

Design and Analysis of a Classical Controller to the Residual Oil in a Small Scale Semibatch Extractor

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Abstract

The main effects of the variables concerned with the isothermal and isobaric supercritical extraction of grape oil are determined using a two levels factorial design. It is verified that the solvent velocity can be manipulated to control the residual oil content in the solids. Disturbances in the mean diameter of the particles can not be controlled during the extraction process. Parameter sensitivity supported the design and the performance investigation of the classical controller to the residual oil in the solid material. The proposed procedure was able to control the extraction process under individual and simultaneous step disturbances in the initial content of oil in the solids and in the solvent inlet concentration.

Keywords: Supercritical Extraction, Semibatch Extractor, Classical Controller

1. Introduction

The solvent extraction process for oilseeds has been considered the most efficient way for extracting vegetable oils from its solid matrixes. The success of this technology is due to its ability in reducing to a minimum the residual oil in the extracted solid material. The hexane has been the widest used solvent, but because of its toxicity and flammability there exist incentive to replace it. Supercritical carbon dioxide has been successfully experienced for extracting natural products, due to its atoxicity, chemical stability, availability and low costs. While too much attention has been directed to know the supercritical - CO₂ performance for extracting a variety of oil-bearing materials, less effort has been dedicated to the optimization and to the control of this extractive process. Several approaches have been proposed and analysed for control of semibatch processes (Garcia-Munoz et. al 2004; Asteasuain et. al. 2004; Srinivasan et. al., 2003; Grau and Puigjaner, 2000; Flores-Cerrillo and MacGregor, 2004) and can be identified the benefits of such approaches to operate the systems at higher performance when compared to manual operation. However, the CO₂ semibatch extractors have peculiar characteristics, specially in their quick dynamic and operating variable interactions, so that the results and conclusions of other types of system may not be directly used. It is important to consider that a good control strategy may lead the process to be operated at high performance, which has a direct impact on the process viability, specially for supercritical extraction, that is an expensive process. The present work intends to contribute to this unexplored field through parametric sensitivity analysis and

investigation of the performance of a classical controller. The first requisite to accomplish this analysis is the availability of a mathematical model and, so, extensive simulation can be carried out.

2. Supercritical Extraction Model

A review on the mechanisms of the solvent extraction process of oilseeds showed that there are at least four steps that can potentially dictate the rate at which the oil is removed from its solid matrixes. They are: 1-solvent penetration in the intraparticle pores; 2-dissolution of the oil adhered to the solid particles by the solvent that fulfill the intraparticle pores; 3-diffusion of the dissolved oil through the solvent in the intraparticle pores until the particle surface; 4-convection of the oil from the surface of the solid particles to the bulk phase of the solvent. A mathematical extraction model that includes all the steps mentioned above requires a considerable number of parameters. A common procedure (Sovová, 1994 and Sovová et al. 1994), here also adopted, is to take in consideration only the two last steps. The following considerations, in addition to the above mentioned, are made: the extraction occurs in a fixed bed of solid particles; the solvent flows continuously through the bed of solids; the oil is treated as a single specie and no axial dispersion and isothermal and isobaric operation are considered. The equations describing the model are composed by material balances of oil in the solid and in the fluid phases:

$$\text{Solid Phase: } \rho_s(1-\varepsilon)\frac{\partial x}{\partial t} = J(x, y) \quad (1)$$

$$\text{Fluid Phase: } \rho_f \varepsilon \frac{\partial y}{\partial t} + \rho_f U \frac{\partial y}{\partial h} = J(x, y) \quad (2)$$

where: t -time of extraction ; ρ_s -density of the solid particles; ε -bed porosity ; x -oil mass fraction in the solids in an oil free basis ; y -oil mass fraction of the solvent in an oil free basis; $J(x,y)$ -rate of extraction; ρ_f -solvent density ; U -solvent velocity ; h -axial distance in the bed. $J(x,y)$ depends on a critical residual oil concentration in the solid particles, x_k . This concentration makes the transition of a period of extraction dictated by convection in the film surrounding the solid particles to a period dictated by diffusion through the solvent inside the particles. The fast extraction rates, in the convection period, are described by:

$$J(x, y) = k_f a_0 \rho_f (y_r - y) \quad (3)$$

when $x \geq x_k$; y_r - oil solubility in solvent; $k_f a_0$ - mass transfer coefficient based on the solvent phase. The slow extraction rates, in the diffusion period, are described by:

$$J(x, y) = k_s a_0 \rho_s x(1 - y/y_r) \quad (4)$$

when $x \geq x_k$; $k_s a_0$: mass transfer coefficient based on the solid phase.

