

Modeling and Simulation of Distillation of a Binary Non-Reacting Mixture Using Rate-Based Approach in Packed Column under Total Reflux

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ABSTRACT: Modeling and simulation of a distillation column are very important in many chemical industries where ever it requires separation or purification of a liquid mixture into its pure components. Particularly in the context of improving efficiency, process scale-up, intensification and control it requires a validated model. In this work, both experimental results and simulation results are obtained. A batch distillation equipment which consists of a reboiler, a packed column and a top condenser is used to distill a mixture of benzene and toluene with various heat inputs to the reboiler. The distillation process in the column is modeled by an evaporation rate-based approach using Raoult's law for driving force. This particular feature is different from the stage-wise equilibrium model. The evaporation mass transfer coefficients are obtained for benzene and toluene in lab-scale apparatus. The material balance equations for the two components are solved using a finite difference method. The distillate compositions at total reflux obtained from experiments and the simulations are compared for validating of model proportionally. Further insights are obtained in regard to throughput and distillation efficiency.

KEYWORDS: Distillation; Rate-based approach; Simulation; Raoult's Law.

INTRODUCTION

Modeling of a distillation process with all its complexity is a challenging area of science and engineering [1]. Yet because of the wide application of distillation in separating and purifying organic liquids in particular there is a need for a reliable model. Also, with the contemporary application of process control for achieving a high degree of separation in distillation apparatus, it requires a model amenable for real-time computation which helps in the suitable selection of controller type and their parameters settings [2]. Keeping in view

the above design considerations and development of control strategies, the equilibrium models are often considered as deficient [3]. An alternate rate-based model requires a distributed parameter model approach [4]. In principle, the rate-based model can be implemented if the values of evaporation rate constants and the activity coefficients are available for a component liquid in a mixture at both sub and super-heated conditions relative to its boiling points. If the rate-based model is successful for steady-state prediction of a packed column performance

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then it can be extended to variants such as reactive distillation and divided column processes for efficient design of such equipment [5, 6].

Although steady state simulations can predict the distillate composition accurately, it still remains perplexing about the start of a packed bed distillation column. This is because as the reboiler contents are heated the generated vapor moves upward due to buoyancy in the voids of the packed column. As the vapor is passing upwards a portion of it condenses and trickles down along the packing. It is difficult to establish whether the packing surface area can be taken as equal to the actual interfacial area between vapor and liquid. So, there is some finite start up time required for establishment of hydro dynamically developed vapor and liquid flows in the packed column. In addition to this thermal steady state also takes some more time for composition in the distillate to reach a steady state value in the case of a batch with total reflux or a continuously fed column. In principle a rate based model also has to incorporate material and energy balance along with interfacial evaporation models [7-9]. A multi component distillation in a batch column was modelled assuming mass transfer resistance on both liquid and vapor phase with Maxwell – Stefan equations [10]. The rate based model was applied to a tray column with assumption of completely mixed liquid phase and plug flow for vapor phase. The error in prediction was found more for higher heat input rates [11]. It was found that the dynamic rate based model needs simplification in order to carry out the computation. Mass transfer coefficients were suggested to be taken as constants for reasonable accuracy in prediction [12]. The rate based model was applied to a tray column for a ternary system. It was further used in economic analysis for finding optimum reflux ratio [13]. The rate based model was applied for a ternary system in a dividing wall distillation column for a reactive system with pseudo homogeneous kinetics [14].

The gap addressed in this work is to determine the mass transfer coefficient correlations from suitable experiments and use for rate based modeling of distillation and further validation. In this work a batch distillation apparatus consisting of a reboiler, packed column and a condenser is fabricated and used. A binary system of non-reacting liquids and non-azeotropic liquids is chosen and its separation into higher light component in the distillate is studied experimentally and also modeled and simulated

using a rate based model approach. Certain assumptions are made in order to make the set of equations solvable. Lab scale experiments were conducted to determine the evaporation mass transfer coefficients of the component liquids in pure form as well as in mixture. Unlike the constant temperature in the reboiler, a constant heat input is applied in order to analyze the performance of the above distillation column with respect to distillate composition, variation of composition along the height of the column. Also the transient of temperature in the reboiler and the time for onset of distillate are obtained experimentally. The model prediction of distillate composition is validated against the experimental data which is found to be good.

