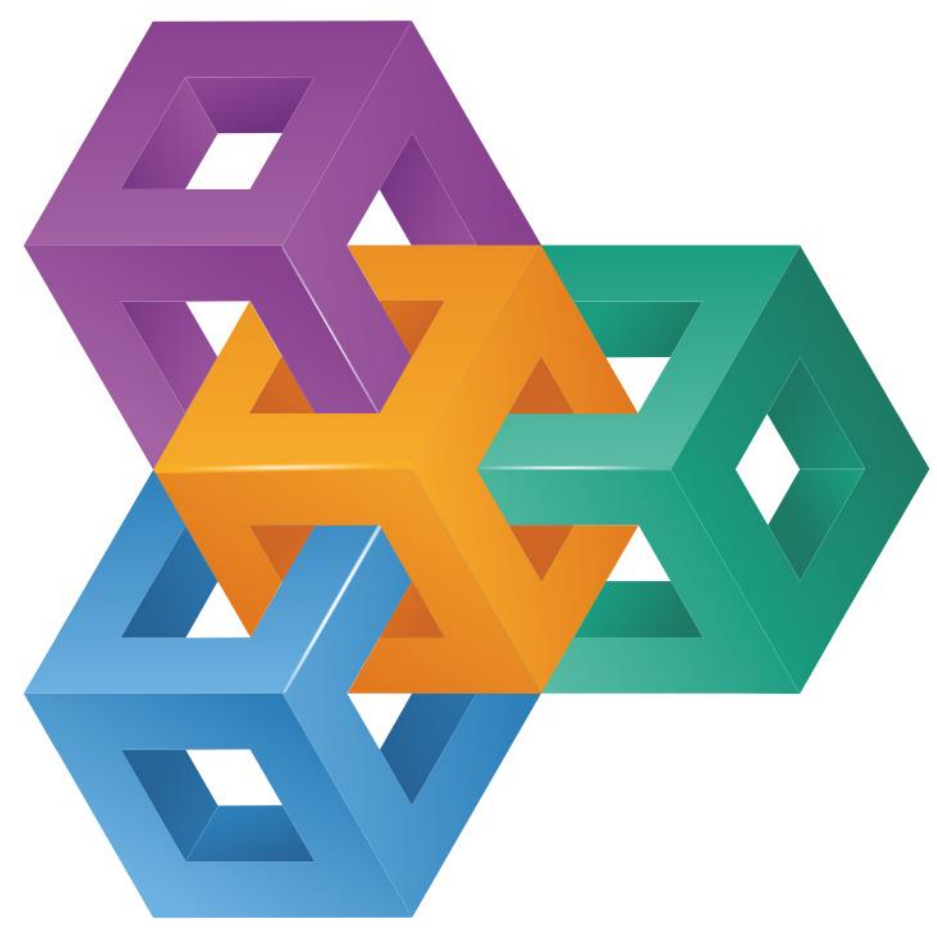




PROPAT

INTEGRATED PROCESS CONTROL

Validation of new NIR spectroscopic sensors for online monitoring and process control

Claudio Avila*, Richard Bourne and Frans Muller

Institute of Particle Science and Engineering, School of Chemical and Process Engineering, University of Leeds (United Kingdom)

* C.R.Avila@leeds.ac.uk

Overview

- Validation plan designed to test the spectral sensors in two industrial applications
- Hardware and software platforms developed for NIR sensor integration/validation
- Preliminary results show platform suitable for process research and development
- Spectral sensors show potential for online monitoring and process control

The validation of the spectroscopic information obtained from the new generations of Near Infra-Red (NIR) sensors is required for the correct prediction of physical and chemical properties from industrial processes, which can be then used to perform robust control actions. In this context, a set of ultra-fast and low-cost NIR sensors developed by VTT and Spectral Engines (Finland) have been integrated into an open source software/hardware platform to perform on-line analysis and chemometric modelling of industrial chemical reactions at Leeds University.

Sensor development



VTT & Spectral Engines

Sensor Validation



Leeds University

Industrial End-users



GSK & MEGARA

For the ProPAT project "Robust and affordable process control technologies for improving standards and optimizing industrial operations", this platform have been used to monitor different esterification reactions, with the final aim of scaling up the system to monitor a high temperature polymerization reaction performed by Megara Resins (Greece). Also, the platform is currently being deployed in a fluidized bed dryer designed to measure the moisture content of pharmaceutical granules, an application required by GlaxoSmithKline (UK).

