Applying Refractometers to the on-line Measurement of Green Liquor Density

Western Canada BLRBAC
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C.A. Vossberg
Agenda:

• Background information

• Refractometer basic principles

• Key issues facing green liquor refractometer measurement

• Experimental installation review
Green Liquor Refractometers:

- On-line measurement of green liquor density or (TTA) at two stages in the process:
  - Outlet of Dissolving Tank (focus of presentation)
  - After Clarifier
- Allows real-time control of the green liquor dilution to meet the target TTA
- Benefits:
  - Indication (and prevention) of excessive green liquor density and impending crystallization
  - Improved white liquor quality
  - Consistent black liquor solids
  - Maintain a desired process solution
    - Accurate Process Control
    - Economical Operation
    - Decreased Offline Testing
Measurement Principle

R.I. (sapphire) = 1.760

R.I. (water 20°C) = 1.33335

If the angle of Light "B" = 30°, then

\[
\frac{(1.760) \sin 30°}{1.33335} = 0.6600
\]

\[
\theta_r = \sin^{-1}(0.6600) = 41.30°
\]

Critical Angle is when \( \theta_r = 90° \)

\[
\frac{(1.760) \sin \theta_i}{(1.33335) \sin 90°} = 0.7576
\]

\[
\theta_i = \sin^{-1}(0.7576) = 49.25°
\]

http://interactagram.com/physics/optics/refraction/
Measurement Principle
**Measurement Principle**

**GREEN LIQUOR T.T.A.**

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**PERCENT T.T.A.**

**REFRACTIVE INDEX**

- 1.369
- 1.370
- 1.371
- 1.372
- 1.373
- 1.374
- 1.375
- 1.376
- 1.377
- 1.378
- 1.379
- 1.380
- 1.381
- 1.382

**PERCENT T.T.A.**

- 6
- 6.25
- 6.5
- 6.75
- 7
- 7.25
- 7.5
- 7.75
- 8
- 8.25
- 8.5

**Graph**

- Plot showing the relationship between percent T.T.A. and refractive index.

**Electron Machine**

- Date: 10/20/2000
- Test: G.L. TTA
- Chem:
- Instr: ABBE
- 1.3825
Key Issue facing Refractometer Green Liquor Measurement:

- Optical coating
- Duration of on-line measurement before optical coating occurs
- Maintenance of cleaning method
- Thermal changes
An optical coating problem associated with green liquor has been an on-going problem resulting in the adaptation of several different cleaning methods:

- **Steam**
  - Inadequate scale removal (may accelerate scaling)
  - Thermal shock
  - Relatively low pressure over process

- **Mechanical removal**
  - Adequate scale removal
  - Frequent service intervals

- **Chemical removal**
  - Adequate scale removal
  - Frequent service intervals

- **High-pressure mill water**
  - Adequate scale removal
  - Poor water quality
  - Thermal change
  - Frequent maintenance intervals
Aggressive scaling can immediately begin occurring after an optical cleaning without time for an accurate measurement:

- Declining industry wide
- Greater attention to green liquor control
  - Reduce variation of TTA
    - You help us we help you
  - Adequate dissolving tank mixing
  - Anti-scaling additives
  - Reducing thermal changes
High maintenance equipment leads to an unsuccessful installation:

- Expensive
- Labor intensive
- Unreliable
- Leads to operator distrust
- Difficult for real-time control
Thermal Changes:

Thermal changes have been observed to aggravate the scaling and coating issues associated with green liquor resulting in increased refractometer maintenance:

- **Prism shock**
  - Steam purge can increase coating
  - Prism cracking
    - Hotter than process
    - Colder than process

- **Cleaning nozzle fouling**
  - Temperature change may contribute to scaling
    - Complete nozzle blockage
    - Water stream deflection
Green Liquor
Experimental Installations:

• Green Liquor Dissolving Tank:
  • Four different installations
    • Custom install locations tailored to individual mill requirements
    • Removable high pressure cleaner adapter
    • Heated water purge system
    • One experimental cleaning nozzle flushing system
  • Cleaning methods
    • High pressure cleaner supplied with heated de-mineralized water
      • Approximately 1500psi
      • Water temperature 160F
    • Boiler Feed Water
      • Approximately 800psi
      • Water temperature 160-200F
  • Significant incremental improvements
Installation 1

Three different dissolving tanks.

