“Understanding and Controlling the Kraft Liquor Cycle using FT-NIR Analysis”

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Needs a consistent high solids black liquor (75%+) with a stable viscosity.

Needs a relatively clean green liquor that has a consistent high TTA (7.5+) with good reduction efficiency.

Needs a clean, strong, consistent, white liquor with low dead load.

Needs a consistent weak black liquor that has low foaming, fouling and plugging characteristics.

Needs a consistent high solids black liquor (75%+) with a stable viscosity and Btu.
Quotes from Lord Kelvin

“To measure is to know”

“If you cannot measure it, you cannot improve it”
FT-NIR Liquor Analyser

- Based on Vibrational Spectroscopy, FT-Near Infrared
- Optical measurement based on absorption of light from chemical constituents in sample
- Remote analysis – fibre optically connected flow cells
- Fully automated system with line flushing
- Increase accuracy, precision, and frequency
  - Improve process optimization
Spectral Regions

- Wavenumber (cm⁻¹)
  - Electromagnetic Spectrum
  - X-ray
    - 700nm
  - Ultraviolet
    - 2500nm
  - Infrared
    - 250000nm
  - Microwave
    - 1000000nm

Absorbance

- Weak
- Strong

Slide is courtesy of FPInnovations
How is it Measured?

Sample

Fiber optic

Sample

Fiber optic
Hardware Components

Spectrometer in MCC room

Spectrometer (back) with 8-detectors
One Mill’s FT-NIR Analysis System

<table>
<thead>
<tr>
<th>Digestion</th>
<th>Recovery</th>
<th>Recaust</th>
</tr>
</thead>
<tbody>
<tr>
<td>(white and weak black)</td>
<td>(unclarified green)</td>
<td>(clarified green and white)</td>
</tr>
<tr>
<td>EA - white</td>
<td>TTA</td>
<td>TTA</td>
</tr>
<tr>
<td>TTA- white</td>
<td>Reduction Efficiencies</td>
<td>Carbonate</td>
</tr>
<tr>
<td>Sulfidity-white</td>
<td>Carbonate</td>
<td>EA</td>
</tr>
<tr>
<td>REA-black</td>
<td>Sulfidity</td>
<td>AA</td>
</tr>
<tr>
<td>Lignin- black</td>
<td>Sulfate</td>
<td>Sulfidity</td>
</tr>
<tr>
<td>Inorganic/organic-black</td>
<td>Thiosulphate</td>
<td>Thiosulphate</td>
</tr>
</tbody>
</table>

- **Digester**
  - FTPA2000 - 260 spectrometer rack
  - Fibre Optic Cables
- **Recovery**
  - Sample station
- **Recaust**
  - EA - white
  - TTA
  - TTA- white
  - Reduction Efficiencies
  - Carbonate
  - Sulfidity-white
  - Carbonate
  - EA
  - REA-black
  - Sulfidity
  - AA
  - Lignin- black
  - Sulfate
  - Sulfidity
  - Inorganic/organic-black
  - Thiosulphate
  - Thiosulphate
What the FT-NIR can Add to the Understanding of the Kraft liquor Cycle

**Traditional Measurements**
- Weak black liquor Beaume
- Firing Temperature
- % Solids (Firing)
- Gun pressure
- Dissolving tank Beaume
- Reduction efficiency (manual test – weekly?)
- TTA (Manual from recaust)
- EA to digester (Manual test)

**Added FT-NIR Measurements**
- Inorganic/organic composition
- REA from the digester
- Lignin content Weak black liquor % solids
- Intermediate Black Liquor % solids , REA
- Critical solids
- BTU of liquor
- Soap/tall oil content
- On line TTA, carbonate, EA sulphidity, sulfate, thiosulphate (from dissolver, equalizing tank and feed to slaker)
- On line WL EA to the digester

*Future*
# FT-NIR and Cycle Needs

## The Evaporator and Recovery boiler

### The Evaporators
- Needs a liquor low in soap
- Needs a consistent liquor with respect to the critical solids of Burkeite and dicarbonate

### The Recovery Boiler
- Needs a consistent, high solids (75%+) with a stable viscosity and BTU
- Needs a liquor low in soap or at least a liquor with a fairly constant soap content

### FT-NIR Analysis on Black Liquor Can Provide
- An inorganic /organic ratio that provides information on the BTU of the liquor and information that can impact viscosity
- REA information which can influence liquor viscosity, soap solubility and evaporator performance
- Lignin determination that may in the future lead to a lignin removal strategy to remove the bottleneck of the recovery boiler
Soap solubility and REA

- Doug Foran (a tall oil expert) has stated that in his presentation at the Tappi Short course that for good soap recovery the REA should be above 6 grams per liter at 20% solids or 2.9% by black liquor weight.

