SLIDE MATERIAL

to the

RECYCLE

PROJECT ADVISORY COMMITTEE

March 6-7, 2000
INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

Atlanta, Georgia

SLIDE MATERIAL

RECYCLE

PROJECT ADVISORY COMMITTEE

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FLOTATION DEINKING FLUID MECHANICS

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FOR

PROJECT F00903

March 6-7, 2000
Institute of Paper Science and Technology
Atlanta, Georgia
FLOTATION DEINKING FLUID MECHANICS

Project F00903
March 6, 2000

Project Budget and Participants

- $114K for FY99-00
- Ted Heindel, Assistant Professor of Engineering, IPST (0.2 man-years)
- Adele Garner, Assistant Engineer, IPST (0.5 man-years)
- Akilu Giorges, Assistant Scientist/Post-Doc, IPST (0.25 man-years)
- Fred Bloom, Professor of Applied Mathematics, Northern Illinois University (0.1 man-years)
Other Projects

- "Bubble Size Control to Improve Oxygen-Based Bleaching" with Tom McDonough, DOE Agenda 2020 project, ~$250K/year for three years.
- "Flow Pattern Identification in Fiber Suspensions Using Neural Networks" with Mostafa Ghiaasiaan, Georgia Tech., IPST/GIT Seed Grant, $38K (IPST), $38K (GIT).
- "Characterization and Removal of Stickie Contaminants from OCC Recycle Mills" with Kevin Hodgson, University of Washington, CG project, $42K (IPST), $50K (UW).
- "Approach Flow Systems", IPST DFRC project, $75K.

Student Projects

- "Flow Regime Mapping in Vertical Gas/Liquid/Fiber Flows," Kelly Ronk, 1st Year IPST MS Student
- "Flow Regime Mapping in Horizontal Gas/Liquid/Fiber Flows," Mark Snyder, 1st Year IPST MS Student
Presentation Outline

- Objectives
- Bubble Size Measurements
- Flotation Modeling
- Conclusions

Flotation Objectives

The objective of this project is to increase flotation efficiency by maximizing contaminant removal from wastepaper while minimizing fiber loss.
IPST Research Line #13

Reduce and/or control contaminants (e.g., stickies, dyes, toners) in recycled fiber pulp using break-through technologies to allow the interchange of recycled pulp with virgin pulp of similar fiber make up at an economical cost.

Flotation Deinking Fluid Mechanics

- Flotation Modeling
  - Develop Mathematical Model
    - Lab Scale Model Validation
    - Mill Scale Validation
    - Predict Flotation Performance
  - Improve Current Flotation Cells
- Flotation Deinking Fluid Mechanics
- Bubble Size Measurement
  - Develop Methods to Measure Bubble Size in Fiber Suspensions
  - Identify Factors That Affect Bubble Size in Fiber Suspensions
- Equipment and/or Process Changes
  - Develop New Flotation Cells
  - Develop Bubble Size Control Strategies

IPST Confidential Information - Not for Public Disclosure
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Presentation Outline

- Objectives
- Bubble Size Measurements
  - FXR Update
  - Bubble Size Control Options
  - Summary
- Flotation Modeling
- Conclusions

Bubble Size Measurements

- Objective:
  - To determine bubble size distributions in a pulp suspension and methods for bubble size control
- Benefits to industry:
  - Improve bubble size control for more effective flotation deinking
  - Experimental techniques can be used in other areas
    - Bleaching with gaseous chemicals
    - Coating deaerator effectiveness
Bubble Size Measurement Goals for April 99 - March 2000

- Verify lognormal distributions for one additional superficial liquid velocity.
  - Status: Completed

- Verify lognormal distributions for one additional fiber type (i.e., ONP).
  - Status: Completed

- Investigate methods to reduce and/or eliminate the formation of large bubbles, especially at “high” consistencies.
  - Status: Completed

- Summarize findings in a Member Company Report.
  - Status: In Progress

FXR Update

- Previous cocurrent studies used copy paper ($C = 0, 0.5, 1, 1.5\%$), superficial gas velocities ($v_g$) of 1, 2, 4 cm/s, and a superficial liquid velocity ($v_L$) of 2 cm/s

- This past year obtained FXR images of
  - copy paper ($C = 1\%$), $v_g = 1, 2, 4$ cm/s, and $v_L = 1$ cm/s
  - ONP ($C = 1\%$), $v_g = 1, 2, 4$ cm/s, $v_L = 2$ cm/s

