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Haute Ecole Spécialisée de Suisse occidentale
Fachhochschule Westschweiz
University of Applied Sciences and Arts Western Switzerland

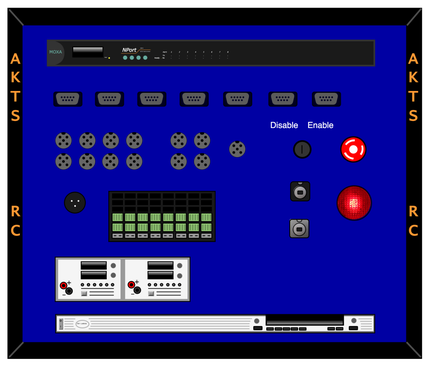
Master of Science HES-SO in Life Sciences

INVESTIGATION OF THE COURSE OF CHEMICAL REACTION DURING CHANGE OF THEIR SCALE FROM GRAMS TO TONS USING REACTION CALORIMETRY APPROACH

Damiano Rickenbach
CHEMICAL DEVELOPMENT & PRODUCTION
EIA-FR
Advisor: Olivier Vorlet // In collaboration with **AKTS SA**

DESCRIPTION

Project based on the study of heat flows, which take place during a chemical reaction. The AKTS MSRC acquisition system, which consists of both an hydraulic and controller module and a control software was used to perform a scale up from a 1 liter reactor, to 5, 10 liters and finally up to a reactor scale of 60 liters.



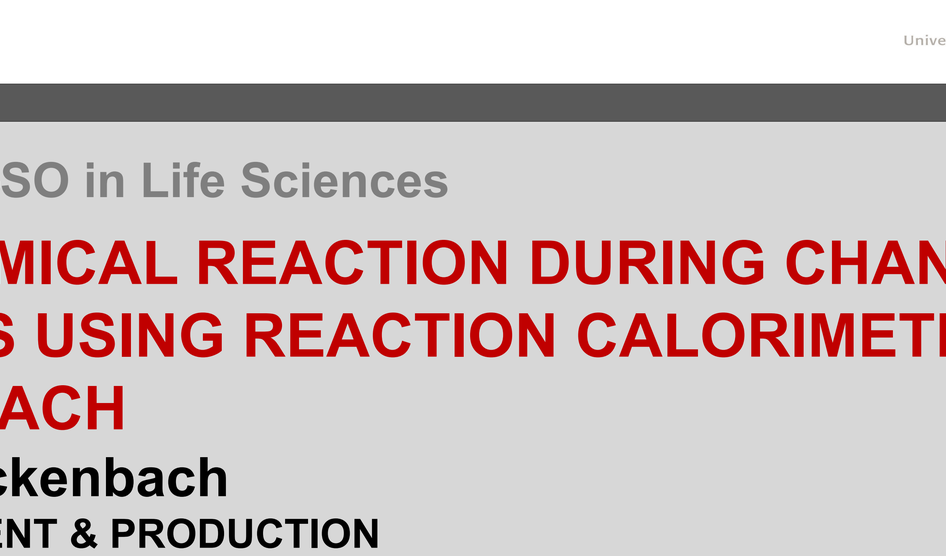
AKTS MSRC acquisition box

A study of measurement repeatability was performed, testing three new calorimetry modes developed by AKTS: Power & Jacket Compensation (PJC), Power & Power Compensation (PPC) and Jacket & Jacket Compensation (JJC).
Simulated chemical reactions, dosing water into water, were carried out to characterise the system (calibration). Then, with the obtained results, it was possible to measure the heat release by the simple chemical reaction between hydrochloric acid and sodium hydroxide ($\triangle H_r$), as well as the specific heat capacities (Cp) of the sodium hydroxide solution, respectively of the sodium chloride formed solution. The dynamic and regulation parameters characterising the 1, 5 and 10 liters reactors were used to mathematically extrapolate these parameters for a 100 liters homothetic reactor. Simulations were performed to verify the feasibility of calorimetric analysis on a 100 liters scale.
To perform calorimetry studies on the 60 liters reactor in the ChemTech laboratories, a stainless steel plate heat exchanger was added to the inlet of the jacket circuit of the reactor. The AKTS hydraulic module was then connected to the heat exchanger, thanks to which it was possible to control the temperature inside the reactor without compromising the availability of the installation. A LabView program, employing MQTT protocol to transfer the signals of the temperature sensors inside the reactor (Tr) and at the inlet and outlet of the jacket (Tj IN and Tj OUT), was developed allowing *the AKTS-MSRC software* to collect the data in real time.
Some promising preliminary tests were carried out.

