MODELLING ETHANOL-WATER PRESSURE SWING DISTILLATION IN AN STRUCTURED PACKED BED COLUMN

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ABSTRACT

Applying the method of pressure swing distillation (PSD) we obtained (97 % v/v) ethanol starting from 95 % v/v ethanol, which shows that the ethanol-water azeotrope has been broken in this study. Experimental work to achieve the final product was carried out operating the column in two stages or modes, these being: (1) Low pressure distillation (LPD), and (2) High pressure distillation (HPD). In the first operating mode, a low degree industrial alcohol (80 % v/v ethanol) is fed to a bench scale distillation column packed with Mellapack structure, where (95 % v/v) ethanol is obtained, working at atmospheric pressure. Then product collected in the first stage is fed to the reboiler, and the HPD process takes place to get 97 % (v/v) ethanol. Here we have shown that it is possible to perform the pressure swing using an auxiliary engineered device, instead of using a second distillation column, in contrast to the opinion of Doherty and coworkers (2001). As part of this work we also have shown how to exploit the pressure sensitive property of an azeotropic mixture, as it is the case of the ethanol-water mixture. The ratio of the volume of product obtained to the energy consumption is 0.7 L. et-OH (97 %)/kWh.

Keywords: Pressure Swing Distillation, structured bed, intensification, azeotrope, non equilibrium thermodynamics.

RESUMEN

Aplicando el método de destilación por oscilación de presión (PSD) se obtuvo (97% v / v) de etanol a partir de etanol al 95% v / v, lo que demuestra que el azeótropo etanol-agua se rompe en este estudio. El trabajo experimental para lograr el producto final se llevó a cabo operando la columna en dos fases o modos, siendo estos: (1) la destilación a baja presión (LPD), y (2) destilación de alta presión (HPD). En el primer modo de funcionamiento, un alcohol industrial de bajo grado (etanol 80% v/v) se alimenta a una columna de destilación a escala de banco, empacado con estructura Mellapack, donde se obtiene etanol al 95% v / v trabajando a presión atmosférica. Luego, el producto colectado en la primera etapa se alimenta al hervidor, y el proceso de HPD tiene lugar para obtener 97% (v / v) de etanol. Aquí hemos demostrado que es posible llevar a cabo la oscilación de presión usando un dispositivo auxiliar de ingeniería, en lugar de usar una segunda columna de destilación, en contraste con la opinión de Doherty y colaboradores (2001). Como parte de este trabajo también hemos demostrado cómo explotar la propiedad sensible a la presión de una mezcla azeotrópica, como es el caso de la mezcla de etanol-agua. La relación entre el volumen del producto obtenido con respecto al consumo de energía es de 0.7 L. et-OH (97%) / kWh.

Palabras clave: destilación, cambio de presión, lecho estructurado, intensificación, azeótropo, termodinámica de no equilibrio.

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1. INTRODUCTION

Separation processes in chemical enginee-ring are used weather to purify raw materials, intermediate species, or final products to satisfy the market requirements.

Distillation is one of the most used separation process in the chemical industry, despite of its high energy consumption and low effi-ciency compared to other separation pro-cesses. Currently research works worldwide is aimed at intensifying processes, whether by designing compact units, or engineering accessories that enable an optimal use of the energy available.

In this study we conduct pressure swing distillation (PSD) in an structured packed bed column at bench scale, with no use of any mass separation agent. The final product obtained is ethanol over its azeotropic value (97 % v/v), after operating the same column at two different pressures.

2. SOME CONSIDERATION FOR CHOOSING PRESSURE SWING DISTILLATION

Chemical engineering has devised a series of separation processes and techniques to make products for the market. Among them, distillation is considered one of the most important separation processes. However, it is important to note that it is not a panacea to solve all separation problems. As a result, it is necessary to take into account certain aspects relevant to its selection as a sepa-ration process.

Some factors to be considered for a prelimi-nary decision making as far as selection of a separation method is concerned are:

- Minimum relative volatility 1.3.

- Thermally stable products.

- Production rate of 5,000 to 10,000 lb / d.

- High rates of corrosion and explosive reac-tions should not take place under conditions of distillation.

Concerning PSD, in the present case we have found that it is necessary to take into account the following considerations, before taking a decision:

- The concentration of the feed should be near to its azeotropic point.

- The azeotrope should be pressure sensitive type.

- Pressure swing should be conducted from low to high pressure, or viceversa, aimed at getting pseudo steady state operation.

2.1 Some applications of distillation

Distillation is widely applied in petroleum processing, for example for separation of propylene / propane. Oil refineries rely on distillation to obtain the products used in modern society, such as fuels, solvents, etc. Distillation processes equipped with heat pumps increase process efficiency. In aeronautics, distillation is used to recycle astronauts' urine.

