Energy Optimization of Reactive Distillation Columns for Biodiesel by Pinch Point Analysis

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Abstract: This papers aims at optimizing the energy consumption of reactive distillation columns for simulation of biodiesel production. The design of the proposed biodiesel production uses lauric acid ($C_{12}H_{24}O_2$) as feedstock in a reactive distillation column. In order to do so, selection has been done by selecting the design parameters of the column: the number of stages, feeding flows, reflux ratio, the height of the transfer unit as well as the analysis of the Sulzer BX packing. ASPEN PLUS^{TM} simulation software has been used to work out the thermodynamic steady state of the system on which the optimization is based on. Energy optimization is carried out through the Pinch Point Analysis. Thus it has been proven that the Pinch Point Analysis allows optimization of the heat exchange network in a distillation column. Results showed that it is possible to reduce drastically the energy requirements of a distillation process up to 60 %. The heat exchange network showed that is promising to design more energy-efficient and environment-friendly distillation process for production of biodiesel.

Keywords: Biodiesel, reactive distillation, method Point Pinch Analysis.

1. INTRODUCTION

In recent years, global existing energy demand and increasing environmental concerns are motivating researchers to engender extensive research on more efficient and environmentally friendly fuels, i.e. biofuels: biodiesel, bioethanol and biomass, among others. In particular, biodiesel is considered as the most viable and popular alternative to fossil-derived diesel [1-4]. Since it is a fuel that presents major advantages over conventional diesel: it does not harm the environment as long as it is yielded from organic material such as vegetable or fats; it is non-toxic and biodegradable; it contains no sulphur and it is better lubricant [4].

Most of studies for new biodiesel production processes are aimed at reducing the use of oil reserves, carbon dioxide emissions and use of alternative energy sources [5-8]. Furthermore, important process intensification policies are taking place in order to replace fossil-derived diesel with biodiesel, for example, the EU´s objective is to replace 10% of fuel consumption with alternative fuels by the year 2020 [9].

In Mexico, for example, a feasibility study on the use of bioethanol and biodiesel as an alternative to transportation fuel took place in 2006. The study estimated a saving of 7.5 million tons of CO_2 released into the environment per year as long as a production of 100,000 ton/year is maintained until 2014, which represents a substitution of 2 to 5 per cent [10]. Nowadays the Mexican annual production of biodiesel reaches roughly 3.7 million of liters [11].

Although many studies for exploring more energy-efficient and environmentally friendly processes and technologies for yielding biodiesel are ongoing, particular attention has to be paid to the separation processes. In particular, separation processes such as distillation are some of the most energy-intensive processes that accounts for almost 3% of the energy world consumption [12-14]. In fact, energy consumption in a chemical power plant accounts for 40% of total consumption [15]. Hence global concerns on distillation processes are taking place in order to reduce both energy consumption and carbon dioxide emissions [16-19]. Several studies have focused on the use of heat integration approaches in the design of separation process, which permits to lay emphasis on the energy requirements and determine the optimal point for operating the reactive distillation based biodiesel process [20-24].

In accordance to all the aforementioned issues, this paper describes the energy optimization of a reactive distillation column for simulation of biodiesel production. In particular, a reactive distillation column is used here because it combines both distillation and chemical reaction stages into a single process unit, besides it reduces the amount of alcohol in the feed stream causing its stoichiometric ratio come close with oil. The effect is that less energy is required for the

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alcohol recovery process [1,15]. In this study, ASPEN PLUS[TM] by AspenTech has been used as the simulation software. It permits to find optimum conditions for the design and operation of the RDC in terms of molar ratio of alcohol to oil, re-boiler temperature, residence time, alcohol to feed location and reflux ratio. The RADFRAC model was used since it is a model that relies on the assumption that the vapor from the stage below and liquid from the stage above are brought into intimate and well-mixed contact in the stage [25]. Whereas *Pinch Point Analysis* was used as optimization technique.

2. METHODOLOGY

2.1. Chemicals and Reagent

The process consists of a production of 2,500 liters per day of biodiesel from lauric acid ($C_{12}H_{24}O_2$) as feedstock reacting with methanol and 2-ethylhexanol (2EH, henceforth).

Lauric acid is a medium-length long-chain fatty acid and it is found in most of the Mexican land as fat and oil derived from plants (e.g. coconut) and animals (e.g. pigs), besides the version of ASPEN PLUS[TM] software used in this study had all of its physical properties available. The software provides methanol (MEOH) and 2-ethylhexanol (2-EH) properties as well.