- Components:
  - (3) MPR E-Scan with High Pressure Cleaning Systems
  - Installed in dissolving tank green liquor recirculation 2” line
  - All units use smelt spout cooling water @ ~160F with drain solenoid to ensure hot water supply
  - Removable high pressure nozzles
  - 9 months ago changed from normal mill water to heated water

- Results with heated water:
  - Before: monthly service interval of sensing head due to cracking prisms and nozzle fouling
  - Now:
    - Using instrument for automated control
    - Sensing head service interval greatly enhanced
    - Nozzle service has been extended marginally

- Pro/Con with install:
  - Ease of isolation for sensor maintenance
  - Operations must insure clean recirculation line
  - Improper cleaning of recirculation line can result in damage to sensing head
Two different dissolving tanks, one boiler.

- Components:
  - (2) MPR E-Scan with High Pressure Cleaning Systems
  - Installed in dissolving tank green liquor recirculation 2” line
    - One tank in service at a time
    - Second tank in weak wash clean
  - Removable high pressure nozzles

- Pro/Con with install:
  - Ease of isolation for sensor maintenance
  - Must have two dissolving tanks
  - Operations must insure clean recirculation line
Installation 3

Three dissolving tanks with two discharge lines and dedicated weak wash supply

- Components:
  - (6) MPR E-Scan
  - Installed in dissolving tank green liquor 6” discharge lines
  - All units use high pressure boiler feed water with drain solenoid to ensure hot water supply
  - Removable high pressure nozzles

- Results:
  - Using instrument for automated control
  - Systems in operation for 6 months with no major issues or servicing

- Pros/Cons with install:
  - Simple system
  - Direct product sampling
  - Low maintenance
  - Availability of high-pressure boiler feed water and associated piping
  - Low risk of green liquor intrusion into boiler feed water
Installation 4

• One dissolving tank
• Components:
  ● (2) MPR E-Scan with High Pressure Cleaning Systems
  ● Both units use “de-min” water @ ~160F with drain solenoid to ensure hot water supply
  ● Removable high pressure nozzles
  ● Installed in 8” dissolving tank green liquor discharge / weak wash supply lines
  ● One unit has automated vinegar nozzle flushing system
**Installation 4**

- **Results:**
  - Before: (1) year sensing head service interval w/purge every 7 minutes for 10 seconds
    - Using normal mill water
    - Replacing nozzles monthly
  - Now:
    - Using instrument for automated control
    - Both purge systems on the heated water
    - Possibly (2+) year sensing head service interval w/purge every 12 minutes for 5 seconds
    - Over 6 months on same nozzle
  - Automated vinegar nozzle flushing
    - 6 month run time
    - Improvements still to be determined
    - May increase service interval of nozzle
    - Enough to justify added maintenance and complexity of system?
Installation 4
Installation 4
Experimental Installations

Conclusion

- Optical coating
  - Adequately removed with heated high pressure water
- Duration of on-line measurement before scaling occurs
  - Increased with less green liquor TTA variation
- Maintenance
  - Heated water
    - Reduction in scaling aggravation
    - Reduced prism shock
    - Reduced issues with cleaning nozzle fouling
  - Refractometer sensing heads installed on green liquor discharge / weak wash supply
    - Feasible when sensing head and cleaning nozzle maintenance is prolonged
    - Alleviates need for separate cleaning of recirculation line
  - Vinegar flush may not be beneficial enough to justify added maintenance
- Thermal changes
  - High pressure water nearly same temperature as green liquor
  - Drain solenoid valve
Green Liquor Refractometer
Summary

- Incremental improvements to refractometer cleaning system
  - Realistic option for on-line green liquor measurement if pipeline scaling allows
  - Accurate process control
  - Low installation cost
  - Reduced maintenance requirements

- Increased green liquor control
  - Safer recovery boiler operation
  - Overall improvement to Kraft process
  - Reduced standard deviation of green liquor variability
  - Better consistency leads to less coating and scaling
Electron Machine Corporation

C.A. Vossberg (President)
Cell (352)-406-1352
cal@electronmachine.com

Brad Osborne (Sales/Marketing Manager)
Cell (352)-267-0118
brad@electronmachine.com