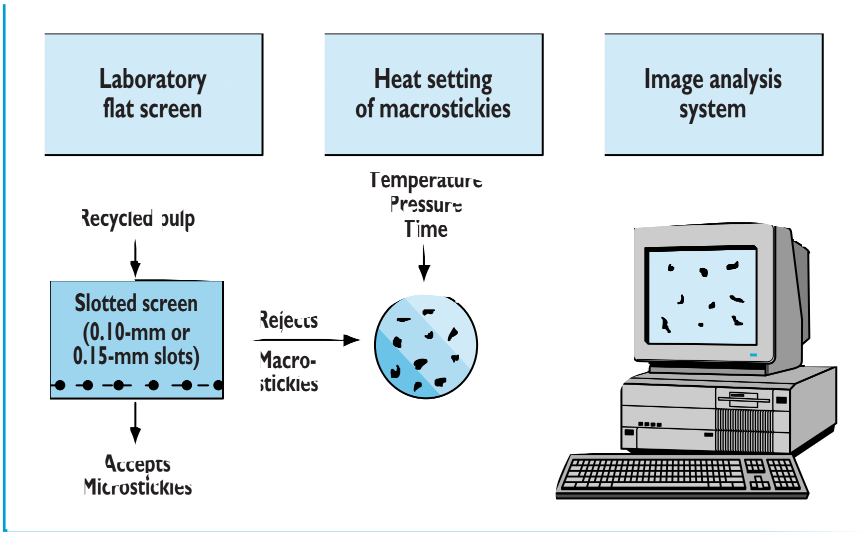
1. The method must measure only the "tacky" contaminants and not all hydrophobic contaminants.
2. Statistics must be in place to support the test method.
3. The time required to conduct the test must be short enough to meet the needs of a paper mill wet/dry lab.

These are the three key design issues guiding any effort to develop a new, improved test method for quantifying stickies.

MICROSTICKIES TEST METHODS

Microstickies in the water circuits of recycling processes and paper machines have a significant impact on paper machine runnability and final sheet quality. In 1994, the European paper mill consortium INGEDE (International Research Association for Deinking Technology) provided funding for two German paper institutes, who were charged with the task of evaluating existing stickies test methods and developing a new, improved method. The institute in Darmstadt focused on macrostickies *(2)*, while the institute in Munich studied microstickies. Although good progress has been made in the development of a method for macrostickies, there has not been a breakthrough in the field of measuring dispersed and/or dissolved colloidal microstickies *(3)*. **Table II** lists the current chemical analytical methods that are used to measure microstickies. As seen in Table II, none of the methods meets the dual requirements of stickies selectivity and quantification.

In North America, the news concerning microstickies is more encouraging: A TAPPI standard practice to quantify microstickies in mill process waters is currently in the ballot process *(4)*.



I. Overview of proposed macrostickies test method

Leykam method
Sulzer–Escher Wyss method
Voith method
Lang method
Haindl method
Berol method
INGEDE method
Chesapeake method
UPM method
Garden State method
Perlen method
Kaipola method
LCC method

* All methods use one of the following flat screens: Somerville, Packer, Valley, Haindl, Pulmac

I. Macrostickies test methods *

FUNDAMENTAL STATISTICS FOR STICKIES TEST METHODS

Statistics determining sample size

Several existing methods use 1.2-g TAPPI handsheets *(5, 6)* or 20-g *(7)* pulp samples to determine stickies content. These sample amounts are insufficient when the stickies concentration in the pulp is low. The ASTM SP D 6149-97 test method recognizes this fact and requires a 100-g sample when a 0.15-mm slotted screen is used. Sample size is reduced to 25–50 g o.d. pulp when a 0.10-mm slotted screen plate is applied *(8)*.

