Prediction of the Operating Conditions in a Batch Distillation Column Using a Shortcut Method

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ABSTRACT: *A shortcut procedure as quick, easy-to use method for design and simulation of multicomponent batch distillation is used to predict the operating condition of recovering xylene from solvent in an existing batch distillation column in benzol refinery. The procedure can be used to investigate the effect of the operating parameters on the operation of column for three possible modes of batch distillation operations; constant reflux, variable reflux and step wise change in the reflux ratio. It is shown that step wise change in the reflux ratio, in comparison with a fixed ratio mode, can significantly reduce the required operation time and makes the operation more economical even at high reflux ratios. Applying the procedure in a batch distillation column (National Iranian Steel Cooperation) with 35 bubble-cap trays, mixed-xylenes were produced in a desired purity of 95% with a recovery of 92.5% from a feedstock containing 40% xylenes.*

KEY WORDS: *Batch distillation, Shortcut, Multicomponent, Xylenes*

INTRODUCTION

Batch distillation is often the process of choice for separation of multicomponent mixtures containing high value chemical specialties, commonly conducted in small volumes. Due to their inherent flexibility, batch columns can generally be used for a variety of systems involving variable compositions and components. Batch distillation may become an important separation method in the recovery and reprocessing of waste solvent mixtures as environmental regulations become more strict and disposal costs become more expensive. Even though batch distillation consumes more energy than continuous distillation, it provides more flexibility and involves less capital investment [1]. The flexibility of this separation method makes it possible to adjust the operating conditions to the required purity specifications. The operation can be carried out with (1) variable reflux and constant product composition, (2) constant reflux and variable product composition and (3) cyclic operation, characterized by two modes of operation, transient total reflux and stripping. The design of a batch distillation column is much more complex in comparison with that of a continuous distillation column as it requires consideration of the unsteady-state behavior. The complexity of the problem increases with the number of components in multicomponent systems. Early dynamic models for distillation, which are restricted to small deviations from the steady-state, have been presented by Mah *et al* [2], Distefano [3] and Howard [4]. Gani *et al* [5], Gani and Cameron [6], Cameron *et al* [7], Ruiz *et al* [8] applied more complex equilibrium-stage models for the simulations of dynamic phenomena in continuously operated columns. In additiuon, Gani *et al* [9] and Gani and Ruiz [10] incorporated considerations of dynamic startup/shutdown operations and column hydraulic into an advanced equilibrium-stage model. Perhaps the most accurate rate-based model for simulation of dynamic deviations from the steady state for tray columns was developed by Kooijman [11] and modified by Polkonen *et al* [12] for

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packed columns. For the simulation of batch distillation, Galindez and Fredenslund [13] and Diwekar [14] presented quasi-static models approximating the process as a sequence of continuous operations with changing feed in each time step. Recently a completely dynamic rate-based model for the simulation of batch columns was presented by Kreul *et al* [15].

Although rigorous dynamic simulators have already reached a commercial stage, their demand in terms of computational time is very large, and therefore they are hardly useful at the decision-making steps of design or production planning of a batch distillation column. Thus, many articles deal with the development of simple models and shortcut procedures, appropriate at these decision-making steps. The shortcut methods are very efficient and can be used for preliminary design and analysis of batch columns. The main features of these methods include lower computational time, lower memory requirements, and its adaptability to design.

Some present literature reports the use of the Fenske-Underwood-Gilliland (FUG) method for continuous distillation to describe the instantaneous separation performance of batch columns. This approach was presented by Diwekar and Madhavan [16] and later on used to solve both design problems, as in Diwekar [17], and simulations, as in Sundaram and Evans [18]. This approach as well as, the shortcut methods developed by Diwekar [19] and Salnomone *et al* [20] allow for a considerable reduction in computation demand, which in turn becomes independent of the number of stages.

In the shortcut methods, the simplifying assumptions adopted to reduce computational time are constant relative volatility, constant molal overflow and zero holdup in the column.

