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New Configuration of a Distillation Process with Reduced Dimensions

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New proposals for distillation such as HIDiC, diabatic and others have been presented and show that the energetic optimization inside a column can reduce equipment dimensions. Based on these, it is proposed a new configuration of a falling film distillation unit operating at atmospheric pressure, where the energy supply is done by a thermosyphon. Tests with ethanol/water have been performed in a pilot-plant conventional column and in the film unit proposed. Despite the new proposal presents a lower purification than the conventional, it indicates the process feasibility once the new heat supply allows the ethanol purification. Now, it is necessary to improve operational conditions to achieve better separation. The main advantage is the reduction of the unit dimensions then this can be a promising result for the petroleum industry once it enables performing distillation in environments with little space.

1. Introduction

Traditionally distillation processes have been intensified by external heat integration. Based on this, alternative technologies of distillation have been studied and in several applications the physical size of a distillation or recuperation unit can be problematic and can even restrain its use, as in offshore oil extraction. The geometry of a distillation unit imposes heights that can be unfeasible depending on local installation. In this sense, some alternatives have been proposed aiming to modify the conventional structure of a distillation column with the objective of improving the energetic efficiency and also reducing the unit dimensions.
For example, Arrison (2000) published a patent on horizontal distillation, whose system includes a series of tanks interconnected by condensation tubes. The main goal of the authors was to decrease the height of the distillation columns once that in the horizontal distillation the trays are replaced by the tanks.
The heat integration concept was first introduced almost 70 years ago. So far, various schemes have been proposed as described in a review published by Jana (2010. Several

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methods, such as intercoolers–interheaters, heat pumps, secondary reflux and vaporization, and multiple-effect columns have been explored. Basically, the idea is to reduce the external energy inputs by effectively using the heat energy from the distillation units and to distribute the heat more uniformly along the length of the columns. Among those important techniques is the Petluk configuration, Dividing-Wall Columns (DWC), Multi-Effect Columns, Heat pump-assisted unities, diabatic distillation and Heat Integrated Distillation Column (HIDiC).

The Dividing-Wall distillation Column includes energy savings and miniaturization. According to Barroso-Muñoz et al. (2010) the DWC configuration can reduce energy consumption by 30–50% over conventional distillation sequences for the separation of some mixtures. Furthermore, this reduction in energy consumption also results in lower column diameter (miniaturization due to reduction in internal flows).

Another configuration that was extensively studied consists of diabatic distillation columns (Johannessen and Røsjorde, 2007). The objective of these units is to obtain energy savings in the process through successive heat introductions and removals in the column. This situation can be improved by spreading the heat requirements over the whole length of a distillation column.

In addition, related to the energetic optimization, many studies for fractioning columns concentrate on the HIDiC proposal. Iwakabe et al. (2006) show savings of 30% for Benzene-Toluene-Xilene distillation and 50% for separation of a mixture of 12 hydrocarbons, when compared to the same separation in a conventional column. Other researches can be cited in this area (Suphanit, 2010 and Huang et al., 2007) and all of them associate the energy saving to the modification in the conventional structure of a fractioning unit.

A very interesting work is the one proposed by Saifutdinov et al. (1999), who patented a compact separation unit that uses a thermal fluid to perform heat transference. As a way of increasing the efficiency of the mass and heat transference, transference tubes in the rectifying section part and/or in the evaporator part have means to provide irregular (variable) amounts of heat between the interior and the exterior of the tube along its height. This reduces the dimensions of a standard unit around 3 to 10 times.

As cited before, all these configurations have optimized energy requirements distributing the heat along the length of the columns and sometimes getting miniaturization with this. Aiming this, the objective of this work is to evaluate a falling film distillation unit with a new energy supply by thermosyphon (vapor chamber with non-condensable gas) in order to increase the efficiency of the heat and mass transfer, which have means to provide irregular amounts (temperature profile) of heat between the interior and the exterior of the tube along its height. Experiments with the water and ethanol mixture have been performed in order to verify the feasibility of the proposal and those have been compared to the pilot conventional column. This comparison was done evaluating which tower tray of separation in the new proposal corresponds to the conventional.

