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STUDY OF THE HYDROUS ETHANOL PURIFICATION STAGE: SIMULATION AND SENSITIVITY ANALYSIS EXTRACTIVE DISTILLATION USING DWSIM SIMULATOR

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Abstract: The ethanol purification stage has a high energy demand. The work therefore aims to simulate the extractive distillation of ethanol-water with ethylene glycol, using the DWSIM v.8.6 simulator. Sensitivity analysis will be used to simulate the extraction column. The results of the analysis revealed that the temperature, solvent flow rate and pressure of the azeotropic mixture are crucial to the process, obtaining ethanol with 98.86% purity, but further studies are needed in order to obtain 99.5% ethanol.

Keywords: Ethanol; Purification; Distillation, azeotropic mixture, DWSIM.

INTRODUCTION

Ethanol is a widely used biofuel, and its production has been significant in Brazil since the late 1970s, driven by government policies for fuel diversification strategies, such as the Proálcool program. In 1997, the Kyoto Protocol reinforced the importance of biofuels in reducing CO₂ emissions, encouraging ethanol production. Public incentives for sugar cane cultivation have led Brazil to become the world's second largest ethanol producer, behind the United States, which produces ethanol from corn (Soares and Junior, 2021).

From the fermentation of sugars, ethanol contains other components that can affect its performance (Martins, 2022), which is why the purification of ethanol is essential to use it in various areas such as the chemical and food industries and, with an alcohol content of 92.5% by mass, hydrated ethanol is used as a biofuel (ANP, 2022). Distillation is a process in which ethanol is separated from water, an azeotropic mixture, in which azeotropic and extractive distillation methods can be used. Azeotropic distillation is characterized by the addition of a third component, such as benzene, pentane and cyclohexane, called a formant azeotrope, which alters the volatility of the mixture, allowing it to be separated.

While extractive distillation uses a drag agent, such as glycols and glycerol, immiscible with the azeotropic mixture, which increases the volatility of the key component, enabling separation (Gil et al, 2014).

This study proposes the simulation of the extractive distillation of the ethanol-water system using the ethylene glycol solvent, followed by the recovery of this solvent. The simulation data will be based on previous results from Purgatto et al. (2016), who used the ASPEN HYSYS[®] Simulation Software, version 8.6 For this work, the free software DWSIM will be used for the simulation, along with the Chemsep package, without the use of solvent recycling. The aim is to analyse the results obtained in the free software and study the system using sensitivity analysis to obtain optimum values for the input parameters, thus maximizing the anhydrous ethanol obtained and minimizing energy consumption.

MATERIAL AND METHODS

The aim of this work is to simulate and analyze the extractive distillation of the ethanol-water system, using the solvent ethylene glycol, followed by the distillation to recover the solvent. The aim of the analysis is to obtain optimum values for the input parameters in order to maximize the production of hydrated ethanol and minimize energy consumption.

To this end, operational models were developed in the DWSIM simulator, version 8.7, in order to reproduce the simulation carried out by Purgatto et al. (2016) in the ASPEN HYSYS simulator, of the extractive distillation of the azeotropic mixture to obtain hydrated ethanol at a mass fraction of 95%, and then use the results to obtain the sensitivity analysis of the system in order to maximize the obtaining of ethanol.

In the simulation, water, ethanol and ethylene glycol were added as components and the NRTL thermodynamic model and

SI Engineering unit systems were used. Two ChemSep extractive distillation columns were used in the simulation flowchart, where the first was called the extractive column and the second the recovery column.

The extraction column consists of 15 plates, with a reflux ratio of 2.825, where the ethanol-water mixture is distilled using ethylene glycol as a carrier. The ethanol-water mixture enters the feed stream in the eighth dish, at a flow rate of 5173 kg/h, a temperature of 111.2°C and a pressure of 3.5 bar, the compositions of ethanol and water are 73% and 27% respectively. While the ethylene glycol stream with a flow rate of 20000 kg/h is fed into the second plate, at a composition of 99% ethylene glycol and 1% water, at 256 °C and 4.5 bar.

The residue generated in the extraction column is composed mainly of ethylene glycol and water and is fed into the recovery column to regenerate the solvent. The recovery column is made up of 10 plates, with the feed occurring in the fifth plate and a reflux ratio of 3. The simulations were carried out in order to obtain a 95% ethanol concentration in the extraction column and 99.9% solvent in the regeneration column, with both columns having an efficiency of 100%.

The simulation data was adjusted using the NRTL (non-random two-liquid) model (Figure 01 and Figure 02), which takes into account the different interactions present between the molecules in the system when calculating the activity coefficient of a solute in a liquid solvent, in totally or partially soluble systems (Renon and Prausnitz, 1968). In addition, it is applied to numerous systems, such as mixtures of polar, partially miscible and non-electrolyte components, as well as non-ideal systems, and is widely used for modeling liquid-vapor equilibrium and liquid-liquid equilibrium (Albuquerque, 2015; Sosa et al., 2017).

By adjusting the bibliographic data (Lei et al., 2002) using the NRTL method, the liquid-vapor equilibrium graphs (Figure 01 and Figure 02) were obtained for the ethanol-water mixture.

