Online Monitoring of Cleaning In Place Systems Facilitated by a Broadly Tuneable Quantum Cascade Laser

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Introduction

The increased performance and commercial availabilty of Quantum Cascade Lasers (QCL) made it possible to move from experimental setups in well protected labs to real world applications in industry. Especially lasers with an external cavity (EC-QCL), supporting a tuning range of up to 600 cm^{-1} in the mid-IR, are well suited for monitoring chemical processes in the liquid phase as they can cover broad absorption bands. Although conventional FTIR-spectrometers cover an even broader spectral range, they are outplayed by sensors based on EC-QCLs because they enable, due to their orders of magnitude increased optical power density, a higher flow cell thickness.

Here, we present a sensor based on an EC-QCL (Daylight Solutions, USA) for monitoring the cleaning behavoir of an industrially used vessel. The contamination of the vessel with residuals of a chemical or biological process taking place in the vessel were simulated by coating the vessel with different substances. During the cleaning process, the rinsing water is monitored with the EC-QCL sensor. The sensor is part of the PATOV-Project (Process Analytical Technology Unit for Online Verification), aiming to optimize the Clean-In-Place processes in pharmaceutical industry.

Experimental Setup

Optical Part:

- Light source: grating tuned EC-QCL (1030-1230 cm^{-1}, 350 mW)
- Flowcell: CaF_2-windows, 160 µm pathlength
- Detector: thermoelectrically cooled MCT-detector (-58 °C)
- Data acquisition: Boxcar Integrator + AD7760 (ADC)

Liquid Handling System:

- Interface between process stream and sensor
- Cleaning agents for the flow cell (ethanol, acetone)
- Injection of calibration samples