How do we integrate our analytical methods into a large Chemical Process?
Transform spectra into a meaningful quantitative - qualitative output

Research System

Small volumes
Well controlled conditions
Close to intrinsic behaviour

VS

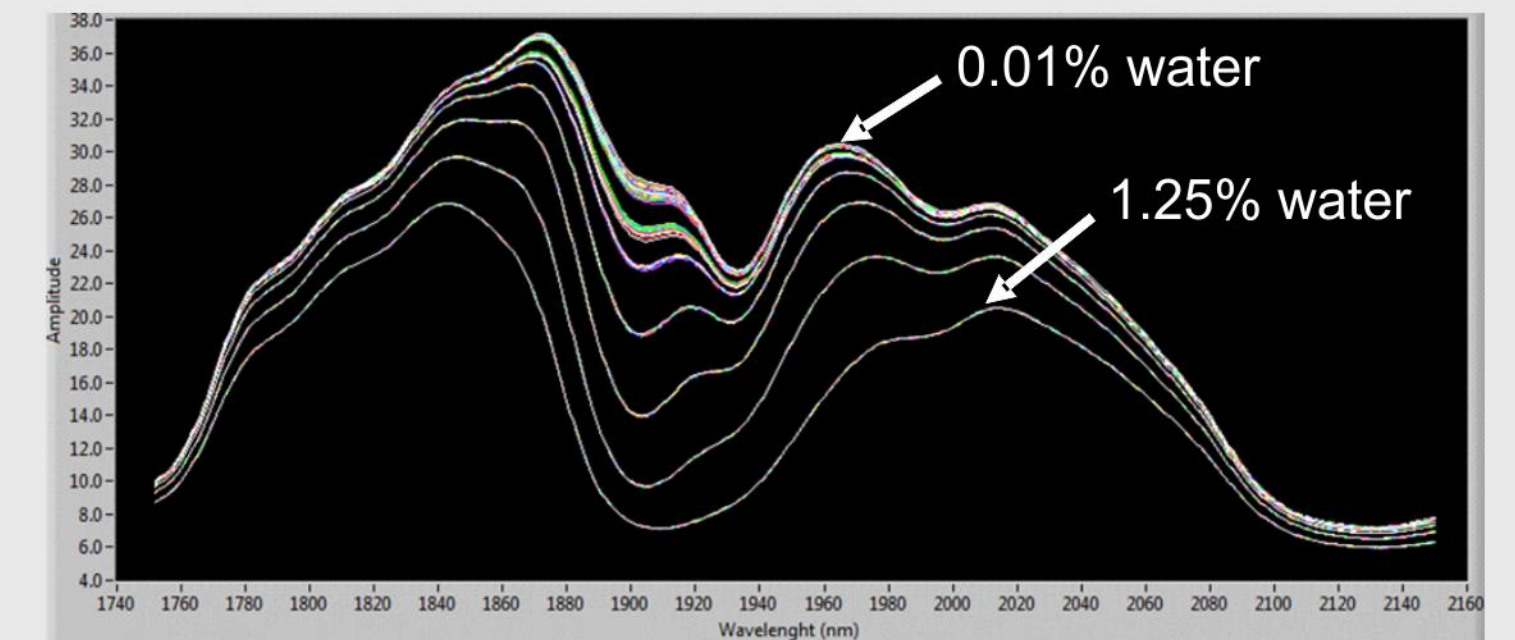
Industrial System

Large volumes
Fluctuating conditions
Mass/energy transfer restrictions
Friction and noise become relevant

"...unfortunately sometimes something that looks nice at laboratory scale cannot be achieved at large scales..."