The accuracy of any stickies test method is an exponential function of the sample size. Test methods that use a small sample size can produce numbers that do not reflect the true stickies concentration in the pulp. Selecting the amount of pulp to be tested is a complex matter that is best addressed using statistical equations

Test method	Stickies quantification	Stickies selectivity
Extraction	+	–
IR spectroscopy	–	+
COD *	+	–
Cationic demand	+	–
Conductivity	+	–

* Chemical oxygen demand

II. Analytical methods for quantifying microstickies load in water circuits

We can chart a straight line showing the required sample size as a function of anticipated stickies *(9)*. Some events are rather rare, and we assume—in a mathematical sense—that stickies in pulp are the exception rather than the rule. With this assumption, the probability of the three-dimensional distribution of stickies in the pulp can be described with the Poisson distribution formula. The Poisson distribution is a mathematical rule that assigns probabilities to the number of occurrences. The only thing we have to know to specify the Poisson distribution is μ, the mean number of occurrences.

$$p(k) = \frac{\mu^k}{k!} e^{-\mu} \qquad (1)$$

where

$p(k)$ = probability of finding k stickies
k = number of stickies in a pulp sample of a given size
μ = mean number of stickies in a pulp sample of a given size

The standard deviation for μ, the mean number of stickies, is one of the properties of the Poisson experiment.

$$s = \sqrt{\mu}$$

The accuracy of the stickies test method can be determined by establishing a confidence interval. The mean stickies number μ will fall with a probability of p within the following confidence interval:

$$x - \frac{s}{\sqrt{n}} \cdot t(p) \le \mu \le x + \frac{s}{\sqrt{n}} \cdot t(p) \qquad (2)$$

where

x = test result
s = standard deviation
n = sample size
t = "student" t factor
p = probability

To establish the standard deviation s, various experimental test rows must be conducted. The Poisson distribution allows us to determine the sample test accuracy directly as a function of sample size by solving Eq. 2 for n.

For a desired 80% probability and a sample size larger 100 g, we apply t = 1.282 *(10)*.

When applying real test numbers, μ is replaced with x. This is done because x is the value from a sample size n, while μ is the value one obtains when analyzing all of the pulp. We thus substitute $s = \sqrt{\mu}$ with $s = \sqrt{x}$.

Combining Eqs. 1 and 2 results in Eq. 3:

$$x - 1.282\sqrt{\frac{x}{n}} \le \mu \le x + 1.282\sqrt{\frac{x}{n}} \quad (3)$$

We can rewrite Eq. 3 defining the relative confidence interval v, as seen in Eq. 4.

$$v = \pm\frac{1.282\sqrt{\frac{x}{n}}}{x} \quad (4)$$

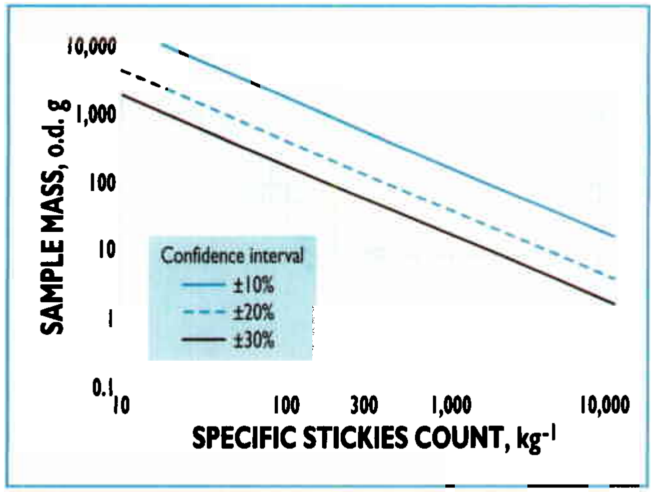
We can then solve for sample size n, as seen in Eq. 5.

$$n = \frac{1.644}{x \cdot v^2} \quad (5)$$

Incorporating the sample size unit (g) and the specific stickies number x (stickies/kg o.d. pulp), we establish Eq. 6, which allows us to calculate the necessary mass sample size m (g) based on a confidence level v (%) and an anticipated stickies number x (kg^{-1}). In Eq. 6, n has been replaced with m. The units of measurement for specific stickies (g^{-1}) was changed to the more practical unit (kg^{-1}), and the confidence level v is now a percent, explaining the "10^7" in Eq. 6.

$$m = \frac{1.644 \cdot 10^7}{x \cdot v^2} \quad (6)$$

The smaller the confidence interval, the larger is the required sample size. The statistical chart in **Fig. 2** is constructed according to predetermined probability and confidence limits. For example, if we anticipate a count of 400 stickies/kg o.d. pulp, and if we desire that 80% of all test numbers fall into a ±20% confidence interval along the straight line, then our statistical chart shows that we need to flat-screen approximately 100 g o.d. pulp.