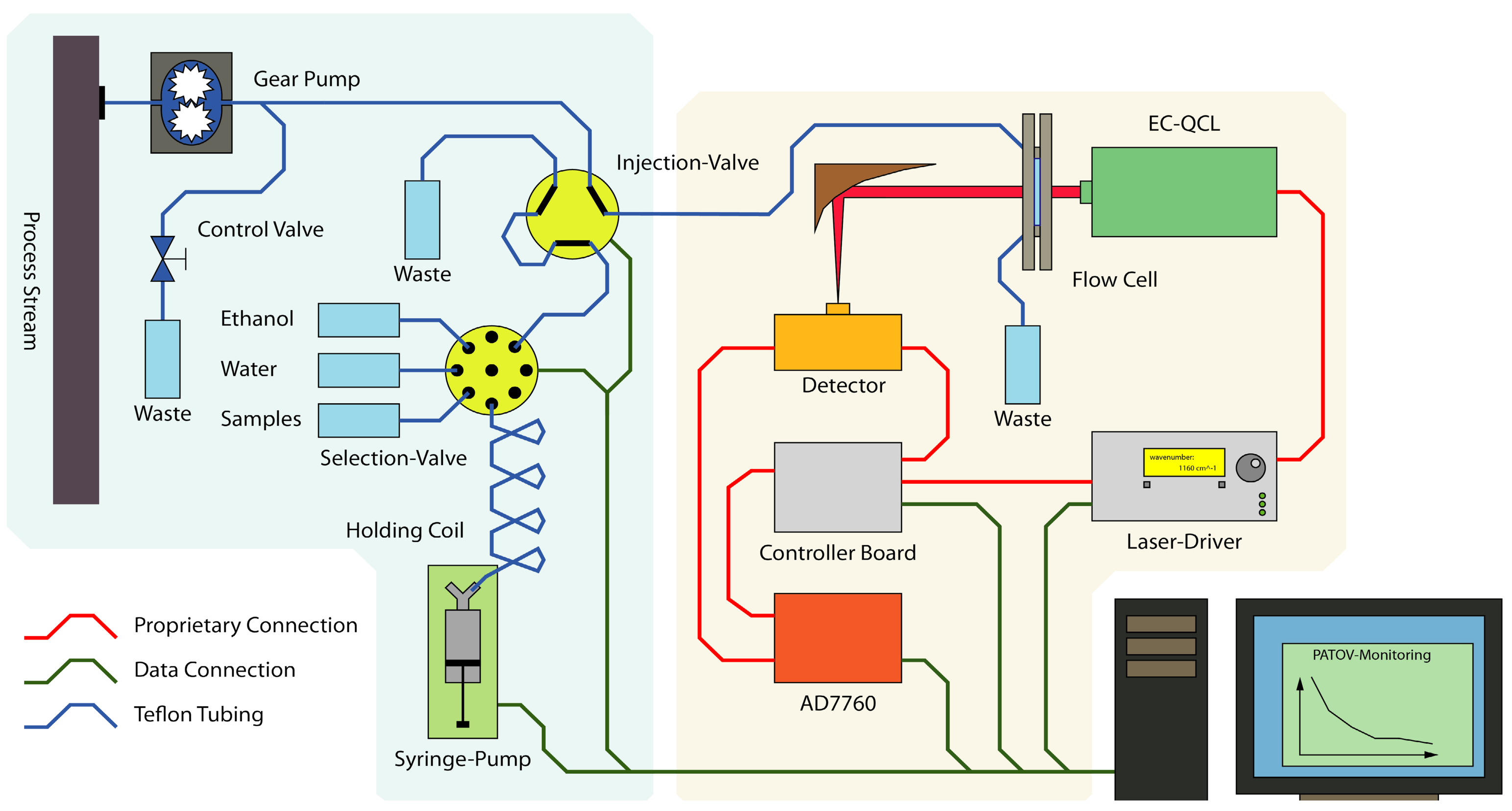

Fig. 1: Scheme of the EC-QCL sensor with the optical system (right) and the sample injection system (left)

On-Site Calibration

- Analytes of interest: glycerol and xanthan (carbon sources in fermentation)
- Calibrations were recorded directly next to the PATOV testing facility (semi rugged environment)

Fig. 2: PATOV testing facility

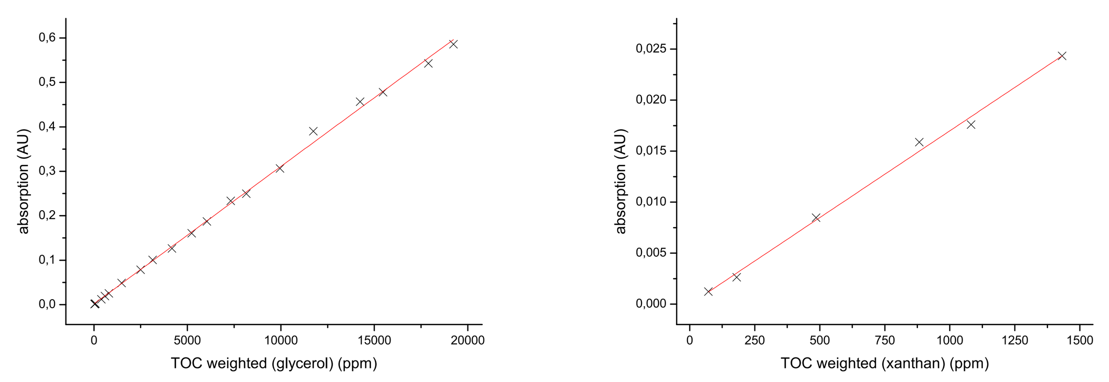

Fig. 3: Calibration curves of glycerol and xanthan

Process Monitoring

Laboratory Vessel

- Vessel filled with water (~8 l)
- Periodically spiked with glycerol (1 ml)
- Gear pump with needle valve used for continous sample injection
- Process monitored at 1113 cm^{-1}, 4 data points per second
- Continuous stired-tank reactor –> sigmoid response expected

