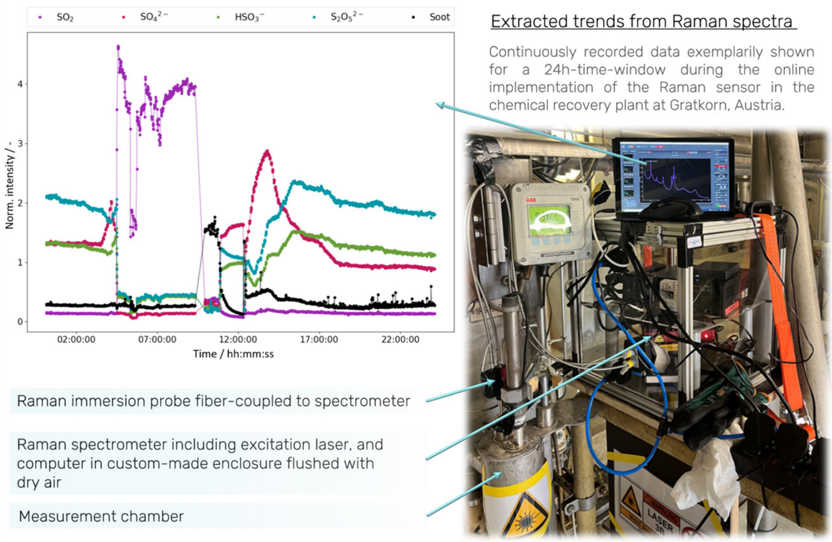
SUCCESS STORY



CHASE
Chemical Systems Engineering

Programme: COMET – Competence Centers for Excellent Technologies

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On-line PAT implementation at plant and extracted trends (© CHASE/Wieland)

CLOSING THE LOOP IN THE CHEMICAL RECOVERY OF THE PULP AND PAPER INDUSTRY USING RAMAN SPECTROSCOPY

DEMONSTRATION OF RAMAN SPECTROSCOPY AS VIABLE PAT-TOOL IN AN ON-LINE IMPLEMENTATION AT THE SAPPI GRATKORN MILL, AUSTRIA

Recovery of chemicals used in the solubilization of lignin from wood is a key process technology in the pulp and paper industry for both ecological and economic reasons. The main chemical ingredient of the “cooking liquor” used in this solubilization is $Mg(HSO_3)_2$. To recover the SO_2 and MgO, the spent cooking liquor is incinerated. SO_2 is recovered from the hot flue gas in a cascade of Venturi scrubbers while MgO is washed and hydrated to form the $Mg(OH)_2$ slurry. Exposing the flue gas to the $Mg(OH)_2$ slurry, the magnesium bisulfite cooking liquor is formed in a 2-step reaction process. Conditions in these scrubbers are harsh (temperatures > 60 °C, pH in the range 4-7, high ionic strength). Together with complex interactions between gas phase and liquid media, these conditions lead to the formation of insoluble salts, which reduces the recovery efficiency, causes unscheduled downtimes, and increases maintenance costs.

An improved process understanding is needed to counteract these precipitations. After thermodynamic modelling of the recovery process (cooperative collaboration with MFP 2.1), Raman spectroscopy was employed as non-destructive, in-situ process monitoring tool investigating the chemical composition in the Venturi scrubbers to bridge the modelling approach with the real-world process. In combination with multivariate regression models, the spectral data is translated into critical process-relevant parameters. The harsh environment proves challenging, as most available chemical process probes cannot withstand the process conditions, and those that do quickly degrade in industrial practice.

We demonstrate the use of Raman spectroscopy to address this challenging PAT task as it can be performed on-line in a non-invasive way. We show that Raman is fit for purpose, by being able to monitor process parameters such as free SO_2, total SO_2 and monosulfite (see figure 1) in real-time – parameters that are crucial for process control, and presently typically determined by manual sampling and titration. Raman spectra collected along the Venturi chain show concentration profiles matching established off-line titration.

Furthermore, the test-wise on-line implementation in a Venturi scrubber at the plant highlighted its potential as an efficient and effective tool for process monitoring and optimization. Accurate, real-time monitoring allows to control the process in a way to avoid precipitation of insoluble salts, hence, reducing the loss in chemicals and ensuring continuous operation with reduced risk of unscheduled downtimes.

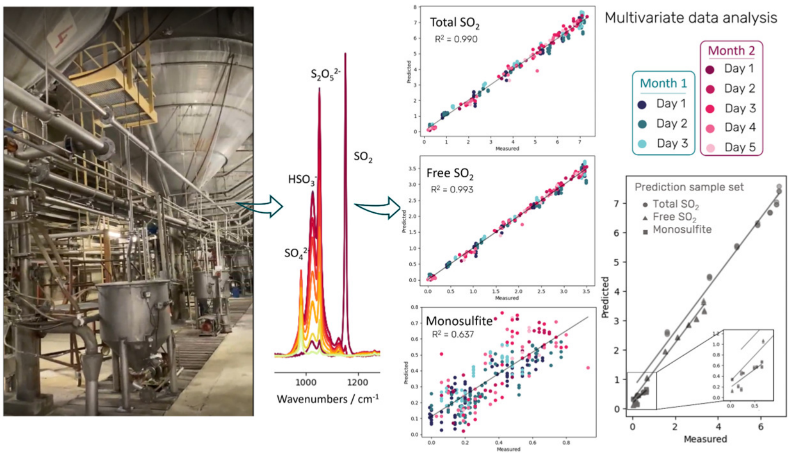

Figure 1: Raman spectra were collected along the chemical recovery chain (cascade of Venturi scrubbers). Different components in the scrubber media are detected via their distinct spectral fingerprint. Multivariate data analysis allows determination of several target variables (total SO_2, free SO_2, monosulfite) from a single Raman spectrum. Cross-validation and prediction results of the multivariate model based on ≈ 300 at-line measurements show good model performance.

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