In the present work, the modified FUG method is used to simulate an existing batch distillation column in benzol refinery unit to be used for xylene recovery from solvent and to predict the operation condition for the desired separation using step changes in reflux ratio.

The Shortcut Method

The shortcut method is based on the reasonable approximation that the batch distillation column can be considered as a continuous distillation column with changing feed. The modified Fenske-Underwood-Gilliland method is used to simulate multicomponent batch distillation column.

Variable Reflux Case

In this case, the top product composition, $x_{D,i}$ is assumed to be constant throughout the operation, the differential material balance can be written as,

$$d \ln B/F = d x_{B,i} / (x_{D,i} - x_{B,i}) \qquad i = 1,2,\ldots,n \quad (1)$$

Where, $x_{B,i}$ is the composition of the i^{th} component in the still at any time t. If component 1 is taken as the reference, then the variation of composition of any other component i with respect to component 1, is given by the equation as bellow,

$$x_{B,i}=x_{D,i}-[(x_{D,i}-x_{F,i})/(x_{D,1}-x_{F,1})] \qquad i = 2,3,\ldots..,n \quad (2)$$

Once the still compositions are known at any time, the FUG method could be applied, resulting in the following equations;

1- Fenske's equation can be used to estimate Nmin,

$$N_{min} = \ln [(x_{D,lk} \cdot x_{B,hk}) / (x_{B,lk} \cdot x_{D,hk})] / \ln [\alpha_{lk} / \alpha_{hk}] \quad (3)$$

2- Underwood equations with the feed at the boiling point, which is the case of batch distillation can be written as,

$$\sum_{i=1}^{n} \alpha_i x_{F,i} / (\alpha_i - \phi) = 0 \quad , \quad R_{min} + 1 = \sum_{i=1}^{n} (\alpha_i - \phi) \qquad (4)$$

The value of ϕ must lie between the values of the relative volatilities of the key components and is found by trial and error.

3- Gilliland equation is as follow,

$$(N - N_{min})/(N+1) = 0.75\{1-[(R-R_{min})/(R + 1)]^{0.5668}\} \quad (5)$$

Since, in this case, the $x_{D,i}$ is fixed, it follows that N_{min} and R_{min} are function of $x_{B,i}$. In order to relate the composition in the still and overhead, the following equation is used [16],

$$x_{D,i} / x_{B,i} = (\alpha_i / \alpha_1)^{c1} (x_{D,1} / x_{B,1}) \qquad i=2,3,\ldots\ldots,n \quad (6)$$

The differential material balance can be written as follows,

$$dx_{B,i} / (x_{D,i} - x_{B,i}) = dx_{B,1} / (x_{D,1} - x_{B,1}) \quad i=2,3,\ldots,n \qquad (7)$$

and the summation equation,

$$\sum_{i=1}^{n} x_{D,i} = 1 \qquad (8)$$

Integrating the material balance equation and substituting in equation 6 yields,

$$(x_{B,i})_{new} = (x_{B,i})_{old} \exp[(\Delta x_{B,1} / (x_{D,1} - x_{B,1})) ((\alpha_i / \alpha_1)^{C1} x_{D,1} / x_{B,1} - 1))] \qquad (9)$$

Combining equations 7 to 9 gives,

$$G(1) = \sum_{i=1}^{n} (\alpha_i / \alpha_1)^{C1} (x_{B,new} / x_{B,1}) x_{D,1} - 1 = 0 \qquad (10)$$

The objective is to determine the value of C_1, using the Newton-Raphson method for the solution of equation 10.

Since, the intermediate values of $x_{B,i}$ may not add up to unity, these values are transformed into normalized variables,

$$x_{BN,i} = x_{B,i} / \sum_{j=2}^{n} x_{B,j} \quad , \quad i=2,3,\ldots,n \qquad (11)$$

These normalized variables are included in equation 10.

Constant Reflux Case

To initiate the design procedure, an initial value of top product composition is assumed. Since the composition is variable in this case, an additional equation is needed to satisfy the extra degree of freedom [16], which can relate R, N, C_1 and compositions,

$$R_{min,g} / R - R_{min,u} / R = 0 \qquad (12)$$

Where $R_{min,g}$ calcuated from Gilliland correlation which can be equated with $R_{min,u}$ calculated from the Underwood equation.

