Development of a packaging test method

CARL HOUTMAN, HANNAH HAYNES, ASHOK GHOSH, SEONGKYUNG PARK, AND COLLEEN WALKER

ABSTRACT: Innovation in packaging design will be facilitated by a simple test method to indicate whether a product is compatible with paper recycling. Three laboratories cooperated on the development of a method and used it on linerboard, coated paperboard, wet-strength paperboard, and white copy paper. This test method includes pulping and screening.

The data presented here illustrate the factors affecting the results of a bench-scale test. Our observations show that a bench-scale test can give reproducible results for yield, < 5%. We also present an approach to contaminant assessment based on current published test methods. A specification with a lower limit on yield of 70% and a contaminant level of less than 5,000 particles/kg is proposed.

Application: The purpose of this work is to provide a validated test method for determining yield during recycling of packaging materials.

For any papermaking operation, fiber is the largest cost. Naturally, papermaking facilities have repulping equipment to recover fiber from internally generated materials, e.g., web break material (often called broke), trimmings, off-specification product, etc. This internal recycle loop has always been a part of papermaking, even before paper machines were invented. In integrated facilities, materials generated during converting operations can also be recovered in a repulper.

Industrial-scale papermakers balance price, performance, and runnability when they are obtaining fiber. Generally, the best recovered fiber comes directly from industrial sources. For example, new double-lined kraft corrugated cuttings (DLK, ISRI 13) [1] is a more desirable material that often sells for $20/ton more than old corrugated cardboard (OCC, ISRI 11) [2]. The Institute of Scrap Recycling Industries (IRSI) circular lists several other sorted grades that often command a higher price. On a tonnage basis, these industrially produced materials account for more than 75% of paper recycled into new products.

The other major source of recovered fiber is products that have been used and discarded, often called post-consumer. At times, consumer sentiment, government rules, and third-party certifications have favored fiber recycling to meet longer term goals. A combination of these factors has driven demand for paper recycling up and down over the last 150 years [3]. In North America and Europe, as machine-made paper became widely available, social practices around hygiene tended to favor disposal or incineration over recycling. Fiber shortages during the World Wars swung the balance towards recycling, but after the demands of the war effort subsided, discarding paper returned to being the norm. In the 1970s, concerns about landfill capacity led to paper recycling mandates and post-consumer content requirements on paper products [4]. In the middle 1980s, cities in North America started to implement curbside recycling programs. With a growing availability of recovered paper, companies responded by adding recycling capacity in the 1990s.

Since 1990, there was a steady increase in the paper recovery rate until 2010, when it began to level off at just under 70% [5]. Aggregation of recovery rates into a single number is misleading. OCC, for example, is recovered at nearly 90%. Other mixed paper grades have recovery rates near 30%. In addition, there have been large shifts in the grades of paper produced. Printing and writing grades have been steadily declining, and board and packaging grades have increased. As the grade mix of production has changed, so have the markets for products using recycled fiber. Corrugated board, paperboard for packaging, and tissue grades are now the major markets for recovered fiber.

As recovery of fiber grew in the early 1990s, interest in designing products compatible with recycling began. The first major push was to improve OCC recovery. To provide resistance to water, corrugated boxes were coated with waxes. In 1994, the Fibre Box Association (FBA) and American Forest and Paper Association (AF&PA) initiated a research study at the Institute of Paper Science and Technology (IPST) to develop a method for determining when a coating interfered with OCC repulpability [6]. After several revisions, the proposed test method became the "Voluntary standard for repulping and recycling corrugated fiberboard treated to improve its performance in the presence of water and water vapor," released in 2013 [7]. The opening sentence of the Purpose section reads: *"The application of this Voluntary Protocol is only for 'linerboard' (including Kraft*

Paper), corrugating medium, combined board, and corrugated products made from these materials, collectively known as 'corrugated fiberboard'." While this method has been recently updated [8] and applied to a wide range of paper grades, its intended purpose was to address water barrier coating on OCC.

In 1994, the United States Postal Service (USPS) approached the U.S. Department of Agriculture's Forest Products Laboratory (FPL) to help develop a test method and specification to determine when a pressure sensitive adhesive on a postage stamp is compatible with paper recycling. Over the course of the next several years, the FPL, the USPS, and a private testing lab collaborated on bench, pilot, and mill-scale tests of 15 candidate adhesives. Analysis of these data showed a strong correlation between the mill and pilot results [9,10]. Further refinement of the bench-scale testing determined that bench scale testing does indeed sufficiently predict the performance of a pressure sensitive adhesive during paper recycling [11]. The United States Executive Order 13148 specified that the test method developed by the USPS be applied to purchases in the U.S. government. With both governmental backing and industry collaboration, the test methods were collected as a standard with the Tag and Label Manufacturers Institute (TLMI). This standard went through many revisions; the most recent revision was released in 2007 [12]. The goal of this test method is to determine when a pressure sensitive adhesive is sufficiently removed by typical paper recycling unit operations. At the time, printing and writing grades were preferred, so slotted screening and flotation were chosen as representative unit operations. The essence of the method is to make handsheets after pulping, screening, and flotation. The handsheets are then dyed to develop contrast between the adhesive particles and the fiber. The results of image analysis are reported with a threshold of 10 ppm in the flotation handsheets as indicating a recycling compatible adhesive. Since their release, the TLMI specifications and test methods have been applied to a wide range of products. This test method is used to test approximately 15 samples per year.

More recently, the Confederation of European Paper Industries (CEPI) has developed a bench-scale test method for assessing recyclability of paper products [13]. This effort is a coordinated response to the European Union's Circular Economy Action Plan (CEAP) adopted in 2015. The test method involves pulping and two screening steps, using a 5-mm hole screen followed by a 0.15-mm slot screen. After the screening steps, the pulp is evaluated for adhesives and other contaminants. In addition, the filtrate water can be tested for dissolved substances. This test method appears to be widely practiced in Europe and is now becoming a standard for many packaging materials.

