redin Revista Facultad de Ingeniería, Universidad de Antioquia, No. 78, pp. 48-54, 2016

Using factorial design to increase the efficiency on a small-scale ethanol distillation

UNIVERSIDAD DE ANTIOQUIA 1803

Utilizando diseño factorial para aumentar la eficiencia en la destilación de etanol en pequeña escala

Flávio Dias-Mayer*, Giseane Fumagalli-Schettert, Paulo Roberto dos Santos-Salbego, Nicholas Islongo-Canabarro, Vanessa Baldo, Marcio Antonio Mazutti, Edson Luiz Foletto, Ronaldo Hoffmann

Department of Chemical Engineering, Federal University of Santa Maria. Avenida Roraima, 1000. CEP: 97105-900. Santa Maria, Brazil.

ARTICLE INFO

Received June 11, 2015
Accepted February 3, 2016

KEYWORDS

Ethanol distillation, factorial design, small-scale, process optimization

Destilación de etanol, diseño factorial, pequeña escala, optimización de proceso

ABSTRACT: This research assessed experimentally the performance of a small-scale ethanol/water distillation column. Statistical analysis was performed using Statistica® 7.0, considering a significance level of 90% ($p < 0.10$), to evaluate if the independent variables (feed stream ethanol concentration and flow rate) influence on the production of ethanol in accordance with the Brazilian legislation, i.e., a Hydrous Ethanol Fuel with ethanol content between 92.5 and 93.8 wt%. The results demonstrated that the influence of the feed stream ethanol concentration and flow rate were significant for both the top product concentration and the recovery ratio. The recovery ratio of ethanol was above 80%, demonstrating that the performance of the small-scale column is satisfactory.

RESUMEN: Este estudio evaluó experimentalmente el comportamiento de una columna de destilación de etanol/agua a pequeña escala. Se realizó análisis estadístico utilizando el software Statistica® 7,0, considerando un nivel de significación del 90% ($p < 0,10$), para evaluar si las variables independientes (concentración de etanol y caudal de alimentación) influencian la producción de etanol de acuerdo con la legislación brasileña, i.e., etanol combustible con concentración de etanol entre 92,5 y 93,8 % en masa. Los resultados demostraron que la influencia de la concentración de etanol y el caudal de alimentación fueron significativos para la concentración del producto destilado y la tasa de recuperación. La tasa de recuperación de etanol fue mayor que 80%, lo que señala que el rendimiento de la columna a pequeña escala fue satisfactorio.

1. Introduction

Ethanol fuel production plays an important role in the economy of several countries, being the world's largest producers the United States, Brazil and China. In Brazil, at 2010 year, the ethanol and sugar sector accounted for 19.1% of the primary energy supply [1]. This is due largely to the Brazilian Alcohol Program, whose incentives have transformed ethanol fuel into an alternative to gasoline [2-5]. This program is considered the largest program for ethanol production in the world [2], leading Brazil to be an important player in the international ethanol trade market [6]. The competitiveness of ethanol fuel compared with gasoline encouraged the popularization of vehicles with flex-fuel engines. Since 2003, 18.5 million light vehicles with this technology have been manufactured in Brazil [7], so called flex engine (gasoline and/or ethanol), and they will account for 47% of the national fleet in 2015 [5]. This scenario favors the increase of demand and also the price of ethanol fuel [8], leading to a grown in the hydrous ethanol fuel (HEF) production. However, this increase in the ethanol production, considering the large-scale traditional model, has some associated disadvantages such as land concentration [9, 10] and rural exodus [11], economic and social risks of monoculture [12], the food versus biofuel dilemma [13, 14], and environmental impacts [15, 16], although the latter question has been more clearly resolved, according to various studies [17-19].

In some regions, the expansion in the ethanol production should be adapted to the local conditions of topography and landholding. In the case of Brazil, the southern State (Rio Grande do Sul) have a differentiated model compared to other States, based on small-scale ethanol production as complementary activity, integrating both production of energy and food. This model requires, in addition to governmental incentive projects, technological development of equipment and processes for the production of ethanol, especially regarding to the distillation column. This unit comprises the largest share of energy consumption and has a high potential to increase its efficiency.

