

Optimization the Continuous Distillation Process of an Aqueous Ethanol Mixture

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Abstract

Worldwide value of ethanol as a renewable source of environmentally friendly energy makes it necessary to continue research and documentation of operating conditions to optimize production process. This study determined conditions of feed flow rate (FR) and reflux ratio (RD) maximizing the distillate concentration (DC) obtained in the distillation of a 20% v/v ethanol aqueous mixture. For this purpose, a continuous distillation unit of seven plates with bells with a 1.01325 bar pressure was used. A factorial experiment 3^2 corresponding to three feed flow rate levels (FR: 0.5 Lh⁻¹, 1.5 Lh⁻¹ y 2.5 Lh⁻¹) and three reflux ratio levels (RD: 1, 3 and 5) were randomly developed under one in-block design. Results showed that interaction of feed flow rate and reflux ratio significantly affected distillate concentration ($p < 0.05$). It was determined that the polynomial function that relates the distillate concentration (DC) with feed flow rate (FR) and reflux ratio (RD) corresponds to equation $DC = 49.15 + 35.54(FR) + 2.52(RD) - 7.50(FR)^2 - 0.52(FR)(RD)$ ($R^2 = 0.98$). Optimum point of the process variables maximizing distillate concentration was found by using the response surface method, with 96.78% v/v being the maximum distillate concentration obtained from a feed flow rate of 2.16 Lh⁻¹ and one reflux ratio of 5.

Keywords: concentration, distillate, optimum, reflux.

INTRODUCTION

Growing world demand of energy requires improving efficient use and generation process. As energy prices keep escalating, energy conservation becomes the main concern for all industries. Distillation is probably the most studied unitary operation in terms of optimization and control. Considering that distillation columns are the main energy units in chemical plants and, given the recent increase in fuel costs, it is essential to find better operating strategies and control systems to optimize these processes. Energy consumption during distillation can have a strong impact on overall profitability; therefore, distillation column optimization should aim at higher production and higher quality of distillate. A distillation column is a multivariate system in which perturbation of any parameter can affect overall performance as well as cause production loss [1].

Current trends towards use of more environmentally friendly fuel sources have generated major changes in public policy of nations aiming at supporting production of alternative fuels; thereby, the European Union plans to replace approximately

6% of diesel and petrol consumption with alternative fuels such as biodiesel and bioethanol by 2020 [2]. Accordingly, ethanol distillation processes are of great industrial interest, not only because they are a renewable source of energy but also because of practicality of synthesis from lignocellulosic material [3] such as some residues from food industry [4]. On the other hand, replacement of fossil fuels such as gasoline with bioethanol can significantly reduce emissions of air pollutants [5]. Distillation is the separation technique most frequently used in the chemical industry for purification of bioethanol [6]. The growing demand for high value-added chemical and biochemicals contained in small mixing volumes as well as great flexibility of operation are some incentives that currently highlights the importance of continuous distillation [7], [8].

Ethanol has different uses, either alone or mixed, ranging from fuels, culinary products, pharmaceuticals to chemical industry and others. A major quality is its renewable property and little affectation to the environment [9], [10]. Continuous research and documentation of aspects such as volume and production efficiency and operating conditions are necessary to allow better yields [11].

Ethanol distillation process is affected by many factors. They include reflux ratio, inlet temperatures, initial feed concentration, feed flow and others. The theoretical determination of these factors is very complicated due to the occurring mass and energy transfer processes and use of approximate calculation parameters under a concept of ideality that detract from the real values of results [12].

Coelho *et al.* (2012) evaluated recovery of bioethanol from banana crop residues as a function of reflux ratio [13]. He used a standard ethanol-water mixture, working at full reflux and with 0.5, 1 and 2 proportions. The best result was obtained with the highest reflux ratio.

Lara and Barroso (2015) explored different scenarios where minimum costs are obtained for different flow conditions and feed concentrations. They concluded that at higher feed flow the cost per kmol of anhydrous ethanol decreases. They also encountered that costs increase as ethanol concentration decreases [8].

Gonzalez and Cardenas (2015) studied operating conditions of a reaction-separation process to produce ethanol. They found that the region of operability is located between 8 and 100 reflux ratio values. They also affirm that steady states of high conversion for ethanol are located at values in the reflux ratio higher than 30, generating an increase in the separation of the products from their reagents [14].

Rodríguez *et al.* (2015) evaluated the behavior of a continuous-flow rectification column from an ethanol-water mixture. They varied the column operating parameters, determining the best operating conditions according to operation reflux and feed temperatures. Results showed that at a higher feed temperature there was more separation in the continuous process, and that, with a higher reflux, the composition of the distillate was obtained [12].

