Fabricio Omar Barroso-Muñoz[1]
Salvador Hernández[1]
Juan Gabriel Segovia-Hernández[1]
Héctor Hernández-Escoto[1]
Vicente Rico-Ramírez[2]
Rosa-Hilda Chávez[3]

[1] Departamento de Ingeniería Química, Universidad de Guanajuato, Guanajuato, Mexico.

[2] Instituto Tecnológico de Celaya, Departamento de Ingeniería Química, Celaya, Mexico.

[3] Instituto Nacional de Investigaciones Nucleares, México, Mexico.

Research Article

Implementation and Operation of a Dividing-Wall Distillation Column

The design and construction of a prototype of a dividing-wall distillation column was possible by integrating previous knowledge in process intensification, energy savings, theoretical control properties, and closed-loop dynamics of thermally coupled distillation sequences. In order to achieve the predicted energy savings for this class of complex distillation column, a dividing wall and a side tank were implemented in order to manipulate the internal flows associated with energy consumption. The reaction between ethanol and acetic acid was conducted within the prototype, and the experimental results indicate that a heterogeneous mixture of ethyl acetate and water is obtained as the top product. The temperature profile measured during the experimental run can be used for controlling the batch distillation column in cyclic operation mode.

Keywords: Dividing-wall column, Energy-efficient design, Reactive distillation

Received: September 11, 2010; *revised:* January 06, 2011; *accepted:* January 12, 2011

DOI: 10.1002/ceat.201000388

1 Introduction

Distillation remains as the most-used separation technique for liquid mixtures despite of its high energy consumption and low thermodynamic efficiency [1]. In this context, several studies have been conducted in order to reduce the energy consumption in distillation; for example, some authors have improved the internals of the column by either using structured packings or by developing new configurations that are capable of reducing energy consumption [2–4]. The most common configurations used to reduce energy demands in distillation are called thermally coupled distillation sequences (TCDS). A TCDS can reduce energy consumption by between 30 and 50 % in comparison to classical distillation sequences for the separation of ternary mixtures or even more components [5–8]. TCDS have gained significance because of the need for more efficient separation alternatives that can be used to mitigate high energy demands. Many theoretical studies have shown that potential energy savings can be achieved through the use of these complex distillation sequences without introducing additional control problems [9]. In this respect, the theoretical control properties and closed-loop dynamic simulations for TCDS have resulted better than those of conventional distillation sequences. Among these complex distillation options, the Petlyuk distillation column has gained importance in industrial practice because its implementation is possible by using a single shell and a dividing wall. The flows around the dividing wall play an important role since the energy consumption depends strongly on the values assigned to these flows.

Based on the many theoretical contributions to TCDS design and control, their pilot-scale and industrial implementations have been possible due to the development of the dividing-wall distillation column (DWDC) [10]. The DWDC is thermodymically equivalent to the most popular thermally coupled system, named Petlyuk distillation column.

The literature reports significant advances towards the real implementation of a DWDC, proposing novel designs like the non-welded DWDC [3]. By considering new trends in process systems engineering, such as process intensification, it is possible to propose new applications of the DWDC configuration [11]. Perhaps the most important illustration of process intensification is the reactive distillation column, which combines reaction and separation in the same unit [12]. Using the same approach, if reactions are carried out in a DWDC, additional benefits are expected, e.g., a reduction in energy consumption and miniaturization of the distillation column (as a result of reduction of energy consumption and internal flows). Furthermore, reductions in energy consumption can be translated into less greenhouse gas emissions and less use of cooling water in the condensers.

In light of these ideas, a DWDC has been designed, implemented, instrumented and controlled to study the reactive distillation. This work presents the details of the energy-efficient design and some preliminary tests on reactive distillation.

Correspondence: Prof. S. Hernández (hernasa@quijote.ugto.mx), Universidad de Guanajuato, Campus Guanajuato, DCNyE, Departamento de Ingeniería Química, Noria Alta s/n, Guanajuato, Gto., 36050, Mexico.

