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Optimization of the recycle structure of multiple stages molecular distillation

Daniela S. Laoretani, Oscar A. Iribarren*

Instituto de Desarrollo y Diseño INGAR CONICET—UTN, Avellaneda 3657, 3000 Santa Fe, Argentina

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ABSTRACT

In cases when the separation achieved by one molecular distillation stage is not sharp enough, it is resorted to multiple stage schemes. This brings the issue of how to recycle the intermediate streams to optimize the process. The present paper proposes to use the source-sink assignment methodology to decide the recycle structure. The counter current scheme is also taken into account because it is usually regarded as the design of choice to recycle streams. The approach was applied to optimize the recycle structure of a two stages process for the separation of free fatty acids from soybean oil deodorizer distillate, to get a concentrate of tocopherols in the residual stream, within specification of acidity. The counter current scheme was also implemented. For this case study the optimal recycle structure gets 10% more product with a profit 0.6% larger than the counter current scheme, even if it has larger operative and investment costs. So, in this particular case, the reward for using a rigorous methodology was not that significant. However, the approach proposed here is systematic and necessarily leads to a structure with a performance greater than or equal to any other, because the superstructure explored includes all of them.

1. Introduction

Molecular distillation (MD) or short path distillation is the partial evaporation of a liquid feed with a small distance between the evaporation and condensation areas (in the order of 2–5 cm) at very low working pressures (in the order of 2–20 Pa). In MD there is no boiling of the liquid nor equilibrium between phases. The phenomenon that occurs is depicted in Fig. 1: some molecules escape from the liquid film that wets the evaporator surface and reach the liquid film that wets the condenser surface. The distance between the evaporating and condensing surfaces is in the order of the mean free path of the molecules, which reduces the collisions that could redirect them back to the evaporator. A low temperature at the condenser is also important to reduce the number of molecules escaping from the condenser liquid film (Burrows, 1960).

With this level of vacuum, MD allows implementing separations at lower temperatures than conventional distillation, reducing hazard of decomposition of thermally unstable compounds. Also, there is a short time of exposure to the working temperature due to the short residence time in the apparatus. MD is applied industrially to obtain vitamin A, vitamin E, free fatty acids (FFAs), fish oil and other products where conventional distillation cannot be applied (Batistella et al., 1999; Pramparo et al., 2005; Martins et al., 2006a; Rodrıguez Posada et al., 2007).

When MD is applied to industrial processes, one single stage of separation may not be enough to obtain the products within the required specifications (Cvengros et al., 2000; Yu et al., 2015). Many articles in the literature approach multistage MD processes, reporting results from experiments or simulations. Contributions by Martins and coworkers will be taken here as the case study due to the detailed information reported by the authors.

In Martins et al. (2006a) the authors thoroughly studied the effect of MD operating variables (temperature and the ratio distillate to feed) to obtain a good depletion of free fatty acids (FFAs) from soybeans oil deodorizer distillate (SODD). One outcome of this exhaustive experimental work was to show the limitation of fractionation in a one stage operation because conditions that result in higher tocopherols concentration in the residue lead to higher losses in the distillate. The authors suggest that a multistage scheme with recycling of selected fractions may overcome this limitation. Consistently, in Martins et al. (2006b) the authors approach the same separation with the five stages separation scheme shown in Fig. 2. The first three stages operate at 120 °C

* *Corresponding author.*
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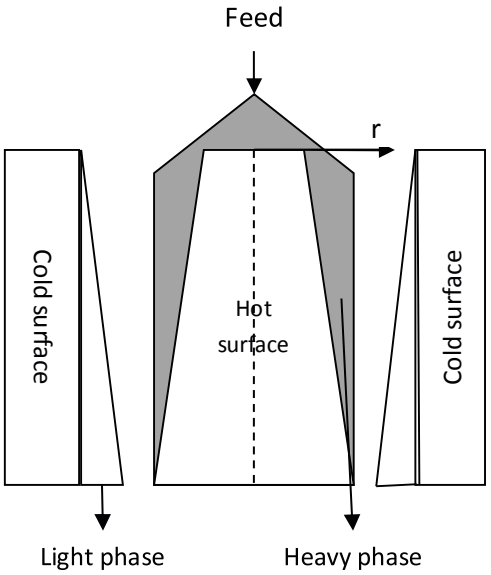

Fig. 1 – A schematic representation of a molecular distillation apparatus.

to remove FFAs in the distillates, which are waste exit streams. The last two stages operate at 185 °C separating tocopherols in the distillates from heavier components, so the residual streams are wastes in this case. The distillate of the last stage is the desired product: the tocopherols concentrate. The waste streams contain tocopherols but recycling them was not contemplated. Rodriguez Posada et al. (2007) propose a similar scheme of successive distillations to recover tocotrienols from palm oil fatty acid distillate. The process consists of three stages operating at 120 °C to remove FFAs in the distillates, and one last step at 160 °C to recover tocotrienols in the distillate, separating them from heavier components. Again there are losses of product in the waste streams, but recycling them within the process was not contemplated.

