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PERFORMANCE INDICES TO DESIGN A MULTI-COMPONENT BATCH DISTILLATION COLUMN USING A SHORTCUT METHOD

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Abstract - In this paper, three quality or performance indices (Luyben's capacity factor, total annual costs, and annual profit) were applied for the design of a batch distillation column working at variable reflux. This work used the Fenske-Underwood-Gilliland short-cut method to solve a problem of four components (benzene, toluene, ethyl-benzene, and ortho-xylene) that needed to be separated and purified to a mole fraction of 0.97 or better. The performance of the system was evaluated using distillation columns with 10, 20, 30, 40 and 50 theoretical stages with a boil-up vapor flow set at 100 kmol/h. It was found that the annual profit was the best quality index, while the best case for variable reflux was the column with 50 stages. It was confirmed that the best case always required a reflux ratio close to the minimum.

Keywords: Batch distillation; Short-cut method; Variable reflux; Performance indices; Luyben´s capacity factor; Total annual cost; Annual profit.

INTRODUCTION

Distillation is the most used separation process in the chemistry and petrochemical industry, mostly in continuous processes, but it is also used in discontinuous processes. Batch distillation is widely used for the separation of specialty and fine chemicals and for the recovery of small amounts of solvent to obtain products of high purity and added value. Batch processing is the main feature of the pharmaceutical, biochemical and specialty chemical industries.

One advantage of batch distillation is the possibility to separate a mixture of several components with only one column and, although a batch distillation column uses more energy than continuous columns (Turton *et al*., 2003, page 59 and 60), many times it is convenient to operate in batch mode. For these reasons the design, analysis and optimization of batch distillation columns have deserved attention from several researchers. (Sundaram and Evans, 1993; Low and Sorensen, 2003; Santos *et al*., 2012; Jana *et al*., 2013; Scanavini *et al*., 2012; Garcia *et al*. 2014).

Figure 1 shows a conventional batch distillation column (batch rectifier) consisting of a re-boiler or pot, a column with plates or packing, a condenser, a reflux tank, and accumulative recipients for products (main cuts) and byproducts (off cuts).

There are two main operating methods for batch distillation columns: constant reflux (and variable product composition of the distillate), and variable reflux (and constant product composition, for one component, of the distillated product). Another oper-

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*To whom correspondence should be addressed ating method is the optimal reflux policy, which tries to employ an economical combination of the two main operating methods, according to established criteria like: minimum time, maximum product, or maximum profit.

The dynamic process of a batch distillation column can be modeled through the balance of mass and energy and coupling them with equilibrium ratios obtained from thermodynamic or experimental vapor liquid equilibrium records.

The models may vary from McCabe Thiele binary distillation with constant or variable reflux, to short-cut or approximate methods (Diwekar and Madhavan, 1991; Sundaram and Evans, 1993; Barolo and Guarise, 1996; Salomone *et al*. 1997) to rigorous methods (Meadows, 1963; Distefano, 1968; Boston *et al*., 1981; Galindez and Fredenslund, 1988).

Performance indices have been used for discriminating between alternatives, not only for design (Luyben, 1971; Quintero-Marmol and Luyben, 1990; Al-Twain and Luyben, 1991, Bernot *et al*., 1993), but mainly for optimization (Diwekar *et al*., 1989; Ki-Joe and Diwekar, 2000; Low and Sorensen, 2003; Santos *et al*., 2012). As shown in Table 1, indices are separated in three classes: batch distillation parameters, energy, and money.

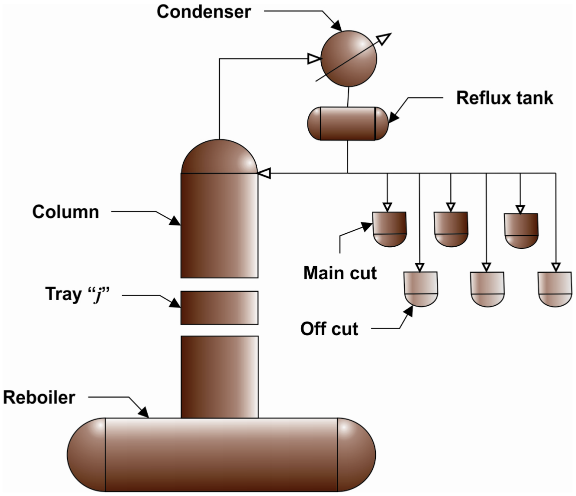


Figure 1: Batch distillation column with three receivers for products (Main cut) and three receivers for mixtures (Off cut).

Table 1: Performance indices used for batch distillation columns.

Class	Performance index name	Performance index equation	Reference
I	Product purity	$x_{D,i,} x_{Bi}$	
I	Product yield	D_i, B_i	Santos *et al*. 2012
I	Batch time	$t_t = t_p + t_f$	
I	Batch distillation capacity	$CAP = \frac{\sum_{j=1}^{NC} P_j}{t_t + 0.5}$	Luyben, (1971), Quintero-Marmol *et al*. (1990), Santos *et al*. (2012)
II	N-feasibility index	$N_f = 1 - \frac{(N_{\min})_{avg}}{N}$	Ki-Joo & Diwekar (2000)
II	R-feasibility index	$R_f = 1 - \frac{(R_{\min})_{avg}}{R}$	Ki-Joo & Diwekar (2000)
II	Thermodynamic efficiency	$\eta = \frac{W_{\min}}{W_{\min} + \varepsilon_{loss}}$	Ki-Joo & Diwekar (2000)
II	Energy	$E = \frac{\int_0^{t_t} Q(t)\,dt}{t_p + t_f}$	Low and Sorensen (2003)
III	Total Annual Cost	$TAC = C_{capital} + C_{operation}$	Luyben (1991)
III	Profit	$P = \sum C_i D_i NB - \frac{C_1 NV}{G_a} - \frac{C_2 V}{G_b} - C_3 \cdot V \cdot t \cdot NB$	Diwekar *et al*. (1989)
III	Profit	$P = \left[\left(P_i C_i - FC_f \right) - \frac{180 V t_b}{8000} \right] NB - 1500 V^{0.5} N^{0.8} + 9500 V^{0.65}$	Santos *et al* (2012)
III	Profit	$P = \frac{\sum_{i=1}^{N_c} C_i H_i \left(t_f \right) - C_{feed} H_{feed}}{t_f + t_s} - K_1 N^{0.802} V^{0.533} - K_2 V^{0.65} - K_3 V$	Low and Sorensen (2003)
III	Annual Profit	*A.* *P.* = (Annual sales revenue-annual feed cost) -*TAC*	This work

As was before for the case for continuous distillation, energy consumption and efficiency are now an area of opportunity in batch distillation. The papers of Nawaz-Khan *et al*. (2012), Jana *et al*. (2013), and Asiedu *et al*. (2014) show that some strategies used for continuous distillation columns (such as vapor recompression and heat integration) are now being applied to batch distillation.

This work presents data for constant reflux operation reported by Narvaez *et al*. (2013) and a variable reflux short-cut method to design a batch distillation system (column, condenser and re-boiler) while evaluating three quality indexes: Luyben's capacity factor (CAP), total annual cost (TAC), and annual profit (AP).

