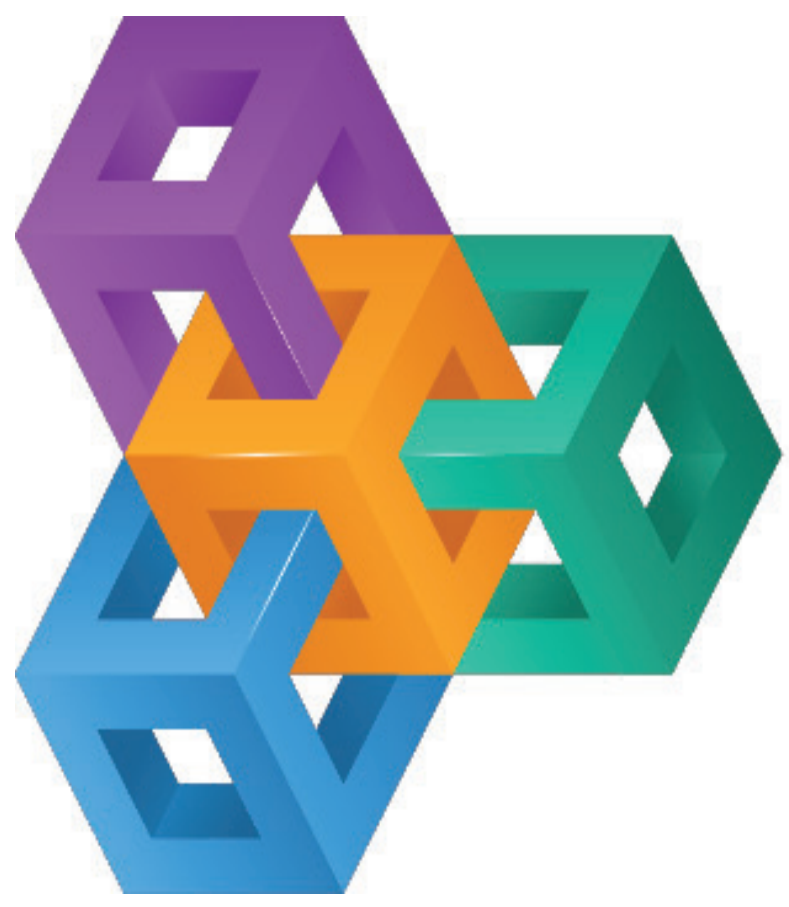




NEAR-INFRARED SPECTRAL SENSORS

PROPAT

INTEGRATED PROCESS CONTROL

Mr. Uula Kantojärvi, Spectral Engines Oy, Helsinki, Finland

Spectral Engines develops and sells high-performance infrared spectral sensors

The core component is same across all our product: the MEMS-FPI technology, which is used to select the measured wavelengths of the IR spectrum.

IR analysis can be used for both qualitative and quantitative chemical analysis.

- Qualitative analysis can be used to identify and classify samples into different groups.
- Quantitative analysis can be performed on samples of known type and different compositional parameters can be estimated.



The spectral sensors are rugged, modular field-ready devices designed to be a platform for development of completely new ways to use IR spectroscopy in new markets, especially where IR spectroscopic methods have previously been too expensive, or not in use at all, such as in the consumer markets. Central vision is to create a large network of NIR devices instead of centralized, resource-heavy testing spots.