2 System Analyzed

Among the several aspects that must be considered during the design and implementation of a DWDC, two critical issues can be identified. The first one is associated with the wall that is introduced in the middle section of the DWDC, since this important element allows the practical implementation of the Petlyuk distillation column. In principle, no heat transfer must occur through the wall in order to obtain a DWDC equivalent to a Petlyuk distillation column; as a result, insulation of the wall might be necessary. However, according to exergy analysis, insulation of the wall is not necessary since heat transfer through the wall might be beneficial [13]. In fact, the net energy balance through the wall does not have a significant effect on the energy performance of the DWDC; therefore, the implementation and costs of insulation of the wall can be avoided.

The second crucial aspect is related to the liquid flows to both sides of the wall in the upper section of the column. It has been reported that the energy demand of the DWDC strongly depends on the values of the interconnecting liquid and vapor flows. For a fixed wall, the flows of gas to both sides of the wall depend on internal pressure drops, but the flows of liquid can be manipulated externally by using a side tank. This external tank plays an important role in fixing the appropriate values of reflux rates to both sides of the wall, leading to reductions in energy requirements.

The system considered in our implementation is the reaction between ethanol and acetic acid to produce ethyl acetate and water, using sulfuric acid as liquid catalyst (Eq. (1)). Since, the reactive system is highly corrosive, Teflon^{TM} was used in the interconnection of the different parts of the DWDC. The internal packings of the distillation column were also designed and constructed by using Teflon.

$$\text{Ethanol} + \text{Acetic Acid} \rightleftarrows \text{Ethyl Acetate} + \text{Water} \qquad (1)$$

3 The Energy-Efficient Design of the DWDC

Using the method reported by Hernández and Jiménez [14], the first step for the design of the DWDC is to calculate the minimum energy required in the reboiler and the amount of energy to be removed in the condenser of the column. The minimum values of cooling and heating requirements are associated with the values of the interconnecting streams. The DWDC system has an internal diameter of 0.17 m, with a total height of the packed bed of 2 m; details of the design of the DWDC system are presented in the work of Hernández et al. [15].

Steam at 5 atm and cooling water at 20 °C are available in the authors' experimental laboratory. The reboiler was designed for a maximum load of 3 kW; it was implemented as a spiral heat exchanger. Stainless steel 316L was considered for the tubing because of the corrosive reactive mixture. The details of the reboiler can be seen in Fig. 1.

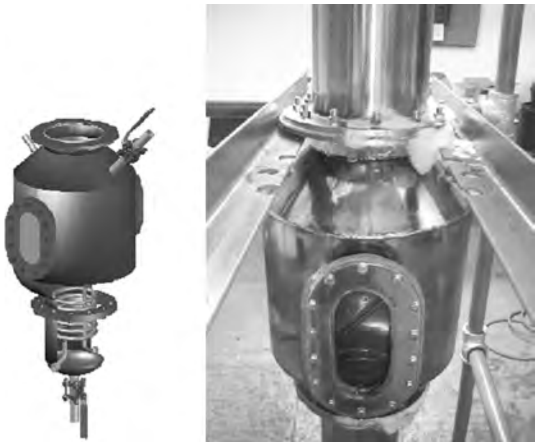
Figure 1. Reboiler of the DWDC prototype.

The condenser was designed for a maximum load of 3 kW. The construction material was also stainless steel 316L because of the direct contact with the mixture in the vapor phase. The resulting condenser uses a double spiral of tubing because of the reduction in space in the experimental laboratory. The final design is shown in Fig. 2.

The body of the DWDC consists of three sections. The upper and lower sections are conventional sections, but it is

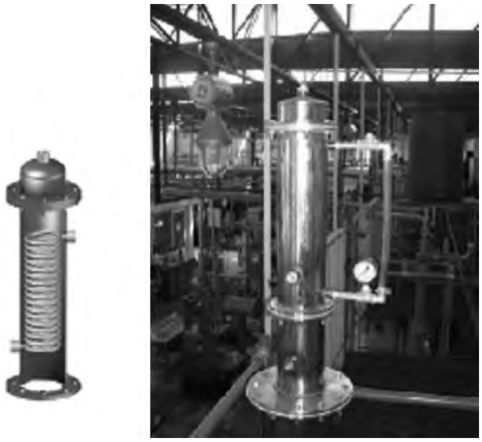
Figure 2. Condenser of the DWDC prototype.


Figure 3. Distributor tray in the upper section of each packed section.

