Chemical Engineering & Processing: Process Intensification 159 (2021) 108211

Contents lists available at ScienceDirect

Chemical Engineering and Processing - Process Intensification

journal homepage: www.elsevier.com/locate/cep

Some insights in experimental studies on the start-up operation of a reactive dividing wall column

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ARTICLE INFO

Keywords:
Dividing wall distillation column
Reactive distillation
DWDC start-up and operation

ABSTRACT

The design, simulation, optimization and control of dividing wall distillation columns have been reported in several theoretical studies, and important energy savings are obtained. Also, important information has been reported in the experimental and industrial implementation of this complex distillation option; but for the case of the reactive option, the information is limited. As a result, in this paper, we report the start-up and continuous operation of a reactive dividing wall distillation column for the production of methyl oleate using an esterification reaction of methanol and oleic acid, catalyzed by sulfuric acid. This experimental DWDC has been used previously for the separation of reactive and azeotropic mixtures in batch operation. The experimental results can be useful for the practical implementation and operation of reactive dividing wall distillation columns.

1. Introduction

Distillation continues being the most used technique for the separation of fluid mixtures despite its high energy consumption and low thermodynamic efficiency [1]. Regarding the energy consumption, several advances in research and development have been reported in order to achieve energy savings [2]. For instance, in the field of complex distillation options, the thermally coupled distillation sequences have reported savings between 30 and 50 % depending on the components and composition of the mixture to be separated [3]. Among the thermally coupled distillation options, the Petlyuk distillation column has been considered the most important option that has been implemented in industrial practice using the concept of dividing wall column. This concept is explained in Fig. 1 for the separation of a ternary mixture (ABC), where the three distillation columns of the conventional sequence (Fig. 1a) are merged into two columns in the Petlyuk option (Fig. 1b), and the two columns of the Petlyuk scheme are embedded in a single shell in the dividing wall distillation column (Fig. 1c).

It is important to note that the dividing wall distillation column (DWDC) is thermodynamically equivalent to the Petlyuk column when no heat transfer occurs through the wall.

Several topics have been studied in DWDCs, for example, thermodynamic efficiency, design and control, optimization, azeotropic mixtures, reactive separations and bio-separations. For instance, in 1965, Petlyuk et al. [4], studied the minimum work required for the separation of multicomponent mixtures. Kaibel [5] reported the industrial implementation of a DWDC using vertical partitions and his results showed that the control behavior of the DWDC was similar or better than that of a conventional distillation sequence. Yildirim et al. [6] reported that DWDCs can present significant energy savings and explored their applications to azeotropic, extractive and reactive mixtures. Egger and Fieg [7] presented a study of the process control of a reactive DWDC, taking into account economic and ecologic aspects in the design of this integrated distillation option. They used the liquid split to control the top composition in one side of the dividing wall.

2. Some experimental studies in DWDCs

The research in the topic of complex distillation columns has gained importance in the academic and industrial research groups due to important reductions in total annual costs and greenhouse gas emissions. Among the complex distillation options, the DWDC has been tested in the separation of reactive and nonreactive systems, given excellent results in energy savings and good dynamic behavior for set point tracking and load rejection.

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https://doi.org/10.1016/j.cep.2020.108211
Received 30 August 2020; Received in revised form 22 October 2020; Accepted 27 October 2020
Available online 2 November 2020
0255-2701/

2.1. Reactive case

Hernández et al. [8] reported the design and control of a DWDC for the production of ethyl acetate using the esterification reaction of ethanol and acetic acid with sulfuric acid as catalyst. The results of the simulation study were used to implement an experimental DWDC, consisting of three packed sections of Teflon™ rasching rings, with a total packing height of 2.5 m. In the design and operation of the DWDCs, it is necessary to perform a search in the interconnecting flows to guarantee the minimum energy demand in the reboiler. As a result, in order to manipulate the interconnecting liquid flows, a side tank was implemented, and the interconnecting vapor flows change according to the pressure drops in the DWDC. The final implemented column is depicted in Fig. 2.

Delgado-Delgado et al. [9] explored the production of ethyl acetate in the DWDC in a batch operation mode, their results showed good agreement between the simulation results obtained in Aspen Plus™ and the experimental results. The distillate of the experimental distillation column splits in two liquid phases, one organic containing ethyl acetate and the aqueous phase.

López-Ramírez et al. [10] explored the production of methyl oleate using an esterification reaction of methanol and oleic acid involving sulfuric acid as catalyst. Initially, the reaction-separation was studied in a glass distillation column and the results were scaled to the experimental DWDC in a batch operation, and these preliminary results indicate that it is possible the production of methyl oleate in the experimental complex distillation column.

Ehlers et al. [11] analyzed the transesterification reaction of n-butyl acetate and n-hexanol using both simulation and experimental studies, finding good correlation between the results.

Egger and Fieg [12] reported simulation studies of a reactive DWDC for the transesterification reaction of butyl acetate with hexanol, using a nonequilibrium stage model. The simulation results were validated using an experimental DWDC, and they reported good agreement between simulation and experimental results. As a result, the proposed model can be used to design this highly coupled distillation option.

