A LITERATURE SURVEY FOR DATA PERTINENT TO THE DETERMINATION OF GENERALIZED

TRAY EFFICIENCIES

Bу

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Thesis Approved:

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CHAPTER I

INTRODUCTION

In the field of chemical engineering one of the largest areas of interest is that which is concerned with distillation. As a result of the studies in distillation, the author believes there is a greater need for improved methods of predicting distillation tray efficiencies.

Recently various studies have been made using the concept of generalized tray efficiencies. The purpose of this paper is to complete a literature survey to find existing distillation data and use these data to evaluate a set of generalized tray efficiencies.

The author hopes that this material will be useful in further understanding those problems which may arise in other such related areas.

CHAPTER II

THEORY

Before approaching the ideas and concepts of the generalized tray efficiency, the existing methods of determining tray efficiencies will be outlined.

Tray efficiency is defined as a measure of the approach of an actual tray to an ideal or equilibrium tray. The ideal tray is one in which the vapor and liquid phases leaving the tray are in mutual thermodynamic equilibrium. Murphree (109) assumed constant molal flow rates along the column to express tray efficiencies in the following manner.

Murphree defined a vapor efficiency as

$$E_{MV} = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}}$$
(1)

where y_n is the composition of a component in the vapor leaving tray n, y_{n-1} is the composition of a component in the vapor entering tray n, and y_n^* is the composition of the vapor leaving the ideal plate in equilibrium with x_n , the actual liquid leaving the real tray. The assumptions are that the compositions y_{n-1} and x_n remain unchanged on going from the actual to the ideal tray. The assumption that x_n remains constant is important in this definition since y_n^* is a function of x_n . x_n must be saturated if y_n^* is to exist in equilibrium with this liquid stream. Similarly, Murphree defined a liquid efficiency as

$$E_{ML} = \frac{x_{n} - x_{n+1}}{\frac{x_{n} - x_{n+1}}{n + 1}}$$
(2)

where $\boldsymbol{\mathrm{x}}_n$ is the composition of a component in the liquid leaving tray n, x_{n+1} is the composition of a component in the liquid entering tray n, and \mathbf{x}_n^\star is the composition of the liquid leaving the ideal plate in equilibrium with y_n , the actual vapor leaving the real tray. The assumptions associated with this liquid efficiency are that the vapor and liquid compositions above the nth tray remain unchanged on passing from the actual to the ideal stage. The assumption that the composition of the vapor, $\textbf{y}_n,$ remains constant is important since x_n^\star is a function of y_n . y_n must be saturated if a saturated liquid phase of composition x_n^* is to exist in equilibrium with the vapor. Figure 1 shows a typical stage with the appropriate subscripts as used in these definitions. Murphree has defined tray efficiencies as the ratio of the actual change to the change which occurs if the tray is considered as an equilibrium stage.



Figure 1. A Simple Distillation Stage

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The definitions of tray efficiency as previously mentioned tend to represent a physical situation only under certain operating conditions. Conditions such as vapor channeling and incomplete liquid mixing are two common cases which result in deviations from the Murphree model. On large diameter trays, the liquid composition can vary across the tray and possibly result in an efficiency of greater than 100 per cent.

A point Murphree efficiency, expressed in terms of vapor compositions, describes the degree of approach to equilibrium between the vapor and the liquid at a single point on the tray. It is defined as

$$E_{OG} = \frac{y_n(p) - y_{n-1}}{y_n^*(p) - y_{n-1}}$$
(3)

where y_{n-1} is the composition of the vapor which enters the tray and $y_n(p)$ is the composition of the vapor leaving point p on the tray. $y_n^*(p)$ is the composition of the vapor in equilibrium with the liquid on the tray at point p.

A similar expression may be given for the liquid point efficiency as

$$E_{OL} = \frac{x_n(p) - x_{n+1}}{x_n^*(p) - x_{n+1}}$$
(4)

where x_{n+1} is the composition of the liquid entering the

tray and $x_n(p)$ is the composition of the liquid leaving point p on the tray. $x_n^*(p)$ is the composition of the liquid in equilibrium with the vapor at the same point p.

