APN1002
Continuous measurement of tall oil and residual effective alkali in black liquor

Key Words

- Pulping
- Black liquor
- Tall oil
- REA
- Residual effective alkali
- Soaps
- FTIR
- Process Spectroscopy
- MIR

Introduction

Tall oil is a valuable byproduct of the Kraft process. It is recovered from tall oil soap that naturally separates from black liquor and is commonly seen floating on the surface of washer filtrate and weak black liquor tanks. The soap itself is made up of various fatty and rosin acid salts/esters and unsaponifiable compounds, and is typically skimmed off and refined into tall oil, providing an additional stream of revenue to mills.

Soap separation also impacts the efficiency of the several unit operations. With decreased tall oil soap concentration in the black liquor, less fouling is seen in the evaporators and recovery boilers. Reduction efficiencies in the recovery boiler have been shown to be increased and the load on the recausticizers is also reduced. Soap separation is impacted by numerous physical and chemical parameters including liquor solids content, liquor temperature and residual effective alkali (REA) concentration. REA affects the solubility of the soap in the black liquor and therefore needs to be well controlled (usually > 6 g / L) to ensure good soap separation and therefore recovery.

Tall oil and REA measurements in weak black liquor are typically obtained offline, using complex and time-consuming methods. Owing to the high cost of these measurements, the data is often intermittent and discrete.

Process spectrometers allow for real time continuous measurements of important chemical concentrations. The IRmadillo is a process analyser built specifically for harsh processing environments like pulp mills. It uses Fourier transform infrared (FTIR) spectroscopy based on static optics, removing the need for moving mirrors or fibre probes, and dramatically improving stability, reliability, and ruggedness.

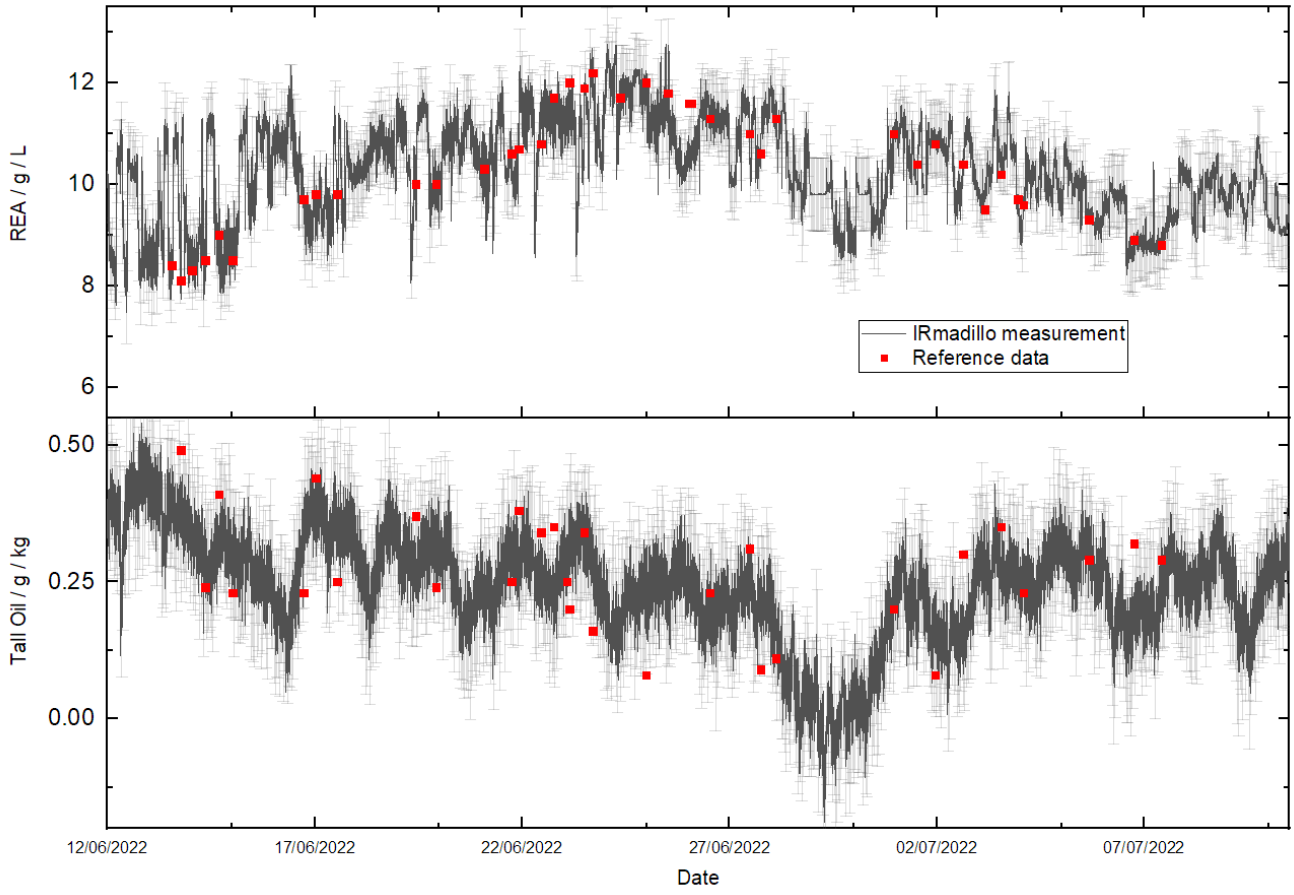

Figure 1: Measurement of REA and tall oil in weak black liquor line as recorded by an IRmadillo analyzer overlaid with laboratory reference data

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Example use case

This application note presents data collected from a single Kraft mill in Europe. An IRmadillo instrument has been installed in the weak black liquor lines after the washing and before the settling tank (see Photo) where the tall oil soap floats to the surface and is removed prior to the boiling of the liquor into a strong black liquor. Calibration models were developed for tall oil and REA allowing for real time continuous measurements that have then been integrated into the DCS, allowing the operators to monitor and measure them in real time. This knowledge has then been integrated into existing control systems to ensure successful tall oil soap separation and removal.

Results

Figure 1 displays the results of calibrations built to measure REA and tall oil in both hard and soft wood. REA required the use of a support vector regression (SVR – a simple type of machine learning) model whereas tall oil worked well with partial least squares (PLS). In many mills, tall oil and REA are not regularly monitored – they are sampled maybe once or twice per shift. Consequently, they are not well tracked throughout the process. If the REA concentration is presumed to be high, though is lower than 6 g L-1 , soap separation will be poor leading to reduced performance in the evaporators and recovery boilers as well as less tall oil soap being recovered.

Having the ability to obtain continuous real time measurements allows operators to know exactly when the REA levels are beginning to fall and therefore when to dose in. By measuring the tall oil in the black liquor, the efficiency of the separation and recovery process can also be monitored.

Conclusions

This work shows that the IRmadillo can be easily installed in a mill, and then used in-line to measure tall oil and REA in black liquor, allowing for real time monitoring and control of tall oil soap recovery. The models presented here include data from both hard and soft woods where Figure 1 clearly shows how each model is able to detect trends that would otherwise be missed with discrete sampling routines.

Photo: Installation of IRmadillo instrument using a simple flow cell mounted on the end of the probe in a weak black liquor line

IRmadillo™

Keep in Mind

The IRmadillo can be calibrated to measure a range of chemicals throughout the pulping process. As well as tall oil and REA in weak black liquor, the IRmadillo has also been shown to be effective in measuring chemicals like Na_2SO_4, NaOH, Na_2S and Na_2CO_3, in black, white and green liquor.

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