Design and Construction of a Novel Oldershaw-type Distillation Column for Measurement and Scale-up of Tray Efficiencies

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Abstract

A new bench-scale, glass distillation column was designed and constructed in this work. This column is quite flexible with adjustable tray spacing and could be used to study various types of systems both foaming and non-foaming, each requiring different tray spacing. Tray efficiencies were measured in this three-inch diameter glass distillation column for two binary systems at total reflux. The results were compared with those obtained for a one-inch diameter Oldershaw-type column and the efficiencies obtained from an industrial column.The results of this work for the cyclohexane/n-heptane system show better efficiency than both the Oldershaw and the industrial column. With regards to methanol/n-propanol system, our data seem to follow the correct trend and are actually closer to the industrial data at lower percentages of flooding.
Keywords: Distillation column, trays, efficiency, scale-up, predictive models

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Introduction

There are two approaches to design distillation columns.One is the equilibrium model and another one which has been considerd recently, is nonequilibrium model.
In nonequilibrium approach, true mass and heat transfer in a packed or tray distillation column are considered to calculate the height of a packed column or the actual number of trays without using any kind of efficiency for the column [1,2].
But, this approach requires accurate mass and heat transfer coefficients to solve the model equations and obtain reliable results. For example, using absorption mass tarnsfer coefficient in this model instead of distillation mass transfer coefficient to design a distillation column may lead to unreliable results[3].
In order to design distillation columns using the equilibrium approach, one needs to estimate the column efficiency. HTU and HETP concepts are used to design packed columns whereas overall and Murphury efficiencies are used to design tray columns [4].
Efficiency for tray columns depends on three main factors: the system involved the flow conditions, and the tray geometry. The following methods are usually used for predicting tray efficiencies:

1. Using the data from an existing similar industrial column.
2. Using empirical correlations.
3. Using theoretical or semi-empirical mass transfer models.

We are using a fourth, seldom-used method here, which uses the efficiency data obtained in a lab-scale column to predict the efficiency for industrial-scale columns. If this could prove to be a valid method, its simplicity would make it very useful for the study of complicated and non-ideal systems. A major disadvantage of this method results from the fact that flow characteristics have a large influence on tray efficiencies. Flow characteristics depend strongly upon tray diameter, and even when all other conditions are the same, the efficiencies of columns with different tray diameters are different.

Therefore, one must consider the mass transfer aspects separately and use local efficiencies in conjunction with models accounting for back mixing in the liquid phase on the tray.
Liquid flow on circular trays is non-uniform having large values from the inlet weir to the outlet weir in the center of the tray and low values near the edges of the tray. This problem has been investigated and it has been shown that flow non-uniformity has a hindering effect on tray efficiency only in columns with diameters larger than two meters [5]. Therefore, the assumption of uniform flow for small-diameter columns seems to provide an adequate model for liquid behavior. Local efficiencies should be the same for columns of different sizes operating under the same or similar conditions. Fair et al. [6] have determined local efficiencies for industrial columns using data from one- and two-inch lab-scale columns.

The objective here is to obtain experimental data and compare them with data from an Oldershaw-type column [6], with FRI data [7], and with data from an industrial column having rectangular-trays [13]. This is done both to verify the performance of our column and also to demonstrate whether or not these data could be used for scale-up.

Experimental Work

The distillation column designed in this work has the following characteristics. The shell is made of glass and all the internal parts including the perforated plates, the down-comers, and the central shaft on which the plates are located are made of stainless steel. The perforation pattern on all trays was triangular. Unlike typical Oldershaw columns, which have a circular pipe in the center as the down-comer, we used half-cylindrical down-comers on the sides. These were rectangular plates extending from tray to tray, the top part of which formed the weir on each tray.
The novelty in the design of this system is the use of a central threaded shaft on which the trays are installed using bolts and gaskets. This allows the adjustment of tray spacing. The typical range of tray spacing is quite wide, from about 6 to 36 inches, depending on the characteristics of the system, most notably its foaming tendency. The ability to change tray spacing provides a great advantage in studying various systems. This flexibility allows us to use the same column height for different numbers of trays. In order to prevent leakage from the annulus between the tray and the shell silicone rings were screwed underneath each tray. This, of course, limits the use of the columns to systems whose components do not react with silicone or do not dissolve it, and to temperatures below the melting point of this material. The head of the shell is removable so that the shaft and the trays installed on it could be taken out and reinserted when necessary. This head was connected to the shell using a flange. The details of the newly designed tray are shown in Figure 1. The various components comprising the column are shown schematically in Figure 2 and the column specifications are presented in Table 1. The specifications of the other columns to which the data of this work are compared are presented in Tables 2 and 3.

1. Tray hole locations *2. Down-comer* *3. Silicone gasket* *4. Central shaft hole*

1. Glass shell *2. Stainless steel trays* *3. Down-comer* *4. Central shaft* *5. Reboiler* *6. Heater*
7. Condenser *8. Shaft support* *9. Flange* *10.Bolt &gasket* *11. Sampling valve*

Figure 1. Details of the adjustable tray and weir.

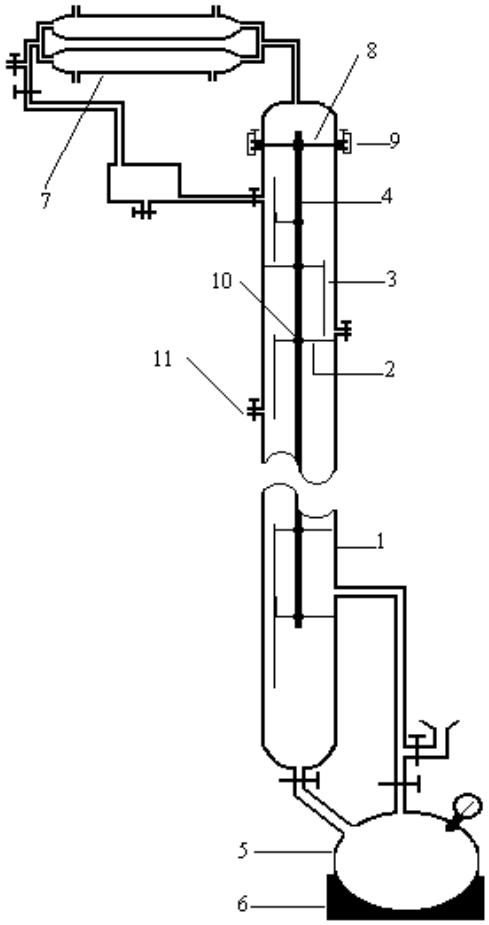

Figure 2. Schematic diagram of the novel distillation column designed and constructed in this work.

Table 1. Specifications of the designed column

Inside column diameter (cm)	7.77
Tray diameter (cm)	7.5
Column cross sectional area (cm^2)	46.575
Active tray area (cm^2)	35.198
Area available for vapor flow (cm^2)	41.687
Down-comer area (cm^2)	4.098
Hole diameter (cm)	0.25
Number of holes	97
Pitch (cm)	0.766
Weir length (cm)	5.25
Weir height (cm)	0.5*
Number of trays	4*
Tray spacing (cm)	10*
Column height (cm)	120
Diameter of central shaft (cm)	1
Tray thickness (cm)	0.08

* adjustable parameters

Table 2. Specifications of the three columns whose data are used for comparison in this work

	This work (Fanni)	Oldershaw [6]	FRI data [7]
Column diameter (cm)	7.5	2.8	122
Tray spacing (cm)	10	2.7	64.8
Hole diameter (cm)	0.25	0.089	1.27
Perforated area (%)	10	8.3	8&14
Outlet weir height (cm)	0.5	---	5.08

Table 3. Specifications of the industrial column with rectangular trays [13]

Weir length (cm)	8.3
Liquid flow path (cm)	99.1
Tray spacing (cm)	15.4
Hole diameter (cm)	0.18
Free area (%)	8
Outlet weir height (cm)	2.5
Inlet weir height (cm)	0.48

Systems studied

Two binary systems were used for which extensive data were available in the literature: cyclohexane /n-heptane and methanol/n-propanol. All chemicals were purchased from Merck and had purities in excess of 99%.
All experiments were carried out at atmospheric pressure and under steady-state conditions at total reflux.

Mathematical framework

The overall column efficiency is obtained from:

$$E_{OC} = N_t / N_a \quad (1)$$

Where,

N_t = theoretical number of trays
N_a= actual number of trays

Murphury tray efficiency is given by Equation (2):

$$E_{MV} = (Y_n - Y_{n-1}) / (Y_n^* - Y_{n-1}) \quad (2)$$

Where,

Y = vapor mole fraction
Y^* = vapor mole fraction in equilibrium with the liquid exiting the tray
n = tray number

The overall and Murphury efficiencies are related as follows:

$$E_{OC} = \ln [1 + E_{MV} (\lambda - 1)]/ \ln\lambda \quad (3)$$

Where,

$$\lambda = mV/L \quad (4)$$

Where,

m = Equilibrium line slope
L = Liquid molar flow rate
V = Vapor molar flow rate

Thus λ is seen to be the ratio of the equilibrium line slope to the operating line slope (L/V). The slope of the operating line is equal to one when distillation is carried out at total reflux. Fair et al. state that for the composition ranges in their work the average value of the slope of the equilibrium line, m, was also close to unity. Therefore, they assumed the overall column

efficiency to be equal to the Murphry tray efficiency [6]. They studied the cyclohexane/n-pentane system at a column pressure of 15 psia, and with an average relative volatility of 1.67, yielding a distillate composition of 85-90 mol%/.

In order to take the effect of liquid mixing and back mixing into account, point efficiency is defined as:

$$E_{OV} = [(Y_n - Y_{n-1}) / (Y_n^* - Y_{n-1})]_{point} \quad (5)$$

Point efficiency and Murphury efficiency are related as follows [9,10]:

$$E_{MV} / E_{OV} = f(\lambda, Pe) \quad (6)$$

Where,

$$Pe = W^2 / (D_E . \bar{t}_L) \quad (7)$$

Where,

Pe = Peclet number

W = Width of flow path

D_E = Eddy diffusivity

$\bar{t}_L$ = average residence time on the tray

Eddy diffusivity can be obtained from the following correlation [11]:

$$D_E = 0.013 U_a^{1.44} + 0.025 h_L - 0.061 \quad (8)$$

Where,

U_a = vapor velocity in the active area (ft^3/s)

h_L = liquid hold-up (in.)

Perfect vapor mixing has been assumed which is a good assumption for small-diameter columns. It has been shown that for efficiencies below 80%, tray efficiency is not a strong function of vapor mixing [12].

Equations (1) through (8) have been used to convert the overall efficiencies given in reference [6] to point efficiencies so that all data could be compared on a single diagram.

Results and discussion

The results of this work for the cyclohexane/n-pentane system have been compared to the data for an Oldershaw column [6] and the data for an industrial column [7] in Figure 3. The results of this work for the methanol/n-pentane system have been compared in Figure 4 to the data for an industrial column with rectangular trays [13], whose specifications are given in Table 5.

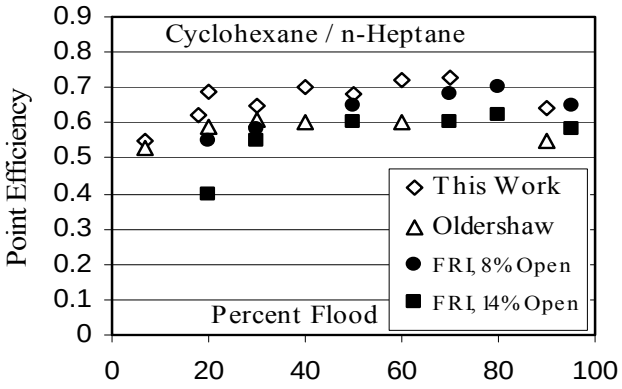

Figure 3. Comparison of the point efficiencies for the cyclohexane /n-heptane binary system.

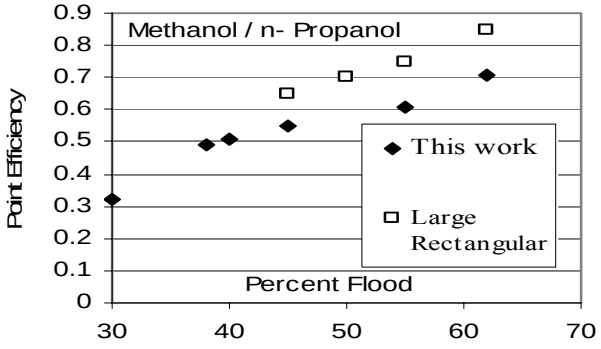

Figure 4. Comparison of the point efficiencies for the methanol/n-propanol binary system.

As can be seen in Figure 3, the results of this work are in good agreement with both the Oldershaw and the industrial column. Our data seem to follow the correct trend and are actually closer to the industrial data.

The data available from the industrial column were limited. Therefore, it may not be wise to draw a firm conclusion from the comparison indicated in Figure 4. Our data seem to follow the correct trend and are actually closer to the industrial data at lower percentages of flooding, thus provide conservative estimates of point efficiencies by about 20 to 30%. These data can be used as a basis for correlating the point efficiencies of industrial columns to those of bench-scale columns. This warrants further experimental work in this regard.

Conclusions and Recommendations

The data obtained in this work seem to confirm the performance of the newly designed column. The data obtained from this new column seem appropriate for determining the lower bound of column efficiencies for industrial columns and could therefore be used for conservative design estimates.

The following recommendations are also made for future work:

- Constructing and testing other types of trays.
- Comparing tray performance with packing performance in the same system.
- Operating the system at reflux ratios other than total reflux.

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