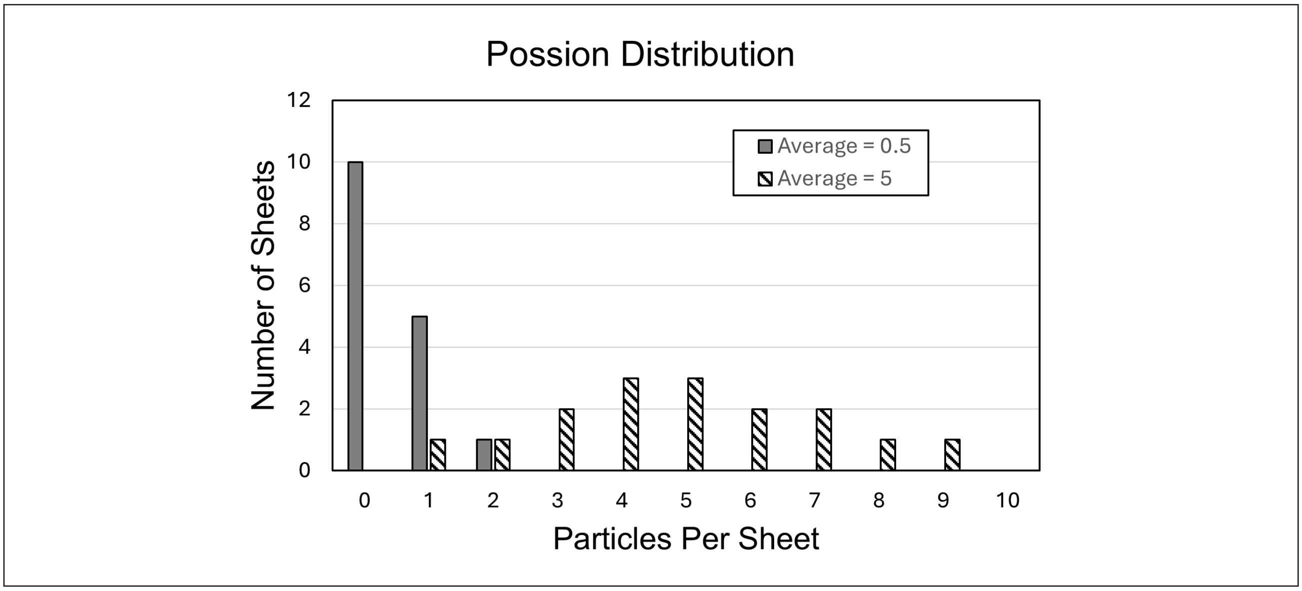
In North America, the certification approach has been taken to encourage brand owners to develop products that are compatible with paper recycling. A fee-based third-party certification process has been developed under the umbrella of GreenBlue's How2Recycle. The How2Recycle program requires a brand owner to apply to have a specific product assessed. The assessment follows a product from being discarded by a consumer, collected, sorted, and reprocessed into a final product. The reprocessing part of the certification is based on taking materials through simulated recycling unit operations. Once a product has been assessed to be recyclable, it can be labeled to help the consumers know how to recycle it.

Most recyclability test methods include yield measurement and stickies assessment. In this context, stickies are defined as any substance that can be found adhering to process equipment or paper webs. Methods for measuring yield provide reproducible results, but fundamental chemical, physical, optical, and statistical difficulties limit reproducibility of stickies measurement.

From the earliest days of recycling post-consumer recovered paper, stickies formation caused operational and product quality problems. Recent analysis shows that stickies in the dryer section of a paper mill are mixtures of a wide range of polymers [14] that are coming into processes as various coatings, polymer films, pressure sensitive adhesives, natural wood extractives, and silicone release coatings. A wide range of polymer chemistries are used in modern packaging. Some materials are tacky at room temperature, and some are not. While there has been significant previous work on method development, no universal method for stickies determination has emerged.

Stickies measurements require a chemical, physical, or optical property to be different than fiber. Many methods apply optically contrasting material, powders, or colored papers onto stickies with pressure and heat, as with TAPPI Standard Test Method T 277 "Macro stickies content in pulp: the 'pick-up' method" and other methods [8,13]. Others use hydrophobic selective dyes [12]. Doshi et al. [15] showed that there is a strong correlation between the number of particles per kilogram of pulp for methods based on powder coatings and the hydrophobic dye. The correlation was less strong for apparent total area of adhesive identified. This is likely due to particles being deformed during application of heat and pressure, which changes their projected area during image analysis. Subsequently, Sithole and Fallon [16] also compared sticky measurement methods. They observed correlations between methods and concluded that methods based on count are more reliable than those based on area.

Once contrast between fiber and particles has been developed, image analysis can be applied. Getting a reliable measurement for a pulp with few particles presents a fundamental statistical problem. Sampling rare events, i.e., particles per sheet for a clean pulp, results in Poisson distributions, which approximates a Gaussian distribution when the number of observed particles becomes larger, as illustrated by **Fig. 1**. Note that with the average number of particles per sheet equal to 0.5, there are no particles found



1. Predicted number of sheets exhibiting the observed particles per sheet for a measurement following Poisson distribution.

in most of the sheets. When the average number of particles per sheet is increased to 5, the distribution becomes more similar to a Gaussian distribution. For methods applied directly to handsheets, e.g., the TLMI method, the error bound on the measure is proportional to the number of particles observed. Thus, one can improve the measurement by making more handsheets. Alternatively, the CEPI method attempts to address this problem by concentrating the particles with a fine screen and transferring rejects to colored filter paper. One is left with a choice, either to make many handsheets from the screen accept pulp, i.e., >16, and see all of the visible particles that are passing through the slotted screen, or to collect the reject particles from the slotted screen on a single filter paper and ignore small particles passing through the screen.

The purpose of the current work is to provide data to support the development of a reproducible test method for determining yield and contaminant levels in fiber-based packaging. The ultimate goal of the work is to get an approved TAPPI Standard Test Method for recyclability. To this end, a group of laboratories have been recruited to conduct this test method on a set of defined materials. The results of this larger study have been used to calculate the repeatability and reproducibility as defined in TAPPI T 1200 “Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility.” As this test method becomes more widely accepted, it is hoped that the staff at How2Recycle will consider using it as evidence that a particular package can be technically reprocessed into a new fiber product.

METHODS

Three commercial products were obtained from Smurfit Westrock representing major classes of paper and paperboard products. White envelope paper was obtained in 1998 from Union Camp as part of the USPS project. This particular paper was specially produced to have very low stickies and dirt. Several large rolls of this material have been retained for use as the background paper when conducting TLMI recycling-compatible adhesive testing. **Table I** shows the major specifications of the papers or boards used in this study.

The boards or papers were cut into 75-mm strips and then passed through a paper shredder to give 75 mm x 6 mm pieces. Initial trials with pieces of the wet strength board that were 300 mm x 6 mm showed that they overloaded the disintegrator’s motor.

Three laboratories—Forest Products Laboratory (FPL), Smurfit Westrock (SWR), and University of Maine (UM)—used their own existing equipment to conduct these experiments. There was a desire to use pulpers that better reflect commercial practice. The pulpers at these three laboratories were similar to Adirondack Pulper 450H (Adirondack Machines; Hudson Falls, NY, USA), however, initial interlaboratory comparisons showed that pulping results lacked reproducibility due to after-purchase modifications of the pulpers. Considering that CEPI have chosen to use a disintegrator for pulping, similar to the one described in TAPPI T 205 “Forming handsheets for physical tests of pulp,” and that most pulp laboratories have this device, it was decided to use it as a pulper for this work.