A practical approach proposed to implement these cascades is fractional condensation, as described in Cvengros et al. (2000). The condenser is divided into consecutive zones, each one producing an exit distillate stream of different concentration: the first fraction is very rich in the most volatile compound, and then the compositions in the following fractions gradually decrease. The authors tell that this apparatus with one evaporator and a divided condenser can be used for fractionation and in cases when recycling of some portion of distillate is convenient.

For a long time there have been proposals of recycling these intermediate streams resembling the counter current arrangement of conventional distillation stages. Burrows (1960) in his book about MD presents a configuration named fractionating still. This configuration has more than one MD stages arranged such that the heavy phase exiting each stage is sent to the previous stage (with a larger concentration of heavier components) and the light phase exiting each stage is sent to the next stage. For a binary separation and four stages, the arrangement would look like Fig. 3, with the feed entering into the cascade in a location according to its composition, e.g. in stage 4 if it is very rich in the light component, in stage1 if it is very rich in the heavy component, or in a middle point (e.g. in stage 2 or 3) if its composition is balanced.

Other arrangements have also been proposed (with appropriate arguments for each separation in study), for example Batistella et al. (1999) explored alternatives for the concentration of carotenes from palm oil and proposed the cascade scheme shown in Fig. 4. The carotenes remain in the heavy phase so the residue exiting the process is the concentrated product. They found that refluxing part of the product to previous stages permitted to increase its concentration.

The reflux stream is drawn as a dotted line in Fig. 4 meaning that it could be recycled to any still. Actually, the authors explored the three alternatives: reflux to stage 3, to stage 2 and no reflux at all, finding that the optimal decision was refluxing to stage 2.

The rest of this paper is organized as follows. Section 2 presents the approach taken in this paper to solve the optimization of the recycle structure of multiple stages molecular distillations. Section 3 introduces the mathematical model which is presented in more detail in Supplementary material. Section 4 presents the results and discussions and finally Section 5 presents the conclusions and future work.

2. Approach proposed in present work

2.1. *The superstructure*

The approach by Batistella et al. (1999) presented in the last part of the previous introduction section and illustrated with Fig. 4 serves as a motivating example to explain the superstructure approach. The dotted line representing the reflux of part of the product that exits the last stage to previous stages is a superstructure: taking as a base case the flow sheet of the process without recycles, Fig. 4 adds splitters and mixers that implement streams that reflux part of the final product to either units 2 or 3. Afterwards, a decision procedure is applied to select optimal flow rates for these new streams (that are also allowed to be zero). In the paper of Batistella et al. (1999) the decision procedure was to simulate a number of discrete values for the reflux ratio: 0, 1, 5 with the reflux stream entering either completely into stage 3 or stage 2.

Actually, there are many more possible recycles: the feed to the process could enter to any stage, and any exiting stream could be directed to feed any other stage, even the same stage from where it is exiting. This technology for generating a superstructure that includes all the possible recycles is not new, it is well established and explained in text books of mass integration as in El-Halwagi (2006) and is a Mass Sources—Mass Sinks allocation optimization problem. For the case study taken in this work (the separation of FFAs from SODD), with just 2 MD stages (to keep it simple yet already multistage) the superstructure generated is shown in Fig. 5.

The countercurrent alternative was also considered: it is always appealing, simpler to optimize, and it is a valuable heuristics for mass integration too (Fischer and Iribarren, 2011). Fig. 6 presents the countercurrent alternative already illustrated in Fig. 3, but in the same format of Fig. 5 for com-

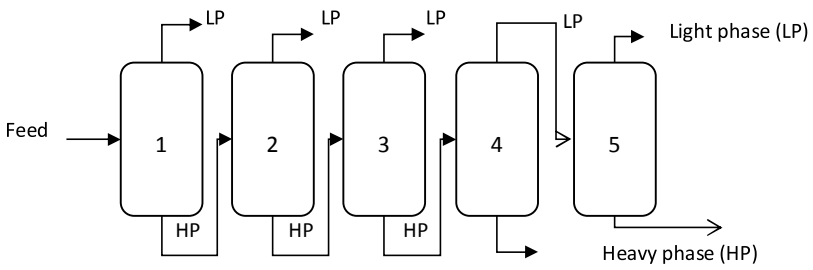

Fig. 2 – Sequence of molecular distillation stage proposed by Martins et al. (2006b).