Equations 10 and 12 can be solved to obtain the unknowns C_1 and $x_{D,1}$ for a given change in $x_{B,1}$. For this, the Newton-Raphson method can be utilized.

The flow chart of the design procedure for the constant reflux case and step changes in reflux ratio mode, used in this work, is shown in Fig.1. Details of the computer program developed for this work is given in Ref. 21.

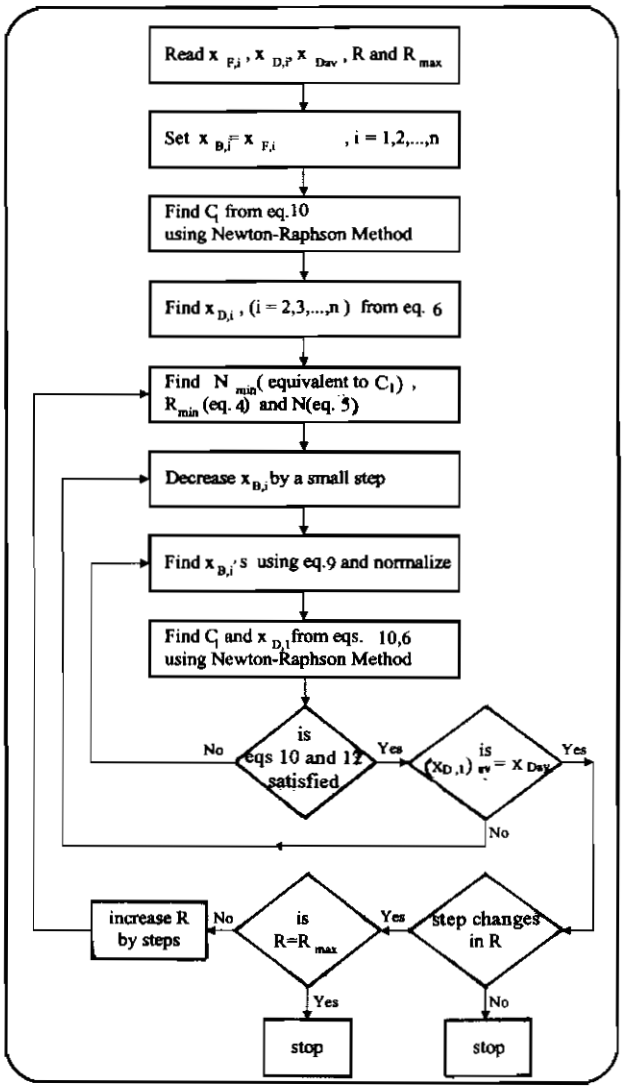

Fig.1: The flow chart for the design procedure

RESULTS AND DISCUSSION

To validate the computer program, a series of experimental reported data and the results obtained by the rigorous methods for different systems are compared with those found by the present shortcut method where a good agreement is observed [21]. Results for two examples are given in Figs. 2 and 3. In both figures, the results by the short-cut method are compared with the experimental data as well as those obtained by Stewart *et al* [22] using the rigorous method. In Fig. 2 results for a ternary system