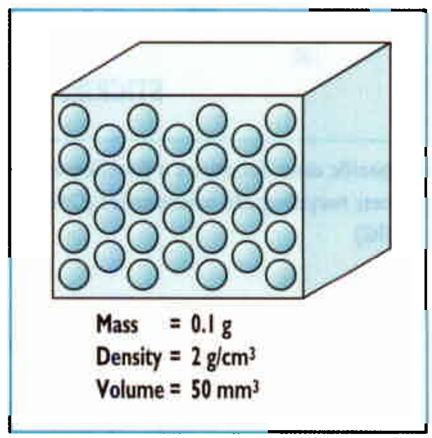


2. Statistical chart showing the effect of desired confidence interval on sample size (mass)

Stickies count, projected area, and stickies heat-set area

Knowing the number of stickies across the recycle process and in the final recycled pulp tells the papermaker only half of the story. Three-dimensional stickies come in many sizes, and counting these stickies by touching them using tweezers or dissecting needles, as often practiced in a recycle mill, is not recommended. Experience has shown that when charting stickies counts across the complete recycle process, a sudden increase in count may occur. This applies to macrostickies screenable with a lab flat-screening device. Such changes are difficult to interpret, and they make it impossible to calculate stickies-removal efficiencies across the machine modules.

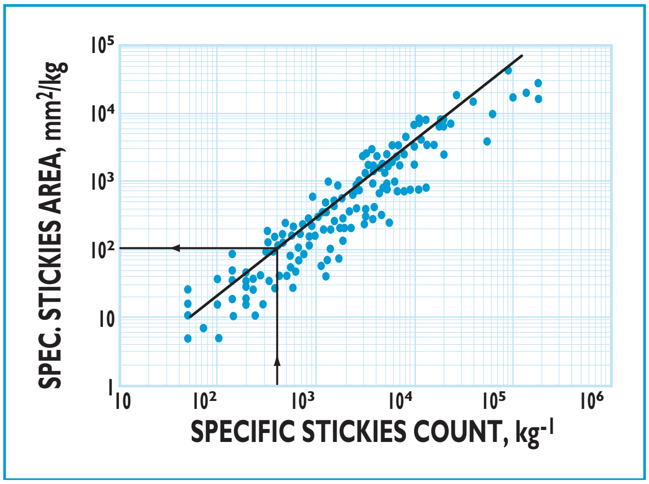
The model depicted in **Fig. 3** and the corresponding data in **Table III** show why methods based solely on counting stickies or on measuring the projected area of the three-dimensional stickies are not recommended. For example, consider a three-dimensional sticky that has been broken down into various smaller stickies by mechanical shear forces in the recycle process. Table III shows different theoretical test results for the same original three-dimensional sticky (Fig. 3) when applying a counting method or a method measuring the projected area. On the other hand, the total



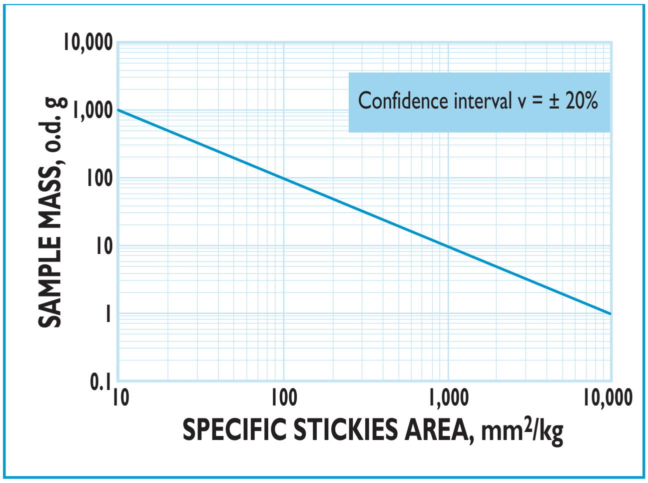
3. Three-dimensional sticky that serves as the model for the calculations presented in Table III

