A NOVEL APPLICATION OF TAPPI T 277 TO DETERMINE MACRO STICKIES DISINTEGRATION AND AGGLOMERATION IN THE RECYCLE PROCESS

Oliver Heise
Director, Technology Center
Voith Sulzer Paper Technology
2200 N. Roemer Road
Appleton, WI 54913-2337
USA

Bangji Cao, Ph.D.
Supervisor, Laboratory Services
Voith Sulzer Paper Technology
2200 N. Roemer Road
Appleton, WI 54913-2337
USA

Dr. Samuel Schabel
R&D Engineer
Voith Sulzer Paper Technology
Escher – Wyss Str. 25
D-88212 Ravensburg
Germany

EXECUTIVE SUMMARY

The TAPPI T 277 test method describes a procedure for determining heat-set area and number of macro stickies in a specified amount of pulp screened. The recycle process is a separation process which removes macro stickies with a high degree of efficiency. Often Stickies balances around these particular process modules do not add up, which leads to the hypothesis that disintegration or agglomeration of macro stickies is taking place in various types of process equipment. The application of statistical mathematical tools yields the ability to calculate the disintegration or agglomeration probability of macro stickies thereby explaining unsatisfactory stickies balances. These calculated findings are of significance, for example, screening equipment could be designed to minimize shear forces on the viscoelastic stickies to remove them more efficiently. Analyzing flotation stickies balances utilizing the hypothesis test (t-test) will lead to a better understanding why in some cases flotation removes and in other cases it does not remove macro stickies.

ABSTRACT

Applying TAPPI T 277, a statistically sound test method, to determine the macro stickies content in recycled pulp revealed that often stickies balances around process modules, e.g., pressure screening and flotation equipment do not add up. Applying fundamental shear force equations within pressure screens and comparing these results to the intrinsic bulk strength of adhesives, gave us indications that adhesives can be fractured in smaller size ranges. Applying statistical mathematical tools allows us to calculate the disintegration or agglomeration probability of macro stickies across process modules in the recycle process.

Application

Determining the disintegration or agglomeration probability of macro stickies in the recycle process will lead to equipment and process improvement separating these #1 pulp degrading contaminants from the recovered paper. A high stickies agglomeration probability with adequate tensile strength prior to fine slotted screening promotes the formulation of a new generation of environmentally benign adhesives.

INTRODUCTION

The North American paper industry perception is that stickies are still the #1 degrading culprit affecting recycled pulp. Stickies cost the U.S. paper industry $650 million a year, according to the American Forest & Paper Association (1).

The PC (personal computer) revolution in the office and at home is creating not less paper consumption but more (2) and unfortunately an increased percentage of adhesive products are included in this paper boom . Shipments of office papers jumped 33% from 1986 to 1997, and experts see no sign this growth will slow (3).

Three additional trends are impacting the ever-increasing level of adhesives in the office paper recycling bins and residential curbside collection sites.

- Mail volume rose 20% from 1992 to 1998,
- Envelope sales were at an all time high in 1997 (3),
- During the last nine (9) years adhesive papers experienced healthy annual growth rates of 6.2%. Forecasters predict a 6.6% annual growth rate until 2003(4).

In 1998 a wood-free recycling mill was the first one to report an increase of stickies, based on a three year study, which monitored stickies entering the recycling process (5).

Of all adhesive types entering the paper recycling stream, the PSA (Pressure Sensitive Adhesives) give the recycling process technology the biggest challenge. In simplified terms we have at the one side of the adhesive product spectrum a general all purpose adhesive which can be described at standard conditions as hard and non-tacky. Standard conditions are defined as a temperature of 23 ± 2ºC and a relative humidity at that temperature of $50 \pm 2\%$ (6). An example is Polyvinylacetate (PVAc) which is commonly used on envelopes. On the other side of the adhesive product spectrum are the soft and tacky more troublesome PSAs (7). PSAs are complex adhesive formulations of polymers, tackifiers, surfactants and rheological modifiers.

We are hypothesizing that adhesives, when exposed to shear forces, are running a probability risk level of being fractured; thus making their removal via screening equipment more difficult. On the other hand, being able to calculate potential stickies agglomeration probability will further enhance the screening separation process. Calculation of the stickies agglomeration/disintegration probability in flotation deinking machines will optimize this module further with regard to micro and macro stickies removal.

Shear Force Fundamentals

Fine slotted pressurized screening has been published as the workhorse of recycling operations for the removal of macro stickies. The most recent long term (three years) operational experience in a mill environment revealed that flotation equipment has a promising future for micro- and macro stickies removal (8). The knowledge around this application is relatively new and the industry data base and experience on macro stickies removal with fine slotted screening far outweighs the knowledge around flotation. Therefore in this section we will focus mainly on the interaction of shear forces within pressure screening equipment and the viscoelastic stickies and how to calculate the effects of their interaction.

We have identified two locations within a pressure screen where shear forces, when exceeding the tensile strength of stickies, can lead to fracturing and disintegration of macro stickies . Based on fundamental calculations the higher shear forces take place in the screen basket slot, compared to the ones around the rotor element (e.g., foil) (9). **Figures 1 and 2** give two examples of this.

