ADVANCED AT-LINE KAPPA MEASUREMENTS WITH NIR-SPECTROSCOPY FOR FIBRELINE OPTIMIZATION

FITNIR ANALYZERS INC.

Thanh Trung
Outline

• Overview kappa measurements and it’s importance for kraft mill
• Available technologies
• Installation and implementation of NIR analyzer
• Results from mill
  – Wet pulp kappa for stockline
  – Dry pulp sheet kappa for final product check/verification
  – Other applications
• Learnings and best practices
• Summary and conclusions
Variable-Costs in Pulp Production

- Five main components to variable costs:
  - Fiber
  - Chemicals
  - Energy
  - Labour
  - Maintenance

- To improve margins
  - Reduce internal variable costs
  - Improve efficiencies
  - Improve yield, product quality

Cost (US$) of producing 1 ADMT of HW kraft pulp ($)
Mill Optimization Opportunities

- Chips
- White Liquor
- Green Liquor
- CaO
- CaCO₃
- Recovery Boiler
- Dissolving Tank
- Evaporators
- Washers
- Heavy Black Liquor
- Weak Black Liquor
- Pulp Bleaching
- Weak Wash
Top Factors Influencing Digester

- Pulping chemistry: WL EA/AA, sulfidity
- Wood species and anatomy
- Chip size distribution
- Chip quality (MC, others)
- Mill digester operations (time & temperature)
- Critical to measure the WBL REA for fast feedback
- All factors influence Kappa number:
  - Critical for feedback for digester as well as feedforward for bleach plant.
Current Standard Kappa Testing

• KMnO4 Titration:
  – First developed by Tasman and Berzins (1957)
  – Measures the residual lignin content of kraft and semi-chemical pulps
  – Kappa < 5 requires larger amount (>10g OD pulp)
    • Method requires 30 – 70% KMnO4 consumption
  – Kappa > 50 requires small amount (< 1g OD)
    • Higher shives, resinous bundles, uncooked fibres
    • Leads to greater error
Pulp Kappa Measurements

• Manual kappa titration suffers from many errors:
  – Shives, pins, and non-representative fibres
  – Inaccurate weight of samples
  – Liquor contamination
  – Chemical concentrations
  – Reaction time (10min.)

• Discrepancies can be observed between testers and between labs.

• Time consuming, resulting in low frequency of analysis

• To date, accurate and rapid kappa determination remains a challenge to the industry, especially with varying chip quality
Benchtop Auto-titration

• Jiang, Audet, van Lierop, and Berry
  – Adapted an autotitrator technique to micro kappa
  – Automated sample tray for up to 17 samples
  – Improved accuracy and std. dev.
  – Still mimics standard titration with measurement time of ~ 20 min. per sample
  – Excellent lab technique – reference method
Online Kappa Analyzer

• Photometric titrations – adapted based on KMnO4, KI absorbance peak
  – Monitors abs. loss with addition of thiosulphate.
• UV-based analyzer
  – Rely on UV and visible spectrophotometer
  – Multiple – discrete wavelength (species sensitive)
  – Well established and accepted
  – Reported to require higher maintenance
  – May not be well suited for high (>70) kappa due to low consistency requirements
• Insertion probe/single point
  – Promising technique and cost effective
• Industry still requires rapid, accurate benchtop kappa measurements
Near-Infrared (NIR) Spectroscopy

- Energy from NIR light is absorbed by the lignin
- Remaining light is diffuse reflected
- Optics collect diffuse reflected light
- Infrared detector registers absorption
- Spectrum contains unique features
Spectrum of Dry Pulp Sheet

- **H2O** at 1920nm
- **Cellulose** at 2100nm
- **Hemi-cel.** at 2330nm
- **1410nm**
- **1580nm**
- **1760nm**
- **Aromatic lignin** at 1690nm
- **Crystalline region of cellulose**
NIR Kappa Analyzer Installation
Demin. Water Booster and Filter

- Booster pump to pump demin. water to 80 psi
- Wash pulp to reduce chemical residuals
- Floc pulp in suspension to allow good pad formation
Sample Preparation Device

- Sample preparation station used to produce sample pad
- Pulp samples thoroughly washed
- Piston dewater and presses to form pad, ~1/2” thick
Sample Analysis

• Blank is scanned first run to zero the analyzer
• Wet (or dry) pulp sample is placed in sample chamber
• Sample type/location and collection time entered
• Analysis time ~ 15 secs
• Results displayed on screen or DCS
Analyzer Calibration and Performance

- Spectral data from analyzer is then correlated with reference values
  - Kappa of wet pulp
  - Kappa of dry pulp sheet
  - Possibility of other properties:
    - Pulp viscosity
    - Airdry content
    - S10 and S18
    - Etc.
KRAFT LINERBOARD APPLICATION

Wet Pulp Kappa Analysis
Kraft Linerboard Application

- Mill-A produces approx. 1200 t/d of linerboard pulp
- Has been utilizing an obsolete photometric kappa analyzer at the digester testing lab.
  - Results showed large variations shift-to-shift
- Comparison with TAPPI method showed ~10 point offset
- NIR data showed excellent linearity with TAPPI method
## Crosschecks with Mill TAPPI Titrations