Distillation strength is based on the fact that it is a process whose flowchart is quite sim-ple, it requires low cost of investment, it is feasible to scale up by the power law 0.6 or less. Its weakness is that it can not be used to separate temperature-sensitive mixtures, or mixtures whose components have close boiling points. In the same way it does not work to separate azeotropic mixtures. Whe-never distillation is not the chosen method of purification, other separation methods such as extraction, adsorption, crystallization, chromatographic separations, membrane separations, as well as hybrid systems come to play for a variety of industrial applications.

3. LITERATURE REVIEW

Lewis (1928) was the first to suggest disti-lling azeotropic mixtures by pressure swing distillation, PSD. This method is in principle similar to vacuum distillation, where the boi-ling point of the mixture is manipulated by the change in pressure of the system.

As it is well known, azeotropic mixtures cannot be separated into its components by traditional distillation, however, there exists some mixtures which may be named as pressure sensitive azeotropes, that may be separated by PSD (Sattler et al., 2008, Seider et al., 1999, Frank T., 1997). If the pressure increases, the azeotropic point is shifted to favor the separation of the mixture, in such a way that the azeotrope and the high boiling point component may be obtained as the distillate, otherwise the low boiling point component may be found as the major species in the distillate. This behavior depends upon the thermodynamic nature of the azeotrope.

Pressure swing distillation is used as an alternative process to azeotropic distillation, reactive distillation or extractive distillation. Thus in PSD the pressure of the system is used to break the azeotrope, and can be used in batch or continuous processes (Phismister et al., 2000). Few results for continuous processes have been found in the literature (Repke et al., 2005). Studies of PSD for the separation of multi component mixtures involving azeotropes may be found in the current literature (Modla et al., 2009).

Variations such as reactive-PSD has been applied to a methanol/methyl acetate azeotropic mixture produced as a waste stream in the polyvinyl alcohol industry. The ethyl acetate (EtAc) is a high value product and the methanol is a raw material for the polyvinyl alcohol industry which may be recycled in the process (Bonet et al., 2006, Phimister et al., 2000; Modla et al., 2008; Klein, 2008).

4. NON EQUILIBRIUM THERMODYNAMICS

Non equilibrium thermodynamics states the existence of systems that produce entropy, in such a way that if we were able to put in contact two entropy producing systems, we might transfer part of the available energy from one system to another, all in accord with the second law of thermodynamics. As a result, we might be able to transform the entropy produced in the system, into work

of separation. In practice it depends upon a series of factors and boundary conditions.

If we think on distillation under non equilibrium thermodynamics domain, we should be able to develop certain kind of auxiliary device, and to match properly the sub domains to transfer energy from side to another.

Now to theorize the statement, let us put forward the theory of entropy production from Onsager (1931).

$$T\sigma = \sum_i J_i X_i \qquad (1)$$

Where σ is the rate of entropy production across the frontier of the domain, T is the absolute temperature, J_i is the flow at steady state, and X_i represent the generalized forces.

Now using the relationship due to Tun Sac (2004), J is described by,

$$J_i = \sum_k L_{ik} X_k \qquad (2)$$

Where are the phenomenological coefficients. These coefficients satisfy the Onsager's relationships. Thus,

$$L_{ik} = L_{ik} \qquad (3)$$

Recalling the work of Palomino (2012), the previous described relationships generate,

$$T\sigma = -\sum_k J_k^* [RT\Delta(\ln y)_k] \qquad (4)$$

Here R is the gas universal constant.

This equation shows that if we are able to harness the entropy produced in a sub domain, we may use it as an separation agent, which is precisely what we have been able to perform in this study.

5. MATERIALS AND METHODS

A distillation column equipped with structured Mellapack type packing randomly ordered, constructed in stainless steel 304, has been used to carry out the experimental part of this study. The diagram of the distillation onset is shown in Figure N º1. The process is conducted in batch mode. In addition to

the distillation facility, standard laboratory glassware has been used for sampling and measurement purposes.

6. PROCESS DESCRIPTION

The diagram of the PSD is shown in Figure No. 2, which in spite of showing two columns of distillation, it rather represents two operating modes of the same distillation column; these being: (1) Low pressure distillation, and (2) High pressure distillation. In fact the second mode is much more relevant to the issue of removing the desired product, out of feeding an ethanol-water mixture near to its azeotrope composition.

