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Project Title: Evaluation of Various Adhesive Contaminant Analysis Methods for the Use in Old Corrugated Container Recycling Plants

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EXECUTIVE SUMMARY

This project involves critically evaluating different test methods for their ability to detect stickies contaminants in old corrugated container (OCC) recycling plants. Tests were broadly classified as either macro or micro stickies test methods based on standard industrial terms.

The macro stickies test methods evaluated were a bleaching and dyeing of handsheets, Port Townsend Method 1 (involves dyeing handsheets with black dye followed by image analysis), Port Townsend Method 2 (involves dyeing lab-screened rejects on filter pads with black dye followed by image analysis), Tappi Method 277 (involves pressing lab-screened rejects against a white coated material that transfers to stickies followed by image analysis), and a deposition method in which stickies are deposited onto a sample of a paper machine wire. The micro stickies test methods evaluated were deposition on a paper machine wire, deposition on a polyethylene bottle, deposition on a polyethylene film, solvent extraction and a Tappi Method for Micro-stickies in Process Water (involves lab-screening of refrigerated and non-refrigerated samples).

The most valuable category of samples subjected to the above stickies tests were series of samples collected after the various major operations of an OCC recycling mill. Sets of samples from two OCC recycling mills were obtained. These samples were useful because it was expected that the stickies concentration should decrease through the recycling process. These samples provided an opportunity to determine on real OCC samples if the tests could "fingerprint" an OCC recycling plant performance through its various stages of pulp processing.

Port Townsend Method 2 displayed a consistently better ability to track a decreasing trend in macro stickies content across the operations of an OCC recycle mill than the other macro stickies tests. Port Townsend Method 2 test results also showed a better reproducibility of repeated tests on the same samples (less scatter of data). The labor demanded and the skills and equipment needed to run Port Townsend Method 2 were reasonable. This is the preferred method of macro-stickies testing to characterize the performance of a recycling mill or an individual pulp processing operation.

Of the micro stickies test methods, the Polyethylene Film Deposition method is the preferred method of micro-stickies testing to characterize the performance of a recycling mill or an individual pulp processing operation. This method showed a clear decrease in micro-stickies across the OCC mill, was reproducible, and was practical to implement.

Fluctuations in stickies content in an OCC recycle mill are are large, with a coefficient of variation of about 30%. Thus, errors in stickies detection due to sampling are expected to be significant. All practical means of alleviating sampling issues should be considered.

Recommended stickies test methods are made for the evaluation of OCC feedstock to a recycle mill, for anti-deposition strategies for the paper/board machine, and for process water evaluation.

INTRODUCTION

Adhesive contaminants and any other contaminants with a propensity to deposit (stickies) are a serious production and product quality problem for old corrugated container (OCC) recycling mills. The ultimate goal for OCC mills is to remove these stickies through equipment or operation modifications. To embark on these improvements, the measurement of stickies must be accurate and precise. Accuracy, the ability to detect a quantity's true value, in stickies detection is important for recyclers to understanding how key unit operations and processing conditions impact stickies removal. Precision (the range of scatter in a measurement) in stickies detection is also important. The variability of a measurement coupled with the natural variability of the process and the finite amount of sampling and testing that can be performed all combine to determine a confidence range in a measurement. It is the difference between confidence ranges that is used to determine if a unit operation or change in process condition is significantly changing the concentration of stickies in the system.

Many stickies detection methods have been proposed and evaluated. However, there are no extensive quantitative comparisons of these methods on the same furnish that would allow a proper comparison of the utility of each method. Further, many of these methods are for bleached pulp or wood containing pulps that are relatively bright, not dark pulps like OCC.

*It is the **objective** of this research to evaluate several different stickies test methods for accuracy and precision in applications of OCC recycling and to evaluate their ability to be practically implemented in an industrial setting. It is the **goal** of this research to then be able to recommend in a ranked list the test methods that would serve the needs of OCC recycling mills with regards to stickies.*

The tests in this research have been grouped as "macro stickies" tests or "micro stickies" tests. Stickies contaminants, retained by a 0.006 inch slotted lab screen are considered to be macro stickies and those that pass through and are still particulate are considered to be micro stickies. Dissolved species were not considered here.

In the year 2001 the focus of the research was on evaluating macro stickies test methods with blends of pulps and also with a series of OCC samples that were obtained after the unit operations in an OCC recycling mill. The same types of experiments were performed on micro stickies test methods in the year 2002. This final report describes all of the tests evaluated and the materials used, summarizes all of the findings, and makes recommendations for which test methods to use for certain applications.

LITERATURE REVIEW

The most recent review on stickies quantification methods was presented by Doshi and Dyer at the 2000 Tappi Recycling Symposium, "Review of Quantification Methods for PSA and Other Stickies". Over 40 references were presented discussing different methods of stickies quantification. Other comprehensive reviews are available [Paper Recycling Challenge, Vol. I Stickies, Doshi and Dyer, Doshi and Associates, Appleton WI, 1997, and Vol. IV Process Control and Mensuration, 1999]. Overviews of the methods utilized at NCSU have also been published [Venditti, Chang, et. al., Vol. I Stickies, Doshi and Dyer, Doshi and Associates, pp. 45-48, Appleton WI, 1997 and Venditti, Chang and Jameel, PaperAge, pp. 18-20, Nov. 1999].

The large number of test methods reflect the (a) differing test needs for different types of pulp (e.g., OCC vs printing and writing grades) or different types of stickies contaminants (e.g., micro and macro stickies), (b) a general desire for an improved, convenient test method and (c) the great importance of stickies detection.

There is in general a lack of information describing the relative performance of the various test methods. In one study Venditti and coworkers have correlated the results from deposition tests performed at NCSU with dyeing/image analysis results performed at the Forest Products Laboratory on the same pulps [Tappi Recycling Symposium, pp. 475-482, 1998]. A linear relationship was found between deposition and image analysis with stickies concentrations from 1 to 10^4 PPM. The correlation coefficients, R^2, were approximately 0.8.

The use of a deposition test with a papermachine wire and with a microfoam packing material was compared by Carre, Fabry and Brun [Paper Recycling Challenge, Vol. I Stickies, pp. 185-189, Doshi and Dyer, Doshi and Associates, Appleton WI, 1997]. Both methods detected a maximum of depositable stickies at the point at which charges in the system were neutralized. It was concluded that the papermachine wire was more sensitive than the microfoam method but this conclusion was not justified.

The accuracy of stickies determinations has been reported in some cases. Gravimetric methods used in a deposition test with known spiked quantities of stickies revealed that the amount of deposits detected (and thus the accuracy of the test) varied for various adhesives in the range of 50-100 % [Paper Recycling Challenge, Vol. I Stickies, pp. 104-110, Doshi and Dyer, Doshi and Associates, Appleton WI, 1997]. Another deposition study with a single PSA type material showed that the recovery of adhesive as deposits could vary from 0 to 100% simply by the presence of interfering substances such as starch and talc [Venditti, et.al., Tappi 99 Proceedings, pp. 681-692, 1999].

Despite the magnitude of research in the area there is not a solid set of data from which to compare test methods. With respect to OCC, the information on stickies detection methods is very scarce. This indicates that a well-controlled set of experiments in which different test methods are used on the same OCC pulp samples would provide the needed information upon which further stickies research could be based.

MATERIALS AND METHODS

Stickies Test Methods: Macro Stickies Tests

Bleaching and Dyeing of Handsheets.
Bleaching was performed in a 4000-ml glass beaker equipped with motorized stirrer in a hot water bath kept at 70°C (in a laboratory hood). 1920-mls of water were placed into the beaker along with 80 OD g of pulp sample. 12 g of sodium acetate and 24-mls of glacial acetic acid were added to the pulp slurry. These two chemicals act as a buffer for the bleaching experiment. After the pulp mixture reached 70°C, 12 g of sodium chlorite was added. This amount of sodium chlorite was also added at 30 minutes and 60 minutes. 15 g of sodium thiosulfate was added to the mixture 30 minutes after the last dose of sodium chlorite to stop the bleaching reaction. After the reaction has stopped, approximately one minute after adding the sodium thiosulfate, the beaker was removed from the hot water bath. The pulp was washed with deionized water on a Buchner funnel with vacuum using a Whatman 541 filter paper. During washing the pulp changed from a yellow to a slight gray color. When the color change had stopped, the pulp was removed and placed in a plastic bag for storage. Standard Tappi methods were used to produce 1.2 gram handsheets.

In a laboratory hood, #1003 Morplas Blue dye solution was made in a 1000-ml volumetric flask by adding 95% Heptane to 0.67 g of Morplas Blue #1003 (Sunbelt Corp., Rock Hill SC) powder. The solution was stirred with a magnetic stirrer overnight. The dye solution was filtered in a Buchner funnel using Whatman 541 filter paper to remove undissolved material. 250-mls of the filtered dye solution was placed into a crystallizing dish. Each handsheet was swirled in the solution for 10 seconds. The handsheets were placed on a line to dry overnight in a hood. Approximately 25 handsheets could be dyed with a 250 ml sample of the dyeing solution.

The Apogee Specscan Image analysis program was used to determine specks in both the bleached and the bleached/dyed handsheets. Subtraction of the two sets of results eliminated the effect of non-sticky dirt on the measurement.
The settings on the Specscan program were as follows:

- Normal Sample
- 6” round sheets
- 256 grayscale
- 600 dpi resolution
- Threshold setting, 80 % of average grayscale value
- Minimum particle size detected 0.02 mm^2
- Set of 5 HS scanned, both top and bottom

Paper Machine Wire Deposition

80 OD grams of the pulp sample was placed into the deposition chamber and diluted to 1 % consistency, approximately eight liters. The sample was maintained at 55°C in a stainless steel beaker in a water bath in a modified PIRA deposition chamber. Four 7.5-cm X 14.5-cm rectangles of paper machine wire obtained from Weavexx Corporation were oven dried (105°C for 30 min) and weighed and placed in the holding paddle. The paddles were counter rotated at 0.75 Hz for 30 minutes in the pulp stock. The paper machine wires were then removed from the paddle and gently rinsed with deionized water and then dried and weighed. The wires were dyed in the same manner as described above for the bleaching and dyeing of handsheets. Image analysis settings with the Apogee system was as follows:

- Normal Sample
- 7 X 14 cm rectangles
- 256 grayscale
- 600 dpi resolution,
- 103 Absolute Threshold
- Minimum particle size detected 0.02 mm^2
- Set of 4 to be scanned, front and back

Tappi Test Method T277

A sample of 20 g OD was disintegrated with a Tappi Disintegrator for 5 minutes and then screened using the Pulmac Masterscreen with 0.006 inch slots. The rejects were captured on a black filter paper, approximately 2.7 g and 20.5 cm in diameter (from Fisher Scientific). The filter pad was placed face up with a special coated paper obtained from Voith Sulzer and pressed using a Carver press at 90°C and 11.6 psi for 10 minutes. The filter pad was then rinsed with a shower of deionized water at 15 psi for 25 seconds. The filter pad was pressed again with the same parameters as before but now with a silicone coated release liner facing the sample. The filter paper was then weighed to obtain a reject weight. The procedure calls for using a black felt pen to hide all of the brown fibers existing on the filter paper. The white coating specks on the black filter paper are then detected using image analysis. The image analysis settings were the same as decribed below for Port Townsend Paper Corp. Method 1 except that the threshold value was set to 66.

Port Townsend Paper Corp. Method 1: Dyeing of Handsheets

20 g OD of sample was disintegrated in a TAPPI disintegrator for 5 min to break fiber bundles. The sample was diluted to 0.5% consistency and 250 ml of the material was used to make a consistency determination. Five 1.2 gram standard Tappi handsheets were then made. Drying of the handsheets was performed by placing the handsheet between a Teflon and metal plate and heating with an Emerson Speed Dryer Model 135 at 300°F for 5 minutes. The sheets were conditioned in a Tappi conditioning room overnight. Handsheets were placed on the surface of a pool of Parker Quick Ink until the ink absorbed through the entire handsheet, typically about 5-10 seconds. The ink was held in a common rectangular aluminum pan (about 9 by 13 inch) with an ink depth of approximately one quarter of an inch. The handsheets were then placed on a single sheet of blotter paper and allowed to dry overnight in air at room temperature. Areas covered

with stickies remained light whereas the fibers were dyed black. Image analysis using the Apogee Specscan program was as follows:

- Reverse Threshold
- 6" round sheets
- 256 grayscale
- 400 dpi resolution
- Threshold setting, 23 manual
- Minimum particle size detected 0.02 mm^2
- Set of 5 handsheets, top and bottom

Port Townsend Paper Corp. Method 2: Screening Method

80g OD of a pulp sample was diluted to a 1.2% consistency and disintegrated for 5 minutes in a TAPPI disintegrator to remove fiber bundles. A 1000-ml sample of the slurry was used to make a handsheet (speed dried) to determine consistency. Three samples were then screened using a Pulmac Masterscreen with 0.006 inch slots and the rejects captured on 20.5-cm white filter paper. The filter paper between a metal plate on the bottom and a Teflon plate on the top was dried using the Emerson Speed Dryer at 300°F for 5 minutes. The filter paper was allowed to condition in the Tappi conditioning room overnight to obtain an accurate reject weight. The filter papers were then dyed with Parker Quick Ink, dried (filter paper down against the single blotter paper). A 20 cm diameter circle was analyzed using image analysis for each of the three filter papers in a similar manner as the Port Townsend Paper Corp. Method 1. Again, the stickies were light brown whereas the fibers were black.