- Details will be reported in Project F00903 Member Company Report 10
  - In preparation
Effect of Superficial Liquid Velocity ($u_l$) on the Air/Water Small Bubble Size Distribution

- $H = 115-140$ cm
- $u_l = 1$ or $2$ cm/s
- $u_g = 1, 2,$ or $4$ cm/s
- No effect of $u_l$ on small bubble size
- Similar effects at $H = 15-40$ cm

Effect of Superficial Liquid Velocity ($u_l$) on the Air/Water/1% Copy Paper Small Bubble Size Distribution

- $H = 115-140$ cm
- $u_l = 1$ or $2$ cm/s
- $u_g = 1, 2,$ or $4$ cm/s
- Negligible effect of $u_l$ on small bubble size
- At $H = 15-40$ cm
  - Slight effect
  - Due to poor mixing in lower column region at $u_l = 1$ cm/s
Effect of Fiber Type on the Small Bubble Size Distribution
($v_g = 4 \text{ cm/s}, u_e = 2 \text{ cm/s}$)

- At $H = 15-40 \text{ cm}$
  - Negligible differences
- At $H = 115-140 \text{ cm}$
  - Air/water produces largest bubbles
  - Due to bubble coalescence
  - ONP produces smallest bubbles
  - Residual chemicals in ONP system and significant mixing reduce bubble size
- Negligible fiber type trends at lower superficial gas velocities
  - Only occurs when mixing is greatest

Methods for Bubble Size Modification

- Various chemical and physical parameters affect bubble size
  - System pressure
  - Suspending medium (i.e., fluid)
  - System chemistry
  - Gas injector design
  - Gas injection flow rate
  - Local shear rate (i.e., turbulent intensity)
Best Option for Bubble Size Control

- Use staggered tube bundles
- Diameter should be at least twice as large as fiber length to prevent stapling (e.g., D > 1 cm)
- Spacing will affect pumping power and flow conditions
  - too close will increase pumping power
  - too loose will not significantly affect bubble size

Bubble Size Measurement Summary

- Additional FXR images have been acquired
  - Effect of superficial liquid velocity on the small bubble size is negligible
  - Effect of fiber type on small bubble size is negligible except at the highest gas velocities studied
- Bubble size modification options have been reviewed
  - Best option for bubble size modifications is a staggered tube bundle
- Details of this work will be presented in F00903 Member Company Report 10
  - In preparation
Bubble Size Measurement Goals for April 2000 to March 2001

- Complete Member Company Report 10
- Design and construct a modified flow chamber for our cocurrent bubble column
  - Use staggered tube bundles
- Obtain FXR images in modified bubble column
- Member Company Report on FXR results

Presentation Outline

- Objectives
- Bubble Size Measurements
- Flotation Modeling
  - Validation Experiments
  - Updated Flotation Model
  - Summary
- Conclusions
Flotation Modeling

- **Objective:**
  - To determine factors that affect bubble/particle interactions.

- **Benefits to industry:**
  - Predict effects of process changes before expensive system trials.
  - Predict changes to improve current flotation cell performance.
  - Predict performance of new flotation cell designs.

Model Validation Goals for April 99 - March 2000

- Summarize current status in a Member Company Report.
  - Status: Completed (Report 9 issued in August 1999)

- Extend the flotation model to include more than one particle per bubble.
  - Status: Completed

- Complete the lab scale model validation.
  - Status: Completed

- Summarize these results in a Member Company Report.
  - Status: In Progress

- Initiate mill scale model validation flotation trials.
Current Model Validation Experiments

- Used Wemco flotation cell
- Used new image analysis system
- Ran flotation trial for various flotation times
  - 30 sec, 60 sec, 90 sec, 2 min, 3 min, 4 min, 5 min, 8 min, 10 min

Wemco Flotation Cell

- Standard laboratory flotation cell
- Operates in batch mode
- Variable speed rotor
  - 300-1500 RPM
- Air injection can be controlled
New Image Analysis System

- **Software:**
  - Image Pro Plus v.4

- **Hardware:**
  - Pentium II 450 MHz computer w/ 19” color monitor
  - High resolution digital camera
  - Autoscanning stage with 8” of travel
  - 8” x 8” backlighting system

Image Analysis Capabilities

- Pixel resolution with current 35 mm lens is 7.1 μm/pixel
- Autoscanning stage and digital camera capture ~83% of entire handsheet area for analysis
- Particles identified with aspect ratio filter
  - aspect ratio must be between 1 and 3
- Minimum particle size set at 21 μm equivalent diameter
Repulping Conditions