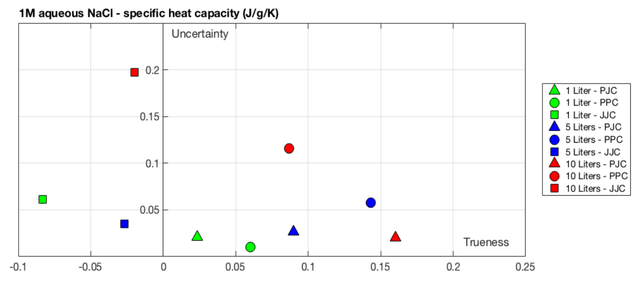
OBJECTIFS

- Acid base reaction
  - Comparison with literature
  - Comparison with simulation
- Evaluating repeatability of the process
- Scale-up reactor and reactor calibration limits investigation
- Larger scale process simulation

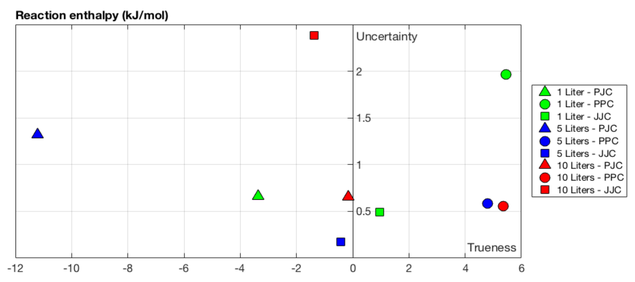
RESULTS



With the current system, the determination of specific heat capacity of NaOH, gives better results in JJC mode and on a 5 liter scale. Measurements with a 10 liter reactor on PPC and JJC modes also give reliable values, although a little less accurate. The 1 liter reactor, on the other hand, has been shown to have the most variability, both in terms of precision and accuracy.



The figure shows that obtained results with 1 liter reactor, on all calorimetry modes, allow results with less than 0.1 J/g/K uncertainty and trueness to be obtained. Compared to the determination of the specific heat of NaOH, all the measured results are tendentially more precise and accurate. The JJC mode performed on the 10 liters reactor provided the least precise, but most accurate data. In contrary, PJC mode, again applied to measurements with the 10 liters reactor, gave the least accurate but precise results.



The determination of the heat of reaction has been also performed. The determination of the heat released by a given chemical reaction gave more reliable results with the JJC mode for the 1 and 5 liter reactor. When performing a reaction with a 10 liter reactor, the PJC mode was the most suitable.

CONCLUSION

A new design of AKTS calorimetry system's hydraulic module has been built and tested, as well as a new heat flow calculation procedure, also developed by AKTS.
An acid-base reaction was carried out under the conditions described above and the obtained results were compared with the reference values. The three new calorimetry modes, developed by AKTS, were tested and the repeatability of the process was evaluated for both the basic acid reaction and the simulated reaction, for the scale of 1, 5 and 10 liter reactor.
Results showed that the AKTS calorimetry system allows to precisely and accurately measure the heat flows that take place during a chemical process up, to the pilot scale. The average value of reaction enthalpy for the reaction between sodium hydroxide and hydrochloric acid, and for all the reactions performed on all the scales, has been calculated and is 56.86 kJ/mol.
The factor that has the greatest influence on repeatability is the variation in ambient temperature between measurements.
From the point of view of the scale-up limits of the reactor and the calibration of the reactor limits investigation, the scale-up was successfully carried out from 1 to 5 and then to 10 liter.