2.2. Equipment Description

The continuous reactive distillation system is shown in Figure **1**. The Reactive Distillation (RD) column is the main unit of the process where the reaction and separation of unreacted methanol and 2-ethylhexanol takes place (A1). For the simulation, the RD column is packed with Sulzer BX packing. So the main design parameters of the column are the number of stages, feeding flows, reflux ratio, the height of the transfer unit as well as the analysis of the Sulzer BX packing. Three heat exchangers (C1, C2 and C3) are considered in the process in order to recover heat from the product (biodiesel). Lauric acid, 2-ethylhexanol and methanol are fed from the tanks T1, T2 and T3, respectively.

2.3. PROCESS DESCRIPTION

According to Figure **1**, three feed streams, one of lauric acid ($C_{12}H_{24}O_2$), other of 2-ethylhexanol (2EH) and other of methanol at room temperature and atmospheric pressure are heated before being fed to the RD column. The lauric acid is heated to 130°C, the methanol and the 2-ethylhexanol streams are heated to 120°C according to operating conditions assumed by Miranda *et al.* [26]. The methanol and lauric acid are fed in a molar ratio of 1:1 into the RD column. The process feedstock is composed of 252.4 kg/h of lauric acid, 26.05 kg/h of 2-ethylhexanol and 40.37 kg/h of methanol at 1.5 bar. The RD column is fitted with 22 stages, 20 are reactive stages using the Sulzer-BX packing as liquid-gas contactor and the remaining stages are the condenser and the re-boiler, and works at atmospheric pressure.

The lauric acid enters the column at the 3rd stage, whereas the methanol enters at the 15th stage. The 2-
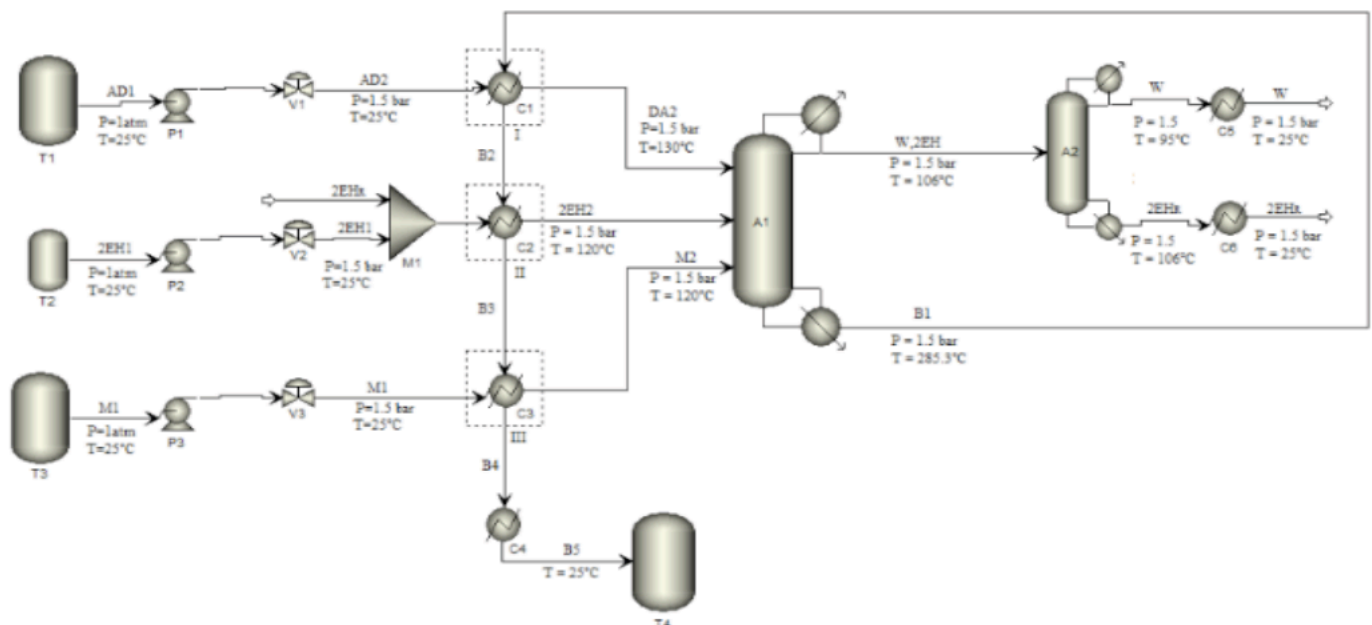