Ethanol production on small scale presents low yields, especially in the distillation stage, with performance of the distillation column of about 66% in the ethanol recovery

* Corresponding author: Flávio Dias Mayer
e-mail: flavio.mayer@ufsm.br
ISSN 0120-6230
e-ISSN 2422-2844

DOI: 10.17533/udea.redin.n78a06

efficiency [20]. The small scale distillation apparatus generally do not achieve the minimum concentration (mass fraction between 92.5 and 93.8% of ethanol) required by the Brazilian National Agency of Petroleum, Natural Gas and Biofuels (ANP) [21]. When this concentration is achieved, a high concentration of ethanol frequently occurs in the bottom product, resulting in 32% of ethanol loss in this stream. In order to solve this problem, a hybrid distiller containing Vigreaux column and Raschig rings in the sections of stripping and rectification, has recently been proposed [20]. However, the optimization process using this hybrid distiller has not been yet investigated. It is known that the experimental factorial design is an adequate optimization method when no mathematical model is available [22]. The use of factorial design methodology for the optimization of continuous distillation process is quite scarce. In some works in literature, the use of factorial design methodology is mainly focused on batch distillation [23], vacuum distillation [24], system using dividing-wall column [25, 26], and membrane distillation [27-29].

Based on the aspects previously mentioned, the aim of this work was to improve the performance of ethanol recovery using a pilot scale distillation column by means of experimental design methodology. For this purpose, a set of experiments was carried out in order to evaluate the influence of feed ethanol concentration and flow rate on the top and bottom products concentration, according to the legal requirements imposed on the market.

2. Experimental

2.1. Apparatus and experimental procedure

Operation analysis of a pilot scale distillation column was investigated by varying the feeding ethanol concentration and mass flow rate using a synthetic solution prepared by the dilution of 92.5 wt% ethanol in water. Schematic diagram of hybrid distillation column used in this work is shown in a previous work [20]. The hybrid distiller (Figure 1) is constituted by the devices and following accessories: a feed pre-heat tank, a reboiler composed of a submerged electrical resistor and an external heating mantle; a fractionating column divided in stripping and rectification modules, composed of 6.0 mm Raschig rings and a Vigreux column, respectively; a condenser, a reflux heating tank, besides temperature and pressure sensors along the distiller connected to a programmable logic controller. The feed stream at 86ºC and 1.0 atm (sub-cooled liquid) was fed to the column using a peristaltic pump previously calibrated according to its rotation velocity. The condenser was refrigerated by water at 20 ºC from a refrigeration unit (cooler). The product samples from the top and bottom were taken in triplicate every 15 minutes throughout the distiller operation. The samples were immediately analyzed on a digital densimeter (Anton Paar, DMA 4500 M) at 20 ºC. The density conversion to mass fraction was performed by the internal routine from digital densimeter, with an estimated accuracy of 0.025 wt%. The desired minimum concentration for the top product was set according to the ANP [21], which establishes a minimum ethanol concentration equal to 92.5% in ethanol fuel (equivalent to 95.1% in volume). The desired ethanol concentration for the bottom product was limited to 0.5 wt%, corresponding to 0.69% in volume) to avoid excessive losses of ethanol. The top and bottom values combined aimed to result as higher as 92% of ethanol recovery. Ethanol recovery as top product was calculated using Eq. (1).

$$\phi = \frac{D.x_D}{F.x_F} \tag{1}$$

Where ϕ is the recovery efficiency, D and x_D are top product mass flow rate and concentration, respectively, and F and x_F are feed mass flow rate and concentration, respectively.

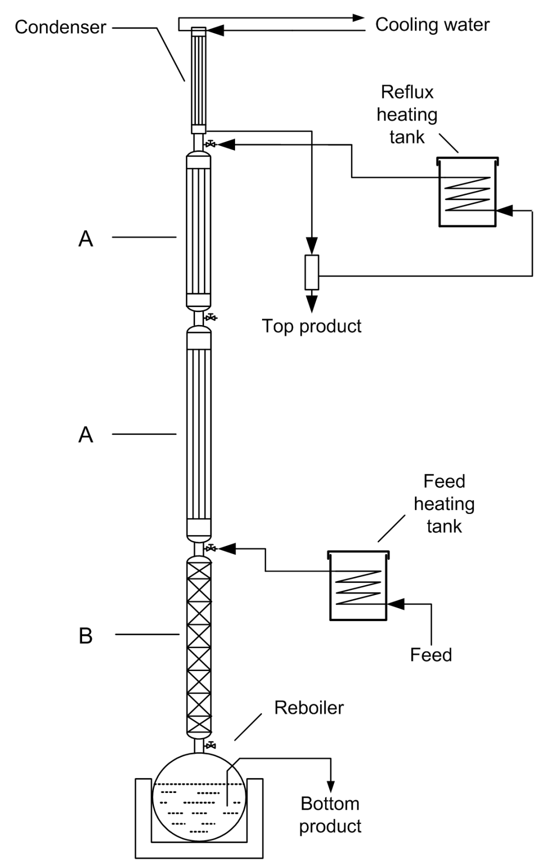


Figure 1 Distillation column flowchart. "A" and "B" refers to the Raschig rings and Vigreux modules, respectively