Stickies Test Methods: Micro Stickies Tests

The below methods were used as micro stickies test methods. In some cases, macro-stickies could affect the results and this is indicated in the procedure with an *.

Paper Machine Wire Deposition*
Same procedure as for macro stickies testing, above.

Bleaching and Dyeing of Handsheets.*
Same procedure as for macro stickies testing, above.

Polyethylene Bottle Method*
The test device consisted of a four-place gang stirrer on top of a base consisting of four individual hot plates. The stirring devices consisted of 3-inch marine-style propellers with a drilled rubber stopper for mounting the polyethylene bottle (250 ml, of known OD weight). The pulp slurries of 500 ml each at 1 % consistency were tested in one-liter glass beakers. The slurries were heated for 10 minutes at 40 ^{o}C (hot plate temperature) before the bottles are introduced. The bottles were attached to the propellers and a few drops of Toluene was added (Aldrich, HPLC grade). The Toluene was added to accelerate the test by forming a ring of depositable material at the air-slurry interface. The slurries were then heated to 60 ^{o}C (hot plate temperature) and held at the temperature for 10 minutes. Once the test was complete the bottles were removed and rinsed thoroughly with cold tap water. This is used to solidify and improve the adherence of the stickies. Then each bottle was dried at 50 ^{o}C for one hour and allowed to cool down to room temperature. The weight of the stickies was determined from the difference between the oven dried bottle weight and the total weight of the bottle and stickies after the test.

Polyethylene Film Method*
A pulp slurry (500 ml at 1 % consistency) was agitated in a one-liter glass beaker (in a 65 ^{o}C water bath) with a marine type impeller at 600 RPM. Five pieces of LDPE film (Associated Bag Company) were cut into 2 X 5 cm pieces and the total weight of the five pieces was determined. The plastic film pieces were added to the slurry and were mixed with the slurry for one hour. The pieces were then taken out of the slurry, dipped into cold tap water to remove fibers and solidify the stickies. The remaining pulp slurry is screened with a slotted vibratory screen (Valley flat screen with a 0.15 mm slot widths). Any rejects from screening are placed onto a 5 X 5 cm piece of film. All six of the pieces of film are air dried overnight and then weighed. Subtraction of the weight of the film pieces alone provided a measure of the micro stickies.

Solvent Extraction*
Pulp at 2% consistency (1000 ml) was placed into a 2000 ml separatory funnel (Fisher Brand, Cat. No 10-437-10F). The solvent used in the extraction was chloroform (Aldrich, HPLC grade). An amount of 150 ml of the chloroform was added to the slurry and shaken 50 times. The extraction liquid was allowed to separate and settle for 4 hours. After this time the organic liquid at the bottom was filtered through a filter paper, pore

size approximately 20 microns (P8 Fisher Brand, Cat. No 09-795D) and allowed to pass into a 500 ml tin pan (known weight after oven drying). Chloroform (50 ml) was again added to the separatory funnel, and shaken 50 times. The sample was allowed to separate for a 2-hour period and the organic liquid taken off the bottom and filtered and placed in the tin pan. The procedure was repeated with another 50 ml of chloroform but with a settling time of only one hour. The chloroform extract was allowed to evaporate in a chemical exhaust hood overnight. Any remaining chloroform was driven off by a final drying step at 50 ^{o}C for 1 hour. The tin pan and remaining extract was weighed. The difference in weight between the tin pan alone and with the extract was considered the micro stickies material.

Microscope Image Analysis Method

Handsheets prepared with the bleaching and dyeing process were used in this procedure. Image analysis was performed using an Olympus BH-2 microscope and ImagePro Plus Version 4.0 image analysis program. For each test, four areas on the top-side of each of 5 handsheets were analyzed. The settings on the microscope were as follows: 5X Lens on the microscope, transmitted light setting of 70%, surface light setting of 40%, and area of regions scanned of 1200 micrometers on edge. The threshold limit was constant at 55. The number of particles and average particle size were determined. A PPM of contaminant was determined.

Tappi Test Procedure for Micro-stickies in Process Water

OCC samples were refrigerated (arbitrary consistency) for a two-week period before testing (40°F). Refrigeration supposedly allowed the micro stickies to precipitate into large particles and screen more effectively using a 0.006-inch slotted screen. After refrigeration was complete the samples were screened and tested for stickies using a prescribed macro stickies testing method. In our case the PT Method 2 was used to determine the stickies content. The stickies content of these refrigerated samples included both micro and macro stickies. Stickies measurements of macro stickies alone were determined by performing the same procedure on non-refrigerated samples. Subtracting the results provided a measure of the micro stickies content.

Materials for Testing

Due to the timeline of the research over two years and the changing approaches and tests used, several sets of pulp samples were evaluated. The sets are described below.

Sample Set A. Highly Contaminated OCC Blends
Two pulps were supplied from an old corrugated container recycling plant, Mill 1. One sample was of the final recycled pulp product before going to the papermachine. The other sample was the rejects from the quaternary screen and was highly contaminated. Stickies tests were performed on blends of the two pulps containing 0, 25, 50, 75, and 100% of the screen rejects pulp.

Sample Set B. Blends of Recycled OCC and Virgin 2001
Two pulps were supplied from Mill 1. One pulp was of the final recycled OCC pulp product just before the papermachine. The other sample was virgin kraft pulp. Each pulp was centrifuged to approximately 30% consistency upon receipt and stored in a cold room (44°F). Stickies tests were performed on blends of the two pulps containing 0, 25, 50, 75, and 100% of the recycled pulp.

Sample Set C. Mill 2: Accept Samples from Various Recycling Operations
Samples were taken after a secondary pulper, cleaners, screens, reverse cleaners, thickener, and disperser from Mill 2. Composite samples at each location were produced by combining specimens taken at 15 minute intervals for four hours.

Sample Set D. Mill 3: Accept Samples from Various Recycling Operations
Pulp samples were obtained from an OCC recycling mill, Mill 3. At the time, the incoming OCC bales were from one single wastepaper broker to reduce the variability of the incoming furnish. Only commercially collected OCC was included, no OCC from households or other sources were present. At the time, the fully processed recycled OCC pulp was re-routed around the high-density storage tank and directly sent from the recycling process to the papermachine, thus making samples at the headbox correspond directly to samples from the recycling process. Composite samples at several points in the mill were taken every 20 minutes over a three-hour period. Sampling points were: HD cleaners, coarse screens, MD cleaners, fine screens, gyro cleaners, disperser, and headbox.

Sample Set E. Blends of Recycled OCC and Virgin Pulp 2002
An OCC sample was obtained from a OCC recycling mill (Mill 2) from the secondary pulper. A virgin kraft pulp sample was taken from a virgin softwood kraft mill (Mill 4) from off the gravity decker prior to refining. These samples were used to make blends with several ratios to determine the minimum detectable concentration of stickies. Triplicate tests were performed on blended samples and a 95% confidence interval was determined. If the confidence interval did not contain the value of zero within it, then that test was deemed to have measured a significant content of stickies. Blends of OCC from the secondary pulper and virgin kraft fiber of 25%, 18, 12 and 0% of the OCC from the secondary pulper were utilized.

A summary of the different materials tested and the tests performed in the research is shown in **Table 1**. It also shows the approximate time-line when testing was complete.

Table 1. Matrix of Samples and Testing Performed

Sample	Date	PM Dep.	Bleach Dye HS	T277	PTM1	PTM2	PE Bottle	PE Film	Solvent
A	6/01	x	x	x	x	x			
B	9/01	x	x	x	x	x			
C	2/02	x	x	x	x	x			
D	7/02	x	x	x	x	x	x	x	x
E	12/02	x					x	x	x

RESULTS AND DISCUSSION

Macro Stickies Results

Macro Stickies: Sample Set A: Highly Contaminated OCC Blends
Our first set of test evaluations for macro-stickies test methods was performed on blends of fully processed OCC and quartenary screen rejects. Each test was performed in triplicate on blends including 0, 25, 50, 75 and 100% screen rejects. Note that the samples containing any amount of screen rejects had high contamination levels. **Figure 1** shows the PPM of stickies detected versus the % screen rejects in the blend for various test methods. **Table 2** lists the test methods and the corresponding R^2 values of the linear regression analysis.

Table 2. Correlation Coefficients for the Sample Set A: Highly Contaminated OCC Blends

Test	R^2
Bleaching and Dyeing	0.986
PT Method 1 (Handsheets)	0.980
PT Method 2 (Screening)	0.027
T277	No Useful Results
Deposition with Image Analysis	0.933
Deposition with Gravimetric Analysis	0.854

It is observed that the R^2 value for the two test methods that utilized handsheets for analysis, i.e., bleaching and dyeing and PT Method 1, were extremely high, around 0.98. These methods are sensitive and linearly related to the blend ratio at the given high contamination levels. The deposition method, either with image analysis or gravimetric analysis to detect the deposits, also was able to detect the stickies. However, the R^2 values were less than that with the handsheet methods.

PT Method 2 had a very poor correlation with the % screen rejects, R^2=0.027. This method concentrates the stickies and other contaminants on a filter pad for subsequent dyeing and image analysis. For all of the blends with screen rejects greater than 25%, the filter pad was completely covered with stickies and other contaminants, which prevented image analysis of the dyed pad from detecting any differences between these samples. The T277 testing method also has screening the pulp as one of its steps to concentrate the stickies. Again, it was found that the rejects filter pad was completely covered with material for all of the blend ratios investigated, rendering the results not useful. It may be possible to modify these tests for heavily contaminated pulps by screening smaller quantities of pulp for analysis.

The results of this portion of the study indicate that the handsheet methods are adequate with very high concentrations of stickies. Also, because PT Method 2 and T277 concentrate the stickies in a sample (advantageous for common levels of stickies, see later), these methods did not work for highly contaminated samples.

Macro-Stickies Sample Set B. Blends of Recycled OCC and Virgin 2001
It was also of interest to evaluate these test methods on pulps with lower stickies concentrations. To do this, the stickies tests as described in the experimental section were performed on blends of recycled OCC and virgin pulp in the ratios of 0, 25, 50, 75 and 100% recycled OCC.

The deposition test on the recycled OCC – virgin pulp blends was found to be insensitive to the % of recycled OCC. It was already known that the deposition test on the 100% recycled OCC resulted in extremely low results; the detected PPM and weight gain due to deposited stickies was 12 PPM and 0.5 milligrams, respectively. These detected quantities were near the minimum practical detection limit for image analysis and gravimetric measurements. It was thus expected that deposition measurements with blends of recycled OCC and virgin pulps would not be useful. In fact, deposition tests with 50% recycled pulp and 50% virgin pulp resulted in 0 PPM and 0.0 milligrams. Also, for the 100% virgin pulp 0 PPM and 0.0 milligrams were recorded. Due to these findings, the other blend ratios were not tested using deposition. It was concluded that *the deposition test is not useful for the low stickies concentration pulps* tested and the experimental procedures followed herein.

The deposition method is different from the other test methods in that it identifies stickies contaminants that deposit on materials such as papermachine fabrics under certain operating conditions. For instance, this could be an issue if the deposition test is performed at 50°C but the stickies present are depositable only at higher temperatures. Also, this can be a disadvantage in measuring total stickies content if detackifying materials in the pulp prevent the deposition of otherwise depositable stickies [10,11]. *However, the deposition test method can be very useful in the evaluation of anti-deposition programs or alternate fabric materials, see later.*

For the other stickies detection methods, the tests were performed in triplicate rounds on all of the blends and the results for one round of testing are shown in **Figure 2**. The two handsheet methods, i.e., bleaching and dyeing and PT Method 1, were consistent, resulting in similar (but not equal) stickies PPM levels versus blend ratio **(Figure 2)**. The average sticky size for the handsheet methods were both about 0.1 mm^2 for all of the blend ratios (data not shown). This indicates that the two handsheet methods are, in general, detecting the same types and quantities of contaminants in the tests.

