EFFECT OF OPERATING CONDITIONS ON THE CONCENTRATION OF MONOGLYCERIDES USING MOLECULAR DISTILLATION

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Abstract: In this work, one-factor-at-a-time experiments and factorial design were carried out in order to study the effect of operating conditions on the monoglycerides (MG) concentration using molecular distillation (MD) process in laboratory scale. Therefore, producers of distilled MG can use the results obtained here to optimize industrial plants. Firstly, a 2^{4-1} factorial design was employed to select the most important operating variables of the process. Among the four variables studied, evaporator temperature (ET), feed flow rate (Q), feed temperature (FT) and condenser temperature (CT), just ET and *Q* are important at the experimental conditions studied. Furthermore, using empirical models and experimental data, it is shown the dependence of the outlet stream compositions to ET and Q, which were selected from the fractional factorial design.

Keywords: molecular distillation; short path distillation; monoglycerides; fractional factorial design.

INTRODUCTION

Molecular distillation (MD) is a gentle distillation method suitable for separation and purification of thermally unstable materials as well as for liquids with low vapour pressure and high molecular weight (Micov *et al.*, 1997).

Basically, there are two kinds of molecular distillators: falling film and centrifugal distillators (Batistella and Wolf-Maciel, 1996). In both models, the separation principle is the vacuum, enabling molecules to evaporate from the evaporator to the condenser, and the formation of a thin liquid film which promotes effective heat and mass transfers. Falling film distillators use gravity force to promote a thin film on the evaporating cylinder (evaporator), usually with a wiping element that mixes and distributes the liquid over the whole evaporator surface (Cvengros *et al.*, 2001), while centrifugal distillators use centrifugal force to cause this thin film. Two product streams are generated: distillate (D), rich in the molecules that escape from the evaporator and reach the condenser, and residue (R), rich in heavier molecules from the evaporator.

In the MD process, vapour molecules find a free path between the evaporator and the condenser. The distance between these two components should be shorter than the mean free path of the evaporating molecules. In these conditions, theoretically, the return of the molecules from the vapour phase to the liquid phase should not occur and the evaporation rate should only be governed by the rate of the molecules that escape from the liquid surface (Batistella *et al.*, 2000). However, in industrial plants, the distance between the evaporating and condensing surfaces is longer than the mean free path of the molecules. In this case, the process is called short path distillation (Chen *et al.*, 2005).

The performance of the process depends on the design of the equipment, such as the geometry of the evaporation space (Kawala and Dakiniewicz, 2002), type of condenser (Cvengros *et al.*, 2000a), presence or not of a entrainment separator (Lutisan *et al.*, 1998), and also on the operating variables of the process, like the operating pressure (P), evaporator ET, Q, FT, CT, and so on. Traditionally, one-factor-at-a-time approach is used to study these operating variables. However, factorial design can be an alternative, since, with a reduced number of experiments, it permits the analysis of many variables simultaneously, isolating the impact of each one. In addition, it provides information about the interaction between variables.

In this work, one-factor-at-a-time experiments and factorial design were carried out in order to determine the effect of operating

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conditions on the manufacturing process of distilled monoglycerides (MG), which are concentrated MG that have been widely used in the food industry for many years to improve physical characteristics of shortenings (Kuhrt *et al.*, 1950), potato and pasta products (Bakels, 1976). Although the experiments were carried out in laboratory scale, producers of distilled MG may use the results obtained here to optimize their industrial plants, due to the fact that the starting material used in this work has the typical composition of the mixtures that are usually fed in the industrial molecular distillers. Thus, once established the proposed methodology, other feed compositions can be studied with little refinement, but this is matter to other paper.

MATERIALS AND METHODS

Molecular Distillator

The model used in this work was a centrifugal distillator from Myers Vacuum Inc. (Kittanning, PA), with an evaporating surface area of 0.0046 m^2, and the evaporator rotation velocity fixed at 1350 rpm. CT and FT were controlled by thermostatic baths, while ET was controlled by an electrical heater system and its control. A simplified scheme of the equipment is shown in Figure 1. A peristaltic pump (Masterflex, Vernon Hills, IL) was used to control Q. The pressure P (16 Pa) was obtained by a vacuum system consisting of a diffusion and a mechanical pumps configured in series. A starting material is fed in the centrifugal distillator and, during the process, lights components such as MG, glycerol (GL) and free fatty acids (FFA) are obtained in the distillate stream while triglycerides (TG) and diglycerides (DG) are obtained in the residue stream, due they present higher boiling temperatures.

