NONISOTHERMAL DYNAMIC MODEL OF A SFE STRUCTURED PACKING COLUMN

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INTRODUCTION

This paper presents a dynamic simulation model for a countercurrent structured packed column running at supercritical fluid conditions. The model accounts for the heat transfer throughout the column and axial effects on temperature profiles are considered. The model is applied to a case study involving the fractionation of a binary mixture of squalene and methyl oleate with supercritical carbon dioxide. The physical properties of the fluid phases were estimated based on data taken from literature. For each physical property a correlation was developed in terms of pressure, temperature and phase compositions. The validation of the model is made by carrying out a series of experiments in a lab-scale SFE apparatus. Disturbances on selected load variables and operation parameters are applied and the dynamic response of the SFE unit tracked.

The reason for developing a dynamic model of a supercritical fluid extraction (SFE) process is that the design and analysis of these processes requires reliable computation tools. By doing a dynamic study one can obtain information on the behaviour of the whole process when subjected to changes in the input variables. With all the information obtained we can also optimize the process making it more efficient.

The purpose of the current work is to develop a dynamic simulation model of a SFE column for the extraction of liquid mixtures. The proposed model is being validated by performing a series of extraction experiments in our lab-scale extraction apparatus, whose internals are filled with structured gauze packing elements. As the process feed we used a mixture of squalene and methyl oleate to partly emulate olive oil residues obtained from the olive oil refining process. The dynamic model is being developed using the commercial process simulation software package gPROMS®. The model incorporates a set of differential equations corresponding to material and energy balances over the column and the algebraic equations that describe the heat and mass transfer kinetics of the process, the hydrodynamic behaviour of the packing at high pressure conditions and the thermodynamic equilibrium of the system. GPROMS is used to solve these equations in order to time and axial position.[1]. The thermodynamic relations, the mass and heat transfer and the hydrodynamic equations were developed from experimental data previously collected by Ruivo [2-4]. The physical and transport properties of the gas and liquid phases are allowed to vary axially and with time. The input variables of the model are the initial pressure and temperature of operation, and the flowrate and composition of the supercritical solvent and liquid feed streams entering the packed column

The differential global material balances the gas and liquid phases are as follows:

$$A\varepsilon \frac{\partial\left[\left(1-h_L\right)\rho_G\right]}{\partial t} = \frac{\partial G}{\partial z} \tag{1}$$

$$A\varepsilon \frac{\partial\left[h_L\rho_L\right]}{\partial t} = \frac{\partial L}{\partial z} \tag{2}$$

Where L and G are the liquid and gas phase molar flowrates, z and t are the dimensional time and axial position variables and h_L is the local liquid holdup.

Concerning the energy balances for gas and the liquid phase, the radial temperature profiles in the column were neglected as a first approximation. The heat transfer to the column wall is taken into account by an effective heat-transfer coefficient h_W based on the local temperature difference between the structured packing and the wall. The gauze packing is assumed to be in thermal equilibrium with the liquid phase due to a uniform thin layer of wetting liquid film. Neglecting the axial conductive heat transport, the differential energy balance in the gas phase can be written as:

$$A\varepsilon\left(1-h_L\right)C_{pg}\rho_g \frac{\partial T_g}{\partial t} + C_{pg}G\frac{\partial Tg}{\partial z} - \frac{A\varepsilon\left(1-h_L\right)}{\tau_s}\lambda_g \frac{\partial T_g^2}{\partial z^2} = aeh_g\left(T_s - T_g\right) \tag{3}$$

Where A is the internal cross-section area of the column, ε and ae are the porosity and specific area of the packing, and h_g is the local heat transfer coefficient for the gas. T_g, G, ρ_g, λ_g and C_{pg} denote respectively the temperature, mass flow, density, thermal conductivity, and specific heat of the gas phase. τ_s is the packing turtuosity. The second derivative of the temperature in order to spatial coordinate accounts for the thermal dispersion inside the column.

The differential energy balance for the liquid plus packing medium is:

$$A\left[\varepsilon h_L C_{pl}\rho_l + \left(1-\varepsilon\right)C_{ps}\rho_s\right]\frac{\partial T_l}{\partial t} + C_{pl}L\frac{\partial Tl}{\partial z} - A\varepsilon\lambda_{zeff}\frac{\partial T_l^2}{\partial z^2} = aeh_g\left(T_g - T_l\right) + 2\pi Rh_W\left(T_W - T_l\right) \tag{4}$$

Where R is the column internal radius, h_W is the packing to wall heat transfer coefficient, T_W is the wall temperature, and ρ_s and C_{ps} are the packing density and specific heat, respectively. L, ρ_s and C_{ps} denote the mass flow, density and specific heat of the liquid phase. λ_{zeff} is the effective thermal conductivity of the liquid plus packing medium; again, the second derivative of the temperature accounts for the thermal dispersion in the column.

1 – MATERIALS AND METHODS

Experimental data is acquired in a lab-scale SFE apparatus described elsewhere [2]. The extraction column is filled with Sulzer EX structured gauze packing. The apparatus allows the possibility to acquire online data on pressure, temperature and mass flows throughout the plant.