Figure 1: Conventional Biodiesel production process with reactive distillation column.

ethylexanol enters the column at the 10th stage in order to complete the reaction and to obtain a more pure product. In the RD column the esterification takes place to form methyl dodecanoate (biodiesel) and a mixture of 2-ethylhexanol and water (by-product). The water-2EH mixture is then fed to a second column (A2) where the 2-EH is separated and recycled to the process. The final biodiesel at (285°C and 1.5 bar), is then fed back to the heat exchanger network until it is brought to 25°C and 1 bar in a tank. Three heat exchanger zones are proposed in this study:

- Zone I: Heat exchange between the biodiesel stream (B1) leaving the RD column and the lauric acid stream (AD2).

- Zone II: Heat exchange between the B2 stream and the mixing of makeup and recycled 2-EH stream (2EH2).

- Zone III: Final heat exchange between the B3 stream and the makeup methanol (M1).

The RD column is simulated using the RADFRAC module of Aspen Plus, and the rigorous continuous stirred tank reactor (CSTR) model available in Aspen Pus is used for the conventional process. The simulation consisted in defining chemical components, thermodynamic models, feed stream conditions such as flows, temperature and pressure, column diameter as well as the height of packing equivalent to one theoretical plate (HETP). The model assumes finite steps of concentration change rather than the continuous change actually occurs in a packed column. In prior study, it is indicated that the actual error involved in using this method is small. The model includes equations for the HEPT in order to calculate the required height in the package to reach the equilibrium in a hypothetical stage [27]:

$$EHTP = P\sqrt{\rho_l} / (2712 + 82.0P) \left(1 + 1.505 \left(\frac{\rho V}{\rho l} \right)^{0.25} \right)^2 * F^{0.42}$$

(1)

Where P is the operating pressure in mmHg, F is the vapour load factor in m/s(kg/m^3)$^{0.5}$, $\rho_{l,v}$ is the liquid and vapor density in kg/m^3. Further discussion of Eq. (1) is available in [27]. Because of the non-ideal behaviour of reaction mixture, the UNIQUAC model is employed to calculate the relevant phase equilibrium properties. The esterification reaction is carried out at a 1:1 alcohol-to-oil molar ratio and a 16% excess of 2-ethylhexanol to complete the reaction. In the RD column, the 2-ethylhexanol has an important role, methanol alone would form an azeotrope with water not removed in the reaction, which would impede the separation by simple distillation. Thus, 2-EH permits to separate the water from the distillation product and avoids degradation of biodiesel purity [28]. A water-2EH stream of 1.46 kmol/h is introduced into the rectification column at a water molar fraction of 0.86 and 100°C, and a 2-EH molar fraction of 0.14 at 195°C.

2.4. Model and Simulation Performance

As a result of the process simulation and models used in this study, Table **1** provides details of the key process streams for the process shown in Figure **1**. Of particular interest is the purity and yield of the biodiesel which are 99.99 % and 270 kg/h, respectively. This is due largely to the use of 2-ethylhexanol as azeotrope inhibitor agent in the rectification column.

The liquid mass fraction profiles of the biodiesel production process by using reactive distillation column (RDC) are plotted in Figure **2**. Almost a complete conversion of lauric acid to biodiesel is obtained with ten reaction stages as shown in the profiles. In fact, a high purity of biodiesel can be obtained from stage 17.

Table 1: Results of the Biodiesel Process Simulation

Parameter	Lauric Acid	Methanol	2Ethylhexanol	Biodiesel	Water
Molar flow rate, [kmol/h]	1.26	1.26	0.2	1.26	1.46
Mass flow [rate, kg/h]	252.4	40.37	26.05	270.08	48.75
Volume flow rate, [l/min]	5.22	457.63	0.59	6.05	0.99
Temperatures, [K]	403.15	393.15	393.15	558.42	379.05
Enthalpy, [kJ/kmol]	39.5	11.22	22.85	31.45	16.91
Molecular weight, [kg/kmol]	200.32	32.04	130.23	214.35	33.39
Specific heat, [kJ/kg-K]	2.56	1.60	3.01	4.14	4.55