MATERIALS AND METHOD

The Problem

The problem was to design (size) a distillation column, with condenser and re-boiler operating at atmospheric pressure with variable reflux, to separate and purify an equimolar mixture of benzene, toluene, ethyl-benzene, and ortho-xylene, containing 100 kmol of each, and needing products of each component with molar fractions of 0.97 or better. The boil-up rate *V* was fixed at 100 kmol/h. The internals of the distillation column could be sieve trays. Also, the area of the condenser and re-boiler is to be calculated, as well as the steam and cooling water needed in the re-boiler and condenser, respectively.

It seems convenient to report three performance indices: Luyben's capacity factor, total annual cost, and annual profit for several alternatives for a number of stages, for example: 10, 20, 30, 40, and 50 theoretical plates, it seems also convenient to report reflux ratio profiles for several runs.

Previous Work and Model Description

It was Diwekar in her 1988 Ph. D. dissertation who first reported the shortcut method for batch distillation columns and the paper by Diwekar and Madhavan (1991) is considered to be the first in a major journal. Their paper described both constant and variable reflux. In 1993, Sundaram and Evans reported their method for simulating batch distillation operation. It only covered constant reflux ratio. Both papers assumed that, at any time, the batch column was identical to the rectifying section of a continuous column and applied the shortcut (FUG) method: Fenske (1932), Underwood (1932, 1946), and Gilliland (1940) assuming that the relative volatility of the components remained constant. Both methods began with the setting $x_{W_i} = x_{F_i}$. Gilliland graphical results were correlated first by Molokanov *et al*. (1972) and then by Eduljee (1975) with the simpler expression Equation (9) in Table 2. Barolo and Guarice (1996) extended their continuous short-cut method to batch distillation columns and used an infinite stage column.

The FUG shortcut method assumes that the vapor and liquid holdup on each tray is negligible. This is true for the vapor phase at the normal low pressures of many distillations. The amount of liquid holdup is higher for tray columns than for packed columns especially those using structured packing. Seader and Henley (2006) wrote in Section 13.5: *A batch rectifier is usually operated under total-reflux condition for an initial period of time prior to the withdrawal of distillated product. During this initial time period liquid holdup in the column increases and approaches a value that is reasonably constant for the remainder of the distillation cycle.*

Seader and Henley (2006) applied the Sundaram and Evans (1993) method and showed how the FUG equations were used to solve a simulation problem with constant reflux. They assume that both vapor and liquid holdup is negligible and presented global (1) and component *i* (2) mass balances:

$$-\frac{dW}{dt} = D = \frac{V}{R+1} \tag{1}$$

$$\frac{d\left(x_{W_i}W\right)}{dt} = x_{D_i}\frac{dW}{dt} \tag{2}$$

Solving Equation (1) for W (using k for the time increment index):

$$W^{(k+1)} = W^{(k)} - \frac{V}{\left(R+1\right)}\Delta t \tag{3}$$

Solving Equation (2) for x_{Wi} (using k for the time increment index), and i for any component:

$$x_{W_i}^{(k+1)} = x_{W_i}^{(k)} + \left(x_{D_i}^{(k)} - x_{W_i}^{(k)}\right)\left[\frac{W^{(k+1)} - W^{(k)}}{W^{(k)}}\right] \tag{4}$$

Equations (3) and (4) together with other algebraic equations form the basis of the calculations. Equation (3) gives a new value for total molar mass at the new time at the bottom of the column, and

Equation (4) gives the concentration of the component (i) at the bottom at the new time.

For constant reflux the reference component was the heaviest, for the problem r=4=o-xylene. For variable reflux and constant distillated composition, the reference is the lightest component, r=1=benzene for the first separation of four components, r=1= toluene for the second or three components separation, and r=1=ETB for the third or two components separation. Equations (3) and (4) may be applied also for variable reflux but it is necessary to relate x_{Di} and x_{Wi} with the Hengstebek-Geddes Equation, (5) and (6) in Table 2.

A part of the procedure followed by Diwekar and Madhavan, 1991 was combined with the procedure used by Seader and Henley (2006) (Section 13.6) for constant reflux for variable reflux and constant composition for a light component that is the reference component, but now the minimum reflux calculation is more general.

Table 2: Actions and equations used in batch distillation column operating at variable reflux.

Step	Action	Equation	Reference
1	Data needed	$N_c,\ \alpha_{i,HK},\ x_{W,i},\ N,\ x_{D,r},\ V,\ W_0$ r=reference component= LK HK=Heavy component (O-Xy)	
2	Find $N_{\min}$ with summation of $x_{D,i}$=1 by using Hengstebeck-Geddes Eq.	Hengstebek-Geddes: $\sum_{i=1}^{N_c} x_W^i \left(\frac{\alpha_{i,HK}}{\alpha_{r,HK}}\right)^{N_{\min}} \left(\frac{x_D^r}{x_W^r}\right) - 1 = 0 \qquad (5)$	E-12 in Diwekar and Madhavan (1991)
3	Find $x_{D,i}$ for the non-reference components. i=2,..Nc	$x_D^i = x_W^i \left(\frac{\alpha_{i,HK}}{\alpha_{r,HK}}\right)^{N_{\min}} \left(\frac{x_D^r}{x_W^r}\right) \qquad (6)$	E-8 in Diwekar and Madhavan (1991)
4	Find θ from Underwood Equation. q=1 for feed as saturated liquid	$\sum_{i=1}^{N_c} \frac{\alpha_{i,HK} x_{w_i}}{\alpha_{i,HK} - \theta} = 1 - q \qquad (7)$	E-9-28 in Seader and Henley (2006)
5	Find $R_{\min}$ from Underwood Equation	$R_{\min} + 1 = \sum \frac{\alpha_{i,HK} x_{D,i}}{\alpha_{i,HK} - \theta} \qquad (8)$	E-9-29 in Seader and Henley (2006)
6	With Gilliland (Eduljee) solve for R with N, $N_{\min}$, and $R_{\min}$	$\frac{N - N_{\min}}{N+1} = 0.75\left(1 - \left(\frac{R - R_{\min}}{R+1}\right)^{0.5668}\right) \qquad (9)$	E-13-28 in Seader and Henley (2006)
7	With the value for R, stepping forward in time and calculate new molar mass at bottom and new molar fraction of all components	$W^{(k+1)} = W^{(k)} - \left(\frac{V}{1+R}\right)\Delta t \qquad (3)$ $x_W^{(k+1)} = x_W^{(k)} + \left(x_{D_i}^{(k)} - x_{W_i}^{(k)}\right)\left[\frac{W^{(k+1)} - W^{(k)}}{W^{(k)}}\right] \qquad (4)$	E-13-22 in Seader and Henley (2006) E-13-23 in Seader and Henley (2006)
8	Verify W, x_W, D, or t		
9	Repeat steps 2 to 8 for several cycles.		
10	Stop		

In this work, to solve the problem, the strategy was to simulate the operation of a batch distillation column with 10 to 50 theoretical stages maintaining the mole fraction of the light component at a value of 0.97, and rank the runs according to three qualification parameters: Luyben's capacity factor, total annual cost, and annual profit. It was expected that the qualification parameters would point to the most convenient combination.