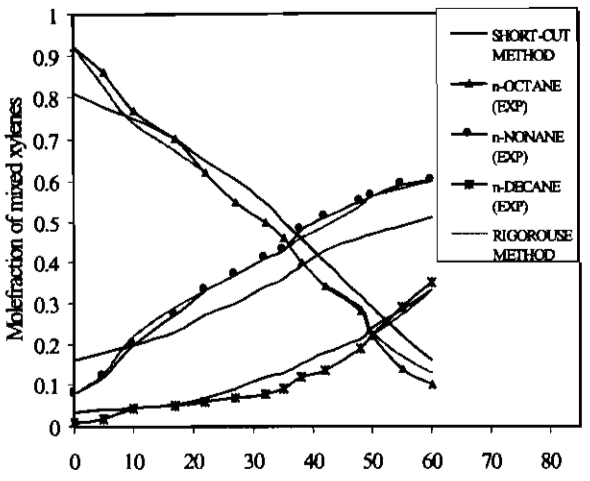

Fig.2: Experimental and theoretical distillation curves , ternary system

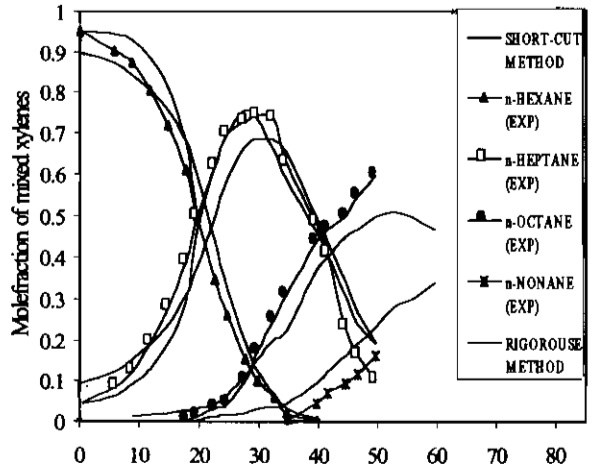

Fig.3: Experimental and theoretical distillation curves , five component system

at constant reflux ratio of 1.15 and holdup of 5.4mol% and in Fig. 3 results for a five component system at reflux ratio of 1.4 and holdup of 3.2mol% are presented. It can be seen that the shortcut method results agree well with the experimental results obtained for small holdup conditions and with those predicted by the rigorous method.

A solvent with the boiling point range of 135 to 170 °C, is a by-product fraction of benzol separation in a batch distillation column of coke and by-products complex of National Iranian Steel Cooperation. Its production rate is 330 tones per year. The GC-analysis of different samples indicate that about 30-40% of the solvent is made up of mixed xylenes. The solvent analysis is given

Table 1: Solvent analysis

Component	Mole %	Weight %
Ethylbenzene	0.5	0.463
p-xylene	16.25	15.06
m-xylene	18.7	17.35
o-xylene	4.7	4.35
Iso-propylbenzene	1.7	1.78
n-propylbenzene	2.6	2.73
1,3,5 trimethylbenzene	35.4	37.14
1,2,4 trimethylbenzene	12.5	13.12
1,2,3 trimethylbenzene	7.65	8.07

in Table 1. It was decided to predict the feasibility of recovery of this amount of xylenes (%90 Purity) in a benzol batch distillation column with 35 bubble cap trays.

The shortcut method was used to simulate this batch distillation column with 21 ideal trays (overall column efficiency is estimated 60%). In this method, the liquid holdup in the column was assumed to be zero. The overall holdup in this column represents less than 3% of the batch charged into the reboiler (60 m^3 solvent) and so it can be neglected.

α in the above eqations is considered to be the average volatility of the mixed xylenes with respect to the heaviest component in feed. It may be assumed that solvent forms an ideal solution, then α is estimated from the vapor pressure of its components using Antoine equation.

Computed prediction variations in mole fraction of mixed xylenes, as a distillate product, at different con-

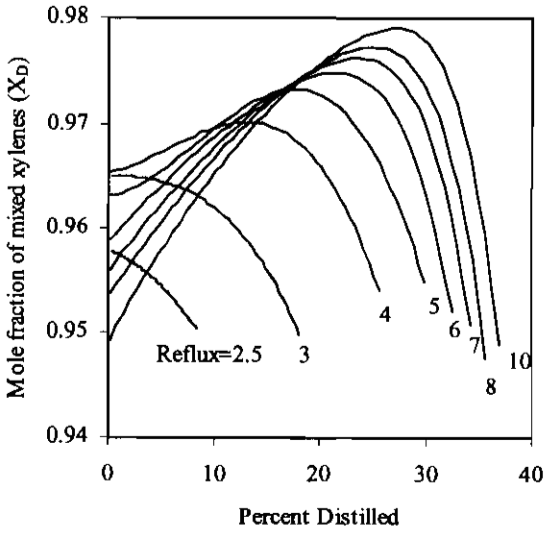

Fig.4: Predicted xylene purity against percentage of recovery at different reflux ratio