In some cases tray efficiencies are related to the separation efficiency of a whole column. Lewis (101) defined the overall column efficiency as the ratio of actual trays in a column to the number of ideal trays that will yield an equivalent separation. This definition suffers from the difficulty that it tries to describe the separation behavior of an entire column with one number even though conditions are changing throughout the column. The actual and ideal comparison tray do not have the same separating ability. Therefore, when the number of plates differs the assumption of equal reflux rates and product concentrations must be made.

Another concept in tray efficiency was first introduced by Carey (18) and reported by Nord (116) for the case of heat transfer on the distillation plate. This temperature, or thermal, efficiency is defined as

$$E_{TV} = \frac{T_n - T_{n-1}}{T_n^* - T_{n-1}}$$
 (5)

where T_n and T_{n-1} are the temperatures of the vapor leaving and entering the tray respectively, and T_n^* is the temperature of the vapor in equilibrium with the liquid leaving the

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tray. Similarly, for the liquid phase, the efficiency is defined as

$$\mathbf{E}_{+\mathbf{L}} = \frac{\mathbf{t}_{n} - \mathbf{t}_{n+1}}{\mathbf{t}^{*} - \mathbf{t}_{n+1}}$$
(6)

where t_n and t_{n+1} are the temperatures of the liquid leaving and entering the tray respectively and t_n^* is the temperature of the liquid in equilibrium with the vapor leaving the plate.

Standart (136) has defined a generalized plate efficiency which is concerned with the change of the extensive properties of a phase across the tray. Standart has so defined this system that the two streams leaving the ideal plate are in equilibrium and satisfy all the heat and material balance requirements of the actual plate.

Standart expresses the concepts concerning this definition of the equilibrium state quantitatively as follows.

For the actual and ideal equilibrium tray, an overall material balance is given as

$$V_{n-1}+L_{n+1} = V_n+L_n = V_n+L_n^*$$
 (7)

which may also be expressed for each constituent, i, as $V_{n-1}Y_{n-1,i}+L_{n+1}x_{n+1,i} = V_nY_{n,i}+L_nX_{n,i} = V_n^*Y_{n,i} +L_n^*x_{n,i}^*$ (8) where V and L are the molal flow rates of the streams

around the nth plate and y_1 and x_1 are the vapor and liquid mole fractions respectively for the ith component. V_{n-1} is the rate of the vapor entering tray n from the tray below and L_{n+1} is the liquid entering tray n from above. V_n and L_n are the rates of the vapor and liquid streams which actually leave the tray. V_n^* and L_n^* are the vapor and liquid rates respectively of the equilibrium stream leaving the ideal tray, asterisk (*) denotes equilibrium.

In conjunction with the material balance, Standart defined an enthalpy balance which is given as $V_{n-1}H_{n-1} + L_{n+1}h_{n+1} - Q_n = V_nH_n + L_nh_n = V_n^*H_n^* + L_n^*h_n^*$ (9) The definitions of V, L, V_{n-1} , L_{n+1} , V*, and L* remain unchanged. Q_n is the rate of heat lost from the nth tray to the surroundings. Here, the assumption is made that the heat lost from the actual and equilibrium tray is the same. H is the molal vapor enthalpy and h is the molal liquid enthalpy.

In addition to these equilibrium conditions, one should note that

$$T_n^* = t_n^*$$
 and (10)

$$\nu \dot{\nabla}_{,1} = \nu \dot{L}_{,1} \tag{11}$$

where μ_1 is the chemical potential of the ith component. This is equivalent to stating

$$y^* = Kx^*$$
 (12)

where K is the distribution coefficient whose value is determined from an assumed value of T_n^* . T_n^* and t_n^* are the temperatures of the vapor and liquid streams leaving the ideal tray.

With this set of equations at hand, one may readily derive the following set of efficiencies. The overall material efficiency is given by Standart as

$$E = \frac{V_n - V_{n-1}}{V_n^* - V_{n-1}} = \frac{L_n - L_{n+1}}{L_n^* - L_{n+1}}$$
(13)