The manipulation of the distillation pressure and the recycling ratio are key factors to carry out the PSD at pseudo steady state.
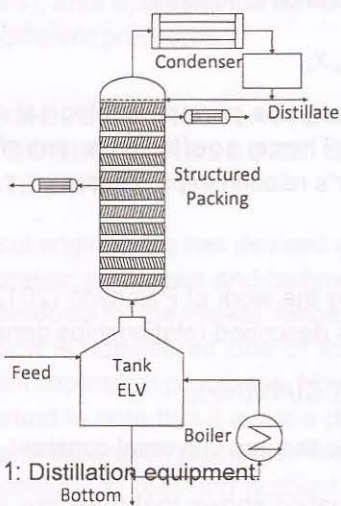
Fig. Nº 1: Distillation equipment

6.1 Low pressure distillation (LPD)

This operation is rather similar to a conventional rectifying distillation, and is conducted at atmospheric pressure (1.013 bar or 1 atm). Thus feeding 80 % v/v industrial alcohol, a product containing a concentration close to the azeotrope is obtained (95 % v/v).

A number of factors are combined to achieve the enrichment of the industrial alcohol, which are the nature of the packing, its contact area and the hydrodynamic of the column, the rate of heating and the recycling ratio; that on the whole configure a process of high energetic efficiency, that is to say, an intensified process.

Let us refer to the work of Wolley et. al. (1999), who stated that by conventional distillation it is possible to achieve around 90 to 92 % v/v, which is lower than our result (95 %).

6.2 High pressure distillation (HPD)

Once the first stage is completed and the ethanol-water mixture has increased it concentration close to its azeotropic point. The high pressure distillation starts by feeding the product obtained in the previous stage. This operation requires a departure condition, which starts at atmospheric condition, then the outlet valve located in the engineered device is closed. Thus the distillation column starts swinging its pressure up to the desired value, that is 2.26 bar in our case. Stabilization of the distillation procedure requires to match the rate of heating with the recycling ratio, to be able to get pseudo steady state operating condition. As a result, starting with 95 % v/v ethanol-water mixture, the separation of the components of the azeotropic mixture is achieved, since the new azeotrope is intrinsically connected to the new pressure of the distillation column. The product obtained at this condition is a mixture of 97 % (v/v), which is above its azeotropic point.
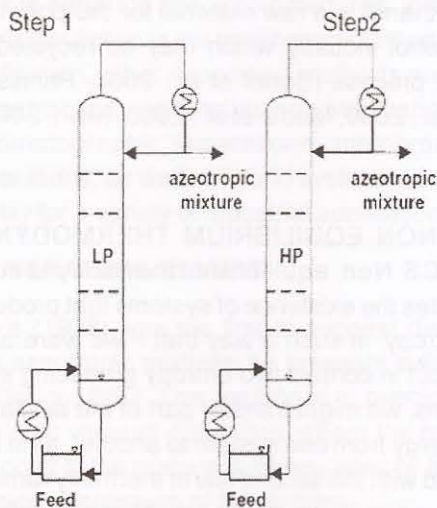
Fig. Nº 2: PSD Process diagram
(Adapted from Repke et al., 2007)

7. MODELLING OF THE PSD DISTILLATION

PSD may be mathematically described based upon the assumption that an intimate contact between two phases (vapor-liquid) coexist through the equilibrium stages, where J stages are idealized being in equilibrium. However, in the real world, such contact does not take place, weather due to the nature of the own packing, the velocity of the ascending vapor, or some anomalies concerning the rate of recycling of the liquid, as well as the distribution of vapor and liquid inside the separating column.

Modelling a real system of distillation is very cumbersome to achieve, due to a series of uncertainties involved, which goes from the proper knowledge of the parameters, as well as the operating conditions.

One way to face the modeling procedure of a real world distillation makes use of the theory of non equilibrium thermodynamics, which enables the possibility of using part of the entropy generated in the system as a separating energy, depending upon the engineered device used to claim such energy (Palomino, 2012).

7.1 Mass and energy balance in the process

Mass and energy balance are derived based on the MESH model. In order to facilitate the formulation of the model Figure N. 3 is presented, where an idealized jth-element of volume is shown. Mass balance equations for each compound:

$$M_{i,j} = L_{j-1}x_{i,j-1} + V_{j+1}y_{i,j+1} + F_j z_{i,j} \\ - (L_j + U_j)x_{i,j} - (V_j + W_j)y_{i,j} = 0 \quad (5)$$

Energy balance:

$$H_j = L_{j-1}h_{L_{j-1}} + V_{j+1}h_{v_{j+1}} + F_j h_{f_j} - \\ \S\S\S\S üⁿ_j + U_j \ h_{L_j} - V_j + W_j \ h_{v_j} - Q_j = \quad (6)$$

7.2 Solution

Equations (5) and (6)) generate a set of non linear algebraic equations that are solved using a highly efficient algorithm developed by Wang and Henke (1966).