EXPERIMENTAL SECTION

The chosen binary system is a mixture of benzene-toluene. It was chosen because it doesn't form azeotrope at or near atmospheric pressure. It is intended to start with an equimolar mixture of benzene and toluene and obtain pure benzene which is the lighter component at the top of the column. The apparatus is a batch packed column with reflux facility. In the first part of the experimental study the evaporation rate constants of benzene and toluene are obtained. In the second part of the experimental study the dynamics of reboiler temperature and steady state distillate composition are determined for various heat input rates to the reboiler. A digital refractometer is used to measure the refractive index of benzene-toluene mixtures and the corresponding composition of any sample is determined using a pre-calibrated chart.

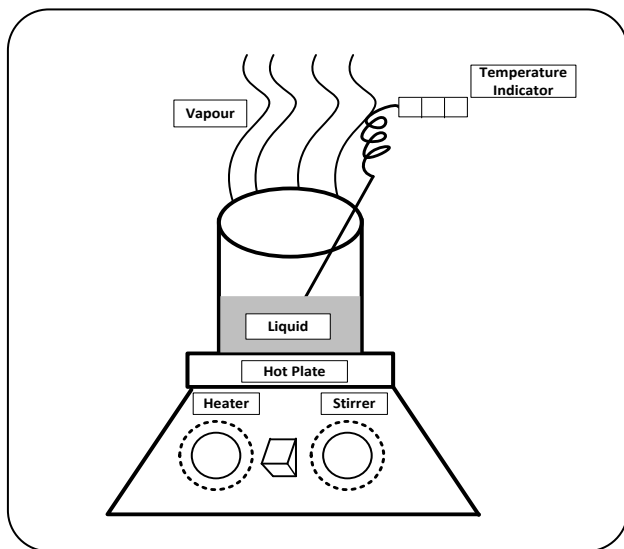
Determination of evaporation rate constants

When a mixture of two completely miscible liquids is heated at an infinitesimally small rate then the mixture reaches its bubble point or boiling point in case of pure liquids at steady state. At this bubble point the temperature remains constant for a small evaporated quantity. The procedure adopted is that a clean empty beaker of 50 mL volume filled with a mixture of benzene and toluene is kept on a hot plate until a steady state temperature is reached. The schematic of this experiment is shown in Fig. 1.

At this juncture the beaker is shifted onto a digital weighing balance with an accuracy of 1 mg, the time taken for one gram loss in weight of the beaker containing the liquid mixture is noted. This data is tabulated in Table 1.

Table 1: Experiment data pertaining to the evaporation of benzene-toluene mixture.

Benzene Volume (ml)	Toluene Volume (ml)	Bubble Point Temperature (°C)	Time taken for one gram loss at Bubble Point (s)
50	0	81.2	27.2
40	10	83.5	25.4
30	20	90.4	22.8
20	30	95.5	22.5
10	40	103.2	24.9
0	50	110.3	26

**Fig. 1: Schematic of a heated beaker used to evaluate the evaporation rate of a liquid.**

Let the components be labelled as benzene(A) and toluene(B). To calculate the evaporation mass transfer coefficients of benzene (K_A) toluene (K_B) the following equations are used similar to the evaporation of pure liquids [15].

