

APPLICATION OF CONSTANT REFLUX BATCH DISTILLATION FOR ARTESAN CRUDE OIL REFINING

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Abstract

Crude oil which has dominated global energy supply in the last two and half centuries has developed the science of distillation being the basis of its refining process. Continuous distillation is the preferred mode of distillation operation given its inherent advantages over batch distillation, however, the latter is gaining acceptance and even prominence in some parts of the world for several reasons. The present work revisits batch distillation design with the aim of using it for the refining of crude oil in small quantities. A constant reflux batch distillation column was designed in this work to refine 100 litres of Bonnylight crude oil of Nigeria. The column gave more than 70% product recovery which is quite high for such processes and also feasible physical column dimensions that allow for easy construction. Finally, results of model computations and software simulation using ASPEN HYSYS process simulator gave a mean deviation of 17.2 which though is significant can be improved upon to increase the predictive accuracy of the model.

Keywords: Reflux, Batch, Distillation, Artesian, Refining

1. Introduction

Industrial processes for large scale production or manufacturing are usually carried out in continuous processes. However, batch operations have the advantage of easier and more simplified designs as well as lower cost of construction and operation.

Distillation as a means of separating a mixture of different liquids has been practised and studied for many centuries. But efforts at developing more modern ways of distillation got to the peak after the discovery of crude oil in 1856 in Pennsylvania in the United States of America [1]. Batch processes for crude oil refining in parts of Africa and Asia have become quite common due to supply gaps that exist in refined petroleum products. Locals and even national governments have resorted to ancient crude distillation processes based on batch operations to refine petroleum and fill these supply gaps [2].

Two basic forms of batch distillation operations are: the constant reflux process (which gives a changing product composition), as is the case with crude oil refining, and the variable reflux process when a constant product composition is desired [3], [4].

For distillation of crude oil in small units which has become attractive lately as mentioned earlier, batch operations with constant reflux of distillate presents a ready and viable

alternative to the conventional continuous distillation processes that have dominated the industry since the late twentieth and twenty first centuries [5]. Objectives of the work are;

- i. Apply standard design equations of constant reflux batch distillation [6] to separate different components of crude oil.
- ii. Determine products specification (i.e. the different types of distillate) for the process.
- iii. Determine physical dimensions of the column and reboiler such as column height, column diameter, tray dimensions etc based on the physicochemical as well as thermodynamic properties of the feed and products.
- iv. Determine column efficiency with parameters such as weeping and flooding [7] in the column.
- v. Fabricate pilot unit for experimental validation of design results.

2. Development of model

2.1 Assumptions of the Model

Constant reflux batch distillation equations in the present work are developed based on the following assumptions:

- i. The crude oil to be separated is assumed to be an ideal mixture. This assumption implies that it

obeys both Dalton’s law of partial pressure and Raoult’s law.

- ii. Stages of the distillation process are also assumed to be ideal which means there is vapour – liquid equilibrium on each stage [8].
- iii. There is equimolar overflow [9] in the column (i.e. vapour holdup in column is constant). The foregoing implies there is negligible heat effects in the process thus model does not required a heat balance equation.

2.2 Products Specification

For successful modelling of the distillation process of any mixture, composition of the mixture must be known and the expected products of separation specified [10]. Crude oil is a multicomponent liquid with a wide range of separable fractions. However, for the purpose of the present work, the desired products of distillation are Naphtha, Kerosene, Diesel and the Residue whose properties are given in Table 1 as adopted from assay data of Bonnylight Crude Oil from the library of ASPEN Technology [11].

Table 1
Bonnylight CrudeOil Cut Yields (2011)

Product Cuts	Temp (°C)	% Vol	% Mole	% Mass
Off Gases	IBP – 70	6.32	13.00	4.6
Naphtha	70 – 221.1	29.03	41.67	26.72
Kerosene	221.1 – 371.1	37.00	32.19	38.51
Diesel	371.1 – 537.8	19.50	10.34	21.27
Residue	537.8 – FBP	7.40	2.80	8.83

2.3 Material Balance

Figure 1 shows the configuration of a batch distillation unit comprising of the Reboiler, Column or Still and a Condenser. Separation of components is effected by stages in the column (which are sieve trays in this case) and the condenser.

