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Sensitivity analysis of a control-oriented model of supercritical extraction

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What are advantages of supercritical fluid extraction?

- CO_2 non-flammable and non-toxic
- properties of CO_2 can be 'tuned' with operating conditions
- the critical point of CO_2 is easily accessible
- high selectivity
- easy separation of the solvent (if there is no co-solvent)

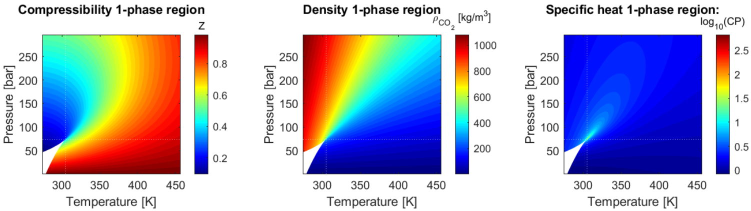

Figure 1: Properties of CO_2

SFE - assumptions

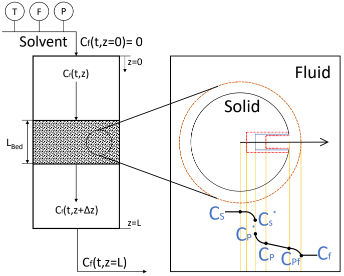

Figure 2: Schematic representation of SFE process

Assumptions:

- Quasi-one-dimensional model
- Negligible external diffusion
- Low-Mach number assumption
- No pressure drop
- Pseudo-single component
- Negligible thermal properties of solid phase
- Uniform distribution of the particles
- Varying void fraction along the extractor

Model of an extractor

(1) Fluid phase mass balance [1]

$$\frac{\partial c_f(t,z)}{\partial t} + \underbrace{\frac{1}{\phi}\frac{\partial\left(c_f(t,z)u\right)}{\partial z}}_{Convection} = \underbrace{\frac{1-\phi}{\phi} r_e(t,z)}_{Kinetic} + \underbrace{\frac{1}{\phi}\frac{\partial}{\partial z}\left(\boxed{D_e^M}\frac{\partial c_f(t,z)}{\partial z}\right)}_{Diffusion}$$

(2) Solid phase mass balance [1]

$$\frac{\partial c_s(t,z)}{\partial t} = -\underbrace{r_e(t,z)}_{Kinetic}$$

(3) Heat balance [2]

$$\frac{\partial\left(\rho_f(T(t,z),P(t))h(t,z)A_f\right)}{\partial t} - \frac{\partial\left(P(t)A_f\right)}{\partial t} = \underbrace{-\frac{\partial\left(\rho_f(T(t,z),P(t))h(t,z)A_f v\right)}{\partial z}}_{Convection} + \underbrace{\frac{\partial}{\partial z}\left(k\frac{\partial T(t,z)}{\partial z}\right)}_{Diffusion}$$

Extraction kinetic [1]

$$r_e(t,z) = -\frac{\boxed{D_i}}{\mu l^2}\left(c_s(t,z)\boxed{k_m} - c_f(t,z)\right)$$

Output function [3]

$$y(t) = g(x(t)) = \int_0^{t_f} Q(t)c_f(t,z)|_{z=L}dt = \int_0^{t_f} u(t)Ac_f(t,z)|_{z=L}dt = \iff \frac{\partial y(t)}{\partial t} = u(t)Ac_f(t,z)|_{z=L}$$

Equation of state - Peng-Robinson

$$P\left(T(t,z),\rho(t,z)\right) = \frac{RT(t,z)}{v(t,z)-b} - \frac{a}{v(t,z)(v(t,z)+b)+b(v(t,z)-b)}; \quad v(t,z) = \frac{1}{\rho(t,z)}$$

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[1] E. Reverchon, Mathematical modeling of supercritical extraction of sage oil, AIChE J 42 (6), 1996

[2] John D. Anderson. Computational fluid dynamics the basic with applications. McGraw-Hill, 1995

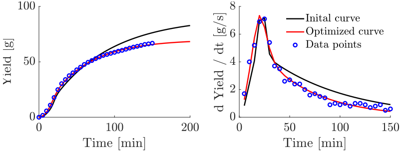
[3] H. Sovova, R. Komers, J. Kucuera, and J. Jezu. Supercritical carbon dioxide extraction of caraway essential oil. Chemical Engineering Science, 1994