Diameter, mm	Total volume of stickies, mm^3	Volume per sticky, mm^3	Calculated number of stickies per total volume	Area per sticky particle (projected), mm^2	Total projected area, mm^2	Average thickness of heat-set sticky particle, mm	Calculated area of heat-set sticky particle, mm^2	Total area of heat-set stickies, mm^2
1	50	0.52	95.49	0.79	95.00	0.03	17.45	1666.67
0.5	50	0.07	763.94	0.20	150.00	0.03	2.18	1666.67
0.25	50	0.01	6,111.55	0.05	300.00	0.03	0.27	1666.67
0.1	50	0.00	95,495.97	0.01	750.00	0.03	0.02	1666.67

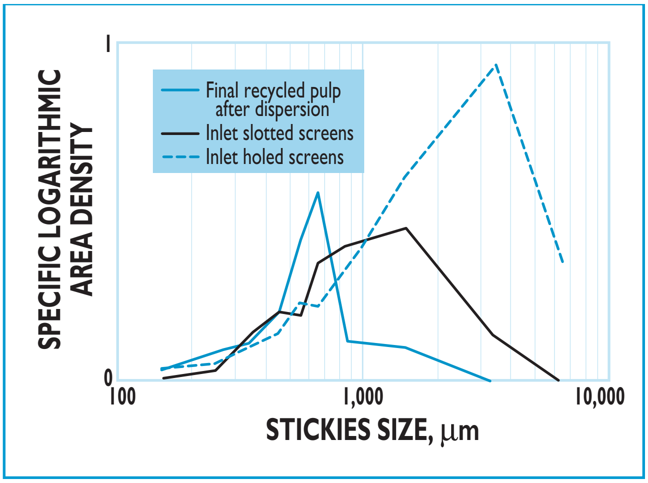
III. Calculated results for 50-mm³ sticky broken down into particles of various size



4. Specific stickies area as a function of the number of stickies



5. Statistical chart showing the sample size required to obtain an 80% probability and a confidence interval of 20% at a given level of stickies content



6. Specific area density as a function of stickies size for a deinking process recycling old newspapers (ONP) and old magazines (OMG)

area of heat-set stickies is the same for different numbers of downsized stickies. In calculating the results in Table III, we assumed a stickies thickness of 30 µm after heat setting. Of course, the assumed thickness in this model would change with different types of stickies.

Stickies count vs. heat-set stickies area

As noted previously, statistics help in determining the required sample size when measuring the stickies numbers. However, statistics are of little help in accurately determining the stickies area per kilogram of o.d. pulp (denoted as specific stickies area). Fortunately, we have access to a database of mill and pilot plant stickies numbers at various locations in the recycle process. Using these data, we developed an average line showing the specific stickies area as a function of the number of stickies, as seen in **Fig. 4**. After combining Fig. 2 and Fig. 4, we can graph the required sample size as a function of the specific stickies area, as seen in **Fig. 5**.

Let us apply this significant chart to two recycled bleached pulps with two different stickies quality specifications: 10 mm²/kg and 100 mm²/kg. We desire an 80% probability and a 20% confidence interval for our test numbers. Figure 5 shows that we need to flat-screen 100 g of pulp for the anticipated 100 mm²/kg stickies. Note that a stickies content of 10 mm²/kg with the same statistical parameters requires 1000 g. This is a key point: Small sample sizes yield secure statistical numbers only when the stickies content (area) is large. *Any* stickies test method that uses a small sample size (e.g., <20 g) needs to be viewed with caution. When measuring the specific stickies area, we recommend classifying the stickies sizes (areas) and stating their distribution. It is obvious that a few large stickies have a larger impact on the total error of the test results than many small stickies. Therefore, we are introducing a new term: specific stickies area density.

Stickies size classification and specific stickies area density

The specific stickies area and the total number of stickies are commonly charted when graphing the stickies test results across a recycle process. We believe that graphing the specific stickies area density yields more information. The specific stickies area density is defined as the specific area in one size class divided by the width of that class.

