

Evaluation of Sticky Contaminant Analysis Methods

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Macro-Adhesive Contaminant Analysis

Scope of Work

- Goal: determine the accuracy and precision of different stickies detection methods *for OCC* pulps
- Indicate the advantages and limitations of each test method for the different types of stickies

Macro-stickies Detection Methods Studied

- **Bleaching and dyeing**
 - Bleach with Chlorite and make Handsheets
 - Perform Image Analysis
 - Dye with Morplas Blue
 - Perform Image Analysis
- **Deposition:**
 - Low Consistency pulp slurry exposed to counter rotating paper machine wires
 - Determine Gravimetrically the Deposits
 - Dye deposits and perform Image analysis

Macro-stickies Detection Methods

- **Tappi Test Method T-277:**
 - Screen pulp with Pulmac Masterscreen (0.006 inch slots)
 - Collect rejects on black filter paper
 - Press against white coated paper : adhesives pick-up white coating
 - Use black marker to darken brown fiber
 - Image Analysis



Macro-stickies Detection Methods

- **Port Townsend Method 1: Handsheets (Steve Nordwell)**
 - Make Handsheets
 - Dye with black ink, stickies do not pick-up ink
 - Perform Image Analysis
- **Port Townsend Method 2: Screening**
 - Screen pulp with Pulmac Masterscreen (0.006 inch slots)
 - Dye rejects with black ink, stickies do not pick-up ink
 - Perform Image Analysis

Effort Required for Test Methods

Macro-stickies Test Methods:	Effort Needed:
– Bleaching and Dyeing Method	4 hrs
– Deposition Testing	
• gravimetric	2 hrs
• image analysis	3 hrs
– Tappi T277 pm-99	1.5 hrs
– Port Townsend 1: Handsheets	1.5 hrs
– Port Townsend 2: Screening	2 hrs

Summary of Macro-Stickies Tests

Weyerhaeuser Mill Samples			
	Avg. 95%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
Inland Mill Samples			
Bleaching and Dyeing	1720	100	1710
PT Method 1	1940	3830	50
PT Method 2	5100	21000	24
T277	21930	128330	17

OTHER MACROSTICKIES QUANTIFICATION

- Fluorescent Speck Counting Method
- Sulzer Escher Wyss Method
- Southeast Paper Method
- Dye Method

FLUORESCENT SPECK COUNTING

- Screen 100 g stock through 6-cut slotted screen.
 - Collect rejects & discard accepts.
 - Blend rejects with bleached virgin fibers.
 - Make handsheet(s).
- Sandwich handsheet between clean filter papers.
 - Hot press at 300⁰F to 350⁰F.
 - Cool under pressure.
 - Separate filter papers from handsheet.
- Examine filter papers under UV light.
 - Count and estimate area of fluorescent specks

SULZER ESCHER WYSS METHOD

- Screen 100 g stock through 6-cut slotted screen.
 - Collect rejects on a filter paper.
 - Discard accepts.
- Place filter paper on a black board.
 - Dry at 200⁰F and hot press at 265⁰F.
- Brush off fibers from the black board.
 - Count and estimate speck area.

SOUTHEAST PAPER METHOD

- Screen 100 g stock through 6-cut slotted screen.
 - Dry and weigh rejects; discard accepts.
 - Blend rejects with bleached virgin fibers.
 - Make handsheet(s).
- Examine handsheet under microscope.
 - Probe specks with a needle.
 - Count and estimate area of specks that feel sticky.