2. Proposal Description

The unit developed is composed by a vertical tube where the distillation by film occurs, assisted by thermosyphon (vapor chamber with non-condensable gas). The heat transferences are carried out on the internal and external surfaces of the distillation tube and thermosyphon tube which work as heat exchangers. The vapor condensation of the mixtures occurs on the external surface, while the heat extraction of the external surface of the exchanger is guaranteed by the operation of the heat tube, more specifically of the type of vapor chamber.

The distillation process of a binary mixture occurs through a falling liquid film (ethanol/water - with 10% of ethanol in volumetric basis) which flows on the internal surface of a vertical tube (mass and heat transference tubes where the distillation occurs). The liquid film flows in countercurrent with the vapor generated while the wall of the tube is maintained at a constant temperature radially (through the vapor chamber) and with a particular profile along the height.

The distillation tube is built in glass with the 3 mm of thickness and 26 mm of internal diameter according to Saifutdinov et al. (1999). The initial calculation of the exchange area was based in classical equations and corresponds to the total area of the bubbling that exists in a conventional column. Based on this value, the design parameters were estimated for the mass and heat transference tubes, i.e., distillation tubes. Then, different combinations between heights and diameters of the tubes were analyzed based on the exchange area calculated and for the 26 mm of diameter, the final height was determined in 1 m. This value was also defined aiming miniaturization.

3. Experimental Results

Tests were carried out with a mixture of 10% in volumetric basis of ethanol and 90% water (volumetric basis). The same mixture was used in experiments in a conventional pilot unit whose responses provided a basis for comparison of the film unit efficiency. The operation conditions of the film distillation are presented in Table 1. The distilled product was analyzed by gas chromatography aiming to analyze the separation efficiency. Methyl ethyl ketone was used as a solvent, in the proportion of 20 μL collected sample + 980 μL methyl ethyl ketone.

Experiments were conducted for approximately 4 hours after steady state and the results refer to this period. In Figure 1(a) is presented the temperature profile in the bottom of the tube of the distillation column and in Figure 1(b), the temperature profile obtained in the vapor chamber (heating source). In this chamber there are 11 temperature sensors (1 - inferior and 11 - superior) to indicate the chamber temperature through the tube length. By analyzing Figure 1(a), we can observe that with the vapor chamber conditions presented the maximum bottom temperature reached was approximately 95^{o}C. The composition results showed a recuperation of the ethanol fraction of approximately 1.0 L.h^{-1} with average mass fraction of 60%, according to Figure 2.

Table 1: Film unit operation conditions used in tests with ethanol-water.

Variable	Value
Temperature in the bottom of the chamber	123°C
Temperature on the top of the chamber	98°C
Temperature of the distillation feeding	78°C
Feeding flow rate	17 L.h^{-1}

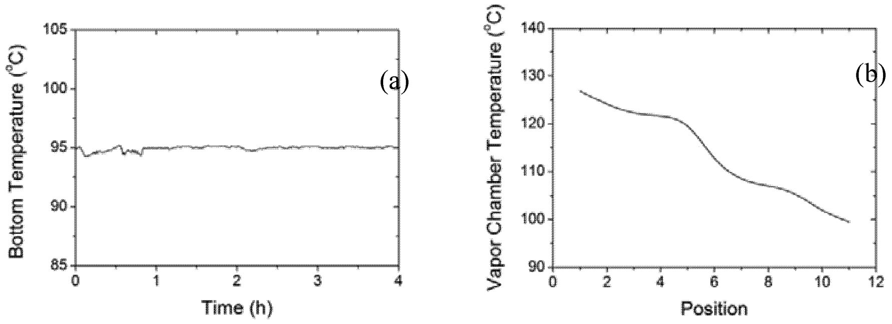

Figure 1: Temperature (a) distillation bottom and (b) profile in the vapor chamber.