$$\text{Evaporation flux of benzene (A)} = \quad (1a)$$

$$K_A (x_A \cdot p_A^{\text{sat}} - y_A \cdot p_t)$$

$$\text{Evaporation flux of toluene (B)} = \quad (1b)$$

$$K_B (x_B \cdot p_B^{\text{sat}} - y_B \cdot p_t)$$

Where,

x_A = Ismole fraction of component 'A' in liquid phase

y_A = Ismole fraction of component 'A' in vapor phase

K_A = Ismass transfer coefficient of component 'A', $\text{gmol/m}^2 \cdot \text{atm} \cdot \text{s}$

p_A^{sat} = Issaturated vapor pressure of component 'A', atm

x_B = Ismole fraction of component 'B' in liquid phase

y_B = Ismole fraction of component 'B' in vapor phase
 K_B = Ismass transfer coefficient of component 'B', $\text{gmol/m}^2 \cdot \text{atm} \cdot \text{s}$

p_B^{sat} = Issaturated vapor pressure of component 'B', atm

p_t = Is Total pressure or atmospheric pressure, atm

In the above equations Eq. (1a) and Eq.(1b) the mole fractions of the evaporating liquids in air are assumed negligible and that the mole fractions in the liquid are assumed same as the starting mixture compositions. The evaporation flux of A and B are evaluated using the following formula.

$$\text{Evaporation flux} = \frac{\Delta m}{\Delta t} \frac{1}{A_{c,b} MW} \quad (2)$$

Where,

Δm = Mass of evaporated liquid, g

Δt = Time of evaporation, s

$A_{c,b}$ = Cross sectional area of the beaker, m^2

MW = Molecular weight of liquid, g/gmol

By substituting the result of Eq.(2) in Eq.(1a) and Eq. (1b) the corresponding evaporation mass transfer coefficients are obtained and plotted in Fig. 2 and Fig. 3.

It can be observed from Fig. 2 that K_A exhibits three different slopes for the entire temperature range of applicability to distillation as such the fitted linear expressions for K_A vs T are as given below in Eq.3(a), Eq.3(b) and Eq. 3(c).

$$K_A = 0.0025 \times T + 0.0578 \quad \text{for } T < 68^\circ\text{C} \quad (3a)$$

$$K_A = 0.009 \times T - 0.4961 \quad \text{for } 68^\circ\text{C} < T < 81.2^\circ\text{C} \quad (3b)$$

$$K_A = -0.005 \times T + 0.6443 \quad \text{for } T > 81.2^\circ\text{C} \quad (3c)$$

Similarly, the expression obtained for K_B vs T from Fig. 3 is of power law model type as given below in Eq. (4).

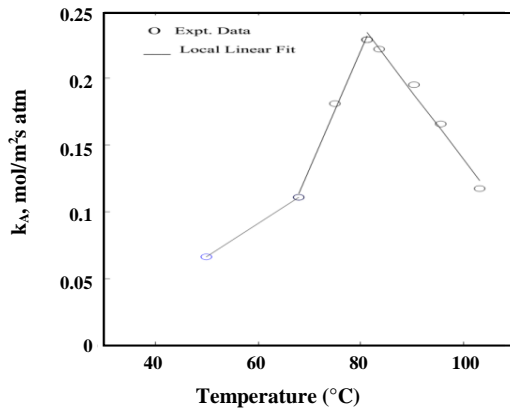


Fig. 2: Evaporation mass transfer coefficients of benzene (A) at various temperatures.

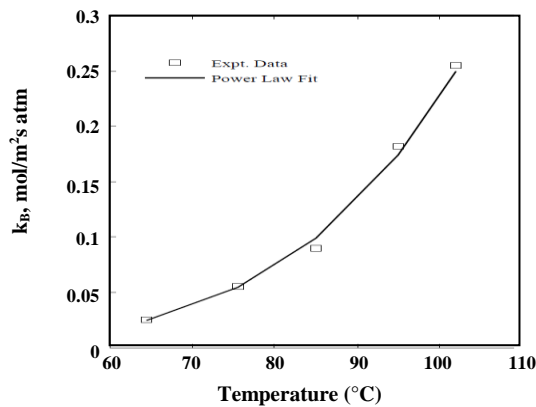


Fig. 3: Evaporation mass transfer coefficients of toluene (B) at various temperatures.