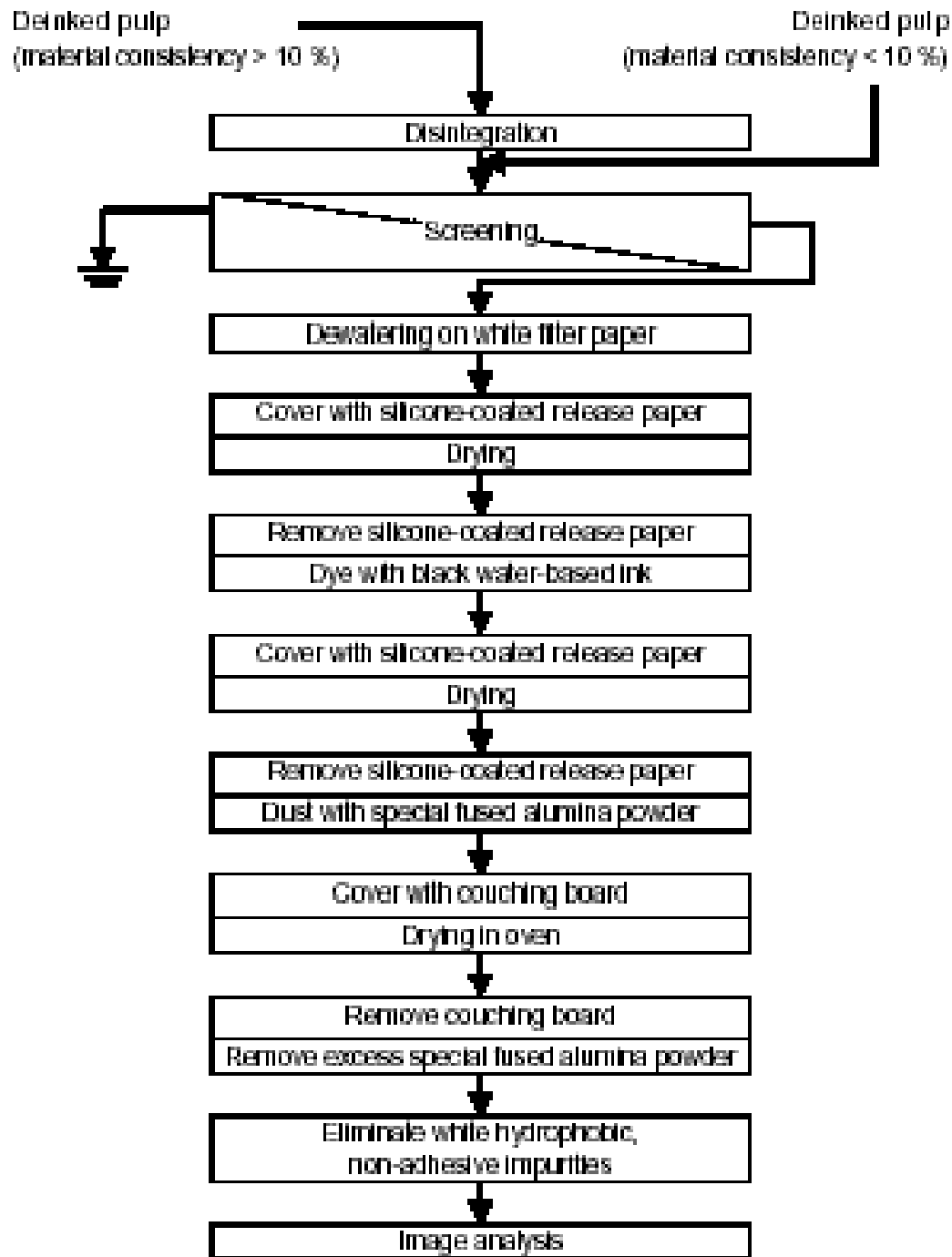
Manual METHOD

- Screen 100 g stock through 6-cut slotted screen (or other level but note + consist)
- Collect rejects on filter paper
- Dry and press against clean sheet
- Manually probe under microscope with pin all spots
- Count sticky spots/100g pulp

Stickies Quantification INGEDE Method

www.ingede.com, Method 4

**“Analysis of Macrostickies in
Deinked Pulp (DIP)”**



INGEDE METHOD

Evaluation of **Micro-Adhesive Contaminant Analysis Methods for the Use in OCC Recycling Plants**

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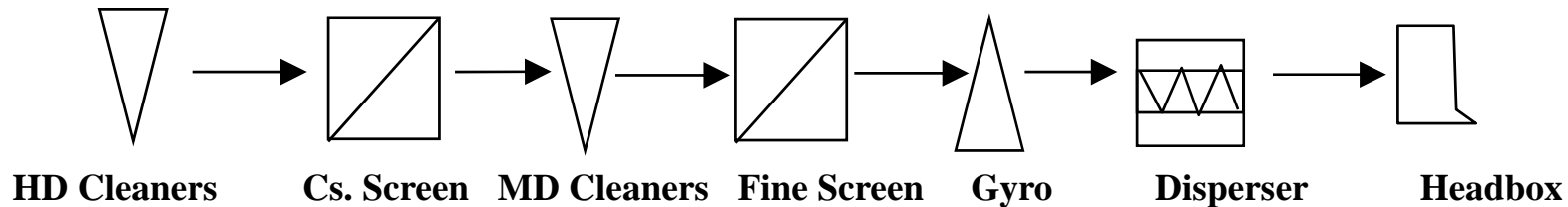
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Micro-stickies Research Methods (Materials):

- OCC pulp samples from various mill locations (see below)
Maysville, KY
- Effort made to use bales from one single broker (only commercially collected OCC)
- Re-routing of pulp around high density storage tank directly to machine
- composite samples obtained at 20 min. intervals for 3 hours



Micro-stickies Research Methods (Tests):

- Bleaching and dyeing of handsheets
- Deposition: papermachine wire
- Deposition: polyethylene bottle
- Deposition: polyethylene film
- Solvent Extraction
- Tappi Method for Process Water (Perform screening stickies test PTM2 on refrigerated and unrefrigerated samples)

Micro-stickies Research Methods (Evaluation):

- Perform each test on each pulp sample in triplicate
- What is the reproducibility of the test result based on the triplicate tests at each sample point?
- Does the test reflect a general decrease in expected stickies content through the recycling process?
- Does the test have a usable range of response values relative to the reproducibility of the test?

Paper Machine Wire Deposition Method (Pira Modified)