All three locations have Somerville-type screens similar to the device described in TAPPI T 275 “Screening of pulp (Somerville-type equipment).” The slotted screens had nominally 0.15-mm slots. The screens at FPL and UM are more than 20 years old, and feeler gauge testing showed that the slots were within 0.02 mm of the correct size. The SWR slotted screen is new and determined to have slots of the correct size. A 5-mm hole screen was made at the FPL

Name	Kraft Liner	Wet Strength	Coated Board	Envelope
Basis Weight, g/m^2	280	320	320	90
Coating	none	clay/carbonate	pigmented barrier	none
Fiber	softwood kraft	softwood kraft	softwood/ hardwood kraft	softwood kraft
Filler	no	no	yes	20% $CaCO_3$
Bleached	no	no	yes	yes
Wet strength	no	yes	no	no

I. Characteristics of the paper and board used for this study ($CaCO_3$: calcium carbonate).

following the CEPI specification. A computer aided design (CAD) drawing can be obtained from the authors.

At FPL, a house-built system for logging motor power was developed. A Motor Power Sensor Model TP-1 (Load Controls Incorporated; Sturbridge, MA, USA) was used to convert power to a 0–10 volts direct current (VDC) signal. This signal was connected to a NI 9215 analogue to digital converter (National Instruments; Austin, TX, USA). A Python program using the NIDAQMX module was used to collect voltages at a rate of 1 Hz. The system was calibrated by measuring the power dissipated into a resistive load of known resistance. The power signal was integrated over the duration of the pulping run to estimate the total energy. An identical run with only water was also integrated to get an estimated base energy. The reported specific energy was the difference of these two measurements divided by the oven-dried weight of the fiber in the pulper.

For a more complete description of the methods, see the proposed test method, which is included in the Appendix.

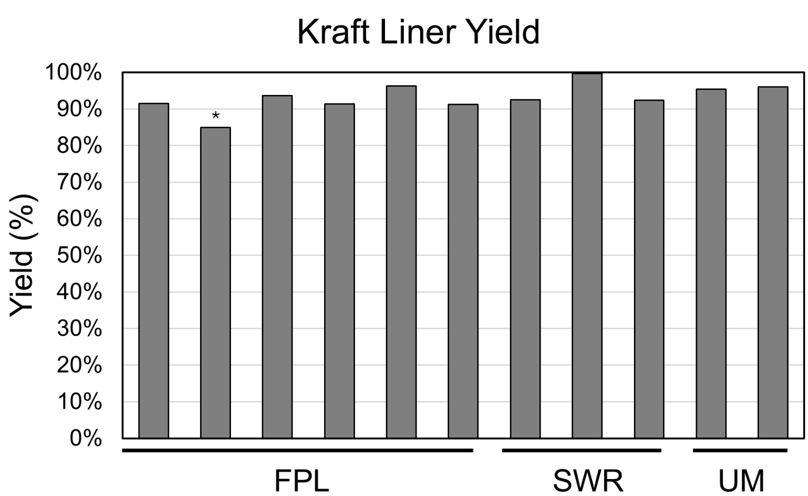
RESULTS

Kraft linerboard

The initial testing was conducted with kraft liner. As expected, with sufficient applied energy, this material can be completely fiberized, with only a minor amount of unpulped tags left on the screen. Inspection of **Fig. 2** shows that most of the yield values were between 90% and 99%.

An outlier was identified by the operator as suspect for physical reasons, and was 4.2 standard deviations from the mean value of the rest of the data, so it was excluded from further analysis. Pairwise t-testing was used to determine if there was a reason to reject the null hypothesis that the data from all three laboratories were the same. The results are shown in **Table II**. Inspection of the p-values in Table II shows that there is no statistical support for the conclusion that the results are different between the laboratories at a 95% confidence level. Given this statistical conclusion, it is reasonable to combine the data into a single grand average, which is also shown in the table.

To determine the end point of disintegrator pulping, we



2. Measured average yield for five determinations. Sequential t-testing showed that one sample could be considered an outlier (p = 0.0002), indicated by * (FPL: Forest Products Laboratory; SWR: Smurfit Westrock; UM: University of Maine).

SWR vs. FPL*	0.132
SWR vs. UM	0.054
FPL* vs. UM	0.636
Grand average yield	93%
95% CI on yield	1%

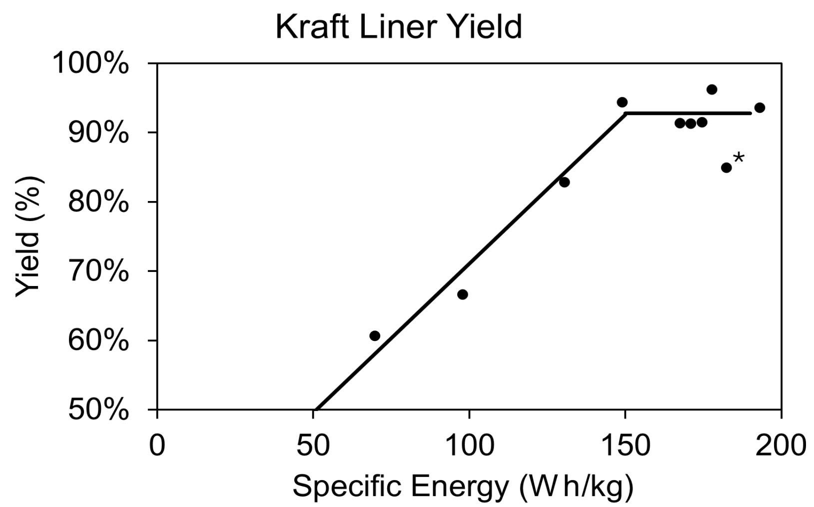
* Outlier removed.
SWR: Smurfit Westrock; FPL: Forest Products Laboratory;
UM: University of Maine; CI: confidence interval.

II. Pairwise* t*-test* p*-values.

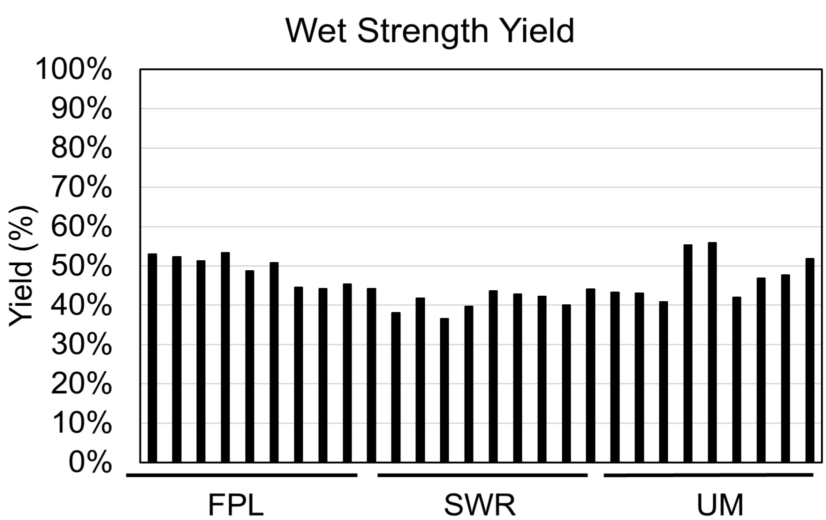
systematically varied the number of revolutions for disintegrating kraft liner (**Fig. 3**). It is a reasonable assumption that any commercial pulper will be operated to completely recover this paper grade. The four data points with energies below 150 W h/kg show a consistent increase in yield as the number of revolutions increased from 10,000, 15,000, 20,000, to 25,000. By 30,000 revolutions, there does not seem to be any further improvement in yield. An analysis of the 30,000 revolution replicates gives an average of 178 ± 9 W h/kg. These data indicate that 30,000 revolutions, which corresponds to 10 min of pulping, are a reasonable end point.