The efficiency for the ith component is given by

$$E_{i} = \frac{V_{n}y_{n,i} - V_{n-1}y_{n-1,i}}{V_{n}^{*}y_{n,i}^{*} - V_{n-1}y_{n-1,i}} = \frac{L_{n}x_{n,i} - L_{n+1}x_{n+1,i}}{L_{n}^{*}x_{n,i}^{*} - L_{n+1}x_{n+1,i}}$$
(14)

and the enthalpy efficiency is given as

$$E_{H} = \frac{V_{n}H_{n} - V_{n-1}H_{n-1} + r_{n}Q_{n}}{V_{n}H_{n}^{*} - V_{n-1}H_{n-1} + r_{n}Q_{n}} = \frac{L_{n}h_{n} - L_{n+1}h_{n+1} + (1 - r_{n})Q_{n}}{L_{n}^{*}h_{n}^{*} - L_{n+1}h_{n+1} + (1 - r_{n})Q_{n}}$$
(15)

 r_n is defined as the fraction of the heat lost by the vapor stream on the nth plate. This latter efficiency is not necessarily less than one.

Unlike the Murphree and Carey efficiencies, these definitions of generalized tray efficiencies are completely symmetrical with respect to liquid and vapor. There is no need to make an assumption that the streams entering or leaving the actual tray are saturated as is the case with the Murphree and Carey efficiencies. In using the generalized plate efficiencies, the attainment of an equilibrium plate is not an elaborate manipulation of the variable conditions, but merely a convenient reference from the computational point of view.

In considering the equilibrium equations of Standart's with the generalized efficiency equations, the fact is apparent that the overall material efficiency is a dependent equation, being dependent upon the efficiency equation for the ith component.

In addition to a discussion of the generalized tray efficiency, the author included a discussion of the method used in determining T* and the other equilibrium terms found in the material and enthalpy balance equations previously discussed.

Certain experimental data must be known before one may start to calculate a generalized tray efficiency. These data consist of liquid and vapor flow rates to and from the tray, as well as their corresponding temperatures, enthalpies, and compositions. With this data at hand, one may perform the following calculations.

For an m-component system, a temperature $T_n^* = t_n^*$ is assumed as the equilibrium temperature on tray n. An important fact to note is that this value of T* is bounded by the temperature of the vapor and liquid streams leaving the plate. Using this T*, a value for K, H*, and h* may be

found for each component. These values are then used in the equilibrium equations.

A material balance may be written for each component, i, as expressed in equation (8). The equations for x_{i}^{*} and y_{i}^{*} may be written as $y_{i}^{*} = k_{i}x_{i}^{*}$, where

$$x_1^* + x_2^* + \dots + x_i^* + \dots + x_m^* = 1$$
 (16)

$$y_1^* + y_2^* + \cdots + y_1^* + \cdots + y_m^* = 1$$
 (17)

This provides 2m + 2 equations with the same number of unknowns. After solving for these unknowns, the values of V_n^* and L_n^* may be substituted into equation (9). The assumed value of T* may be checked with the values calculated for H* and h*. This procedure is continued until the assumed value of T* checks with the results calculated in the enthalpy expression.

Once these equilibrium quantities are found, the efficiencies may be determined as outlined previously.

CHAPTER III

TRAY EFFICIENCY AND DISTILLATION STUDIES

In recent years a great deal of research has been done in the field of tray efficiencies. Many factors which affect the distillation process, such as foaming, surface tension, entrainment, liquid mixing effects, froth height, and thermal effects have been studied.

Empirical Correlations

One of the approaches used to predict tray efficiencies involves correlations of an empirical nature. This empirical approach involves investigating and correlating large quantities of data for any or all design, operating, and system property variables encountered during rectification.

Two correlations, one which predicts tray efficiencies as a function of liquid viscosity and the other which predicts tray efficiencies as a function of liquid viscosity and component relative volatility have found wide use. These correlations are empirical and represent adequately

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only those systems upon which they are based, and thus, their application to other systems is doubtful.

Drickamer and Bradford (32) utilized plant test data from fifty-four refinery fractionating columns to develop a simple correlation between overall column efficiency and feed stock molar average viscosity. All of the tests were made on bubble-tray columns with diameters in excess of 4 feet. The correlations found apply only to this type of tray and for the hydrocarbon systems studied.