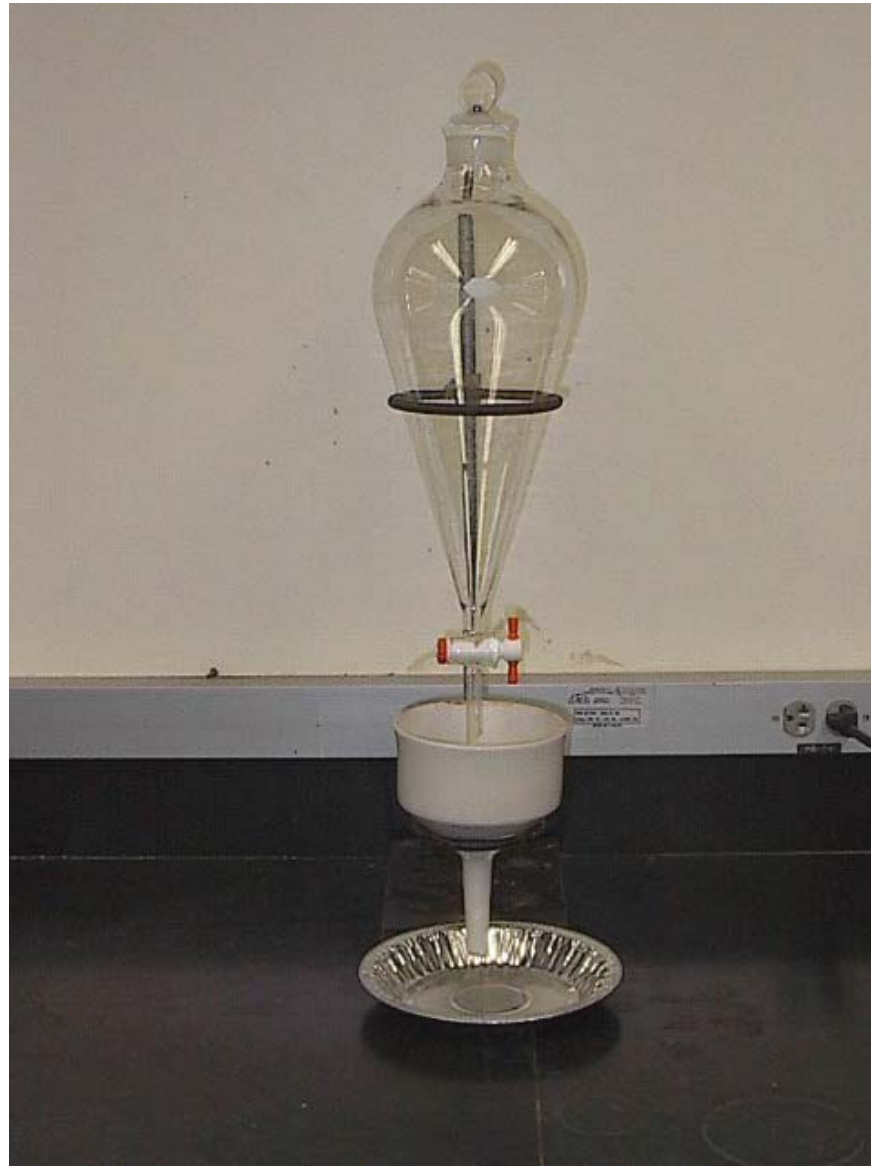
PE Bottle Deposition Method



PE Film Deposition Method



Solvent Extraction



Pulmac Master Screen



Bleaching and Dyeing

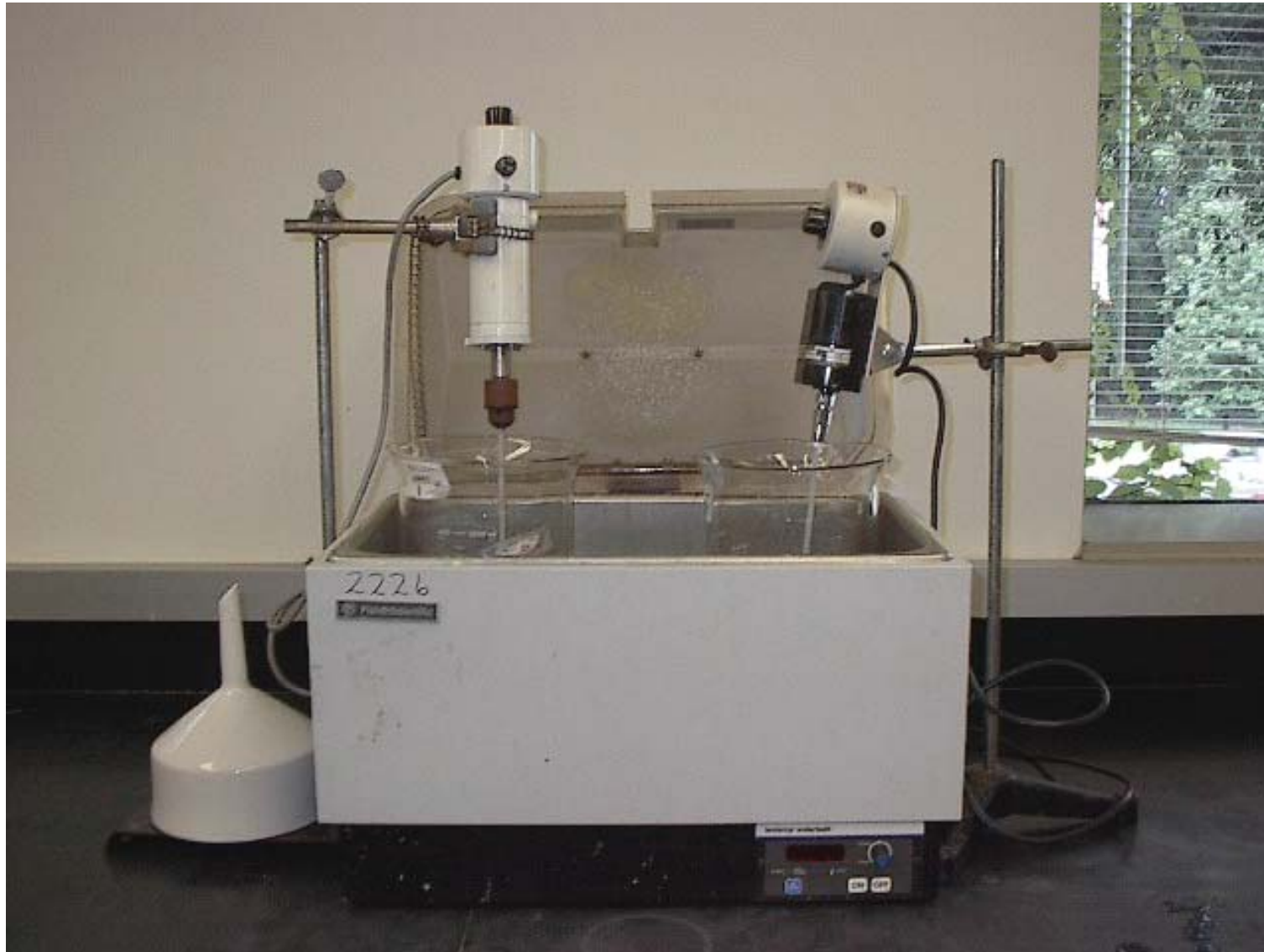


Figure 1. Stickies weight as measured by the Deposition Test Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated.