Wet-strength paperboard

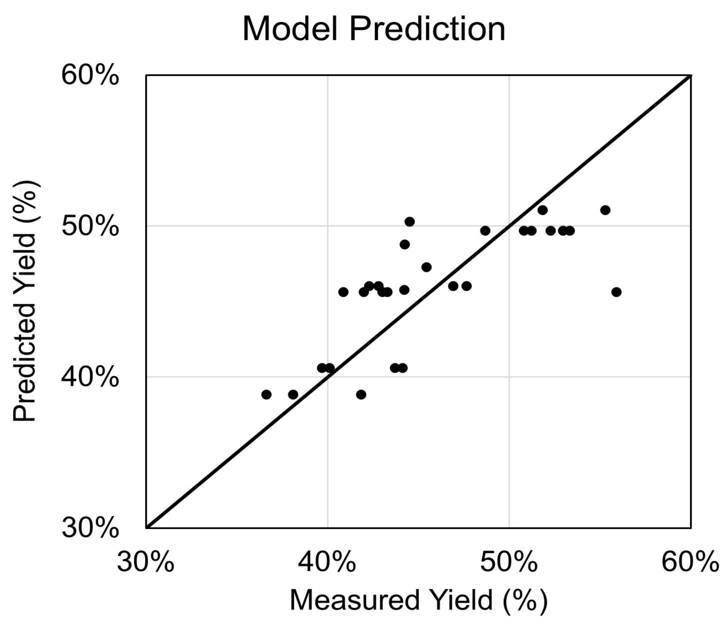
When a sheet treated with wet-strength agent was tested,



3. Kraft liner yield plotted vs. measured specific energy. The specific energy was reduced by reducing the number of pulper revolutions. The sequence was 10,000; 15,000; 20,000; 25,000; and 30,000 revolutions. The data point marked by * was identified as an outlier.



4. Yield results for experiments with the wet-strength board (FPL: Forest Products Laboratory; SWR: Smurfit Westrock; UM: University of Maine).



5. Plot of the linear model prediction vs. the measurements.

the yield was between 40% and 50%, as Fig. 4 shows. This reduction in yield was due to fibers not being fully liberated from a cross-linked network and fiber tags, incompletely pulped material, being collected on the slotted screen. The base sheet for this material is an unbleached softwood kraft similar to the kraft liner discussed previously, so one would expect a yield greater than 90%.

Initial statistical analysis shows that the SWR results are significantly different from FPL and UM, so further investigation was warranted. A total of 26 trials were conducted. At the beginning of the project, we did not confirm adherence to TAPPI T 275 "Screening of pulp (Somerville-type equipment)," specifically with respect to flowrate of the water. This test method also does not specify temperature. The temperature was measured and varied between 10°C and 46°C. We also observed that there was often fiber continuing to be collected at the specified 20 min, so some experiments were conducted with a 40-min total screening time. A linear statistical model was developed for yield, with flow rate, temperature, and time as independent variables. In equation form, the model was:

$$\text{yield} = \beta_0 + \beta_1{*}\text{flowrate} + \beta_2{*}\text{temperature} + \beta_3{*}\text{time}$$

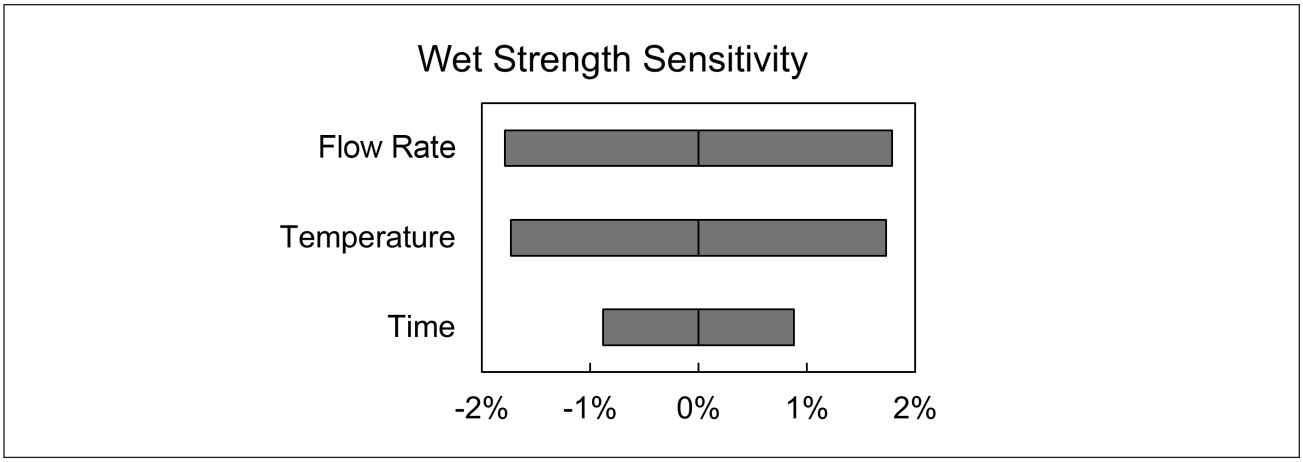
In this equation, the β's were estimated coefficients. The coefficient of determination (R^2) was 0.56, so the model was able to capture 56% of the variation in the data and provided a standard error of the measurement of 3.7%. A plot showing the model prediction vs. observations is shown in **Fig. 5**.

To characterize the dependence of the yield on the independent variables, a sensitivity analysis was conducted by sequentially increasing and decreasing the dependent variables by 25%. The results shown in **Fig. 6** indicate that flow rate and temperature have the largest impact. Time shows a lower impact because the majority of the fibers has been collected in the first 20 min and the fibers collected in the second 20 min were likely less than a few percent of the total mass. After conducting these experiments and this sensitivity analysis, we concluded that a flowrate of 8.6 L/min, a temperature of 46°C, and a time of 40 min should be specified. Putting these values into the statistical model results in an average yield of 46%. If warm water is not available for the screening, the statistical model predicts that the yield will drop to 41% when cold (10°C) water is used for the screening.