2.2. Non-reactive cases

Mutalib et al. [13] investigated the temperature control of a DWDC using simulation and experimental tests, and they reported that the DWDC exhibited good dynamic responses for disturbances in the feed. This work constituted the basis for experimental studies in the field of complex distillation systems.

Buck et al. [14] studied the temperature control in a pilot DWDC, reporting that the experimental results were in agreement with the previous dynamic simulations. As a result, the formulated mathematical model can be used for additional control studies of this complex distillation scheme.

In the case of bio-separations, García-Ventura et al. [15] explored the purification of a binary mixture of ethanol-water with a purity near to the azeotropic point. The experiments were conducted in the DWDC in a semicontinuous operation using glycerol as entrainer. The results indicated that the production of high purity ethanol (> 99 % wt) was possible using the distillation column and glycerol as entrainer.

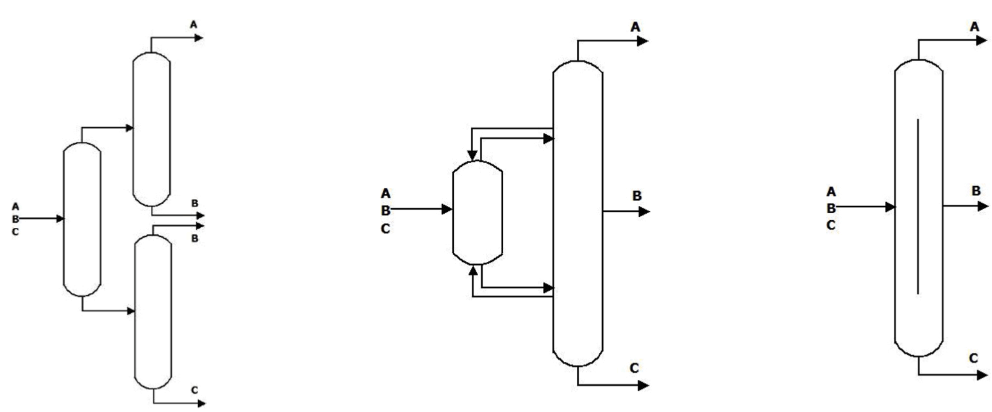
Lavasani et al. [16] studied, by using CFD simulations and experiment tests, the pressure drops and turbulence of the vapor-liquid mixture in a DWDC with several types of downcomers. They reported good correlation between the experimental and theoretical results.

Staak and Grützner [17] studied the separation of narrow-boiling or azeotropic mixtures in an extractive DWDC. They reported the case of an extractive DWDC implemented in the Lonza AG that was successfully designed using simulation runs and no experimental tests in a pilot plant were required. The designed extractive DWDC has been in operation, and the design specifications have been achieved during the complete operation.

Li et al. [18] announced the industrial implementation of a DWDC, and they tested the performance of the implemented distillation column using a mixture of alcohols, reporting 25.43 % of thermodynamic efficiency, a 33.39 % savings in energy consumption, and 28.73 % reduction in total annual cost. Finally, they concluded that these results can contribute to the industrialization of this complex distillation option.

