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MOBILE NIR IN-LINE DEVICE FOR MONITORING OF SUPERCRITICAL CO₂ BASED PROCESSES

Outline

What we are doing



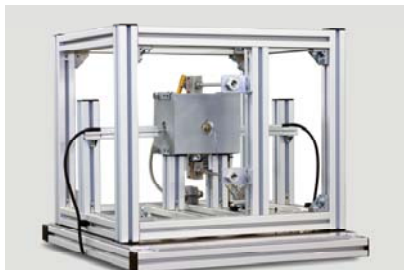
In-line monitoring for
 • Extraction: **Make processes more selective and efficient**
 • Reactions: **Measure kinetic data for process optimization**



• In-line NIR spectroscopy
 • On-line GC for solubility data necessary for NIR calibration



✓ Extraction in supercritical CO₂
 > Selective extraction, efficient termination
 (Example: **De-oiling of metal parts**)
 ✓ Reaction in-line-monitoring
 > Kinetic data for process optimization
 > Process control e.g. for safety reasons or selectivity
 (Example: **Partial oxidation of hexanal to hexanoic acid**)



State-of-the-Art

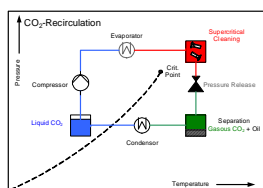
- Standard cells in market: 300°C at 25 bar OR 50°C at 300 bar
- Demand for extraction: 40-80°C up to 750 bar
- Demand for reaction: 40-200°C up to 350 bar (or even higher)



SINASCO: max. 180°C, max. 1000 bar

Examples

Cleaning Process using Supercritical CO₂

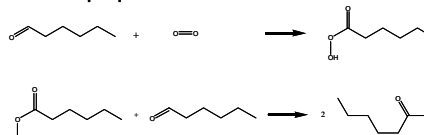


- ✓ Replaces problematic solvents
- ✓ De-Oiling of abrasive slurry (glass, metal)
 > **Recycling of glass and metal**
- ✓ Cleaning of metal formed components
 > **Clean parts**
- ✓ Recycling of cutting and grinding oils
 > **Limit for de-oiling: 0.5%**

N. Dalmen, J. Schön, and E. Dinjus, Proceedings of the 7th International Symposium on Supercritical Fluids, Vol. 1, 2000, 369.
 J. Schön, K. Buchmüller, N. Dalmen, P. Grottelner, P. Schwab, H. Wild, Wissenschaftliche Berichte, FZKA-6759 (October 2003)

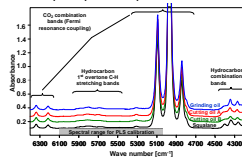
Autoxidation for Synthesis of C4-C13 Carboxylic Acids

So far: **Liquid phase oxidation with air in solvent: 2 Phases**

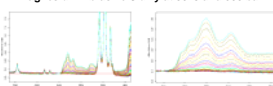


New: Homogeneous reaction in supercritical CO₂

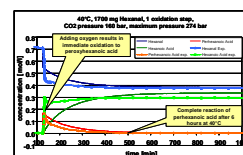
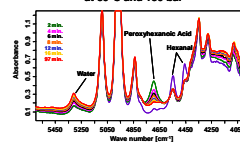
NIR Absorption Spectra of Squalene and Real Oils in CO₂



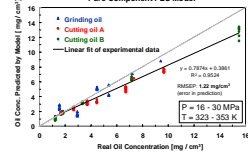
In-line-Monitoring for Supercritical CO₂ Cleaning of Magnesium Abrasive Slurry at 50°C and 300 bar



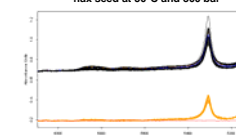
NIR Spectra of Hexanal Oxidation with Synthetic Air at 80°C and 180 bar



Correlation of Real and Predicted Cutting Oil Concentration Using the Pure Component PLS Model



In-line Monitoring for Supercritical CO₂ Extraction of flax seed at 50°C and 300 bar



Résumé

Benefit

- In-line-Monitoring system
 - Compact
 - Mobile
 - Flexible
- Easy, fast integration into available extraction plant
- Savings in costs for resources
- Faster processing of orders
- Higher production rates

Potential Applications

(Source: Internet Pages DEGUSSA, Uhde, Natex, Separex)

- Hops: Extraction control, termination
- Cocoa: Hops: Extraction control, termination
- Nutmeg: Selective extraction for detection of oil/butter transition
- Spices, Rape: Selective extraction, extraction control, termination
- Pepper: Selective extraction, extraction control, termination
- Essential Oils: Selective extraction, extraction control, termination
- Cork: Extraction of TCA* in ppm Range
- Coffee, Tea: Extraction control, termination
- Rice: Removal of pesticides, conversion of starch (big market in Asia)
- Determination of loading curves for unknown systems by in-line measurements
- Hostalen-Production at Dupont in USA: Reaction in supercritical CO₂

*2,4,6-trichloroisocyanate

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