Also, the two screening methods, i.e., PT Method 2 and T277, were consistent, resulting in similar (but not equal) stickies PPM levels versus blend ratio **(Figure 2)**. As screening concentrates the stickies the PPM of the screening methods was higher as expected than the handsheet methods. The average sticky size for both of the screening methods was about 0.4 mm^2 (data not shown). The larger average sticky size detected for the screening methods relative to the handsheet methods is understandable, as the screening operation eliminates the small particles from subsequent detection.

The correlation coefficient (R^2), for each test and each round are shown in **Table 3**. The average R^2 values of the three rounds for each test is also shown in **Table 3**. The average R^2 values for the two test methods that utilized handsheets for analysis, bleaching and dyeing and PT Method 1, were high, greater than 0.9. The two screening methods, PT Method 2 and T277 also had very high average R^2 values, greater than 0.94. Thus, all four of these methods were sensitive and linearly related to the blend ratio and accordingly, the stickies content.

Table 3. Results of linear regression analysis of detected stickies vs. the % recycled OCC in a blend of virgin kraft/recycled OCC for various test methods.

		R^2 For Best Fit Line			
Testing Method	Meas. per Test	Round 1	Round 2	Round 3	Avg.
Bleach/Dyeing	5	.974	.852	.995	.940
PT Method 1	5	.934	.875	.940	.916
PT Method 2	3	.983	.915	.952	.950
T277	1	.980	.982	.976	.979

Sample Set C. Mill 2: Accept Samples from Various Recycling Operations
Samples were taken after a secondary pulper, cleaners, screens, reverse cleaners, thickener, and disperser from Mill 2. Composite samples were produced by combining specimens taken at 15 minute intervals for four hours at each sample location.

It was found that the deposition method was not useful on these samples as the amount of deposits was zero or near the threshold of our detection limits. The results for the bleaching and dyeing method, PT Method 1, PT Method 2, and T277 are shown in **Figures 3-6**, respectively. It is observed that PT Method 1 and PT Method 2 demonstrate a generally decreasing stickies content as the pulp is processed through the various operations. This decreasing stickies content is more pronounced with PT Method 2. (It is assumed that all of the operations except for thickening should decrease the macro stickies content.) Bleaching and dyeing and T277 produced results showing *increased* stickies content after several operations. *This unlikely scenario indicates that the test results from bleaching and dyeing and T277 on mill samples across the mill are not reliable.*

To quantitatively compare the different testing methods a comparison of the relative scatter of the test measurements for each test was performed as follows. The confidence interval, CI, the range that the true average value is expected to exist with a 95% confidence was calculated for each test method and sample. The confidence interval is determined using the following formula:

$$95\%\ \mathrm{CI} = \bar{x} \pm t_{0.95} \frac{s}{\sqrt{n}}$$

where $\overline{x}$ is the average test result, $t_{0.95}$ is a tabulated statistic, s is the standard deviation and n is the number of samples. These intervals are plotted in **Figures 3-6**. Then, the average CI for each test method was determined over all of the sampling locations, **Table 4**. The overall range of test responses for each type of test was also determined, i.e., the difference between the average stickies content after the secondary pulper minus after the disperser, (SPAN), **Table 4**. The quantity, 100% * Avg CI / SPAN, was then determined, **Table 4**. Useful test methods will have good reproducibility (i.e., small CI) and a large range of measurement values across the mill (i.e., large SPAN) and thus a smaller value of 100% *Avg CI / SPAN. The value of 100% *Avg CI /SPAN is the lowest for PT Method 2 (52%), **Table 4**. Note that for all of the other tests, the expected CI of a measurement is greater than the total span of measurements expected across the entire recycle process. This indicates that these methods are not sensitive measures of stickies when compared to the total range of stickies measurement results expected from the beginning to the end of the recycling process.

It is important to note that the magnitude of the confidence intervals is a function of both the test method and the variability in the pulps. However, since all of the test methods used the same pulps for analysis, a useful comparison of the testing methods can be performed. *From **Table 4,** PT Method 2 shows a significant advantage over the other techniques in analyzing the mill samples across the mill.*

Table 4. Stickies Test Results (PPM) from Samples after Various Recycling Operations

	Avg. 95% Confidence Interval, CI	SPAN	100%*Avg CI / SPAN
Bleaching and Dyeing	3900	1330	300
PT Method 1	6060	5800	104
PT Method 2	8920	17360	52
T277	12540	7690	164

In the same study, separate time-dependent samples were obtained from the secondary pulper and the disperser every 15 minutes for 4 hours to investigate the expected fluctuations in the stickies concentration versus time. The stickies test results are shown using PT Method 2 for the samples in **Figure 7**. It was found that the coefficient of variation (100% * standard deviation / average) of the stickies concentration after the secondary pulper and after the disperser were 28% and 40%, respectively, **Figure 8**. *Thus, it has been demonstrated that in this mill (a typical OCC recycle mill) the fluctuations in stickies concentrations are considerable and it is expected that this is not unusual for OCC recycling mills.*

Sample Set D. Mill 3: Accept Samples from Various Recycling Operations
It was deemed of interest to evaluate the stickies tests across another OCC recycling mill. Pulp samples were obtained from an OCC recycling mill, Mill 3. A much tighter control over the feed OCC was maintained so it was expected that the pulp samples would be less variable relative to Mill 2. Composite samples at several points in the mill were taken every 20 minutes over a three-hour period. Sampling points were: HD cleaners, coarse screens, MD cleaners, fine screens, gyro cleaners, disperser, and headbox.

The results are shown in **Figures 9-12**. The results for the HD cleaners samples are considered invalid due to an extreme amount of unpulped material being in the sample. (The deposition method on these samples is discussed in the following section.) If we consider only the samples after the HD cleaner, the results indicate that PT Method 2 shows a good reproducibility and an expected downward trend in the stickies content, **Figure 11**. Bleaching and dyeing results do not indicate a downward trend in stickies through the process, **Figure 9**. PT Method 1 does show a downward trend but with large variability in the measurements, **Figure 10**. T277 shows a downward trend and reasonably low variability in the measurements, **Figure 12**.

Table 5 shows the value of 100% *Avg CI /SPAN for the different test methods. The effort to maintain a somewhat homogenous feed to the pulper taken here make the values of 100% *Avg CI /SPAN lower than those for the same tests for Mill 2, **Table 4**.
PT Method 2 and T277 displayed low values of 100% *Avg CI /SPAN for this trial, 24 and 17%, respectively. The PT Method 1 had a significantly higher value of 100% *Avg CI /SPAN equal to 50%. The bleaching and dyeing method was found to be completely ineffective with a value of 1710%. *The results indicate that PT Method 2 and T277 are the most effective stickies test methods based on these samples across the mill.*

Table 5. Stickies Test Results (PPM) from Samples after Various Recycling Operations

	Avg. 95% Confidence Interval, CI	SPAN	100%*Avg CI / SPAN
Bleaching and Dyeing	1720	100	1710
PT Method 1	1940	3830	50
PT Method 2	5100	21000	24
T277	21930	128330	17

Micro Stickies Results

Sample Set D. Mill 3: Accept Samples from Various Recycling Operations
In our first evaluation of micro-stickies test methods, accept pulp samples from HD cleaners, coarse screens, cleaners, the screens, gyro cleaners, disperser and headbox were used (same samples as in the section above for Macro stickies test methods). **Figures 13-17** show the results for all of the micro-stickies test methods except for image analysis. The 95% confidence intervals are indicated on the figures.

It was found that the HD cleaner sample had too much unpulped material and this contributed to an even larger variability in the test result, see **Figure 16** as an example. This was due to a large amount of unpulped debris interfering with the screening involved in the test. In fact, for any test method with screening involved, the results of the HD cleaner sample are considered invalid by the investigators.

The papermachine wire deposition, the bottle method, and the film method are related deposition methods and all show similar results, **Figures 13-15.** A large stickies content was determined for the coarse screen sample (with a large variability) relative to all of the other samples. All of the samples after the coarse screen had stickies contents that were about three times smaller than the coarse screen sample. Even though stickies contents detected were low after the coarse screen, a downward trend in the data is observed in general from the cleaners to the *disperser* samples.

The results of the Tappi Test Procedure for Micro-stickies in Process Water are shown in **Figure 16**. There is not a downward trend as expected. The data indicates that this method is not a useful technique for these samples. In fact, the procedure is better suited for extremely low consistency waters. Part of the difficulty in this method lies in the fact that the reported result is the subtraction of the results of the PT Method 2 test on a refrigerated sample from an unrefrigerated sample. Inspection of the refrigerated sample test results showed that the refrigerated and unrefrigerated samples had essentially the same stickies content. Subtraction basically generates noise from the data. Refrigeration did not agglomerate a significant amount of stickies that were screened. *The Tappi Test Procedure for Micro-stickies in Process Water is deemed not sensitive to accept samples across an OCC recycle mill.*

Solvent extraction data in **Figure 17** shows the expected downward trend in stickies content from coarse screen to disperser. The headbox sample does have a significantly higher test result, due perhaps to the additives and the accumulated materials in the papermachine section. *This increased stickies content was also observed for all three deposition methods, **Figures 13-15**, indicating that either sticky material is accumulating in the paper machine section or some additive is being detected as a sticky.*

For the microscopic image analysis technique, samples after the coarse screen and the disperser were analyzed. The results showed that there were a larger number of smaller particles after dispersion than after the coarse screens, reasonable considering the disperser objective to break-up large particles. However, it was unknown whether these

particles were sticky or non-sticky. *The microscopic image analysis method was found to be extremely time-consuming and tedious and not recommended for standard sticky detection.* With both bleaching and dyeing and then the use of microscopic image analysis required, the technique consumed about 8 hours for the analysis of a single pulp sample and it was deemed not of use to further investigate this technique.

Table 6 shows the 100% *Avg CI /SPAN over the samples from coarse screen to disperser. All of the deposition tests and the solvent extraction test had low values of 100%*Avg CI/SPAN, indicating utility for the methods. Further, inspection of **Figures 13-15** indicated that these methods showed expected downward trends. *Of the deposition methods used in micro-stickies testing, the data in **Table 6** and in **Figures 13-15** suggest that the polyethylene film method may be a more sensitive method for testing.* In contrast the Tappi Test Procedure for Micro-stickies in Process Water did not show the expected downward trend and also had a high value of 100%*Avg CI/span. It is concluded that the Tappi Test Procedure for Micro-stickies in Process Water is not useful in an OCC mill with fiber containing samples. Although the solvent extraction method had an acceptable value of 100%*Avg CI/span equal to 19%, it suffers from the fact that it requires the use of an organic solvent.

Table 6. Micro-Stickies Test Results (PPM and Weight) from Samples after Various Recycling Operations

Inland Mill Samples			
	Avg. 95% Confidence Interval, CI	SPAN	100%*Avg CI / SPAN
Deposition	.00248	.0199	12
Polyethylene Bottle Method	.00825	.0284	29
Polyethylene Film Method	.00208	.0318	7
Solvent Extraction	.05298	.2759	19
Tappi Method for Process Water (PPM)	5260	4050	130

Sample Set E. Mill 2. Blends of Recycled OCC and Virgin Pulp 2002

Micro-stickies test results on blends of OCC from the secondary pulper and virgin kraft pulp are shown in **Figures 18-21** for blends containing 25%, 18, 12 and 0% of the OCC from the secondary pulper. The 95% confidence intervals are indicated on the figures. **Table 7** shows the average test value result and the upper and lower limit of the 95% confidence interval for all tests and blends. A negative lower limit of the 95% confidence interval indicates that the test result is not significantly different than a zero stickies content.

At both the 0 and 12% levels of secondary pulper OCC none of the four tests were able to recognize a non-zero sticky content. Only the solvent extraction method was able to identify a non-zero sticky content for both the 18 and 25% levels. From this data, the solvent extraction method is deemed the most sensitive. However, a weakness of the solvent extraction method is the low quantitative values of the method, even at a 18% level (circa 2 milligrams), which would be difficult to reproduce with accuracy in a dynamic industrial setting.

Table 8 shows the average 95% confidence interval over the blend samples from 25% to 0% secondary pulper OCC, the span, defined as the (stickies content of 25% minus the 0% secondary pulper OCC sample, and the 100%*Avg CI/span. A lower value of 100%*Avg CI/span indicates a better performing test. The relatively lower value of 100%*Avg CI/span for the solvent test relative to the three other tests is in agreement with the statement that the solvent test is more sensitive than the others. The relatively high 100%*Avg CI/span for the paper machine wire deposition indicate that it is not as sensitive as the other three methods and is therefore not recommended for use.