Equations (1), (2), (3) and (4) are subjected to the following initial and boundary conditions: $x(h, t = 0) = x_0$, $y(h = 0, t) = y_0$, $y(h, t \leq t_{lim}) = y_r$

where: x_0 -initial oil content in the solids; y_0 -inlet solvent oil content; y_r -equilibrium solubility of the oil in the solvent; t_{lim} -minimum time required to the no saturated solvent reach a point h of the bed. Equations (1) to (4) were solved numerically with the fourth order Runge-Kutta method in respect to time ($\Delta t = 0.1$ s) and finite differences were used for the spatial coordinate discretization ($\Delta h = 0.00005$ m). Values for the mass transfer parameters may be found in Sovová et al. (1994) for the supercritical extraction of grape oil. The pressure is 280 bars and the temperature is 40°C. The laboratory scale extractor is 5.0 cm of useful length.

3. Solvent and Solid Oil Contents at the Extractor Exit

Important information for the equipment design as well as for the definition of the operating strategy is that related to the extraction time regarding to the residual oil contents, in both, solids and solvent. Figures 1 and 2 show, respectively, the residual oil in the solids and in the solvent at the extractor exit as a function of the extraction time. The magnitude of the numerical errors in presented in the right y-axis of Figure 1 (dashed line). In terms of extraction, it can be seen that in the initial phase of the convection period, the extraction is very slow, as consequence of the high solvent oil content. As soon as fresh solvent achieves the extractor exit, the extraction becomes faster. This zone, in the convection period, characterized by fast rates of extraction, is called extraction front region. When the residual oil critical concentration in the solids is attained, at the diffusion period of extraction, the extraction rate decreases, and the residual oil content in the solids changes very slowly. The design and performance evaluation of control strategies require the knowledge of the effects of the variables concerned with the extraction process. To ensure that the obtained effects are meaningful in a computational solution, it is important to know the magnitude of the numerical errors. As can be seen in Figure 1, the relative numerical errors in the computation of the residual oil in the solids is smaller than about 2.5%.

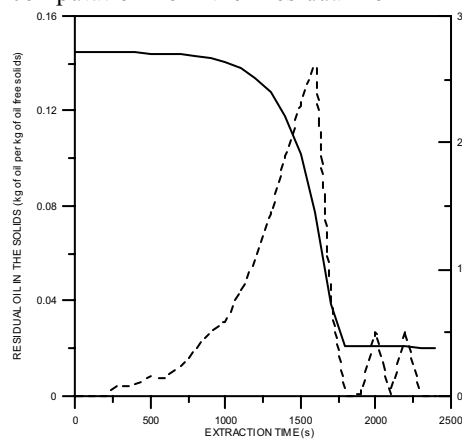


Figure 1. Solid residual oil at the extractor exit.

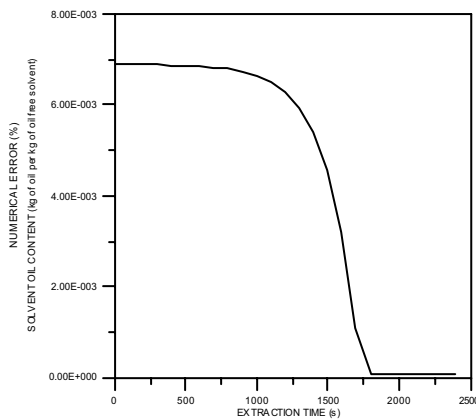


Figure 2. Solvent oil content at the extractor exit as a function of extraction time.