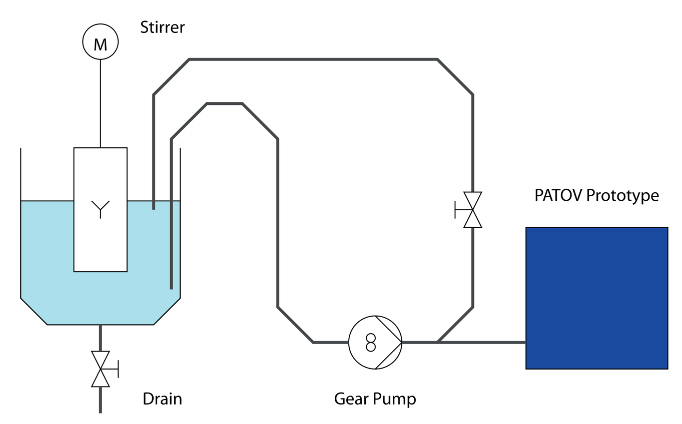

Fig. 4: Scheme of the laboratory vessel

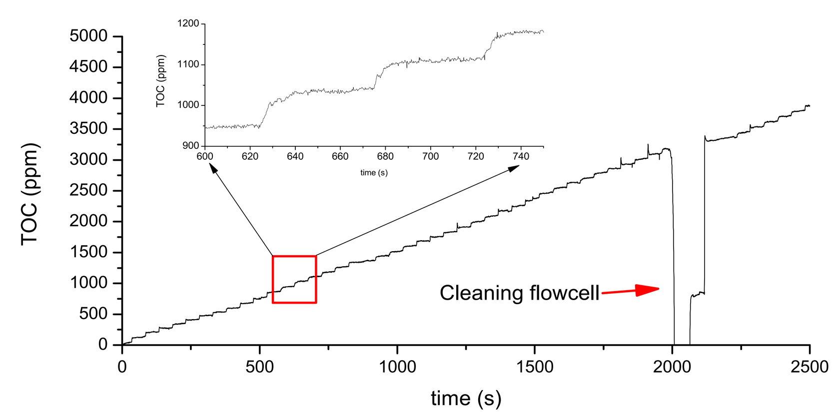

Fig. 5: Results of adding glycerol to the laboratory vessel iteratively (1 ml per step)

PATOV Testing Facility

- Walls of the vessel (~950 l) coated with glycerol (~800 g)
- Cleaning started and monitored at 1113 cm^{-1}, 1 data point per second
- Expected behaviour: sharp increase at beginning, then exponential decay

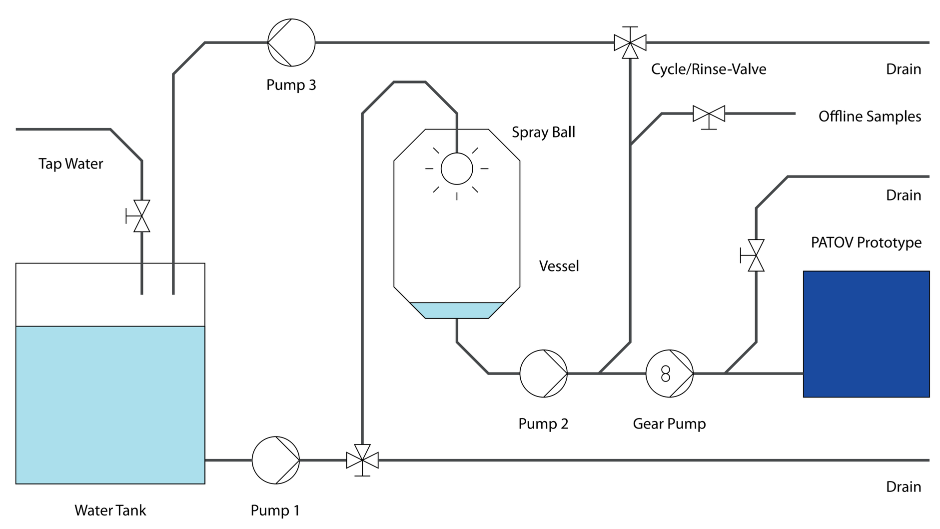

Fig. 6: Scheme of the PATOV testing facility

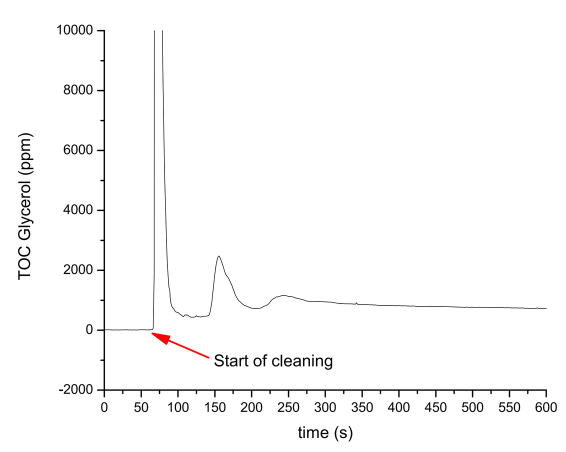

Fig. 7: Rinsing the PATOV testing facility after coating the walls of the vessel

Outlook

Upcoming experiments will include:

- Integration in an PLC equipped testing facility
- Evaluation on a differently sized/real life testing facility
- Expansion of the testing system towards protein contaminations

Project Partners: