MATHEMATICAL MODEL OF MEMBRANE MODULE FOR SOLVENT RECOVERY FROM SOYBEAN OIL MISCELLA

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***Abstract*— A mathematical model was developed to predict the performance of the solvent recovery process in vegetable oil extraction. It consists of a mathematical programming-oriented model for optimizing a biorefinery superstructure. Published experimental data was recompiled related to the PVDF-10SI membrane. For the case study, the mathematical model provides much more conservative design results than when considering just punctual experimental data. To concentrate miscella from 25% wt to 38% wt of oil, the required membrane area for the mathematical model is 275.22 m^2, while the design according to punctual experimental data results in a membrane area of 224.23 m^2. Besides, the mathematical model allows evaluating the separation performance of each intervening compound through their permeabilities. This is an important contribution since the results obtained using punctual experimental data are not accurate enough for membrane process design in oil desolventizing.**

***Keywords*— Solvent recovery, Miscella, Membrane module, Optimization process**

I. INTRODUCTION

The edible vegetable oil industry applies a productive process that involves several stages that vary according to the type of oilseed grain and the implemented technology and are intended to achieve the separation of the oily phase (crude oil) from the solid phase (flours). The flour containing some residual oil is subjected to extraction of this oil by using solvent. This stage (called solvent extraction) generates miscella as a first co-product (Fereidoon, 2005). Miscella is a homogeneous solution composed of solvent and 20 to 30% crude vegetable oil. The solvent commonly used to extract vegetable oil is hexane due to its solvent properties and market availability. It is estimated that 1.7 kg of hexane vapor per ton of processed oilseed grain is released to/lost in the atmosphere during recovery by distillation (Salehi, 2014), which results in high economic and environmental costs. The solvent present in miscella is recovered through different stages (Ferrero, 2016). This part of the process presents some disadvantages such as high energy consumption and solvent loss into the environment, among others. The need of counteracting these disadvantages brings about the implementation of membrane technology in this area.

Membrane technology applied to the vegetable oil industry presents attractive features: low operating cost; simple operation; high scalability at industrial level; no waste generation (clean technology) (De Morais Coutinho *et al*., 2009). Moreover, when compared with processes such as distillation, oil is not subjected to high temperatures, thus preserving product quality (Hafidi *et al.*, 2005). These characteristics make this technology attractive when making decisions on process synthesis and design.

There is extensive literature about membrane studies on solvent recovery and deacidification of miscella. In the early 1990s, different membranes were experimentally analyzed using reverse osmosis and ultrafiltration to remove solvent from miscella with different solvents such as hexane, isopropyl alcohol, and ethanol (Köseoglu *et al*., 1990). Pagliaro *et al*. (2011) analyzed the initial oil content in miscella, flow rate, and transmembrane pressure for three types of membranes, obtaining optimal process conditions. Also, they observed the unimportant fouling formation in all analyzed experiments. Badan Ribeiro *et al*. (2006) obtained similar results to those previously mentioned for polymer membranes. Raman *et al.* (1996a) experimentally analyzed six commercial membranes for stability and hexane permeability, oil concentration in miscella from 20 to 45% wt. The conclusion shows that the introduction of membranes causes a 50% reduction in energy costs of hexane evaporation. These authors also propose a design of membrane stages for deacidificating soybean oil with methanol as a solvent. Darvishmanesh *et al*. (2011) analyze different membranes and different solvents at different feed pressures to desolventize miscella, obtaining the highest permeate flow at the highest analyzed pressure of 20 bar for all studied membranes. De Morais Coutinho *et al*. (2009) analyzed a wide variety of membrane processes applied to the different stages of vegetable oil production. Their research work shows that membrane technology achieves promising results in miscella solvent recovery in a concentration range of approximately 25 to 45 %wt oil. So far, research has tended to focus on specific process variables such as optimization of temperature, pressure, oil concentration in miscella, among others, rather than on the process design itself. Nevertheless, recent studies (Firman *et al*., 2020) have concentrated on membrane module modeling and some aspects of the process design. The literature provides important models of membrane equipment, including the equation-oriented approach (Zarca *et al*., 2019; Rall *et al*., 2020). However, these models are intended for other processes and do not apply for

Table 1. Experimental data published by Firman *et al*. (2017) to PVDF-10SI membrane