Validation approach:

- NIR acquisition from pilot plant operations
- Fast algorithm to acquire and resolve unknown spectral signals applying chemometric modelling
- Generate output to the user and compare with data obtained from alternative analytical methods

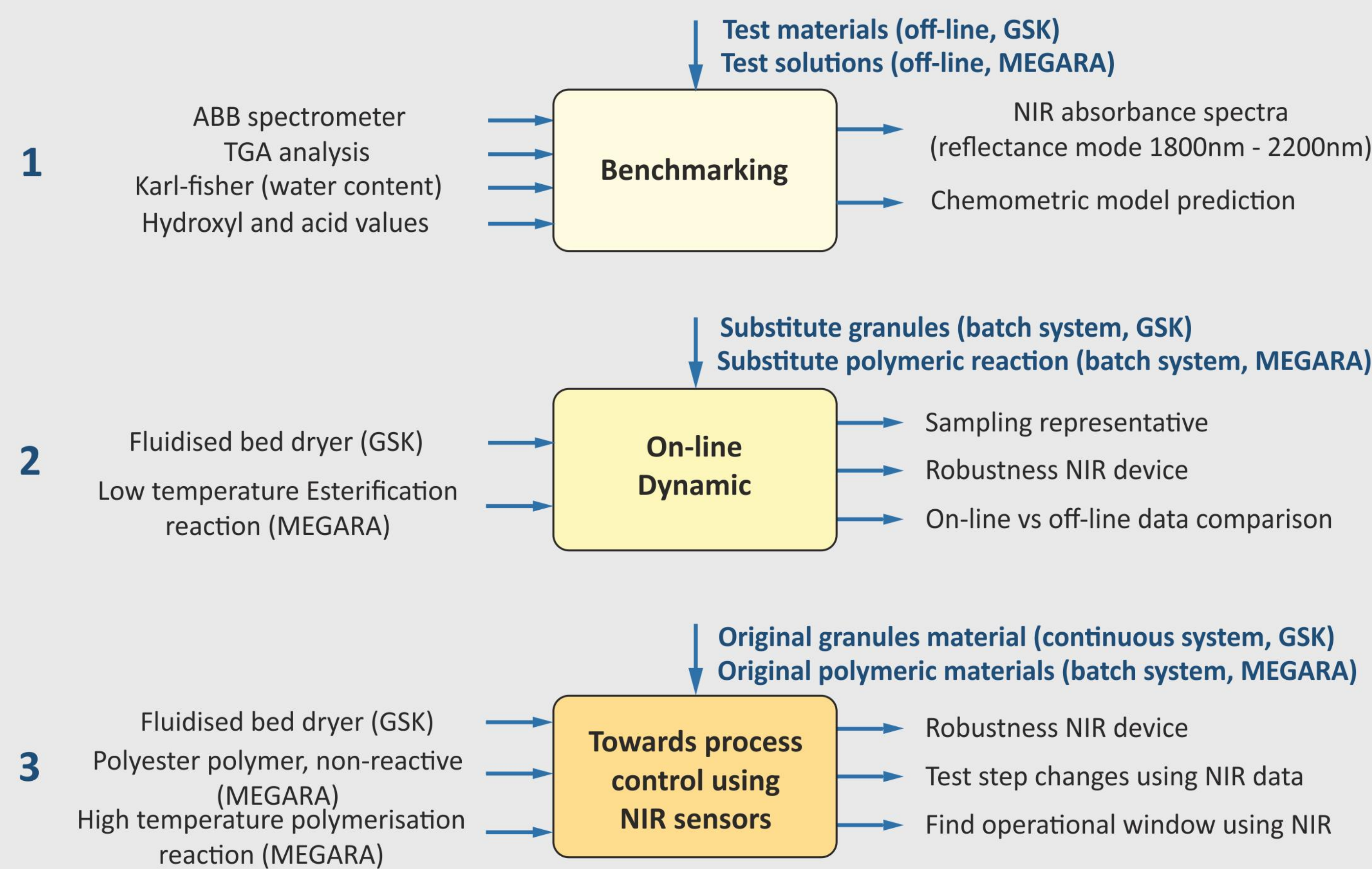


Water determination in pharmaceutical applications

The sensor validation platform developed at Leeds University support any type of spectral sensor in addition to those initially mentioned, allowing simultaneous acquisition and data processing of up to 50 spectral reading per second. This fast flow of information allow us to compare the performance of different spectral sensors operating at different wavelength ranges (NIR, FTIR, UV-VIS), and operating under controlled laboratory conditions.