The high yield and purity of the biodiesel is due largely to the consumption in excess of the 2-ethylhexanol from stage 12. On the contrary, methanol to lauric acid feed molar ratio is high from stage 2 to stage 7, then it decays because of the yield of biodiesel.
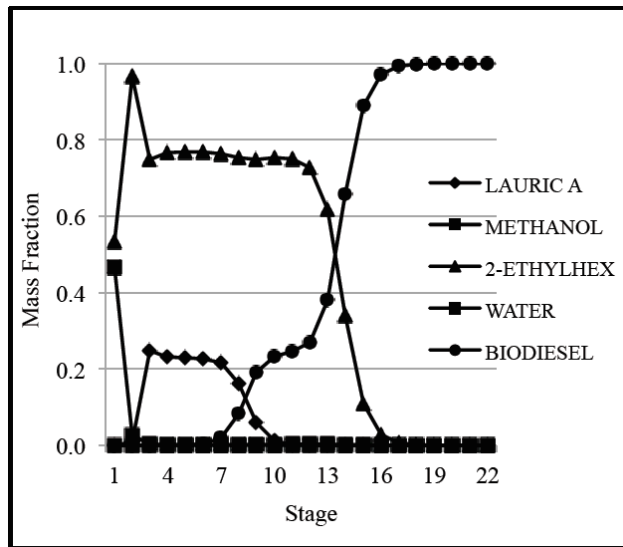

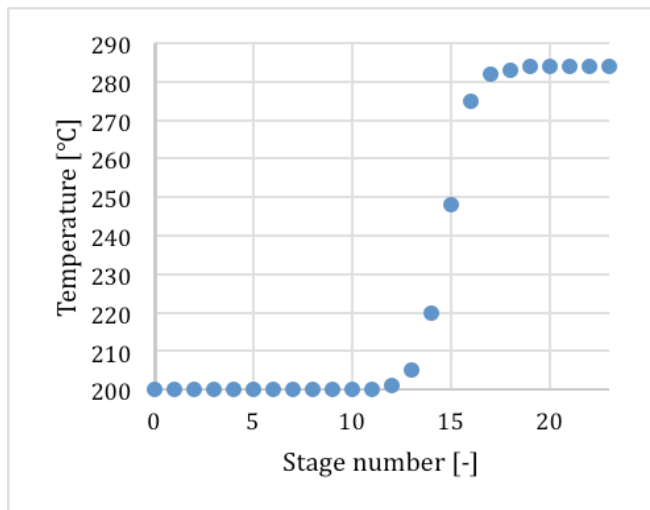**Figure 2:** Liquid composition profiles within the reactive distillation column.

Figure 3: Temperature profile within the reactive distillation column.

Figure **3** plots the temperature profile in the reactive distillation column proposed herein. The RDC is operated at temperatures ranging from 200°C to 280 °C, what suggests the need for taking advantage of the energy that can be used downstream for optimizing the process. The high temperature shown in stage 22 is due mainly to the re-boiler installed at the end of the process. Figure **4** shows the energy consumption in the re-boiler as a function of the molar reflux ratio. From studying this effect, the minimum condition for the re-

boiler to operate is that the molar flux ratio is 3 with a re-boiler energy consumption of 92 kW in average.
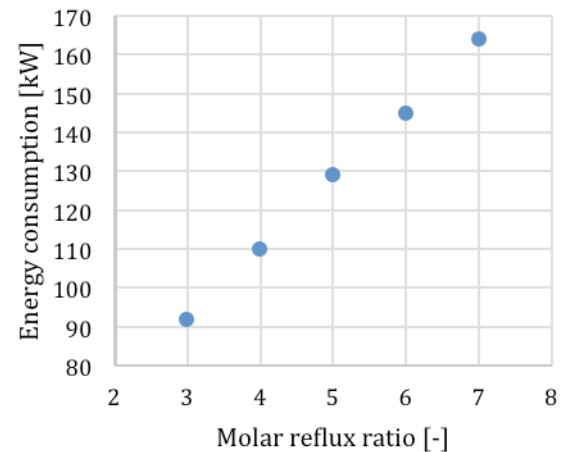

Figure 4: Energy requirement in the re-boiler at different molar reflux ratios.