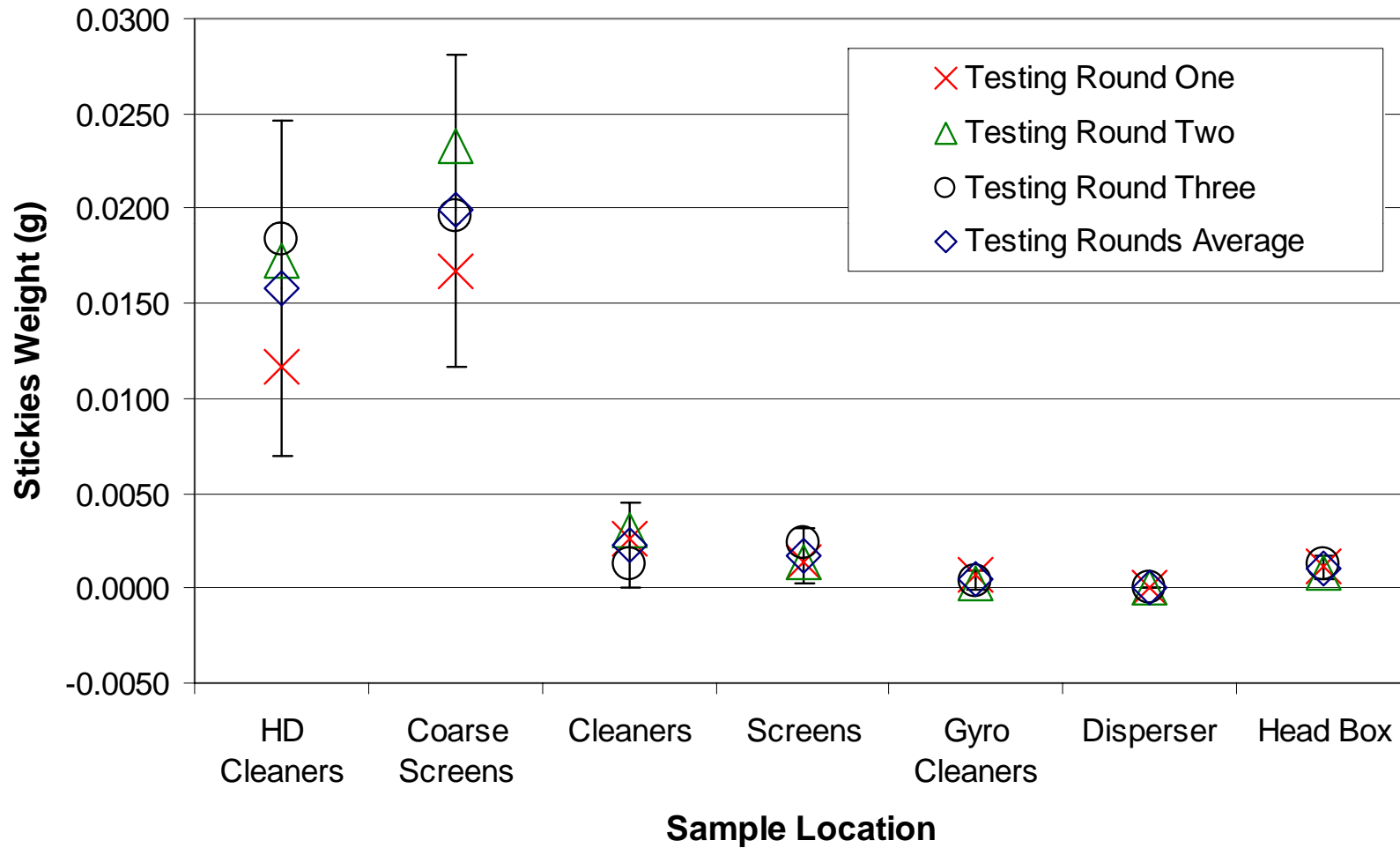


Figure 2. Stickies weight as measured by Polyethylene Bottle Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated.