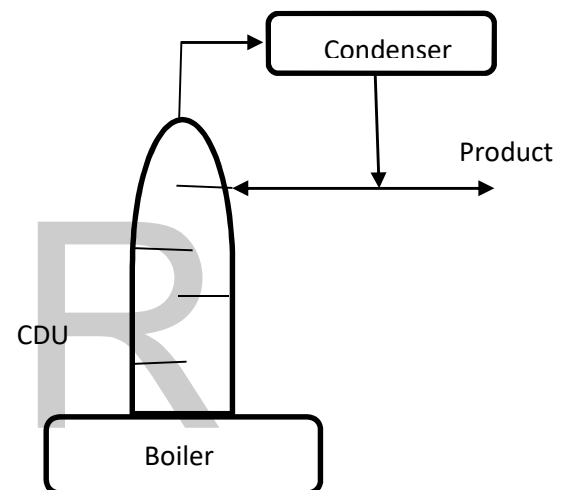


Fig. 1. Schematic Diagram of a Batch Distillation Unit

Overall component balance for the process where y moles of vapour of component (i) in the still and x moles of liquid of component (i) in reboiler are generated from xf moles of feed was given by Lord Rayleigh [12] as;

$$y^i dF = d(x_B^i F) \tag{1}$$

Expanding (1) yields;

$$y^i dF = x_B^i dF + F dx_B^i \tag{2}$$

Integrating (2) gives;

$$\int_F^L \frac{dF}{F} = \int_{x_F^i}^{x_B^i} \frac{dx_B^i}{y^i - x_B^i} \quad (3)$$

The amount of liquid left in still (or heater) at the end of a batch L, is given by integration of (3). The right hand side of Equation (3) is integrated graphically using the Simpson's rule by a plot of $\frac{1}{y^i - x_B^i}$ against dx_B^i shown in Figure 4. Relationship between y^i and x_B^i is given by the relative volatility of components [13] in the mixture (in this case under conditions of equimolar overflow) as;

$$y^i = \frac{\alpha x_B^i}{(\alpha - 1)x_B^i + 1} \quad (4)$$

But relative volatility α is also given by;

$$\alpha = \frac{P_1^o}{P_2^o}; \quad i = 1, 2 \quad (5)$$

For determination of other process parameters such as reflux ratio (R) and distillate composition x_D etc, material and component balance is taken over each stage (n) of the process respectively and shown.

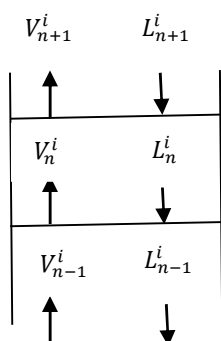


Fig. 2. Diagram of Material and Component Balance per Stage

$$V_n^i + L_n^i = V_{n+1}^i + L_{n-1}^i \quad (6)$$

And

$$y_n^i V_n^i + x_n^i L_n^i = y_{n+1}^i V_{n+1}^i + x_{n-1}^i L_{n-1}^i \quad (7)$$

But taking a component balance over the condenser gives;

$$y_n^i V_n^i = x_n^i L_n^i + D x_D^i \quad (8)$$

Substituting (8) in (7) yields;

$$y_n^i = \frac{L_{n-1}^i}{V_n^i} x_{n-1}^i + \frac{D}{V_n^i} x_D^i \quad (9)$$

The assumption of constant vapour and liquid holdup gives;

$$V_n^i = V_{n+1}^i \quad (10)$$

And

$$L_n^i = L_{n-1}^i \quad (11)$$

Equation (9) thus reduces to;

$$y_n^i = \frac{L_n^i}{V_n^i} x_n^i + \frac{D}{V_n^i} x_D^i \quad (12)$$

Also, the reflux of distillate back into the column through the top tray to enhance separation is given as the ratio (R) of liquid refluxed to the distillate drawn;