Based on the simulation data previously presented, sensitivity analyses were carried out in order to find out which parameters have the greatest influence on the energy consumption of the process, making it possible to reduce energy consumption without compromising the quality of the end product. This is especially relevant in the context of the search for more sustainable and economical processes, highlighting the importance of studies such as the one presented in this paper.

RESULTS AND DISCUSSION

The distillation process flow chart is shown in Figure 3, which shows an extraction column with a 73% feed input Ethanol and 23% water, and 99% ethylene glycol solvent. Next is the recovery column, which receives the flow from the base of the extraction column, this separates the water and ethylene glycol.

Using data from Purgatto et al. (2016), a simulation was carried out in which unsatisfactory results were obtained, with a 45% mass fraction of ethanol at the top of the extraction column and an energy flow in the reboiler of - 4677 kW. As a result, a sensitivity analysis of the system was carried out using the DWSIM software. The solvent recovery column performed satisfactorily and constantly, achieving 99% ethylene glycol at the bottom of the regenerative column, so no sensitivity analysis was applied at this stage. The first parameter studied in the sensitivity analysis, carried out only on the extraction column, was the mass flow rate of the solvent. As the flow rate increased, there was a continuous increase in the molar fraction of ethanol (Figure 4a), reaching a maximum value of 99.85 % (molar) at a flow rate of 16,000

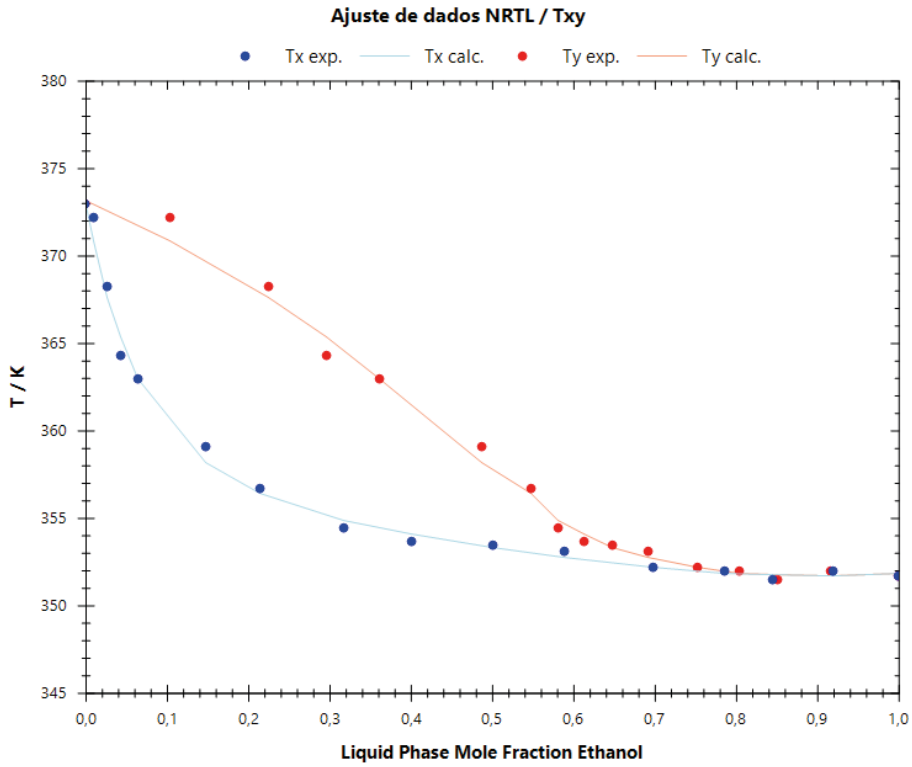


Figure 01. T-x-y diagram with NRTL data adjustment
 Source: Own collection (2024).