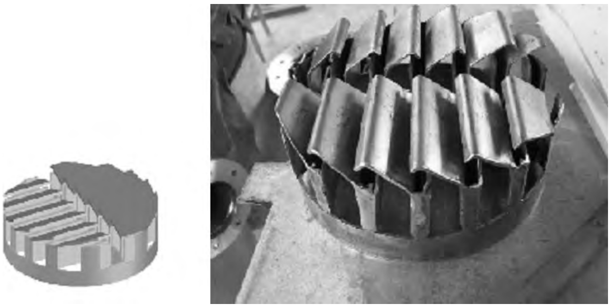

Figure 4. Support tray in the lower section of each packed section.

necessary to consider the design of the distributor of the liquid in the upper section (Fig. 3) and the support stage (Fig. 4) in the lower section.

On the contrary, the middle section is characterized by a wall placed inside the main shell, which plays an important role in the operation of the DWDC [16]. Many practical implementation problems need to be addressed. For instance, the wall and shell are constructed of stainless steel 316L and, therefore, the fit is not perfect; then, vapor and liquid may be transferred between both sides of the wall. In order to avoid this problem, it was necessary to incorporate a thin flat sheet of Teflon between the wall and the cylindrical body of the shell. The wall can be seen in Fig. 5.

The side tank is also considered a key part in the design and operation of the DWDC prototype. The liquid leaving the upper section of the distillation column is withdrawn to the side tank, using two valves, as depicted in Fig. 6; two liquid flows are returned to both sides of the dividing wall. These valves are used for manually controlling the flows. It is possible to implement control valves in order to manipulate the flows using digital control.

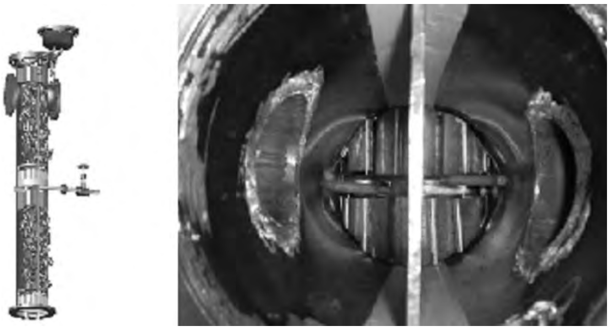

Figure 5. Wall in the middle section of the DWDC prototype.

Figure 6. Side tank implemented in the DWDC prototype.

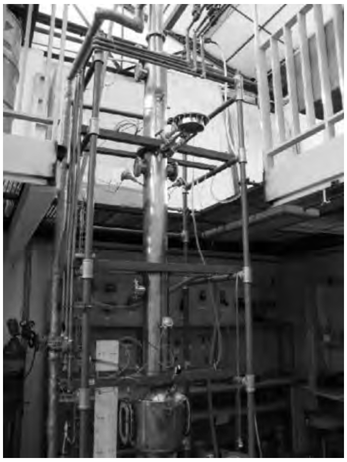

Figure 7. Final implemented DWDC.

Once the DWDC scheme is implemented, the instrumentation has to be completed. The experimental column is instrumented using six thermocouples connected to a computer via a Siemens acquisition data. Thermocouple T_1 is used to measure the temperature in the reboiler. Thermocouple T_2 is located in the middle section of the packed section mounted on the reboiler. Thermocouples T_3 and T_4 measure the temperatures at both sides of the dividing wall section. T_5 is inserted in the packed section below the condenser and T_6 is in the condenser section.

The amount of vapor supplied to the reboiler is controlled by using a pneumatic valve connected to the computer via the same electronic system. This control loop is currently under operation. The final implemented prototype of the DWDC is shown in Fig. 7.

4 Results

Currently, the experimental DWDC prototype is operated at total reflux, since operating the column in cyclic batch mode presents some benefits according to the work reported by Peng et al. [17]. The cyclic operation mode implies changes from total reflux to total withdrawal, and vice versa, for certain periods of time. This operation mode has its benefits, like maximum separation capacity, appropriate operation and control, and a reduction in time, in comparison to the classical constant reflux or constant distillate composition operation policies.

After introducing stoichiometric amounts of ethanol and acetic acid into the reboiler and adding sulfuric acid as catalyst, the heating is turned on by using the control interface. The cooling water valve is opened in manual operation mode.

Figure 8. Two liquid-phase products obtained in the distillate of the DWDC.