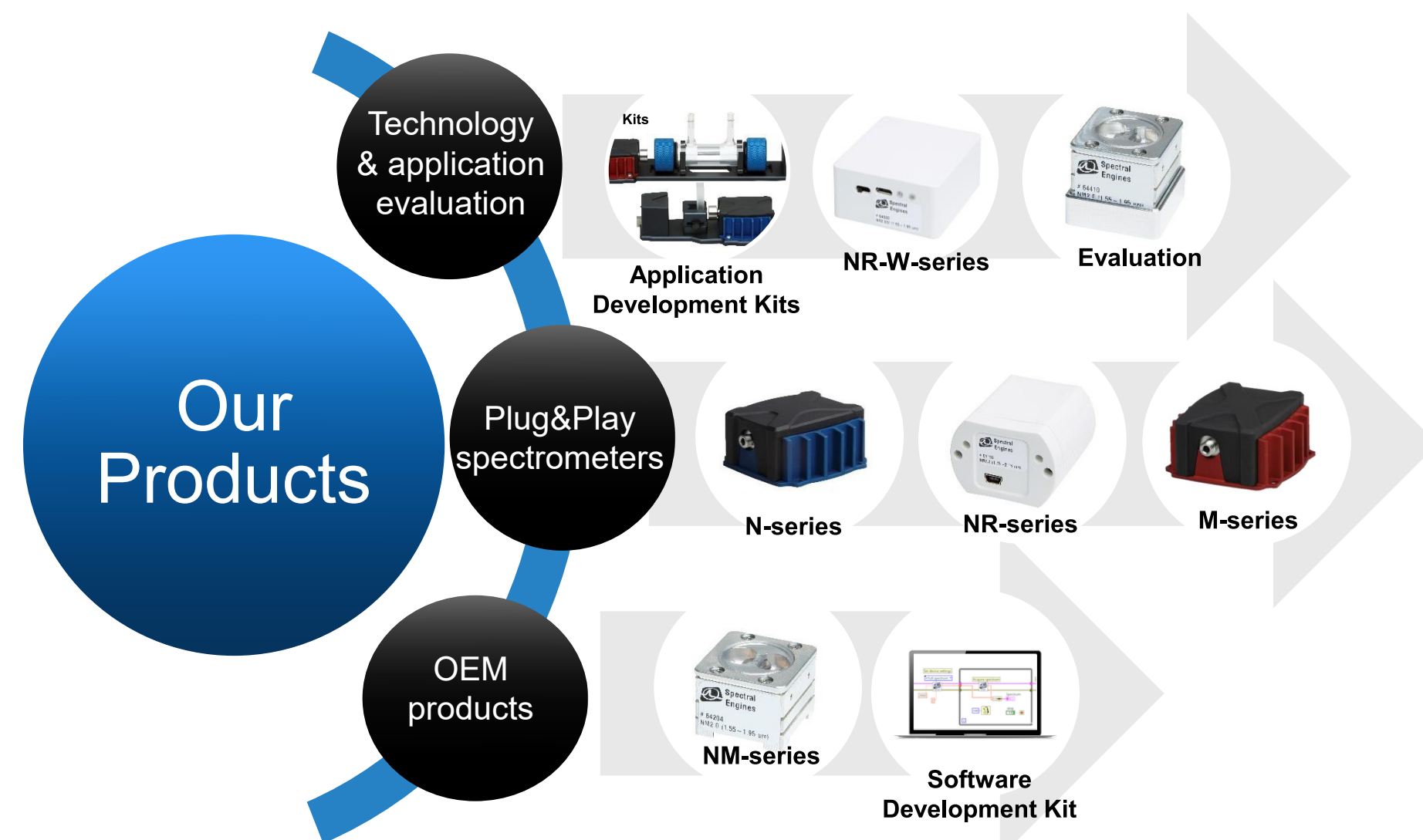
Instead of assessing quality of harvested crops all at once after the harvest, they produce would be tested at the harvester itself. Raw cow milk quality could be determined during the milking with a sensor device integrated into the milking equipment and not at the central laboratory only. The type of recycled polymers could be identified and sorted automatically.

Products							
	Type	Light sources	SMA90S	Integrated reflection optics	Integr. tungsten light sources	Data and power connection	Wireless
N-sensors	Modular	External	Yes	No	No	USB	No
NR device	Reflection measurements	Internal	No	Yes	Yes, 2 x	USB	No
NR-W wireless devices	Reflection measurements	Internal	No	Yes	Yes, 2 x	USB	Yes
NM modules	Modular	Internal, External	Optional	Optional	Yes, 2 x	I2C, UART, USB	No

Our products for the near-infrared spectral region includes N-series spectral sensors, NR-series reflection devices, and NM-series spectral modules.

The typical features of our sensors include that they are designed for specific wavelength ranges that must be specified on order. The N-, NR-, NR-W, and NM type units can be made to work in any of these specified NIR wavelength ranges.

- 1.7: 1350 to 1650 nm
- 2.0: 1550 to 1950 nm
- 2.2: 1750 to 2150 nm
- 2.5: 2000 to 2450 nm



New MEMS-FPI development by VTT

Dr. Anna Rissanen and Dr. Bin Guo, VTT Technological Research Centre of Finland

VTT Technological Research Centre of Finland and its VTT Memsfab Ltd. are both partners in the EU-ProPAT project. As part of the project new near- and mid-infrared spectral sensor technologies have been developed. The new N2.5 NIR spectral sensor has the VTT's MEMS-Fabry-Pérot interferometer for the range 1900 - 2500 nm as its core component.