Table 2:   Final Parameters for the Reactive Distillation Column

Parameter	Value
Stages number	22
Reactive stages	20
Molar reflux ratio	3
Lauric acid feeding stage	3
2-Ethyhexanol feeding stage	10
Methanol feeding stage	15
Biodiesel purity, [% wt]	99.99
Reboiler energy consumption, [kW]	92
Condenser heat losses, [kW]	72
HETP, [m]	0.23
Column diameter, [m]	0.33

3. ENERGY OPTIMIZATION OF THE RDC

3.1. Energy Balance

Distillation columns are regarded as the mainstay of chemical process industry (they are used for about 95% of all fluid separation in the chemical industry [13, 14]) and are quite energy intensive because of their low thermal efficiency which is around 5-20% [29, 30]. As a rule, the idea behind the energy optimization of distillation columns is to improve its global thermal efficiency. To put it another way, the idea is to reduce the external energy inputs by effectively utilizing the heat energy from distillation units and to distribute it

more uniformly along the length of the column [12]. In that case, a thermal analysis is required since it identifies energy targets within the reactive distillation column, that is to say condensers and re-boilers heat loads, as well as technical proposals to redesign the column to make it more efficient.

Considering all the biodiesel production process, the simulation results provide the heat duty of each one of the equipments (Table **3**).

Table 3:   Heat Duty of Biodiesel Production Equipments

Equipment	Energy consumption, [kW]
Reactive distillation column	40.57
Rectification column	16.12
Heat exchanger C1	23.27
Heat exchanger C2	2.15
Heat exchanger C3	2.62
Heat exchanger C4	-2.15
Heat exchanger C5	-1.53
Heat exchanger C6	-41.1
Pump P1	0.25
Pump P2	0.25
Pump P3	0.25
Total Balance	**40.7**

As expected, the most energy-consuming equipment turned out to be the reactive distillation column, 40.6 kW. This was due to the high purity of the biodiesel. Heat exchangers C4, C5 and C6 show negative values because they are equipments used to cool down the fluid going through them, leading to a reduction of the global energy consumption of the process.

3.2. Heat Integration

The conventional process of the biodiesel, as the one studied herein, shows a heat duty of 40.7 kW, approximately. Hence the need for applying a technical methodology to reduce its energy consumption. In this context, the *Pinch Point Analysis* is an engineering method usually used for the optimization of energy recovery systems. In order to simplify the process, the reactive distillation column where the methodology is applied is shown in Figure **5**.

For the most energy recovery in the reactive distillation column, two hot streams are to be cooled

down to ambient temperature: one is the biodiesel and another is the water-2-EH mixture. While three streams are to be heated: one is the lauric acid feedstock (DODE-AC), the other is the 2-ethylhexanol stream (2-ETHEX) and the third is the methanol feed stream. Table **4** summarizes the technical temperature
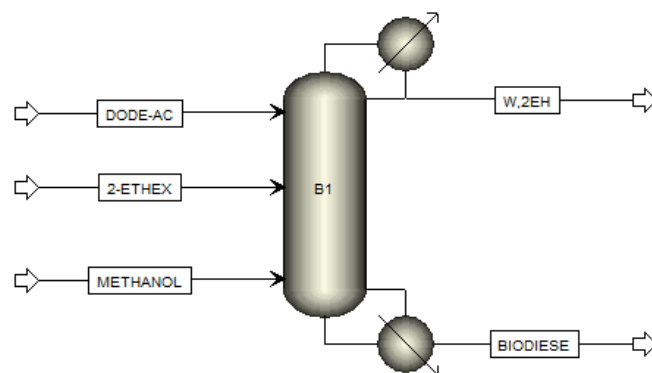

Figure 5: Schematic process diagram for application of the pinch point analysis.

changes permitted in each stream, their heat capacity rate and enthalpy change rate.

Table 4: Temperature Interval for the Process Stream

Stream description	Temperature interval, [K]		Heat capacity rate, [kJ/(s° C)]	Enthalpy change rate, [kJ/s]
	In	Out		
Lauric acid (DODE-AC)	298.15	403.15	0.18	18.9
Ethylhexanol (2-ETHEX)	298.15	393.15	0.018	1.71
Methanol	298.15	393.15	0.022	2.09
Water-ethylhexanol (W, 2EH)	379.05	298.15	0.49	-39.86
Biodiesel	558.50	298.15	0.229	-59.62

The sets of temperature versus enthalpy rate values that are established for the streams that are to be cooled and those that are to be heated are plotted in Figure **6**. This is its composite diagram for the heat integration of the biodiesel process.

4. RESULTS

Table **5** shows the design parameters utilized in the reactive distillation column as a result of the simulation of the biodiesel production process. Here it is observed a mass fraction of 0.999 for the purity of the biodiesel

stream. Such purity can also be detected in the composition profile shown in Figure **3**, in which it can be seen that biodiesel is highly pure and free of impurities as product of the column bottom.