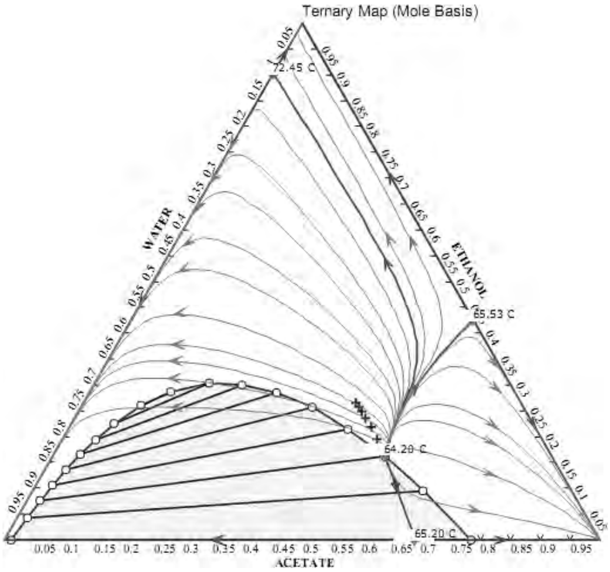

Figure 9. Ternary map for the system water/ethanol/ethyl acetate at 604 mm Hg.

Operation at total reflux takes place until the temperature profile registered in the interface remains constant. At that time, product is taken off as distillate. The product obtained is illustrated in Fig. 8. It can be seen that two liquid phases are present. In fact, the top product is a heterogeneous azeotrope that forms two liquid phases (Fig. 9). One liquid phase is rich in ethyl acetate and the second one is mainly water. Interestingly, this result is in agreement with the dynamic simulations reported by Hernández et al. [15] for dual temperature control, since the control of the composition of ethyl acetate was made using inferential control. The dynamic responses of the composition of the distillate were around 0.65 mole fractions of ethyl acetate, which is close to the composition of the heterogeneous azeotrope (0.67 mole fractions of ethyl acetate).

The temperature profile indicated in Fig. 10 can be used for the control of the cyclic operation mode. This temperature profile can be used for cyclic control since, in practice, the online implementation of a gas chromatograph is difficult and expensive. Also, the implementation of an online measurement of composition can lead to control problems because of the delay in the measurements. Additionally, the temperature registered in the top section of the DWDC is around 63 °C, which is close to the boiling temperature of the heterogeneous azeotrope (65.2 °C) indicated in Fig. 9. This result supports that the composition at the top of the DWDC is close to the heterogeneous azeotrope ethyl acetate-water, at an operational pressure of 604 mm Hg that is considered constant.

It is important to mention that the experimental run is in agreement with the closed-loop dynamic behavior expected for the reactive case. This result corroborates the potential application of DWDC for reaction and separation according to the principles of process intensification. As ongoing research, more results about the control of the DWDC will be obtained shortly. This is important because the comparison has been made between the steady-state results of the simulation and the experimental results under total reflux conditions.

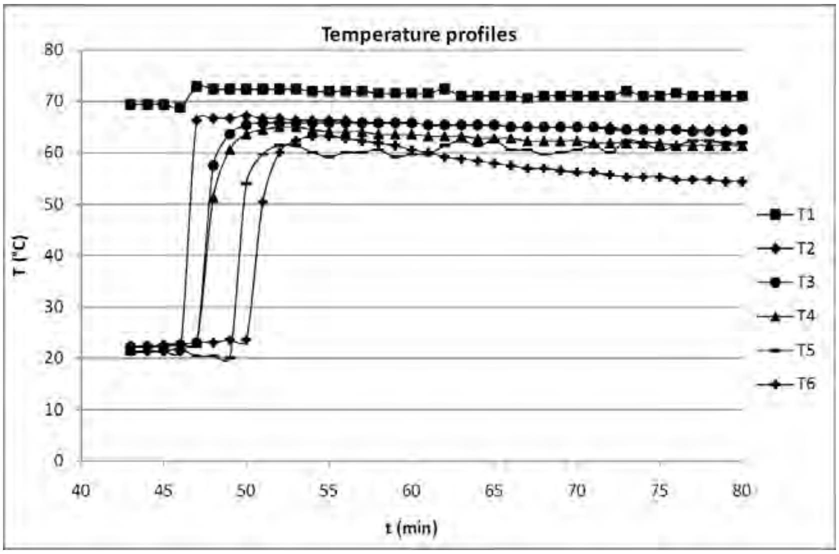

Figure 10. Experimental temperature profile of the DWDC.