Equilibrium Ratios and Relative Volatility Used for the Shortcut Method

For liquid vapor equilibrium the binary experimental data reported in Gmehling *et al.* (2000) were used that compare well with predicted values using Raoult's law and Antoine vapor pressure and with runs using Aspen plus with the thermodynamic model recommended by the experimental data in Gmehling *et al.* (2000). Relative volatilities taking the heaviest component ortho-Xylene as reference were calculated for the top and bottom of distillation column and a geometrical average provided: $\alpha_{B\text{-}O\text{-}Xy}=6.7$, $\alpha_{T\text{-}O\text{-}Xy}=2.8$, $\alpha_{ETB\text{-}O\text{-}Xy}=1.3$, $\alpha_{O\text{-}Xy\text{-}O\text{-}Xy}=1.0$.

It is convenient to note in Table 2 that, for variable reflux calculation, the reference component is the light component, while for constant reflux operation the reference component is the heaviest.

Quality or Performance Indices

Luyben's Capacity Factor (CAP)

Quintero-Marmol and Luyben (1990) did not use the shortcut method, but studied and reported simulation results for binary separations covering the following practical aspects of optimal design: Number of trays, reflux ratio, tray holdup, and amount of initial charge to the still. For a complete batch cycle they included: startup time or equilibration time (t_E), time needed to bring the column to the steady state condition in total reflux; overhead product withdrawal or process time (t_p), period during which on-specification distillate product is produced; Final bottoms product purification time (t_F), time required to remove enough low boiler from the still pot and column trays so that the bottoms product will be on-specification when the liquid in the column drains down into the still pot. They established that the design objective was to maximize capacity and they defined the capacity as the amount of total on-specification products produced per unit time (*i.e.* the sum of distillate and bottoms). They concluded that, as is usually the case in continuous distillation columns, the optimum combination of reflux ratio and number of trays is usually in the direction of many trays and low reflux ratio.

Al-Tuwaim and Luyben (1991) used simulation results of a rigorous model for binary and ternary mixtures with the following assumptions: Theoretical trays, equimolar overflows, constant relative volatilities, constant tray holdup (1 lb-mol), constant reflux drum holdup (10 lb-mol), and total slop recycle. Their approach was to find the optimal reflux ratios for columns with various numbers of trays and specify the capacity factor proposed by Luyben (1971), which, applied to a multi-component mixture, resulted in the following:

$$CAP = \frac{\sum_{j=1}^{NC} P_j}{t_E + t_P + t_F + t_{r\&r}} \cong \frac{\sum_{j=1}^{NC} P_j}{t_P + t_F + 1.5} \tag{10}$$

It was observed that the equilibration time t_E, the time needed to reach total reflux and the product removal and the recharging time $t_{r\&r}$ were additive and assumed a value of 1.5 hours.

Total Annual Cost (TAC) for Comparison

Total annual cost given by:

$$\begin{aligned} \text{TAC} = {} & \text{Annual cost of equipment} \\ & + \text{Annual cost of utilities} \end{aligned} \tag{11}$$

This is an index used for comparison of alternatives that take into account the annual capital and operational costs of equipment: Distillation column (plates and shell), re-boiler and condenser, and steam for re-boiler and cooling water for the condenser. With the boil-up fixed at V=100 kmol/h and the external reflux ratio $R=L/D$ calculated by the computer program, the liquid flow rate ($L=R^*D$) and internal ratio L/V can be calculated as:

$$\frac{\dot{L}}{V} = \frac{R \cdot D}{R \cdot D + D} = \frac{R}{R+1} \tag{12}$$

With the molar flow rates of liquid and vapor, the diameter of the column can be calculated using physical properties (ρ, μ) and geometric parameters to predict the flooding velocity (U_f) and fixing the operational vapor velocity as a fraction (f≅0.8) of it, the column diameter is (Seader and Henley 2006, Page 217):

$$D_{col} = \sqrt{\frac{4 \cdot V \cdot M_v}{f \cdot U_f \cdot \pi \cdot \left(1 - \frac{A_d}{A_{col}}\right) \cdot \rho_V}} \tag{13}$$

The calculation of the flooding velocity U_f was first proposed by Fair (1961) plotting the Souders and Brown capacity factor (C_f) versus a kinetic energy ratio (F_{LV}). Treybal (1980) and Doherty and Malonne (2001) adapted it to analytical determination.

With the molar flow of the vapor V that must be condensed and the heat of condensation of distillate vapor, Q_C was calculated, and from it the condenser area A_C was determined with the heat transfer equation using the overall heat transfer coefficient $U_C \cong 850$ W/m^{2o}C suggested by Turton *et al.* (2003) (Table 9.11, page 345). ΔT_C is the logarithmic mean of the temperature driving force between the cooling water and organic condensation temperature. Additionally, the flow rate of cooling water was obtained.

$$\begin{aligned} Q_C &= V \cdot \Delta H_{cond} = U_C \cdot A_C \cdot \Delta T_C \\ &= m_{CW} \cdot Cp_{CW} \cdot \Delta T_{CW} \end{aligned} \tag{14}$$

At the bottom of the column, heat was needed to vaporize V kmol/h of the liquid and, with the heat of vaporization of the liquid, the bottom re-boiler area A_R was obtained using $U_R \cong 1140$ W/m^{2o}C (Turton *et al.*, 2003, Table 9.11, page 345), as well as the flow rate of the steam needed as main utility.

$$\begin{aligned} Q_R &= V \cdot \Delta H_{vap-bottommixture} = U_R \cdot A_R \cdot \Delta T_R \\ &= m_{steam} \cdot \Delta H_{vap-steam} \end{aligned} \tag{15}$$

Annual Profit (AP) for Comparison

A complete economic analysis would require the calculation of annual gain or profit:

$$\begin{aligned} \text{Annual profit} &= \text{total annual income} \\ &\quad - \text{total annual cost} \end{aligned} \tag{16}$$

Annual income is the amount of money collected when the four harvested products are sold. Table 6.4 of Thurton *et al.* (2003) provides averaged values from the Chemical Market Reporter for January-April, 2001 for some compounds. Below is the cost for three:

Chemical	**Cost (USD/kg)**	**Typical shipping capacity or basis for price**
Benzene	0.349	Barge, Gulf Coast
Toluene	0.322	Barge, Gulf Coast
Ortho-xylene	0.446	Railroad tank cars

The selling price for ethyl-benzene was assumed to be equal to that of ortho-xylene.