Validation Plan

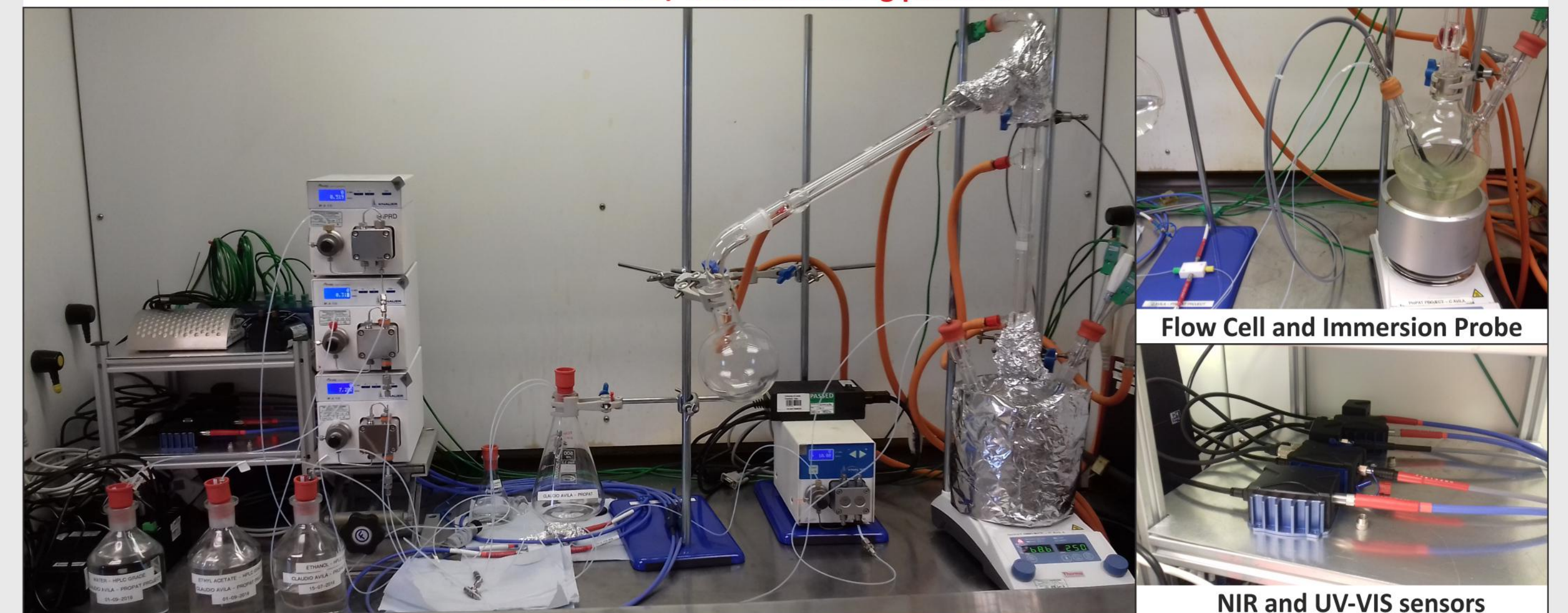
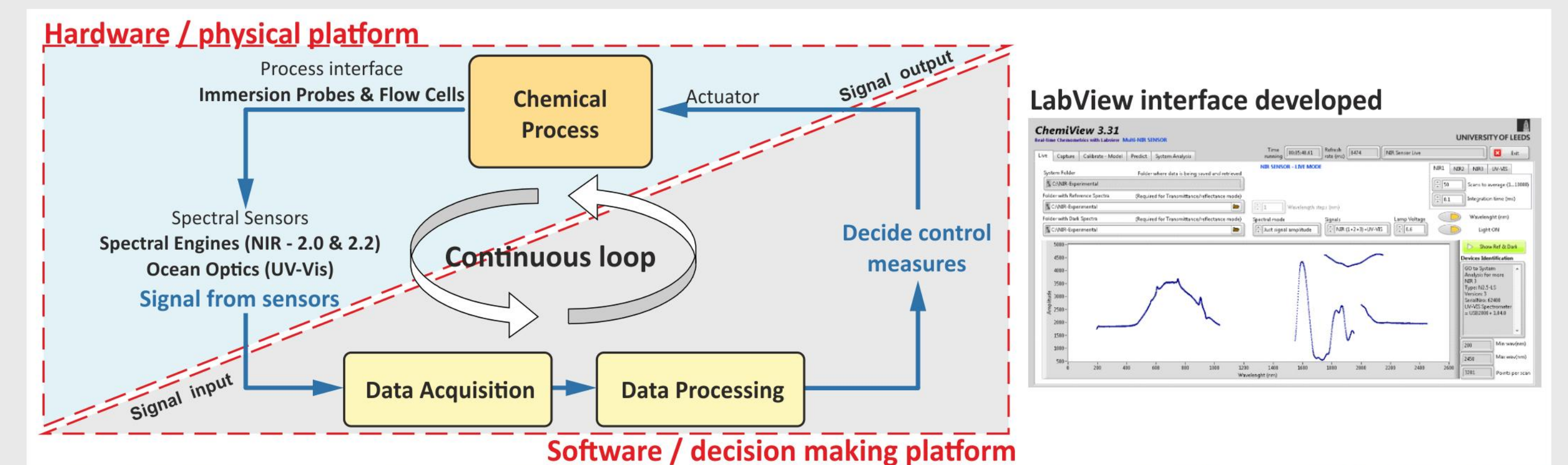
The validation plan for the NIR spectral sensors considers three stages: a) off-line validation under controlled laboratory conditions to baseline the instrument performance; b) on-line validation of the sensors operating under batch mode to assess the robustness and response under more complex operational requirements; and c) on-line validation of the sensors operating under a continuous mode to assess its potential for process control.