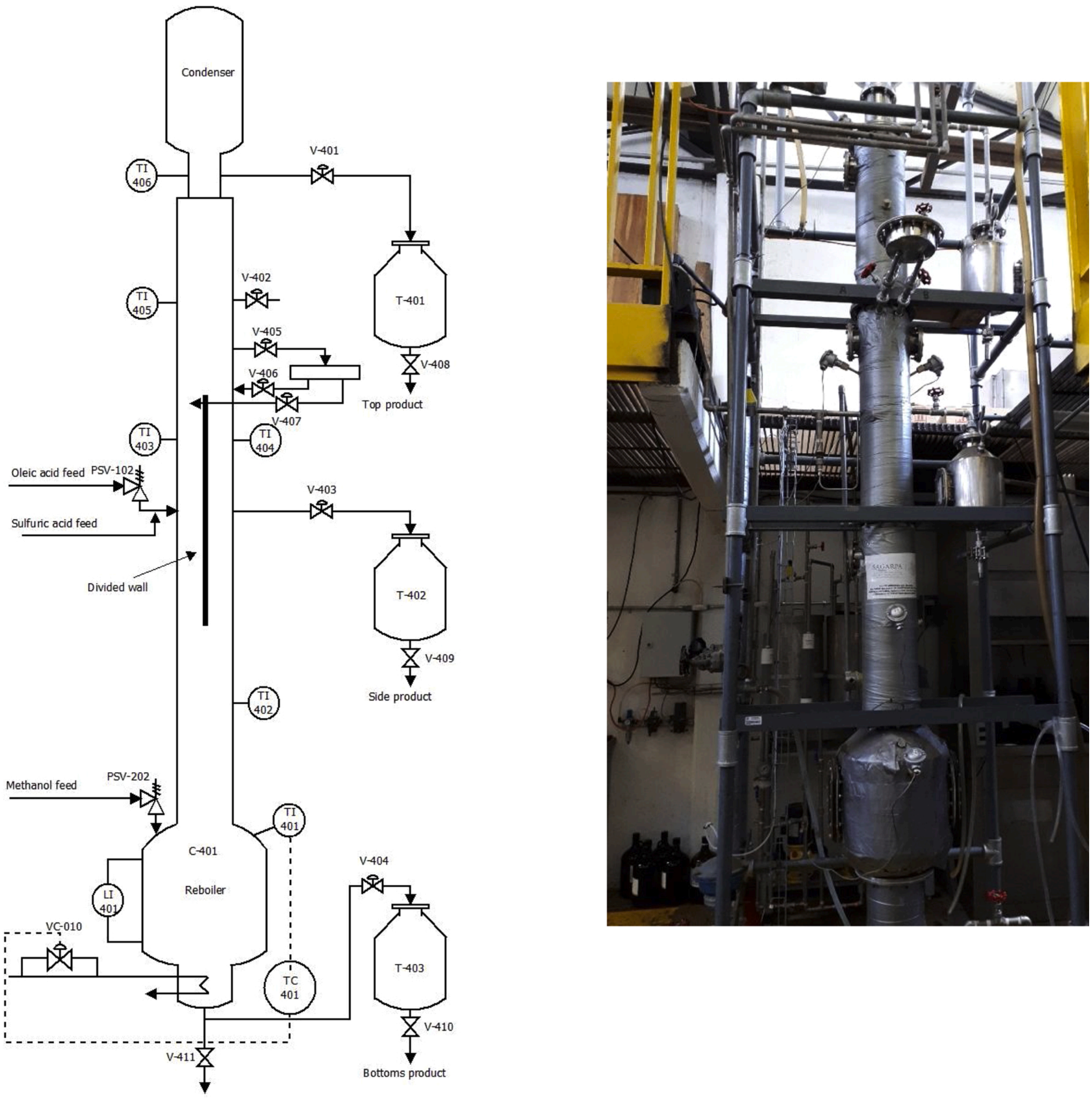
3. Start-up analysis in distillation

In the processing industry, there is the need to approach the operation of industrial equipments, so they increase their energy efficiency, leading to more economical and environmentally oriented processes. A feasible way to achieve these purposes lies in the optimal dynamic operation of industrial operations. The start-up of reactive distillation (RD) columns has also been addressed, but to a lesser degree. Alejski and Duprat [19] described a dynamic model to model kinetically controlled RD processes. In their work, they described the column start-up and disturbance rejection behavior during continuous operation. They found



a) Conventional distillation sequence   b) Petlyuk column   c) Dividing wall column

Fig. 1. Distillation sequences for the separation of a ternary mixture.



a) Schematic representation b) Real implementation

Fig. 2. Implementation of a dividing wall distillation column.

that the calculated dynamic temperature profiles were similar to their experimental data, but the concentration profiles differed from them. They also suggested that tray hydraulics must be taken into consideration in dynamic models. Monroy-Loperena and Alvarez-Ramıírez [20] analyzed the multiplicity maps for the ethylene glycol column and suggested a possible start-up procedure, based on the shape of the bifurcation diagram. Bisowarno and Tadé [21] used the SpeedUp process simulator to study several start-up scenarios for the ethyl tert-butyl ether column, while analyzing the potential problems that were due to input multiplicities embedded in the system. Scenna and Benz [22] using HYSYS, simulated the start-up of the closed-loop ethylene glycol column. Although they do not give details on the controllers used, they successfully analyzed different start-up policies and suggested the introduction of a feed below the reactive trays to reduce start-up time. Reepmeyer et al. [23] described "optimal time" strategies for column start-up, although they did not solve any optimization problems. In fact, using gPROMS, and a rigorous tray-by-tray model, they simulated different start-up heuristic strategies to determine which one required less time. They do not recommend the use of start-up strategies described for conventional distillation to RD, because they may lead to longer start-up times.

Compared to the number of publications related to the design, simulation, and control of RD columns, the number of works devoted to compute optimal start-up and steady state transition trajectories is rather scarce. Ruiz et al. [24] were among the first to use optimization numerical techniques to address the computation of optimal operating policies. Cervantes and Biegler [25] used the simultaneous dynamic optimization formulation to minimize the heat required to start-up a RD column. Raghunathan et al. [26] formulated and solved a hybrid optimal start-up problem where modeling switches were used to take care of different types of models, because of different operating conditions emerging during dynamic transition operations.

In the specific case of the start-up and operation of DWDCs, the number of experimental studies is reduced in contrast to the those reported using simulation analysis. For instance, Niggemann et al. [27] developed a model for the simulation and analysis of a DWDC during the start-up for the separation of a mixture of fatty alcohols. The developed model takes into account the heat transfer across the wall and the split of the vapor in the lower part of the divided wall.

Wu et al. [28] have reported a start-up procedure that has been successfully applied to a DWDC pilot plant used for the high purity separation of a mixtures of aromatic compounds. The start-up procedure involves four steps, including total-reflux, benzene withdrawn, toluene withdrawn, and continuous feeding.

Egger and Fieg [29] presented an interesting study of the start-up and open loop behavior of an experimental DWDC for the enzymatic catalyzed butyl acetate transesterification with hexanol. Their experimental tests demonstrated a stable and safe operation of the reactive DWDC pilot plant. Also, they developed a dynamic model that was validated using the experimental results.