Starting Material

The commercial MG feeding the molecular distillator was donated by Braswey S.A. (Pirapozinho, SP, Brazil). It is produced from partially hydrogenated vegetable oil and has the typical composition of the mixtures that are usually fed in the industrial molecular distillers, as fallows: 10.8% of TG, 37.7% of DG, 43.6% of MG, 0.7% of FFA and 7.2% of glycerol (GL).

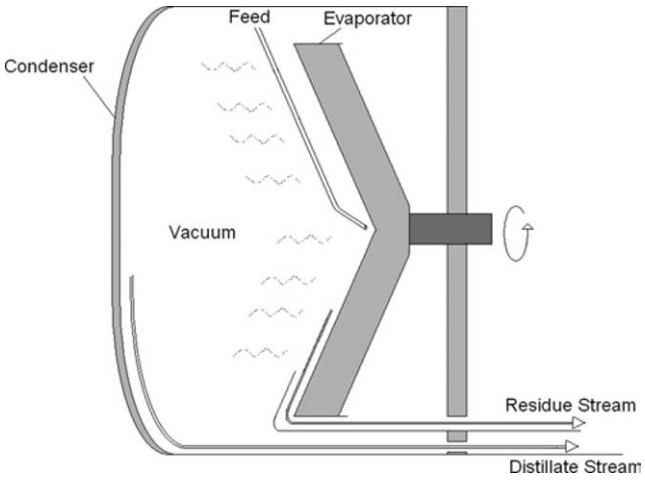

Figure 1. Scheme of equipment of molecular distillation.

Method of Analysis

Gel-permeation chromatography, also called high-performance size-exclusion chromatography (HPSEC), was used for the acylglycerols, FFA and GL analyses (Schoenfelder, 2003). The chromatographic system consists of an isocratic pump, model 515 high-performance liquid chromatography (HPLC) Pump (Waters, Milford, MA), a differential refractometer detector model 2410 (Waters), and an oven for columns thermostatted at 40°C by a temperature control module (Waters). The samples were injected using a manual injector (model 7725i, Rheodyne, Alltech, Deerfield, IL), with a 20-μL sample loop. Two HPSEC columns Styragel HR 1 and HR 2 (Waters), with dimensions of 7.8 × 300 mm and particule size of 5 μm, packed with styrenedivinylbenzene copolymer were connected in series. The mobile phase used was HPLC-grade tetrahydrofuran from Tedia (Fairfield, OH) and the flow rate was 1 mL min^{-1}. The typical pressure at this flow rate was 450 psi (3102 kPa). All the standards were obtained from Supelco (Bellefonte, PA). The data processing was done by the Millenium software 2010 Chromatography Manager Software from Waters.

Screening Design Methodology

Firstly, a set of experiments was designed to determine the effects of four operating variables (ET, Q, FT and CT) on the mass ratio (D/F). This mass ratio is calculated as:

$$D/F = (\text{mass of distillate})/(\text{mass of feed}) \quad (1)$$

and denote the efficiency of the molecular distillation process. Different combinations of operating conditions impact on different values of D/F and, consequently, on different outlet stream compositions. The fixed composition of the starting material is similar to the typical composition of the mixtures of TG, DG and MG produced industrially, by glycerolysis reaction at temperatures above 200°C, using inorganic catalysts (Fregolente *et al.*, 2005a).

The half-factorial design that was employed to conduct this study is shown in Table 1. All data were treated with the aid of STATISTICA 7 from StatSoft Inc. (Tulsa, OK). It is a 2^{4-1} experimental design, with three central points (runs 9, 10 and 11) (Barros Neto *et al.*, 2003). These central points are important since they are replicates in the centre of the experimental range. The variation between them reflects the variability of all designs (Carvalho *et al.*, 1997). In Table 1, actual values (in parenthesis) of the operating variables are shown

Table 1. 2^{4-1} fractional design.

Run	FT (°C)	CT (°C)	ET (°C)	Q (mL min^{-1})	D/F
1	−1 (60)	−1 (50)	−1 (150)	−1 (5)	0.044
2	+1 (110)	−1 (50)	−1 (150)	+1 (10)	0.018
3	−1 (60)	+1 (90)	−1 (150)	+1 (10)	0.018
5	−1 (60)	−1 (50)	+1 (250)	+1 (10)	0.101
6	+1 (110)	−1 (50)	+1 (250)	−1 (5)	0.327
7	−1 (60)	+1 (90)	+1 (250)	−1 (5)	0.265
8	+1 (110)	+1 (90)	+1 (250)	+1 (10)	0.150
9	0 (85)	0 (70)	0 (200)	0 (7.5)	0.082
10	0 (85)	0 (70)	0 (200)	0 (7.5)	0.057
11	0 (85)	0 (70)	0 (200)	0 (7.5)	0.064