The experimental results obtained will build a matrix of data, comparing the performance of the NIR sensor against standard instruments and additional off-line analysis. The success criteria was defined using specific quantitative and qualitative indicators to be obtained from the experimental work, focusing on the reliability of the data provided by the sensor and the suitability of using the data acquired to implement process control at an industrial scale.

Experimental System

The experimental system developed incorporates two main components: a) the hardware: a physical platform including the process interfaces e.g. immersion probes, flow cells and fibre optics, and the spectral sensors; and b) the software: a decision making platform implemented in LabView including data acquisition, calibration, and processing algorithms, and an option to output the predicted data.



One of the experimental systems implemented at Leeds University (esterification): Left) Reactor vessel with temperature and flow control; Right) High temperature NIR Immersion probe and Spectral sensors used.

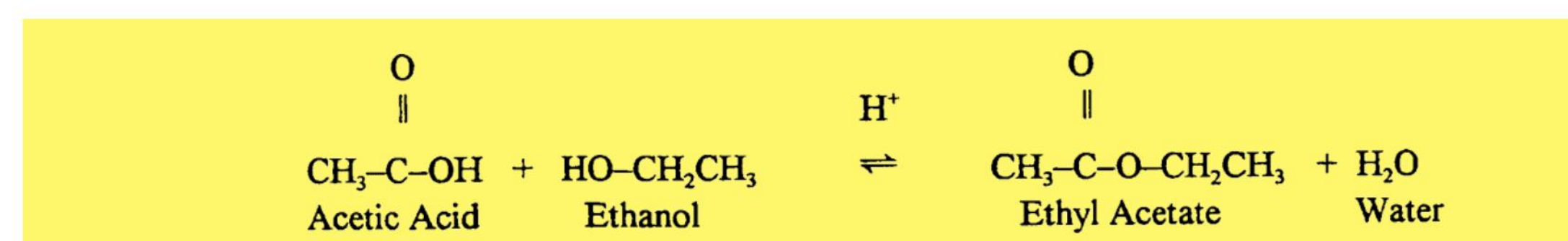
Preliminary Results

Two model chemical reactions have been used to evaluate the performance of the NIR sensors under batch mode (Left side: Low temperature ethanol esterification; Right side: mid-temperature butanol esterification). In both cases, the sensors output fed a basic PLS chemometric model from which dynamic chemical compositions were predicted for each system.