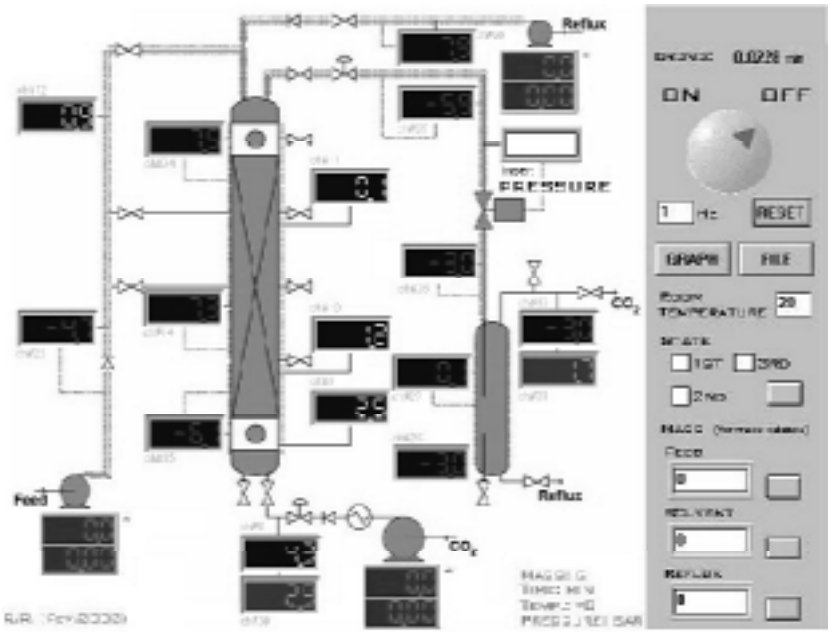

Fig. 1: Data aquisition layout

The column is divided in three zones; a heating tape with the help of a temperature controller heats each zone.

The validation of the models is made by comparing the temperature and composition profiles in the column with the ones predicted by the simulator.

Figure 2 shows a temperature profile of the packed column at 18 MPa and 4.92 kg/h of CO_2 mass flowrate, Bottom, Middle and Top designate the temperature profiles in the column at the bottom, middle and top of the column.

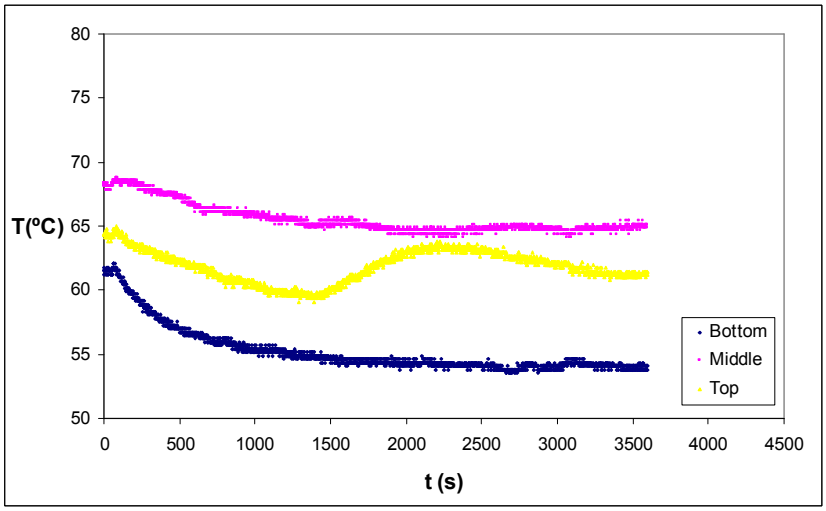

Fig. 2: Temperature profiles in the extraction column

Figure 3 presents a comparison between the temperature of the gas exiting the column obtained experimentally with the predicted by the present model:

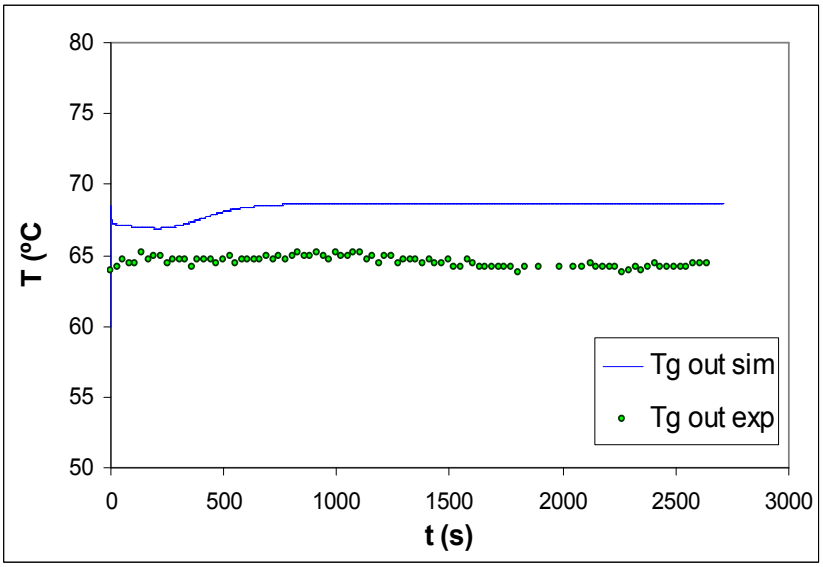

Fig. 3: Gas outlet temperature; experimental and predicted data

The simulation model is currently being improved by comparing both the experimental temperature and composition profiles through the packed column with the predicted ones.

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