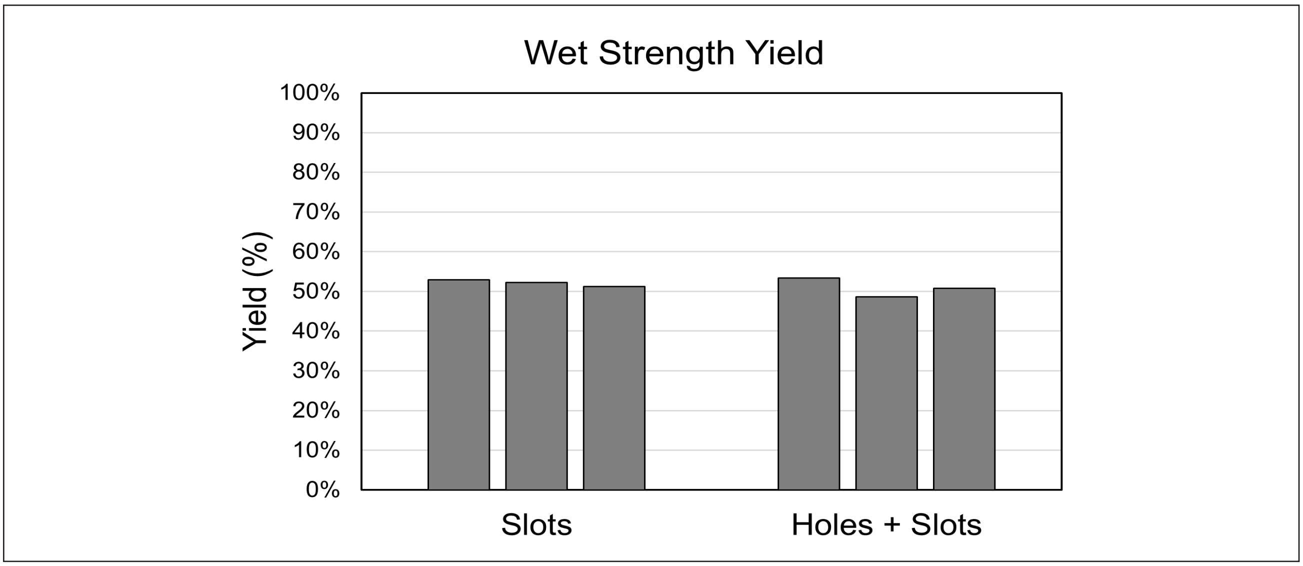
Five-millimeter hole screening

The European CEPI test method uses a sequential screen method. They first use a 5-mm hole screen and then a 0.15-mm slotted screen. This sequence replicates a rather common arrangement in paper recycling mills. It also has the potential to improve the slotted screening efficiency by avoiding blinding with unpulped tags. Initial tests with kraft liner, coated board, and envelope paper showed that there was no material collected on the hole screen, but tests with the wet-strength board showed significant quantities of material would not pass through the 5-mm holes, so replicate trials with wet-strength board were conducted.

The data, shown in **Fig. 7** and identified as "Slots" and "Holes + Slots," differ in that the first is the result from slotted screening for 40 min, and the second is the result from



6. Sensitivity of the yield on increasing and decreasing flow rate, temperature, and time by 25%.



7. Measured average yield for five determinations. Slots are used at the Forest Products Laboratory (normal method) and Holes + Slots are also used by FPL (hole screen followed by slotted screening).

40 min of hole screening followed by 40 min of slotted screening. Statistical analysis of these two data sets indicates that there is no support for concluding that these results are different with p=0.44. The dry weight of the rejects collected on both screens was determined. The percentages of material collected on the hole screen and the slotted screen were 32% and 78%, respectively.

Coated board

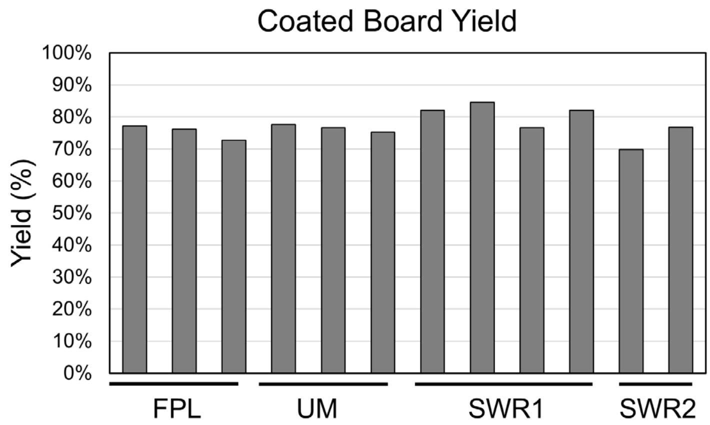
The yield for the coated board was lower than the yield for kraft liner, as shown in **Fig. 8**. Pairwise t-testing indicates that there is no support for concluding that the three labs are giving different results. Even the difference between SWR1 (without labels) and SWR2 (with labels) is not statistically significant. The average of all these data is 77% ± 3%. Note that the 95% confidence interval (CI) on this yield is three times the value of the kraft liner, likely indicating that there is more variation in processing this sample. The reduction in yield appears to be due to loss of fines or filler.

Envelope paper

As part of the USPS Recycling Compatible Adhesives Project, 25 tons of a clean white envelope paper was generated. This material was specifically produced to be low in stickies and dirt and was bleached softwood kraft pulp with approximate 25% calcium carbonate filler. Three trials with this envelope paper were conducted at FPL and resulted in a yield of 74.1%.

Losses

This method involves collecting screen accepts pulp on a 150-mesh screen and yield determination by making five handsheets on a standard mold as described in TAPPI T 205 "Forming handsheets for physical tests of pulp." Both



8. Measured average yield for five determinations (FPL = Forest Products Laboratory, UM = University of Maine, SWR1 = Smurfit Westrock, and SWR2 = Smurfit Westrock [with the addition of 2.9 g of PSA labels]).

Sample	Yield	95% CI	Rejects	95% CI	Loss	95% CI
Kraft liner	93%	1%	2%	2%	5%	2%
Wet strength	46%	3%	45%	3%	9%	2%
Coated board	77%	3%	0%	0%	22%	2%
Envelope	74%	2%	0%	0%	26%	2%
CI: confidence interval.						

III. Average yield, rejects, and estimated loss values.

operations resulted in loss of small particles, such as clay, calcium carbonate, and fiber fines. In addition, screen rejects were collected, dried, and weighed. By subtracting the mass of accepts and rejects from the initial mass, the yield loss was estimated. As expected, the various samples exhibited significantly different levels of loss. For three of the products, the screen reject mass was less than 2%, but for the wet-strength board, 45% of the mass was found in the screen rejects. **Table III** shows the average yield, rejects, and estimated loss values.