Feed			Permeation			J (l/hm^2)	Retentate			R%
Mass fraction			Mass fraction				Mass fraction			
Oil	FFAs	Hexane	Oil	FFAs	Hexane		Oil	FFAs	Hexane	
0.09910	0.00090	0.8991	0.0262	0.00159	0.9722	35.1	0.12476	0.000733	0.8745	79.00
0.24775	0.00225	0.7478	0.0559	0.00160	0.9425	19.3	0.26619	0.000658	0.7331	79.00
0.34685	0.00315	0.6469	0.0951	0.00163	0.9033	14.3	0.3804	0.000694	0.6189	75.00

solvent recovery from miscella.

In the present work, a mathematical model of membrane module for the recovery of solvent from crude vegetable oil miscella is developed. The model represents the behavior of miscella and solvent throughout the entire membrane process, thus enabling an efficient equipment design and global economic performance estimate. Also, it is a mathematical programming-oriented model, so that it can be used in a superstructure and thus allow the process synthesis, simulation, and optimization.

This paper is developed in sections. Section II A presents the approach of the conducted study based on experimental data and all the analyses carried out to obtain the necessary information to reach the objective. Section II B describes the development of the mathematical model here proposed. Results and discussions are exposed in section III. Finally, conclusions are drawn and proposals for future work are presented.

II. METHODS

A. Approach proposed in present work

Membrane permeability studies are usually aimed at identifying the optimum operation point, so few experimental analyses are performed for operating conditions far from the identified optimum. This generates a certain lack of information necessary to rigorously model the behavior of the membrane under operating conditions in industrial processes (possible operation points far from the optimum). When globally optimizing a process, the final operating point of the membrane is hardly the experimentally determined optimum for certain initial conditions. The development of this work is based on experimental data obtained by Firman *et al*. (2017) for the PVDF-10SI membrane. The Poly (vinylideneflouride) PVDF-10SI membrane had the highest hexane permeate flow and oil recovery. This membrane showed the highest amount of experimental data in the previously mentioned work. It has hydrophobic characteristics. From the experimental analyses carried out on the PVDF-10SI membrane at a pressure of 20 bar and a temperature of 30 ºC, three fixed operating points were obtained for three different initial miscella conditions (10, 25 and 35% wt of oil). Table 1 shows hexane, oil, and FFAs composition of feed streams and permeate and retention streams as well as the total permeate and rejection coefficient for each feed stream.

It has been determined (Firman *et al*., 2020; Firman *et al*., 2013) that the behavior of this type of membranes corresponds to Darcy's law (Eq 1). See Fig. 1 for their application trough a membrane layer in cross flow pattern.

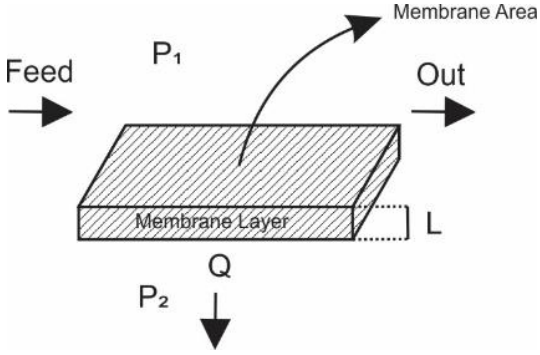

Figure 1. Darcy law schematization.

$$Q = -k \cdot \frac{A}{\mu} \cdot \frac{dp}{dl} = k \cdot \frac{A}{\mu} \cdot \frac{(p_1 - p_2)}{l} \quad (1)$$

In this expression, Q is the volumetric flow rate (m^3/h), A is the cross-sectional area (m^3), μ is the absolute viscosity (N/m), dp and dl are the pressure (Pa) and thickness differentials (m). Normally, the parameters of k/μ are called membrane permeability (L_m). In this sense, permeability is a property of the membrane that depends on the permeate component viscosity. The set of parameters $k/(\mu \cdot l)$ is also referred to as permeability (L, in $m^3/(h \cdot m^2 \cdot Pa)$), but in this case the set also includes membrane thickness. To distinguish these last parameters from an intrinsic property of the membrane, we would rather call them permeance. Thus, the mass transfer through membrane area can be expressed as:

$$J = L \cdot \Delta p \quad (2)$$

where J is the permeate flux (normally in $m^3/(h \cdot m^2)$ or $l/(h \cdot m^2)$). In addition to the overall membrane permeate flux (J (l/hm^2)) for the different initial oil concentrations in the miscella (shown in Table 1), Fig. 2 shows individual permeances of hexane, oil, and FFAs components according to concentration, which is more appropriate for the process mathematical modeling.