Pan et al. [30] analyzed the start-up and operation of a Petlyuk-type dividing wall distillation column for the separation of a quaternary mixture of alcohols: methanol, ethanol, n-propanol and n-butanol. The proposed start-up procedure includes total reflux, batch operation and continuous rectification. The control strategy included a four temperature control loops, and a good dynamic performance was obtained.

Our research group has conducted several simulation studies in synthesis, design, optimization and control of DWDCs for the separation of hydrocarbons, azeotropic mixtures, extractive and reactive systems, and in this paper, we focus on the start-up and continuous operation of an experimental DWDC for a reactive system.

4. Start-up and continuous operation of the reactive DWDC

In a methodological way, initially the batch operation of this DWDC was addressed, and now the continuous operation is reported. In the previous section, some important experimental studies are described briefly, and they constitute the basis for the next experimental stage. This is important since some parts of the DWDC; for instance, the reboiler and condenser should be redesigned for the continuous operation.

Fig. 3 presents a detailed process flow sheet considered in the experimental operation of the DWDC.

In the experimental start-up of the DWDC, the methanol is introduced in the reboiler using Pump P-201 and Valve V-201; while the oleic acid is fed in the middle section of the column using Pump P-101 and Valve V-101. The sulfuric acid is introduced in the oleic acid pipe using Pump P-301 and Valve V-301. In the first stage of the operation, the distillation column is operated at total reflux, and the energy required in the reboiler is supplied using steam vapor. The total condenser operates using cooling water.

The total reflux operation continues until the temperature profile is constant; this can be concluded from the measurements of five thermocouples distributed in the column and connected to a laptop via the SIMATIC WinCC of SIEMENS.

Previously, it is important to know the values of the flows through valves V-406 and V-407, since these values are associated to the minimum energy consumption required in the reboiler, and they are obtained using an optimization procedure.