RESULTS AND DISCUSSION

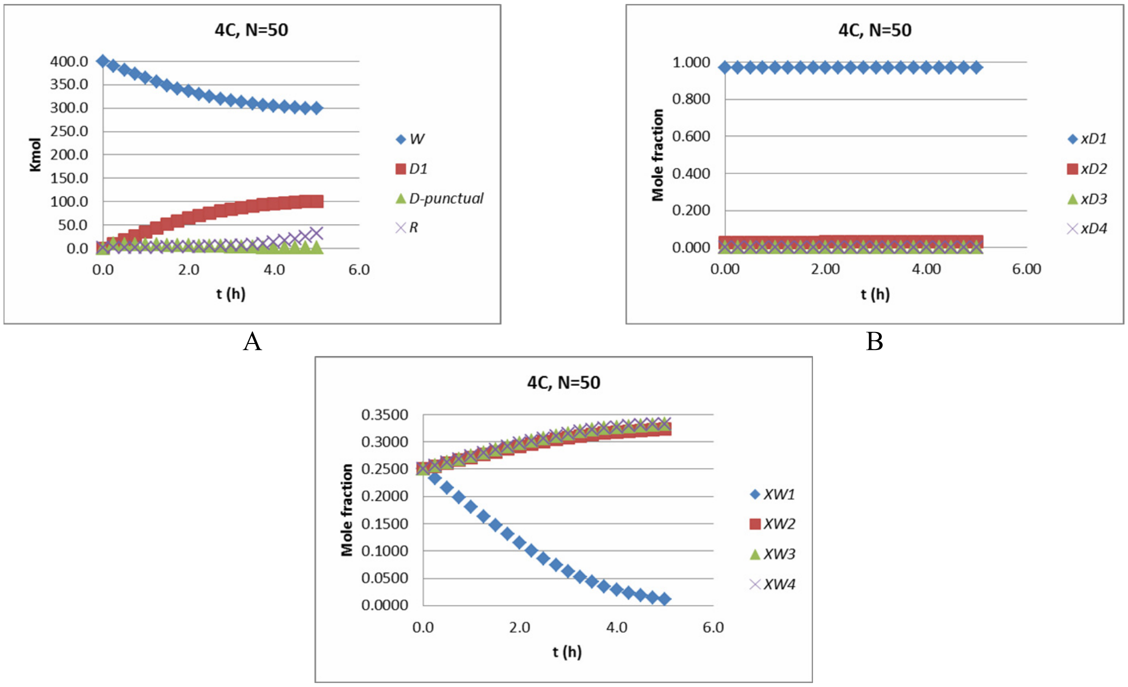
Four Component Separation

With the equations of Table 2 computer programs in Excel were prepared, validated with the data of Diwekar and Madhavan (1991) and then applied to the four component problem. For this separation, benzene is collected as distillated product with mole fraction of 0.97 and the reflux ratio is adjusted (increased) to get that result; the mixture of toluene, ethyl-benzene and o-xylene are concentrated in the pot.

Benzene and toluene have a relative volatility of 2.39 compared with toluene and ethyl-benzene of 2.15, and ethyl-benzene and o-xylene of 1.3. This separation is easy and distillation batch columns with 10, 20, 30, 40, and 50 theoretical stages may do the job. Table 3 and Figure 2 show partial results.

Table 3: Comparative results for 4 component separation.

4C	***N*=10**	***N*=20**	***N*=30**	***N*=40**	***N*=50**
Time (h)	7.0	5.50	5.0	5.0	5.0
X_{W1}	0.0056	0.0056	0.0049	0.0050	0.0050
W	299.94	299.13	298.94	298.97	298.97
$D1$ (kmol)	100.06	100.87	101.06	101.03	101.03
$D_{punctual}$ (kmol/h)	8.69-0.48	9.63-0.93	9.91-0.95	9.61-0.95	9.61-0.95
N_{min}	4.05-7.59	4.05-7.74	4.05-7.87	4.05-7.80	4.05-7.80
θ	4.23-6.46	4.23-6.49	4.23-6.50	4.23-6.50	4.23-6.50
$R\text{-}t_0$	1.88	1.60	1.60	1.60	1.60
$R\text{-}t_f$	58.74	32.3	31.74	31.63	31.63
Sum of reflux area	97.69	39.67	38.88	38.85	38.85
R-average	15.53	8.70	8.53	8.52	8.52



A B

C

Figure 2: Results for distillation of benzene from the 4 component mixture with 50 stages.

Figure 2A shows the depletion of total mass (W) at the bottom, the accumulated distillate ($D1$) at the top, the reflux ratio (R), and the actual distilled flow rate ($D_{-punctual}$) that decreased with time because the reflux ratio (R) increased to keep constant the mole fraction of the reference light component.

It is convenient to observe the increase of the reflux ratio R over time from 1.60 to about 31.6 in order to maintain constant the value of x_{DA}=0.97.

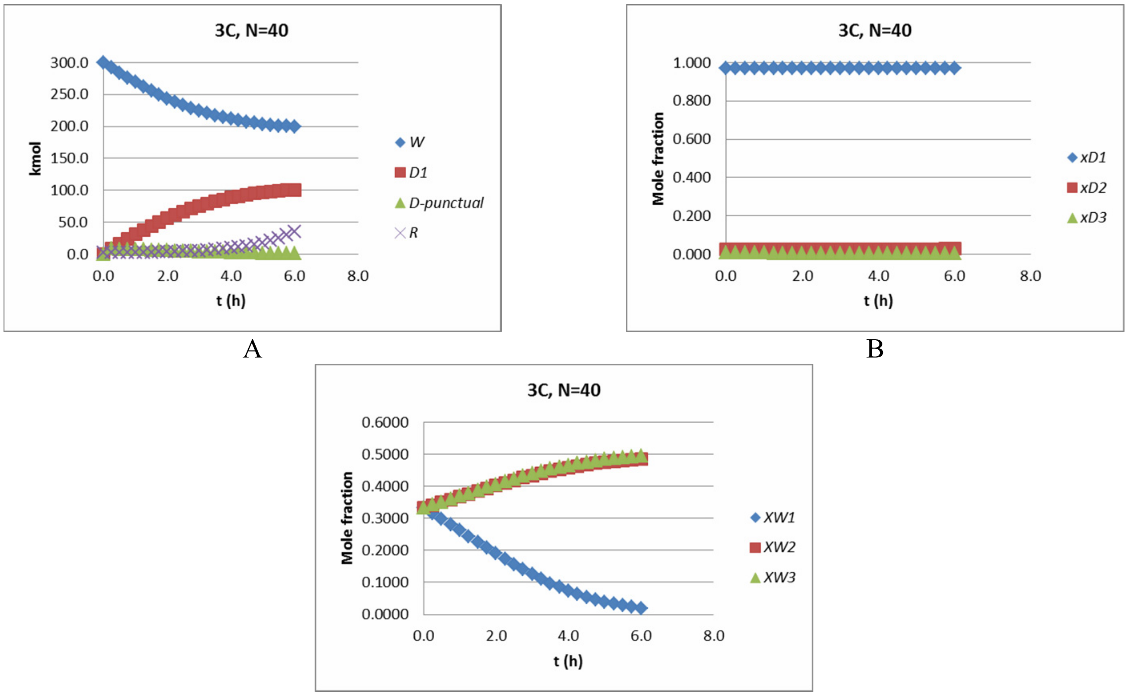
Three Components Separation

It is assumed that the new run will begin with 300 kmol of an equimolar mixture of toluene, ethyl-benzene and ortho-xylene. Now toluene is the light and reference component. Relative volatility between toluene and ETB is now 2.15 instead of 2.39 for benzene and toluene. Although the separation is more difficult, the five batch distillation columns may still do the job of getting a mole fraction of 0.97 for toluene at the second distillate receiver by adjusting the reflux ratio and concentrating ethyl-benzene and ortho-xylene at the bottom, both with a mole fraction of about 0.5. Table 4 and Figure 3 show partial results of this operation at variable reflux.

Note that in Table 4 the row with the values of R-t_0 and R-t_f present values for initial (t_0) and final (t_f) reflux ratio that increase with time to maintain constant the value of X_{DT}=0.97. In this row there seems to be no difference, but a difference is clear in the last rows of the sum of reflux area and average reflux ratio.