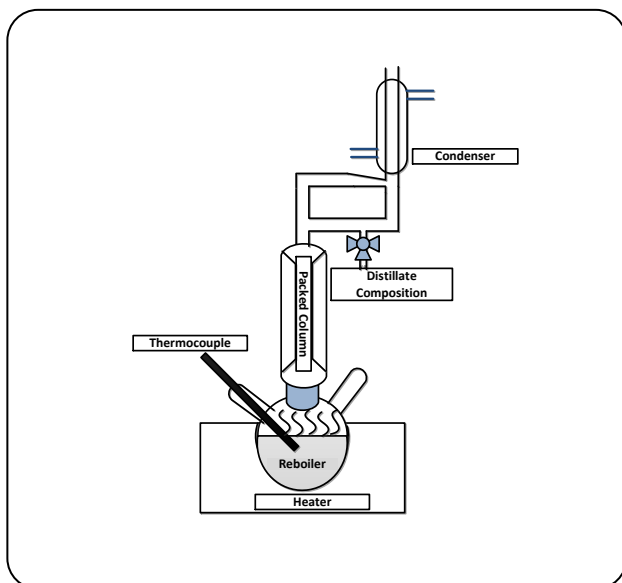


Fig. 4: Schematic diagram of experimental apparatus.

$$K_B = 1.5339 \times 10^{-11} \times T^{5.0844} \quad (4)$$

The regression R-square value for all the above fits were very close to 1 which indicates a good fit. For benzene the boiling point is 81.2 °C where as that of toluene is 110.3 °C. Hence as we heat a mixture of benzene-toluene at some point of temperature the benzene is in superheated state thereby increasing P^{sat} by large factor exponentially. Hence, when mass transfer coefficient is calculated from Eq. (1a), the magnitude of it drops below that of its value at its normal boiling point. Therefore, there is a maximum in mass transfer coefficient of benzene in the given range of temperatures i.e., 50°C – 110 °C approximately.

Batch distillation under total reflux

Experiments were carried out on binary mixture of benzene and toluene at various reboiler heat input rates. The experimental apparatus used is shown schematically in Fig. 4.

It consists of a round bottom flask of one-liter capacity surrounded with an electric heater. A temperature probe of PT-100 type is provided in the reboiler with a digital display to record the temperature manually. The heater power can be varied by changing the applied voltage with the help of a variac. An ammeter is provided to display the current passing through the heater. By taking the product of applied voltage and current the power input is calculated. A packed column of 0.5m height is provided on the top of the round bottom flask. The packing was made of raschig rings of approximately 1 cm size. Both the packing and the column wall are made of the glass. The top of the column is connected to a vertical condenser with a three-way valve and recycle tubing. The coolant water is available at room temperature of 30 °C which is circulated in the vertical condenser at a moderate flow rate so that there is very less vapor losses.

For all the experimental runs a mixture of 0.5 mole of benzene and 0.5 mole of toluene is added to the reboiler. It gives a volume of 450 ml. The heater is set to a certain applied voltage with the help of variac. The recorded voltage, current and calculated power are tabulated in Table 2 for three runs.

The temperature indicated of the reboiler liquid is noted at regular time intervals. Actually the time is noted for every 1 degree centigrade rise in temperature. The obtained temperature dynamics are plotted in Fig. 5.

Table 2: Experimental data at steady state.

Run No	Voltage (Volts)	Current (Amp)	Power (Watts)
1	104	0.503	52.3
2	144	0.7	100.8
3	175	0.853	149.2

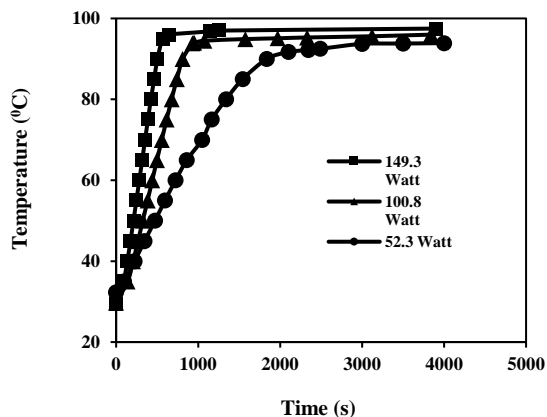


Fig. 5: Temperature dynamics for three different heat inputs to the reboiler for mixture of benzene-toluene with a mole fraction of 0.5.