Model discretization

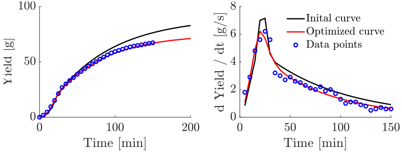
$$\frac{dx(t)}{dt} = \begin{bmatrix} \frac{dc_{f_1}(t)}{dt} \\ \vdots \\ \frac{dc_{f_{N_z}}(t)}{dt} \\ \frac{dc_{s_1}(t)}{dt} \\ \vdots \\ \frac{dc_{s_{N_z}}(t)}{dt} \\ \frac{dh_1(t)}{dt} \\ \vdots \\ \frac{dh_{N_z}(t)}{dt} \\ \frac{dP(t)}{dt} \\ \frac{dy(t)}{dt} \end{bmatrix} = \begin{bmatrix} F_1\left(c_f(t), c_s(t), T(t), y(t); \Theta\right) \\ \vdots \\ F_{N_z}\left(c_f(t), c_s(t), T(t), y(t); \Theta\right) \\ F_{N_z+1}\left(c_f(t), c_s(t), T(t), y(t); \Theta\right) \\ \vdots \\ F_{2N_z}\left(c_f(t), c_s(t), T(t), y(t); \Theta\right) \\ F_{2N_z+1}\left(c_f(t), c_s(t), T(t), y(t); \Theta\right) \\ \vdots \\ F_{3N_z}\left(c_f(t), c_s(t), T(t), y(t); \Theta\right) \\ F_{3N_z+1}\left(c_f(t), c_s(t), T(t), y(t); \Theta\right) \\ F_{3N_z+2}\left(c_f(t), c_s(t), T(t), y(t); \Theta\right) \end{bmatrix} = F\left(t; \Theta\right)$$

where $x \in \mathbb{R}^{N_x = 3N_z + 2}$ and $\Theta \in \mathbb{R}^{N_\Theta = N_\theta + N_u}$

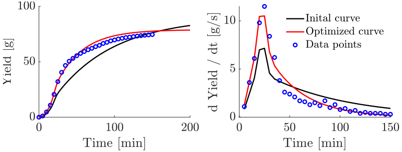
Results of the simulation



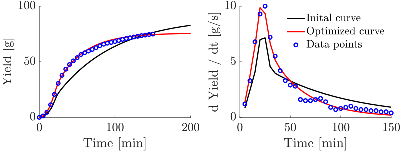
(a) Experiment at $40^\circ C$ and 200 bar



(b) Experiment at $50^\circ C$ and 200 bar



(c) Experiment at $40^\circ C$ and 300 bar



(d) Experiment at $50^\circ C$ and 300 bar

Sensitivity Analysis

Local derivative-based methods involve taking the partial derivative of the output with respect to an input parameter. In sensitivity analysis the original system of equations is solved simultaneously with $\frac{\partial F(x(t);\Theta)}{\partial \Theta}$, where Θ is a vector of all the parameters and the control variables.

$$Z = \frac{\partial x(t,\Theta)}{\partial \Theta}$$

$$\dot{Z} = \frac{\partial Z(t,\Theta)}{\partial t} = \frac{\partial}{\partial t}\left(\frac{\partial x(t,\Theta)}{\partial \Theta}\right) = \frac{\partial}{\partial \Theta}\left(\frac{\partial x(t,\Theta)}{\partial t}\right) = \frac{dF(x(t);\Theta)}{d\Theta}$$

$$\begin{aligned}\frac{dF(x(t);\Theta)}{d\Theta} &= \frac{\partial F(x(t);\Theta)}{\partial x(t)}\frac{\partial x(t)}{\partial \Theta} + \frac{\partial F(x(t);\Theta)}{\partial \Theta} \\ &= J_x(x(t);\Theta)S(x(t);\Theta) + J_\Theta(x(t);\Theta)\end{aligned}$$

$$\underbrace{\boxed{\frac{dF(x(t);\Theta)}{d\Theta}}}_{(N_x \times N_\Theta)} = \underbrace{\boxed{\frac{\partial F(x(t);\Theta)}{\partial x(t)}}}_{(N_x \times N_x)}\underbrace{\boxed{\frac{\partial x(t)}{\partial \Theta}}}_{(N_x \times N_\Theta)} + \underbrace{\boxed{\frac{\partial F(x(t);\Theta)}{\partial \Theta}}}_{(N_x \times N_\Theta)}$$

[4] T. Maly, L. R. Petzold, Numerical methods and software for sensitivity analysis of differential-algebraic systems, Applied Numerical Mathematics 20 (1-2), 1996