$$R = \frac{L_{n-1}^i}{D} \quad (13)$$

Therefore substitution of (13) in (12) gives the operating line equation in terms of reflux ratio as;

$$y_n^i = \frac{R}{R+1} x_n^i + \frac{1}{R+1} x_D^i \quad (14)$$

A plot of y_n^i against x_n^i in (14) gives slope and intercept respectively as;

$$\text{Slope} = \frac{R}{R + 1} \quad (15)$$

And

$$y_{intercept}^i = \frac{x_D^i}{R + 1} \quad (16)$$

Assuming the condenser to be a total condenser, then without reflux of distillate, the rate of vaporization of the volatile component is equal to the rate of change of composition in the still, thus;

$$dF = dD \quad (17)$$

and

$$x_D^i dD = d(x_B^i dF) \quad (18)$$

The general form of the Rayleigh's equation given in (3) thus becomes;

$$\int_F^L \frac{dF}{F} = \int_{x_F^i}^{x_B^i} \frac{dx_B^i}{x_D^i - x_B^i} \quad (20)$$

To maintain a constantly changing composition of distillate as required by the present separation (which has different fractional compositions), a constant reflux ratio is maintained in the McCabe – Thiele graph [3] at different distillate compositions having the same number of theoretical trays as shown in Figure 4.

2.4 Reboiler Parameters

i. Reboiler Heat Duty

As shown earlier in Figure 1, the unit is comprised of the reboiler, column (or still) and the condenser. Reflux in the

column is made possible by the heat provided in the reboiler.

Therefore, the total heat supplied to the reboiler is used essentially to provide reflux for the column given as [7]:

$$Q_{Reboiler} = \int_0^D \phi R dD = \phi R D \quad (21)$$

Reboiler and Column physical dimensions were determined using standard correlations but suffice to note that the column efficiency was determined in this work using the O'Connell's correlation [4] given in equation (22) while the number of actual trays is given by equation (23).

$$E_c = 51 - 32.5 [\log(\mu_{avg} \alpha_{avg})] \quad (22)$$

$$N_{Actual} = \frac{N_{Theoretical}}{E_c} \quad (23)$$

3. Results and Discussion

Design of the Constant Reflux Batch Crude Oil Distillation

Unit was based on Crude Oil assay data of Bonnylight Crude Oil from ASPEN HYSYS library (2011). The design was based on Crude Oil throughput of 100 litres per hour. The vapour - liquid equilibrium data of the system is given in Table 2 and Figure 3. Relative volatility of components α in the mixture was determined by application of equation (5) using vapour pressures of the adjacent fractions of naphtha and kerosene as given [14].

Table 2
Computational Parametre for Vapour – Liquid Equilibrium

x_B^i	αx_B^i	$\frac{(\alpha - 1)}{x_B^i + 1}$	y^i
0.1	1.6	2.5	0.65
0.2	3.2	4	0.80
0.3	4.8	5.5	0.87
0.4	6.4	7	0.91
0.5	8.0	8.5	0.94
0.6	9.6	10	0.96
0.7	11.0	11.5	0.97
0.8	12.8	13	0.98
0.9	14.4	14.5	0.99
1.0	16	16	1.0
$\alpha = 16$			