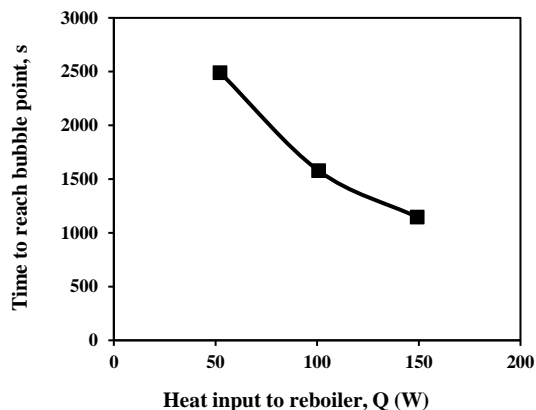


Fig. 6: Time to reach the bubble point for three heat inputs to the reboiler.

The time to reach the bubble point temperature (94.2°C for 0.5 mole fraction of benzene) is noted and plotted in Fig.6. The distillate doesn't appear or formed immediately as the bubble point is reached. It observed that the hot vapor keeps moving up in the column at a slower rate than the temperature ramp rate of the reboiler liquid. This is because the hot vapor losses heat owing to the sensible heat required for heating the packing and the glass wall for the column. The time at which the first droplet of distillate appears is also noted as the time of onset of distillation and it is plotted in Fig.7.

The distillate composition is measured using a digital refractometer and pre-calibrated data is presented in Table 3 and as a chart in Fig.8.

The measured distillate composition in terms of the mole fraction of benzene is plotted for three different heat inputs in Fig.9.

It can be observed from Fig. 9 that the distillate purity of benzene is highest for lowest heat input rate. Also, the distillate composition seems to vary monotonically with the applied heat input rate to the reboiler. Qualitatively lower heat inputs have lower vapor and liquid flow rates leading to high contact residence time in the column giving higher rectification. This particular aspect needs

to be predicted by the help of a steady state rate based model in the following sections.

THEORETICAL SECTION

Modeling

The aim is to develop a steady state model where the reboiler composition is specified, heat input rate to reboiler is specified and distillate composition has to be predicted for total reflux condition in the column as shown in Fig. 2. Some assumptions had to be made in order to arrive at a reasonable output. They are:

- The steady state temperature in the reboiler is assumed equal to the bubble point temperature at 0.5 mole fraction, which is a linear function as $T_R = 109.6 - 30.8 \cdot X_{AR}$. this assumption is applied for all three heat input rates.
- The vapor generation rates of A and B in the reboiler are assumed to be proportional to the mole fraction in the liquid or the mole fraction of benzene ensuing from the vapor in reboiler is considered equal to 0.5 which is the initial mole fraction in the reboiler contents.
- Energy balance is bypassed and a linear temperature profile is assumed along the height of the column. The expression is $T(z) = T_R - (z/H)(T_R - T_C)$ where T_C is the column temperature at top most point. It is assumed as $T_C = 65^{\circ}\text{C}$. Here H is the height of the column.

Table 3: Refractive index of a benzene-toluene mixture.

Volume fraction of benzene	Volume fraction of toluene	Refractive Index (R.I.) at 30 °C
1	0	1.4963
0.8	0.2	1.4958
0.6	0.4	1.495
0.4	0.6	1.4942
0.2	0.8	1.493
0	1	1.4919

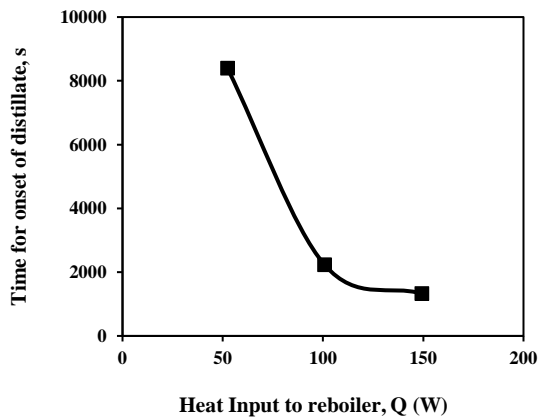


Fig. 7: Time for onset of distillation for three heat inputs to the reboiler.