Specific energy

The FPL developed a method for measuring the specific energy applied to the pulp during the pulping cycle. The energy measured with the pulper containing an equivalent volume of water was subtracted from the energy measured during pulping. Pulping energy is a complex function of board strength, fiber length, and particle size of non-fiberized materials. [17] As shown in **Table IV**, the long, relatively stiff fibers in kraft liner resulted in a relatively high energy consumption. The short fibers and high filler content in coated board and envelope resulted in lower energy consumption. These specific energy levels are significantly higher than typical mill operations. For example, for kraft liner a typical value is 30–50 kWh/metric ton. The difference is likely due to the inefficiency of pulping action of a disintegrator, which was not designed for pulping. This pulping method is less aggressive than commercial-scale pulping and will tend to preserve fiber length, and it will be more sensitive to wet strength agents.

Sample	Specific Energy, W h/kg	95% CI, W h/kg
Kraft liner	178	16
Wet strength	154	24
Coated board	57	5
Envelope	74	5
CI: confidence interval.		

IV. Average specific energy (30,000 revolutions).

Dilution of sample for handsheets

During the processing of the samples, the accepts from the slotted screening are collected on a screen and then diluted with water to a known total mass. The initial method had the mass set at 5 kg. Observation showed that getting a uniform aliquot of a 1% slurry of long fibers was difficult. It was decided to modify the method to make this dilution target be 10 kg. We conducted a statistical test on the results from the two different levels of dilution. The 95% CI for dilution to 5 kg was 4.1% and 10 kg dilution was 2.7%. The probability that this difference is due to random error was determined to be 6.6%, so there is support for setting this dilution at 10 kg. The only practical impact is that a larger aliquot pulp is required to make a 1.2-g handsheet.

DISCUSSION

The results presented here show that reproducible measurements of yield can be made under well-defined pulping and screening conditions. A similar conclusion can be drawn about other published methods of yield determination. The method described here has the particular advantages of using standard laboratory equipment, a simplification of the method advocated by CEPI, and favoring products that give usable fiber. Developing a specification based on the proposed method is relatively straightforward. Many mills will be able to recover fibers at a similar level as observed with the proposed method. A common choice would be to set a threshold value between 70% to 80%.

Turning to contaminant analysis, there is no strong consensus. As discussed previously, quantifying stickies is complicated by a wide range of chemical and physical properties of polymeric materials used in packaging. Foil, ink, and dirt particles that exhibit a natural contrast to fiber can also present problems for paper recyclers. For all of these contaminants, there is the statistical problem of sampling relatively low concentrations of particles, which can lead to poor reproducibility. TAPPI T 277 "Macro stickies content in pulp: the 'pick-up' method" and TAPPI T 563 "Equivalent Black Area (EBA) and count of visible dirt in pulp, paper and paperboard by image analysis" have been developed to apply image analysis to quantify contaminants in pulp. Both of these methods have reasonable repeatability, approximately 20%, but poor reproducibility, 100%. For this discussion we are using the TAPPI T 1200 "Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility" definition of repeatability, which represents the inherent variability in the experimental system, and reproducibility, which includes variability between laboratories as well. Additionally, TAPPI T 563 includes the recommendation that the counting precision can be controlled by setting a target of observing 100 particles. Practically, this could be achieved by making and scanning more handsheets. The same arguments hold for using the blue-dye method as codified by TLMI.

Previous research on stickies determination suggests that developing a specification based on count is the best choice. In addition, because quantifying contaminants that pass through a fine screen is what matters to recycling mills, a method based on analysis of handsheets formed from screen accepts is desirable. The CEPI method "Visual Impurities decision tree" [18] specifies that finding less than 10 small particles per accept handsheet is acceptable. Because this is based on 1.8 g handsheets, 10 particles per handsheet corresponds to 5,500 particles/kg. While this seems like a relatively high level of contamination, it is likely reasonable for current uses of recovered fiber in the markets for board, packaging, and tissue.

Given that there are standard methods for developing contrast for stickies, a specification could be developed based on the following general steps:

(1) Make handsheets following TAPPI T 205 "Forming handsheets for physical tests of pulp" from the screen accepts, and speed dry them.
(2) Follow a current test method to develop contrast between stickies and fiber.
(3) Calibrate a flatbed scanner using a known optical target.
(4) Use image analysis to count the number of contaminant particles including both stickies and dirt.
(5) Continue counting the number of particles in the handsheets until the counting precision drops below 10%, i.e., finding more than 100 particles. This step may require making more handsheets. If all of the screen accepts are consumed, then stop and report the results.
(6) Estimate the average number of particles per handsheet and the 95% CI for the measurement. For cases when some of the handsheets contain no particles, a statistical method based on Poisson distribution may be required.
(7) Use the weight of the handsheets and the average count to express the results as counts/kg of dry pulp.
(8) Determine if the CI includes the value of 5,000 particles/kg.

CONCLUSIONS

A reproducible test method for typical fiber yield during recycling has been developed. By controlling temperature, time, and flow rate during pulping and slotted screening, one can get results with a reproducibility of less than 5%. Setting a specification lower limit on yield of 70% is likely a reasonable value, so if 70% is within the 95% CI of the measurement, the product should be considered recyclable.

Assessment of contaminants is much less certain. A process has been proposed that includes analysis of handsheets made from screen accepts. Either a powder- or dye-based method is used to highlight stickies. The contaminants are counted using image analysis. The average number of particles per handsheet and associated 95% CI are estimated and expressed as number of particles/kg of dry pulp. If the specification limit of 5,000 particles/kg is within the CI, the product is considered acceptable.

The proposed method and associated specification are chosen to be similar to the method advocated by CEPI. If a brand owner has a presence in both Europe and North America, results should be similar, but confirmation should be the subject of future research. **TJ**

LITERATURE CITED

1. "REMA's ISRI specifications," Institute of Scrap Recycling Industries, Washington, DC. Available [Online] https://www.isrispecs.org/ <02Feb2026>.
2. Kasimi, H., "Unravelling recovered paper price trends," *Recycling Product News*, Baum Publication Ltd., Vancouver, BC, Canada, Available [Online] https://www.recyclingproductnews.com/article/43457/unravelling-recovered-paper-price-trends <02Feb2026>.
3. Cooper, T., *Hist. Res.* 80(214): 710(2008) https://doi.org/10.1111/j.1468-2281.2007.00420.x.
4. Ince, P.J., "Recycling of wood and paper products in the United States," General Technical Report FPL–GTR–89, U.S. Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, WI, USA,1996. https://doi.org/10.2737/FPL-GTR-89.
5. "Advancing sustainable materials management: 2018 tables and figures," U.S. Environmental Protection Agency, Washington, DC, 2020.
6. Hutten, I.M. and Banerjee, S, "Repulpablity of waxed corrugated board," Report of Project 3900, Institute of Paper Science and Technology Atlanta, GA, 1994.
7. "Voluntary standard for repulping and recycling corrugated fiberboard treated to improve its performance in the presence of water and water vapor," Fibre Box Association, Elk Grove Village, IL, USA, 2013.