2.2. Experimental design

In the factorial design, the concentration and the feeding flow rate were defined as independent variables and the concentration of top and bottom products were defined as dependent variables. The minimum and maximum feed concentrations were 4.1 and 6.7 wt% of ethanol, respectively. These concentrations are within the ethanol concentration range obtained by fermentation in small scale ethanol production. Concentration below 4.1 wt% results in excessive heat demand at distillation, while concentration above 6.7 wt% is toxic to yeast during the fermentation step. Therefore, the average mash concentration is around 5.2 wt%. Each experiment was performed considering the maximum internal throughput obtained by adjusting the heating power at the reboiler. Previous tests demonstrated that the adequate temperature of the feed stream was around 86ºC because it led to a better stages distribution between the sections of stripping and rectification. The top product flow rate was defined according to a mass balance that resulted in the highest recovery ratio. The effects of feed stream ethanol concentration and bottom products concentrations as well as mass flow rate on the top and the ethanol recovery efficiency were evaluated by means of a central composite rotatable design (CCRD) [30] for two independent variables, with a total of eleven experimental runs. Table 1 presents the levels of each independent variable investigated. All results were analyzed using Statistica® 7.0 (Statsoft Inc, Tulsa, OK, USA), considering a significance level of 90% ($p < 0.10$). It was used a 90% confidence interval because this study comprises a larger scale of experiment than commonly used in the laboratory, resulting in a greater experimental variation [31].

3. Results and discussion

Table 1 presents the top and bottom products concentrations as well as the ethanol recovery efficiency obtained through the CCRD. The ethanol concentration in the column top ranged from 88.17 wt% (run 3) to 92.39 wt% (run 11), when the bottom ethanol concentration ranged from 0.21 wt% (run 5) to 2.63 wt% (run 4). The recovery efficiency ranged from 80.02 % (run 1) to 94.99 % (run 3). The data from Table 1 show that none of the experiments resulted in a top product with concentration to attend the Brazilian regulation. Also, it was verified that the experimental condition that led to the highest recovery efficiency (run 3) is not in agreement to the Brazilian regulation because the ethanol concentration was below 92.5 wt%. Moreover, the highest ethanol concentration (run 11, 92.39 wt%) was obtained in a condition where the bottom ethanol concentration was too high for a small scale ethanol production, with recovery efficiency around 80%, in addition to not attend the minimum concentration (92.5 wt%).

The analysis of the distillation operation as a function of the feed ethanol concentration, which results from both ethanol concentration and feed flow rate, reveals an important relationship with the reflux ratio. From Table 1 data, it was observed that, for equal feed flow rate, the reflux ratio decreases with the increase of feed ethanol concentration. This can be explained by the mass balance within the distiller: higher amount of ethanol (from both feed concentration and flow rate) implies in a greater productivity of top product, in order to keep constant the recovery ratio. Because the distiller operation has always worked close to its maximum capacity (liquid and vapor flows does not change between the experiments), increasing the top product withdrawal decreases the reflux ratio (L/D).

Although ethanol concentrations obtained for the top products had similar values (average concentration of 90.46 wt% ± 1.34), there was a small relationship between the reflux ratio and the top product concentration. The efficiency of packing columns with finite reflux is similar to the efficiency with total reflux [32]. For that reason, it is expected little influence of the reflux ratio on the packed column efficiency, resulting in a small variation in the top product concentration. This behavior could be verified by comparing the following runs: (1) and (3), where the feed concentration is maintained at a constant value, varying the feed flow rate. The reflux ratio in run (1) was approximately 3.5 times greater than in run (3) and the ethanol concentration in the top and bottom products were lower in run (3); and (9), (10), and (11), which one represents the central point of the study, with identical feed concentration and feed flow rate, resulting in similar reflux ratios and, consequently, in similar concentrations to the top product.