By considering shell of thickness 'dz' and performing material balance and component balance, the following set of differential Eq. are obtained for composition and molar flow rate variations along the height of the column.

$$\frac{dV}{dZ} = \frac{dL}{dZ} \quad (5)$$

At infinite reflux distillate flow rate almost tends to zero and Eq.(5) gives $V = L$.

Component Balance for 'A' in vapor phase using evaporation rate based model

$$\frac{d(y_A \cdot V)}{dz} = K_A \cdot A_c \cdot a_c (x_A \cdot P_A^{\text{sat}} \cdot \gamma_A - y_A \cdot P_t) \quad (6)$$

Component Balance for 'B' in vapor phase

$$\frac{d(y_B \cdot V)}{dz} = K_B \cdot A_c \cdot a_c (x_B \cdot P_B^{\text{sat}} \cdot \gamma_B - y_B \cdot P_t) \quad (7)$$

Component Balance for 'A' in liquid phase

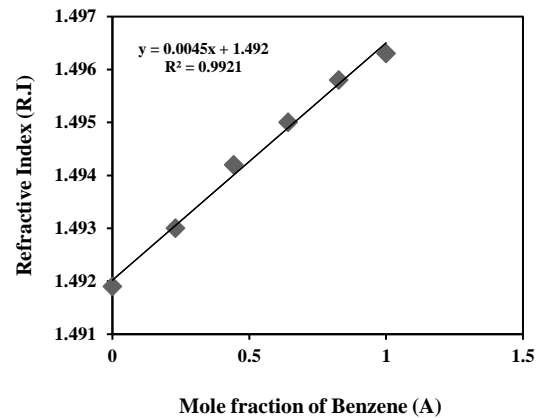


Fig. 8: Calibration chart of Refractive Index vs mole fraction of benzene in benzene-toluene mixture at temperature of 30 °C.

$$\frac{d(x_A \cdot L)}{dz} = K_A \cdot A_c \cdot a_c (x_A \cdot P_A^{\text{sat}} \cdot \gamma_A - y_A \cdot P_t) \quad (8)$$

Component Balance for 'B' in liquid phase

$$\frac{d(x_B \cdot L)}{dz} = K_B \cdot A_c \cdot a_c (x_B \cdot P_B^{\text{sat}} \cdot \gamma_B - y_B \cdot P_t) \quad (9)$$

In the present study at steady state, the reboiler temperature is assumed to be at bubble point of the mixture which is given by

$$T_R = 109.6 - 30.8 X_{AR} \quad (10)$$

Temperature variation in the column is assumed to vary linearly from reboiler to the condenser as below

$$T(z) = T_R - (T_R - T_C) \times (z/H) \quad (11)$$

At first node at the bottom of the column the vapor molar flow rates are related to the heat input rate as below

$$Q = V_A \cdot \lambda_A + V_B \cdot \lambda_B \quad (12)$$

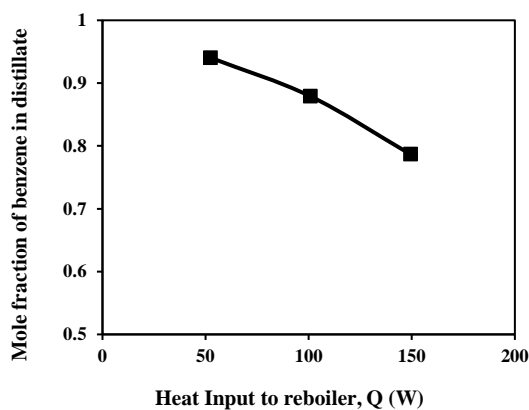


Fig. 9: Experimental distillate composition of benzene for three heat inputs to the reboiler at total reflux.