Table 4: Comparative results for 3 component separation

3C	N=10	N=20	N=30	N=40	N=50
Time (h)	11.0	6.0	6.0	6.0	6.0
X_{W1}	0.0246	0.0090	0.0068	0.0068	0.0069
W	199.95	199.93	199.36	199.40	199.40
$D1$ (kmol)	100.05	100.07	100.64	100.60	100.60
$D_{punctual}$ (kmol/h)	6.99-0.18	8.23-0.81	8.16-0.81	8.16-0.81	8.16-0.81
N_{min}	4-85-8.81	4.85-8.68	4.85-10.8.85	4.85-8.85	4.85-8.85
θ	1.92-2.72	1.92-2.72	1.92-2.72	1.92-2.73	1.92-2.73
R-t_0	2.57	2.03	2.06	2.06	2.06
R-t_f	146.10	36.06	36.06	36.06	35.56
Sum of reflux area	383.26	56.25	55.89	55.73	55.73
R-average	36.47	10.06	10.03	9.99	9.98



A B

C

Figure 3: Results for distillation of toluene from the 3 component mixture with 40 stages.

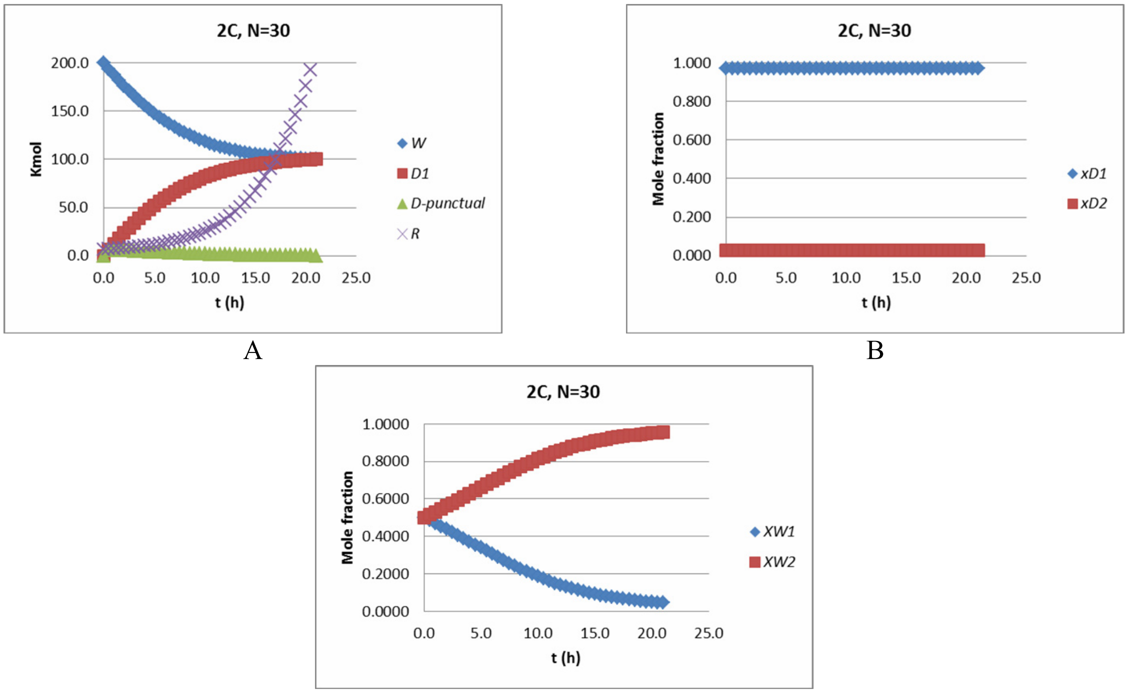
Two Components Separation

It is assumed that this run will begin with 200 kmol of an equimolar mixture of ethyl-benzene and ortho-xylene with a relative volatility of 1.3 that is smaller than for the previous separations. Now ethyl-benzene is the light and reference component. Table 5 and Figure 4 show partial results.

For this two component separation, a longer time and higher averaged reflux ratio are observed than those needed for 3 and 4 component separation. This happens because the separation is the most difficult. Note in Table 5 that the minimum number of stages range from 13-25 and then the columns with 10 and 20 theoretical stages will not be able to perform the separation of ethylbenzene and ortho-xylene and we are left with only three options: 30, 40, and 50 theoretical stages.

Table 5: Comparative results for 2 component separation.

2C-ETB	*N*=10	*N*=20	*N*=30	*N*=40	*N*=50
Time ETB (h)			21	15.5	14.5
X_{W1}			0.0443	0.0467	0.0436
W			99.98	99.98	99.54
$D1$ (kmol)			100.02	100.02	100.56
$D_{punctual}$ (kmol/h)			6.51-0.26	6.87-0.56	6.87-0.64
N_{min}			13.23-24.96	13.23-24.74	13.23-25.02
θ			1.13-1.28	1.13-1.28	1.13-1.28
$R\text{-}t_0$			6.20	6.27	6.27
$R\text{-}t_f$			210.03	99.00	87.73
Sum of reflux area			1108.66	423.27	350.25
R-average			55.4	28.80	25.56
2C-O-Xylene					
Time- ortho-xylene (h)			25.5-21=4.5	18-15.5=2.5	16-14.5=1.5
x_{W1}			0.0297	0.0280	0.0297
x_{W2}			0.9703	0.9719	0.9703
W			98.42	97.99	98.06
$D_{punctual}$ (kmol/h*)*			0.26-0.12	0.56-0.30	0.64-0.42
$R\text{-}t_o$			210.03	99.00	87.73
$R\text{-}t_f$			443.30	183.45	134.01
Sum of reflux area			2526.07	766.52	514.35
R-average			103.34	45.05	34.14



A

B

C

Figure 4: Results for distillation of ETB and O-Xy from the 2 component mixture with 30 stages.

The first eleven rows present the data from the computer program written in Excel for re-collection of ethyl-benzene in the distillate with the desired x_{TB}=0.97 obtained by adjusting the reflux ratio and noting than now it requires higher values than the other two separations.

The last eight rows of Table 5 present the data from the computer program for the purification of ortho-xylene to get the desired composition at the pot or re-boiler. This last operation was also at variable reflux. It should be noted that the purification of ortho-xylene begin where the distillation of ethyl-benzene stops. The operation continues at variable reflux. These values for reflux ratios are the highest. The ranges of reflux ratio are 210-443 for N=30, 99-183 for N=40, and 88-134 for N=50.

Luyben's Capacity Factor (*CAP*)

From Tables 3, 4 and 5 it is observed that the first three components reach 100% recovery of 100 kmol and from the bottom part of Table 5 it is observed that ortho-xylene recovery ranges from 97.99 to 98.42 depending of the number of theoretical stages.

Luyben's capacity factor for the distillation columns compared are:

$$CAP_{N-30}=\frac{100+100+100+98.42}{5.0+6.0+21.0+4.5+1.5}=10.48\frac{\text{kmol}}{\text{hr}} \quad (17)$$

$$CAP_{N-40}=\frac{100+100+100+97.99}{5.0+6.0+15.5+2.5+1.5}=13.05\frac{\text{kmol}}{\text{hr}} \quad (18)$$

$$CAP_{N-50}=\frac{100+100+100+98.06}{5.0+6.0+14.5+1.5+1.5}=13.97\frac{\text{kmol}}{\text{hr}} \quad (19)$$

Note that the penultimate term in the denominator is the finalization term (t_f) and the last term (1.5 hours) is for equilibration plus the removal and recharging time. Probably the last term should be larger, but 1.5 hours was used for this work.