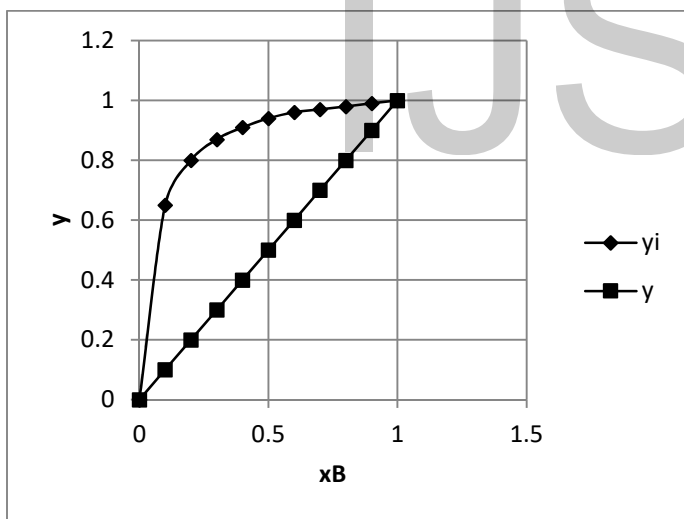


Fig. 3. Vapour - Liquid Equilibrium of the Process

From the McCabe - Thiele's graphs shown in Figure 4 to Figure 8, it is seen that the profiles of y_2, y_3, y_4 and y_5 are at a constant reflux ratio of 2.33 and the column requires a minimum of five theoretical trays to achieve the desired degree of separation. Efficiency of the distillation column

was determined as 54.2 % using the O' Connell's correlation as given in Equation (22). The foregoing therefore implies that nine actual trays are required to achieve the desire degree of separation in the present system.

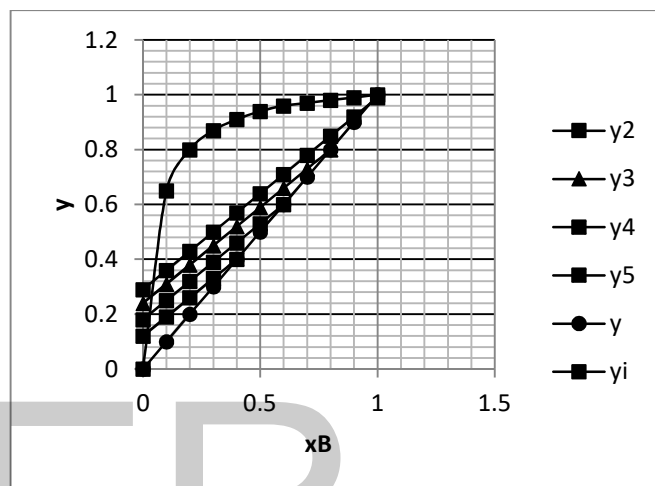


Fig. 4. McCabe - Thiele Graph with Constant Reflux Ratio

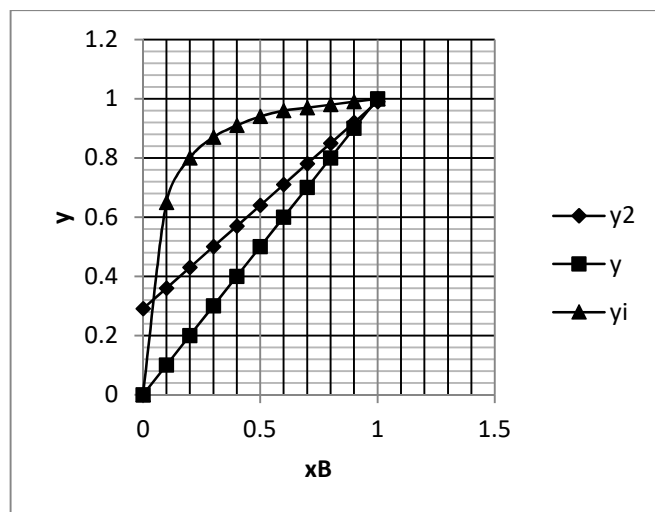


Fig. 5. McCabe - Thiele Graph with $x_D = 0.95$

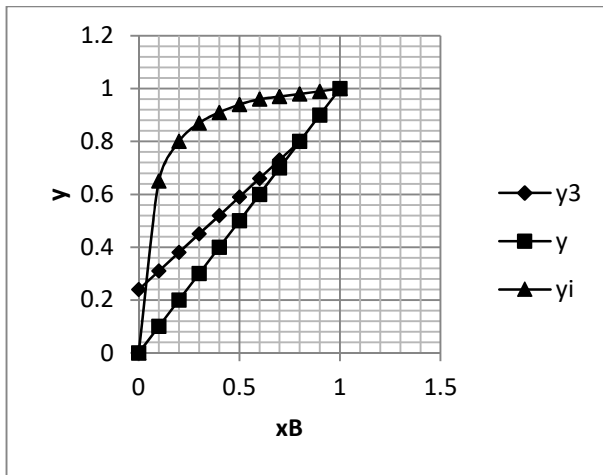


Fig. 6. McCabe - Thiele Graph with $x_D = 0.8$

recovered from the Feed is given as 47.9%, with individual component recoveries shown in Table 4.