stant reflux ratio is shown in Fig. 4. At higher reflux ratios, distillate reveals that the composition of the mixed xylenes is leaner since, larger column holdup is associated with higher reflux ratio. As distillation proceeds, the high reflux ratio favours the separation resulting in richer distillate xylenes. As the reflux ratio increases, the xylene recovery will increase, so at reflux ratio of 10, the maximum recovery of 92.5% can be achieved. The changes of the distillate composition with time at different reflux ratio obtained by the shortcut procedure are given in Fig. 5, indicating the high operational time with high constant reflux ratios. Reflux ratios of higher than 6 makes the operational uneconomic, since the ratio of R/R_{min} in Fig. 6, at a period of estimated time of operation, is obtained to be greater than 1.4. The optimum reflux ratio in batch distillation operation is normally expected to be between 1.0 to 1.4 [23].

The changes in the overhead temperature with time, obtained from the product ASTM distillation curve, at different reflux ratios are presented in Fig.7. Mixed xylenes with the desired purity of 95% are practically obtained by batch distillation at atmospheric pressure when the overhead temperature varies from 138 °C initially to 144 °C.

An optimum reflux policy may require multiple reflux ratios during a single batch separation. Therefore, the response of a distillation column to the step wise changes in reflux ratio is presented in Figs. 8 to 10. When the reflux ratio in this mode of operation changes, R/Rmin ratio will change in the normal range, which is less than 1.4 even at the reflux ratios of higher than 6, as it is shown in Fig. 8. Therefore, the step wise change mode can make the column operation economical, at high reflux ratios. As it is shown in Figs. 9 and 10, the required operation time can be reduced from 30 hours when constant reflux ratio of 10 is fixed to 17 hours when the reflux ratio varies from 3 to 10 in four steps.

Based on the computer prediction, the operation of the already-mentioned batch distillation column was carried out using the step change mode operation by changing the reflux ratio. Mixed xylenes product in a purity of 95% was produced with a recovery of 93% from a feedstock containing 40% xylenes. Overhead measured temperature against time operation is compared with those predicted by model, in Fig. 11. The step changes in reflux ratio cause a significant drop in overhead temperature at the transition period. Obviously, variation in overhead temperature is a result of change in top product compositions with reflux ratio.

In Fig. 12, the bottom temperature Predicted by the model is compared with the experimental result. Though the profile obtained do not agree on an absolute scale, due to inaccuracy in the temperatue measurement and ASTM distillation prediction, it can be seen that the trend of the profiles obtained by the shortcut method is close to that obtained by the experimental result.

CONCLUSIONS

The shortcut method is used in this work to simulate a batch distillation column and to predict the operation condition for the separation of solvent. Computer results predict that the mixed-xylenes could be recovered from solvent in a desired purity of 95% with recovery of 92.5% using the benzol batch distillation column. Computer prediction also indicates that step wise changes in the reflux ratio can significantly reduce the required time of operation and obtain optimum operation parameters. Applying the theoretical prediction, the operation of an existing batch distillation column was carried out in step change mode operation by changing the reflux ratio from 3 to 10 in four steps (as shown in Figs. 11 and 12). Mixed xylenes with desired specification were produced from the solvent. More investigation should be done to find the optimum operative parameters regarding the energy consumption in this batch distillation column.