The image-analysis systems that are used to count dirt and stickies provide classification of the test data. These data can be manipulated to graph the specific logarithmic stickies area density on the y-axis vs. stickies size (or equivalent diameter) on the logarithmic x-axis, as seen in **Fig. 6**. The results in **Table IV** illustrate the required mathematical calculations. For example, consider the size class range between 500–1000 µm:

$$2.303 \cdot \frac{0.232\,\text{mm}^2}{500\,\mu\text{m}} \cdot 750\,\mu\text{m} = 0.8\,\text{mm}^2 \qquad (7)$$

Size (diam.) class range, µm	TEST DATA Measured area, mm^2	CALCULATED DATA Average size (diam.), µm	Specific logarithmic area density*, mm^2
100–200	0.029	150	0.1
200–500	0.186	350	0.5
500–1000	0.232	750	0.8
1000–2000	0.145	1500	0.5
2000–5000	0.037	3500	0.1

* Logarithmic area density = $2.303 \dfrac{\text{measured area}}{\text{net range for size class}} \cdot \text{average size}$

IV. Calculation of specific logarithmic area density

The total area beneath the curves in a chart such as the one depicted in Fig. 6 provides a good visualization of total stickies for each sample.

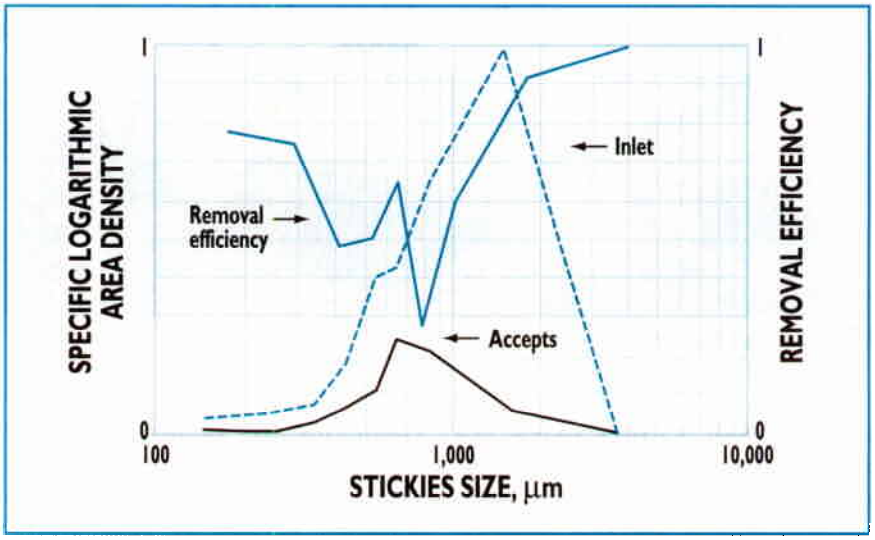
We chose a logarithmic x-axis for better visual interpretation, since stickies sizes span two orders of magnitude. The multiplication with the mean stickies size in the corresponding class—and the use of the correction factor—ensures that the visual impression of the area under the curve corresponds to the stickies area distribution despite the logarithmic scale *(11)*. Application of these multiplication steps ensures that the area beneath these curves represents the total stickies area. The specific logarithmic area densities on the y-axis fall between 0 and 1 and do not have an ISO unit (mm^2) because we divide each number by the maximum logarithmic area density number.

Applications

Figure 6 illustrates the advantage of the proposed graphing method. The results in Fig. 6 are from a deinking process recycling old newspapers (ONP) and old magazines (OMG). The coarse holed screens removed a majority of the specific stickies area. The curve for the finished recycled pulp after dispersion shifts dramatically, showing a smaller total area beneath the curve covering smaller stickies size classes.

Besides the improved informational benefits of these curves, they can also be used as a tool to check the quality of the stickies test. When the correct sample size is used for the test, the curve of the logarithmic stickies area density should be similar to a bell-shaped distribution curve. If the curve shows erratic changes, this indicates that the desired accuracy has not been achieved.

