Development of Screenable Wax Coatings and Water-Based Pressure Sensitive Adhesives

Project DE-FC36-04GO14309

Principal Investigator:
Steve Severtson - University of Minnesota

Research Partners:
USDA FS Forest Products Laboratory
Franklin International
The International Group
Boise Cascade Corporation
Generation of Contaminants from PSA Films and Wax Coatings

PSA films and wax coatings fragment during repulping.

Partial removal via conventional cleaning techniques.

Aggregation and deposition.

Reduced energy efficiency, lost production and diminished product quality.

Retention in the sheet.
Annual Cost to U.S. Paper Producers

MILL PRODUCTION
Reduced Efficiency, Added Cleaning and Waste Handling Costs

$527 MM

RAW MATERIAL
Downgrading of Recovered Paper

$150 MM

LANDFILLING
Increased Process Waste

$14 MM

In addition to the economic impact, it is estimated that an extra 800 MWh are consumed by industry due to PSA and wax contamination and greater than 1 million additional tons of fiber is landfilled.

**Project Objective**

Development of product engineering approaches to provide for enhanced PSA and wax coating removal during the screening of recycled fiber

- Identification of properties that govern PSA and wax coating removal and development of benign formulations
- Characterization of coating-substrate adhesion and development of techniques for manipulating adhesion to enhance removal
- Development of wet-end recipes for paper that promotes PSA and wax removal

The impact on paper recycling operations should be a design parameter in the development of all adhesive and coating systems formulated for paper applications
Project Structure
A Complimentary Partnership

- University of Minnesota
- Franklin International
- Boise®
- Forest Products Laboratory
- The International Group, Inc.
Research Strategy

Input
Commercial Assessment of New Product Approaches

Identification of Key Characteristics for Benign Materials

Synthesis and Formulation of Model and Commercial Systems

Database Generation & Analysis

Laboratory and Pilot Testing of Screening Removal Efficiencies

Characterization of Bulk Mechanical and Surface Properties

Output
Commercially Feasible Benign Products

Input
Access to Existing Product Lines
Commercialization of Benign Hot-Melt PSA
Project DE-FC07-00ID13881

PS Laminate Engineering - matching the right PSA with the facestock properties required to eliminate the negative impact of the adhesive on paper recycling

Research on water-based PSAs and coating wax are an extension of this project.

Advanced labels are designed to meet environmental guidelines – specifically executive order EO13148 – Greening the Government Through Leadership and Environmental Management. This product utilizes a repulpable face stock and repulpable adhesive making it recyclable. The Xonad™ process further reduces the amount of adhesive required on each sheet. Advanced Labels are guaranteed not to jam in any laser or Inkjet printers or photocopy equipment.

Benefits:
- Environmentally Friendly
- Repulpable EnviroSensitive™ Adhesive and Facestock
- Multi-purpose
- Contamination-free technology
- Compatible with all label software
- Premium quality construction
- Lay-flat/stay flat construction
- Guaranteed to work

To place your order contact Customer Service @ 1-800-777-2879

Business Media Division
Buffalo, New York
Hot-Melt PSAs

- 20-50 wt. % Base Polymer
- 30-60 wt. % Tackifier
- 0-25 wt. % Plasticizer

Easily Shaped for Characterization

G', G'', Tan δ

Melt Processing
**Water-Based PSAs**

**Polymerization**
- Monomers
- Emulsifier(s)
- Initiator(s)
- Biocide(s)
- Crosslinking Agents
- Buffer(s)

**Adhesive Emulsion**
- Tackifying Dispersion(s)
- Rheology Modifiers
- Wetting Agent(s)
- Defoamer(s)

**Formulation**
- Characterization samples restricted to thin films with properties dependent on formulation
- *G', G'', Tan δ ?*

**Formulated PSA**
- Processing requires coating of low energy substrate and drying
No evidence for dependence of screening removal efficiency on PSA properties such as:

- Tack
- Peel
- Shear
- Tensile Properties (E, TS,%EL,…)
- Glass Transition Temperature
- Surface Energy

Unlike that found for hot-melt PSA, dry mechanical properties and phase behavior for water-based PSA do not correlate with repulping and screening performance.
**PSA Properties and Wet Processing**

**Probe Tack**

- 1 mil Film @ 25°C
- Soaked in water for 1 min

**Tensile Strength**

- 1 mil Film @ 25°C
- Threshold of RE

Water-based PSA
Monitoring Influence of Moisture

- Monomer Composition
- Surfactant System
- Particle Size Distribution
- Crosslinking Degree
- Other Additives

Film Formation

Latex Properties

Film Analysis

in situ PSA Film Characterization

Mechanical Testing (AFM, Nanoindentation)

Adsorption (QCM, Microgravimetry)

Desorption (Chemical Analysis, Surface Tension)

Water Immersed

Cryo-SEM

Film Formation

Latex Properties
Current Status of Project

• Influence of processing additives on PSA film properties and on screening removal efficiencies was examined.

• Approach developed for preparing laboratory PSA films for transfer coating containing only the adhesive latex.