5 Conclusions

The successful design and implementation of a DWDC were achieved by applying basic process intensification principles. The instrumentation, acquisition data, and the control interface were also implemented for future studies in the control of a dividing-wall reactive distillation column in cyclic batch operation mode. For the reactive system considering total reflux, the results indicate that an azeotrope (ethyl acetate-water azeotrope) is obtained as the distillation product. This result is in agreement with simulations. An experimental temperature profile can also be measured, and it can be used for controlling the distillation column in the cyclic batch operation mode.

Acknowledgment

We acknowledge the financial support provided by Universidad de Guanajuato and CONCyTEG (Mexico).

The authors have declared no conflict of interest.

References

[1] M. Mascia, F. Ferrara, A. Vacca, G. Tola, M. Errico, *Appl. Therm. Eng.* **2007**, *27*, 1205. DOI: 10.1016/j.applthermaleng.2006.02.045

[2] Ž. Olujic, M. Jödecke, A. Shilkin, G. Schuch, B. Kaibel, *Chem. Eng. Process.* **2009**, *48*, 1089. DOI: 10.1016/j.cep.2009.03.004

[3] N. Asprion, G. Kaible, *Chem. Eng. Process.* **2010**, *49*, 139. DOI: 10.1016/j.cep.2010.01.013

[4] L. Y. Sun, X. W. Chang, Y. M. Zhang, J. Li, Q. S. Li, *Chem. Eng. Technol.* **2010**, *33*, 395. DOI: 10.1002/ceat.200900422

[5] D. W. Tedder, D. F. Rudd, *AIChE J.* **1978**, *24*, 303. DOI: 10.1002/aic.690240220

[6] M. Khalifa, E. Mansour, *Clean Technol. Environ. Policy* **2009**, *11*, 107. DOI: 10.1007/s10098-008-0171-6

[7] I. Malinen, J. Tanskanen, *Ind. Eng. Chem. Res.* **2009**, *48*, 6387. DOI: 10.1021/ie800817n

[8] I. Malinen, J. Tanskanen, *Ind. Eng. Chem. Res.* **2009**, *48*, 6372. DOI: 10.1021/ie800818y

[9] R. C. van Diggelen, A. A. Kiss, A. W. Heemink, *Ind. Eng. Chem. Res.* **2010**, *49*, 288. DOI: 10.1021/ie9010673

[10] A. A. Kiss, J. J. Pragt, C. J. G. van Strien, *Chem. Eng. Commun.* **2009**, *196*, 1366. DOI: 10.1080/00986440902935507

[11] D. Reay, *Appl. Therm. Eng.* **2008**, *28*, 2011. DOI: 10.1016/j.applthermaleng.2008.01.004

[12] A. Stankiewicz, *Chem. Eng. Process.* **2003**, *42*, 137. DOI: 10.1016/S0255-2701 (02)00084-3

[13] B. Suphanit, A. Bischert, P. Narataruksa, *Energy* **2007**, *32*, 2121. DOI: 10.1016/j.energy.2007.04.006

[14] S. Hernández, A. Jiménez, *Comput. Chem. Eng.* **1999**, *23*, 1005. DOI: 10.1016/S0098-1354 (99)00257-4

[15] S. Hernández, R. Sandoval-Vergara, O. F. Barroso-Muñoz, R. Murrieta-Dueñas, H. Hernández-Escoto, J. G. Segovia-Hernández, V. Rico-Ramírez, *Chem. Eng. Process.* **2009**, *48*, 250. DOI: 10.1016/j.cep.2008.03.015

[16] F. O. Barroso-Muñoz, S. Hernández, H. Hernández-Escoto, J. G. Segovia-Hernández, V. Rico-Ramírez, R.-H. Chávez, *Chem. Eng. Process.* **2010**, *49*, 177. DOI: 10.1016/j.cep.2010.01.002

[17] B. Peng, X. Li, M. Sheng, S. Song, G. Zhao, X. Li, *Chem. Eng. Process.* **2007**, *46*, 769. DOI: 10.1016/j.cep.2006.07.008