During the first 18 months of the project, the main goal of VTT has been to advance the maturity level of VTT's most recent MEMS Fabry-Pérot (FPI) platforms with specifications provided by end users from process industry. VTT focused on providing:

- a) working MEMS FPI chips which are packaged and meet specifications, including optical modelling and opto-electrical characterization measurements;
- b) statistics of mass-produced FPI chips which are essential for future commercialization and cost effective production of VTT's FPI technology;
- c) improvement in MEMS design, process and layout in order to further increase the performance of the microspectrometer sensors.

Wavelength Chipsize	2500nm 3x3mm	3700nm 4x4mm
Tuning cap 2nd order	2590nm	3800nm
Range	1900 -2500nm	2900 -3700nm
Poly Si	158nm	250nm
Air-gap	538nm	850nm
Oxide cap	375nm	593nm
Structure	2 air gaps	3 air gaps
Aperture	0.9mm	1.4mm
Measured FWHM (3,4°)	17nm@2156nm 12.7V	22nm@3290nm 25V

The role of VTT Memsfab Ltd in the ProPAT project is to manufacture tuneable MEMS FPI filters. New technologies, especially in fabricating the moveable Bragg mirrors using Silicon-air layer pairs, as well as in dicing the fragile wafers using the stealth dicing process, have been developed. The infrared sensors based on the MEMS components have been developed, assembled and calibrated by Spectral Engines. This has been accomplished by doing optical, electrical and mechanical design steps. A significant effort has been focused on the design for manufacturing in order to reach high manufacturing yield. Environmental testing campaign has been carried out to test the sensors.

SENSOR USE IN EU-PROPAT PROJECT

Spectral sensors in polyester characterization and production process analysis

Dr. Sebastian Opitz, ZHAW Zürich University of Applied Sciences, Switzerland

For Megara Resins, a partner in the ProPAT project, the production process of polyesters was investigated both with NIR spectroscopy and with off-line analytical methods to determine the progress in the production of their products. Samples that were taken from the production process at different time points were analysed with titration methods, in order to monitor the changes during the production of the polyester, e.g. the reduction of monomeric educts that are consumed and incorporated in the polymer. At the same time, these samples were also analysed with NIR spectroscopy, in order to ideally relate the measured concentration of e.g. the monomeric educts to a signal in the NIR spectra. This way the NIR sensors can be calibrated.

For the standard off-line measurements the acid and hydroxyl titration methods were applied. All samples were analysed in triplicate with both titration methods. Another important qualifier for the product is its viscosity, which was solely determined beforehand by Megara at the production site. For acid titration, different approaches were tested to check robustness of the method. The first approach was manual titration with buret and visual detection of equivalence point, which is also performed at production site. The second approach was a semi-automatic titration with automatic dosing (Dosimat) and visual detection of equivalence point and the third approach was fully automatic potentiometric titration with evaluation of the recorded pH curve. We were able to obtain very good reproducibility for the acid titration results and the values between the different methods at ZHAW and the values measured at Megara were very similar. The hydroxyl titration has a longer reaction time and lower robustness. Hydroxyl titration is also more sensitive to small volume changes, which results in a higher uncertainty with higher variability. Despite these variables which can increase measurement errors the hydroxyl titration was also used for calibration of the NIR sensors.

NIR spectroscopy was initially done at room temperature in reflectance mode using the three commercially available first batch Spectral Engines sensors (N 1.7, N 2.0, N 2.2) as well as with a bench top NIR instrument from Bruker as reference. Later the new N2.5 spectral sensor was also tested in the laboratory setting. Near-infrared spectroscopy utilizes the light radiation from 800 nm to 2500 nm. With the three sensors, the spectrum is covered from 1350 nm (N 1.7) up to 2200 nm (N 2.2). N2.5 increases the maximum from 2200 to 2500 nm. In a second phase we performed measurements at high temperatures of around 230 °C in transmittance mode with a high temperature probe to investigate the influence of temperature on the spectra. We were able to show that absorbances change considerably with temperature and that NIR-spectra recorded at room temperature are unlikely applicable for extrapolation to high temperatures. It is therefore important to measure directly at high temperatures to successfully calibrate the sensors. The N2.5 was not tested in the elevated temperature measurements.