Considering the experimental results, when the feed oil concentration in the miscella is 10 %wt (including FFAs), a retentate is concentrated only at 12.5 %wt, and an impoverished permeate stream at 2.6 %wt of oil. For this operating point, permeance L from oil is 0.6644 L/hm^2, and from hexane is 34.3939 L/hm^2. These permeances are rigorous only in the range of 10-12.5 %wt of oil. If the objective is to concentrate the miscella (in oil) with an initial oil content of 10 %wt up to 35-40 %wt (normally implemented values in membrane process), the results with these permeances can be inaccurate. For higher accuracy, the next permeation experiment should be performed with a 12.5 %wt oil input. However, the next point with information is for a miscella with 25 %wt of oil. This is a wide range of inlet oil concentration, in which the permeance of components is

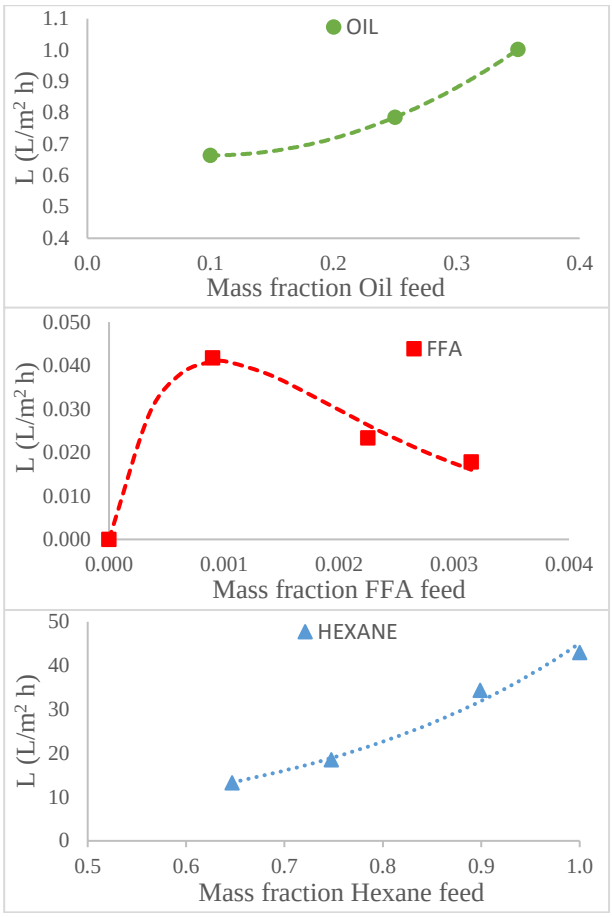

Figure 2. Permeance for each compound.

quite variable (with miscella inlet conditions), as depicted in Fig. 2. Thus, by using conceptual model is it not possible to make a rigorous design of a membrane equipment to concentrate the oil from 10 %wt to 25 %wt or to 35 %wt. Furthermore, estimating the required membrane area would also lead to inaccurate results.

In order to represent the mass transfer phenomena according to Darcy law, permeances are correlated with experimental data. For the published experimental data, the following correlations were found for permeance of hexane, oil and, FFAs:

$$L_{hexane}[\mathrm{L/m^2h}] = 1.4366 * e^{(3.4471 * hexane\ mass\ fraction)} \quad \text{(for 65 to 100 \% wt hexane)} \quad (3)$$

$$L_{oil}\left[\frac{\mathrm{L}}{\mathrm{m^2h}}\right] = 5.3984 * (oil\ mass\ fraction)^2 - 1.078 * oil\ mass\ fraction - 0.7182 \quad \text{(for 0 and 35 \% wt oil)} \quad (4)$$

$$L_{FFA}\left[\frac{\mathrm{L}}{\mathrm{m^2h}}\right] = 0.1181 * e^{-621.9 * FFA\ mass\ fraction} - 0.1181 * e^{-1657 * FFA\ mass\ fraction} \quad \text{(for 0 and 0.315 \% wt FFAs)} \quad (5)$$

with these correlations, we can estimate an L permeance that is representative of the miscella composition for which each membrane stage is being fed in the mathematical model.