4. Parameter Sensitivity Analysis

The objective of this section is to understand how the variables affect the residual oil content in the solids (time dependent) at the extractor exit. The variables analyzed were: mean solvent velocity (U); oil mass fraction of the solid material (x_0); oil mass fraction of the solvent at the extractor entrance (y_0), and mean solid particle diameter (dp). The values of the variables related to the extraction process in a standard condition are: $U=5.0E-4$ m/s; $x_0=0.144$; $y_0=0.0$ and $dp=2.7 E-4$ m. Figure 3 shows the main effects as a function of the extraction time. It can be seen that, in the initial extraction time (limited by convection), only the effect of the initial oil content of the solid particles is not null. In the extraction front region, however, all the variables have significant effects. The effects of the solvent velocity and of the initial oil content in the solids have positive effects on the residual oil, while the solvent velocity has a negative effect. The mean solid particle diameter shows an inversion in the signal of the main effect, in the region of the front of extraction. Firstly, the signal is negative, indicating that the residual oil in the extractor exit is lower for greater particles. After, the signal becomes strongly positive, signifying that as bigger the particles, greater will be the residual oil content in the solids. In the period of extraction rates limited by diffusion inside the solid particles, the particle mean diameter is the unique variable with important effect on the residual oil in the solids. The analysis made above indicates that the solvent velocity may be manipulated to control the residual oil concentration at the extractor exit, after disturbances in the solid and inlet solvent initial oil contents. The analysis of the impact of changes in the particle mean diameter is also useful, since although it may not be controlled during the extraction, an appropriated treatment in the particles before the extraction will be an effective procedure for high performance process operation

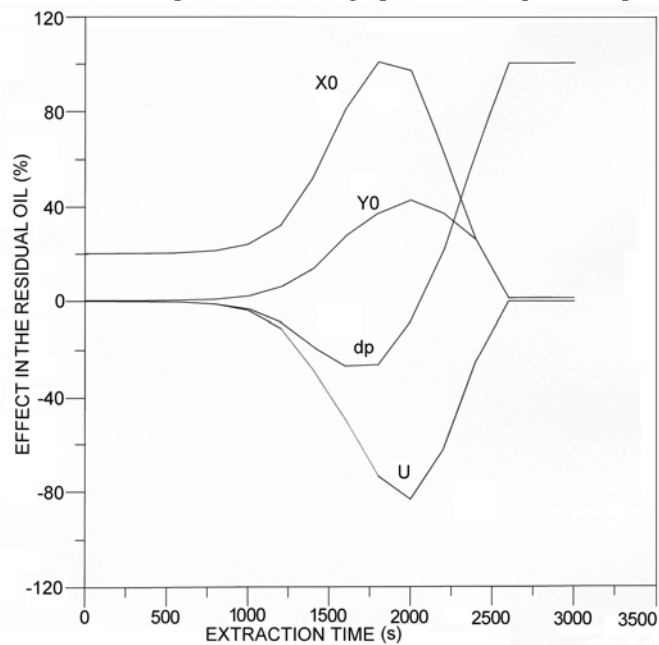


Figure 3: Effects of variables on the residual oil in the solids at the extractor exit.

5. Performance of a Classical Proportional Feedback Controller

As the semi-batch extraction process is intrinsically transient, it was necessary to define a reference extraction curve as a guide for the controller. This extraction curve was adopted as the time dependent residual oil at the extractor exit, for the variable levels considered in Figure 3. The effect of the sampling period on controller's performance was observed in Rezende (1998). The larger sampling period that still leads to a good performance of the controller was 200 seconds. This was adopted in this work. It was, also, noted that the addition of the integral and derivative actions could not improve the classical controller performance. The controller is investigated for keeping a time dependent residual oil concentration at the level dictated by the reference extraction curve, after individual and simultaneous step disturbances (5 and 15 %) in the initial solid oil content and in the inlet solvent oil concentration. The effect of the proportional controller on a time dependent curve for the residual oil concentration at the extractor exit, after positive step disturbances (5 and 15 %) in the initial solid oil content is such that, after 1400 seconds, the profiles of the controlled extraction joins the reference extraction one. Concerned with the solvent velocity after disturbances in x_0 , it can be seen that as soon as the period of slow rates of extraction is reached, the velocity backs to its standard value although the error is essentially null, and this velocity is no more an efficient variable to control the extraction. The performance of the proportional controller after disturbances of 5 and 15 % in the solvent concentration, at the extractor entrance, is presented in Figure 4a. Curves representing the controlled extractions are seen to follow the reference extraction curve. The behavior of the manipulated variable (the solvent velocity) can be observed in Figure 4b. Again, when the final period of slow rates of extraction is reached, the standard value of the solvent velocity is recovered. The performance of the proportional controller is shown in Figure 5a. In spite of disturbances in these two variables, the controller is still able to guide the extraction to the neighborhood of the reference extraction curve. The behavior of the solvent velocity is shown in Figure 5b.