- If soap stays soluble this can cause poor performance on evaporators, increased steaming on the recovery boiler and it can influence the bed formation.
REA and Soap Solubility

Hourly Extraction REA

Grams per liter

Softwood
Hardwood
Softwood

REA soap solubility line

Hourly REA Readings over 6 days
REA and Viscosity

![Graph showing the relationship between REA and Viscosity.](image_url)
Lignin Precipitation

• Overloaded recovery boilers could possibly be relieved through lignin precipitation
• This is now commercially available from Lignoboost
• To be effective it would be advantageous to know the lignin content to determine how much lignin should or could be removed
• REA determination could provide insight into the carbon dioxide to be used in the precipitation process and to determine optimum conditions for soap recovery which could interfere with the precipitation process
Lignin Changes with Species

Black Liquor Lignin Content

Dec 5th to 15th

% Lignin

HW

SW

Lignin

Hardwood/Softwood
Organics and Black Liquor Viscosity

Organic/ Inorganic Content and Viscosity

Organic content
Inorganic content
Viscosity at strong tank

Hourly Reading Nov 2-9
FT-NIR and Cycle Needs
The Recaust

The Recaust Process Needs

- A clean green liquor
- That has a consistent TTA that is fairly strong (7.5)
- That has low dead load (high reduction efficiency)

The FT-NIR on Green Liquor

- Provides on line TTAs with associated carbonate, sulphidity and EA on a continual basis at the dissolving tank the equalizing tank and after the green liquor clarifiers
- It provides on line information of recovery boiler reduction efficiency with associated information on sulphates and thiosulphates
Manual density/ auto density and FT-NIR derived TTA

TTA tracks dissolving tank density extremely well

Manual beaume tests
TTA Improvement at Equalizing Tank Using Trim

Dissolving Tank and Equalizing Tank TTAs for One Month

- Dissolving Tank GL TTA
- Equalizing Tank G.L. TTA

Grams per Liter

Hourly Readings
TTA Control (analyser-based)
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Density Control

TTA Control

Equalization Tank
GL clarifier
WL storage
Digester WL
Reduction Efficiency and Sulphate (steady state)
Reduction Efficiencies
Responses to Air Changes (Jan 11th 2008)
Causticizing Efficiency (CE) Control

Feed forward CGL liquor compositions

From Green Liquor Clarifier

Flow

FT-NIR

Slaker

Feedforward Control

Kinetic Model

Feedback Correction

Lime Availability

CE Target

Temperature

FT-NIR

FT-NIR

Causticizers

To White Liquor Clarifier

Feedback Caust. and WL liquor compositions

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### Recaust. Variability Reduction

<table>
<thead>
<tr>
<th>Process Parameters</th>
<th>Standard Deviation</th>
<th>% Change</th>
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<tbody>
<tr>
<td></td>
<td>Before</td>
<td>After</td>
</tr>
<tr>
<td>GL TTA</td>
<td>3.7</td>
<td>2.6</td>
</tr>
<tr>
<td>CE</td>
<td>3.6</td>
<td>2.0</td>
</tr>
<tr>
<td>Settling Rate</td>
<td>12.7</td>
<td>8.0</td>
</tr>
<tr>
<td>WL EA</td>
<td>3.2</td>
<td>1.9</td>
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The Impact of Dead Load

• The difference between a low and high dead load operation might amount to as much as 125 kg/metric tons of pulp (mtp).

• Differences in energy use between a low and high dead load operation can be as much as 0.7 GJ/mtp.

• A 1% increase in causticizing efficiency reduces dead load by 6-7 kg/mtp, and a 1% increase in reduction efficiency decreases dead load by 2-3 kg/mtp. (Grace and Tran, Tappi Journal July 2009)
Concluding Remarks

• The FT-NIR can measure BL, GL, WL liquor properties online (with same unit)
• Maintenance is low (compared to existing technologies)
• This technology has allowed for measurements on a more consistent basis than possible manually
• Measurements are being made on “new” liquor properties that may allow for a better control of the liquor cycle
• New control strategies are being developed and implemented (based on new liquor properties) to allow for optimization of the Kraft liquor cycle
• We are at the “beginning” of the maximization of the liquor cycle from an energy, quality and operation perspective