B. Mathematical model of membrane module for solvent separation

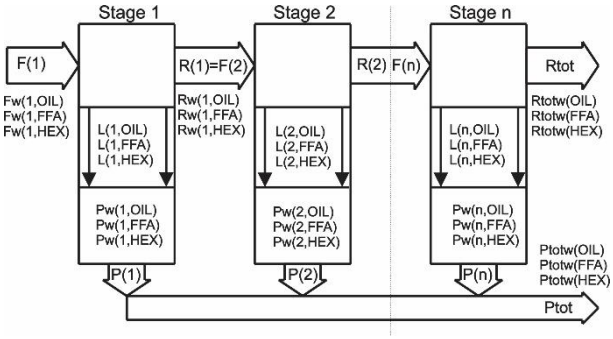

Figure 3. Mathematical model schematization.

Several mass transfers models for membrane modules can be found in the literature (Firman *et al*., 2020; Raman *et al*., 1996b). This work presents a membrane model that can be applied to a spiral or hollow fiber membrane module. Some basic assumptions are required to obtain a compact model:

- There is no variation of temperature along permeation (isothermal model).
- In both retentate and permeate side, plug flow and perfect mixing is considered (concentration polarization and stagnant layer are negligible).
- Transmembrane pressure is constant (negligible pressure drops in both retentate and permeate side).

The proposed mathematical model involves a series of algebraic equations, which makes it possible to use optimization software —such as GAMS (General Algebraic Modeling System)— for their resolution. Figure 3 shows a schematization of the mathematical model for the membrane process.

The objective is to minimize the total membrane area (AreaT),

$$\min.\ \mathrm{AreaT} = \mathrm{Area} * \mathrm{n} \quad (6)$$

where n is the membrane stage number and Area is the stage membrane area.

The objective is subject to a number of constrains. Total mass balances in each membrane stage n are represented by

$$\mathrm{F_{(n)}} = \mathrm{R_{(n)}} + \mathrm{P_{(n)}} \qquad \forall\ \mathrm{n} \quad (7)$$

where $\mathrm{F_{(n)}}$ represents miscella stream input (kg/h) to membrane stage n, $\mathrm{R_{(n)}}$ and $\mathrm{P_{(n)}}$ are retentate (kg/h) and permeate (kg/h) respectively, both streams output from membrane stage n. Here, $\mathrm{F_{(n)}}$ is problem data.

The mass balance for each compound i at each stage n is

$$\mathrm{F_{comp(n,i)}} = \mathrm{R_{comp(n,i)}} + \mathrm{P_{comp(n,i)}} \quad \forall\ \mathrm{n, i} \quad (8)$$

where ($\mathrm{F_{comp(n,i)}}$, $\mathrm{R_{comp(n,i)}}$ and $\mathrm{P_{comp(n,i)}}$) are mass streams of each compound (kg/h) for each stage n in feed, retentate, and permeate, respectively.

Equations 9 to Eq. 17 represent the compound mass fraction ($\mathrm{F_{w(n,i)}}$, $\mathrm{R_{w(n,i)}}$, $\mathrm{P_{w(n,i)}}$), and their relationship with total mass stream for feed, retentated and permeate, for each stage n. The sum of mass fractions should be equal to 1.

$$\mathrm{F_{(n)}} = \sum_i \mathrm{F_{comp(n,i)}} \quad \forall\ \mathrm{n} \quad (9)$$

$$\mathrm{F_{w(n,i)}} * \mathrm{F_{(n)}} = \mathrm{F_{comp(n,i)}} \quad \forall\ \mathrm{n, i} \quad (10)$$

$$\sum_i \mathrm{F_{w(n,i)}} = 1 \qquad \forall\ \mathrm{n} \quad (11)$$

$$R_{(n)} = \sum_i R_{comp(n,i)} \quad \forall\, n \tag{12}$$

$$R_{w(n,i)} * R_{(n)} = R_{comp(n,i)} \quad \forall\, n, i \tag{13}$$

$$\sum_i R_{w(n,i)} = 1 \quad \forall\, n \tag{14}$$

$$P_{(n)} = \sum_i P_{comp(n,i)} \quad \forall\, n \tag{15}$$

$$P_{w(n,i)} * P_{(n)} = P_{comp(n,i)} \quad \forall\, n, i \tag{16}$$

$$\sum_i P_{w(n,i)} = 1 \quad \forall\, n \tag{17}$$

The density of each feed, permeate and retentate streams, was obtained according to its composition as

$$\delta_{(n)} = \left(\sum_i F_{w(w,i)}/\delta_{(i)}\right)^{-1} * F_{(n)} \tag{18}$$

for feed stream.