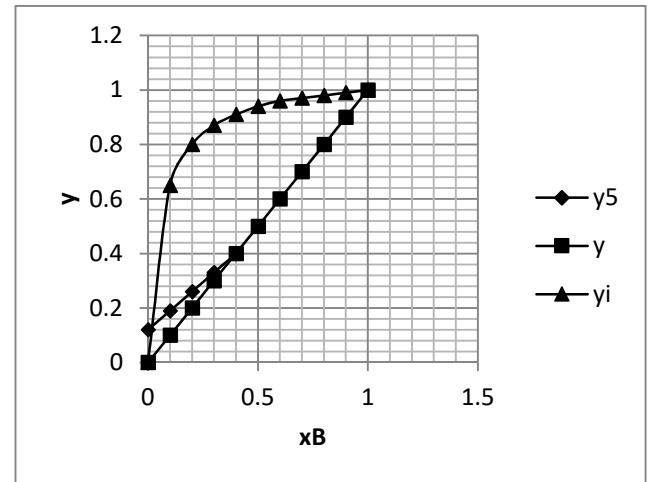


Fig. 8. McCabe - Thiele Graph with $x_D = 0.4$

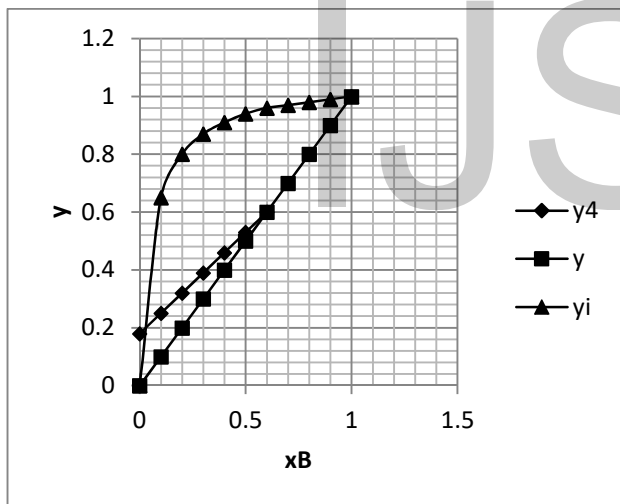


Fig. 7. McCabe - Thiele Graph with $x_D = 0.6$

Detailed procedure of the McCabe - Thiele's method for determination of the number of trays for a distillation column can be found in standard texts [3], [4]. Applying Simpson's Rule for graphical integration to Equation (20) as shown in Table 3 and Figure 9, the amount of distillate

**Table 3
Data for Graphical Integration of Design Equation (Simpson's Rule)**

x_D^i	x_B^i	$x_D^i - x_B^i$	$\frac{1}{x_D^i - x_B^i}$
0.9	0.5	0.4	2.5
0.8	0.2	0.6	1.67
0.7	0.12	0.58	1.72
0.6	0.08	0.52	1.92

The constant reflux batch distillation model developed in this work thus gives about half of the feed being recovered as product. This is a reasonably good performance for a batch distillation column where product recovery is usually quite low compared to the continuous process. There are three Off - Cuts products for the present design. Off - Cuts of the recovered products which is a fundamental feature of the batch distillation process was determined by assuming a 5% fractional contribution each of both the lower and upper

components of adjacent fractions and also shown in Table 4. Consequently, the effective amount of products recovered from the Feed in this model is 41.2 %.