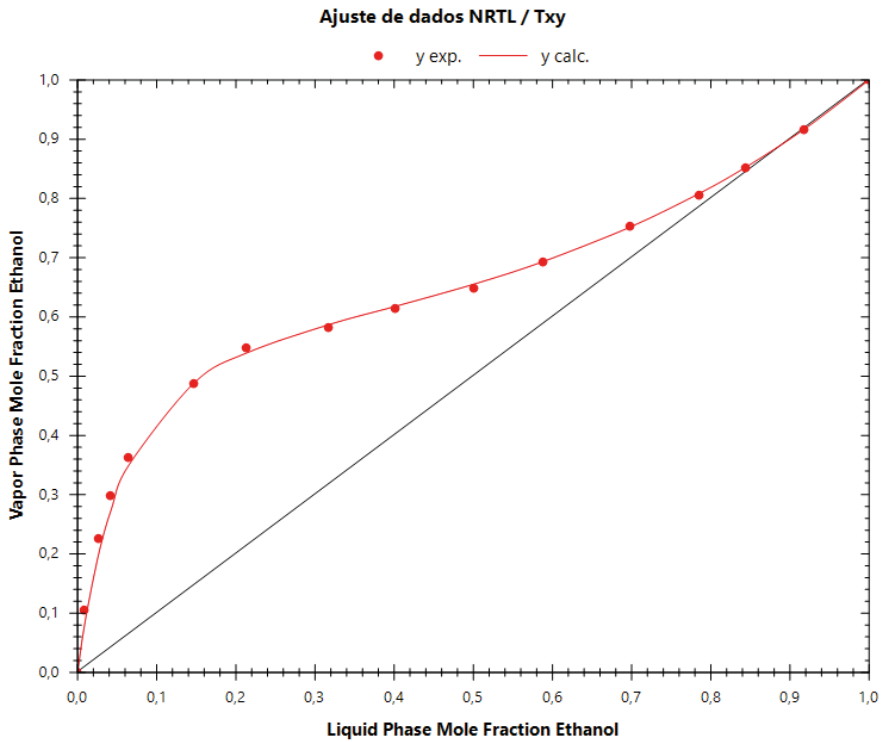


Figure 02. x-y diagram with NRTL data adjustment.
 Source: Own collection (2024).

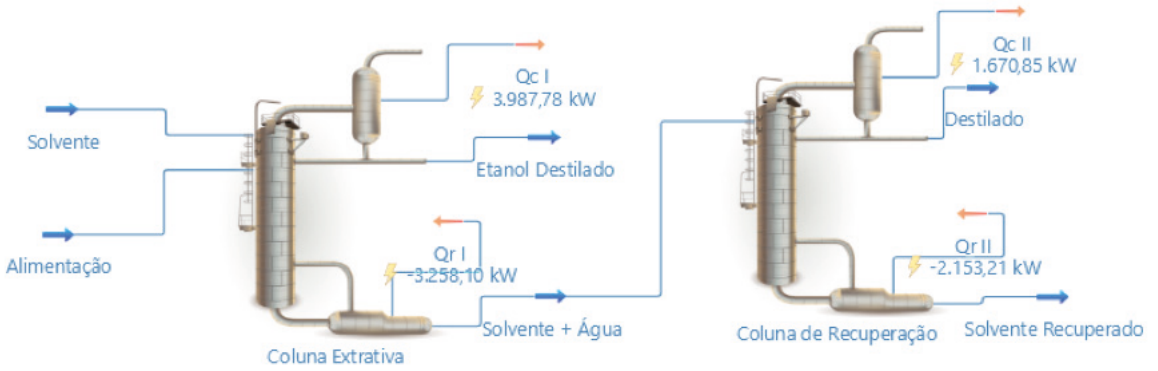


Figure 03. Flowchart of the simulation of ethanol distillation and solvent recovery.

Source: Own collection (2024).

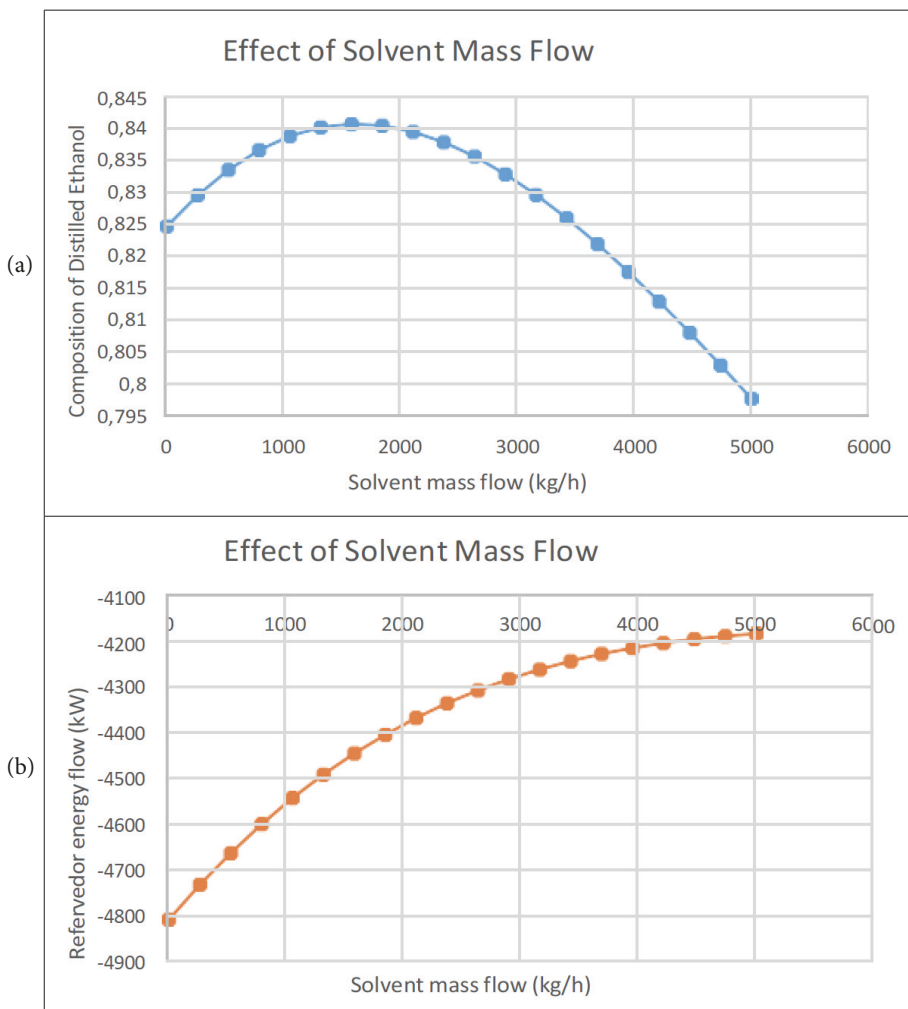


Figure 04. Effect of varying the mass flow of the solvent (a) on the composition of the ethanol in the distillate and (b) in the energy load of the refrervor.