For both reactions, two spectral sensors acquired data simultaneously by using two different process interfaces: a) NIR 2.0 (1550-1950 nm), sensing from a flow cell with 10 mm pathlength using a continuous by-pass loop; and b) NIR 2.2 (1750-2250 nm), sensing from a NIR Hellma immersion probe, transfectance mode with 10 mm of total pathlength, directly immersed into the reactor.

Preliminary results show excellent stability of the sensor signals, with good experimental reproducibility and high signal to noise ratios. These conditions allowed good chemometric predictions for both reactions. This also showed that the noise and the different outcomes generated by the sensors were related to the process itself e.g. bubbles generated by the boiling crossing through the immersion probe, and intermittent flow passing through the flow cell; rather than malfunctions or issues with the NIR sensors.

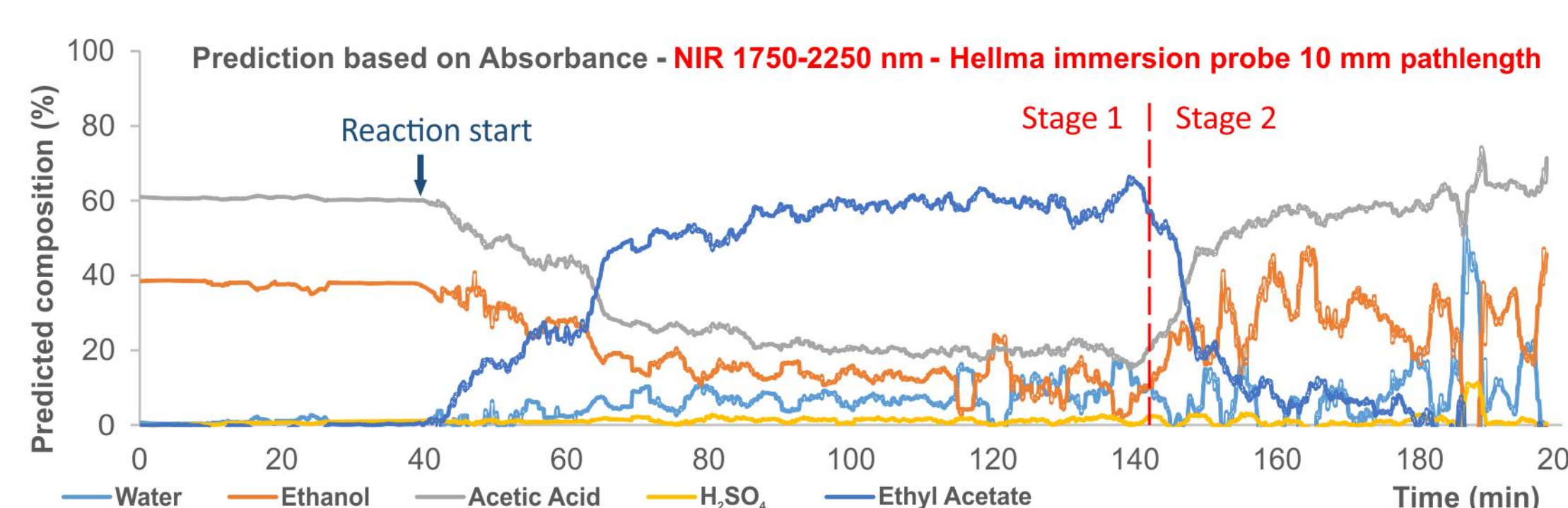
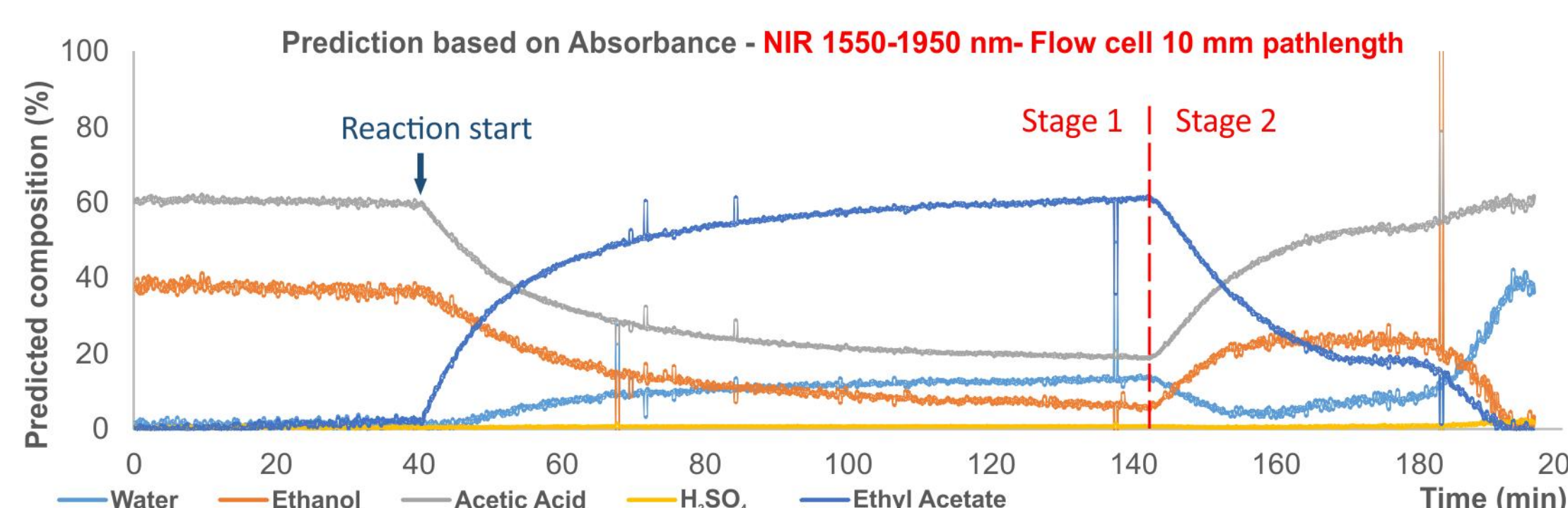
Model reaction 1. Ethanol esterification and product recovery