For Luyben's *CAP* the higher the better. The performance index says that the 50 stage column provided better capacity per unit time.

Total Annual Cost

The CAPCOST Excel program from Thurton's Book (2003) was used to estimate the cost of equipment: condenser (floating head), reboiler (Kettle) and sieve tray column all on stainless steel. Ten year was taken for the depreciation period. The same program was used to estimate the annual operating cost: cooling water and heating steam. Two calculation procedures were done: I.- With the heat duty on condenser and re-boiler in W=Joule/s multiplying it by the time in seconds of the operating time in one year. It was assumed that the distillation column will operate 48 of the 52 weeks, then providing a ratio of

0.923 and using the cost in USD/GJ (0.354 for cooling water and 7.78 for low pressure steam) the amount of USD for each utility was calculated; II.- The flow rate of cooling water and heating steam in kg/h is multiplied by the number of hours worked in one year and then multiplying it by the cooling water (14.8 USD/(1000 m^3) cost and heating steam (16.22 USD/1000 kg) cost. The results of both methods were close. In both cases the cost of heating steam is more than 25 times that of the cooling water.

The annual cost for equipment and steam and cooling water for each column with 30, 40 and 50 theoretical stages was calculated for each separation: 4 components (Benzene), 3 components (Toluene), two components (Ethyl-benzene and O-xylene). Table 6 shows some specific values. It is observed that the two component separation requires the more expensive column because it has the biggest diameter and because more steam is spent. However, the difference is small: 1.02 m versus 1.00 for diameter and 1.201 kW versus 983 kW for re-boiler duty. In order to get the total annual cost, several pondered averages were tried, but it was decided to use the conservative values obtained with the two component column.

Table 7 shows the summary of estimated total annual cost and also the other performance indices. Variable reflux as well as some constant reflux cases are included. For *TAC* the lower the better, this quality index says that the 30 stage distillation column is the one with the lower total annual cost. This is opposite to the first performance index that says that the 50 stage column performs better (18.88>13.43>10.58).

Annual Profit

Equation (16) was used to estimate the total annual income that is calculated by adding the USD obtained from selling each product. Using data from Tables 3 to 5 and using molecular weight (kg/kmol) the kmol of each compound were converted to kg and then multiplied by the compound cost. For the 30 stage column it was obtained:

$$\begin{aligned}\text{Annual income}_{N-30} &= 2726.04 + 2965.62 \\ &\quad + 4736.52 + 4659.93 \\ &= 15088.11\end{aligned} \tag{20}$$

Table 7 shows the summary of annual profit that results from subtracting the cost of the raw material and the total annual cost from the annual income.

For annual profit the higher the better: the 50 stage batch distillation column is the best option.

Table 6: Results for several batch distillation simulations at variable reflux ratio.

	B *N30*	B *N40*	B *N50*	T *N30*	T *N40*	T *N50*	ETB *N30*	ETB *N40*	ETB *N50*
Averaged reflux ratio	9	9	9	10	10	10	56	29	26
Time (h)	5.0	5.0	5.0	6.0	6.0	6.0	25.0	18.0	16.0
Tc (^{o}C)-final	81.4	81.4	81.4	111.9	111.9	111.9	136.5	136.5	136.5
Qc (kW)-final	856	856	856	923	923	923	988	988	988
m-H_2O-enf (kg/h)	49.101	49.101	49.101	52.950	52.950	52.950	56.705	56.705	56.705
Ac (m^2)-final	34.62	34.62	34.62	21.91	21.91	21.91	17.61	17.61	17.61
Dc-top (m)-final	0.90	0.90	0.90	0.96	0.96	0.96	1.02	1.02	1.02
Tr (^{o}C)-final	127.4	127.4	127.4	139.9	139.9	139.9	144.0	144.0	144.0
Qr (kW)-final	983	982	983	1.002	1.002	1.002	1.021	1.021	1.021
m-VH_2O-cal (kg/h)	1.691	1691	1.691	1.724	1.724	1.724	1.757	1.757	1.757
Ar (m^2)-final	124.6	124.6	124.6	127.0	127.0	127.0	129.5	129.5	129.5
Dc-Bottom (m)-final	1.00	1.00	1.00	1.02	1.02	1.02	1.02	1.02	1.02
Condenser (USD*10^3)	118.5	118.5	118.5	114.9	114.9	114.9	115.6	115.6	115.6
Re-boiler (USD*10^3)	782.4	782.4	782.4	799.6	799.6	799.6	817.4	816.9	817.4
Column shell (USD*10^3)	304.8	380.8	455.9	289.2	359.3	428.6	307.8	382.9	457.2
Plates (USD*10^3)	48.2	64.3	80.3	49.1	65.4	81.8	49.1	65.5	81.9
Total equipment(USD*10^3)	1.254	1.346	1.437	1.253	1.339	1.425	1.290	1.381	1.472
Equipment annual cost(10)(USD*10^3)	125.4	134.6	143.7	125.3	133.9	142.5	129.0	138.1	147.2
Steam +water (USD*10^3)	231.4	231.4	231.4	236.4	236.3	236.4	241.5	241.4	241.5
TAC-2C- (USD*10^3)	365.8	366.0	375.1	361.7	370.2	378.9	370.5	379.5	388.7
TAC-pond-1 (USD*10^3)							366.8	374.8	383.7
TAC-pond-2 (USD*10^3)							368.1	376.4	385.5

Table 7: Summary of performance indices for the three distillation columns.

Variable reflux	*N*-30	*N*-40	*N*-50
Reflux ratio (*R*) for B			
T	1.6 to 31.74	1.60 to 31.63	1.60 to 31.63
ETB	2.06 to 36.06	2.06 to 36.06	2.06 to 35.56
O-Xi	6.20-210.03	6.27 to 99.00	6.27 to 87.73
	210.03 to 443.30	99.00 to 183.45	87.73 to 134.01
CAP (kmol/h)	10.48	13.05	13.97
TAC (USD)	370 500	379 600	388 700
AP (USD)	2 732 025	3 518 527	3 630 583
Constant reflux	*N30-ABE*	*N40-CDE*	*N50-CDE*
	R=2, 5, 20	*R*=10, 15, 20	*R*=10, 15, 20
CAP (kmol/h)	9.29	7.38	7.35
TAC (USD)	370 500	379 600	388 700
AP (USD)	2 727 847	1 860 731	1 852 031
Constant reflux	*N30-ABC*	*N40-ABC*	*N50-ABC*
	R=2, 5, 10	*R*=2, 5, 10	*R*=2, 5, 10
CAP (kmol/h)	12.01	12.41	12.94
TAC (USD)	370 500	379 600	388 700
AP (USD)	3 299 318	3 419 916	3 582 214

ANALYSIS

The assumption of perfect separation simplifies calculations and makes comparison easy. In a real operation the second separation would begin with a feed with 1 or 2 kmole of benzene (B) and about 33 kmole of T, ETB and O-Xy. In the second separation, B will go to the first distillate, that will be rich in T and will have a little ETB. The probable mole fractions of the first distillate at the second separation could be $x_{D,B}\approx 0.02$, $x_{D,T}\approx 0.97$, $x_{D,B}\approx 0.01$, $x_{D,B}\approx 0.00$. Similar phenomena will happen for the third separation.