A set of constitutive equations of Soave-Redlich-Kwong, UNIQUAC, Wagner, and the fugacity coefficient are used to perform the complete solution of the PSD problem.

NTU is calculated from the following equation:

$$z = NTU * HETP \quad (7)$$

Where HETP is obtained through a system of equations developed by Rocha-Bravo-Fair for Mellapack type structured packings. This model is based on the interaction between the descending liquid film and the ascending vapor occupying the space available. Also, the physical properties and the geometry of the packing are taken into account (Bravo et al., 1996). This model is used to predict the pressure drop, liquid retention, and the effective area of mass transfer.

Hydraulic effects

$$\frac{\Delta P}{\Delta z} = \frac{\Delta P_{seco}}{\Delta z}\left[\frac{1}{1 - \dot{K}h}\right]^5 \quad (8)$$

Liquid retention

$$h_l = \left(4\frac{F_t}{S}\right)^{\frac{2}{3}}\left(\frac{3\mu_l U_l}{\rho_l(sen\theta)\varepsilon g_{eff}}\right)^{\frac{1}{3}} \quad (9)$$

Fig. Nº 3: General scheme for heat and mass balance (Adapted from Seader et al., 1998)

Effective area

$$\frac{a_e}{a}=\frac{29.12 F_{se}\left(We_l Fr_l\right)^{0.15} S^{0.359}}{Re_l^{0.2}\,\varepsilon^{0.6}\left(1-0.93\cos\theta\right)\left(sen\alpha\right)^{0.3}} \quad 10)$$

Mass transfer coefficient in gas phase

$$\frac{K_g S}{D_g}=0.54\left(\frac{\left(U_{geff}+U_{leff}\right)\rho_g S}{\mu_g}\right)^{0.8}\left(\frac{\mu_g}{D_g\rho_g}\right)^{0.33} 11)$$

Mass transfer coefficient in liquid phase

$$K_l=2\sqrt{\frac{D_l C_E U_{leff}}{\pi S}} \quad (12)$$

8. RESULTS

The following figures show the limits of operation for each case, observing that stage LP (figure 4) works as a conventional distillation, below the azeotrope in terms of the more volatile component, and the stage HP (Figure N ° 5) operates according to the least volatile component and above the azeotropic point.

Tables No. 1 and 2 show the values of the mass and energy balance of each operation.

Clearly the low pressure distillation is less energetically efficient than the high pressure distillation, thus it may be observed that the HPD is 40 % more efficient than the LPD.

Simulation of the process show that the structured packed bed is equivalent to 15 theoretical stages.
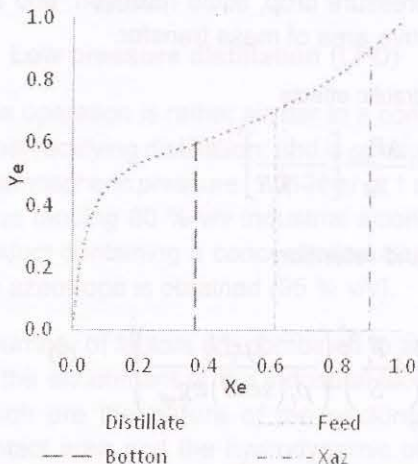
Fig. N° 4: Vapor -liquid equilibrium during LPD.
Source: Elaborated by the authors.
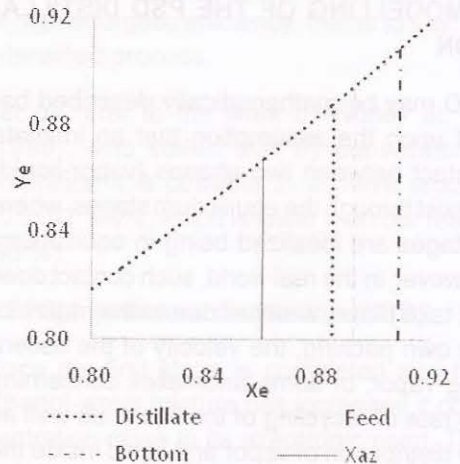
Fig. N° 5: Vapor-liquid equilibrium during HPD .
Source: Elaborated by the authors.