Source: Own collection (2024).

kg/h. There was also a continuous drop in the energy load of the reboiler (Figure 4b), showing that the highest flow rate analyzed, 30,000 kg/h, had the lowest energy load of approximately 3965 kW. In both solvent mass flow rate analyses, there were few variations in the results from 10,000 kg/h onwards.

The next parameter analyzed was the solvent temperature, in which lower temperatures require a greater energy load from the reboiler (Figure 5a), compared to the ethanol mole fraction (Figure 5b), the mole fraction drops to approximately 70%. Therefore, it is recommended not to use temperatures higher than 200°C. Despite the lower heat required, the mole fraction decreases considerably. This is due to the boiling temperature of pure ethylene glycol being 197.4°C at a pressure of 4.5 atm (NIST, 2023).

The pressure of the solvent stream had little influence on the system, as seen in Figure 06, from 1 Bar onwards it behaved approximately constantly.

However, the variation in feed pressure shows how the mole fraction (Figure 7a) increases for pressures greater than 3 bar, but the energy load on the reboiler (Figure 7b) increases considerably, so that pressures above 3 bar are not justifiable.

For the effect of feed temperature, there is a change in temperature behavior between 100 and 150°C. Both the energy load of the reboiler (Figure 8a) and the mole fraction of ethanol (Figure 8b) decrease as the feed temperature increases. As this drop in molar fraction does not vary enough to significantly affect the final product, a temperature of 150°C or less is recommended.

The feed flow rate proved to have little influence on the system, with a linear behavior in relation to the energy load on the reboiler (Figure 9a) from 2,000 kg/h onwards. Therefore, in order to preserve a high level

of production in relation to the composition of ethanol in the distillate (Figure 9b) and the optimum mass flow rate, it is feasible to use a flow rate of 5,000 kg/h, as was done by Purgatto et al. (2016).

The feed mass flow rate used was 5,173 kg/h, while the solvent mass flow rate was 20,000 kg/h, resulting in an amount of solvent 3.86 times greater than the feed. Although a solvent/feed ratio of 80% is commonly adopted, the analysis of the simulation by Purgatto et al. (2016) showed that a greater amount of solvent was used in the operation, possibly due to the effect of implementing the recycle current in the simulation.

Analyzing the effect of the number of stages, we observed that as the number of stages increases, less energy is required in the reboiler (Figure 10a) of the extraction column, as well as an increase in the molar fraction of ethanol (Figure 10b) in the distillate, with little variation after 20 stages.

Checking the results according to the sensitivity analysis carried out shows a promising result, reducing the energy load on the reboiler and increasing the molar and mass quantity of ethanol distilled. The configuration used is shown in Table 01.

Number of internships	20
Solvent inlet position	3
Power input position	11
Solvent temperature (°C)	197
Supply temperature (°C)	150
Solvent mass flow rate (kg/h)	4000
Feed mass flow rate (kg/h)	5000
Solvent pressure (bar)	1,01
Supply pressure (bar)	3
Solvent/feed ratio (S/F)	0,8
Backflow ratio	2,825

Table 01. Input data according to optimizations.

Source: Own collection (2024).