The approximate run-time for the solvent test is estimated at 8 hours, much longer than the other three tests with run-times of about 30 minutes.

The small gravimetric values of the solvent test, the long run-time and the issues surrounding the use of the quantity of solvent needed are disadvantages for the solvent test. The paper machine wire deposition test suffers from low sensitivity. *The polyethylene bottle or film method appears to be a more practical and useful test in an industrial setting.*

Table 7. Micro stickies test averages and 95% confidence intervals for each test method for blends of OCC from the secondary pulper and virgin kraft pulp.

Micro Stickies Testing Methods			
Test Method and % OCC in Blend	Average Stickies Weight (g)	95% Confidence Intervals	
		Upper Limit	Lower Limit
Deposition			
0 %	0.0000	0.0000	0.0000
12%	0.0000	0.0004	-0.0003
18%	0.0007	0.0012	0.0002
25%	0.0026	0.0055	-0.0003
Polyethylene Bottle Method			
0 %	0.0000	0.0000	0.0000
12%	0.0001	0.0003	-0.0001
18%	0.0073	0.0115	0.0031
25%	0.0094	0.0207	-0.0020
Polyethylene Film Method			
0 %	0.0000	0.0000	0.0000
12%	0.0000	0.0001	0.0000
18%	0.0008	0.0018	-0.0002
25%	0.0049	0.0073	0.0026
Solvent Extraction			
0 %	0.0000	0.0000	0.0000
12%	0.0002	0.0005	-0.0002
18%	0.0022	0.0037	0.0007
25%	0.0498	0.0498	0.0394

Table 8. Micro Stickies Test Results from blends of OCC from the secondary pulper and virgin kraft pulp

Micro Test Methods			
	Avg. 95% Confidence Interval, CI	SPAN	100%*Avg CI / SPAN
Deposition	.0009	.0026	35
Polyethylene Bottle Method	.0016	.0121	13
Polyethylene Film Method	.0003	.0051	6
Solvent Extraction	.0007	.0435	2

Evaluation of Stream-lined Tests

There was an interest in evaluating ways of "stream-lining" stickies testing methods. **Table 9** lists the proposed modifications for the "stream-lined" test methods and the results of our investigations. From our findings it is not recommended to perform any of these modifications as the time saved is marginal and in most cases the test results are less reproducible.

Table 9. Listing of strategies to "stream-line" stickies testing methods and comments on the results.

PE Bottle and PE Film	Result
Reduce test to one bottle (film) rather than four.	The use of one bottle rather than four used one fourth of the pulp and the variability of the experiment was increased accordingly. Performing 4 bottles rather than 1 is not a critical factor in the time consumed in the test.
Use Microwave to heat pulp.	Microwave heating took about 5 minutes and did not provide significant decreases in time to do experiment.
Use an heated air gun to dry bottle.	An air gun tended to knock off some deposits and is therefore not recommended
Reduce deposition time from 10 to 5 minutes.	A noticeable decrease in deposit weight occurred at 5 minutes relative to 10 minutes. This decrease caused the test to have deposit weights lower than the balance sensitivity, 1 milligram.
PT Method 1 and PT Method 2	
Use a heated air gun to dry dyed handsheet.	Using a heated air gun took about the same time as a speed drier and resulted in wrinkled handsheets.
Attempted to add ink to handsheet rather than to dry filter pad followed by a second drying step.	The entire handsheet turned black, removing all contrast between contaminants and fiber, destroying the ability of the test to detect contaminants.
Manual count	Manually counting of spots on handsheets does not save time and increases the labor on the technician and the variability of the results. Manual counts or qualitative evaluation of the handsheets are possible in order to eliminate the purchase of an image analysis system.
T277	
Press filter pad and coated sheet for 5 min. instead of 10.	In order for the coating material to transfer to the stickies contaminants the sample must be dried. However, at 5 minutes pressing time the sample was wet and no coating material transferred. Attempts at 6, 7, and 8 minutes showed the same result. It is preferred to simply use the 10 minute pressing time.
Replace hot pressing after rinsing step with the use of drying on a speed drier.	Using the speed drier was an acceptable method to use. The modification saved about 7 minutes.
Manual count	(see comment above for PT Method 1)

Estimates of Time and Labor Necessary for the Various Stickies Test Methods.

From a practical standpoint the actual time and labor that is required to perform stickies test methods is of importance. Clearly, sampling issues are of extreme importance when trying to characterize quantities of pulp flowing in the 100 tons per day range with tests that analyze less than 100 grams of pulp. Thus, given a limited amount of testing resources, convenient and rapid testing methods will allow for increased sampling and testing and more precise measurements.

Estimates of the total time and labor required for macro and micro stickies test methods are shown in **Tables 10 and 11**, respectively. These values are estimates intended to demonstrate the relative efforts needed among the different testing methods. It is very plausible that the total time and the labor needed could be decreased by modifications to the described procedure. For example, drying (estimated as taking 1 hour) could be replaced with a more rapid technique, such as by using a speed dryer.

The labor required in the macro stickies test methods was about three hours for the handsheet methods (bleaching and dyeing and PT Method 1) and only about one hour for the screening methods (PT Method 2 and T277), **Table 10**. *Thus, the screening methods show a significant advantage in decreased labor relative to the handsheet methods.*

The paper machine deposition method does not require more labor than the other macro stickies techniques and in fact when analyzed gravimetrically has one of the lowest labor demands. However, it does require the fabrication of non-commercially available equipment. *With real OCC mill samples the actual amount of deposited material is extremely low, approaching the limitations of common gravimetric balances and image analysis systems. This is a severe limitation of the paper machine wire deposition method for routine stickies testing.*

With regards to the micro stickies test methods, all of the techniques show a relatively low amount of labor demanded, **Table 11**. However, the solvent extraction technique has a significantly longer run time, approximately 13.7 hours, relative to the other techniques. *The small gravimetric values of the solvent test, the long run-time and the issues surrounding the use of the quantity of solvent needed are disadvantages for the solvent test and make it impractical for routine stickies testing.*

Table 10. Estimates of the total time and labor required in hours for the various "macro"-stickies test methods.

Testing Method	Bleaching & Dyeing		PM Wire Dep. Image Analysis		PM Wire Dep Gravimetric		PT Method 1		PT Method 2		T277	
	Total Time	Labor	Total Time	Labor	Total Time	Labor	Total Time	Labor	Total Time	Labor	Total Time	Labor
Test Preparation	1	.3	1	.5	1	.5	1	1	.5	.5	.5	.5
Test Run Time	2	.5	1	*	1	*	*	*	1.5	.5	.5	.2
Handsheet Preparation	.3	.3	*	*	*	*	.3	.3	*	*	*	*
Drying	1	*	1	*	1	*	.8	*	.5	*	.2	*
Rinsing	*	*	*	*	*	*	*	*	*	*	.1	.1
Weighing	.2	.2	.2	.2	.2	.2	.2	.2	.1	.1	*	*
Dyeing	.5	.5	.3	.3			.5	.5	.2	.2	*	*
Drying	1	*	1	*			1	*	1	*	.2	*
Weighing	*	*	*	*			*	*	*	*	.1	.1
Rinsing	.5	.5	.3	.3			*	*	*	*	*	*
Drying	1	*	1	*			*	*	*	*	*	*
Image Analysis	1	1	.8	.8			1	1	.3	.3	.1	.1
Total	8.5 HR	3.3 HR	6.6 HR	2.1 HR	3.2 HR	.7 HR	4.8 HR	3 HR	4.1 HR	1.6 HR	1.7 HR	1 HR

Table 11. Estimates of the total time and labor required in hours for the various "micro"-stickies test methods.

Testing Method	PM Wire Deposition Gravimetric		LDPE Bottle Method		LDPE Film Method		Solvent Extraction	
	Total Time	Labor	Total Time	Labor	Total Time	Labor	Total Time	Labor
Test Preparation	1	.5	1	.5	1	.5	.5	.5
Test Run Time	1	*	.2	*	.2	*	8	.3
Evaporation in Hood	*	*	*	*	*	*	4	*
Drying	1	*	1	*	1	*	1	*
Weighing	.2	.2	.2	.2	.2	.2	.2	.2
Total	3.2 HR	.7 HR	2.4 HR	.7 HR	2.4 HR	.7 HR	13.7 HR	1 HR

Evaluation of Stickies Removal Across a Water Clarifier

It is of interest to determine if water clarification decreases the amount of stickies in process water. Samples were taken before and after a dissolved air flotation clarifier in Mill 3. **Table 12** shows the solvent extraction test results on the clarified and unclarified process water. The clarification process indeed decreased the stickies content by about 20% in the water, as measured by the solvent extraction method. From the limited data, the solvent extraction method appears to be useful in analyzing the clarification process.

Table 12. Solvent Extraction Data for Non-Clarified Water and Clarified Water Samples

	Stickies Weight (grams) as measured by Solvent Extraction				
Sample Location	Test One	Test Two	Test Three	Average	95% CI
Non-Clarified Water	.0565	.0619	.0626	.0603	.0083
Clarified Water	.0496	.0481	.0477	.0484	.0025

Another stickies test method was evaluated on the clarified and unclarified water of Mill 3, an Institute of Paper Science and Technology (IPST) method based on total organic carbon (TOC). A test description follows. The sample is filtered through Whatman 4 filter paper that has an approximate pore size of 25 microns. Part of the filtrate is directly tested for TOC and another part of the filtrate is ultra-filtered through a membrane that allows compounds less than 3,000 molecular weight only to pass through and the TOC of this ultra-filtrate is determined. Subtraction of the two filtrates provides a measure of the TOC in the original sample with size less than 25 microns and molecular weight greater than 3,000. This corresponds to high molecular weight compounds based on carbon that are supposedly the tacky, depositable stickies material.

The TOC based method does not show a decrease in stickies after clarification, **Table 13**. This is reasonable as clarification works best on large suspended solids but doesn't affect dissolved species. This is in contrast to the solvent extraction results showing an about 20% decrease in **Table 12**. This suggests that the solvent extraction method is detecting a decreased amount of large stickies (> 25 microns) for which the TOC method is insensitive.

Table 13. IPST TOC Data for Non-Clarified Water and Clarified Water Samples

	Stickies PPM by Weight in Water
Non-Clarified Water	242
Clarified Water	295

Overall Stickies Test Methods Recommendations for Various Applications in an OCC Recycle Mill

Test Methods to Evaluate an OCC Recycling Process or an Individual Pulp Processing Operation

Two major types of experimental designs were performed in this research, performing test evaluations on (1) blends of two pulps with differing amounts of stickies contaminants and (2) on accept samples from the major unit operations across the OCC recycling mill. In general, after completing this study, it is felt that the evaluations on accept samples across the OCC recycle mill produced data that is a better foundation for developing conclusions on the utility of the test methods. Thus, most of the evaluations that follow are based on these sets of data. **Table 14** categorizes the different test methods as to their ability to track stickies contaminant levels across an OCC recycle mill or simply across a single unit operation. The qualitative comments that follow are based on the shapes of the graphs of stickies detected versus unit operation, the confidence intervals calculated and the quantitative value of 100%*Avg CI/span.

Table 14. Overall utility of stickies test methods to track stickies contaminant levels across an OCC recycle mill.

Category	**Macro Stickies Test**	**Micro Stickies Test**
Preferred Method	Port Townsend Method 2	PE Film Deposition
Good Method	T277	PE Bottle Deposition
Not Recommended	Bleaching/Dyeing of HS PM Wire Deposition – IA PM Wire Deposition – Grav PT Method 1	Solvent Extraction Microscopic IA PM Wire Deposition – IA PM Wire Deposition – Grav Tappi MicroStickies Process Water

Macro Stickies Test Methods

- Port Townsend Method 2 showed consistently better (from the two OCC mills studied) ability to show a trend in decreasing macro-stickies contents across the operations of an OCC recycle mill than the other tests. Port Townsend Method 2 test results also showed better reproducibility on repeated tests of the same samples (less scatter of data). The labor demanded and the skills and equipment needed to run the test were reasonable. This is the preferred method of macro-stickies testing to characterize overall mill or a single operation performance based on this research.

- T277 was shown to be adequate in its ability to track stickies in one mill trial but not another. It is for this reason that it is not the preferred method. However, if a mill has a specific need that indicates that this test would be desirable, it would be of value to

pursue its utility relative to Port Townsend Method 2. Its labor, equipment and skills demands were reasonable.