When total reflux operation ends, the continuous feed of methanol

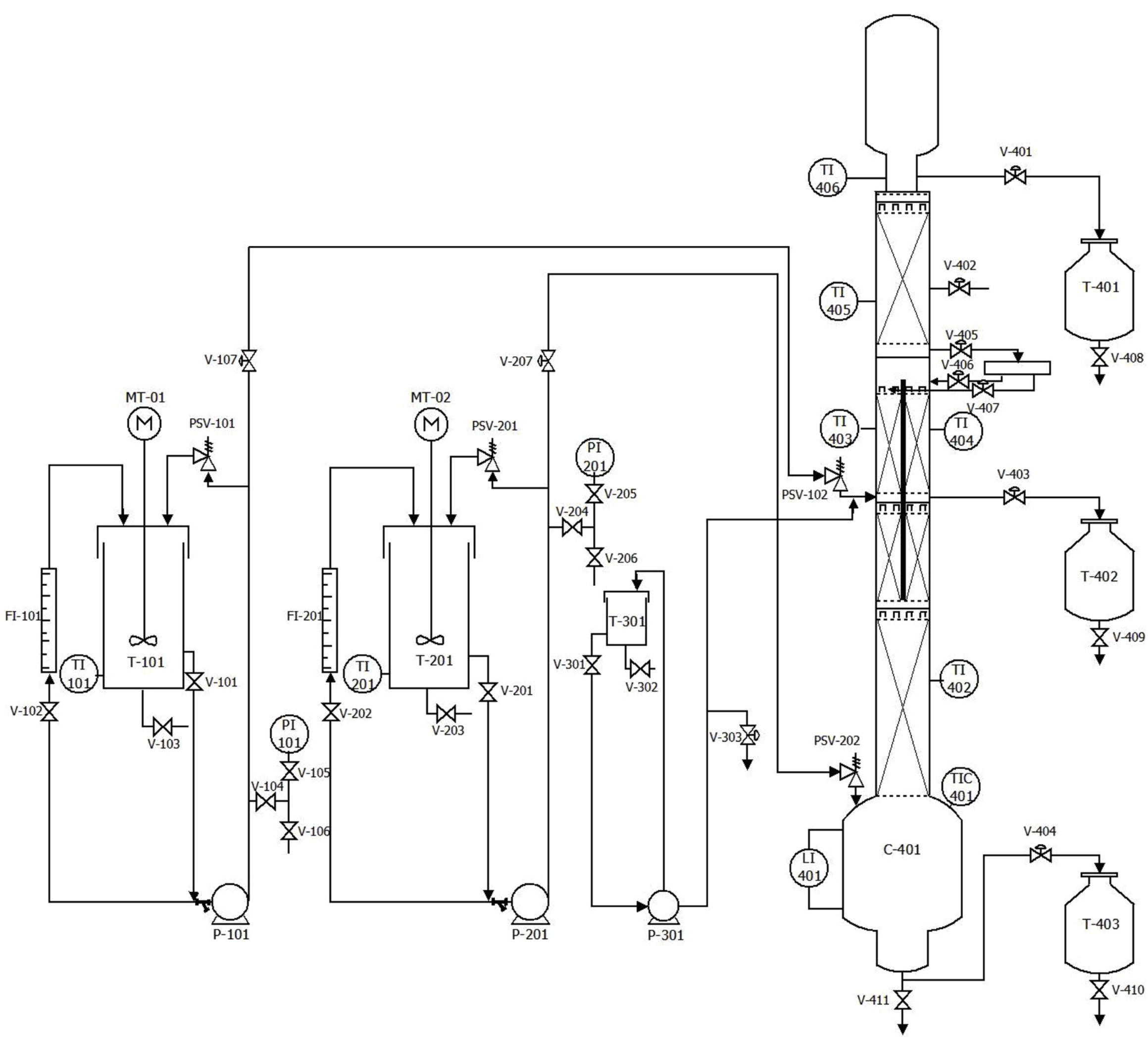


Fig. 3. Process flowsheet for the continuous operation of the reactive DWDC.

and oleic acid are manipulated by stroke adjustment knob in P-201 and P-101 while V-207 and V-107 are fully open. Sulfuric acid feed is controlled by stroke and speed adjustment knob in P-301. At this point, distillate and bottoms products are obtained. Reflux ratio and the distillate product are varied using Valve V-401; the bottoms product can be regulated by Valve V-404, and the side stream product is adjusted using Valve V-403.

For the end of the operation, Pumps P-201, P-101 and P-301 are shut down to cut the reactants to the distillation column. Also, the distillation product and the bottoms product are cut. Heat duty to the reboiler is interrupted and the supply of cooling water continues until the temperatures allow to manipulate the distillation column in a safe mode.

In a general procedure, the first stage corresponds to the charge of the reactants in the reboiler, the second period corresponds to the total reflux operation until constant temperatures are observed, the third moment is the continuous feed of reactants and the opening of the valves in the streams of the products. Finally, the proposed start-up strategy was complemented from experiences in the batch and semi-continuous operations of the DWDC for reactive and nonreactive mixtures.

5. Case of study

Barroso-Muñoz et al. [31] presented an experimental study of the pressure drops in a DWDC and reported the detailed design, that consists of three packed sections of Teflon rasching rings of a total height of 2.5 m, where the main section is divided by a non-insulated wall. Also, a side tank is required in order to manipulate the liquid flows to a distributor tray located in the top of the divided section. This point is important because some distributors were implemented to reduce the mal distribution of liquid (Fig. 3). The detailed design of the DWDC shown in Fig. 2b is reported in the previous papers of Hernández et al. [8] and Barroso-Muñoz et al. [31], but it is important to comment the steps required in the final implementation of the DWDC.

The preliminary design of the DWDC was obtained using the sections method described in Hernández and Jiménez [32]. In this method, the number of stages, the interconnecting stages, and the side stream stage are determined by using the shortcut method of Fenske-Underwood-Gilliland. When the basic structure of stages in the DWDC is determined, initial values are assigned to the interconnecting liquid and vapor streams, and they are varied until de minimum energy consumption is obtained for the proposed tray structure. In this stage, rigorous steady-state simulations are conducted using the equilibrium stage model, and the vapor-liquid equilibrium is modeled using the NRTL model.

The DWDC with the minimum energy consumption, for the esterification reaction in the reboiler, was subjected to closed-loop dynamic simulations using Aspen Dynamics considering proportional integral controllers. The dynamic results indicate that the DWDC can achieve changes in the set points (product compositions) and eliminate the effect of disturbances in the feed composition. The detailed mechanical design of the complex distillation column is reported in Hernández et al. [8].

It is important to highlight that the reported DWDC was designed for the esterification reaction of ethanol and acetic acid in a batch operation mode. Now, we present results for the reaction of esterification of methanol and oleic acid catalyzed by sulfuric acid to produce methyl oleate, because in the future we are going to produce biodiesel using this type of reactions, and conduct a comparison of the energy required in the reactive DWDC and a conventional reactive distillation column. As a result, new information considering the start-up and continuous operation of the complex distillation column is reported. The experimental tests were carried out at an ambient pressure of 0.8 atm.

6. Results

The connection between simulation and experiments is necessary because we are using an existing experimental DWDC. Also, the simulation studies reduce the number of experimental runs, saving reactants and time. As a result, in the first part of the results, the optimal operational conditions for the experimental distillation column, for the feed flows of Table 1, are presented. These results are generated using the equilibrium stage model of the RADFRAC module of the process simulator Aspen Plus™, an equilibrium kinetic model, and the UNIQUAC-RK model for the vapor-liquid equilibrium.

As reported previously, in the optimal design of the DWDCs is important to minimize economic, ecological and safety indicators; however, in the present study, we are using an existing distillation column, and we only determined the minimum energy consumption by performing a search in the interconnecting streams. For the simulation study, it is important to mention that the DWDC is modeled in Aspen Plus using a pefractionator fully coupled to a main distillation column, i. e., Petlyuk distillation column. As a result, the interconnecting streams correspond to the side of wall where the feed of oleic acid is introduced.