The accuracy of the test results in the various size classes can also be checked by plotting the stickies size removal efficiency. **Figure 7** shows the stickies size removal efficiency for a pressure screen. Note that small, erratic changes in the feed and accept samples over the 700–1000-µm stickies size range cause large swings in the curve depicting stickies size removal efficiency. Further, the drop of the curve in the 100–900-µm range lacks explanation, since it suggests that the efficiency decreases with larger stickies sizes. In such a case, we have two options to rectify the problem. Rerun the test with a larger sample size, or adjust the curve via mathematical corrections. For example, we could adjust the curve by weighting the results for the various size classes as a function of their statistical accuracy. This would provide greater weight to the more accurate test results while diminishing the effect of the less accurate numbers *(12)*.



7. Specific area density and stickies removal efficiency as functions of stickies size for a pressure screen

So far we have covered statistics and data charting, which are independent of the stickies test method being applied. The reliability of the final test results depends on the test method, hardware, and test procedures. Results for different test runs are comparable only when the same method is used.

SUMMARY OF PROPOSED MACRO-STICKIES TEST METHOD

Laboratory procedure

The laboratory procedure is described in **Fig. 8**. (Note that two of the illustrations in Fig. 8 (C and E) depict lab equipment commonly used in Europe.) A sample of unbleached recycled pulp is screened using a laboratory flat screen with 0.15-mm (0.006 in.) slots. In North America, bleached recycled pulp is commonly

8. Laboratory procedure

screened with 0.10-mm (0.004 in.) slots. The rejects from the screen are transferred to a black-dyed wet-strength filter. A specially designed coated paper is then placed on top of the filter paper, and both items are heated and pressed under controlled conditions (time, temperature, and pressure). When the filter paper and the coated sheet are separated after heat treatment, the tacky particles on the black filter remove the white coating from the paper, and a high contrast level is achieved. (This can be described as a "pickup" method.) The filter paper is then washed under standardized conditions with a spray nozzle to remove nonsticky debris particles. The contrast created on the black filter paper can be measured with an image analyzer, which counts particles with a diameter greater than 100 μm.

Hardware

To implement the new test method, we recommend the hardware identified in **Table V**. Note that the proposed method yields different test results for different flat-screening devices or when different operating conditions are applied using the same equipment. A comprehensive study has shown that some flat-screening devices are more aggressive than others in washing macrostickies through the slotted screen plate, thus lowering the final stickies test results. For example, the specific stickies area (mm²/kg) for a bleached recycled pulp was three times higher when it was measured with a traditional flat screen (Valley screen) compared with a fully automated laboratory screening device (Pulmac Masterscreen) *(13)*. We recommend that the image-analysis system have a minimum optical resolution of 65 μm/pixel (400 dpi) and 256 gray levels, satisfying TAPPI T 563. Speck counting starts at a minimum particle diameter of 100 μm (0.0078 mm²).

Table V describes the design specifications for the special coated paper used in the proposed method. We have found this paper to be extremely suitable for this test method.

Sample size

As discussed previously, Figs. 2 and 5 determine the required sample size. The required sample size must be estimated to achieve an 80% probability with a 20% confidence interval. Test results may require an adjustment to the sample size. We recommend initially using a minimum sample size of 100 g o.d. pulp.

RESULTS AND DISCUSSION

Our lab sometimes receives more than 50 samples a day from field and pilot trials. We needed to be certain that the proposed stickies test method would be applicable to the wide variety of bleached and unbleached recycled pulps manufactured in North America and Europe. The new method would also have to be applicable to the complete recycle process—starting with the pulper and ending in the approach-flow screening—covering stickies concentrations of 25,000 mm²/kg down to 5 mm²/kg. Finally, it was crucial that the test method be able to produce statistically reliable results within 30 min.

Over the past six years in North America, we applied a stickies method that was a mix of the Chesapeake/FPL "dye" method *(5)* and the 300-g flat-screen Voith method *(14)*. Our European colleagues applied the Escher Wyss "black board" method *(15)*. The newly developed stickies test method has several significant practical advantages over these other methods.