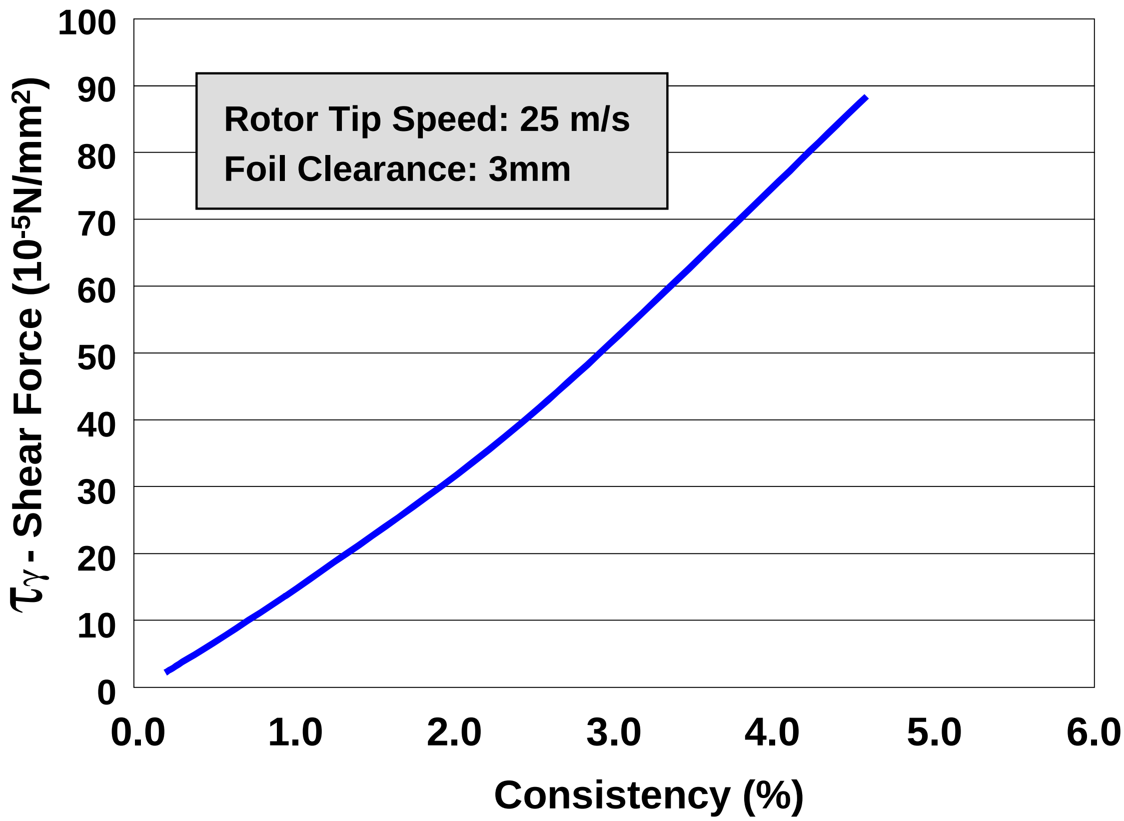


Figure 1. Shear force between rotor element and basket.

Kerekes and Bennington researched extensively tensile strengths of pulp networks and determined power requirements to fluidize the pulp. All their equations show the significant impact of the variable “consistency” on fluidization (10 & 11). In both locations the shear forces are increased with an increase of pressure screen feed consistency.

Applying the TAPPI T 277 macro stickies test method in various deinking screening systems confirmed our theoretical work. Screening systems operating at higher consistencies, requiring aggressive fluidization rotors, revealed lower stickies removal efficiencies compared to screening systems operating in the 1-2% range with gentle fluidization rotors (12).

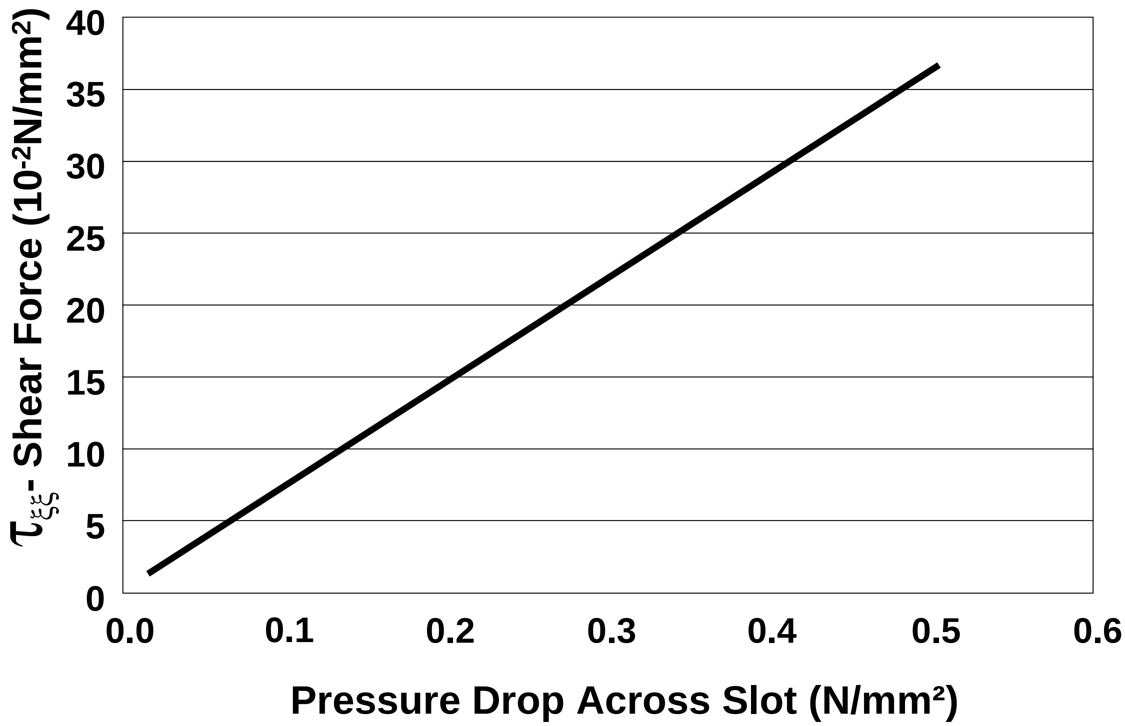


Figure 2. Shear force within 0.15mm pressure screen slot.

Two Industries – Two Different Testing Objectives

Like the research theme done by Wikström and Fredriksson (13), who analyzed the debris breakdown in pressure screening linking it to rheolological pulp properties and basic hydrodynamics, we in turn focused on the research of stickies disintegration and agglomeration in equipment for the recycle process. We learned very quickly that the current test standards applied in the adhesive industry have a different focus compared to the strength testing concepts applied in the paper industry and their machine suppliers. Our desire was to utilize a bulk property test commonly applied in the adhesive industry to determine the inherent cohesive bulk strength of adhesives. This would allow us to compare these numbers to theoretically calculated shear stress numbers in the process equipment. Unfortunately a "straight forward " tensile test for adhesives yields little valuable information for the final adhesive product performance. Therefore commonly applied tests in the adhesive industry are predominantly quantifying tack properties of the adhesive. Contacting adhesive manufacturers for typical intrinsic bulk tensile numbers was an unsuccessful endeavor.