The vapor generation rates of A and B in the reboiler are assumed to be proportional to the mole fraction in the liquid as below

$$\frac{V_A}{V_B} = \frac{x_{AR}}{(1 - x_{AR})} \quad (13)$$

In the above Eq.s the notation for various symbols is as follows

Q = Is heat supplied to reboiler (Watts)

L = is the molar flow rate of liquid from column to the reboiler (gmol/sec)

V = is the molar flow rate of the vapor from reboiler in to the column (gmol/sec)

x_A = mole fraction of component 'A' in liquid phase at a location in the column

y_A = mole fraction of component 'A' in vapor phase at a location in the column

K_A = mass transfer coefficient of component 'A' (gmol/m².atm.s)

A_c = Cross sectional area of the column (m²)

a_c = specific surface area available inside the column in the form of raschig rings (m²/m³)

p_A^{sat} = saturated vapor pressure of component 'A' at a location inside the column (atm)

γ_A = activity coefficient of component 'A' in liquid phase

x_B = mole fraction of component 'B' in liquid phase at a location in the column

y_B = mole fraction of component 'B' in vapor phase at a location in the column

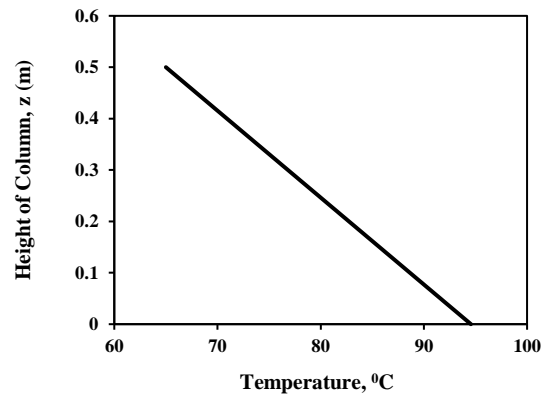


Fig. 10: Temperature variation along the height of the column at steady state.

K_B = mass transfer coefficient of component 'B' (gmol/m².atm.s)

p_B^{sat} = saturated vapor pressure of component 'B' at a location inside the column (atm)

γ_B = activity coefficient of component 'B' in the liquid phase

p_t = Total pressure or atmospheric pressure (atm)

Simulation

The set of differential equations in Eq. (5) to Eq. (9) are solved numerical using explicit Euler forward finite difference method. It should be noted that the molar flow rate of vapor V is in upward direction and molar flow rate of liquid L is in downward direction. About 51 nodes are placed at equi-distance along the height of the column. This gives 50 segments of 1 cm each. The obtained results are presented in Fig. 10 for temperature versus local height in the column, Fig. 11 for benzene (A) mole fraction x_A in liquid along the height of the column, Fig. 12 for benzene (A) mole fraction y_A in vapor along the height of the column and Table 4 indicating the composition of distillate at total reflux obtained from simulation and experiments.

In Fig. 11 and Fig. 12, the difference in vapor and liquid mole fractions of the components mainly depends on vapor and liquid flow rates. Since we are using a small heat input rate the distillation or the rectification seems to occur at a quasi-steady state implying that the streams leaving each node in the finite difference method of simulation are nearly at equilibrium. One important analysis taken up is the evaluation of local relative volatility in the column at various locations as compared to the equilibrium value.

Table 4: Simulation results as compared to experimental data.

Run No.	Heat input rate to Reboiler (Watts)	y_{D_expt}	y_{D_sim}	$L_{condenser}$ (mol/s)	$L_{A,condenser}$ (mol/s)
1	52.3	0.94	0.93	0.0016	0.0015
2	100.8	0.88	0.835	0.0025	0.0021
3	149.2	0.79	0.763	0.0037	0.0028

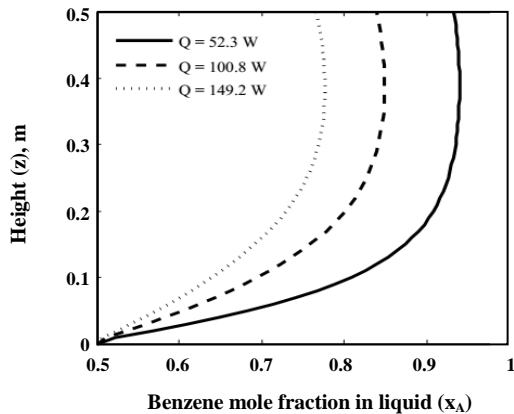


Fig. 11: Benzene mole fraction in liquid along the height of the column.