- Fused laser printed image on cellophane
  - immerse in water and fused toner falls off
- Add fused toner to unprinted copy paper
- Repulp in British disintegrator
  - pH: neutral
  - chemicals: none
  - temperature: ambient
- Slurry contains "non-hairy" toner particles
  over a wide particle size distribution

Experimental Conditions

- Furnish: Fused toner particles added to unprinted copy paper
- Surfactant: Triton X-100
- Surfactant concentration: 20 mg TX-100/(g o.d. fiber)
- Initial stock volume: 3 liters
- Sample size at each time interval: 30 ml
- Temperature: ambient
- Air injection rate: 3 liters/min
- Impeller speed: 900 RPM

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Experimental Procedures

1. Add surfactant to 3L of stock in Wemco
2. Mix for 1 min w/o air flow
3. Turn air flow on for 30 sec
4. Remove foam
5. Stop mixing and air flow
6. Take 30 ml sample
7. Turn mixer and air flow back on for additional time period
8. Repeat 4 – 8 until floated for 10 min

Data Analysis Procedures

- Make handsheet with 30 ml sample in a *clean* British Handsheet mold with filter paper over the wire
- Put dried handsheet on autoscanning stage and capture images (~83% of entire handsheet)
- Neglect “particles” with aspect ratio larger than 3 and equivalent diameters less than 21 μm
- Subtract “correction” factors for each size range
  - average particle count from 5 handsheets made following identical procedures w/o added toner particles
- Obtain removal efficiency
Wemco Particle Concentration

- Feed has wide particle size distribution
- Particle concentration decreases with increasing flotation time

Wemco Removal Efficiency vs. Particle Size

Removal Efficiency =
Feed Conc. - Accept Conc.
Feed Conc.

- Correction makes small changes only for small particle sizes
- Small particles initially better removed
- After 10 minutes (hyperfloation) 100-300 \(\mu\)m particles best removed

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Average Removal Efficiency vs. Flotation Time

- Selected particle size ranges
- Efficiency increases with increasing flotation time
- Differences between particle size ranges are negligible

New Modeling Effort

- Have formulated a coupled set of equations
- The first describes the number of free particles in the system and includes the possibility of having more than one particle per bubble
- The second describes the number of particles leaving the system
- Detailed equations are in the status report
  - Will also be included in Member Company Report 11
Parameter Values Used in the Initial Model Calculations

- \( R_p = 75 \mu m \)
- \( \rho_p = 1.2 \text{ g/cm}^3 \)
- \( R_b = 0.75 \text{ mm} \)
- \( \rho_l = 1 \text{ g/cm}^3 \)
- \( \mu_l = 1 \text{ cP} \)

- \( \eta_b = 12 \text{ cm/s} \)
- \( \sigma = 35 \text{ dynes/cm} \)
- \( \varepsilon = 6.7 \text{ W/kg} \)

- \( n_{p0} = 1.41 \times 10^7 \text{ particles/} \) (m\(^3\) of 1% stock)
- \( n_B = 6.28 \times 10^7 \text{ bubbles/} \) (m\(^3\) of 1% stock)

Effect of Stability Parameter “A” on the Predicted Flotation Efficiency with \( d_p = 125-150 \mu m \)

- Predictions very sensitive to \( A \)
  - when \( A = 1, \) \( P_{slag} \approx 1 \)
- \( A = 0.5 \) matches experimental data very well

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Effect of Stability Parameter "A" on the Predicted Flotation Efficiency with \( d_p = 100-125 \ \mu m \)

- Only changed \( d_p = 125 \ \mu m \)
- With \( A = 0.5 \), model over predicts the data

Effect of Stability Parameter "A" on the Predicted Flotation Efficiency with \( d_p = 150-175 \ \mu m \)

- Only changed \( d_p = 175 \ \mu m \)
- With \( A = 0.5 \), model under predicts the data
- Conclusion - the stability parameter A could be a function of particle size
Effect of the Average Number of Particles on a Bubble (\( \bar{X} \)) on the Predicted Flotation Efficiency

- Only changed average number of particles per bubble
- Removal efficiency increases with increasing number of particles on a bubble
  - approaches a constant value as the number of particles per bubble increases

Effect of Surface Tension (\( \sigma \)) on the Predicted Flotation Efficiency

- Only changed surface tension
  - affects \( P_{\text{stab}} \)
  - experimental values varied between 32-42 dynes/cm
- Efficiency increases with increasing surface tension
- Change not very significant over experimental range
Effect of Turbulent Energy Density ($\varepsilon$) on the Predicted Flotation Efficiency