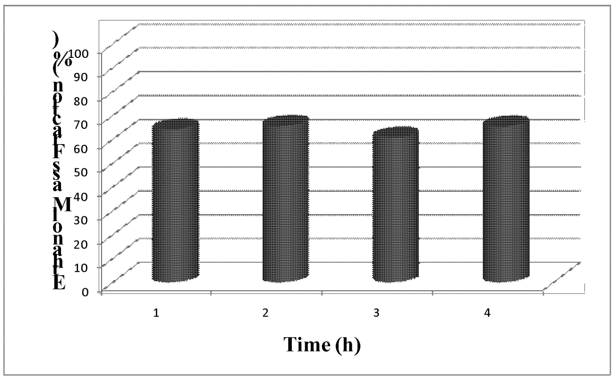

Figure 2: Result os mass fraction of ethanol obtained with the falling film distillation.

In those experiments was observed that the vapor velocity (estimated by computational fluid dynamic simulation) inside the distillation tube was around 2.0 m/s – value greater than desired for a suitable separation. According to Saifutdinov et al (1999) the value of vapor velocity must be maintained around 0.027 m/s – at high velocity of vapor along vertical surfaces a film ceases to be uniform and heat and mass transfer processes become unstable.The result is reflected in compositions (ethanol fraction) lower than expected (around 90%). The low residence time and the high velocity of the flow make an increasing in the film unit´s separation ability impossible.

Another variable that have direct influence on the separation ability is the operation pressure and its variation (ΔP). The testes performed so far have been conducted at atmospheric pressure (different of the most falling film distillations), so that the vapor generated can have a short mean free path, and it condenses inside the tube. We are interested in verifying the effect of an optimized energy supply in a distillation unit and compare to a conventional one, so the atmospheric pressure is essential.

At last, these results were compared with the obtained in the pilot experimental unit and are presented in Table 3. By analyzing the relation between the feeding and the distillate product flow rates, we can observe a greater production of distillate (proportional to the feeding flow rate) for the film unit. However, it is important to notice that this unit could never operate with the flow rates similar to rates of the conventional processes. Despite of this, analyzing the ethanol mass fraction from the film unit it is possible to observe that is lower than desired (90% - conventional).

After the experimental tests in conventional unity, simulations based on these results were performed on Hysys® to estimate the ethanol mass fraction inside the column, i.e. in the trays, which were not possible to estimate experimentally. The value in the distilled product obtained for the falling film process (60% in mass) corresponds to what was found on the seventh stage of the separation in the conventional column (13 trays – zero on reboiler).

An additional, but simplified analysis was done on the energy supply. It shows that in conventional distillation is necessary around 13 kW of power supplied only on the bottom, while in the process by film 0.74 kW are used distributed along the tube. If those values are normalized in relation to the flow rate, we have a ratio of 0.04 kW per liter of liquid fed for conventional unit and also 0.04 kW for distillation by film. It is important to remember that the one conventional still has heat contributions due to the feeding stream and reflux that was counted in the total heat supply to the conventional distillation. A further analysis is necessary, but it is possible to believe that the energy provided in the distillation by film is feasible and relatively better distributed than the conventional one. This is a preliminary result once it also important to remember that was not obtained the same composition in the two configurations and the energy supply in film distillation could change with new operational conditions.

Table 3: Results obtained for the separation with the film and conventional distillation.

Variable	Conventional Unit	Unit by film
Feeding flow rate	300 $L.h^{-1}$	17 $L.h^{-1}$
Temperature in the bottom of the column	98°C	95°C
Mass Fraction of ethanol on the distillate	90%	60%
Distillate product flow rate	5 $L.h^{-1}$	1 $L.h^{-1}$

4. Conclusions

The tests performed have shown the proposal feasibility. They are promising in relation to the conditions of separation obtained versus the quantity of energy provided, especially if we take into account that the process is operating at atmospheric pressure, while all the reports in literature on distillation by film are carried out with vacuum.
Further studies are being carried out in order to evaluate the influence of residence time of the liquid with the efficiency of the separation. Surfaces improved in the distillation tube are enabling to increase the residence time, considering the analysis of the separation the next step.

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