Repeatability

Upon implementation of the new macrostickies test method in our labs, we achieved a coefficient of variation (COV) below 20%. Results from a bleached pulp sample with controlled stickies concentration levels are included in **Table VI**. A recycled old corrugated container (OCC) sample was also tested, denoted as "unbleached fiber" in Table VI.

As expected, the COV at a confidence level of ±20% is also a function of the specific stickies numbers, as seen in **Fig. 9**. The good news is that nearly all test numbers fall on or below the theoretical curve. This demonstrates that the statistics established for the sample size are crucial for the accuracy of the entire test method. All other issues, such as the impact of lab technicians and the accuracy of the image-analysis system, have less impact on the accuracy of the method if the test procedure is implemented correctly.

Experiences with various recycled pulps

- The new method can be applied to high-quality pulps with extremely low stickies numbers (<50 mm²/kg). For these types of pulps, statistics predetermine sample size.
- Only particles that become tacky when exposed to temperature and pressure are measured.
- The new method discriminates between tacky wax and nontacky wax (a quality issue in OCC recycled pulps).
- The new method measures "glass type" and "black" stickies (a quality issue in mixed office waste (MOW) recycled pulps). Dye methods (applied in North America) usually require two tests with two different contrast levels to pick up both types of stickies.
- The new method uses lab screening devices with slots that are the same width as those in commercial pressure screens (0.15 mm or 0.10 mm).

Hardware	SUPPLIER North America	SUPPLIER Europe
Flat screening device designed to process minimum sample size of 100 g o.d. pulp	Pulmac Somerville Valley	Haindl Packer Somerville
Filtering system with vacuum Silicon-coated paper Black, wet-strength filter paper Glass-fritted funnel	Any labware supplier	Any labware supplier
Special coated paper Recommended size: max 320 mm × 210 mm Plybond resistance: 2 ± 0.5 kN/m [a] 5.5 ± 1.5 kPa [b] Base paper: 70 g/m², wood free, sized One side coated: 50–55 g/m² ISO brightness: 85 ± 3%	Voith Sulzer	Voith Sulzer
Drying/pressing system	Heated Carver press	Rapid–Köthen sheet former
Image analyzer	Microscanner Optomax	Dot Counter

[a] Per DIN 54516 *(16)*
[b] z-direction tensile strength per TAPPI T 541 om-89

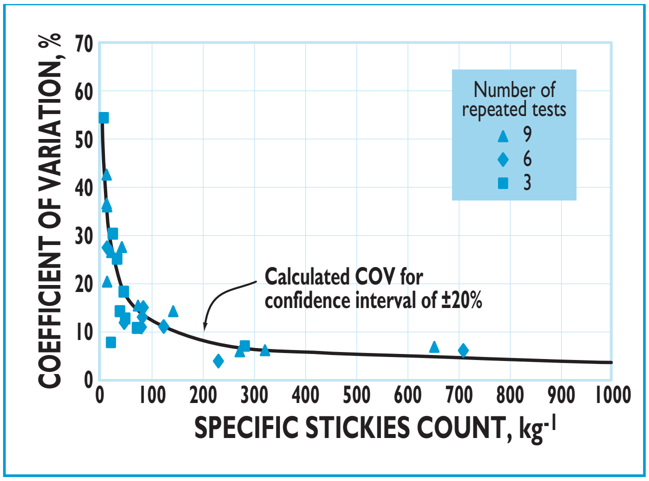
V. Recommended hardware for proposed stickies test method

Trial ID	SPECIFIC AREA, mm²/kg: Bleached fiber Sample A	Bleached fiber Sample B	Unbleached fiber Sample C
1	1518.0	1537.4	3466.2
2	837.8	1889.6	3346.0
3	1489.8	2146.4	3810.8
4	1162.8	2373.8	3218.8
5	1106.6	1918.2	2572.4
6	1411.6	1677.6	3723.8
7	1381.2	1693.8	2924.0
8	…	…	3392.6
Average (x)	1272.5	1891.0	3306.8
Standard deviation (S_e)	247.4	291.1	406.3
COV, % [a]	19.4	15.4	12.3
Repeatability (r), % [b]	38.1	30.2	24.1