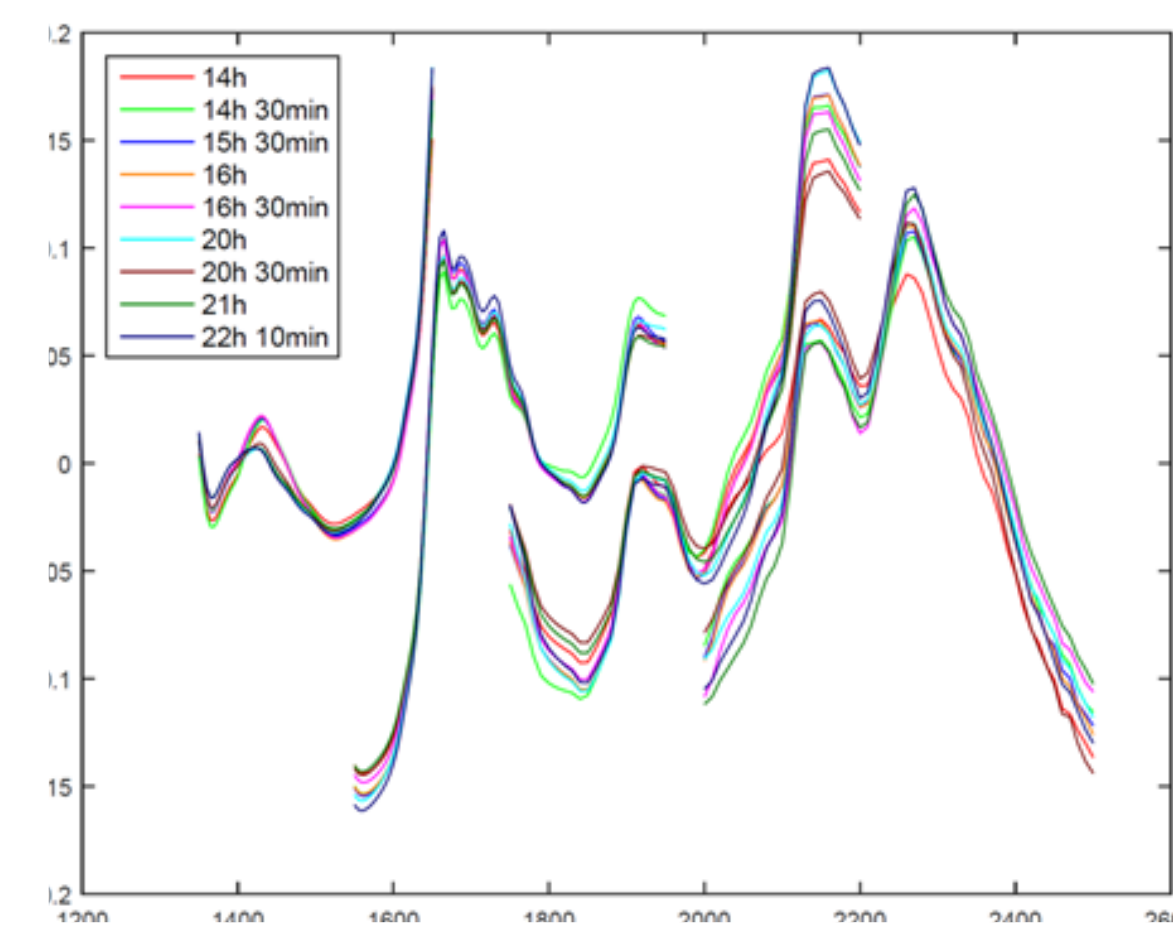


Figure: The different samples from the polyester production batches were also analyzed also with N 2.5 in addition to the three sensors (N1.7, N2.0 and N2.2) at room temperature using compressed polymer tablets. Pictured are the measurements of Pudralac 7383 with four spectral sensors, N1.7, N2.0, N2.2 and N2.5 at room temperature.

Spectral sensors in pharmaceutical production processes

Dr. Claudio Avila, University of Leeds, United Kingdom

University of Leeds have examined the feasibility of Spectral Engines' sensors to detect moisture content of solvent samples. Solvent samples including ethanol, acetone, and ethyl acetate containing trace amounts of water were tested.

The moisture content of the solvent samples varied from 0.01% up to 1.0% moisture, and could be detected using the N2.0 spectral sensor coupled to a 10mm liquid flow cell. This information could be used in the future to develop models for moisture analysis, such as those required by GlaxoSmithKline (GSK) for their process tableting pilot line, which is set as one of the goals for the ProPAT project. GSK is one of the world's largest pharmaceutical and healthcare company, and an industrial partner in the ProPAT consortium.