Each compound density $\delta_{(i)}$ was considered to be 913.8 kg/m^3 for oil, 883 kg/m^3 from FFAs and 655 kg/m^3 from hexane.

Equations 19 to 21 represent permeance $L_{(n,i)}$ for each compound i at each membrane stage n in function of mass fraction for each stage.

$$L_{(n,i)} = 1.4366 * e^{(3.4471*F_{w(n,i\epsilon hexane)})} \quad \forall\, n, i\epsilon hexane \tag{19}$$

$$L_{(n,i)} = 5.3984 * (F_{w(n,i\epsilon oil)})^2 - 1.078 * F_{w(n,i\epsilon oil)} + 0.7182 \quad \forall\, n, i\epsilon oil \tag{20}$$

$$L_{(n,i)} = 0.1181 * e^{(-621.9*F_{w(n,i\epsilon FFA)})} - 0.1181 * e^{(-1657*F_{w(n,i\epsilon FFA)})} \quad \forall\, n, i\epsilon FFAs \tag{21}$$

The last part of the mathematical model analyzes the final output stream of the membrane equipment considering all stages n of the performed discretization. Rtot represents el total retentate and is obtained in the last membrane stage n (Eq. 22). Equations 23 and 24 are necessary to calculate mass fractions $Rtot_{w(i)}$ and mass stream of compounds i $Rtot_{comp(i)}$ to total retentate. Ptot represents total permeate and results from the sum of permeates of all analyzed n (Eq. 25). Equations 26, 27 and 28 obtain mass fraction $Ptot_{w(i)}$ and mass stream of compounds i $Ptot_{-comp(i)}$ to total permeate.

$$Rtot = R_{(n)} \quad \forall\, n\epsilon ord(n) = card(n) \tag{22}$$

$$Rtot_{w(i)} = R_{w(n,i)} \quad \forall i, n\epsilon ord(n) = card(n) \tag{23}$$

$$Rtot_{comp(i)} = R_{comp(n,i)} \quad \forall i, n\epsilon ord(n) = card(n) \tag{24}$$

$$Ptot = \sum_n P_{(n)} \tag{25}$$

$$Ptot_{w(i)} * Ptot = \sum_n P_{comp(n,i)} \quad \forall i \tag{26}$$

$$Ptot_{comp(i)} = \sum_n P_{comp(n,i)} \quad \forall i \tag{27}$$

$$\sum_i Ptot_{w(i)} = 1 \tag{28}$$

The following equations represent the connection between the retained stream of stage n, with the feed stream of the following stage (n + 1).

$$R_{(n)} = F_{(n+1)} \quad \forall n\epsilon ord(n) \neq card(n) \tag{29}$$

$$R_{comp(n,i)} = F_{comp(n+1,i)} \quad \forall i, n \epsilon ord(n) \neq card(n) \tag{30}$$

Mass balances for each membrane stage n and the total process are necessary for the equipment membrane design. The total equipment membrane area AreaT shows equipment size (Eq.31); and the area for each stage n is obtained through permeate stream $P_{comp(n,i)}$ and permeance $L_{(n,i)}$ (Eq.33).

$$Area = \frac{AreaT}{card\ n} \quad \forall\, n \tag{31}$$

$$P_{comp(n,i)} = Area * (L_{(n,i)}) * \delta_{(i)}/1000 \quad \forall\, n, i \tag{32}$$

When a final oil concentration in miscella is desired, the next equation is added to the model.

$$R_{-w(n,i\epsilon oil)} = R_{oil\ desired} \quad \forall n \epsilon ord(n) = card(n) \tag{33}$$

This mathematical model makes it possible to analyze the behavior of permeate and retentate along the membrane area. It can be used to determine the membrane area needed for obtain specified miscella concentrations from an initial concentration. Also, it can be used to obtain the miscella concentrations from a specified membrane area. Expressing the mass transfer phenomenon in this way enables the analysis of this process operation simultaneously with other processes operations. Therefore, the model can be used in a biorefinery superstructure for synthesis, design, and process optimization.