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Digester</th>
<th>Standard Kappa*</th>
<th>Analyzer Kappa</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>A</td>
<td>112.9</td>
<td>112</td>
</tr>
<tr>
<td>S2</td>
<td>B</td>
<td>111.1</td>
<td>111</td>
</tr>
<tr>
<td>S3</td>
<td>B</td>
<td>114.4</td>
<td>113</td>
</tr>
<tr>
<td>S4</td>
<td>B</td>
<td>114.6</td>
<td>114</td>
</tr>
<tr>
<td>S5</td>
<td>B</td>
<td>115.6</td>
<td>115</td>
</tr>
<tr>
<td>S6</td>
<td>B</td>
<td>115.1</td>
<td>115</td>
</tr>
<tr>
<td>S7</td>
<td>A</td>
<td>104.7</td>
<td>105</td>
</tr>
<tr>
<td>S8</td>
<td>A</td>
<td>109.3</td>
<td>110</td>
</tr>
<tr>
<td>S9</td>
<td>A</td>
<td>91.7</td>
<td>91</td>
</tr>
<tr>
<td>S10</td>
<td>B</td>
<td>102.9</td>
<td>103</td>
</tr>
</tbody>
</table>

RMSEP = 0.68 Kappa
Analyzer Measurements @ Start-up

Digester-1

Digester-2
CONVENTIONAL KAMYR APPLICATION

Wet Pulp Kappa Analysis
## Conventional Kamyr Kappa Testing

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Location</th>
<th>Tester 1</th>
<th>Tester 2 (Control)</th>
<th>NIR Kappa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Blow-line</td>
<td>39.6</td>
<td>38.2</td>
<td>38.15</td>
</tr>
<tr>
<td>2</td>
<td>Upper Ex</td>
<td>41.4</td>
<td>40.5</td>
<td>40.4</td>
</tr>
<tr>
<td>3</td>
<td>Decker</td>
<td>37.7</td>
<td>36.4</td>
<td>36.1</td>
</tr>
<tr>
<td>4</td>
<td>Blow-line</td>
<td>27.7</td>
<td>29.6</td>
<td>30.2</td>
</tr>
<tr>
<td>5</td>
<td>Upper Ex</td>
<td>35.0</td>
<td>31.4</td>
<td>31.5</td>
</tr>
<tr>
<td>6</td>
<td>Blow-line</td>
<td>25.4</td>
<td>25.5</td>
<td>25.8</td>
</tr>
<tr>
<td>7</td>
<td>Blow-line</td>
<td>22.8</td>
<td>22.6</td>
<td>22.3</td>
</tr>
</tbody>
</table>
Mill Validation Results

\[ y = 0.9948x + 0.1438 \]

\[ R^2 = 0.9844 \]

Stdev Kappa = 1.0
NIRS Kappa Analyzer

- Accurate measure of true kappa number
- Method reduced tester-to-tester variability
- No chemical requirement
- Measurement can be completed in < 5min.
- Allow for higher frequency testing
FINAL PULP INSPECTION

Pulp Sheet Kappa Analysis
Finishing Line Quality Control

- Quality control at the finishing line requires onerous testing, especially when it comes to chemical and physical testing.
- For mills producing brown pulp for specialty applications (cement board), customer requires kappa number of pulp with shipment.
- Tedious and time consuming manual kappa means that product could be out of compliance and shipment could be rejected or called back.
Pulpsheet Kappa with NIRS

• Based on the same principle of operation, applied NIRS to measure pulp sheet kappa
• Pulp sheets taken at pulp machine, cut into ~6” discs
• System zeroed and sample is measured as is.
Pulpsheet Kappa Performance

\[ y = 0.9761x + 0.7143 \]
\[ R^2 = 0.9879 \]
Cross Machine Variability Analysis

- Pulpsheet samples from all bales across the machine were sampled
- Analysis done for all 8 samples
- Drop #4 chosen to perform crosscheck
- Cross machine direction data shows that kappa is consistent, within 1-kappa unit
Machine Direction Kappa
Machine and Washer-55 Kappa
Summary

- NIR technology, as applied, has been successfully implemented for mill’s day-to-day kappa testing:
  - Simplified analysis and removed sources of errors
  - Allowed for greater frequency of testing
  - Is currently being used to track digester operations, allowing for optimization
  - Eliminates hazardous chemicals
  - Savings on chemical upwards of $100K/year, based on purchasing of KMnO4 and acid

- Can also be used for other applications:
  - Pulp sheet kappa and other properties
  - Pulp sheet viscosity
Learnings and Best Practices

• Manual testing of kappa:
  – Must follow standard TAPPI or PAPTAC procedure to ensure accurate and meaningful values
  – Pulp should be washed thoroughly to ensure no contamination from dissolved lignin
  – Sample dry weight is critical
  – If chemical strengths are suspect, discard all and restart with blank titration
  – Avoid shives and pins which could highly impact analysis

• NIR Measurements:
  – Run blank (15s) every hour or before each test
  – Provide ~sufficient sample size to produce pad
Theme: Industry of the future:
“Chemical-Free Lab”
THANK YOU