- Only changed turbulent energy density
  - affects collision rates and stability
- Increasing $\varepsilon$ decreases flotation efficiency
- Turbulent energy density has a significant influence on the predictions

Effect of Contact Angle ($\theta$) on the Predicted Flotation Efficiency

- Only changed contact angle
  - affects $P_{stab}$
- Higher contact angles (more hydrophobic) produce higher removal efficiencies
- Typically a range of $\theta$ in flotation cells
- A good estimate of $\theta$ is needed for good predictions
Effect of $h_0/h_{crit}$ on the Predicted Flotation Efficiency

- Only changed $h_0/h_{crit}$ affects $P_{adv}$
- Increasing $h_0/h_{crit}$ decreases removal efficiency considerably
- Accurate value of $h_0/h_{crit}$ is needed

Improved Flotation Model Summary

- The new model predicts the correct trends in the experimental data
- Model is sensitive to accurate estimates of selected parameters
  - stability parameter “A”
  - turbulent energy density, $E$
  - contact angle, $\theta$
  - initial-to-critical film thickness, $h_0/h_{crit}$
- Report on model validation is currently being prepared
- Currently trying to get a better handle on “A” and $h_0/h_{crit}$

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Field Trial Planning

- A mill has not yet been identified
- Need accurate measurements or estimates of
  - particle size, concentration, and density
  - bubble size, concentration, and rise velocity
  - surface tension
  - contact angle
  - turbulent energy density
  - gas holdup
  - average number of particles on a bubble
- Also need $h_0/h_{crit}$ and "A" functional relationships

Flotation Modeling Goals for April 2000 to March 2001

- Determine theoretical values of $h_0/h_{crit}$ ($P_{ast}$) and "A" ($P_{stab}$) in terms of fundamental parameters (i.e., $R_p$, $R_B$, etc.)
  - Member Company Report on these parameters
- Identification of methods and techniques needed for mill-scale model validation
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Conclusions

- Bubble Size Measurements
  - Effect of superficial liquid velocity on the small bubble size is negligible
  - Effect of fiber type on small bubble size is negligible except at the highest gas velocities studied
  - Best option for bubble size modifications is a staggered tube bundle

- Flotation Modeling
  - The new model predicts the correct trends in the experimental data
  - Model is sensitive to accurate estimates of selected parameters
Technology Transfer Opportunities

- Gas flow visualization method can be used in other areas where gas flow information is needed
  - gas injector design and operation
  - gas mixer design and operation
  - deaerator design and operation
- Bubble size control options have been suggested
ONLINE STICKIES SENSOR

SLIDE MATERIAL

FOR

PROJECT F042

March 6-7, 2000
Institute of Paper Science and Technology
Atlanta, Georgia
Online stickies sensor

March 2000

Suresh Shrauti
Sujit Banerjee

Started: July 1999 ($73K, 0.7 FTE)

- Establish first-cut feasibility of the individual steps (done).
- Design unit (done).
- Build & lab test device with PSA (June '00).
  - added task: test with mill stickies (underway).
- Field work (FY01).
- Refine (other stickies, mixtures).
Approach

- Screen out stickies from fiber.
- Homogenize stickies with a Polytron homogenizer with a non-carbon (silicate) surfactant.
- Analyze with a TOC analyzer.

Size distribution of homogenized PSA

<table>
<thead>
<tr>
<th>Size</th>
<th>Coarse (15 sec)</th>
<th>Fine (2 min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;100μ:</td>
<td>65%</td>
<td>96%</td>
</tr>
<tr>
<td>100-200μ:</td>
<td>27%</td>
<td>4%</td>
</tr>
<tr>
<td>&gt; 200μ:</td>
<td>8%</td>
<td>0%</td>
</tr>
</tbody>
</table>

Note that most stickies are <100μ
Screening efficiency

- Acrylate PSA was cured, & coarse-homogenized in 100 mL with silicate.
- PSA in Pulmac streams (0.006”, 150 μ screen) was determined by filtering & weighing.
- Most of the PSA can be screened & recovered. They are mostly in the rejects, suggesting agglomeration.