The following illustration (**Figure 3**) summarizes the four most common " strength" tests applied by adhesive manufacturers: the 180 degree peel strength, RT (room temperature) shear, loop and Polyken probe tack test (6 & 14). The RT shear test is the test, which comes the closest with regard to determining the force required for the cohesive split of the bulk material. Unfortunately the adhesive industry measures the failure time when applying the RT shear test at a constant weight and constant initial tack area.

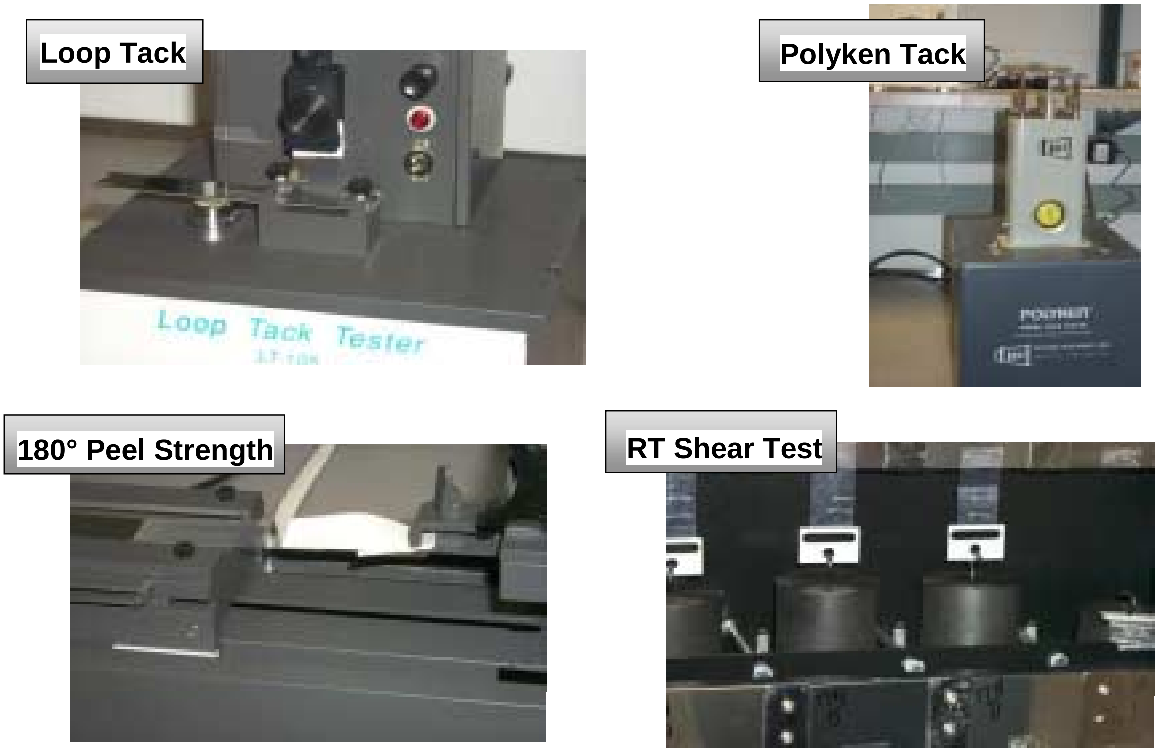


Figure 3. Common adhesive strength tests.

A market analysis of the adhesive industry revealed that the waterborne acrylics have the highest annual growth rate numbers, whereas the solvent born adhesives show decreasing market share. Water based acrylic PSAs also dominate the label market (15).

Therefore waterborne PSA materials are of interest and concern for the recycle process which can have RT shear strength values as low as 0.03 N/mm^2 (15). As mentioned previously, this number is, for our purpose, of little value since the shear stress failure is a function of time in this test. The same strength value was published but utilizing the PSTC - 7 test (6) for a moisture-resistant waterborn PSA adhesive (16).

A typical stickies composition across a wood-free deinking process was given by, formerly known as, Union Camp (5) revealing that the Styrene Acrylates and Acrylates were the most difficult to remove. Fort James identified Poly 2-ethylhexyl acrylate as the culprit passing two fine slotted screening systems in one of their wood-free deinking mills (17).

To get an understanding of the intrinsic bulk tensile strength of commercially available adhesives, we tested one Polyacrylate, Polyvinylethyleneacetate (EVA) and a PVAc at various strain and temperature rates. The purpose of this testing was to support our message that the tensile strength failure numbers can fall in a dangerous band-width of the theoretically calculated shear forces in pressure screening equipment (9). We first tested a Polyacrylate PSA under dry conditions showing higher tensile strength failure numbers at higher strain rates, which revealed the viscoelastic behavior of the polymer. We repeated these experiments, soaking this particular adhesive for three hours at different pH and temperatures, duplicating typical paper recycling conditions. We plotted the two curves (**Figure 4**) for a pH 7.5 over temperature, demonstrating the significant impact of temperature and the liquid environment on the stress at failure.