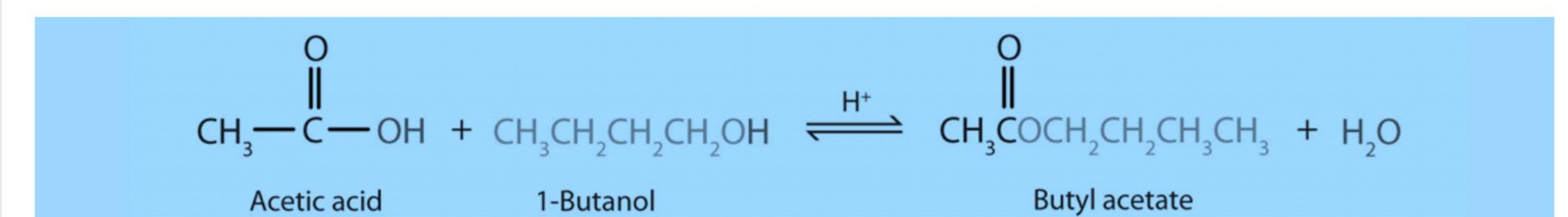
Reacting volume: ~1000ml
Temperature ~67-75°C, 1atm
Ethanol: Acetic Acid 1:1.3

Stage 1: Under reflux condition

Stage 2: Distillation (bottom monitoring)