In the runs (5) and (6) where the feed concentration was varied at the extremes of the experimental planning (3.7 and 6.7 wt%), a significant variation in the top and bottom product concentration was not noticed, showing that the use of extreme values are poor operational conditions for the distiller. As for the bottom product, only runs (5) and (8) resulted in a concentration within the established limit, possibly due to the difficulty caused by the use of a Vigreux type column in the stripping section. This is the reason of tests using solutions of 4.1 and 5.2 wt%. Flooding in the tower was also observed in some feed concentrations when the power of the heating mantle was higher than 50% of the total power. The flooding point was premature, probably because of bottlenecks between the module connections.

The experimental results presented in the Table 1 were used to establish the effects of the studied variables, i.e. the effects of feed stream ethanol concentration and bottom products concentrations as well as mass flow rate on the top and the ethanol recovery efficiency. The effects were expressed in the form of Pareto chart, which are presented in the Figure 2. For the top product concentration, the quadratic terms for feed concentration and mass flow rate were statistically significant (Figure 2(a)), whereas other terms as linear and interaction were not significant in the studied range ($p < 0.1$). The negative signs of the quadratic terms indicate the presence of a maximum point. For the bottom ethanol concentration (Figure 2(b)), it was observed that neither the studied variables (concentration and feeding flow rate) were significant in the evaluated range. This indicates that the bottom concentration is statistically the same, regardless the values of the process variables probably due to the design of the stripping section (Vigreux). For the recovery efficiency (Figure 2(c)), linear and quadratic terms for feed concentration as well as the interaction between the feed concentration and mass flow rate were statistically significant ($p < 0.1$). Increasing the feed concentration led to a decrease in the recovery efficiency, whereas the positive sign of quadratic term for feed concentration indicates the presence of a minimum point in the system.

Table 1 Mean ethanol concentration for top (Y_1) and bottom (Y_2) products, reflux rate and ethanol recovery efficiency

Run	Feed ethanol concentration (wt%)[a]	Feed flow rate (kg h^{-1})	Ethanol concentration (wt%)		Reflux ratio (L/D)[b]	Recovery Efficiency (%)[c]
			Top	Bottom		
1	4.1 (-1)	2.20 (-1)	91.33	0.95	6.95	80.02
2	6.3 (1)	2.20 (-1)	90.82	1.86	2.87	83.78
3	4.1 (-1)	3.93 (1)	88.17	0.69	1.94	94.99
4	6.3 (1)	3.93 (1)	89.77	2.63	1.26	83.14
5	3.7 (-1.41)	3.02 (0)	88.55	0.21	3.91	90.93
6	6.7 (1.41)	3.02 (0)	90.65	0.51	3.81	83.50
7	5.2 (0)	1.85 (-1.41)	89.67	1.27	4.06	84.21
8	5.2 (0)	4.39 (1.41)	90.40	0.31	3.32	81.75
9	5.2 (0)	3.02 (0)	91.36	1.59	3.39	80.70
10	5.2 (0)	3.02 (0)	92.01	1.34	3.77	81.27
11	5.2 (0)	3.02 (0)	92.39	1.02	3.58	81.60

[a] Code of experimental planning point in parenthesis; [b] Reflux ratio is the ratio of reflux (*L*) and top product (*D*) flow rate; [c] Calculated according to Eq. (1).