• Properties and removal efficiencies of >25 commercial water-based PSAs were measured. This required the development of new “in-situ” tests involving the building of in-house equipment not commercially available.

• Potential dominant properties governing removal efficiencies were identified and are currently being pursued through model formulations.

• Several model water-based PSAs are currently under study.
Wax Deposition and Removal

Mesophase Region

Deposition (%) vs. Temperature (°C)

Removal Efficiency (%) vs. Temperature (°C)
Wax Composite and Nanocomposite

**Composite**
- Dispersant
- Paraffin Wax
- Clay Filler
- Melt Processing

**Nanocomposite**
- Paraffin Wax
- OrganoClay
- Ultrasonic Processing
- Surfactant intercalation

Performance Testing of Nanocomposite and Scale Up

Screening Removal Efficiency

Both Laboratory and Pilot-Scale Testing

Barrier and Strength Properties

Permeation of Gasses and Liquids and Influence of Exposure on Strength

Commercially Feasible Compounding

Solid Feed

Mechanical Compounding

exfoliated and evenly distributed platelets
Current Status of Project

- Both laboratory and pilot scale tests developed for gauging screening removal efficiencies.
- Structure (molecular, nano- and micro-) and bulk mechanical and surface properties of IGI commercial coating waxes were characterized.
- Screening removal efficiencies and deposition tendencies were measured as a function of temperature.
- Clay-wax composite properties and performance were examined. This included pilot-scale testing of the IGI product. Results led to the development of an organoclay-wax nanocomposite produced via sonication.
- Mechanical properties of the composite and melt were characterized and its compounding using a laboratory compounding equipment below the melt point was demonstrated.
<table>
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<th>ID No.</th>
<th>Description</th>
<th>Planned Completion Date</th>
<th>Actual Completion Date</th>
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<td>1</td>
<td>Characterization and removal testing of Franklin label grade water-based PSAs</td>
<td>10/05</td>
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<td>2</td>
<td>Characterization and removal testing of standard wax coatings</td>
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<td>3</td>
<td>Characterization and removal testing of new model water-based PSAs</td>
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<td>Study on the role of facestock properties in determining removal of PSAs</td>
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<td>Development of new benign commercial PS labels</td>
<td>11/07</td>
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<td>Study on the role of board properties in determining removal of wax coatings</td>
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<td>Development of new benign commercial treated corrugated containers</td>
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Acknowledgements

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Questions and Discussion
Testing PSA Removal Efficiency

PSA film is pressed onto handsheets
labels are attached to sheets in a sample of copy paper
PSA content = 0.5%

removal efficiency is quantified gravimetrically

Cellulose Dissolution & Resin Oxidation (when required)

Removal Efficiency

IA Area Fraction

Paper Shredder

1/4" strips

Valley Flat Screen
15-cut screen (0.38 mm slots)

Accepts

Rejects

Adirondack Formax 1800H & 450H Pulpers
• consistency = 10%
• 60 Hz (∼690 rpm)
• typical time = 20 min.

TAPPI Method T-205 om-88
Testing the Removal Efficiency of Wax

Valley Flat Screen
15-cut screen (0.38 mm slots)

ASTM standard D590-93

Wax removal efficiency = \frac{\text{Weight of wax screening rejects}}{\text{Weight of initial wax in wax-coated boards}} \times 100\%
**Modeling the Fragmentation Behavior of Thermoplastic PSA Formulations**

<table>
<thead>
<tr>
<th>PSA</th>
<th>$T_{50}(^\circ C)$</th>
<th>$\alpha(^\circ C)$</th>
<th>SAFT($^\circ C$)</th>
<th>$0.10\Delta T(\circ C)$</th>
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<td>58</td>
<td>3.6</td>
<td>57</td>
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<td>72</td>
<td>7.3</td>
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<td>61</td>
<td>7.5</td>
<td>62</td>
<td>6.9</td>
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Removal Efficiency = \[
\frac{\exp\left(\frac{T_{50} - T_R}{\alpha}\right)}{1 + \exp\left(\frac{T_{50} - T_R}{\alpha}\right)} \times 100\% \]
Temperature-Dependent Repulping Results

- 14 Thermoplastic PSA formulations with rosin ester and C₅ hydrocarbon tackifying resin systems
- Predicted removal efficiency vs experimental removal efficiency ⇒ Slope = 0.99, Intercept = 0.07, R² = 0.98 (140 points)
Predicting Thermoplastic PSA Recycling Performance

**Commercial PSA 1**
- SAFT = 48.9°C
- ΔT = 56.6°C

**Commercial PSA 2**
- SAFT = 79.4°C
- ΔT = 63.5°C

**Commercial PSA 1**
- T_{50} = 49°C
- α = 5.7°C

**Commercial PSA 2**
- T_{50} = 79°C
- α = 6.4°C

α = 0.10ΔT
PSA-Face Stock Adhesion

Face Stock Tensile Loss

Low Surface Energy, Non-Fiberizing Face Stock

Increasing Removal Efficiency

Increasing fiberous rejects via screening

Complete fiberization of face stock

No Influence on Removal Efficiencies from Face Stock

High Surface Energy, Readily Fiberized Face Stock