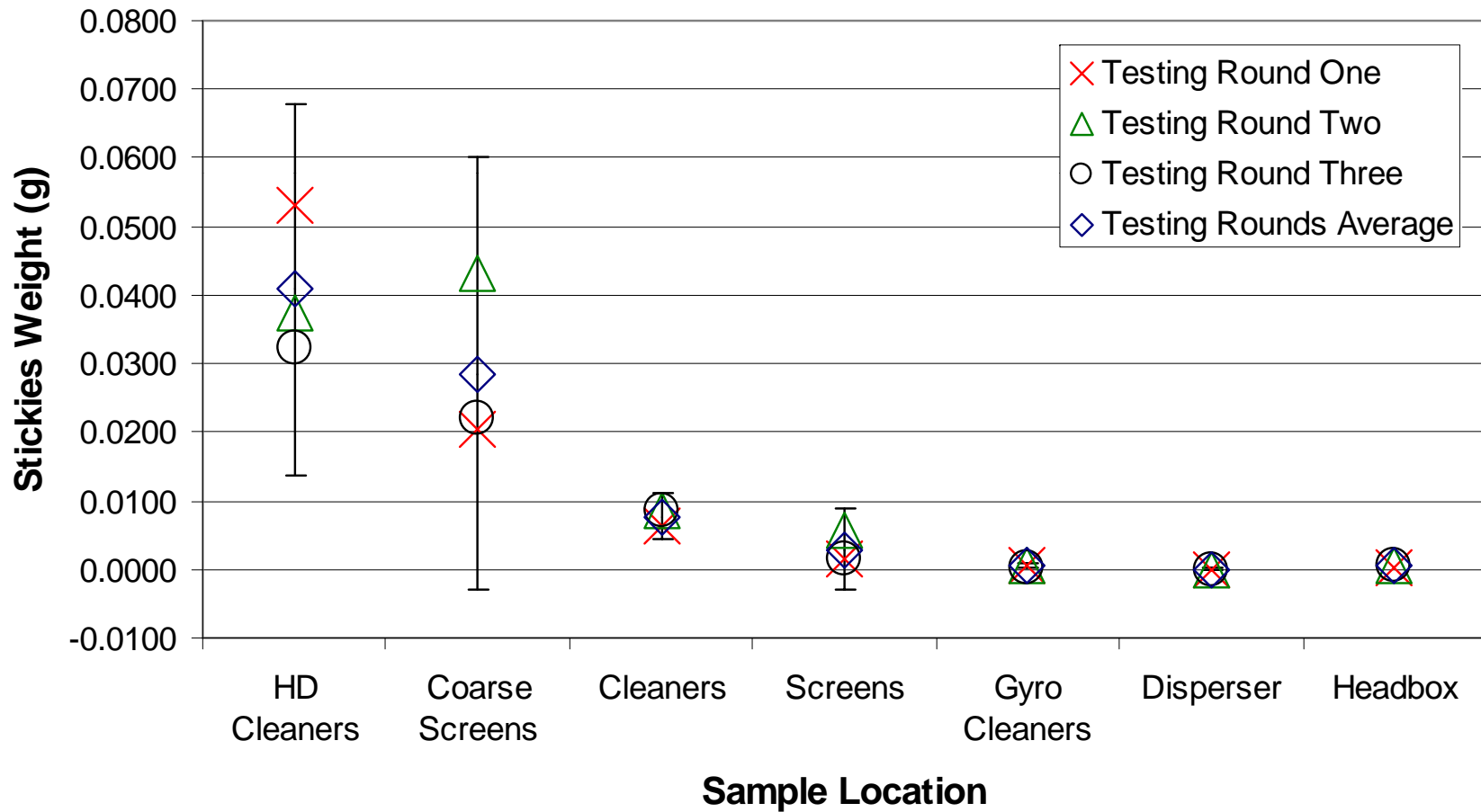


Figure 3. Stickies weight as measured by Polyethylene Film Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated.

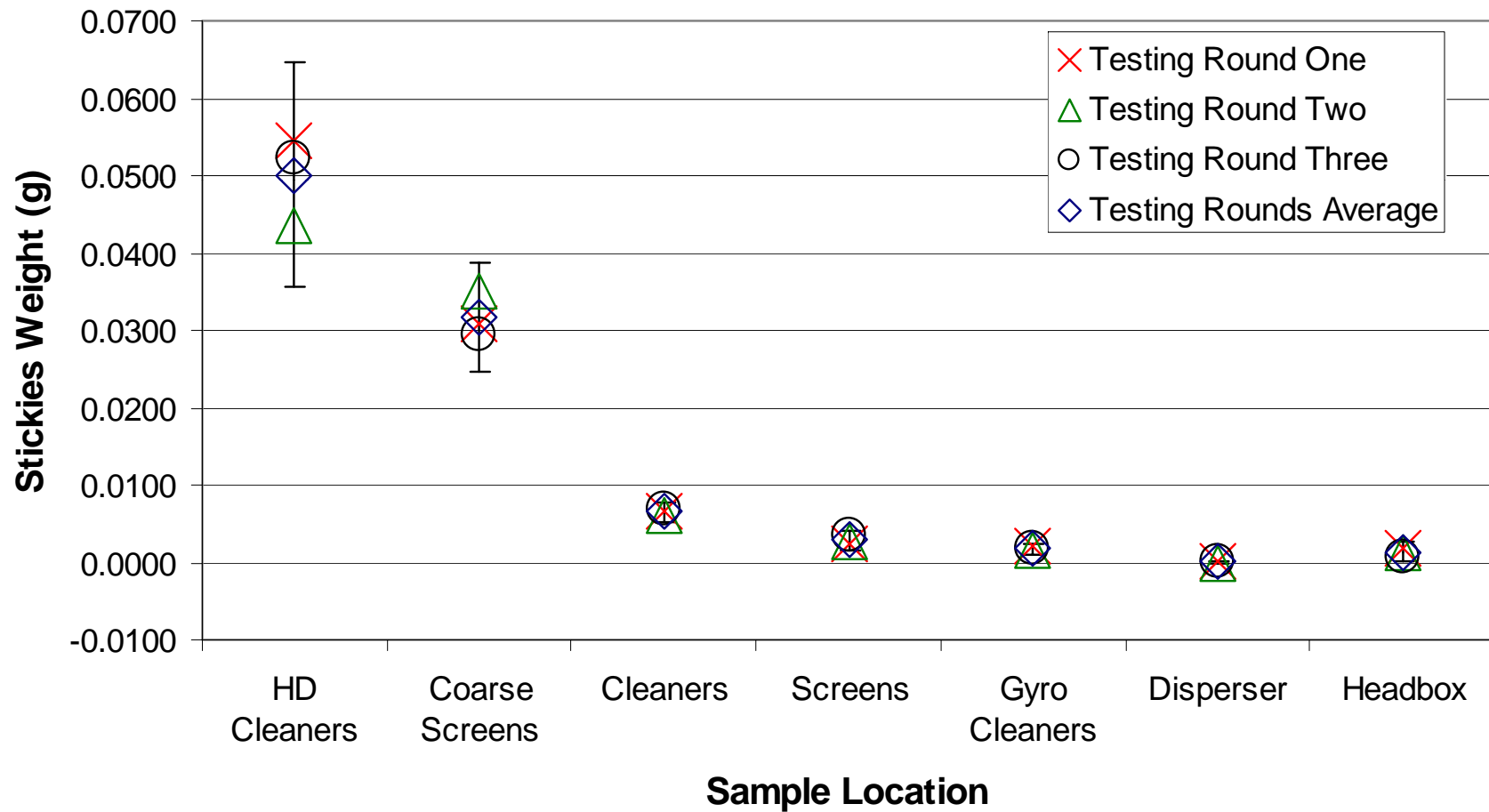


Figure 5. Stickies weight as measured by Solvent Extraction on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated.