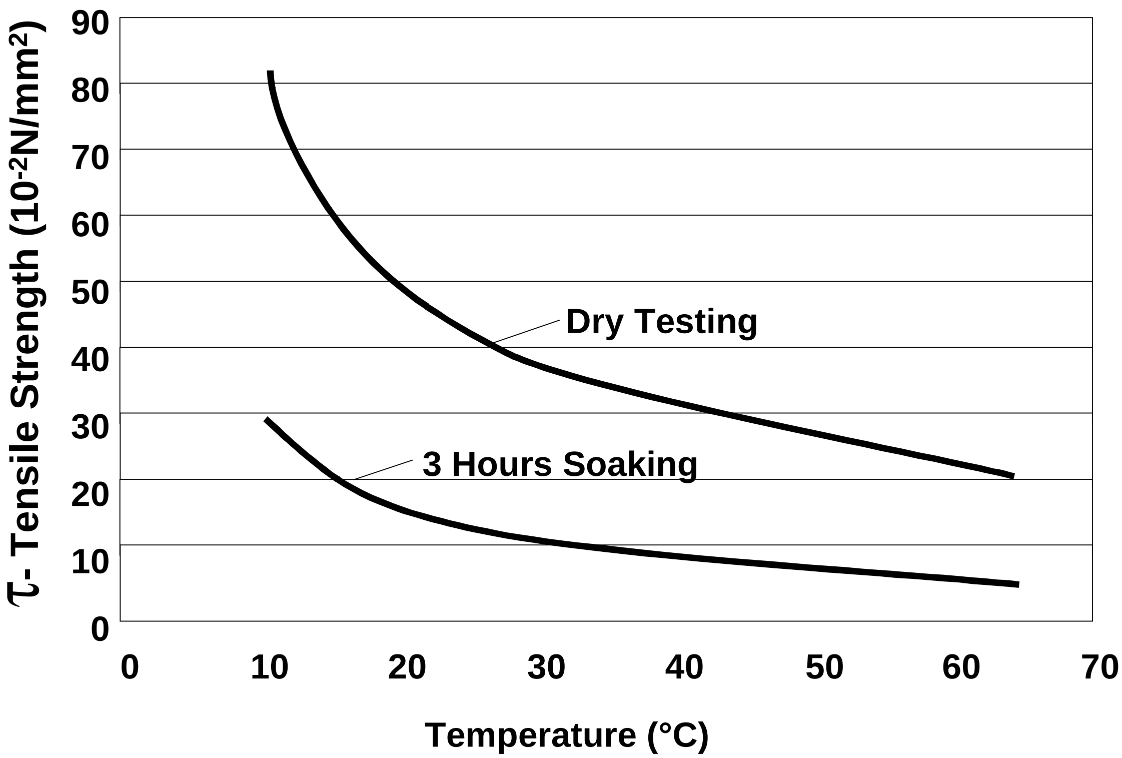


Figure 4. Tensile strength of a dispersible Polyacrylate adhesive.

Paper recycling operations have proven to themselves that the polymer stress-strain curve is a function of the process temperature and that lowering temperatures across their screening modules maximizes stickies removal (18, 19 & 20). Young`s modulus for visco-elastic polymers increases significantly as the temperature is lowered. This implies that the risk of extrusion through a fine slotted screen basket increases with higher temperatures. An interesting phenomena we observed within screening equipment and "aggressive" lab screening devices when applying TAPPI T 227 to determine the macro stickies count, was that PSA type stickies are transformed to a " worm " shape giving evidence of the impact of shear forces (**Figure 5**). We did not see this as pronounced of a change of aspect ratio in "gentle" type of screening devices. These observations were also observed by Venditti et al., using the term "string-like" shape and they attributed this change mainly to the interaction of PSA particles and the recovered paper components (21). Venditti pointed out that this change of shape has a negative impact on the screening efficiency.

In addition to PSA we tested the stress at failure of a PVAc and EVA in a dry environment. The EVA hot-melt showed only a stress of failure at temperatures higher than typically applied in the recycle process. Lower temperatures caused a stress at failure of the substrate, which were carton strips in this particular testing program. The PVAc caused substrate failure covering the whole test temperature range from 20 to 85ºC.

Based on the above testing, we conclude that PSAs are the leading culprits within the multifaceted group of stickies. New thinking and standards on how to test PSAs with regard to their intrinsic bulk strength and how these numbers correlate to shear forces inherent in the recycle equipment is needed.

"aggressive" "gentle"

Figure 5. T 277: Example of "worm shaped" PSA stickies.

Disintegration and Agglomeration Probability

It has been our experience and most recently the experience of others (21) that stickies balances around certain process modules do not add up despite a statistically sound macro stickies test method as TAPPI T 277. This experience refers to specific stickies area and total stickies count measured with TAPPI test method T 277. As previously discussed we have enough indications that the magnitude of shear forces, for example in pressure screening equipment, can disintegrate or fracture macro stickies. **Figure 6** gives a good illustration of our hypothesis of a high stickies disintegration probability when the heat-set stickies area of reject and accepts is smaller then the pressure screen feed.

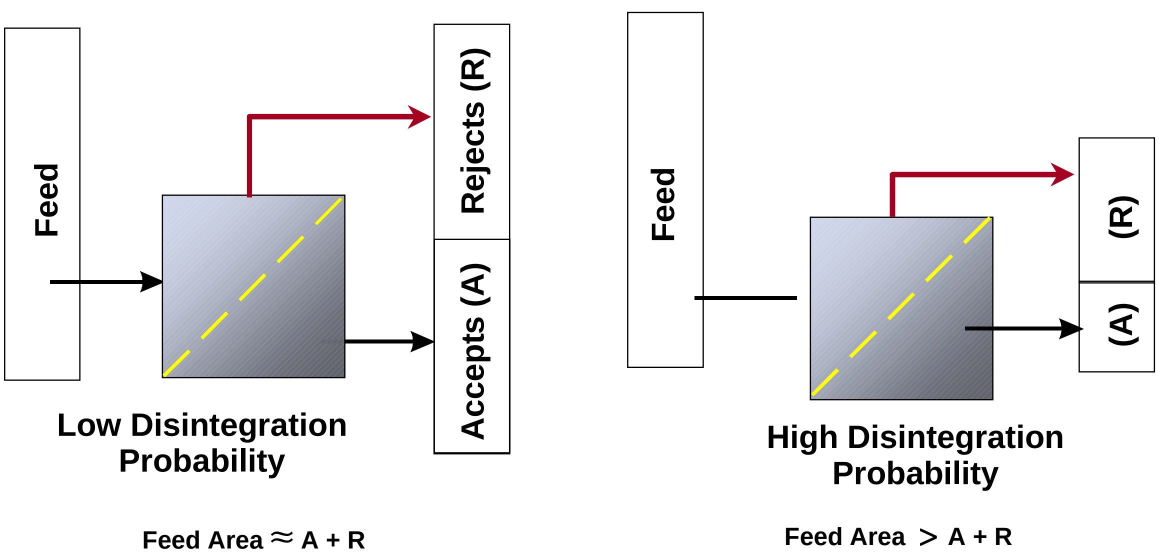


Figure 6. Balancing stickies areas to evaluate the disintegration probability.