Notation

B	Bottoms product flow rate
C_1	Constant
F	Amount of feed
N	Number of plates
N_{min}	Minimum number of plates
R	Reflux ratio
R_{min}	Minimum reflux ratio
R_{max}	Maximum reflux ratio
x	Liquid mole fraction
α	Relative volatility
ϕ	Underwood equation constant

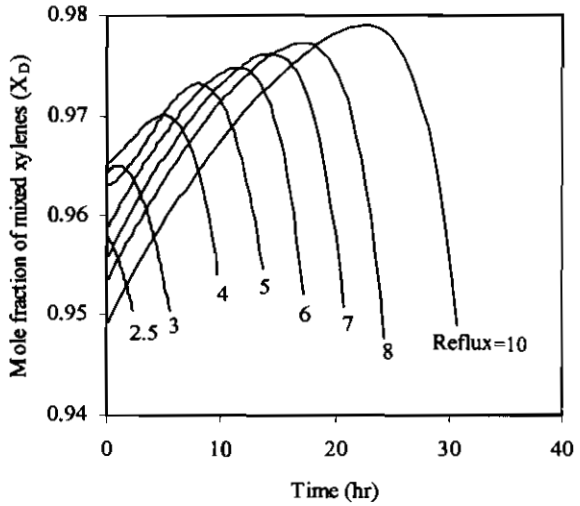

Fig.5: Predicted xylene purity against time of operation at different reflux ratio

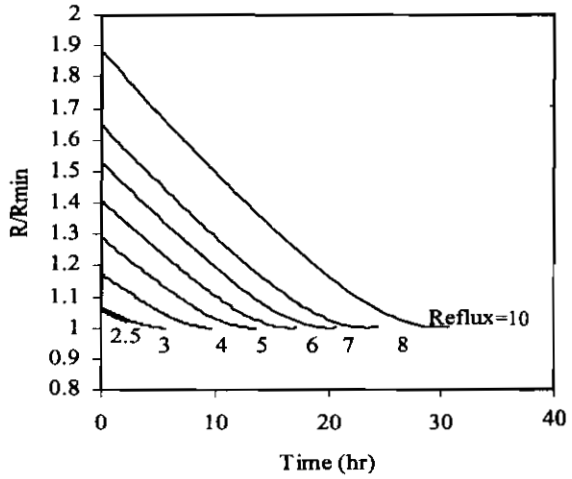

Fig.6: Effect of reflux ratio on R/Rmin ratio with time of operation

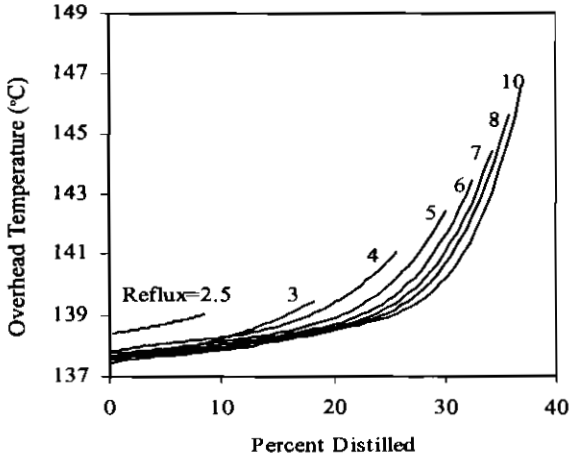

Fig.7 : Predicted overhead temperature against percentage of mixed xylene recovered

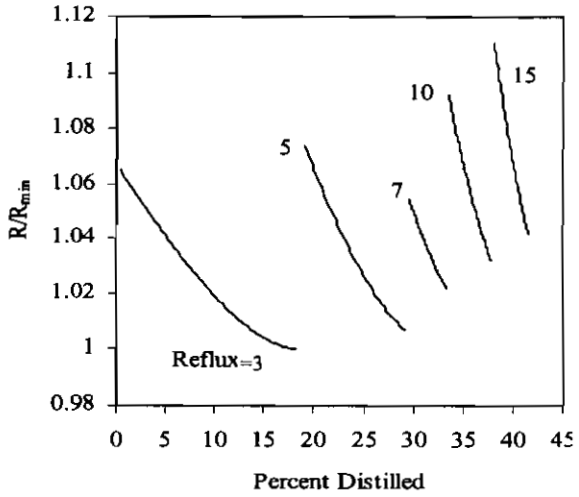

Fig.8: Effect of step wise changes in reflux ratio on R/Rmin against percentage of xylene