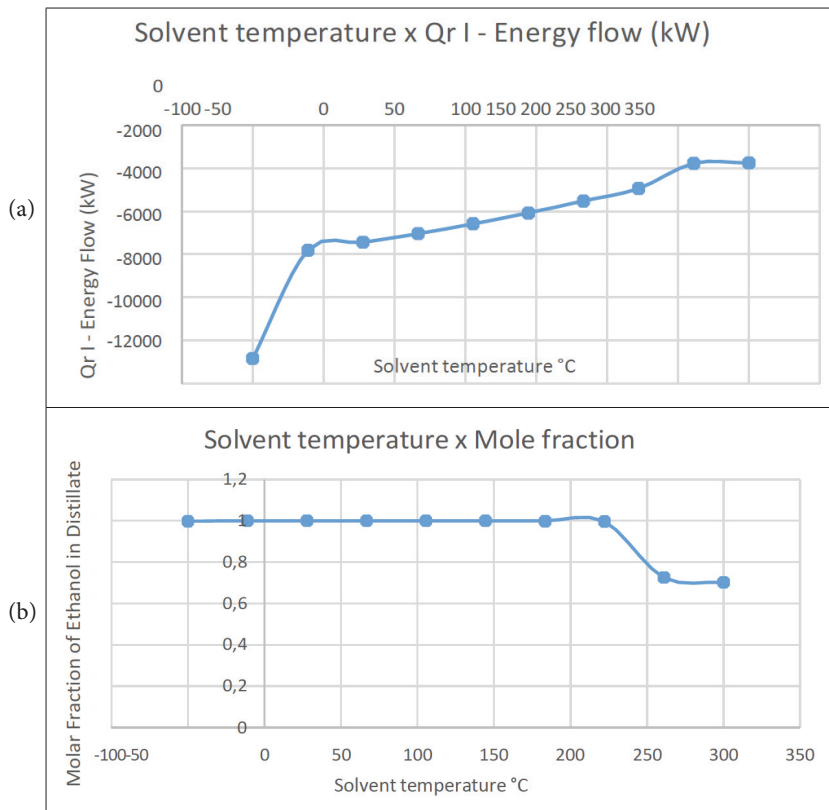


Figure 05. Effect of varying the solvent temperature (a) on the energy load of the reboiler and (b) on the composition of ethanol in the distillate.

Source: Own collection (2024).

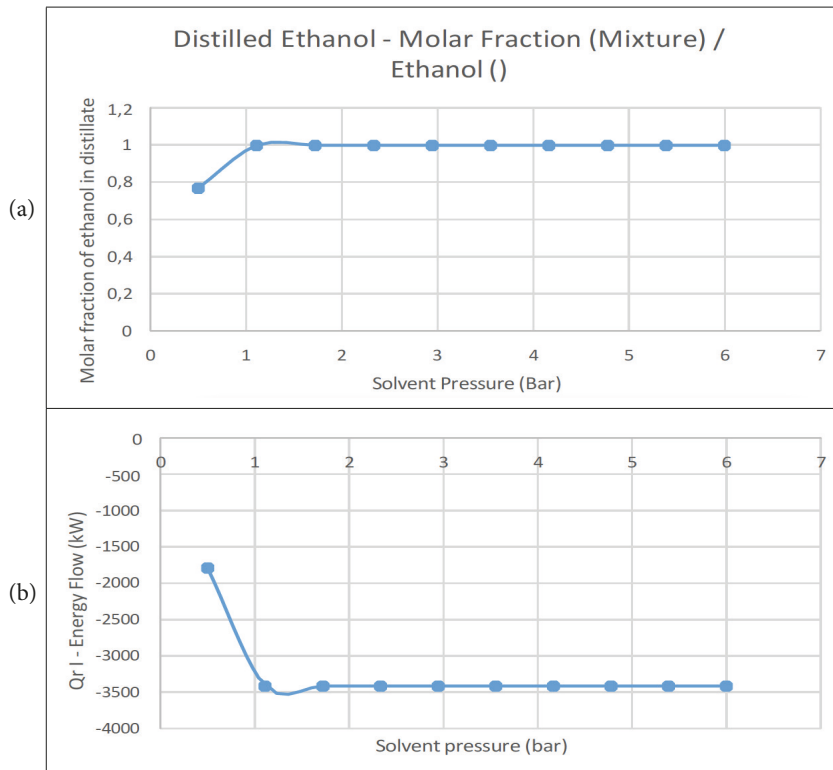


Figure 06. Effect of solvent pressure (a) on the composition of ethanol in the distillate and (b) on the energy load of the reboiler.

Source: Own collection (2024).