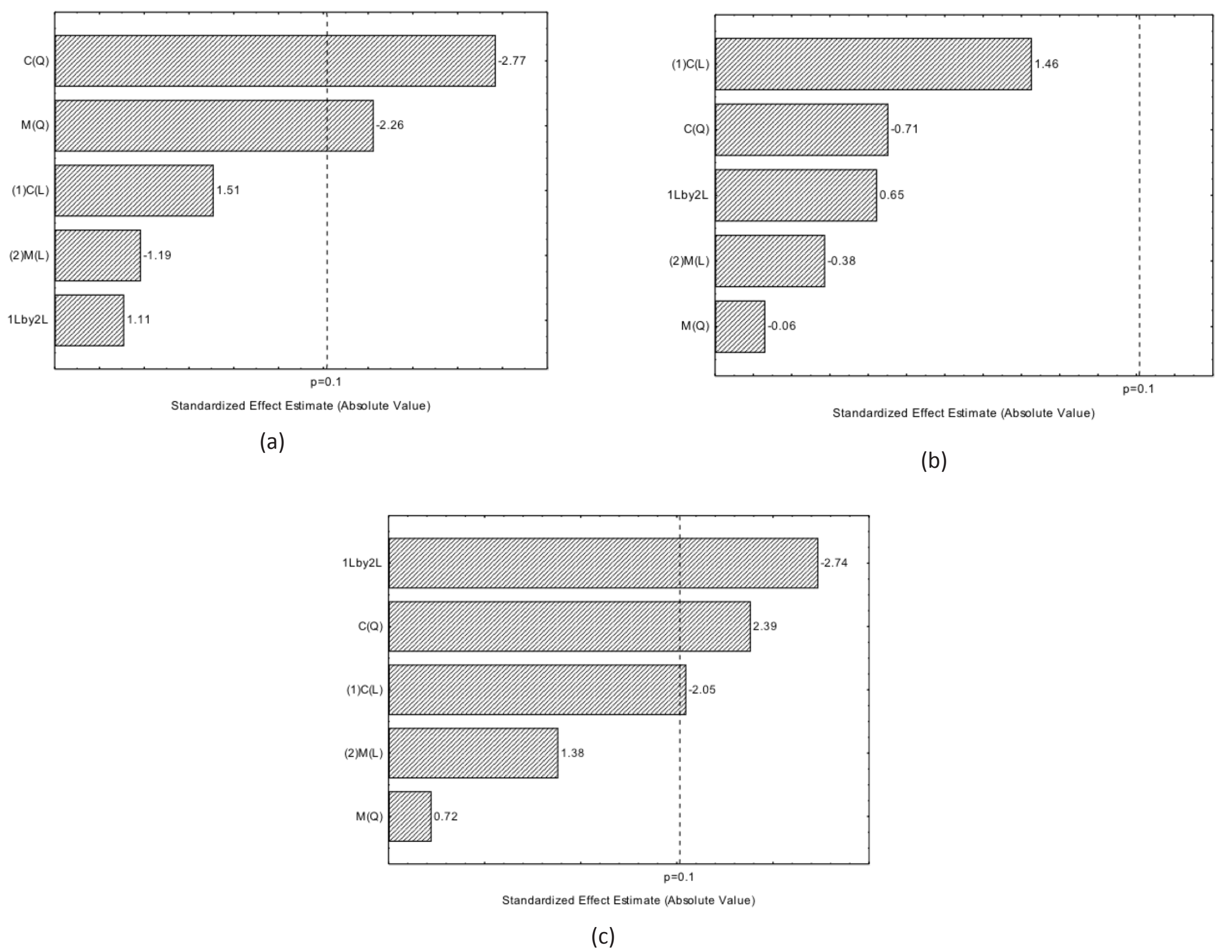


Figure 2 Pareto chart showing the effects of linear, quadratic and interaction terms of independent variables on top ethanol concentration (a), bottom ethanol concentration (b) and recovery efficiency (c)

In order to optimize the experimental conditions to obtain a maximum top product concentration and also higher recovery efficiency, two empirical models were applied considering the significant effects from the assessed parameters. The model is represented below, where Eq. (2) and Eq. (3) represent the ethanol concentration in the top product and the recovery efficiency, respectively. The significance of each term for ethanol concentration in the top and recovery efficiency can be found in Table 2 and Table 3, respectively.

$$Eth = 91.92 - 1.11.C^2 - 0.89.M^2 \quad (2)$$

$$\phi = 81.19 - 2.32.C + 3.10.C^2 - 3.90.C.M \quad (3)$$

Where: *Eth* is the ethanol concentration in the top product (wt%), ϕ is the recovery efficiency, *C* and *M* are the coded feed concentration and mass flow rate, respectively. These models were validated by analysis of variance (ANOVA). The calculated F-test for Eq. (2) and Eq. (3) were about 1.7 and 1.3 times greater than the tabulated ones for significance at $p = 0.1$, and the determination coefficients (R^2) were 0.7474 and 0.7942, respectively. The values for the determination coefficient indicate satisfactory fitting of experimental data, allowing the use of such models to predict process performance as well as a tool for process optimization.