The values of the interconnecting streams play an important role in the energy requirements; for this case, Fig. 4 shows that the reboiler duty decreases as the interconnecting vapor flow (FV, vapor flow in the side of the fed) dimishes, while the liquid flow (FL, liquid flow in the side of the fed) variation does not have important effects. However, the observed reduction on reboiler duty is small. For the same case, the effect of the interconnecting flows on the composition of the methyl oleate, obtained as bottoms product is presented in Fig. 5. The increment in the interconnecting vapor flow gives a higher composition of methyl oleate and the interconnecting liquid flow does not cause a notable effect. It is worthy to note that a high composition of methyl oleate can be obtained using an existing reactive DWDC.

Fig. 6 presents the composition profiles in the DWDC for the optimal conditions detected in the optimization procedure. It is important to note that the methyl oleate is obtained as bottoms product just mixed with methanol and other components.

The experimental runs were conducted using oleic acid (Cosmopolita Drugstore), anhydrous methanol (J.T. Baker), and sulfuric acid (Jalmek). As commented earlier in this paper, we can measure five temperatures along the column. A temperature profile for a representative experimental run is showed in Fig. 7. The reboiler of the reactive DWDC is pre-charged with a mixture of methanol and oleic acid, with a molar ratio of 9:1 and 1% of sulfuric acid as catalyst; the start-up and continuous operation procedure described in the previous section are followed. In this experimental temperature profile, TI-401corresponds to the temperature of the reboiler and TI-406 is the measured temperature in the top of the distillation column.

A temperature difference between TI-403 and TI-404 is observed because in the TI-403 side the oleic acid feed is located. After 86 min of operation, temperature decreases because of reactants are feed at room temperature, this highlights the importance of the preheating of reactants in order to avoid disturbances in the continuous operation. Therefore, an IKA RW-20 mixer and a PID temperature controller MAXWELL MTA-48 for preheating were installed in the oleic acid and methanol tanks to carry out new experiments. This temperature profile indicates the start-up and stabilization periods occur during the first 40 min and then the continuous operation proceeds until 112 min.

The bottoms product was analyzed using *Attenuated Total Reflectance* (ATR) technique and a representative spectrum is presented in Fig. 8. The C = O signal for esters at 1743 cm^{-1} was observed, verifying the production of the methyl ester. The C = O signal for acids at 1719 cm^{-1} and O-H signal for water and methanol at 3370 cm^{-1} indicate that not all the oleic acid was consumed and there is a little quantity of methanol and water at bottoms product.

At this stage of the experimental research, we are focused on the start-up and continuous operation of the reactive DWDC, and of course the production of methyl oleate that is corroborated in a qualitative form using the ATR technique. In our future research, some important points must be considered, for instance, the quantitative measurements of the methyl oleate and other products. Also, for the specific case of this

Table 1
Flows for the experimental dividing wall distillation column, operated at 0.8 atm.

Feed	Flow (kg/h)	Products	Flow (kg/h)	Composition (mole fraction)			
				Methanol	Oleic acid	Methyl oleate	Water
Methanol	14.24	Distillate	11.97	0.9146	0	0	0.0854
Oleic acid	13.95	Side	1.39	0.7099	0	0	0.2901
Sulfuric acid	0.13	Bottoms	14.82	0.1108	0.0278	0.8571	0.0042

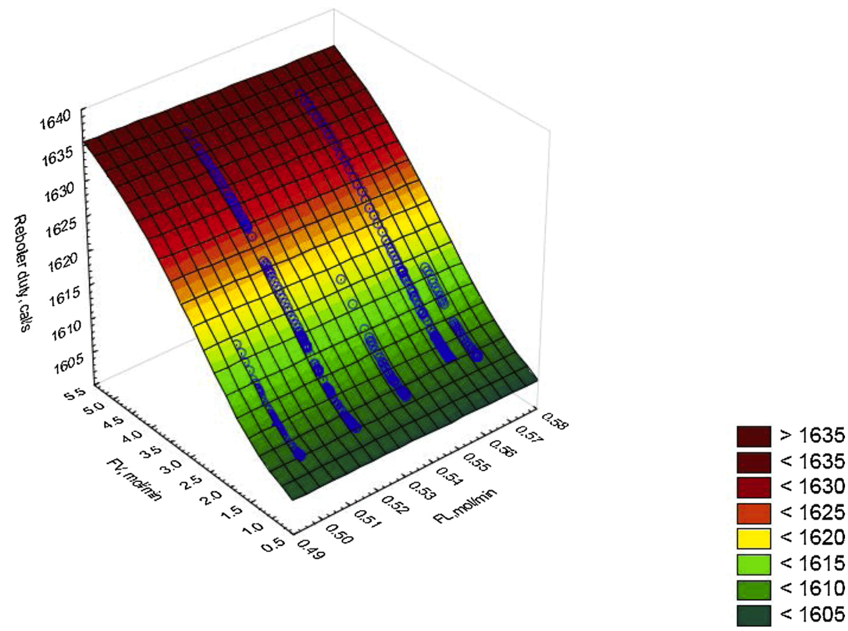


Fig. 4. Search for the minimum energy consumption in the reactive dividing wall distillation column.