- Bleaching and dyeing of handsheets is not a recommended test method. Simply put, the method failed to detect changes in stickies concentrations in two OCC recycling mill trials. The method is also very labor intensive and demands skill and consistency from the technician. The limitation in sample size, due to the labor intensive handsheet making process, makes this method impractical.

- Paper machine wire deposition test method, both gravimetric and image analysis is not recommended. In general, this method was not at all useful for the detection of stickies through the major unit operations across an OCC recycle mill. With typical contaminant levels in accepted pulp streams, the amount of deposits recorded with this technique is extremely low, near the sensitivity limit of gravimetric and image analysis. (This technique is better used to study relatively high concentrations model stickies for stickies control systems for a papermachine.)

- Port Townsend Method 1 showed no ability to track stickies in one mill trial and a marginal ability in the second mill trial. Based on these results it is not recommended to use this technique. The limitations in sample size, due to the labor intensive handsheet making process, make this method impractical.

Micro Stickies Test Methods

- Polyethylene Film Deposition method displayed a good reproducibility of test results on repeated tests of the same samples (less scatter of data). Further this method showed an ability to detect decreases in stickies across the operations of an OCC recycle mill. The labor demanded and the skills and equipment needed to run the test were reasonable. This is the preferred method of micro stickies testing to characterize overall mill or a single operation performance.

- Polyethylene Bottle Deposition was shown to be adequate in its ability to track micro stickies. The polyethylene film method performed marginally better than the bottle method. It is for this reason that it is not the preferred micro stickies method. However, if a mill has a specific need that indicates that this test would be desirable, it would be of value to pursue its utility relative to film method. Its labor, equipment and skills demands were reasonable.

- Solvent Extraction method was found to be able to track micro stickies very well. A consistent downward trend in stickies concentrations across the operation of an OCC recycling mill were found using this method. The reproducibility of the method was good. This method is considered to be sensitive to papermaking chemicals in the papermachine area. This may be advantageous or disadvantageous depending on the intended use of the test. Overall, although the test works, it suffers from two significant disadvantages: it is time consuming to perform and it necessitates the use

of relatively large quantities of organic solvent. Due to these two disadvantages, it is not recommended for routine stickies testing in an OCC recycle mill.

- The microscopic image analysis method was found to be extremely time-consuming and tedious and not recommended for standard sticky detection. Distinguishing dyed blue adhesive particles from dark contaminants under the microscope was not possible, further weakening its ability to detect stickies.

- Paper machine wire deposition test method, both gravimetric and image analysis. In general this method was not at all useful for the detection of stickies through the major unit operations across an OCC recycle mill. With typical contaminant levels in accepted pulp streams, the amount of deposits recorded is extremely low, near the sensitivity limit of gravimetric and image analysis.

- The Tappi Test Procedure for Micro-stickies in Process Water was not able to detect any decreases in stickies contents across the major operations of an OCC recycling mill. This result indicates that this method is not a useful technique for stickies detection in pulp containing samples and is thus not recommended for routine stickies testing. (There may be some utility in this method for fiber-free water streams.)

Test Methods as a Method to Evaluate OCC as a Raw Material

The evaluation of recovered OCC as the raw material to an OCC recycle mill is of significant importance. The following is simply the author's opinion on bale inspection for stickies and wax. When evaluating bales of recovered paper the first issue that comes about is sampling. It is recommended to inspect several (3-5) bales in a shipment after they have been broken open. For each bale inspected a simple count (or visual inspection) of a portion ($1/20^{th}$) of the bale for wax-OCC versus non wax-OCC is probably the most productive activity to evaluate quality. This count should be performed for 3-5 bales and an average and range reported. About 5% wax boxes (6 weight %) have been thought to be acceptable feedstock. Thus, a count of 1/20 wax-OCC/non wax-OCC or less is acceptable. Another step in bale inspection is to look for a preponderance of the following items: plastic films and foams, food, metals, oil or grease contamination, chipboard or other paper grades, and other large contaminants.

In very rare and critical cases it may be of value to determine a stickies content of a shipment. The following is simply the authors speculation on the topic. Sampling is the major issue. In this case a device that will take a core out of a bale is needed. A hand-held drill with an ability to cut about a 2-inch core 2 feet into a bale should be used. Two to three cores should be taken from several bales in the shipment (5-15). The cores should be mixed in a drum. Enough material should be taken and pulped to perform a stickies test. It may be possible to indirectly detect adhesive material by using a very coarse screen (1/4 to ½ inch mesh) to determine unpulped material. A simple wet or dry weight of the rejects should be indicative of stickies contaminants as found on tapes,

adhesives, wax etc. that are hard to repulp. If a critical need exists, Port Townsend Method 2 may be attempted to more closely evaluate stickies.

Test Methods as a Method to Evaluate Issues Involving Paper Machine Deposits

In this case stickies deposits should be scraped off and elemental analysis performed to determine the metal content of the deposit. A simple ash content is always useful to perform. Further, chemical spectroscopy composition analysis, such as used to detect fatty acids, can be performed to determine organic components in the deposits. Many chemical vendors will perform these analyses as a service to customers.

Evaluating paper machine anti-deposition strategies is an area in which the paper machine wire deposition test may be useful. In order to develop useful results, the papermachine water should be simulated with respect to pH, conductivity and types/concentrations of ionic species. Artificially high concentrations of model stickies should be used in the test to improve detection. A control experiment with no anti-deposition control system should be performed and compared to all of the anti-deposition control strategies of interest.

Test Methods as a Method to Evaluate Mill Water System

From the limited testing results on the clarified and unclarified water from Mill 3 the following comments can be made.

- Solvent extraction can detect changes in stickies materials across a clarifier. This statement is based on the assumption that all species soluble in the solvent are potential stickies. As a water clarifier is known to remove suspended solids and not dissolved solids, the solvent extraction method is sensitive to the suspended solids removed by the clarifier.

- The IPST-TOC method is capable of measuring stickies in water with size less than 25 microns and molecular weight greater than 3,000. This corresponds to high molecular weight compounds based on carbon that are supposedly the tacky, depositable stickies material. Thus, this method is useful for dissolved stickies in water. However, this method is not useful in the analysis of a clarifier because a clarifier has no effect on dissolved species in process water.

CONCLUSIONS

Of the macro stickies test methods, the Port Townsend Method 2 is the preferred method of macro-stickies testing to characterize overall mill or single pulp processing operation performance. This macro stickies test method showed a clear decrease in macro-stickies across the OCC recycle mill, was reproducible and was practical to implement.

Of the micro stickies test methods, the Polyethylene Film Deposition method is the preferred method of micro-stickies testing to characterize overall mill or single pulp processing operation performance. This micro-stickies test method showed a clear decrease in micro stickies across the OCC mill, was reproducible and was practical to implement.

Fluctuations in stickies contents at any given part of an OCC recycle mill are expected to be large, having a coefficient of variation of about 30%. Thus, errors in stickies detection due to sampling issues are expected to be significant. All practical means of alleviating sampling issues should be implemented.

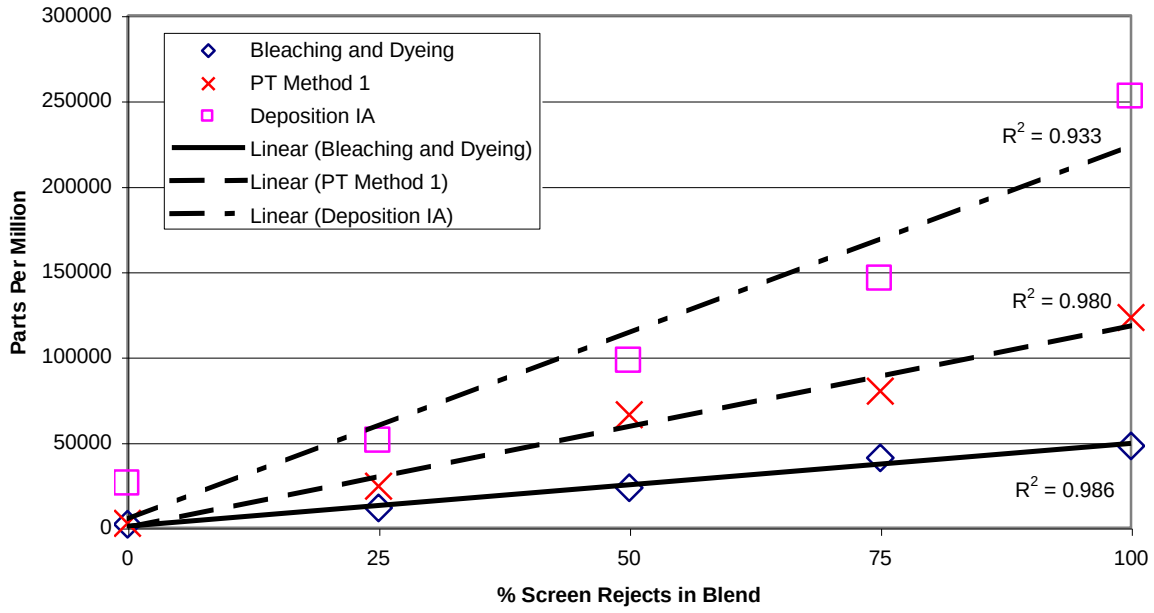

Figure 1. PPM of stickies detected versus the % screen rejects in a blend of recycled OCC/ screen rejects for various test methods. The original deposition results have been multiplied by a constant factor of 2000 for presentation purposes. (Sample Set A)

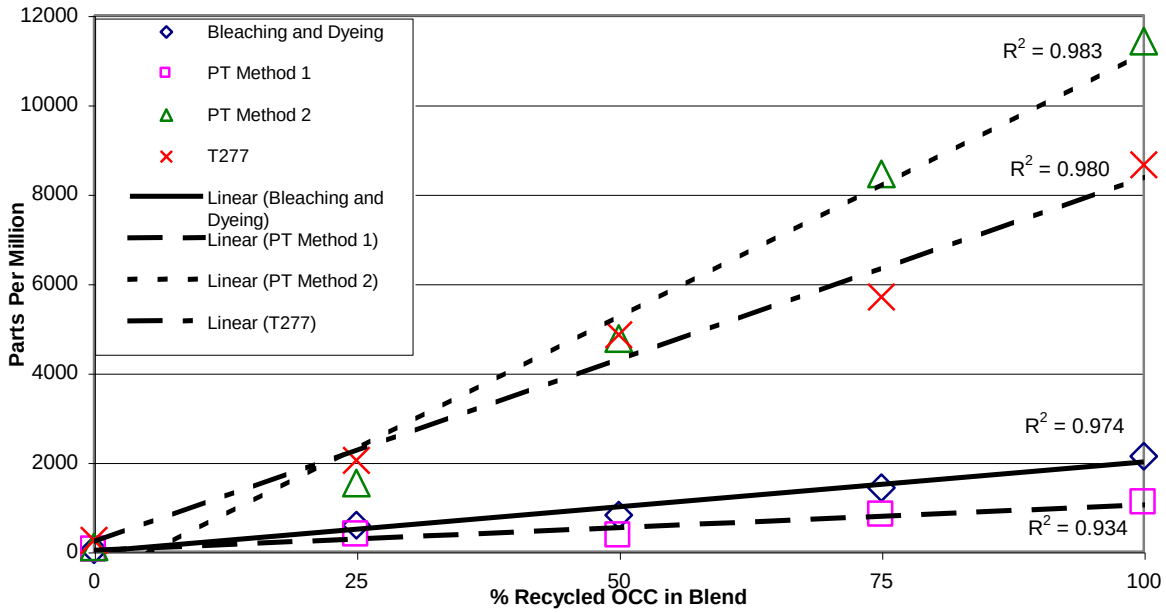

Figure 2. Average PPM of stickies detected versus the % recycled OCC in a blend of virgin kraft/ recycled OCC for various test methods. (Sample Set B)