Figure 3(a) shows the contour curve response for the top product concentration. It is possible to observe the existence of an optimum operational region with high top product concentration, as a function of feed ethanol concentration and feed flow rate. This region is located at a feed concentration ranging from 5.0 and 5.8 wt%, and at a feed flow rate from 2.85 and 3.20 $kg.h^{-1}$. It is important to mention that in this optimum operational region would be possible to obtain a top product ethanol concentration in accordance with the ANP regulation [21]. The range

Table 2 Estimated regression coefficients for ethanol concentration in the top product (*Eth*)

Factor	Coefficients	Standard error	*t(5)*	*p-value*
Mean/interaction	91.92	0.548	167.629	<0.001
C (L)	0,50748	0.673	1.510	0.191
C (Q)	-1,10875	0.803	-2.771	0.039
M (L)	-0,39720	0.673	-1.186	0.289
M (Q)	-0,89125	0.803	-2.225	0.077
C x M	0,52750	0.950	1.111	0.317

Table 3 Estimated regression coefficients for recovery efficiency (ϕ)

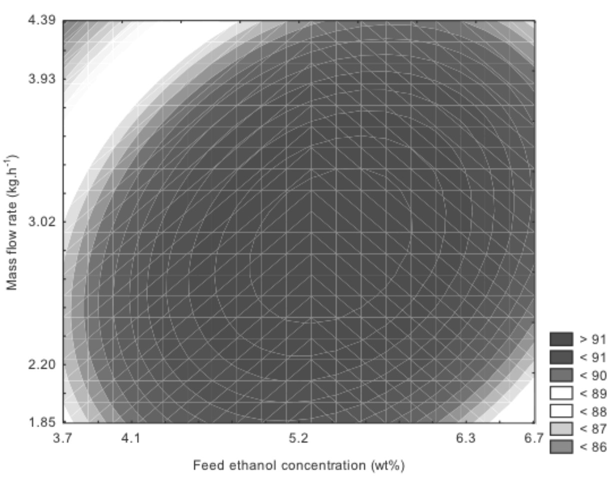
Factor	Coefficients	Standard error	*t(5)*	*p-value*
Mean/interaction	81.19	1.752	46.332	<0.001
C (L)	-2,32470	2.149	-2.049	0.096
C (Q)	3,10875	2.565	2.385	0.063
M (L)	1,35638	2.149	1.384	0.225
M(Q)	0,99125	2.565	0.724	0.501
C x M	-3,90250	3.035	-2.736	0.041

Table 4 Validation of model predictions for top ethanol concentration in experimental conditions around the optimum point

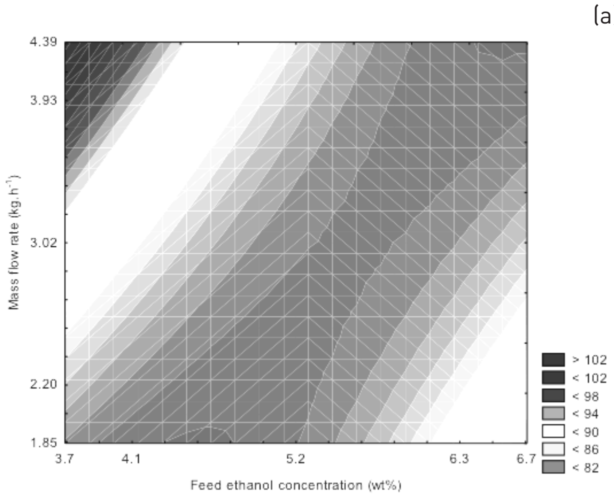
Feed ethanol concentration (wt %)	Feed flow rate (kg h^{-1})	Experimental Top ethanol concentration (wt %)	Calculated Top ethanol concentration (wt %)
6.0	2.56	93.18	90.80
6.0	2.56	93.13	90.80
6.0	2.56	92.65	90.80
6.0	3.48	92.92	89.86
6.0	3.48	92.49	89.86
6.0	4.39	92.87	88.65
5.2	3.02	87.22	91.92

of concentration defined in this study requires a quality control in the fermentation step, in a way to avoid excessive energy demand to distillate low ethanol concentration feed. Moreover, ethanol feed concentrations above the maximum value (6.7 wt%) tends to result in high concentration of ethanol in top product, but also an excessive loss of ethanol in bottom product, because of the low height of the stripping section.

Nevertheless, the recovery effectiveness at the optimized region for top ethanol concentration was between 82 and 86% (Figure 3(b)). The highest recovery efficiency was obtained at a feed ethanol concentration around 4 wt% and feed flow rate ranging from 3.50 to 4.39 kg·h^{-1}. In order to validate the Eq. (2), Table 4 presents some experiments aiming the validation of model prediction and to confirm the optimized condition. As can be seen, there is a satisfactory agreement among predicted and experimental top concentration for all experiments. These results confirm that the model is a reliable tool to apply in process optimization.