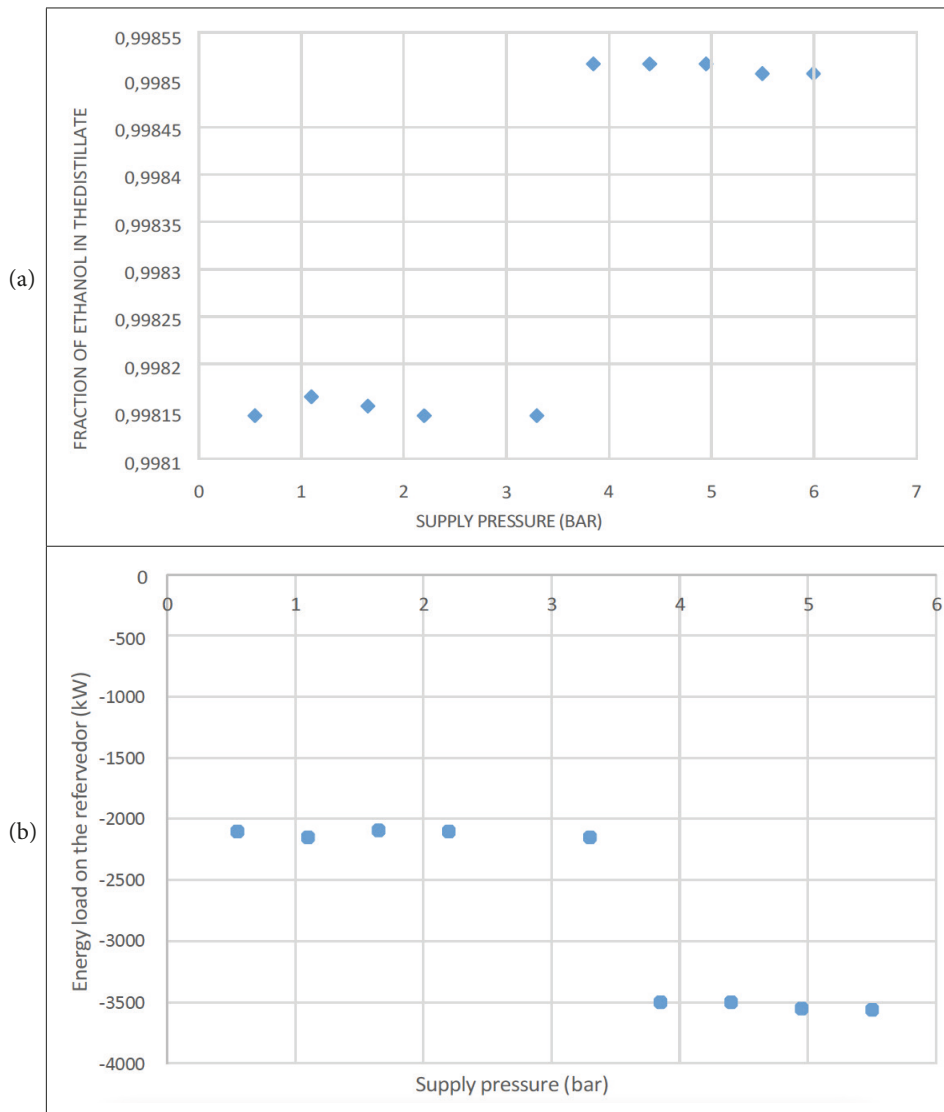
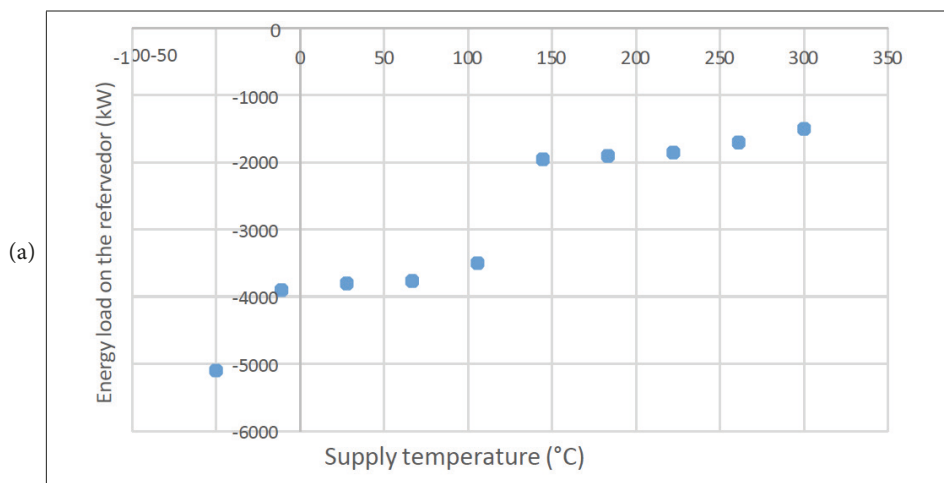


Figure 07. Effect of varying the feed pressure (a) on the composition of ethanol in the distillate and (b) on the energy load of the reboiler.

Source: Own collection (2024).



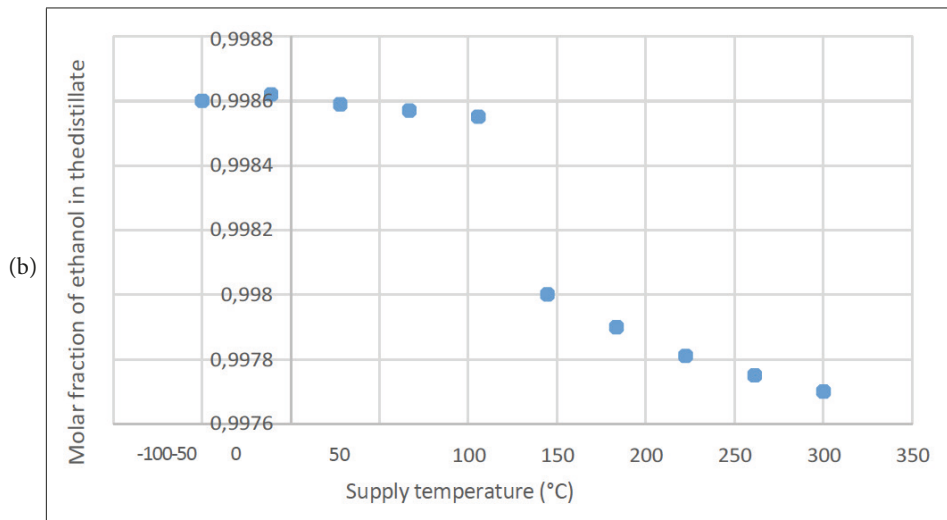


Figure 08. Effect of feed temperature on (a) energy flow and (b) composition of ethanol in the distillate .
Source: Own collection (2024).