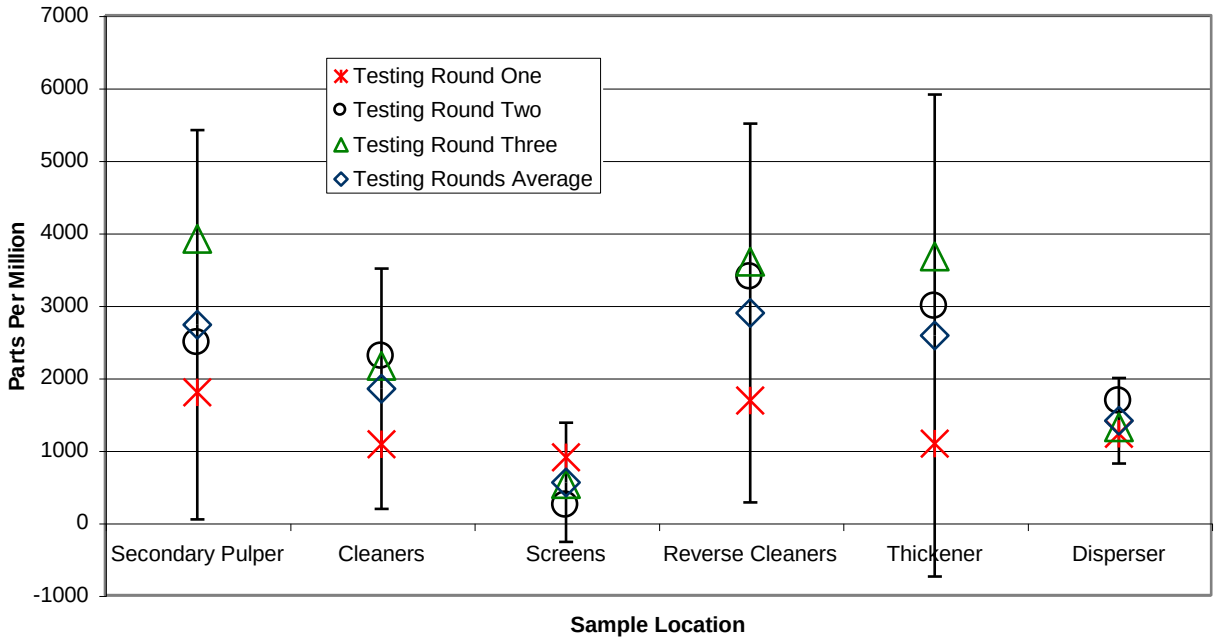

Figure 3. Stickies PPM as measured by the Bleaching and Dyeing Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set C)

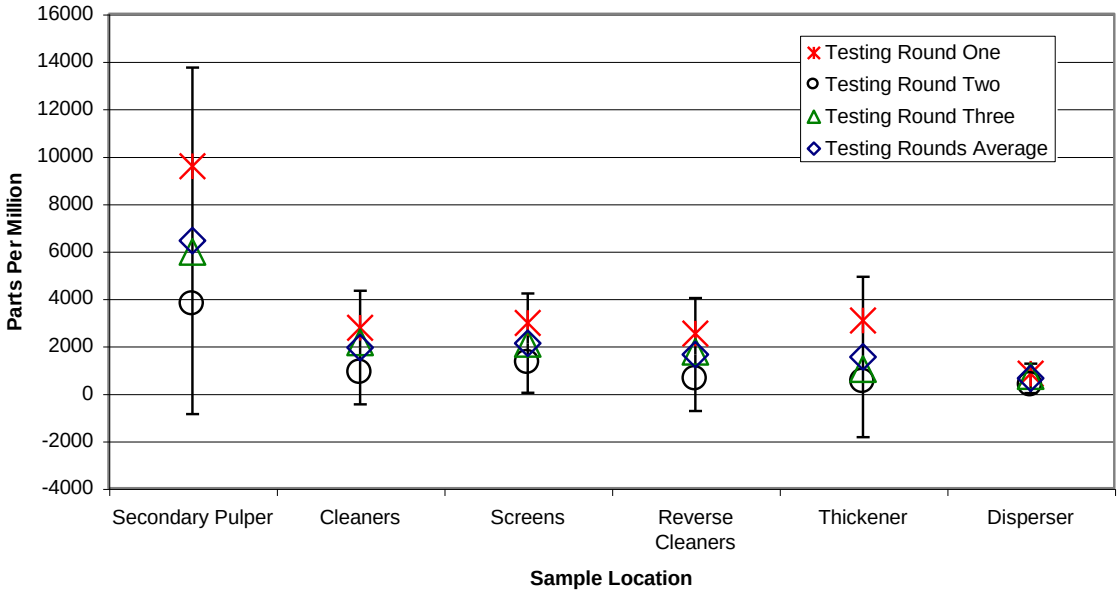

Figure 4. Stickies PPM as measured by the PT Method 1 on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set C)

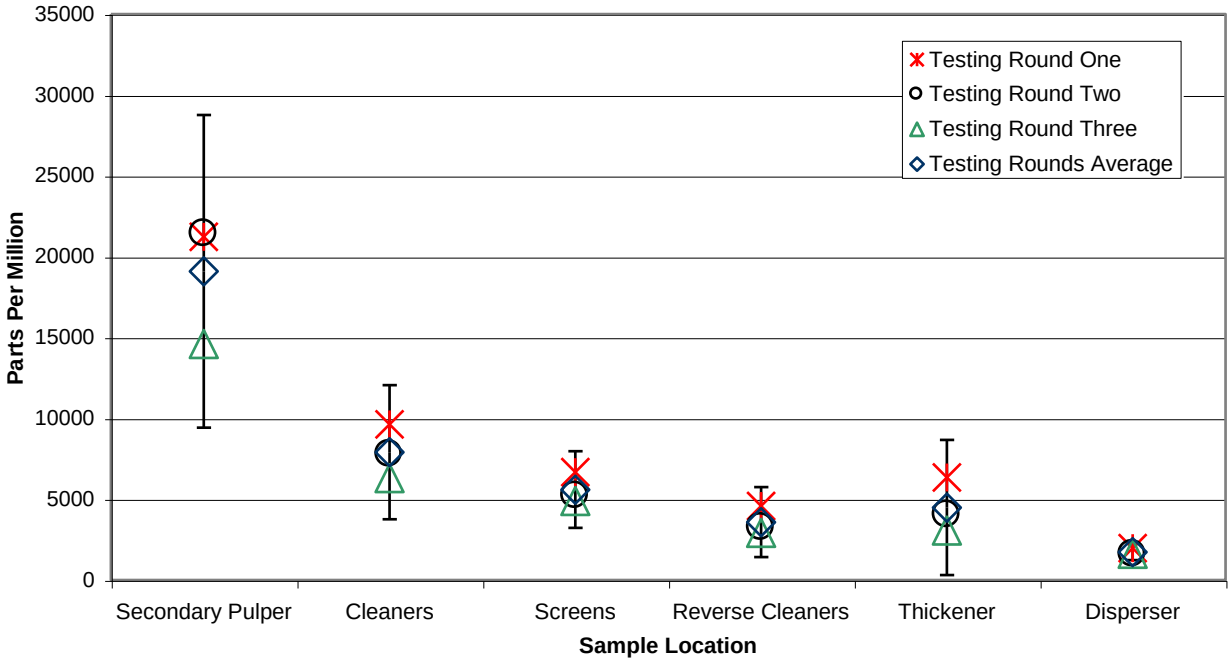

Figure 5. Stickies PPM as measured by the PT Method 2 on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set C)

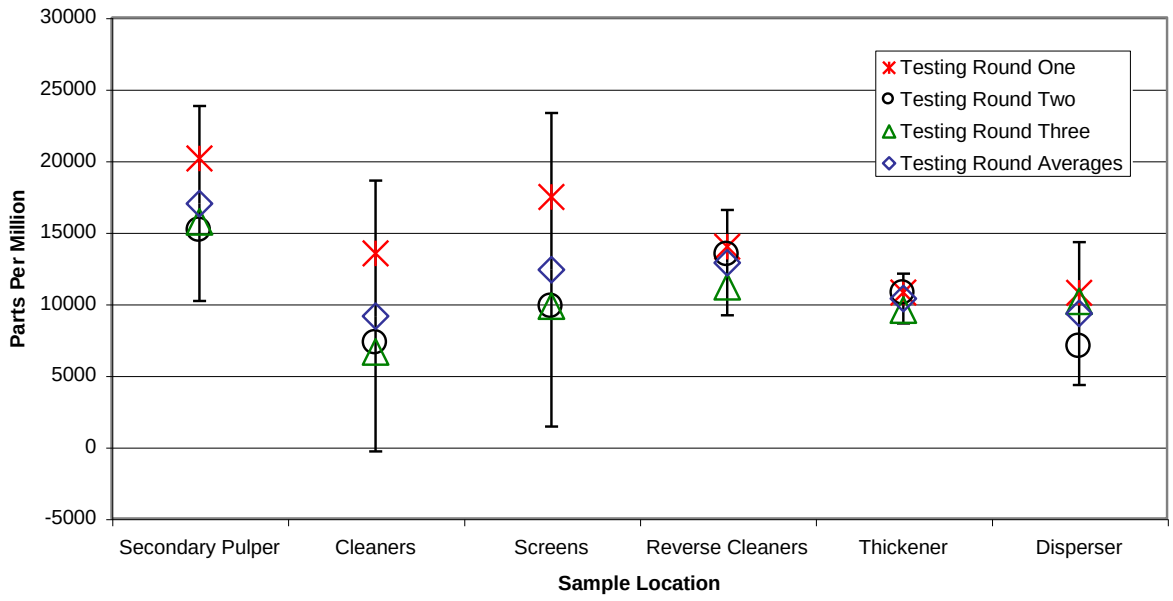

Figure 6. Stickies PPM as measured by the T277 on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set C)

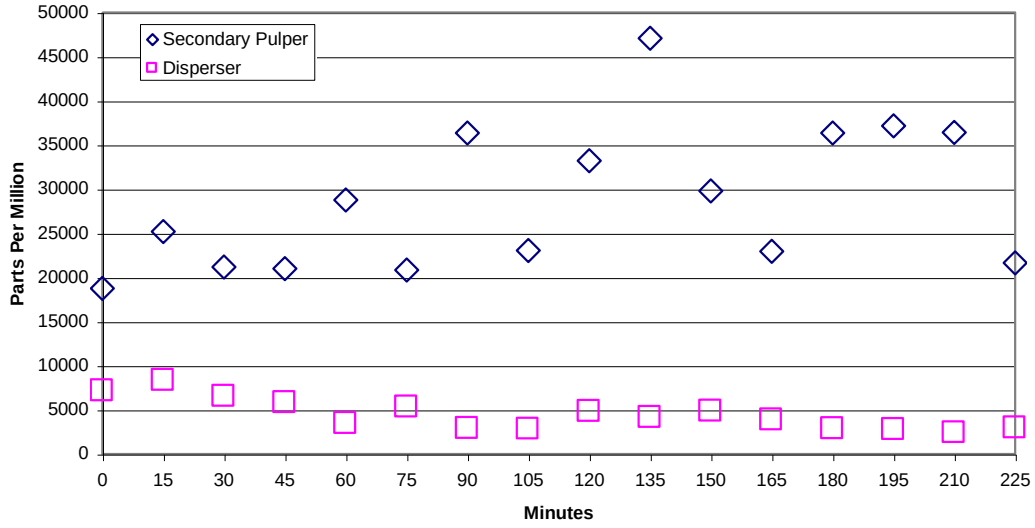

Figure 7. Stickies PPM versus Time as measured by PT Method 2 on time samples. (Sample Set C)

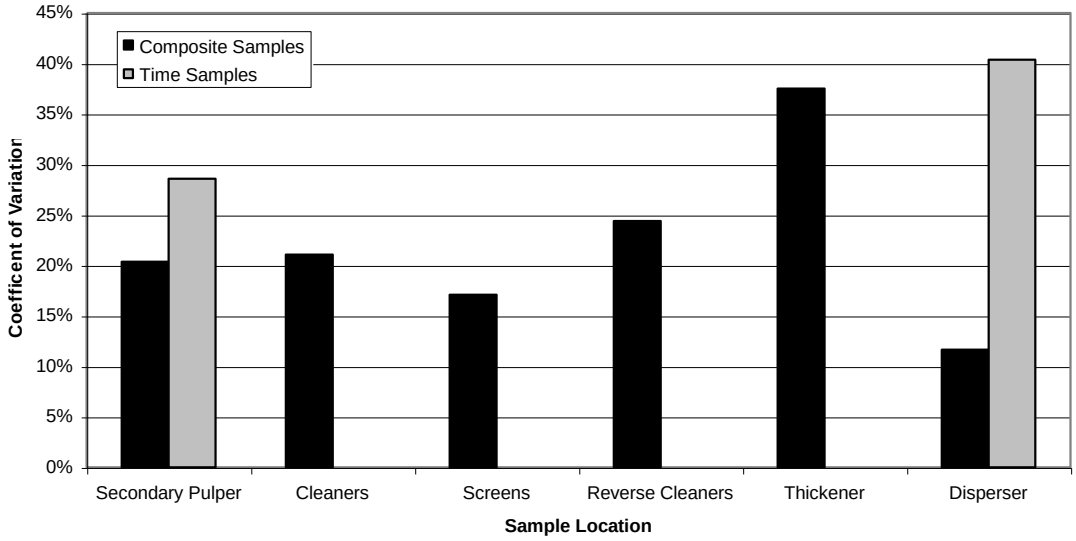

Figure 8. Coefficent of Variation for composite samples (dark bars) and for time samples (light bars). (Sample Set C)