Table 5: Design Parameters of Distillation Column

Parameter	Value
Stages	22
Reactive stage	20
Column pressure, [bar]	1.5
Reflux ratio	3
Acid feed stage	3
2-Ethylhaxanol feed stage	10
Methanol feed stage	15
Biodiesel purity, [wt %]	99.99
Reboiler heat consumption, [kW]	110
Condenser heat loss, [kW]	90
HETP, [m]	0.11
Diameter, [m]	0.28

Besides, as product of the distillate process, a mixture of water and excess of 2-ethylhexanol was obtained, which is recuperated in the second column for recirculation. The majority of the reagents consumption occurs in between trays 3 and 10.
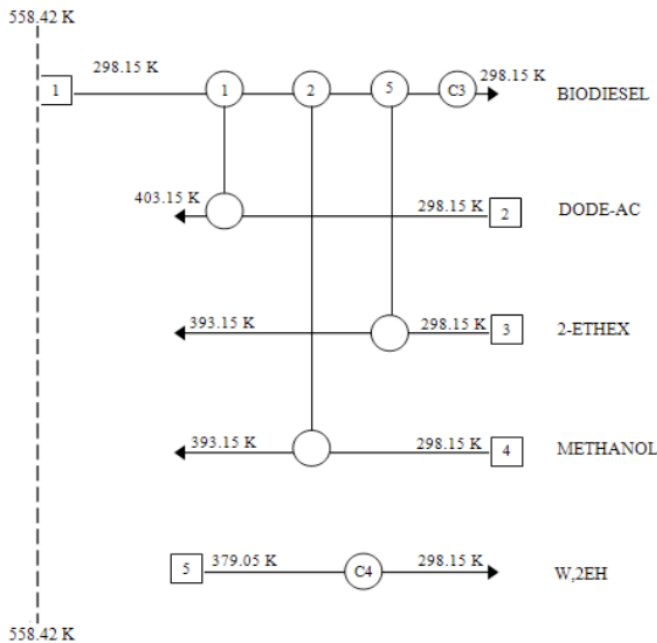

Figure 6: A schematic of the heat exchange network.

From the energy balance, it was possible to find that the total energy consumption (Q_T) utilized by the distillation column and the energy required for increasing inlet streams temperatures accounted for 67 kW. So, as mentioned previously, the idea was to reduce the energy requirements, which was accomplished as explained below.

By application of the Pinch Point Analysis, it was possible to design a new heat exchanger network, refer to Figure **4**. It is clear to see that heat exchangers 1, 2 and 5 are able to exchange heat from the biodiesel stream to the feed streams. Exchangers 3 and 4 represent the cooling services needed for reducing the temperature of the hot streams down to the ambient temperature. Likewise, it is noted that due to the thermodynamic properties of the biodiesel output stream, a temperature of 558.5 K and a WCp of 0.229 kW/K, it is possible to give off energy to the cold streams, thereby eliminating the heating services of the column input streams. As a result the overall energy consumption was reduced to $Q_T`= 41$ kW, which represents a 60 % of energy savings in the reactive distillation column.

5. CONCLUSIONS

This work presented the analysis of a reactive distillation process to obtain biodiesel through esterification of free fatty acids from used frying oil. It has been found that the 2-Ethylhexanol is an excellent water separator agent achieving a biodiesel purity of 99.99 % wt. Furthermore; it has been proven that the Pinch Point Analysis allows optimization of the heat exchange network in a distillation column. Results showed that it is possible to reduce drastically the energy requirements of a distillation process up to 60 %.

Another important conclusion is that the specific heat capacity, C_P, of the heat exchange fluid influence directly on the overall efficiency of the system, since a higher specific heat capacity of the fluid a lower number of trays in the RDC and hence a reduction of duty in the re-boiler and condenser.

The heat exchange network showed that is promising to design more energy-efficient and environment-friendly distillation process for production of biodiesel. Finally, it remains only to validate these results by experimental results which are underway.

GLOSSARY

Symbol	Description
F	Feed flow [kmol/hr]
D	Distllate flow [kmol/hr]

B	Bottoms flow [kmol/hr]
H_i	Enthalpy of each stream [kW/kmol]
Q_r	Re-boiler heat [kW]
Q_c	Loss heat in the condenser [kW]
HETP	Height equivalent to a theoretical plate [m]
Q_T	Total energy [kW]
Q_T	Total energy whit pinch method [kW]

ACKNOWLEDGMENTS

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