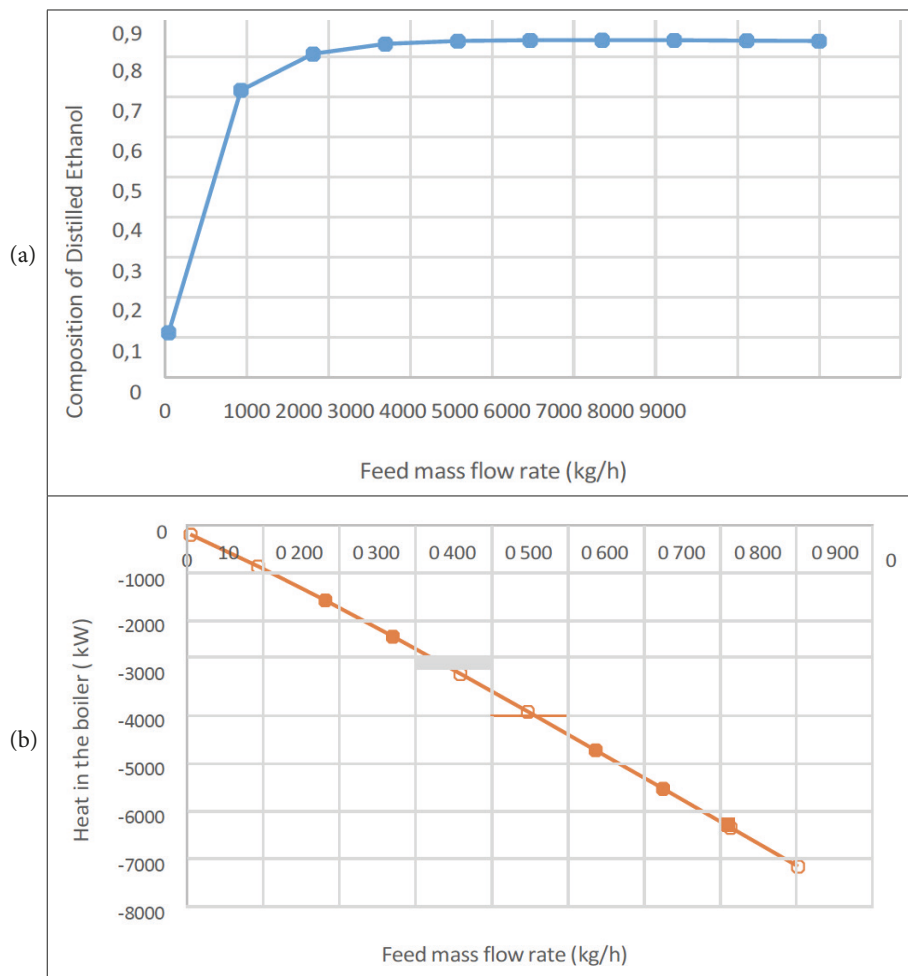


Figure 09. Effect of feed flow rate as a function of (a) refferedor energy load and (b) distillate ethanol composition.
Source: Own collection (2024).