The mathematical derivation of the disintegration and probability calculation is divided into a two-step approach. We applied as a first step the work by Hermann. Some of his work covered the analysis of limestone air separation technology efficiency. Hermann utilized statistical equations to correct the size distributions of coarse and fine limestone fractionation balances (22). Our second step entailed the application of classical textbook statistics: t-test (Hypothesis test).

The definition of the test statistic is given by the following equation (23):

$$t = \frac{\bar{x} - x_h}{s / \sqrt{n}}$$

x_h = hypothesized population mean

s = standard deviation

n = sample size value

$\bar{x}$ = measured average

In simplified terms we need to first correct the stickies balances taking also the accuracy of the test method into account, which will yield a hypothesized mean value of a normal population. Then we solve for t and determine the probability that a difference exists between this hypothesized value and the measured value, applying the t distribution curve (**Figure 7**).

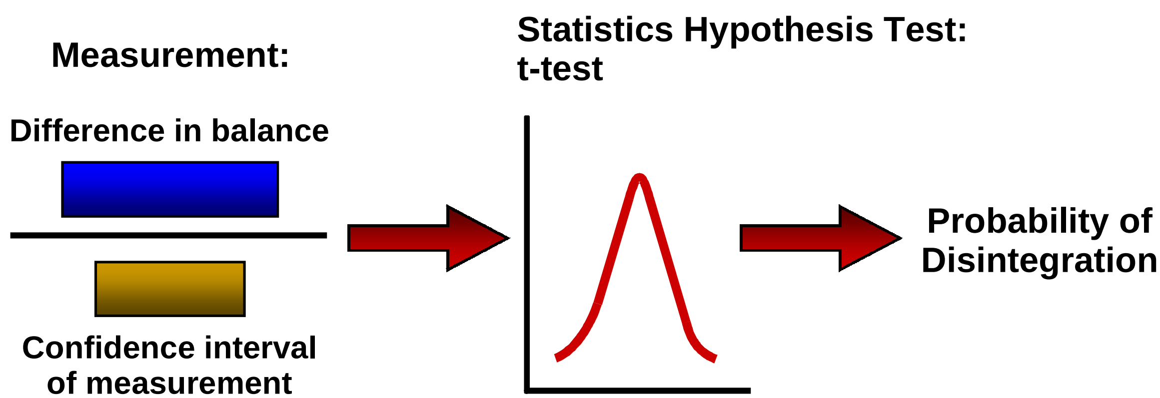
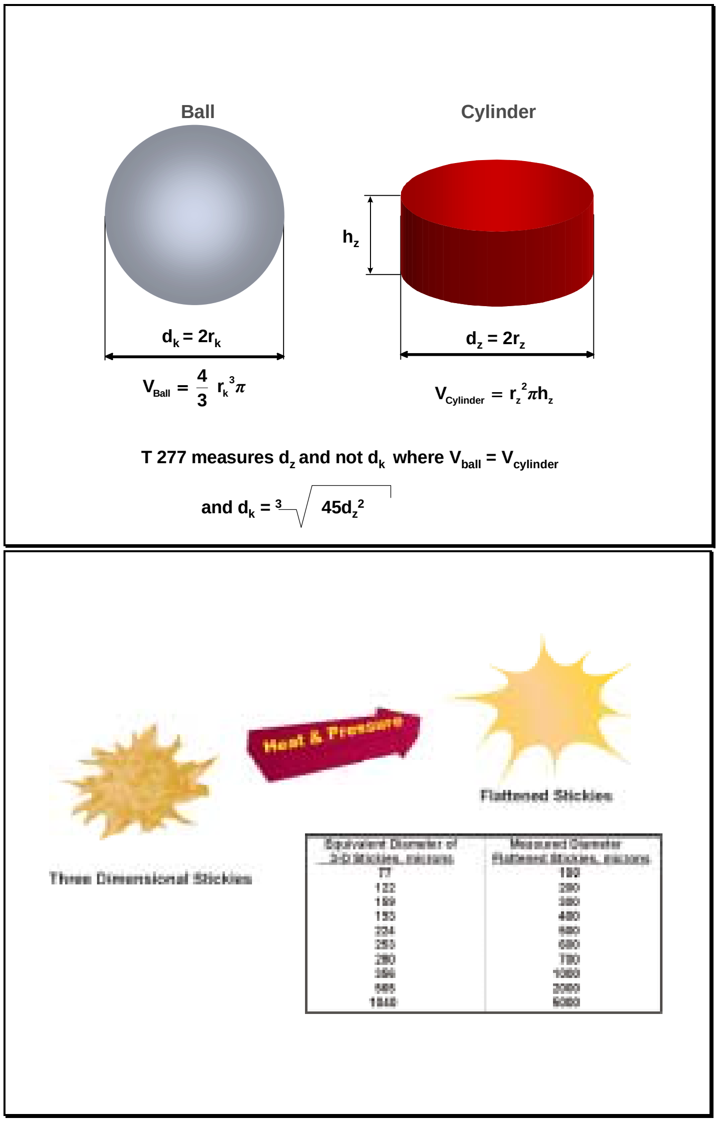


Figure 7. Evaluation of disintegration probability.

TAPPI T 277 macro stickies test method divides the stickies in 10 different sized classes, starting to count at a heat-set diameter of 100 microns. Stickies are tabulated by measured stickies numbers and area (24).

Figure 8 reminds us that TAPPI T 277 converts the three dimensional sticky, due to the controlled heat setting process, into a two dimensional sticky and that a heat set diameter converts to a smaller three dimensional volume diameter (25). Figure 8 reveals the mathematical relationship between the two diameters. Explaining that for the methodology below we started counting with the size class m = 2..n.



Equivalent Diameter of 3-D Stickies, microns	Measured Diameter Flattened Stickies, microns
77	[illegible]
122	280
[illegible]	[illegible]
153	400
224	[illegible]
[illegible]	600
280	700
[illegible]	1000
[illegible]	2000
[illegible]	5000

Figure 8. T 277 Heatsetting of cubical stickies.