III. RESULTS

In this section, mathematical modeling is used to determine the membrane area needed to obtain a certain miscella concentration at the end of the process. For this case, Eq. (33) is fixed at the desired oil concentrations. Also, a number of membrane stages n=50 was used, verifying that above n=50 there are no significant differences in results.

The miscella feed was 10,000 l/h and was considered as a solution composed of oil, FFAs, and hexane, whose molecular weights for each compound are 920 g/mol, 280 g/mol, and 86.18 g/mol respectively. Table 2 shows oil concentration in miscella of 10 %wt and 25 %wt (including FFAs), which are concentrated to obtain miscella with 25 %wt and 35 %wt of oil respectively (the next analyzed concentration in the experimental data). Table 3 considers the same initial concentrations of miscella as those in Table 2 but concentrated to final oil concentration of 38.04 %wt (excluding FFAs), which is the maximum concentration obtained in the experimental data. Results are compared with those that would be obtained using the conceptual design, taking the punctual experimental values of permeance published by Firman *et al*. (2017). As it can be noted in both tables, the total membrane area obtained by using mathematical model is greater than that obtained by using the conceptual model with punctual experimental data on permeance.

Table 2 shows membrane areas required to concentrate the miscella. For concentrating miscella from 10 to 25 %wt demands a 41.40 % bigger area than when considering the mathematical model if compared to the conceptual model. For concentrating miscella from 25 to 35 %wt, the concentration range is narrower, but the difference in the obtained areas for each model is greater (47.81%).

Table 2. Membrane areas required

%wt oil in miscella	%wt oil in retentate	Total area m^2	
		Conceptual design	Mathematical model
10*	25	182.05	257.42
25*	35	215.54	318.60

* Oil includes FFAs

Table 3. Membrane areas required for different initial miscella concentration up to final oil concentration of 38.04 %wt

%wt oil in miscella	%wt oil in retentate	Total area m^2	
		Conceptual design	Mathematical model
10*	38.04	222.20	333.23
25*	38.04	224.23	275.22

* Oil includes FFAs

Table 3 shows membrane areas required to concentrate the miscella up to 38.04 %wt of oil. For an initial miscella concentration of 10 %wt of oil, the membrane area obtained by the proposed mathematical model was 333.23 m^2. This area is 50% higher than the one determined for the conceptual model. For a miscella of 25 %wt initial oil concentration, only a 22.74% bigger area is obtained. It is worth noting that the required area with the conceptual model to concentrate miscella from 9.91%wt to 38.04 %wt (excluding FFAs) is 222.2 m^2; while it is 224.23 m^2 to concentrate from 24.77 %wt to 38.04 %wt (excluding FFAs). The conceptual model requires a more extensive area for lower oil enrichment. These results show that estimating the membrane area by using only specific experimental data could lead to erroneous results, and the performance of a determined area can be poorly estimated.

Rigorous analysis of mathematical model results

For a more exhaustive analysis of the design performance through the mathematical model, different tests were carried out. Here, a miscella feed of 10,000 l/h was considered as in the previous analyses. The initial composition of the miscella was 24.775 %wt oil, 0.225 %wt FFAs and 74.78 %wt hexane. The density of the miscella is obtained in the model as a function of composition. The objective was to determine the membrane area needed to achieve a 38.04 %wt oil concentration in miscella.

Figure 4 shows the variation of total mass flows in the stream of each membrane stage n; while Fig. 5 shows the concentration of each component of the miscella (hexane, oil, FFAs) at the feed stream of each membrane stage n. The figure depicts s how the concentration of oil increases at each stage of the retained stream, as well as the decrease in hexane concentration.

Hexane concentration in the miscella feed in each membrane stage n decreased from 74.8 %wt to 61.7 %wt (a reduction of 17.5%). This solvent reduction, considering the volumes handled at industrial level, involves a large decrease in recovery energy consumption. One of the contributions of this work is the possibility of grasping the behavior of the permeate as the composition of the miscella changes along their way in the membrane equipment. As shown in Fig. 6, the permeate composition presents a similar pattern (for oil and hexane) to that of the feed stream at each membrane stage n, only with different mass fractions.

