MOBILE NIR IN-LINE DEVICE FOR MONITORING OF SUPERCRITICAL CO_2 BASED PROCESSES

G. Wiegand*, S. Johnsen, S. Michel
Forschungszentrum Karlsruhe GmbH, ITC-CPV, P.O.B. 3640, D-76021, Germany,
gabriele.wiegand@itc-cpv.fzk.de, Fax: 0049 7247 82 22 44
B. Zehnder
SITEC-Sieber Engineering AG, Aschbach 621, CH-8124 Maur/Zurich, Switzerland

Supercritical CO_2 is widely used as solvent for separation processes and chemical reactions. Here, in-line tools for monitoring concentrations of chemical species involved indicating the progress of e.g. an extraction process or a chemical reaction, and for control of constant concentrations of substances prior to supercritical particle formation processes are often desired, but not readily available.

Accordingly, the presented NIR in-line monitoring device SINASCO[1] has been developed as a fast, continuous and non-invasive analysis and characterization method for extraction or reaction constituents. It can be operated in batch or continuous circulation mode up to 500 °C and 100 MPa maximum operating conditions. Fiber-optic adapters allow to connect the high-pressure view cell to the FT-NIR spectrometer. The Partial Least Squares Calibration features facilitate the calibration within the temperature-pressure-concentration area of interest compared with the classical methods.

In a technology transfer project between Forschungszentrum Karlsruhe and SITEC-Sieber Engineering AG the NIR in-line monitoring set-up is developed from a stationary laboratory apparatus to a mobile demonstration unit, which is easy to handle, allows for coupling of the instrument to an industrial process unit and will be commercially available, soon.

In industrial applications like CO_2 based extraction processes this in-line monitoring system is a useful tool to lower the production costs by monitoring for the selectivity and for the efficient termination of the extraction procedure (e.g. hops, spices, essential oils, decaffeination, transformation from rice starch, cleaning of metal parts, removing pesticides from rice).

Also for CO_2 based reactions, the NIR in-line monitoring is useful for observing chemical reactions and allow for modeling in regard to process optimization.

As an example, for the cleaning process with supercritical CO_2, where cutting or grinding oils are to be removed from metal parts or metal working residues, the calibration with the reference system squalane/CO_2 proofed to be valid for defining an efficient termination criterion according to which the de-oiling process can be stopped saving time and energy [1, 2, 3]. The autoxidation of aldehydes to carboxylic acids in supercritical CO_2 has also been monitored to control the progress of chemical reactions and to collect data for kinetic modeling of the process [4].

Measurements on customer's demand are available for extraction and reaction processes in supercritical CO_2.

[1] G. Wiegand, J. Bürck, S. Roth, H. Mathieu, K. Krämer, Chemical Engineering Transactions. ***2002***, 2; 579-584
[2] J. Bürck, G. Wiegand, S. Roth, H. Mathieu, K. Krämer, Journal of Near Infrared Spectroscopy. **2004**, 12, 29-36;
[3] J. Bürck , G. Wiegand, S. Roth, H. Mathieu, K. Krämer, TALANTA. **2006**, 68 (5): 1497-1504
[4] G. Wiegand, H. Ederer, J. Bürck, S. Roth, H. Mathieu and K. Krämer, ISSF **2005**, May 1–4 Orlando, FL (2005) (#217, on CD).

[1] Set-up for in-line NIR spectroscopic analysis in supercritical carbon dioxide