Luyben's capacity factor indicates that the best option is the 50 stage column, providing 13.88 kmol of product per hour, almost 30% more than the 30 stage column.

Total annual cost indicates that the best option is the 30 stage distillation column because it only needs to spend 370 500 USD instead of 388 700 USD for the 50 stage batch distillation column.

The original two quality indices used do not indicate the same option, so the third quality index (annual profit) was used. This one says that the 50 stage distillation column is the best option because it provides 3 630 583 USD, almost 33% more than the 30 stage distillation column, but only 3% more than the 40 stage batch distillation column.

Comparing the separation benzene–toluene performed first, the relative volatility between the key components is 2.39, while the value for the second separation toluene – ethyl-benzene is 2.15, and the relative volatility for the third separation ethylbenzene-o-xylne is 1.3. Comparing Tables 3 to 5 it is observed that the separation of ethylbenzene and o-xylene is where there are big differences are when comparing the columns with 30, 40 and 50 theoretical stages.

Table 6 provide information that compare the three separations: benzene (B), toluene (T), and ethylbenzene (ETB) from several perspectives. It shows that the column diameter is about the same. This is because the vapor flow rate is constant (100 kmol/h) and physical properties are not very different.

Qc and *Qr* stay close in values because the ratio *L/V* does not change much because setting the boilup of vapor stream inside the distillation column to a fixed value (100 kmol/h) and using this value for all the columns and runs tested provided homogeneous and almost standard parameters because of the assumption of constant vapor and liquid flow rate inside the column. The reboiler heat duty required for each run is proportional to *V*. The heat duty required at the condenser is also proportional to *V*, as is the diameter of the column.

Because of the variable reflux policy, all benzene, toluene, and ethyl-benzene as product get the desired purity. There is only a small fraction of about 2 kmol of o-xylene that is stored as off cut sub-product.

Comparison with operation at constant reflux. Narvaez *et al.* (2013) presented the same four component separation in a batch distillation column, but working at constant reflux. The reflux ratios used were: 2.0 represented by A, 5.0 represented by B, 10.0 represented by C, 15.0 represented by D, and 20.0 represented by E. A recommended sequence was a 30 stage batch distillation column to obtain benzene working at a reflux ratio of 2.0 (A); toluene separation at a reflux ratio of 5.0 (B); ethyl-benzene and ortho-xylene separation is performed at reflux ratio of 20.0 (E). This run was named N30ABE because it used 30 theoretical stages (N30) and reflux ratios of 2.0 (A) for the first separation (four components), 5.0 (B) for the three component separation, and 20.0 (E) for the two component separation.

Good sequences are also N40-CDE and N50-CDE. Chemical engineering experience in distillation also recommends the runs N30-ABC, N40-ABC and N50-ABC because they use low reflux, probably close to the minimum. Table 7 present the basic data for all the runs. Globally the runs increment reflux ratio and time to compensate for the difficulty in separation due to the lowering of relative volatility from 2.39, 2.15, and 1.3.

CONCLUSIONS

From the three batch distillation operations, the two component separation was the more difficult because it has the lowest relative volatility of 1.3 compared with 2.15 for the three component separation and 2.39 for the four component separation. The two component separation had 25 as the minimum number of theoretical stages and is a kind of limiting factor. Although 10 and 20 theoretical stages may be applied for the three and four component separations, for the last separation only the batch distillation columns with 30, 40, and 50 theoretical stages are able to perform the three separations needed.

Of the three performance indices used, annual profit was the best. Luyben´s capacity factor for variable reflux chose 50 stages as the best, but for constant reflux some times chose 30 stages as the best. Total annual cost (*TAC*) always choses the 30 stage column as the one with lowest TAC, but the annual profit always selects the combination with the highest annual profits.

Operating at variable reflux, the highest annual profit is given by 50 stages, followed by the 40 stages and last the 30 stages column.

Operating at constant reflux requires less operating time and more runs may be performed in the annual operating time. With this operation procedure of constant reflux, the annual profit for the 30 stage column was higher than operating at variable reflux; however, variable reflux operations using 40 and 50 stages columns were 2.8 and 21.4% higher, respectively, which makes them better choices in annual profit than their counterparts operating at constant reflux.

The highest annual profit was provided by the 50 stage batch distillation column operating at variable reflux with a total of $ 3 630 583 USD by year.

NOMENCLATURE

A_c	Area of column (m^2)
A_d	Area of down-comer (m^2)
$\mathrm{A_C}$	Area of condenser (m^2)
AP	Annual profit (USD)
A_R	Area of re-boiler (m^2)
C_i	Cost of product for the i^{th} fraction (USD/mole)
C_{feed}	Unit cost of feed (USD/mole)
C_1	Amortized incremental unit investment cost (USD/(m^2-stage-year))
C_2	Amortized incremental unit investment cost-heat exchanger (USD/(m^2-year))
$\mathrm{C_3}$	Cost for steam and coolant for 1 kg of distillate (USD/kg)
$C_{capital}$	Capital cost (USD)
$C_{operation}$	Operational cost (USD)
CAP	Luyben's capacity factor (kmol/h, or kg/h)
$C_{P\text{-}CW}$	Heat capacity of cooling water (J/kg-K)
D	Distillate flow rate (kmol/h)
D_{col}	Diameter of column (m)
$D1$	Accumulated distillate (kmol)
E	Energy (Joule)
f	fraction (dimensionless)
Ga	Allowable vapor velocity (kg/h/m^2)
Gb	Vapor handling capacity (kg/h/m^2)
H	Enthalpy (Joule/kmol)
H_{feed}	Amount of feed (mole)
H_i	Amount of accumulated product I (mole)
L	Liquid flow (kmol/h)
K_1	Guthrie's correlation coefficient for shell cost
K_2	Guthrie's correlation coefficient for exchanger cost
K_3	Guthrie's correlation coefficient for utility cost
m_{CW}	Flow rate of cooling water (kg/s)
M_V	Molecular mass of vapor (kg/kmol)
m_{steam}	Flow rate of steam (kg/s)
N	Number of theoretical stages (dimensionless)

NB	Number of batches per year (dimensionless)
N_C	Number of components (dimensionless)
N_{min}	Minimum number of theoretical stages (dimensionless)
P	Product (kmol or kg)
P	Profit (USD)
q	Index for thermal condition of feed (0.0 for saturated vapor, 1.0 for saturated liquid)
Q_C	Heat at condenser (J/h)
Q_R	Heat at reboiler (J/h)
R	Reflux ratio (L/D) (dimensionless)
R_{min}	Minimum reflux ratio (dimensionless)
t	Time (h)
t_E	Time of equilibration (h)
t_F	Time of finalization (h)
t_P	Time of process (h)
T	Temperature (K or °C)
TAC	Total Annual Cost (U. S. Dollar)
T_{CW}	Temperature of cooling water (K)
T_R	Temperature of heating steam (K)
U	Global heat coefficient (J/s-m^2-K)
U_f	Flooding velocity of vapor (m/s)
V	Boil-up vapor (kmol/h)
x	Molar fraction on liquid (dimensionless)
y	Molar fraction in vapor (dimensionless)
W	Liquid at bottom of column (kmol)
W_{min}	Minimum work (Joules)