Unlike the existing publications that provide information about a particular experiment under certain conditions and certain miscella concentrations in which a single value of permeate and retentate is obtained, this research work allows observing the membrane performance and the behavior of the different components throughout the whole membrane module.

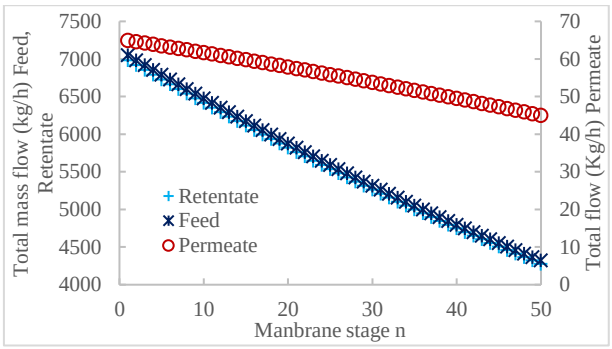

Figure 4. Total flows (kg/h) of feed, permeate, and retentate streams at each membrane stage n.

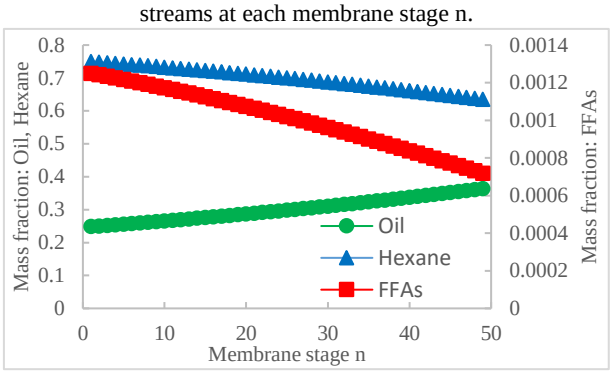

Figure 5. Mass fraction (% wt) for each component present in the mixture at the feed stream at each membrane stage n.

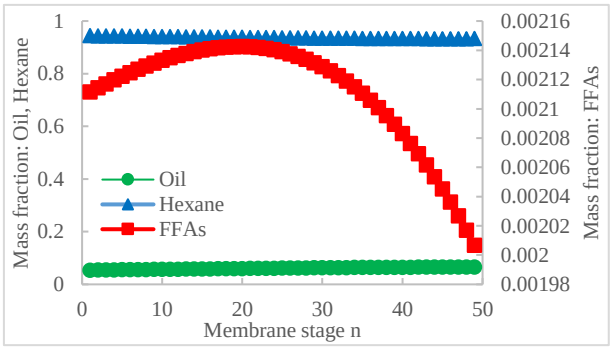

Figure 6. Mass fraction (% wt) of permeate stream for each component at each membrane stage n.

IV. CONCLUSIONS

This work presents a mathematical model for analyzing the nanofiltration membrane module performance in the separation of solvent contained in soybean oil miscella. The mathematical model was developed considering experimental permeance data published by Firman *et al.* (2017). The results obtained using the proposed mathematical model were compared with those obtained using the conceptual design (taking the punctual experimental permeance values). The result obtained with the mathematical model showed a more conservative equipment design than that obtained with punctual experimental permeance values. Membrane area size showed the greatest difference when oil concentration in miscella was 10

%wt. For this case, the required membrane area in the mathematical model is 50 % larger than that in the conceptual model. The mathematical model was also evaluated over a wide range of oil concentrations for the feed miscella, and in all cases it behaved conservatively in concerning the conceptual model with punctual permeance.

The mathematical model allows a rigorous analysis of the membrane module performance when introducing membrane technology into the traditional desolventizing process. It can facilitate technical, economic, and environmental analyses. The use of membrane technology allows a decrease in FFAs content in miscella. This is an important characteristic that can reduce the size of the subsequent deacidification process and the amounts of residues such as soap. Also, it allows its use as part of a more complex model representing a superstructure of the process design alternatives for obtaining vegetable oils by using solvents. In this way, the mathematical model can be used for the synthesis, design, and optimization of hybrid processes −such as those in biorefinery− that include membrane technology.

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