(a)



(b)

Figure 3 Contour plots showing the influence of independent variables on top product ethanol concentration (a) and recovery efficiency (b)

4. Conclusions

The analysis of the experiments in the distillation column demonstrates that the top product concentration (dependent variable) was influenced by the independent variables: feed ethanol concentration and flow rate. Therefore, it was possible to establish an optimum operating region for the hybrid distiller. This requires a good quality control on fermentation step and in the operating conditions of distillation in order to obtain a product suitable for the market. However, it was verified that the independent variables or their interaction had no significant influence on the bottom product concentration, showing that the effects were not relevant for the stripping section. The experimental design methodology proved to be an important tool to improve the operation of a bench scale distillation column. In this work, ethanol fuel was not obtained in accordance with the Brazilian laws (around 92.5 wt%) as the maximum concentration for the top product was 92.39 wt% (run 11), using a feed concentration of 5.2 wt% in ethanol and 3.09 kg h^{-1}. The performance of the hybrid distiller was satisfactorily demonstrated by the ethanol recovery ratio, reaching values above at 80%, being that in two tests, over 90%. Also, it was found a recovery ratio around 83% in the optimized condition, which reinforces consistency as compared with common systems for producing ethanol on small-scale.

5. References

1. Empresa de Pesquisa Energética, *Balanço Energético Nacional, Rio de Janeiro*, 2011. [Online]. Available: https://ben.epe.gov.br/downloads/Relatorio_Final_BEN_2011.pdf. Accessed: Jun. 9, 2015.
2. J. Puppim, "The policymaking process for creating competitive assets for the use of biomass energy: the Brazilian alcohol programme", *Renew. Sustain. Energy Rev.*, vol. 6, no. 1-2, pp. 129-140, 2002.
3. F. Rosillo and L. Cortez, "Towards ProAlcool II - A review of the Brazilian bioethanol programme", *Biomass and Bioenergy,* vol. 14, no. 2, pp. 115-124, 1998.
4. J. Rico, S. Mercedes and I. Sauer, "Genesis and Consolidation of the Brazilian Bioethanol: A Review of Policies and Incentive Mechanisms", *Renew. Sustain. Energy Rev.,* vol. 14, pp. 1874-1887, 2010.
5. G. Sorda, M. Banse and C. Kemfert, "An Overview of Biofuel Policies Across the World", *Energy Policy,* vol. 38, pp. 6977-6988, 2010.
6. M. Rajcaniova, D. Drabik and P. Ciaian, "How policies affect international biofuel price linkages", *Energy Policy,* vol. 59, pp. 857-865, 2013.
7. Associação Nacional dos Fabricantes de Veículos Automotores (ANFAVEA), Anuário da Indústria Automobilística Brasileira, 2013. [Online]. Available: http://www.anfavea.com.br/anuario2013/anuario2013.zip. Accessed: Jun. 9, 2015.
8. X. Du and M. Carriquiry, "Flex-fuel Vehicle Adoption and Dynamics of Ethanol Prices: Lessons from Brazil", *Energy Policy*, vol. 59, pp. 507-512, 2013.