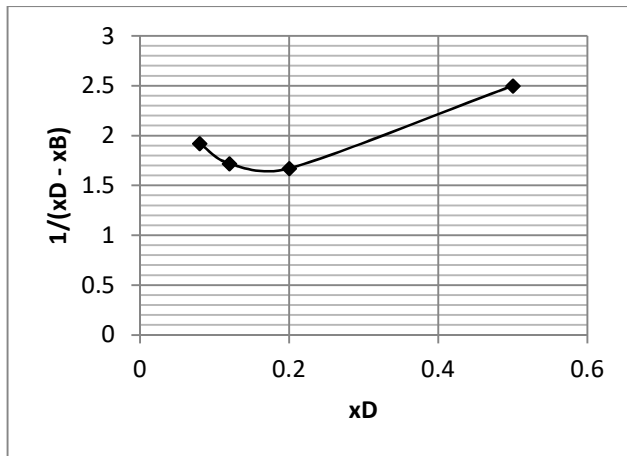


Fig. 9. Graphical Integration by Simpson's Rule

**Table 4
Percent Vaporization of Components (Adjusted Decimals)**

Product Cuts	Temp (°C)	Vol. Fraction	Distillate (D)%
Off Gases	IBP – 70	0.063	3.0
Naphtha	70 – 221.1	0.276	13.2
Off Cut - 1	-	0.033	1.6
Kerosene	221.1 – 371.1	0.333	16.0
Off Cut - 2	-	0.029	1.4
Diesel	371.1 – 537.8	0.180	8.6
Off Cut - 3	-	0.014	0.7
Residue	537.8 – FBP	0.072	3.4
Total	-	1.00	47.9

Physical dimensions of the distillation unit were also determined using the model herein developed. The batch equipment comprises of the reboiler, the Distillation Column and the Condenser. The Column and Condenser dimensions

are given in Table 5. Results of model computations and ASPEN HYSYS simulation of the process gave a mean deviation of 17.2. Finally, column characteristics and performance as defined by Column weeping and flooding gave impressive results within prescribed design limits [5].

**Table 5
Results of Column Dimensions for Model Computations and HYSYS Simulation**

Dimensions	H_c (ft)	D_c (ft)	Time (Mins.)	Reflux Ratio (R)
Model Computation	16.57	1.97	58.90	2.33
HYSYS Simulation	10.79	0.73	44.00	2.50
Deviation	5.78	1.24	14.9	0.17
Mean Deviation	17.2			

4. Conclusion

Crude Oil refining in simple and small units and equipment as alternative to the conventional plants is increasingly becoming attractive. While several reasons can be adduced for this trend, the divestment of business interest in Crude Oil by the major Oil companies globally is at the center of it all. New and emerging Crude Oil refiners are thus predisposed to revolutionize refining by redefining its scope. Local refiners in parts of the developing world have resorted to the crudest options of distillation for this purpose.

Batch distillation which is traditionally preferred for small operations thus perfectly fits into new regime of refining. Constant reflux batch distillation operation gives product composition that varies with time, thus is appropriate for distillation of Crude Oil which has varying product compositions.

The model in this work gave a total product recovery of 47.9% which is considered reasonable for the batch process. Also, the model gave physical dimensions of the batch equipment with high constructability, whence can be easily implemented. Column performance in terms of Flooding and Weeping in column was also within prescribed limits. Finally, determination of process physical dimensions by model computations and ASPEN HYSYS simulation gave a mean deviation of 17.2, though significant can be improved upon to increase the predictive accuracy of the model.

Notations

A_s – Surface Area

D – Volume of Distillate

F – Volume of Feed

L – Volume of Liquid

P_1^o – True Vapour Pressure of Naphtha

P_2^o – True Vapour Pressure of Kerosene

Q – Heat Duty of Crude Oil Boiler

t – Distillation time

T_{Hin} – Temperature of Hot fluid in

T_{Cout} – Temperature of Cold fluid out

T_{Hout} – Temperature of Hot fluid out

T_{Cin} – Temperature of Cold fluid in

U – Overall heat transfer coefficient

V – Volume of vapour

V_{Cond} – Volume of condenser vapour

x_B^i – Liquid fraction of component i in Boiler

y^i – Vapour fraction of component i from Boiler

α – Coefficient of Relative volatility

θ_m

– Long mean temperature difference of condenser fluids

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