Manayay *et al.* (2015) determined optimal parameters for the continuous distillation of an ethanol-water binary mixture. They found that with a 300mBar pressure and a 0.9 reflux, a maximum concentration of 98.78% (v/v) ethanol was obtained [15].

The objective of this research study was to find the operating conditions of the continuous distillation process in terms of feed flow rate and reflux ratio maximizing the efficiency of the separation process in terms of the obtained distillate concentration. This study allows the improvement of operating conditions leading to greater performance of a common and important process in various industrial sectors; in addition to contrasting theoretical foundations that govern this operation and contributing to future research.

MATERIALS AND METHODS

A continuous distillation unit (Elettronica Veneta®, model UDCA / EV) consisting of seven plates with bells, a boiler and a condenser was used for determination of feed flow rate (FR) and reflux ratio (RD) conditions; maximizing concentration of distillate (CD) obtained in the distillation of a 20% v/v ethanol aqueous mixture.

A factorial experiment 3^2 corresponding to three levels of feed flow rate (FR) was developed: 0.5 Lh⁻¹, 1.5 Lh⁻¹ and 2.5 Lh⁻¹, and three levels of reflux ratio (RD): 1, 3 and 5; under a completely random block design, with a 0.05 significance level. Three replicates were performed for a total of twenty-seven experimental trials.

To startup the distillation process, the system worked under a 1.01325 bar pressure. Feed tank and boiler were filled with the 20% v/v ethanol mixture which was brought to boiling point by operating the column at full reflux for about 5 minutes until the distillation unit was stabilized under a recorded 78 ° C temperature profile in the tower's upper plate. The system was fed according to the feed flow rate (FR) levels to be operated and the reflux ratio (RD) was set according to the test levels. For each test performed according to the evaluated treatments, the respective distillate concentration (DC) was determined through an Al-Ambik® alcoholemeter.

For the data statistical treatment, the R program v.2.9.1® (The R Foundation for Statistical Computing, 2009) was used. MATLAB® software package was used to estimate the optimum operating conditions that maximize ethanol concentration.

RESULTS AND DISCUSSION

Analysis of variance ANOVA showed that factors feed flow rate (FR) and reflux ratio (RD) were statistically significant ($p < 0.05$) for the response variable distillate concentration (CD). The interaction of the feed flow rate-reflux ratio factors (FR: RD) was also statistically significant indicating that there is a greater effect given by the simultaneous action of these factors on the response variable obtained from the distillate concentration.

Values of respective linear and quadratic effects and their interaction for the factors are shown in Table 1. The linear and quadratic effects of feed flow rate (FR and FR²) were significant ($p < 0.05$); indicating that there are differences between distillate concentration obtained from a feed flow level of 2.5Lh⁻¹ and that obtained with a feed flow of 0.5Lh⁻¹. In addition, it was possible to establish that this effect is positive, i.e. the concentration of ethanol obtained in the distillate stream increases as feed flow increases.

The significance of the feed flow quadratic effect means that there is a curvature in the distillate concentration response variable when the feed flow is increased. It could be stated that this effect is given by a slight downwardly open curvature which has a relative maximum in the distillate concentration obtained between the feed flow levels employed.

Table 1: Analysis of variance (ANOVA) disaggregated.

Effects	Grades of Freedom	Sum of Squares	Mean Square	F_0	Value-p
FR	1	2244.50	2244.50	1683.38	<2.20E-16*
FR ²	1	337.50	337.50	253.13	3.15E-11*
RD	1	180.50	180.50	135.38	3.21E-09*
RD ²	1	1.50	1.50	1.13	3.05E-01 ^{NS}
FR * RD	1	18.75	18.75	14.06	1.75E-03*
FR * RD ²	1	0.25	0.25	0.19	6.71E-01 ^{NS}
FR ² * RD	1	0.25	0.25	0.19	6.71E-01 ^{NS}
FR ² * RD ²	1	0.75	0.75	0.56	4.64E-01 ^{NS}
Residuals	16	21.33	1.33		

* Indicates significance of the effect ($p < 0.05$)

^{NS} indicates non-significant ($p > 0.05$)

The reflux ratio linear effect (RD) was significant ($p < 0.05$). This indicates that distillate concentration response at the 1 reflux ratio level is different from the response obtained from a 5 reflux ratio. It was further established that such an effect is positive, i.e. the concentration of the distillate is increased by increasing reflux ratio.

The linear effects interaction of factors feed flow rate and reflux ratio (FR * RD) was equally significant ($p < 0.05$). Averages

obtained in the distillate obtained according to evaluated treatments are shown in table 2.