Mathematical Derivation

The error $E_m(x)$ of the stickies area balance is calculated for each measured stickies size class m from the integral stickies areas $Q_{o,m}(x)$(feed), $Q_{1,m}(x)$(accept) and $Q_{2,m}(x)$(reject) by

$$E_m(x) = Q_{0,m}(x) - v_1 \cdot Q_{1,m}(x) - v_2 Q_{2,m}(x)$$

The mean relative amounts of stickies area in accept $\bar{v}_1$ and reject $\bar{v}_2$ are calculated by averaging the values for all n size classes:

$$
\begin{aligned}
\eta_m &= Q_{0,m} - Q_{2,m} \\
\xi_m &= Q_{1,m} - Q_{2,m} \\
\bar{v}_1 &= \frac{\sum_{m=1}^{n} \xi_m \eta_m}{\sum_{m=1}^{n} \xi_m{}^2} \\
\bar{v}_2 &= 1 - \bar{v}_1
\end{aligned}
$$

The standard deviations of the measured values for feed, accept and reject $s_{0,m}(x)$, $s_{1,m}(x)$, $s_{2,m}(x)$ are calculated from the number concentration of measured stickies $a_{i,m}(x)$ and the stickies area $Q_{i,m}$ using the Poisson distribution as described in (22):

$$
s_{i,m}(x) = \frac{\sqrt{\alpha_{i,m}(x)}}{\alpha_{i,m}(x)} \cdot Q_{i,m}(x)
$$
$$
\text{with } i = 0..2, m = 2..n
$$

This results in the weighted sum of variances

$$
\alpha_m(x) = s_{0,m}^2(x) + v_1^2 \cdot s_{1,m}^2(x) + v_2^2 \cdot s_{2,m}^2(x)
$$

from which the corrected cumulative stickies area distributions for all size classes can be calculated.

$$
\begin{aligned}
K_{0,m}(x) &= Q_{0,m}(x) - \frac{s_{0,m}^2(x)}{\alpha_m(x)} \cdot E_m(x) \\
K_{1,m}(x) &= Q_{1,m}(x) - \bar{v}_1 \cdot \frac{s_{1,m}^2(x)}{\alpha_m(x)} \cdot E_m(x) \\
K_{2,m}(x) &= Q_{2,m}(x) - \bar{v}_2 \cdot \frac{s_{2,m}^2(x)}{\alpha_m(x)} \cdot E_m(x)
\end{aligned}
$$

Without disintegration or agglomeration effects the amount of stickies for each size class is balanced. We can perform a t-test (Hypothesis Test) to prove the hypothesis that $Q_{i,m}(x)$ and $K_{i,m}(x)$ represent the same value within the confidence interval given by the accuracy of the measuring method. The probability for this hypothesis to be true is a measure of the absence of disintegration or agglomeration. The probability $p_{i,m}(x)$, that $Q_{i,m}(x)$ and $K_{i,m}(x)$ represent the same value is given by

$$
p_{i,m}(x) = P\left[K_{i,m}(x) - t_{i,m}(x) \cdot \frac{s_{i,m}(x)}{\sqrt{a_{i,m}(x)}} \le Q_{i,m}(x) \right]
$$

where t is the Student factor. With $t_{i,m}(x)$ calculated from the above equation and the degree of freedom, which in this case is the number concentration of stickies $a - 1$, the probability $p_{i,m}(x)$ we are looking for results from Student's t-distribution. Tabulated data or numerical software can be used to apply the t-distribution curve. For each size class *m* we use the largest probability $p_{i,m}(x)$ from feed, accept or reject to plot the curves of disintegration versus the heat-set stickies diameter.

Applications

The methodology described above has been applied to a number of pilot plant and mill trial results to obtain information on stickies disintegration in pressure screens and agglomeration in flotation deinking equipment (26).

Applying TAPPI T 277 allows us to chart the heat-set diameter of the stickies on the x-axis and the calculated stickies disintegration probability on the y-axis. Probably the most talked about issue around pressure screening equipment is the desire to screen at higher consistencies to minimize capital cost. Unfortunatly higher fluidization shear forces are required to achieve this objective, which is detrimental to the overall objective of screening macro stickies with high efficiency (8).

The following two graphs give two examples at two different feed consistencies which require different designed pressure screen rotors for fluidization purposes. The stickies disintegration probability of a pressure screen equipped with a high pulse drum-type rotor and a 0.20mm slotted basket at high consistency is plotted in **Figure 9**.