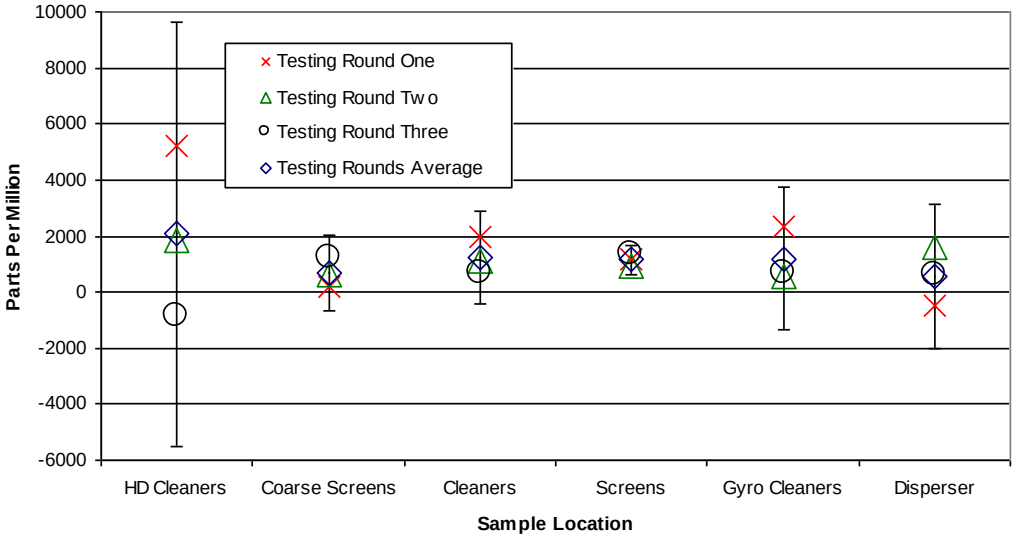

Figure 9. Stickies PPM as measured by the Bleaching and Dyeing Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

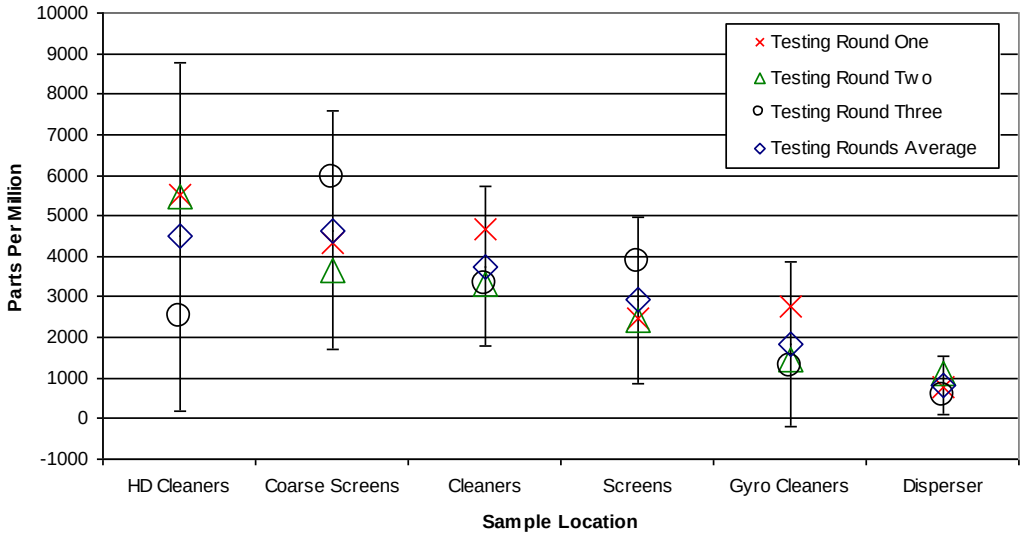

Figure 10. Stickies PPM as measured by PT Method 1 on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

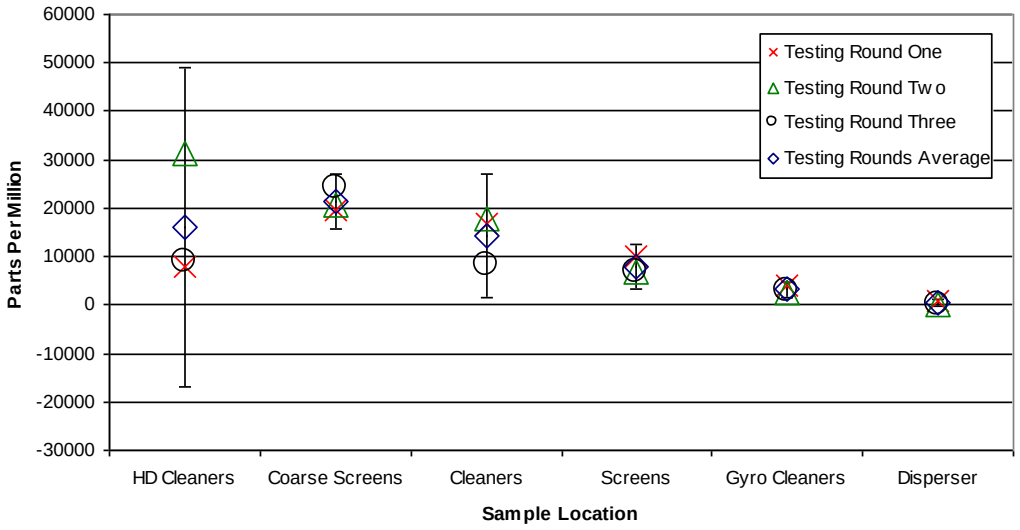

Figure 11. Stickies PPM as measured by PT Method 2 on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

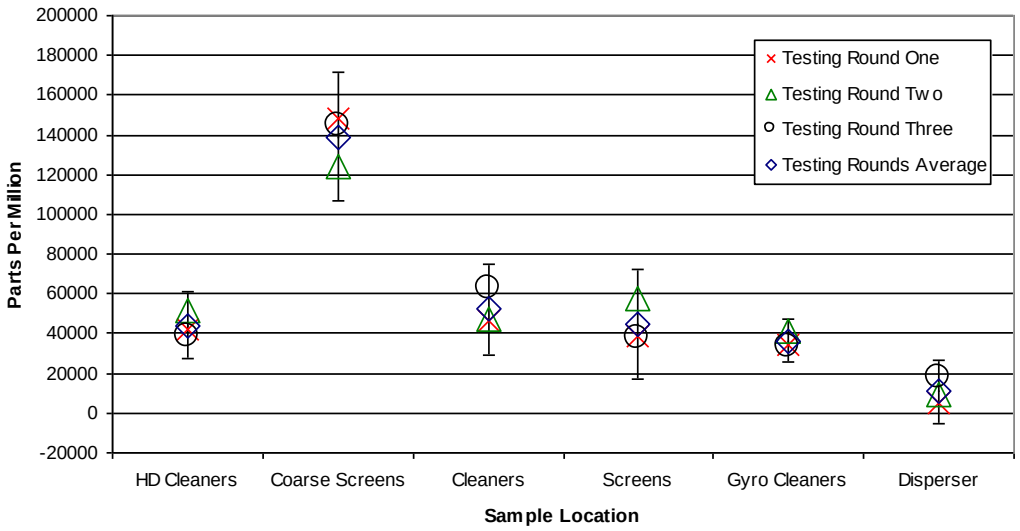

Figure 12. Stickies PPM as measured by T277 on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

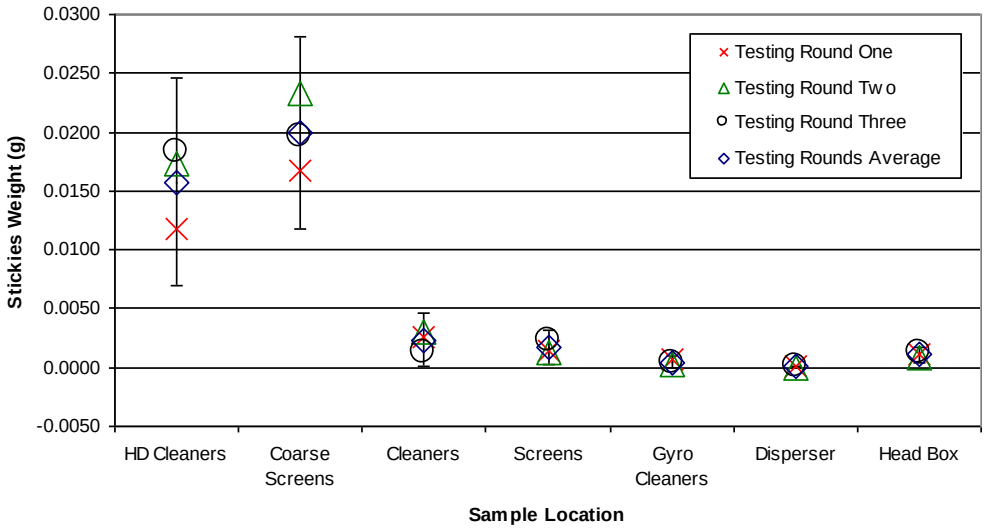

Figure 13. Stickies PPM as measured by the Deposition Test Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

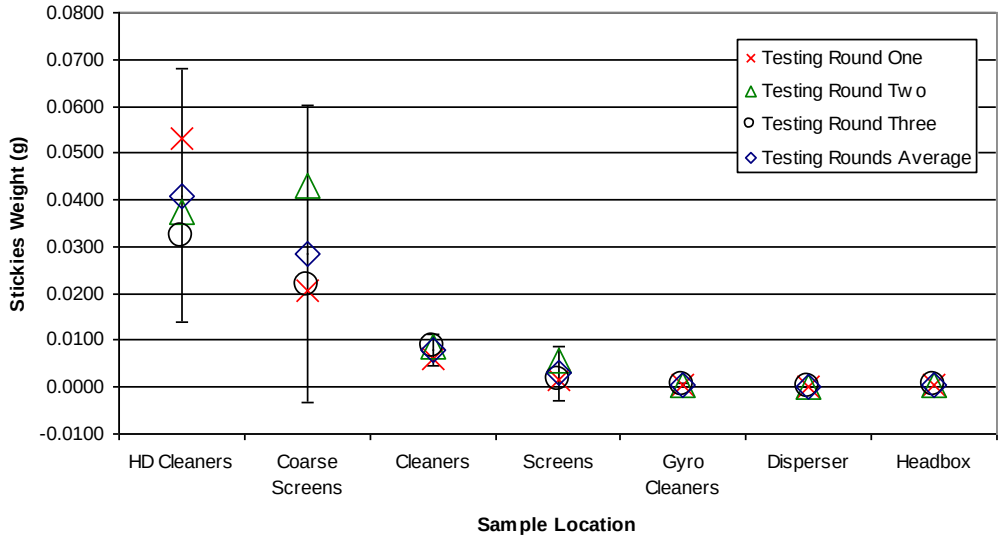

Figure 14. Stickies PPM as measured by the Polyethylene Bottle Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

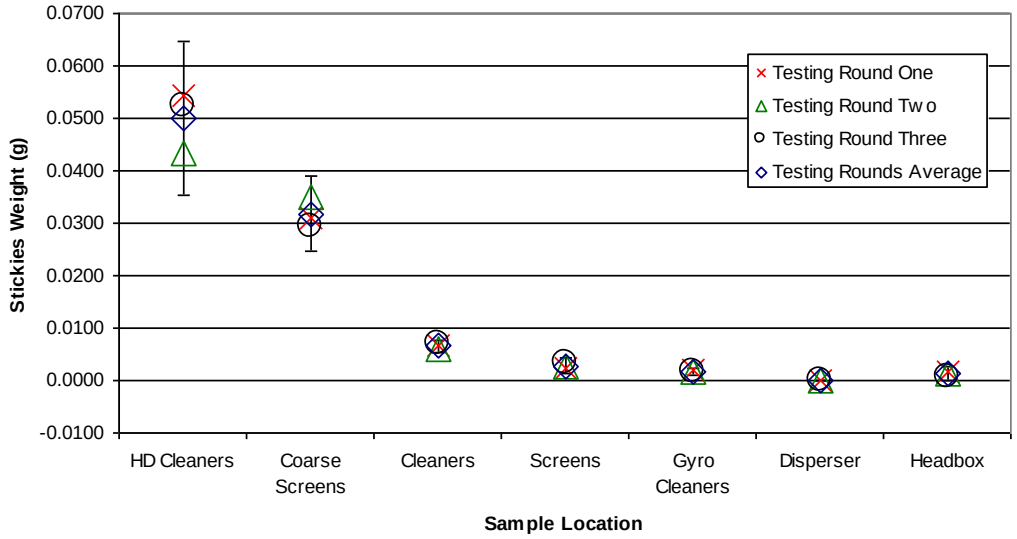

Figure 15. Stickies PPM as measured by the Polyethylene Film Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