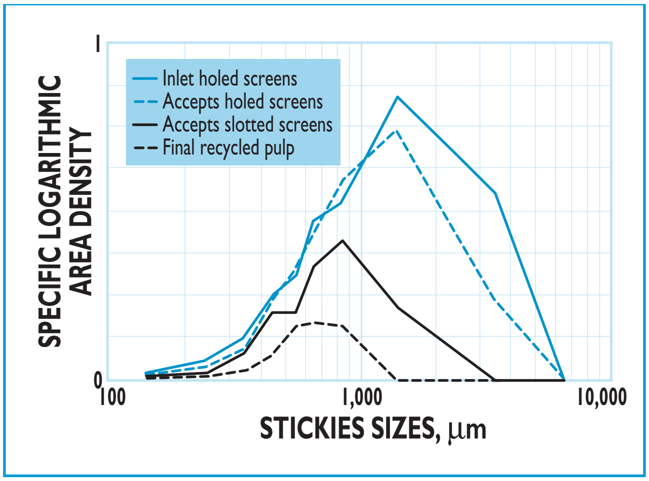
[a] $\text{COV} = 100 \times \frac{S_e}{x}$ (per TAPPI T 1206)

[b] $r = 100 \times \frac{2.77 \times \frac{S_e}{\sqrt{2}}}{x}$ (per TAPPI T 1206)

VI. Measurements of stickies content obtained using the proposed test method



9. Coefficient of variation as a function of specific stickies count; curve represents calculated COV for confidence interval of ±20%



10. Specific stickies area density across the various process modules in a recycle system

Estimate sample size to achieve 80% probability with a 20% confidence interval (minimum sample size = 100 g o.d. pulp)
Flat screen sample with 0.10-mm or 0.15-mm slotted screen
Transfer screen rejects onto black filter
Cover filter with special coated paper
Dry 10 min at pressure = 0.8 bar and temp = 90–95°C
Remove coated paper and rinse filter at 1 bar, 20–25 s, 10 L/min with flat spray nozzle
Post dry
Image analysis of particles ≥100 μm; measure area of coated specks on black filters
Check: Measured stickies area vs. sample size

VII. Proposed test method

Test method application: process optimization

Charts describing the specific stickies area density across various process modules in a recycle system can be used to optimize the equipment and the process. Comparing **Fig. 10** with Fig. 6, note the higher stickies area density in Fig. 10 for the midsize-range classes feeding the holed pressure screens. Note also that the stickies area density curve for the final recycled pulp runs differently compared with Fig. 6. The process and the machine design were different for both charts. Clearly, such charts can be a powerful tool for fine tuning the efficiency of pressure screens in recycle systems.

CONCLUSIONS

A new, improved macrostickies test method has been developed. In developing this test method, we incorporated the research findings of the European paper mill consortium INGEDE. This proposed test method is consistent with the standard practice described in ASTM D 6148, and in addition, it details a specific procedure in which the measurement is based on the thermal properties of stickies. **Table VII** summarizes the steps for the proposed method. The Poisson distribution allows us to define the required test sample size to achieve statistically sound results. The specialty coated paper used in the new stickies pickup test ensures that the method measures only contaminants that are tacky under conditions of elevated pressure and temperature. The design specifications of the specialty coated paper are described in Table V.

A significant new test term—stickies area density—has been introduced. This measurement is useful in fine tuning the recycle process and its equipment. The method has been applied in Europe and North America with success on bleached and unbleached recycled pulps. It is hoped that this method will stimulate the current efforts within TAPPI, CPPA, ASTM, ISO, SCAN, DIN, and ZM, all of whom are searching for a standardized macrostickies test method. The proposed test method described here will be put to a vote by the TAPPI Pulp Properties Committee under Committee Assignment Number CA 980809.2.

TAPPI's Process and Product Quality Division—through a joint ad hoc committee that included experts from TAPPI, CPPA, ASTM, and ISO—successfully developed and approved a documented consensus on automated image analysis of dirt *(17)* (TAPPI T 563, "Equivalent black area (EBA) and count of visible dirt in pulp, paper, and paperboard by image analysis"). Perhaps a similar approach would work in standardizing a global macrostickies test method. **TJ**

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