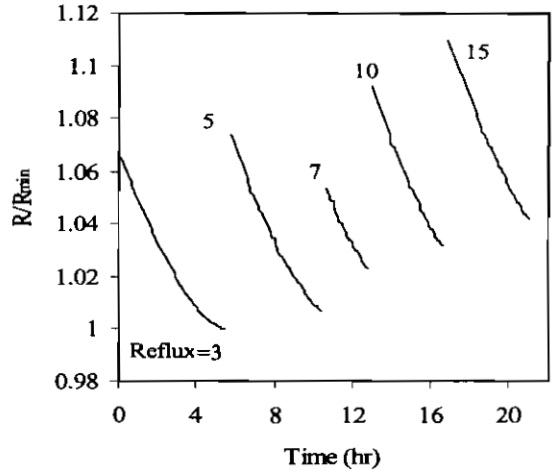

Fig.9: Effect of step wise changes in reflux ratio on R/Rmin against time of operation

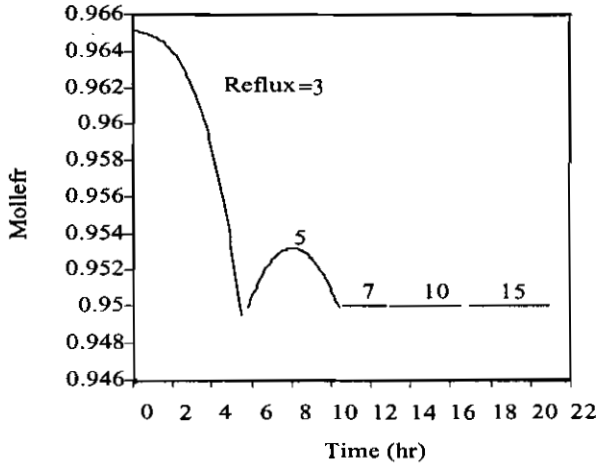

Fig.10: Predicted time of operation in step wise changes of reflux ratio mode

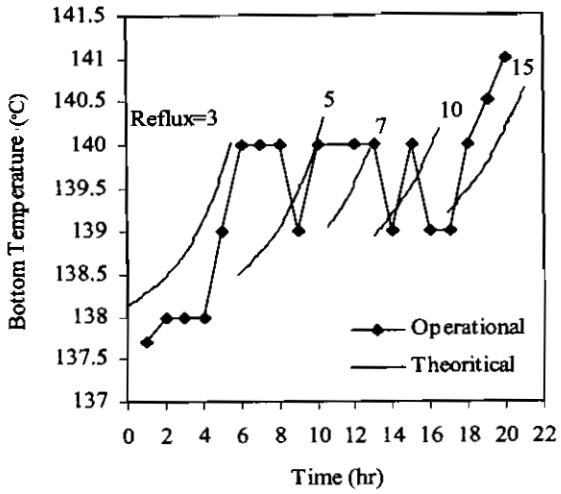

Fig.11: Comparison of operational and predicted column overhead temperature

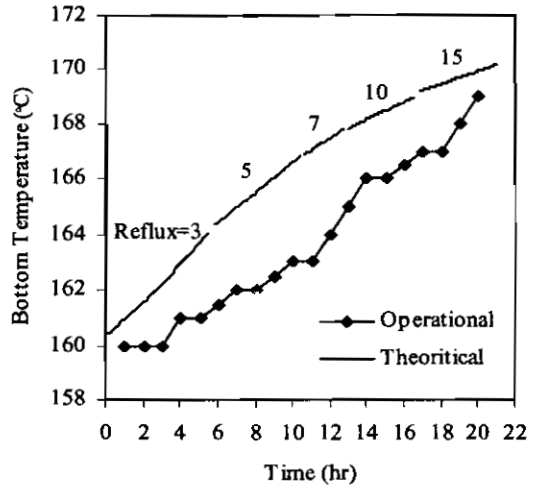

Fig.12 : Comparison of operational and predicted column bottom temperatures

Subscripts

B	Bottoms
D	Distillate
D_{av}	Specified average composition of component 1
F	Feed
hk	Heavy key component
i	Component
lk	Light key component
n	Number of components

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