<table>
<thead>
<tr>
<th>ppm</th>
<th>feed</th>
<th>acc</th>
<th>rej</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,500</td>
<td>93</td>
<td>0.8</td>
<td>86</td>
</tr>
<tr>
<td>5,000</td>
<td>87</td>
<td>1.1</td>
<td>81</td>
</tr>
<tr>
<td>7,500</td>
<td>91</td>
<td>0.9</td>
<td>93</td>
</tr>
<tr>
<td>10,000</td>
<td>98</td>
<td>4.6</td>
<td>94</td>
</tr>
</tbody>
</table>

Homogenizing at 90°C

- PSA in water (100 mL) containing silicate was coarse-homogenized for 15 seconds, fine-homogenized for varying periods, & injected into the TOC analyzer.
- Recovery is too low & is too variable!
- Probable cause: agglomeration.
Strategy: add detackifier

- Tried BRD 2360, Nalco 7520, 7527 T-square (SR-5), colloidal silica (Nalco 8671) & talc.
- Visually, talc & silica gave the best results.
- Based on previous work we settled on a 2% talc suspension; the pH was raised to > 10 to prevent agglomeration.

Results with & without talc

- 800 ppm cured PSA without talc: found 750 ± 640.
- 800 ppm with talc: found 800 ± 350.
- 1,100 ppm with talc: found 1,200 ± 170.

Measurement uncertainty is reduced by talc.
**Mill stickies**

- Composite from screens & cleaners in Westvaco's Tyrone mill.
- Furnish: undeliverable bulk business mail.
- Contained large (>1 mm) agglomerates (ink, shives, stickies).
- Since these would not occur in the fiber stream, the agglomerates were removed, & the remainder mixed with fresh pulp.

**Processing Westvaco rejects**

- Rejects were combined with 1.5 times their weight of bleached SWD pulp & diluted to 1% consistency.
- This was subjected to 1,000 revolutions in a British disintegrator at 140°F, & fed to a Pulmac analyzer. The Pulmac rejects were homogenized & analyzed by TOC.
Work-up of Westvaco stickies

<table>
<thead>
<tr>
<th></th>
<th>feed A</th>
<th>feed B</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH (1%)</td>
<td>0 g</td>
<td>3.45 g</td>
</tr>
<tr>
<td>talc suspension</td>
<td>0 g</td>
<td>11 g</td>
</tr>
<tr>
<td>sodium silicate</td>
<td>0 g</td>
<td>1.35 g</td>
</tr>
</tbody>
</table>

The rejects were suspended in 100 mL of water & (i) analyzed by TOC, and (ii) filtered & weighed; the filter paper was stained & image-analyzed.

Analysis

<table>
<thead>
<tr>
<th></th>
<th>feed A (no chemicals)</th>
<th>feed B</th>
</tr>
</thead>
<tbody>
<tr>
<td>weight</td>
<td>0.1369 g (100%)</td>
<td>0.0132 g (100%)</td>
</tr>
<tr>
<td>TOC</td>
<td>0.048 g (35%)</td>
<td>0.0074 g (56%)</td>
</tr>
<tr>
<td>counts (10-300μ)</td>
<td>2170</td>
<td>330</td>
</tr>
<tr>
<td>counts (10-1000μ)</td>
<td>3660</td>
<td>470</td>
</tr>
</tbody>
</table>

Ink particles could account for the lower TOC recovery. The counts & the TOC correlate well.
Lake Superior stickies

- The tertiary screen rejects were processed in the Pulmac.
- The Pulmac rejects contained very few stickies, but a large amount of shives.
- The stickies were separated through flotation, homogenized, & analyzed by TOC & staining/counting. No chemicals were added.

Analysis

<table>
<thead>
<tr>
<th>weight</th>
<th>0.011 g. (per 100 mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TOC</td>
<td>55%</td>
</tr>
<tr>
<td>counts (10-300μ)</td>
<td>123</td>
</tr>
<tr>
<td>counts (10-1000μ)</td>
<td>214</td>
</tr>
</tbody>
</table>

The results are similar to the Westvaco values.
The sensitivity of the TOC analyzer is estimated at 2 mg/100 mL.
Next steps

- Reduce measurement uncertainty by optimizing syringe bore.
- The procedure can be used now if a Pulmac is available.
- If not, determine how to agitate screen (ultrasonic or mechanical).
- Assemble working unit.
- Should we look at pitch?
- Potential trial sites?

Instrument schematic (step 1: sample collection)
Step 2: flush line with surfactant

Step 3: homogenize & measure TOC
Commercialization

- Ionics, the manufacturer of the TOC machine, wants to commercialize.

- We work with Ionics on an online BOD sensor that is a precursor to this work. Thanks to G-P for getting Ionics involved.

- Projected cost: $2.5K.

- A patent disclosure has been filed. Should we proceed with an application?