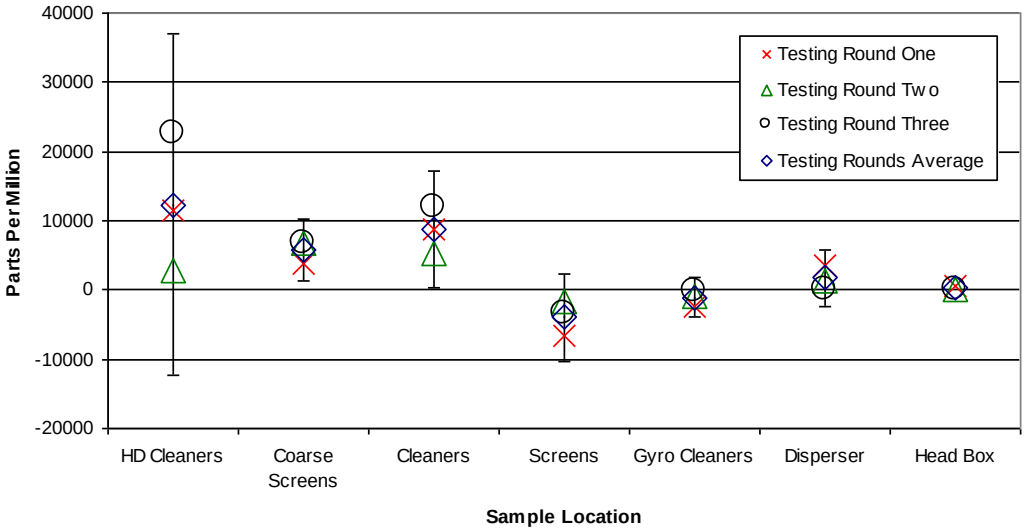

Figure 16. Stickies PPM as measured by the Tappi Method for Micro-stickies in Process Water on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

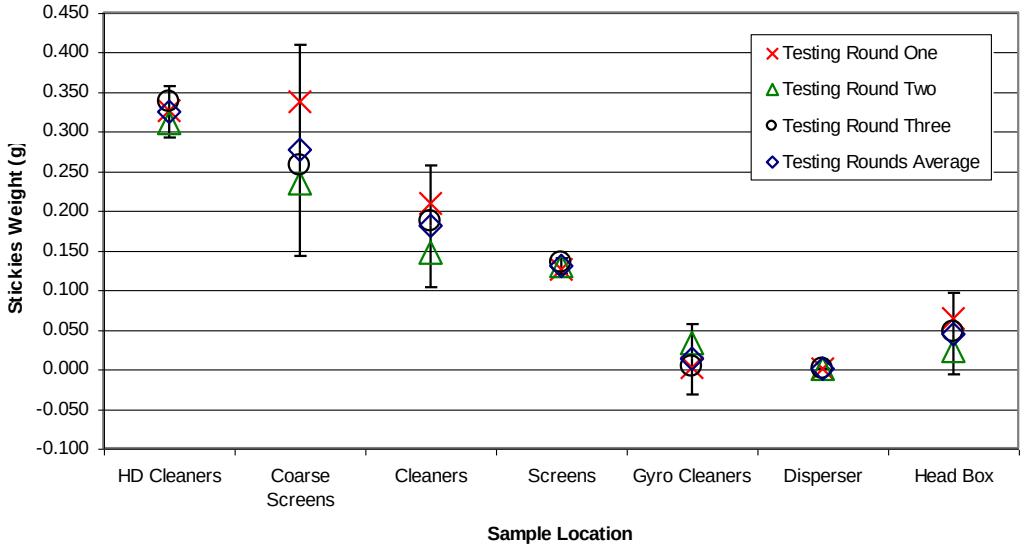

Figure 17. Stickies PPM as measured by Solvent Extraction on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set D)

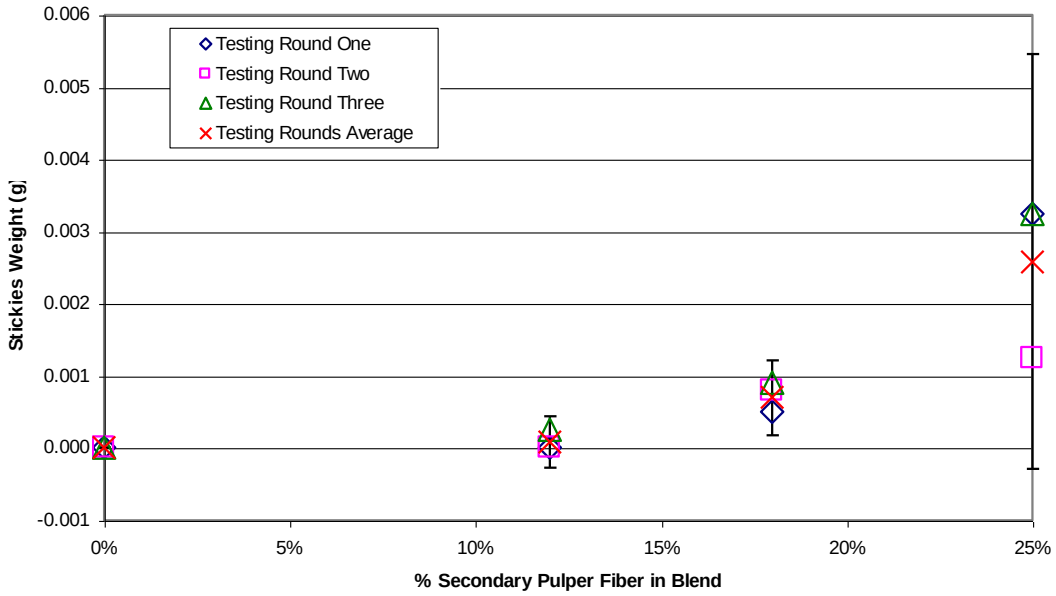

Figure 18. Stickies weight as measured by the Deposition Test Method. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set E)

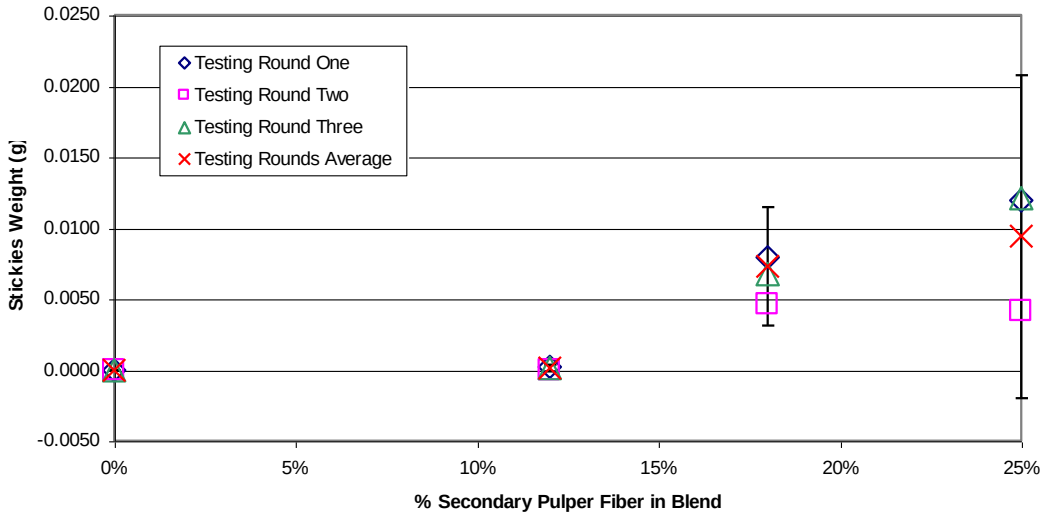

Figure 19. Stickies weight as measured by the Polyethylene Bottle Method. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set E)

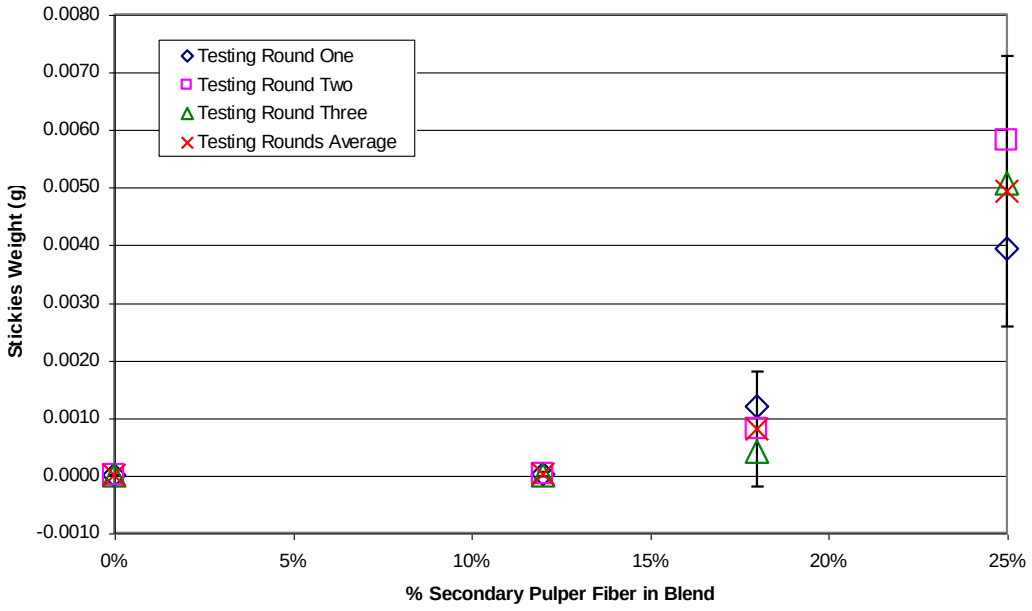

Figure 20. Stickies weight as measured by the Polyethylene Film Method. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set E)

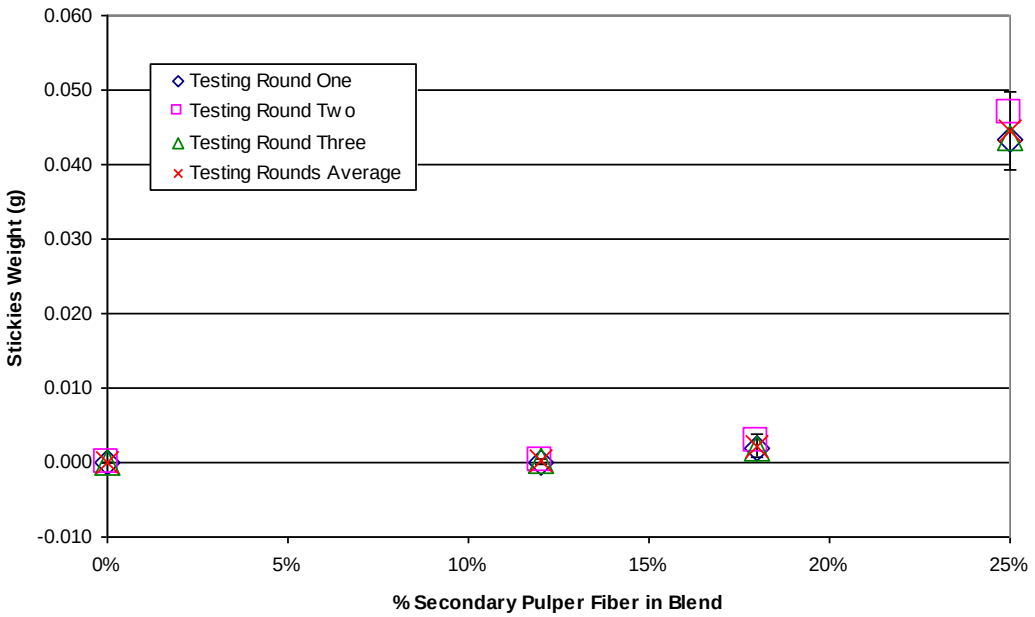

Figure 21. Stickies weight as measured by Solvent Extraction. Upper and lower limits of the 95% Confidence Interval are indicated. (Sample Set E)