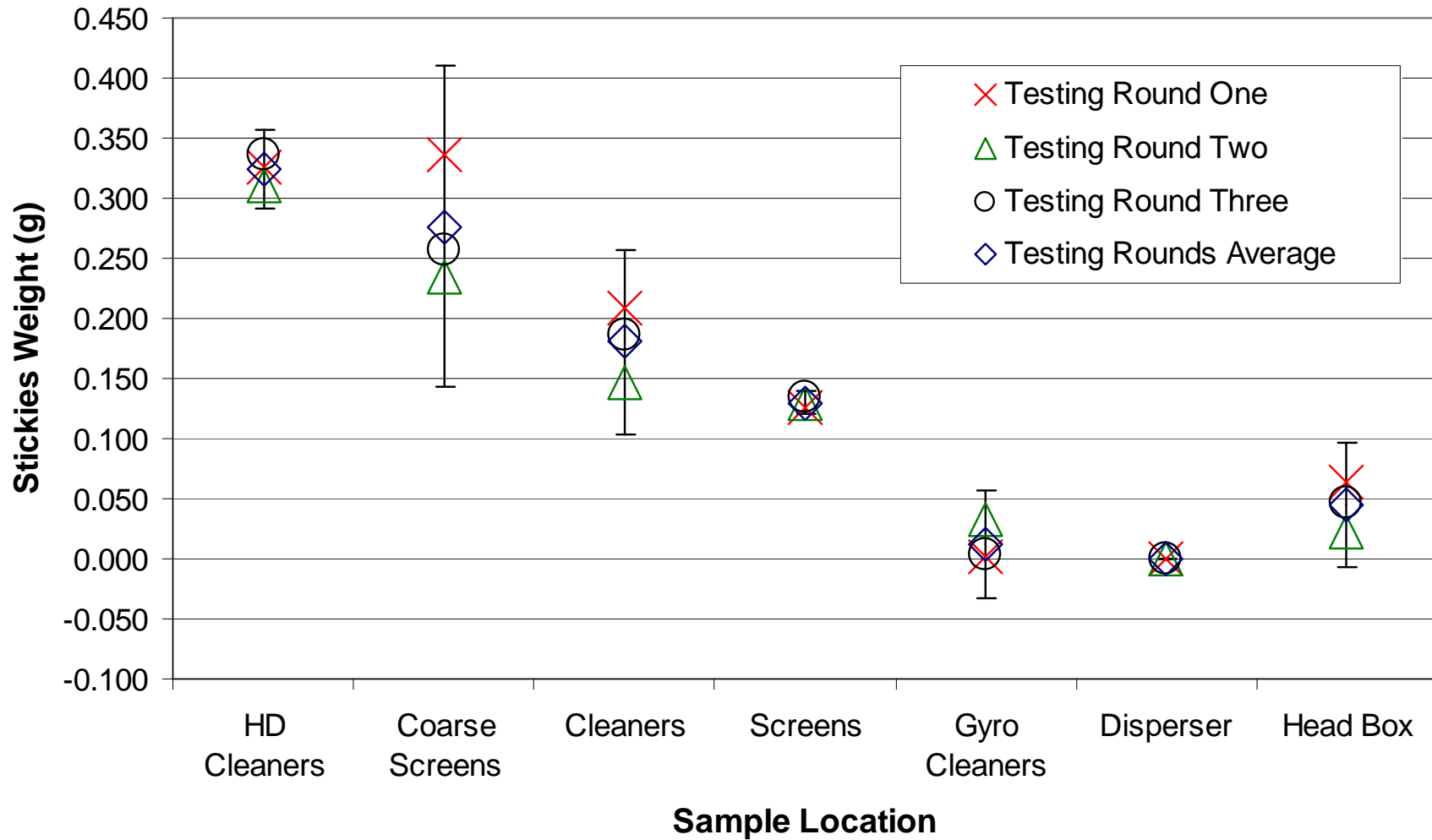


Figure 4. Stickies PPM as measured by Tappi Method for Micro-stickies in Process Water on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated.