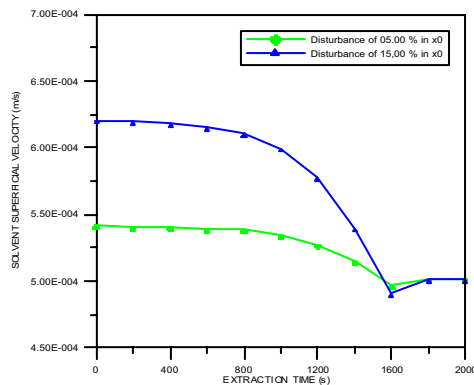


Figure 4a. Performance of the proportional controller after disturbances of 5 and 15% in the entrance solvent oil content.

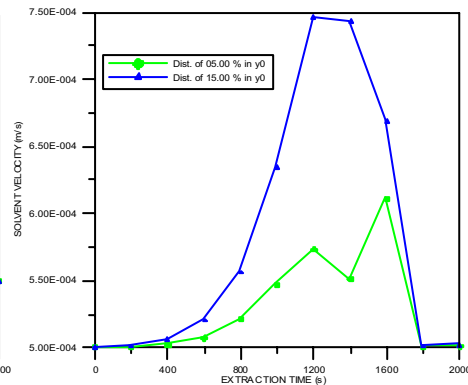


Figure 4b. Proportional controller in the solvent velocity after disturbances in the inlet solvent concentration.

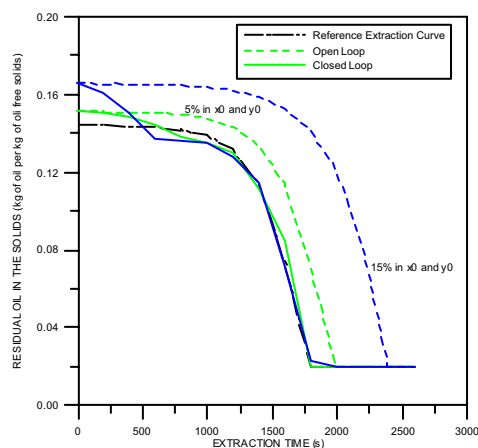


Figure 5a. Performance of the proportional controller after simultaneous disturbances of 5 and 15%.

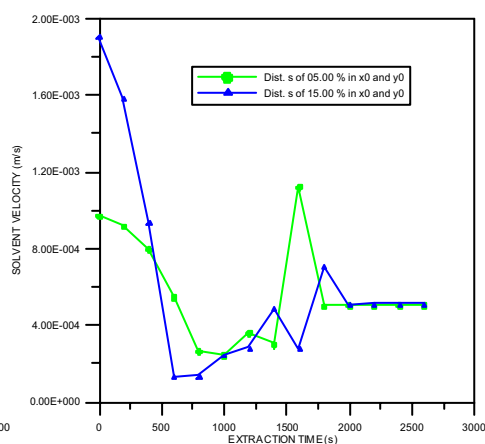


Figure 5b. Proportional controller in the solvent velocity after simultaneous disturbances.

6. Conclusions

The parameter sensitivity analysis required to the design of control systems showed important features for the period of extraction controlled by the oil convection at the vicinity of the solid particles. The proportional controller is able to drive the time dependent curve for the residual oil in the solids towards a predefined extraction pattern, during the period of extraction limited by the oil convection. This control action is made manipulating the solvent velocity after individual or simultaneous disturbances in the initial solid oil content and in the solvent inlet concentration.

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