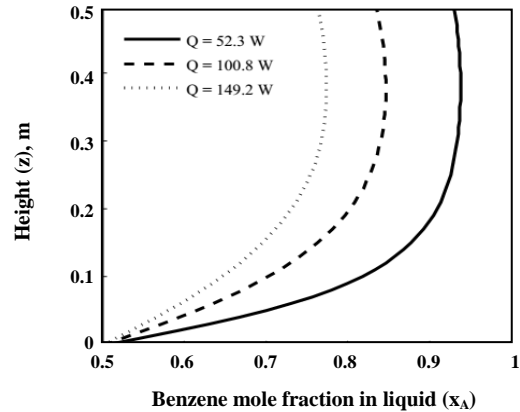


Fig. 12: Benzene mole fraction in vapor along the height of the column.

The local relative volatility $\alpha_{simulation}$ is obtained from the definition $(y_A/(1-y_A))/(x_A/(1-x_A))$ and $\alpha_{equilibrium}$ is obtained from ratio of saturation pressures of A and B at the prevailing local temperature as P_A^{sat}/P_B^{sat} . These two are compared in Fig. 13.

The data evaluated and plotted in Fig. 13 is from Fig. 11 and Fig. 12. Usually relative volatility of 1 implies azeotrope situation. But owing to the rate based model approach of simulation, such an azeotrope situation is overcome. Owing to the quasi steady state as mentioned earlier, the relative volatility is close to 1. It also can be inferred that half of the column height itself is sufficient to obtain the maximum possible rectification. Also, the temperature variation in the column is assumed to be linear as per Eq. (11). Hence it could be a minor factor that the intersection point of Fig. 13 is due to such an assumption and also the reason for relative volatility less than 1. The top and bottom locations of the column are now indicated in Fig. 13. It indicates that the more volatile benzene is rectified majorly near the reboiler itself and after half a height of the column there is no much rectification.

CONCLUSIONS

Experimental studies were carried out in a lab scale batch packed bed distillation column with various heat input rates to the reboiler. For the system of benzene-toluene smaller heat input rates were found to give higher purity of lighter component i.e., benzene in distillate at total reflux. The evaporation rate based model for non-reacting mixture (toluene-benzene system) was developed which was used in predicting the distillate composition accurately. The evaporation mass transfer coefficients were determined experimentally and their fitted curves or expressions were used in the simulations. The mass transfer coefficients for the heavier component had a good power law fit in the entire range of temperatures starting from reboiler to the condenser. Whereas the mass transfer coefficient of the lighter component had three regimes of linear fit differing in slopes as well as sign of slopes owing to superheated condition of the lighter component in the reboiler and also to some extent in the column.

It was found that the local relative volatility is close to one than equilibrium value which provides substantiation and justification for simulating real world distillation processes

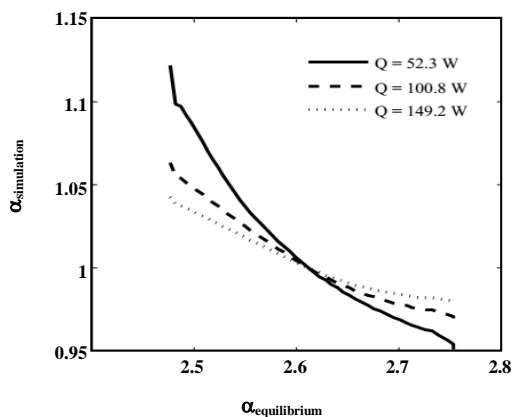


Fig. 13: Local relative volatility as obtained from steady-state simulation composition and from temperature.

using the rate-based model. This has application in modeling reactive distillation such as solid catalyzed esterification reactions in a reboiler connected with a packed distillation column above it.

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