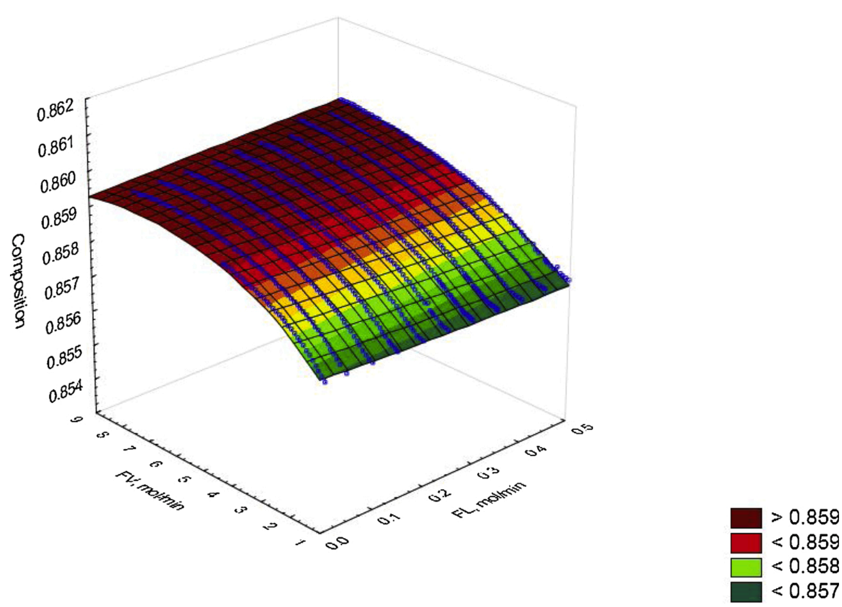
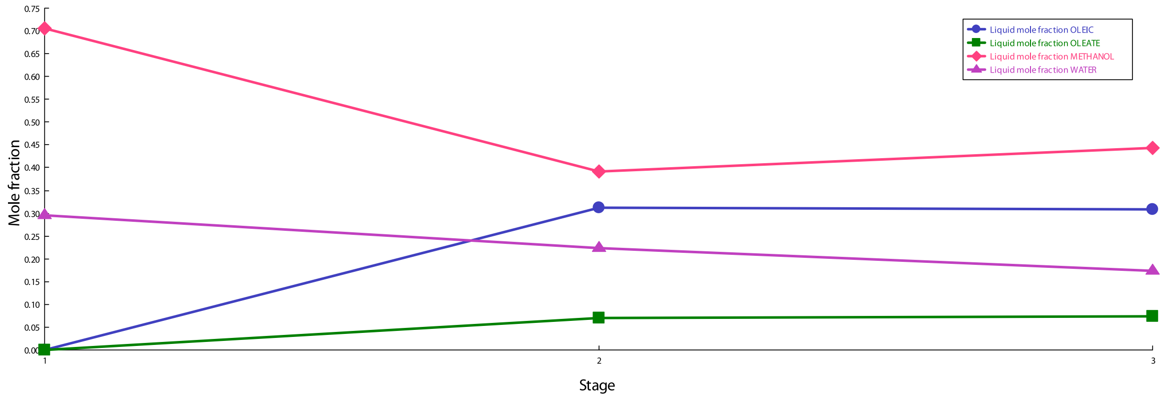


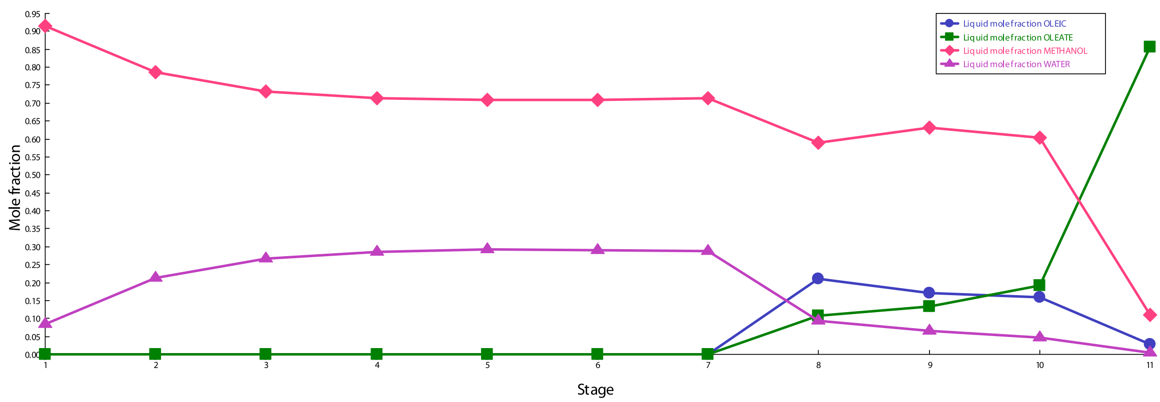
Fig. 5. Effect of the interconnecting flows on the purity of the methyl oleate obtained as bottoms product.

reaction catalyzed using sulfuric acid, the dimethyl ether could be present in the top and bottoms product; as a result, it is important to implement a system to detect the presence of this dangerous component in the working area.