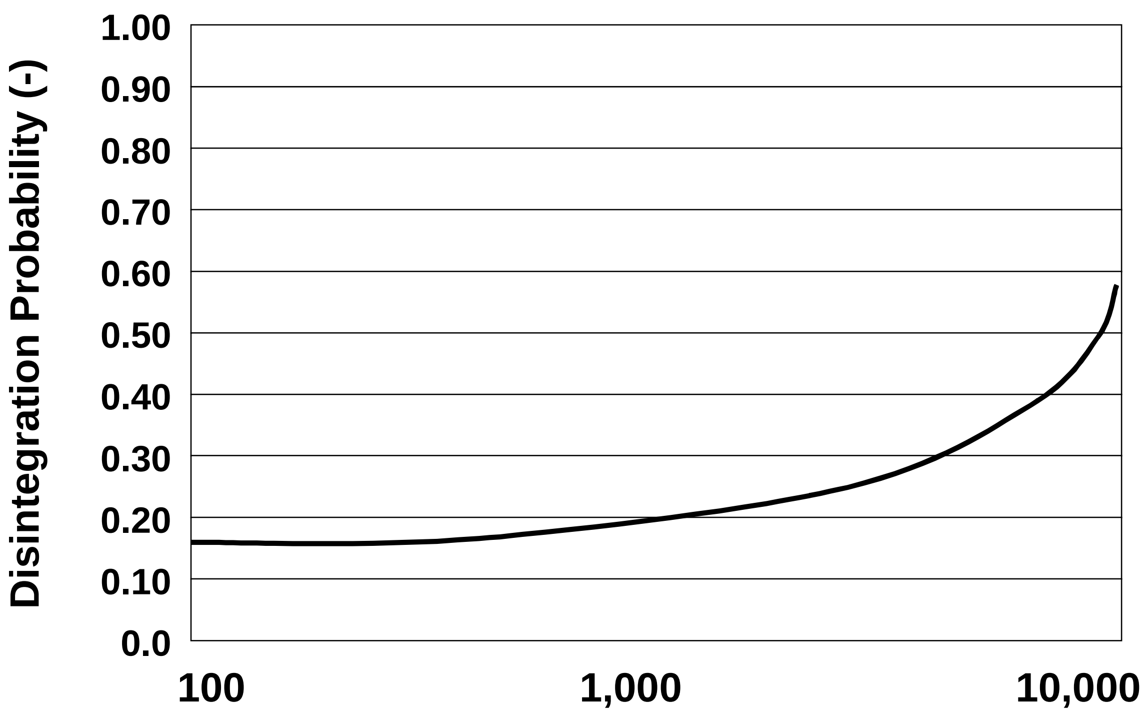


Figure 9. Stickies disintegration in a pressure screen at C_s = 4.4%.

Comparing these results to a pressure screen operated at 1.5% consistency with a low pulse foil rotor and a 0.15mm slotted basket show differences in disintegration effect (**Figure 10**) in the smaller stickies size classes.

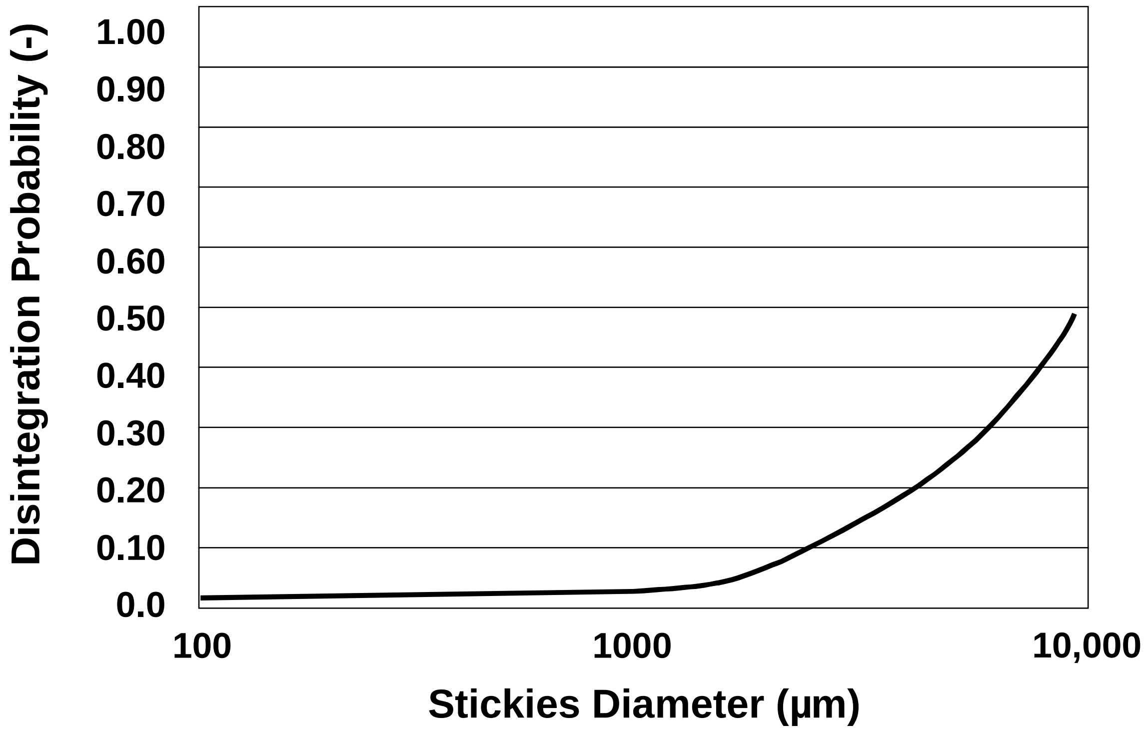


Figure. 10. Stickies disintegration in a pressure screen at C_s = 1.5%.

Another example of applying the proposed mathematical methodology is stickies removal across flotation deinking equipment. **Figure 11** was developed across a preflotation machine in an old newsprint/old magazine (ONP/OMG) mill. Our probability calculations revealed stickies agglomeration.

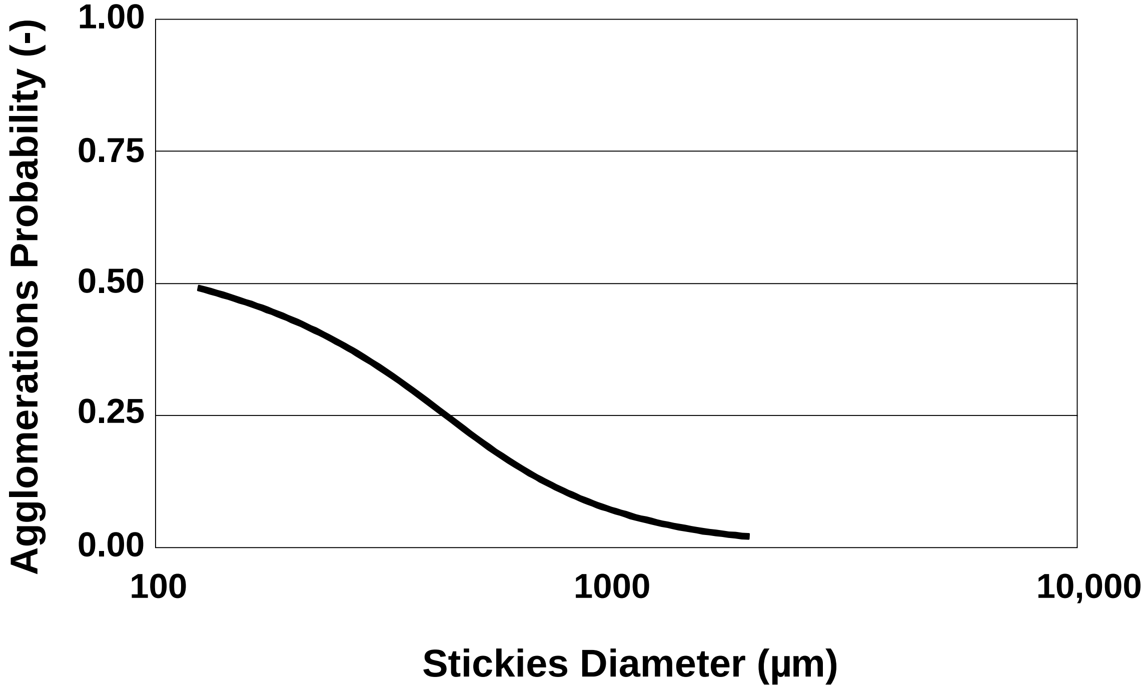


Figure 11. Stickies agglomeration in flotation.

Summary

Pressure screening equipment designed for higher consistencies (C_s > 2-2.5%), causes stickies disintegration due to required fluidization shear forces. Current testing standards in the adhesive industry are not tailored nor can they be applied to analyze this phenomena . Thus making it a difficult task to compare the intrinsic bulk tensile strength with shear forces in this type of recycle equipment. The paper recycling industry has identified the soft and highly tacky PSAs as the most difficult stickies to remove from the recycle process. Based on tensile strength testing results of various commercially available adhesives, especially PSAs, we received indications that the stress of failure can fall into a band-width of shear forces, typically encountered in pressure screening equipment especially at higher consistencies. Therefore we developed a method to determine the disintegration or agglomeration probability of stickies in recycle equipment. Beyond pressure screening equipment this method can be applied to analyze stickies agglomeration removal in flotation deinking equipment, helping us to explain better why stickies removal in some case works and in other cases it doesn't work. Further adhesive formulations could be evaluated beforehand in a pilot plant environment to minimize the risk factor of disintegration in the mill recycle process. A high stickies agglomeration probability with adequate tensile strength prior to fine slotted screening promotes the formulation of a new generation of environmentally benign adhesives.

This paper was presented at the TAPPI 2000 Recycling Symposium in Washington, D.C., and received the best paper award in the category for innovation.

NOMENCLATURE

E	=	error in cumulative size distribution
K	=	corrected cumulative size distribution
Q	=	cumulative size distribution
α	=	number concentration of stickies area [1/kg]
n	=	number of size classes for stickies measurement
p	=	probability
s	=	standard deviation
t	=	Student's factor
ν	=	relative amount of stickies area
η	=	difference in cumulative size distribution. (feed - reject)
ξ	=	difference in cumulative size distrib. (accept - reject)
x	=	heat-set stickies diameter (μm)

SUBSCRIPTS

$_i$	=	feed (0), accept (1), reject (2)
$_m$	=	size class

REFERENCES

1. Seller, E., USA Today "Paper Recyclers Unable to Lick Sticky Problem" 3A (10-7-97).
2. Watson, T., USA Today "Paperless Office Still a Pipe Dream" 12B (3-8-99).
3. O'Brien, J., *Paper Age*, 115(5):10 (1999).
4. Fales, G., *Walden's North American Pulp & Paper Report*, 1(6):4 (1999).
5. Meese, G. and Pikulin, M. *International Recovered Paper IX Proceedings, II-2* (May 1998).
6. Pressure Sensitive Tape Council, "Test methods for pressure sensitive adhesive tapes" (12th ed.) Chicago.
7. McLennan, I. J, *Presentation Handout at PAPTAC 5th Research Forum on Recycling*, Ottawa, CAN (Sept. 1999).
8. Heise, O., Kemper, M., Wiese, H., et al., *TAPPI Recycling Conference Proceedings*, TAPPI PRESS, Atlanta, p. 693 (March 1999).
9. Heise, O., Schabel, S. and Cao, B., et al., *1999 PAPTAC 5th Research Forum on Recycling Proceedings*, Ottawa, CAN, p. 187.
10. Kerekes, R. J., Sozzyunski and Tam Doo, P. A., *Fundamental Research Symposium Transactions*, Oxford, England, p. 265 (Sept. 1985).
11. Bennington, C. P. J. and Kerekes, R. J., TAPPI JOURNAL 79(2):253 (1996).
12. Fursey, M., *TAPPI Pulping Conference Proceedings*, Atlanta, p. 433, (Oct. 1999).
13. Wikström, T. and Fredriksson, B., *1999 PAPTAC 5th Research Forum on Recycling Proceedings*, Ottawa, CAN, p. 197.
14. *1998 Annual Book of ASTM Standards*, Vol. 15.06 Adh., West Conshohocken, PA
15. Blombergen, S., McLennan, I. J. and Schmaltz, C. S., *Adhesive Technology* (Vol. 16) p. 10, (Sept. 1999).
16. Merkel, P. L. and Lydrinski, S. E., Tappi J. 74(12): 114 (1991).
17. *Personal Communications with Robert de Jong, Research Fellow*, Fort James, Neenah, WI (Oct. 1999).
18. Abubakr, S., Thomas, L. A. and Bormett, D., *5th PIRA International Recycling Technology Conference Proceedings*, Paper 13, (Feb. 1998).
19. Ortner, H. and Pfalzer, L. WfP 15:W535-W539 (1983).
20. Pearson, N. and Fullick, C., *Progress in Paper Recycling* 6(4):82 (1997).
21. Venditti, R. A, Chang, H. and Jameel, H., *PaperAge* 115(2): 18 (1999).
22. Herrmann, H., *Chem. Ing. – Tech* 51(11):1140 (1979).
23. Devore, J. and Peck, R. *Statistics: The Exploration and Analysis of Data*, Second Edition, p. 456 (1993).
24. TAPPI T 277 pm-77 Test Method, "Macro Stickies Content in Pulp, the 'Pick-Up' Method", TAPPI Press (July 1999).
25. Heise, O., Cao, B., Dehm, J., et al., *TAPPI JOURNAL* 82(2):143 (1999).
26. Schabel, S., Rienecker, R. and Schweiss, P., *PTS Stickies Symposium Proceedings, Munich, 10-1* (Nov. 1999).

Received: December 21, 1999
Accepted: April 5, 2000

This paper was accepted for abstracting and publication in the November issue of *TAPPI JOURNAL*

TAPPI Website: www.tappi.org