Results of the sensitivity analysis - Internal diffusion

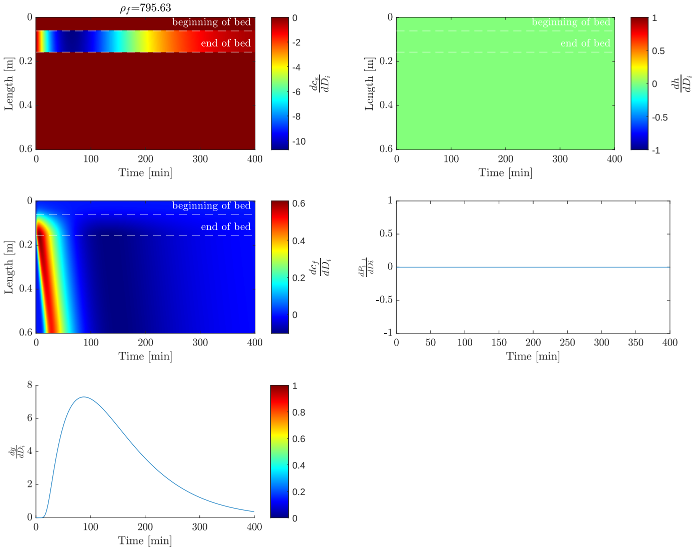


Figure 4: $50^{\circ}C,\ 200\ bar$

Results of the sensitivity analysis - Flow-rate

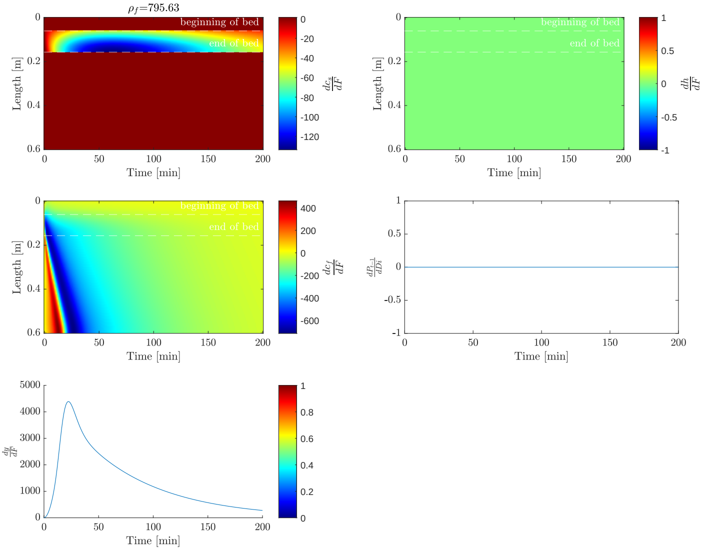


Figure 5: $50^{\circ}C,\ 200\ bar$

Results of the sensitivity analysis - Flow-rate

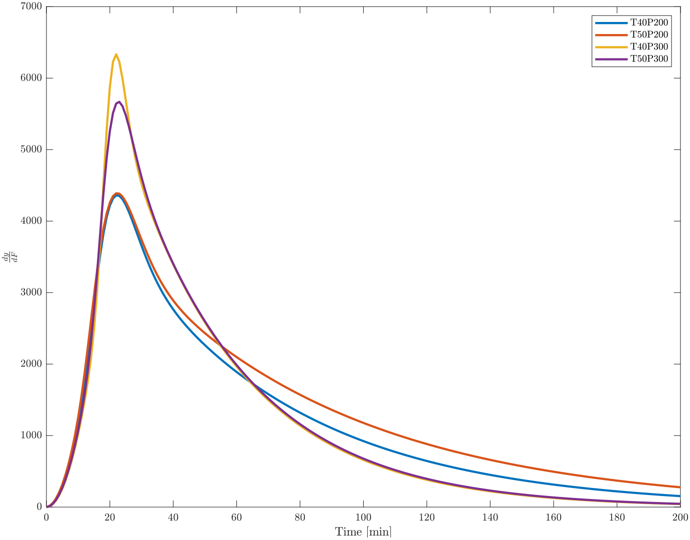


Figure 6: $40 - 50^\circ C,\ 200 - 300\ bar$

Conclusion

The forward sensitivity analysis allows to

- better understanding of the analysed model
- evaluated an influence of controls and parameters
- identify sensitive and none-sensitive parameters
- spot modelling errors