Table N° 1: Mass and energy balance for LPD
ΔP=3.2mmHg

Stage	Mass (kmol/s)	Energy (kW)	Exergy (kW)
Feed	7.16E-05	-2.854	-0.512
Top	-3.27E-05	1.164	0.206
Bottom	-3.89E-05	1.364	0.265
Q condenser		-5.059	-0.766
Q reboiler		5.384	0.868
Summatory	0.00E+00	0	0.061
Recycle			2.976
Calculated thermal efficiency			0.400
Experimental thermal efficiency			0.500
NTU			15

Source: Elaborated by the authors

Table N° 2: Mass and energy balance for HPD
ΔP=50mmHg

Stage	Mass (kmol/s)	Energy(kW)	Exergy (kW)
Feed	2.61E-04	-64.474	-81.843
Top	-1.80E-04	43.883	56.309
Bottom	-8.11E-05	19.750	25.403
Q condenser		-5.579	-1.118
Q reboiler		6.420	1.299

Summatory	-9.458E-11	2.148E-05	0.049
Recycle			1.385
Calculated thermal efficiency			0.728
Experimental thermal efficiency			0.900
NTU			14

Source: Elaborated by the authors

9. DISCUSSION AND ANALYSIS

According to the results obtained in this work, it appears that the idea proposed by Palomino (2012) is quite plausible, informed by the theory of the non equilibrium thermodynamics, since here we have used an engineered device inside the column, which made possible the separation of the azeotropic mixture of ethanol-water. This fact may demonstrate the fact that part of the entropy generated in the process has been converted into separating work by mean of the engineered device.

It is interesting to note that in order to succeed in the purpose of breaking the azeotrope, it is important to consider the following facts:

- The concentration of the feed should be near to its azeotropic point.
- The azeotrope should be pressure sensitive type.
- Pressure swing should be conducted from low to high pressure, or vice versa, aimed at getting pseudo steady state operation.

10. NOMENCLATURE

C_E: Surface Renovation Factor

D_g: Diffusivity Of Vapor Phase

D_i: Diffusivity Of Liquid Phase

Ft: Correction Factor Of Liquid Retention

Fv: Loading Factor For Vapor Phase

NTU: Number Of Transfer Units

K_g: Mass Transfer Coefficient For Vapor Phase

K_{ij}: Binary Interaction Paraments

K_i: Mass Transfer Coefficient For Liquid Phase

L: Rate Of Flow Of Liquid Phase

P: Total Pressure Of System

Pv_i: Vapor Pressure Os Component I

T: Absolute Temperature V: Rate Of Flow Of Vapor Phase

V: rate of flow of vapor phase

V_m: Molar volume

X_a: Molar fraction of water at liquid phase

X_e: Molar fraction of ethanol at liquid phase

X_i: Molar fraction of component i at liquid phase

Y_a: molar fraction of water at vapor phase

Y_e: molar fraction of ethanol at vapor phase

Y_i: molar fraction of component i at vapor phase

Z: compressibility factor

a: nominal surface area of the bed

a_e: effective interfacial area

f: friction factor

g: gravity acceleration constant

g_{eff}: effective gravity

h: liquid retention

s: corrugation length

z: height of packed bed

Greek letters

α: corrugation angle

αik: relative volatility of the light key

δop: average thickness of descendent film

θ: contact angle

λ: depletion factor

μg: vapor phase viscosity

μl: liquid phase viscosity

ρl: liquid density

ρv: vapor density

σ: tension energy

Є: vacuum fraction

Dimensionless Numbers

Re_g: Reynolds number of vapor phase

Re_l: reynolds number of liquid phase

Fr_l: Froude number of liquid phase

We_l: weber Number of liquid phase

10. CONCLUSIONS

➢ Pressure swing distillation at bench scale was performed, by switching the pressure from low (1.013 bar) to high (2.26 bar), thus distilling at 99.9°C. The final product was obtained in the second stage getting ethanol with 97 % v/v concentration, from a feed with 95 % v/v ethanol. This fact indicates that the ethanol-water azeotrope has been broken in the process.

➢ The model produced in this work is validated by simulation, with an error of 0.546 %, as being contrasted with experimental results.

➢ The topology of the structured packed bed, favored the process of contacting descending liquid with the ascending vapor inside the column. This is one of the most important components in the process of separating the mixture by the PSD method; the other one being the rate of vapor production and the velocity of recycling.

➢ The efficiency of the fractionating column in the LPD mode may be considered to be high, since the concentration jumps from 80 % v/v to 95 % v/v, which is very close to its azeotropic point.

➢ This process may be qualified as intensified, since the energy efficiency is high (73%). This value is far over a conventional distillation efficiency, which is 57%.

➢ Distillation under conditions of non equilibrium thermodynamics, produce entropy in the system. This energy is harnessed to improve the efficiency of the PSD, which explains why the energy efficiency is really high.

11. ACKNOWLEDGMENT

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