It is important to say, that the control of this reactive DWDC is under analysis, because only, a PID controller has been installed to regulate the opening of the valve installed in the water steam pipeline. The valves associated to the distillate, side product and bottoms products, respectively, are operated manually.



a) Prefractionator



b) Main column

Fig. 6. Composition profiles for the optimal conditions in the reactive DWDC.

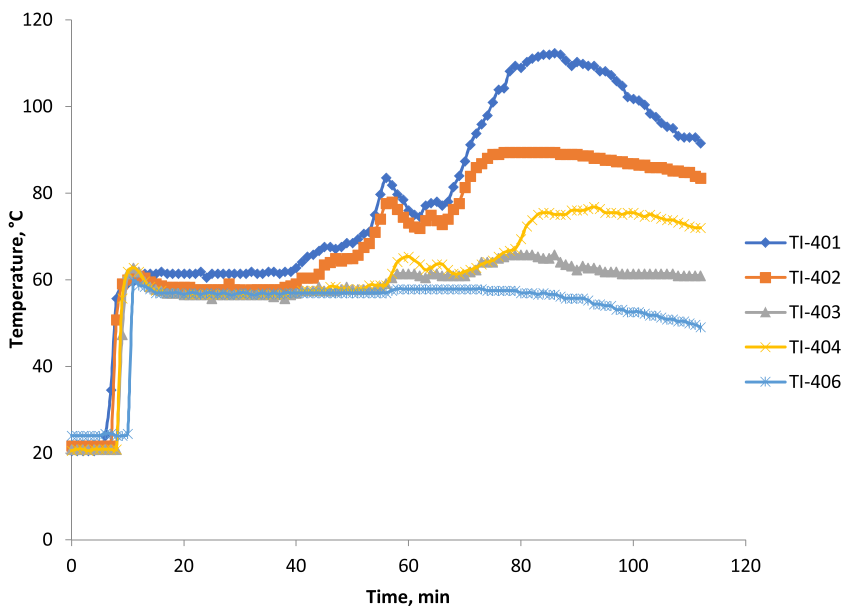


Fig. 7. Representative experimental temperature profiles in the operation of the dividing wall distillation column.

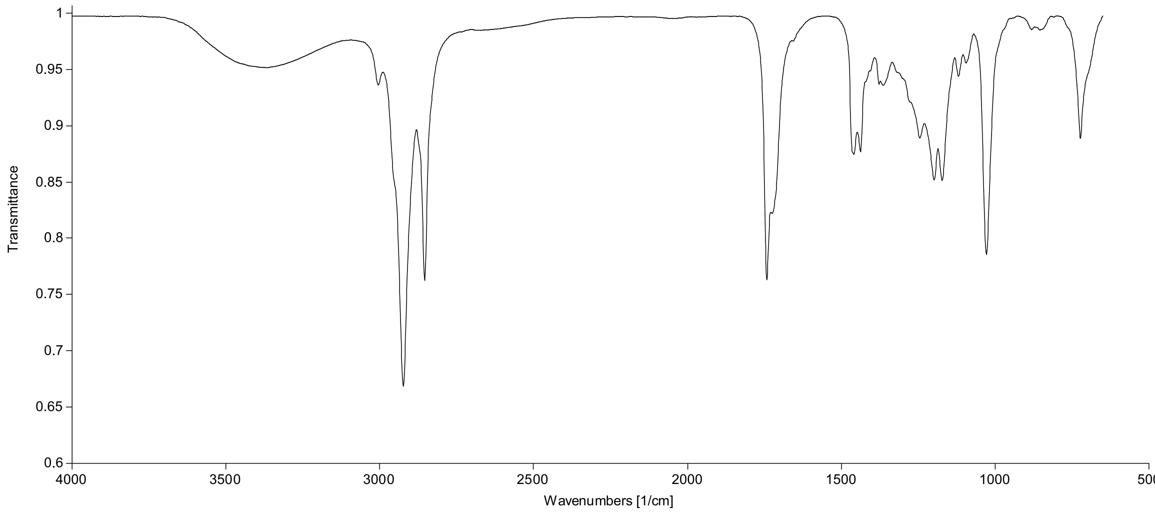


Fig. 8. Representative ATR spectrum for a representative bottoms product.

7. Conclusions

This work shows how conceptual work carried out in a simulation framework supported the operation of an experimental reactive DWDC to produce methyl oleate using an esterification reaction of methanol and oleic acid with sulfuric acid as catalyst. The experimental runs, using the operational conditions determined from Aspen Plus™ simulations, showed that methyl oleate with a high composition can be obtained as bottoms products in the continuous operation of a reactive DWDC. These results can be useful in the industrial implementation and operation of reactive complex distillation columns. This is important since most of reported results for reactive DWDC has been obtained using simulation studies, and in the case of experimental start-up and continuous operation of this complex reactive scheme, the reported information is scarce. The objectives of future work in the area of reactive DWDC involve the following issues: (a) to consider simultaneous design and control as a way to achieve economical design with operability characteristics, (b) to use different types of models during the start-up period by approaching the problem as a hybrid dynamic optimization problem.

Declaration of Competing Interest

We do not have any conflict of interest.

Acknowledgement

This research has been supported by CONACyT and Universidad de Guanajuato, México.

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