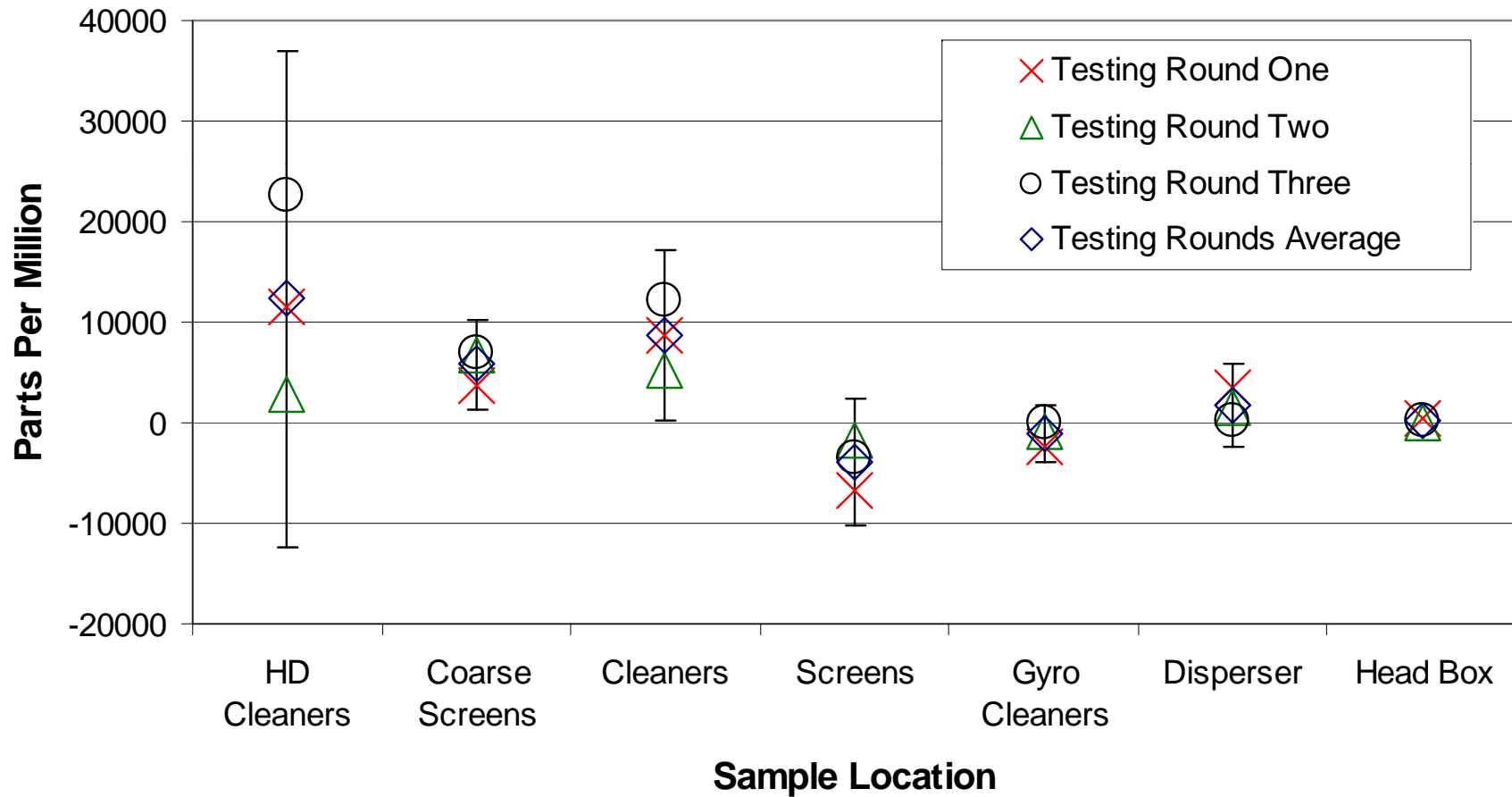
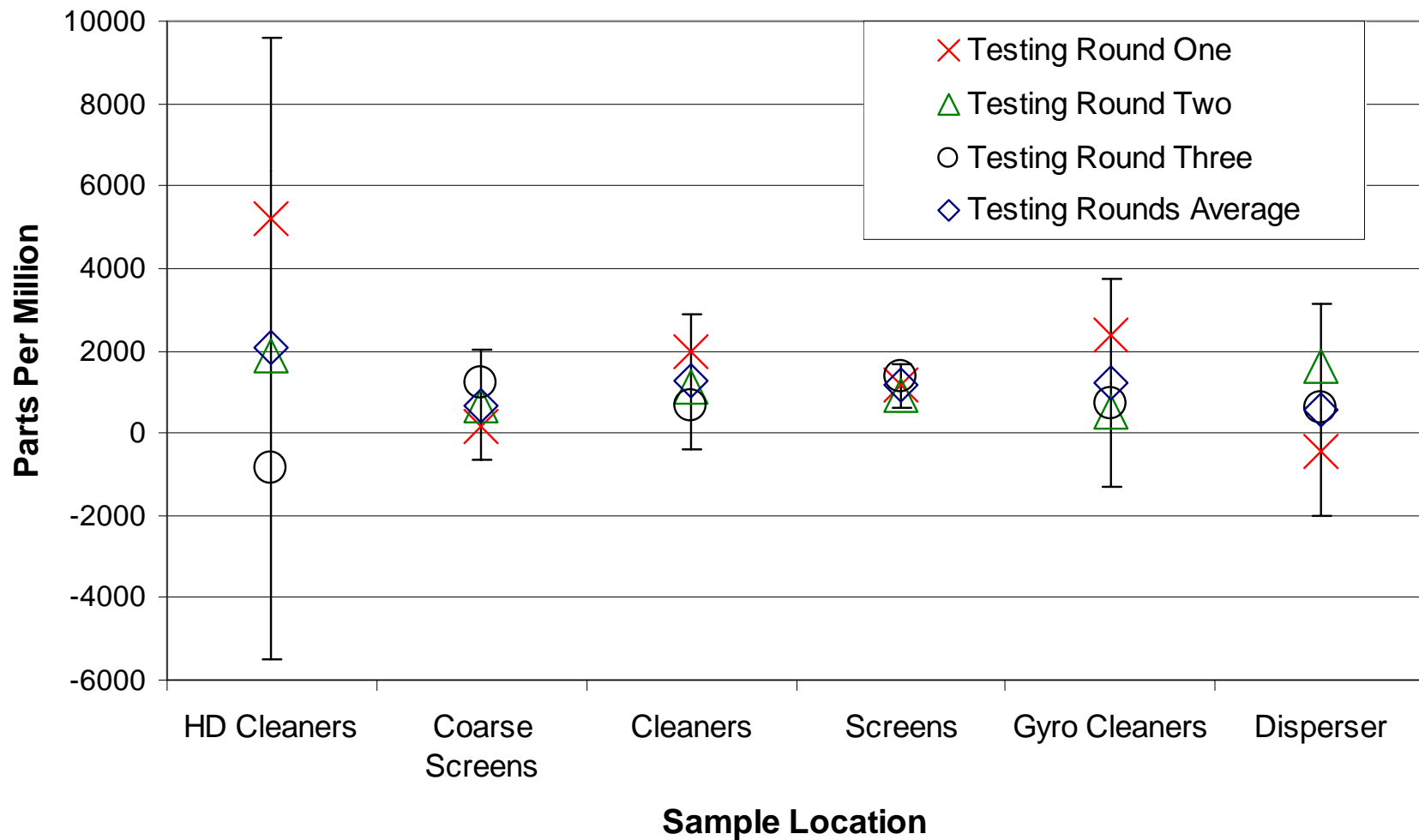


Figure 6: Stickies PPM as measured by the Bleaching and Dyeing Method on composite samples. Upper and lower limits of the 95% Confidence Interval are indicated.