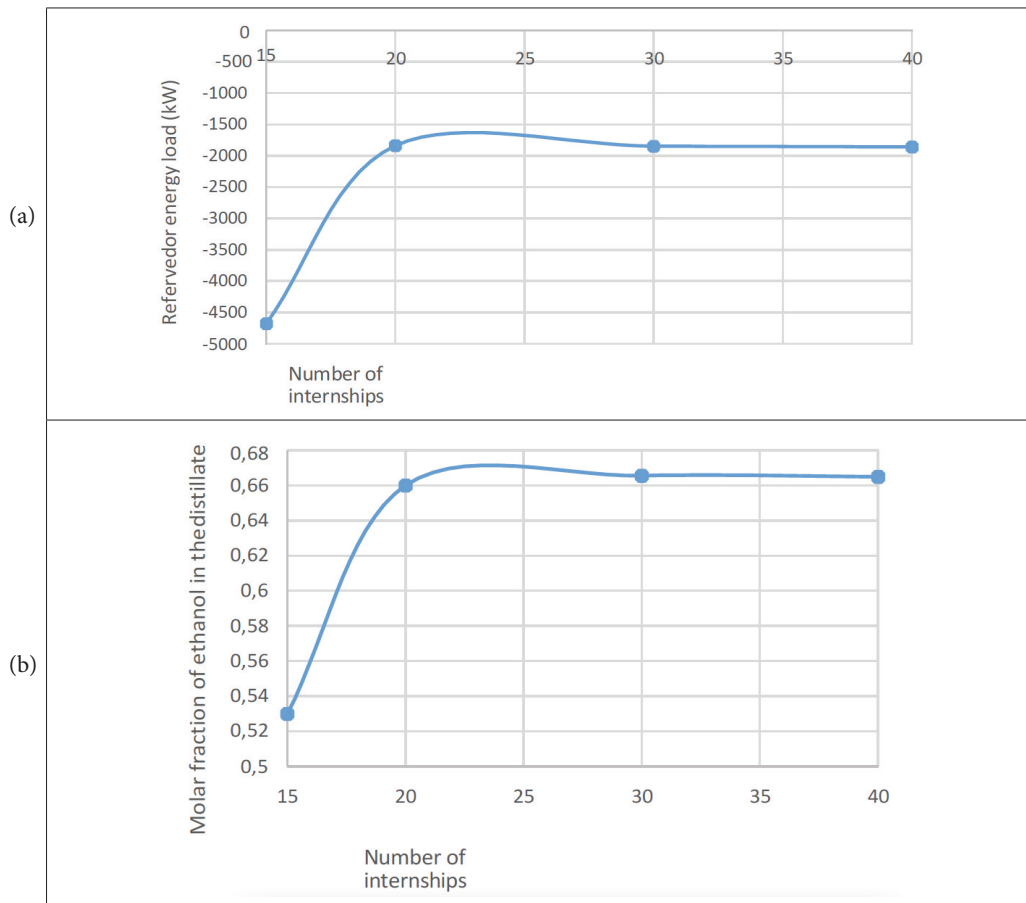


Figure 10. Effect of the number of stages (a) on the energy load (b) on the composition of ethanol in the distillate.
Source: Own collection (2024).

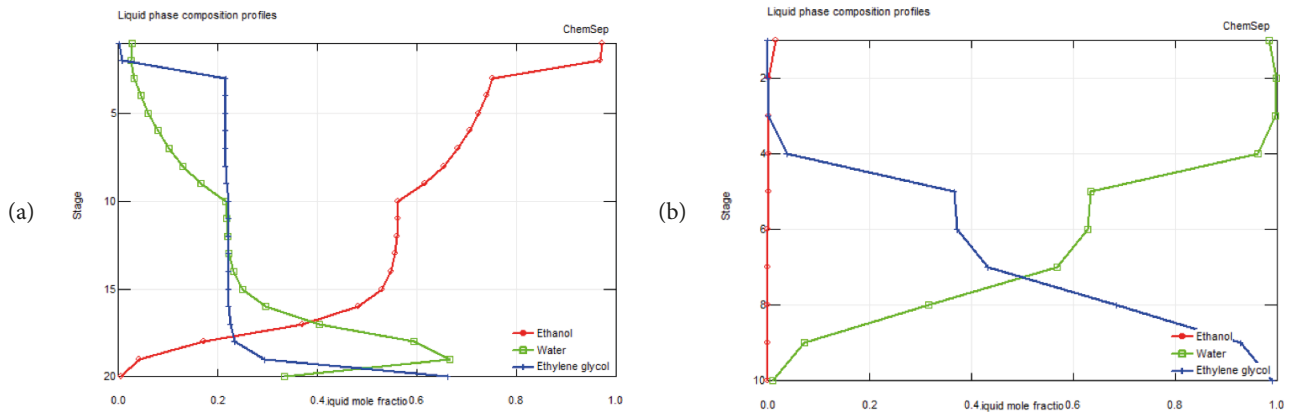


Figure 11. Number of stages as a function of composition for (a) extraction column and (b) regeneration column.
Source: Own collection (2024).

It is possible to observe the behavior of the compositions throughout the stages, disregarding the condenser as a plate. In the extraction column (Figure 11a), the feed enters at plate 10, while the ethylene glycol enters at plate

2. At the top of the column, in plate 1, ethanol is found in its maximum concentration, while in the last stage the maximum concentration of ethylene glycol is found, which drags the water with it. In the regeneration column (Figure 11b), the entry of solvent and water is observed in plate 4, with a clear separation, where water is directed to the top of the column in plate 1, while ethylene glycol is directed to the last plate, where it is at its highest concentration. Ethanol is found in minimal concentrations in the regeneration column.

CONCLUSION

In short, this study focused on identifying the parameters that have the greatest impact on the energy consumption of the process, based on a sensitivity analysis aimed at obtaining optimum values for the input parameters. The objective was to maximize the production of hydrous ethanol while minimizing energy consumption.

It can be concluded that the temperature of the solvent in the input stream and the mass flow rate are very high, values such as 197°C and 4000 kg/h, with a ratio of 80% solvent to feed, showed values that were more favorable to the operation of the process, such as the reduction in the energy flow required in the reboiler and the higher composition of ethanol in the distillate. Another aspect observed was that the solvent stream can operate at atmospheric pressure without harming distillation. In addition, it was found that the use of 20 stages in the column performed better both in terms of energy consumption and the amount of ethanol produced. The

regeneration column performed constantly, recovering 99% ethylene glycol and 98.5% water.

By changing the configuration and input parameters of the extractive distillation, it was possible to achieve satisfactory results, with an alcohol content of 98.86% ethanol by mass and an energy consumption in the reboiler of -2237.16 kW.

The temperature of the solvent and of the azeotropic feed had the effect of reducing the amount of energy that the reboiler will supply to the system, so if the inlet temperatures are not above boiling temperatures, they will not cause damage to the composition of the ethanol present at the top of the extraction column. However, if the inlet temperatures are significantly higher than the boiling temperatures, they will be vaporized, because the excessive increase in temperature leads to an increase in the volatility of the components, compromising the thermal exchanges that are essential for the efficient separation of the components.

The supply pressure has resulted in an increase in the energy load required by the reboiler. Above 3 bar, it requires twice as much energy compared to lower pressures. This increase is due to the fact that higher pressure raises the boiling point of the components, requiring more energy to vaporize them.

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