Table 2: Average Distillate Concentration % v/v ethanol

	FR: 0.5Lh ⁻¹	FR: 1.5Lh ⁻¹	FR: 2.5Lh ⁻¹
RD: 1	67	87	92
RD: 3	72	91	94
RD: 5	76	93	96

The linear effects interaction of factors feed flow rate and reflux ratio (FA*RD) means that the distillate concentration obtained from a feed flow (FR) with the 1 reflux ratio level is statistically different from that obtained with the 5 reflux ratio level; with a positive effect. This indicates an increase in ethanol concentration when passing from a reflux ratio from 1 to 5.

Table 3: Analysis for the multiple linear regression model.

Parameter	Estimate	Value-p
Intercepto	49.1458	<2.00E-16***
FR	35.5417	5.69E-16***
RD	2.5208	7.08E-08***
FR ²	-7.5000	1.44E-12***
FR · RD	-0.6250	2.89E-03***

* Indicates significance of the effect (p < 0.05)

Regression Analysis (Table 3) lists each of the factors coefficients that were statistically significant. For the model, the determination coefficient of R² was 0.987; indicating that the model represents a 98.7% of the phenomenon described, i.e. the percentage of ethanol as a function of the factors studied, feed flow and reflux ratio.

From Table 3 all model parameters were statistically significant (p < 0.05), therefore, the equation (1) representing distillate concentration (DC) as a function of feed flow rate (FR) and reflux ratio (RD) is:

$$DC = 49.1458 + 35.5417 \cdot FR + 2.5208 \cdot RD - 7.5000 \cdot FR^2 - 0.6250 \cdot FR \cdot RD \quad (1)$$

where:

DC = Distillate concentration (% v/v ethanol)

FR = Feed flow rate (Lh⁻¹)

RD = Reflux ratio

Figure 1 represents the behavior of distillate concentration as a function of feed flow rate and reflux ratio, according to the given equation. The operating conditions that maximize the distillate concentration correspond to a 2.1611 L h⁻¹ feed flow rate (FR) and a 5 reflux ratio (RD); from which a 96.78% v/v optimum ethanol concentration value is obtained.

Results showed that optimum point is reached under maximum value of reflux ratio and with a feed flow value close to the upper end of the study range. These results agree with studies carried out by Coelho et al. (2012) [13], Rodriguez et al. (2015) [12] and Gonzalez and Cardenas (2015) [14], who found a higher yield of ethanol for a higher reflux ratio; and with the studies of Zhao et al. (2016) [16] who had higher yield through higher feed flow rate. In addition, to higher food flow rate, operating costs are reduced [8].

Although increasing the reflux ratio increases the distillate concentration obtained, it must be considered that an excess in the reflux ratio increases energy consumption to partially condense vapor flow [14]. For this reason, it is necessary to evaluate process energy under the optimal operating conditions encountered.

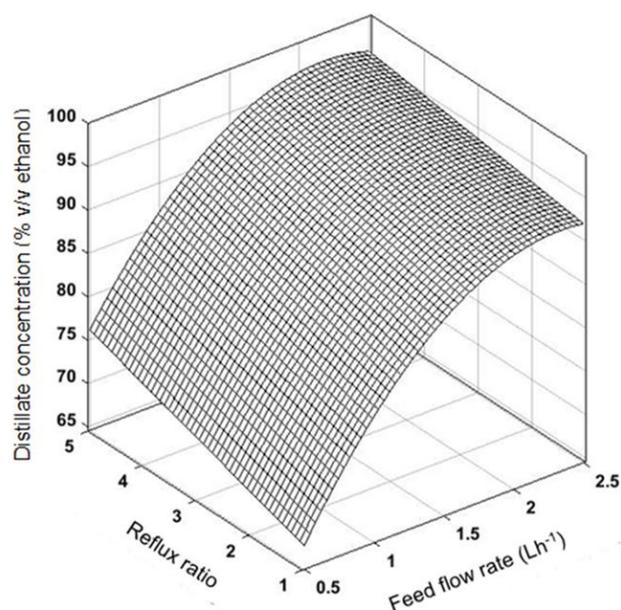


Figure 1: Response surface for distillate concentration

The obtained 96.78% v/v maximum distillate concentration was lower than the optimum concentration determined by Manayay et al. (2015) [15], who obtained a 98.78% v/v ethanol but working under pressures below the atmospheric one (300mbar) and a 0.9 reflux ratio.

CONCLUSION

It was established that the optimal point of process variables maximizing distillate concentration of a 20% v/v aqueous ethanol mixture is given by a 2.16 L h⁻¹ feed flow rate and a 5.0 reflux ratio. With these conditions, a maximum ethanol concentration of 96.78% v/v is obtained using the Elettronica Veneta® continuous distillation column of 7 plates with bells, operating at atmospheric pressure. It was found that as the reflux ratio increases, the distillate concentration also increases, while the feed flow rate generates a quadratic effect on the response variable ethanol concentration, given by a slight downward curvature with a maximum value corresponding to a 2.16L^{h-1} feed flow rate.

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