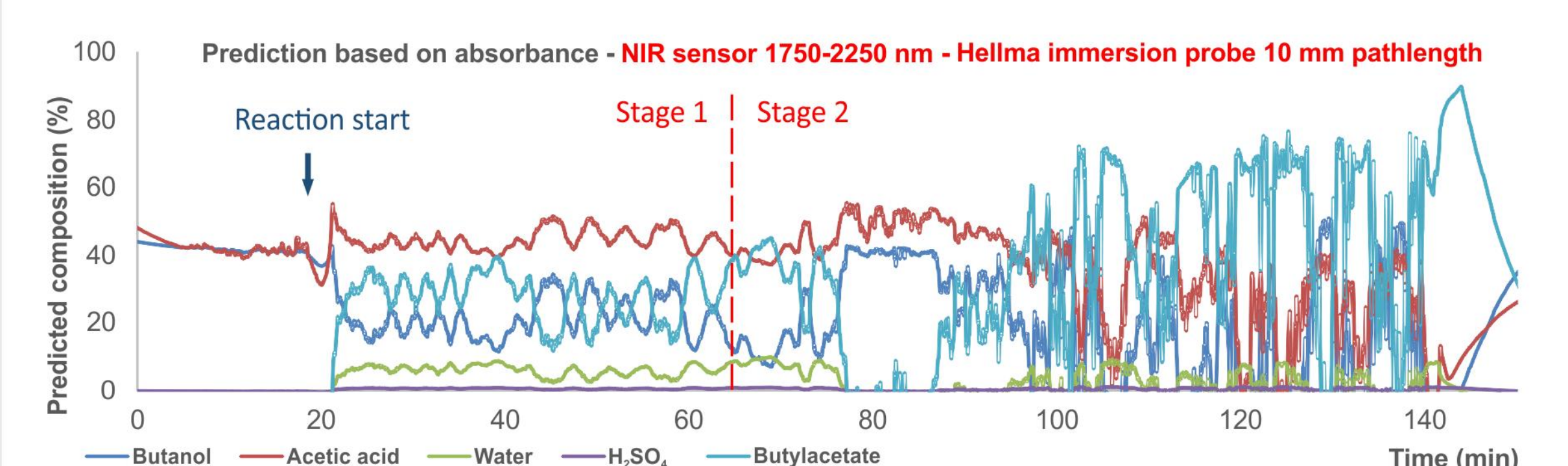
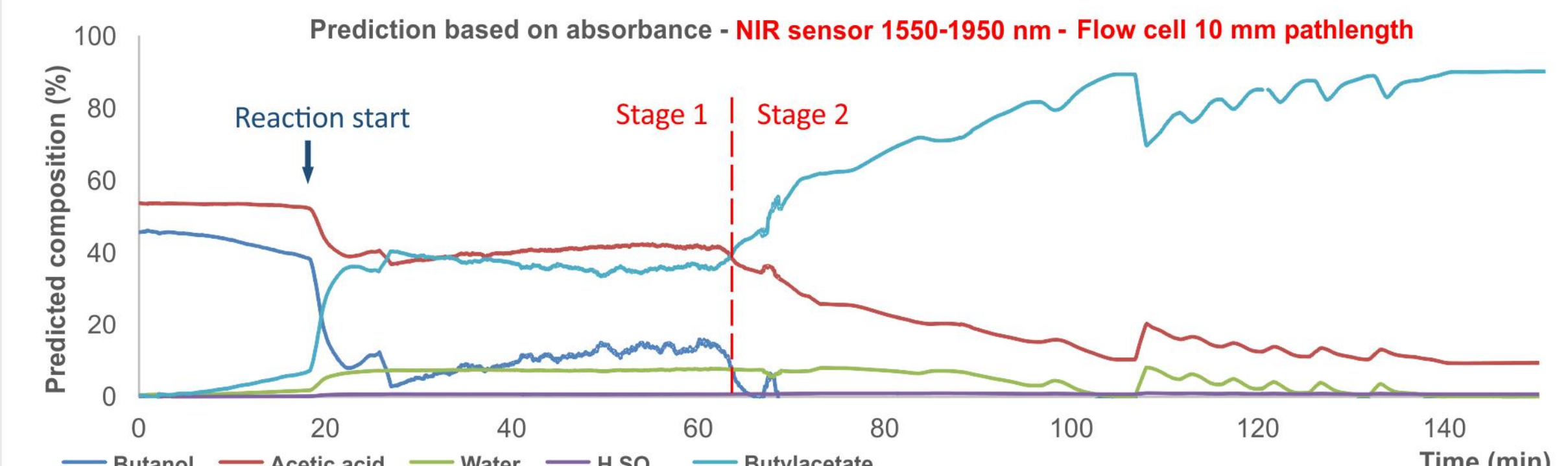
Model reaction 2. Butanol esterification and product recovery



Reacting volume: ~1000ml
Temperature ~100-130°C, 1atm
Butanol: Acetic Acid 1:1.3

Stage 1: Under reflux condition

Stage 2: Distillation series (Azeotropic mixture)



PARTNERS:



The research project receives funding from the European Community's Framework programme for Research and Innovation Horizon 2020 (2014-2020) under grant agreement number 637232