Evaluation of Micro-stickies Test Methods Using Confidence Intervals:

- Calculated the confidence interval for each test method/location
 - calculated the average confidence interval for each test method
- Determined the span of measurements for each test method:
 - Span = avg. stickies content after *coarse screen* minus after disperser
- Best stickies test method should have:
 - small confidence intervals
 - large span (useful range of measurement values across an entire process)
 - Smallest value of:

$$100\% * \frac{\overline{CI}}{SPAN}$$

Summary of Micro-Stickies Tests

	Avg. 95% 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
Bleaching and Dyeing	1720	100	1710

Summary of Micro-Stickies Test Results

- Deposition tests are reproducible, however the absolute quantities detected are small and near detection limits
- Tappi Method (Refrigeration/Screening) not useful for OCC pulp samples, complex
- Solvent Extraction: Reproducible and adequate range of response, use of organic solvent
- Bleaching and Dyeing Method not adequate, complex to perform, not a microstickies test

OTHER MICROSTICKIES QUANTIFICATION

- **Pulmac master screen with modification**
- **EMMA = Effective Measurement of Microstickies Accumulation, IPST/EKA**

Microstickies Measurement

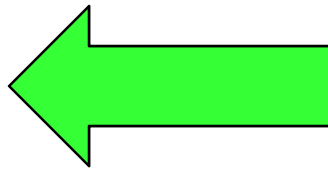
Accelerated Agglomeration Method



Pulmac Master Screen

2 Filter Papers

Macro/Micro



**Concentrator
Mesh Screen
Separator**

Agglomeration:

No chemicals

**Simulates
agglomeration
conditions on PM**

Micro-Stickies Classifier

OTHER MICROSTICKIES QUANTIFICATION

- **IPST/EKA Method**
- **EMMA = Effective Measurement of Microstickies Accumulation**
- **(Measures Total Organic Carbon or TOC) between 25 micron and 5000 Dalton**

EMMA

**TOC of material between 25 micron
and 5000 Dalton**

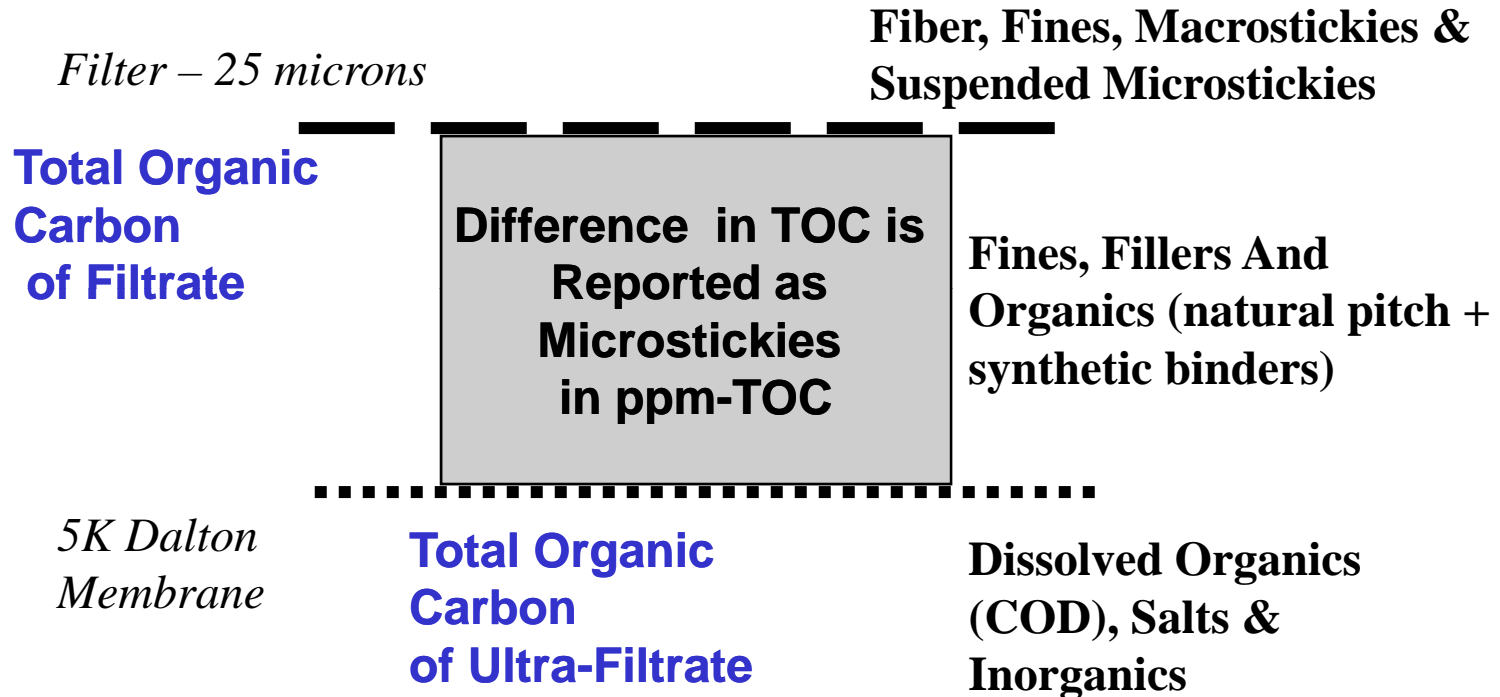
EQUALS

**(TOC in filtrate through 25 μm
Whatman filter)**

MINUS

**(TOC in filtrate through 5000 Dalton
ultrafiltration membrane)**

Microstickies by Difference in TOC after Fractionation



EMMA*	=	Dispersed, Colloidal & Dissolved Microstickie	=	Total Organic Carbon of Filtrate	-	Total Organic Carbon of Ultra-Filtrate
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*EMMA = Effective Measurement of Microstickies Accumulation