Currently most of the work done has been integrating the sensors in the experimental platform at the University. The initial goal is to compare the performance of the sensor against alternative analytical methods used for process monitoring. Preliminary information obtained showed that the sensors can be used to follow chemical reactions in which specific chemical compounds shows light interactions (absorbance or transmittance) within the sensor's wavelength range. A chemometric algorithm developed for the sensors allowed the detection of concentration changes during the esterification reaction between ethanol and acetic acid. This reaction produces ethyl acetate and water, and serves as model case which is currently studied as a preliminary step for the ProPAT project.

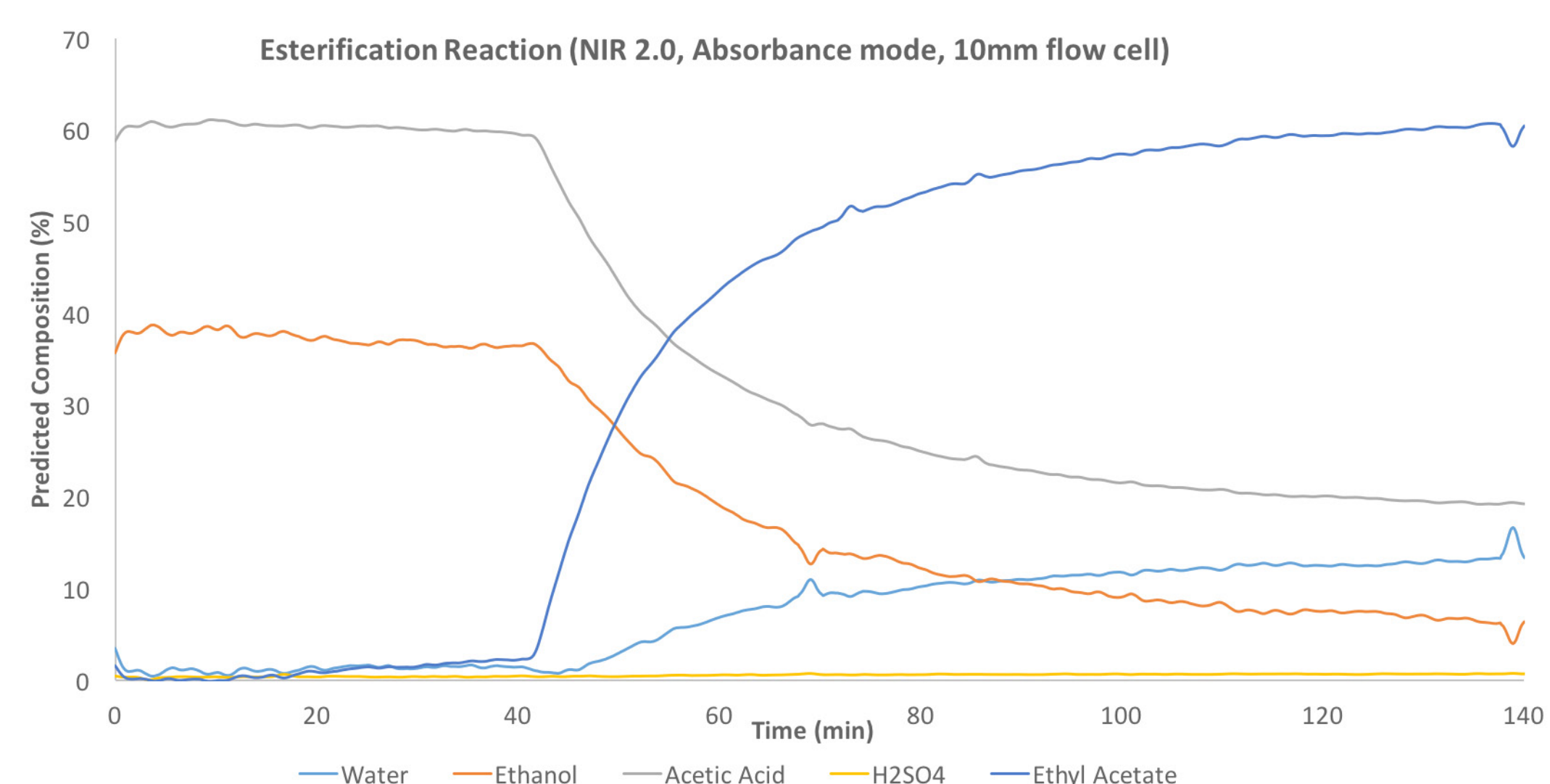
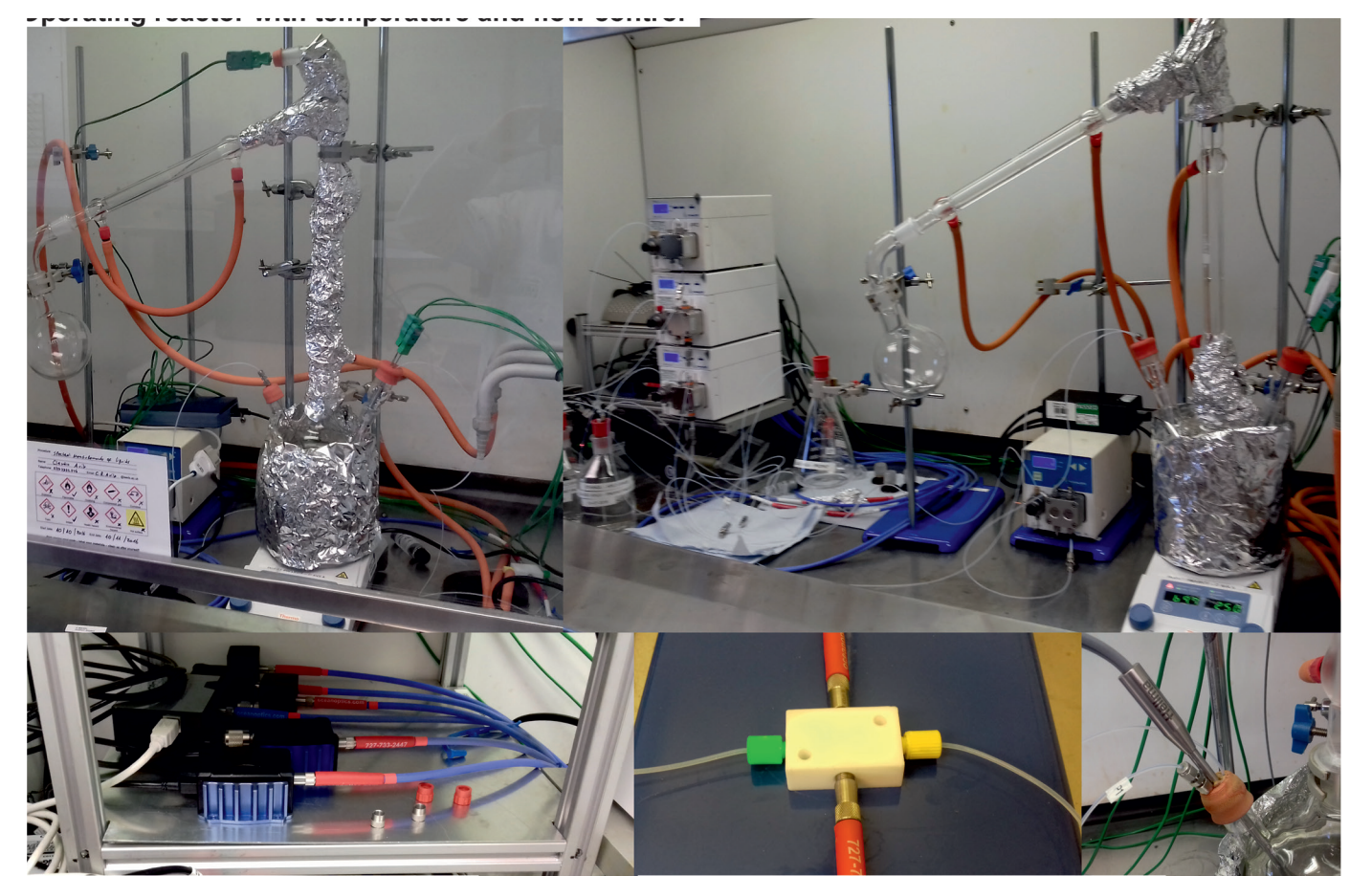


Figure 1: Compositional changes predicted from the sensor data combined with a chemometric algorithm for the esterification of ethanol and acetic acid to generate water and ethyl acetate (reaction performed in batch mode).