ABOUT THIS PAPER

Cite this article as:
Houtman, C., Haynes, H., Ghosh, A., et al., *TAPPI J.* 25(2): 82(2026). https://doi.org/10.32964/TJ25.2.82

DOI: https://doi.org/10.32964/TJ25.2.82

ISSN: 0734-1415

Publisher: TAPPI Press

Copyright: ©TAPPI Press 2026

About this journal

ABOUT THE AUTHORS

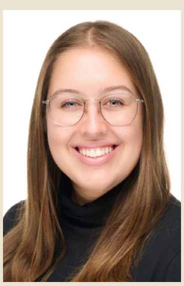
In North America, companies that want to get a package certified as recyclable have to go through a complex testing process. This current test method is based on a method developed for OCC and has several aspects that do not apply to modern packaging. We have a long history in developing recycling compatible adhesives, and this study is a natural extension. The major goal is to provide a simpler method that companies can use as they develop innovative fiber-based packages.

The most difficult aspect of this work was developing consensus around a method that represents the wide range of recycling process designs that exist in the industry. In our findings, it was surprising to see that water temperature affects screening efficiency. The most interesting discovery was that the yield measurement for most of the samples was very reproducible, i.e., <10%.

Because the method is simple and uses normal lab equipment, most mills should be able to screen new products for their recycling performance. Our next step is to get the proposed method, which is included here as an Appendix, approved as a TAPPI Standard Test Method. Reader comments and suggestions on the procedure outlined in the Appendix are earnestly requested and should be sent to the TAPPI Standards Department at Standards@tappi.org.

Houtman is research chemical engineer at the U.S. Department of Agriculture's Forest Product Laboratory in Madison, WI, USA. Haynes is R&D scientist and Ghosh is senior manager of R&D at the Smurfit WestRock Pilot Plant in Henrico, VA, USA. Park is senior scientist and Walker is director at the University of Maine Process Development Center in Orono, ME, USA. Email Houtman at carl.houtman@usda.gov.

Houtman



Hayes

Ghosh

Park

Walker

8. "Voluntary standard for repulping and recycling coated or treated corrugated fiberboard and its equivalents in the old corrugated containers (OCC) recycling stream," Fibre Box Association, Itasca, IL, USA, 9 September 2025. Available [Online] https://www.fibrebox.org/assets/2025/09/Volunatary_Standard_9.17.25.pdf <02Aug2026>.

9. Seiter, D.F., Pikulin, M.A., Meese, R.G., et al., "Environmentally benign USPS stamps: Baseline pilot recycling results," *TAPPI Pulping Conf.*, TAPPI Press, Atlanta, GA, 1998, p. 193.

10. Houtman, C.J., Seiter, D., Sutherland, N.R., et al., "Can laboratory and pilot recycling trials predict adhesive removal in commercial recycling systems? Results from the USPS Environmentally Benign Stamp Project," *TAPPI Fall Tech. Conf. Trade Fair*, TAPPI Press, Atlanta, 2002.

11. Houtman, C., Scallon, K., and Oldack, R., *Prog. Pap. Recycl.* (17)2: 26(2008).

12. "Sustainability," Tag and Label Manufacturers Institute, Nashville. Available [Online] https://tlmi.com/insight/sustainability/ <02Feb2026>.

13. "Harmonised European laboratory test method to generate parameters enabling the assessment of the recyclability of paper and board products in recycling mills with conventional process (Part I)," Confederation of European Paper Industries, Brussels. Available [Online] https://www.cepi.org/wp-content/uploads/2025/02/Cepi-Test-Method-27.02_FINAL.pdf <02Feb2026>.

14. Xu, K., Mo, W., Zhang, F., et al., *Ind. Eng. Chem. Res.* 63(17): 7950(2024). https://doi.org/10.1021/acs.iecr.4c00483.

15. Doshi, M., Moore, W.J., Venditti, R.A., et al., *Prog. Pap. Recycl.* 12(3): 34(2003).

16. Sithole, B. and Filion, D., "Assessment of methods for the measurement of macrostickies in recycled pulps," *Recycl. Res. Forum, 8th*, TAPPI Press, Atlanta, 2007.

17. Saville, F., Martinez, M., and Olson, J., *Can. J. Chem. Eng.* 94(3): 446 (2016) https://doi.org/10.1002/cjce.22421.

18. "Annex documents part I – recycling mill with conventional process," Confederation of European Paper Industries, Brussels, January 2025. Available [Online] https://www.cepi.org/wp-content/uploads/2025/03/4evergreen-Annexes-PART-I-25.02.pdf <02Feb2026>.

APPENDIX

RECYCLING COMPATIBLE PACKAGING TEST METHOD

1. Scope

1.1. The results of this test method provide an indication of whether the materials used in the construction of fiber product are adequately repulped and are unlikely to contribute to runnability or product quality problems for a paper recycling facility.

2. Significance and Use

2.1. The facility age and level of capital investment ranges widely in the paper recycling industry. Some facilities are limited in types and amounts of contaminants they can remove, and others can produce high quality fiber from mixed paper bales from sorting facilities. In addition, there are a wide range of products produced from recycled fiber. Given this diversity in processes and end uses, this test method was designed to represent the minimum unit operations common to all processes.

2.2. While packaging materials are only a small part of the recovered paper stream, the purpose of this test method is to encourage choices of materials and designs that make fiber recycling more efficient. As such, a particular product is tested by itself.

2.3. While this test method provides results on the minimum requirements, subsequent unit operations or testing could be conducted to make other claims about a particular material. For example, one could apply flotation or high-density cleaning to test the removability of a particular contaminant, e.g., ink, metalized layers, etc. Pulp brightness or strength properties could be determined, if those properties are important for the end use.

3. Requirements

3.1. The products covered by this method consist of fiber products, including trays and inserts. Board or sheet materials that have not been converted to final products may also be tested using this method.

4. Equipment & Materials

4.1. Equipment
4.1.1. Balance capable of weighing to 20 kg with 1 g resolution
4.1.2. Balance capable of weighing to 300 g with 0.01g resolution
4.1.3. Thermometer
4.1.4. Paper shredder capable of cutting into ¼" strips
4.1.5. British Disintegrator as specified in TAPPI T 205
4.1.6. Slotted screen separator/analyzer with 0.152 mm (0.006") slots. See TAPPI T 275 or TAPPI T 278 for design and operation of these devices
4.1.7. 150 mesh screen box to collect the accepts from the slotted screening
4.1.8. 105°C drying oven
4.1.9. Plastic buckets
4.1.10. Stopwatch or timer
4.1.11. Stand mixer (Lightning Model NAR-25 or equivalent)
4.1.12. Sheet mold as specified in TAPPI T 205

4.2. Materials
4.2.1. Water supply, controlled to 46°C
4.2.2. Aluminum foil or containers for drying screening rejects
4.2.3. Blotter paper as specified in TAPPI T 205

5. Paper Feed Stock Preparation

5.1. Determine the moisture content of a sample of the product by weighing material before and after drying to a constant weight in a 105°C oven. Note, the sample used to determine the moisture content should not be included in the materials used in the test.

5.2. After determining the moisture content of the material, use this value to determine the amount of material required to reach a weight of 50 g OD. For example, if the material is 5% moisture the total weight is 50/0.95 = 52.6 g.

5.3. If the package weighs less than 50 g, use as many as are required to reach the desired weight.

5.4. If a single package weighs more than 50 g, cut pieces to reach the required weight.

5.5. If partial packages are required, attempt to include a prorated amount of material that contains adhesive, e.g., flap closures, laminations, etc.

5.6. If plastic or metal pieces are part of the selected materials and if these pieces are large enough to cause problems during shredding and pulping, they can be removed and directly included in the screen rejects.

5.7. Take a picture of the material, as documentation.

6. Pulping

6.1. Shred all the material into ¼” wide strips in the paper shredder. The shreds should be approximately 3 inches long (7.62 cm).

6.2. Measure sufficient tempered soft water, 46 ± 2°C, to bring the total weight to 2000 g, i.e., subtract the mass of fiber from 2000 g. For example, 2000 – 52.6 = 1947.4 g of added water. This results in a disintegrator consistency of 2.5%.

6.3. Add the strips to the disintegrator and add the water. Mix by hand to uniformly saturate the fiber. Let the fiber soak for 10 min.

6.4. Measure and record the temperature on the data sheet. Start the disintegrator and run for 15,000 revolutions (5 min.). At the end of the first disintegration stage, open and push any unpulped material off wall with a rubber spatula. Repeat the disintegration cycle for another 15,000 revolutions.

6.5. After the second disintegration cycle, turn off the disintegrator. Measure and record the temperature on the data sheet.

6.6. Remove disintegrator contents.

6.7. Clean the disintegrator.

7. Slotted Screening

7.1. Prior to starting, ensure that a 6-cut screen is installed in the screen.

7.2. Run a cleaning cycle to flush the screen and manually adjust the screening water temperature to 46 ± 2°C and the total flow rate to 8.6 L/min.

7.3. Screen the entire amount of pulped material from step 6.6 in four aliquots.

7.4. Fill the screen with water and add a 500 ml aliquot of the pulp slurry. Start and run the screen for one 40-minute cycle. During the cycle add the other three 500 ml aliquots of pulp every three minutes.

7.5. The accepts are collected and drained in a 150-mesh screen box. The rejects are anything left in the screening chamber and any plastic or metal removed before shredding.

7.6. Collect the rejects in a pre-weighed aluminum container for oven drying at 105°C.

7.7. Clean all sections of the screen unit, including the screen and under the screen.

7.8. After screening process is complete, collect the accepts from the screen box in a previously tared bucket.

7.9. Dilute the accepts in the bucket by adding sufficient water to bring the total weight to 10 kg. Mix the slurry with a stand mixer for at least 5 minutes to ensure homogeneity prior to handsheet making.

7.10. During the formation of the test sheets for handsheet making, the target weight is 1.2 g. The aliquot of the material in the 5-gallon bucket required to achieve this weight is used to determine the consistency of the pulp fiber. For example, if 240 g of slurry gives a 1.2 g dry sheet, the consistency is 0.5%. Make at least five test sheets. The test sheet should be dried with a speed drier between blotter papers. It should be weighed while it is still hot. Report the average and 95% confidence interval for the consistency. Using this consistency and the initial sample dry weight, calculate the total yield and corresponding confidence interval.

7.11. After rejects have dried in the oven overnight, weigh them and record the dry weight.

8. Calculations

8.1. Mass of dry sample may be calculated as:

$$Mass\ of\ dry\ sample\ (g) = \frac{Sample\ mass\ (g)*(\ 100 - \text{ Sample moisture } (\%))}{100}$$

8.2. Pulp slurry consistency may be calculated as follows:

$$Csc\ (\%) = 100* \frac{Mass\ of\ dried\ sheet\ (g)}{Mass\ of\ pulp\ aliquot\ (g)}$$

8.3. Screen yield may be calculated as follows:

$$Yield\ (\%) = \frac{Csc\ (\%)*100}{Mass\ of\ dry\ sample\ (g)}$$

8.4. Average yield may be calculated as follows:

$$Average\ Yield\ (\%) = \frac{\sum Yield_i}{N}$$

(i = 1…N) with N = Number of measurements

8.5. Standard deviation of the yield may be calculated as follows:

$$Standard\ deviation\ (\%) = \sqrt{\frac{\sum \left(Average\ Yield - \ Yield_i\right)^2}{N-1}}$$

(i = 1…N)

8.6. 95% confidence interval of the yield may be calculated as follows:

$$95\%\ CI\ (\%) = \ t_{0.025,N-1} * \frac{Standard\ deviation\ (\%)}{\sqrt{N}}$$

$$t_{0.05,N-1} = Student's\ T\ statistic\ for\ 2.5\%\ and\ (N-1)\ degrees\ of\ freedom$$

9. Reporting

9.1. The report will consist of the following:

9.1.1. Copies of Data Log Sheets

9.1.2. Pictures of the starting materials and rejects.

9.1.3. Dry weight of rejects from the slotted screen.

9.1.4. The screened yield and 95% confidence interval.

10. Precision

Four different paper and board samples to shipped to nine different laboratories. The characteristics of the paper and board used for this study are shown below.

Name	Kraft Liner	Wet Strength	Coated Board	Envelope
Basis Weight (g/m^2)	280	320	320	90
Coating	none	clay/carbonate	pigmented barrier	none
Fiber	softwood kraft	softwood kraft	soft/hardwood	softwood kraft
Filler	no	no	yes	20% CaCO3
Bleached	no	no	yes	yes
Wet Strength	no	yes	no	no

The methods described in T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility" were used to calculate repeatability and reproducibility. Inspection of the table below shows that for samples that have little or no wet strength the maximum repeatability was 8% and the maximum reproducibility was 10%. When wet strength is present, as demonstrated by significant unpulped tags in the screen rejects, one can expect approximately twice the variability.

	Kraft Liner	Wet Strength	Coated Board	Envelope
Grand Mean	93%	45%	78%	75%
Repeatability	7%	15%	8%	6%
Reproducibility	8%	28%	9%	10%

11. Keywords (Mandatory)

Recycling, Pulping, Yield Determination

12. Additional Information

12.1. Effective date of issue: To Be Assigned

13. Referenced Documents

13.1. T 205 sp-24 "Forming Handsheets for Physical Tests of Pulp"
13.2. T 275 sp-23 "Screening of pulp (Somerville-type equipment)"
13.3. T 278 sp-21 "Pulp screening (Valley-type screening device)"
13.4. T 1200 sp-19 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility"

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.