9. A. Veiga and P. Ramos, "PROÁLCOOL e evidências de concentração na produção e processamento de cana-de-açúcar", *Informações Econômicas*, vol. 36, pp. 48-61, 2006.
10. A. Hira, "Sugar Rush: Prospects for a Global Ethanol Market", *Energy Policy,* vol. 39, pp. 6925-6935, 2011.
11. L. Ortiz, S. Schlesinger and S. Noronha, *Agronegócios e Biocombustíveis: Uma Mistura Explosiva*, 2006. [Online]. Available: http://www.natbrasil.org.br/docs/biocombustiveis/biocomb_port.pdf. Accessed: Jun. 9, 2015.
12. B. Ribeiro, "Beyond Commonplace Biofuels: Social Aspects of Ethanol", *Energy Policy*, vol. 57, pp. 355-362, 2013.
13. C. Runge and B. Senauer, "How Biofuels Could Starve the Poor", *Foreign Aff.*, vol. 86, pp. 41-53, 2007.
14. M. Harvey and S. Pilgrim, "The New Competition for Land: Food, Energy, and Climate Change", *Food Policy*, vol. 36, pp. S40-S51, 2011.
15. A. Buyx and J. Tait, "Biofuels : Ethics and Policy-Making", *Biofuels, Bioprod. Biorefining*, vol. 5, pp. 631-639, 2011.
16. H. Azadi, S. Jong, B. Derudder, P. Maeyer and F. Witlox, "Bitter Sweet: How Sustainable is Bio-Ethanol Production in Brazil?", *Renew. Sustain. Energy Rev.*, vol. 16, pp. 3599-3603, 2012.
17. R. Janssen and D. Rutz, "Sustainability of Biofuels in Latin America: Risks and Opportunities", *Energy Policy*, vol. 39, pp. 5717-5725, 2011.
18. A. Walter *et al.*, "Sustainability Assessment of Bio-Ethanol Production in Brazil Considering Land Use Change, GHG Emissions and Socio-Economic Aspects", Energy Policy, vol. 39, pp. 5703-5716, 2011.
19. H. Nuñez, H. Önal and M. Khanna, "Land Use and Economic Effects of Alternative Biofuel Policies in Brazil and the United States", *Agric. Econ.*, vol. 44, pp. 487-499, 2013.
20. F. Mayer, R. Hoffmann and R. Hoffmann, "An Innovative Project Involving an Appropriate Hybrid Distillation System for Small-Scale Ethanol Fuel Production", *Chem. Eng. Commun.*, vol. 200, pp. 563-574, 2013.
21. Agência Nacional de Petróleo, Gás Natural e Biocombustíveis (ANP), *Resolução ANP n° 07/2011*. [Online]. Available on: http://nxt.anp.gov.br/NXT/gateway.dll/leg/resolucoes_anp/2011/fevereiro/ranp%207%20-%202011.xml. Accessed: Jun. 9, 2015.
22. C. Vasconcelos, M. Wolf, "Optimisation, Dynamics and Control of a Complete Azeotropic Distillation: New Strategies and Stability Considerations", in *International Conference on Distillation and Absorption*, Baden-Baden, Germany, 2002, pp. 1-20.
23. F. Mayer, G. Schettert, R. Michel Jr., E. Foletto and R. Hoffmann, "Operation Parameters of a Small Scale Batch Distillation Column for Hydrous Ethanol Fuel (HEF) production", *Ing. Investig.*, vol. 35, pp. 31-35, 2015.
24. A. Oterhals, B. Kvamme and M. Berntssen, "Modeling of a Short-Path Distillation Process to Remove Persistent Organic Pollutants in Fish Oil Based on Process Parameters and Quantitative Structure Properties Relationships", *Chemosphere*, vol. 80, pp. 83-92, 2010.
25. N. Long and M. Lee, "Design and optimization of a dividing wall column by factorial design", *Korean J. Chem. Eng.,* vol. 29, pp. 567-573, 2012.
26. N. Long, Y. Kwon and M. Lee, "Design and Optimization of Thermally Coupled Distillation Schemes for the Trichlorosilane Purification Process", *Appl. Therm. Eng.*, vol. 59, pp. 200-210, 2013.
27. G. Chen, Y. Lu, W. Krantz, R. Wang and A. Fane, "Optimization of Operating Conditions for a Continuous Membrane Distillation Crystallization Process With Zero Salty Water Discharge", *J. Membr. Sci.*, vol. 450, pp. 1-11, 2014.
28. P. Onsekizoglu, K. Bahceci and J. Acar, "The Use of Factorial Design for Modeling Membrane Distillation", *J. Membr. Sci.*, vol. 349, pp. 225-230, 2010.
29. A. Sun, W. Kosar, Y. Zhang and X. Feng, "Vacuum Membrane Distillation for Desalination of Water Using Hollow Fiber Membranes", *J. Membr. Sci.*, vol. 455, pp. 131-142, 2014.
30. M. Bezerra, R. Santelli, E. Oliveira, L. Villar and L. Escaleira, "Response Surface Methodology (RSM) as a Tool for Optimization in Analytical Chemistry", *Talanta,* vol. 76, pp. 965-977, 2008.
31. M. Rodrigues and A. Iemma, *Planejamento de Experimentos e Otimização de Processos*, 2^{nd} ed. Campinas, Brazil: Cárita Editora, 2014.
32. H. Kister, *Distillation Design*, 1^{st} ed. New York, USA: McGraw-Hill, 1992.