Greek Letters

α	Relative volatility (Ki/Kj) (dimensionless)
ΔH	Heat of condensation or vaporization (J/kg-K)
θ	Relative volatility at minimum reflux (dimensionless)
ε_{loss}	Lost work (Joules)
η	Thermodynamic efficiency (dimensionless)
μ	Viscosity (kg/(m-s))
ρ	Density (kg/m^3)

Subscripts

$acum$	Acumulated
C	Condenser
D	Distillate
F	Feed
f	Final, flooding
i	Component
R	Re-boiler
$r\&r$	Removal and recharging
t	Total
V	Vapor
W	Bottom
0	Initial

Superscripts

k	Time index

REFERENCES

Al-Twain, M. S., Luyben, W. L., Multicomponent batch distillation. 3. Shortcut design of batch distillation columns. Ind. Eng. Chem. Res., 30, 507-516 (1991).

Asiedu, N. Y., Hildebrandt, D., Glasser, D., Batch Distillation Targets for Minimum Energy Consumption. Ind. Eng. Chem. Res., 53, 2751-2757 (2014).

Barolo, M., Guarise, G. B., Batch distillation of multi-component system with constant relative volatilities. Trans. IChemE, 74-A, 863-871 (1996).

Bernot, C., Doherty, M. F., Malone, M. F., Design and operating targets for nonideal multicomponent batch distillation. Ind. Eng. Chem. Res., 32, 293-301 (1993).

Boston, J. F., Britt, H. I., Jirapongphan, S., Shah, V. B., An Advanced System for the Simulation of Batch Distillation Operation. In: Foundations of Computer –Aided Chemical Process Design, vol. II, AIChE, Mah, R. H. S., Seider, W. D., (Eds.), 203-237 (1981).

Distefano, G. P., Mathematical modeling and numerical integration of multicomponent batch distillation equations. AIChE J., 14, 190-199 (1968).

Diwekar, U. M., Simulation, design, and optimization of multicomponent batch distillation columns. Ph. D. Dissertation, Indian Institute of Technology, Bombay, India (1988).

Diwekar, U. M., Madhavan, K. P., Multicomponent batch distillation column design. Ind. Eng. Chem. Res., 30, 713-721 (1991).

Diwekar, U. M., Madhavan, K. P., Swanky, R. E., Optimization of multicomponent batch distillation column. Ind. & Eng. Chem. Res., 28, 1011-1017 (1989).

Doherty M. F. and Malone, M. F., Conceptual Design of Distillation Systems. McGraw Hill (2001).

Eduljee, H. E., Equations replace Gilliland Plot. Hydrocarbon Processing, 56(9), 120-122 (1975).

Fair, J. R., How to predict sieve tray entrainment and flooding. Petro/Chem. Eng., 33, 45-62 (1961).

Fenske, M. R., Fractionation of straight-run Pennsylvania gasoline. Ind. Eng. Chem., 24, 482-485 (1932).

Galindez, G., Fredenslund, A., Simulation of multicomponent batch distillation processes. Comput. Chem. Eng., 12, 281-288 (1988).

Garcia, A. N., Loria, J. C. Z., Marin, A. R., Quiroz, A. V. C., Simple multicomponent batch distillation procedure with a variable reflux policy. Braz. J. Chem. Eng., 31(2), 531-542 (2014).

Gilliland, E. R., Multicomponent Rectification. Ind. Eng. Chem., 32, 1101-1106, (1940).

Gmehling, J., Onken, U., Arlt, W., Vapor-Liquid Equilibrium Data Collection, Aromatic hydrocarbons. Vol 1, Part 7, 7a, 7b, DECHEMA (2000).

Jana, A. K., Nawaz Khan, M. M., Maiti, D., Improving energy efficiency and cost-effectiveness of batch distillation for separating wide boiling constituents. II: Internal versus external heat integration. Chem. Eng. & Proc., 72, 122-129 (2013).

Ki-Joe, K., Diwekar, U. M., Comparing batch column configurations: Parametric study involving multiple objectives. AIChE J., 46, 2475-2488 (2000).

Low, K. H., Sorensen, E., Simultaneous optimal design and operation of multi-vessel batch distillation. AIChE J., 49, 2564-2576 (2003).

Luyben, W. L., Some practical aspects of optimal batch distillation design. Ind. Eng. Chem. Process Des. Dev., 10, 54-59 (1971).

Meadows, E. L., Multicomponent batch-distillation calculations on a digital computer. Chem. Eng. Symp. Ser., 59, 46-48 (1963).

Molokanov, Y. K., Koroblina, T. P., Mazurina, N. I. and Nikiforov, G. A., Approximated method for calculating the basic parameters of multicomponent fractionation. Int. Chem. Eng., 12 (2), 209-212 (1972).

Narvaez-Garcia, A., Zavala-Loria, J. C., Vilchiz-Bravo, L. E. and Rocha-Uribe, J. A., Design of batch distillation columns using short-cut method at constant reflux. Journal of Engineering, Vol. 14, ID 685969, 14 pages, http://dx.doi.org/10.1155/2013/685969 (2013).

Nawaz Khan, M., Bhaskar Babu, G. U., Jana, A. K., Improving energy efficiency and cost-effectiveness of batch distillation for separating wide boiling constituents. 1. Vapor recompression column. Ind. & Eng. Chem. Res. 51, 15413-15422 (2012).

Quintero-Marmot, E., Luyben, W. L., Multicomponent batch distillation. 2. Comparison of alternatives slop handling and operating strategies. Ind. Eng. Chem. Res., 29, 1915-1921 (1990).

Salomone, H. E., Chiotti, O. J., Iribarren, O. A., Short-cut design procedure for batch distillations. Ind. Eng. Chem. Res., 36, 130-136 (1997).

Santos, J., Jin-Kuk, K., Smith, R., Operational optimization of batch distillation systems. Ind. Eng. Chem. Res., 51, 5749-5761 (2012).

Scanavini, H. F. A., Ceriani, R., Meireles, A. J. A., Cachaca distillation investigated on the basis of model systems. Braz. J. Chem. Eng., 29(2) 429-440 (2012).

Seader, J. D. and Henley E. J., Separation Process Principles. 2nd Ed., John Wiley & Sons (2006).

Sundaram, S., Evans, L. B., Shortcut procedure for simulating batch distillation operations. Ind. Eng. Chem. Res., 32, 511-518 (1993).

Thurton R., Bailie, R. C., Whiting, W. B., Shaeiwitz, J. A., Analysis, Synthesis, and Design of Chemical Processes. Prentice Hall (2003).

Underwood, A. J. V., Theory and practice of testing stills. Trans. Inst. Chem. Eng., 10, 112-158 (1932).

Treybal, R. E., Mass Transfer Operation. 3th Ed., Mc Graw Hill (1980).

Underwood, A, J. V., Fractionation distillation of multi-component mixtures. Calculation of minimum reflux ratio. J. Inst. Petrol., 32, 614-626 (1946).