O'Connell (119) extended Drickamer and Bradford's correlation to include the overall efficiency as a function of the product of the average liquid viscosity based on the feed composition and the relative volatility of the key components. By inclusion of the relative volatility, O'Connell was able to extend somewhat the Drickamer-Bradford correlation to systems with high relative volatility

Utilizing only one or two variables, as did Drickamer, Bradford, and O'Connell, tends to oversimplify the complex problem associated with distillation efficiencies. To more accurately account for the complexity of this problem, Chaiyavech and Van Winkle (20) attempted to evaluate the effect of several system property variables in a 1.0 inch diameter perforated plate distillation column. The

variables considered were surface tension, relative volatility, viscosity, density, and diffusivity of both liquid and vapor phases. Data obtained from this column were correlated to give an equation for the Murphree plate efficiency in terms of the system properties mentioned.

English and Van Winkle (45) improved upon the correlation of Chaiyavech and Van Winkle by extending the correlation to include column design and operating variables as well as system property variables. The variables selected by English and Van Winkle were fraction free area, weir height, reflux ratio, vapor mass velocity, relative volatility, liquid Schmidt number, and surface tension. The data used in this correlation were from tests involving only binary systems with both bubble-caps and perforated trays.

Fundamental Correlations

The fundamental approach to tray efficiency research involves the use of mass transfer theory to characterize the transfer relationships which occur on a tray. One of the earliest major studies in this area was the project of the A.I. Ch.E. Research Committee. A five-year study starting in July, 1952, was carried out at three universities.

Their primary goal was to develop a correlation for predicting bubble tray efficiencies in commercial distillation columns.

The four main factors with which the A.I.Ch.E. Research Program were concerned were the rate of mass transfer in the vapor and liquid phases, the degree of liquid mixing on the tray, and the amount of liquid entrainment between trays. As a result of the findings, the A.I.Ch.E. Research Committee developed separate correlations for each of these factors and used these correlations to predict efficiencies.

The major steps in this prediction method were first to predict point Murphree efficiencies from the mass transfer relationships of the vapor and liquid phases. The variables affecting the resistance to mass transfer were the physical characteristics of the tray, the vapor and liquid flow rates, and mass transfer relationships of the fluid phase. The mass transfer relationships considered in the gas phase were gas rate, mole fraction of the component in the phase, mass transfer coefficient, total system pressure, interfacial area on the tray, and gas holdup. In the liquid phase the mass transfer relationships considered were liquid rate and density, mass transfer coefficient, interfacial area of the tray, and liquid holdup on the tray.

To relate this point efficiency to the Murphree tray efficiency, a liquid mixing model on the tray was developed. The variables considered were the distance of liquid travel across the tray, the eddy diffusion coefficient, and residence time of the liquid on the tray.

The final step in predicting the tray efficiency involved a correction of the Murphree tray efficiency for entrainment. The variables involved with this step were surface tension, vapor velocity, and tray spacing. Once this had been completed, a value for the overall column efficiency was obtained from this corrected Murphree tray efficiency.

Application of the A.I.Ch.E. correlation is limited to bubble-tray towers and, for most cases, to binary systems. This method may also be applied to systems in which little or no previous experience exists.

Literature Survey of Prior Experimental Observations

In the extensive literature survey which was undertaken in the preparation of this paper, several articles were found which had a definite connection to this particular field of interest. Many articles had no direct bearing in relation to what the author was searching for but were

important for the references which they contained. Therefore, these articles will be referenced and may be found in the bibliography. (1-3, 5-17, 19-31, 33-44, 46-59, 61, 62, 64-81, 83-108, 110-115, 117, 118, 120-134, 136-148,150-165).

Of all the articles surveyed, there were six papers with sufficient data to merit a discussion of these papers in relation to a generalized plate efficiency.

In June of 1963, Bakowski (4) presented an article discussing the effects of mass transfer and plate efficiency in a bubble-cup column. The apparatus used by Bakowski consisted of a boiler, a distillation column and a watercooled condenser. The column measured 4.0 inches in diameter and contained one plate. The temperature on the plate was measured by a thermometer fitted in a thermowell. Samples of the condensate entering the plate were taken at a position at the bottom of the column where the condensate was preheated before being returned to the top of the column. The samples of the liquid leaving and entering the plate were taken simultaneously. At this time a liquid rate was measured and the vapor rate was calculated from this liquid rate.

The majority of the runs were made at total reflux. Assuming that there was no loss in the condenser, the composition of the vapor entering the plate was the same as the

composition of the liquid returning from the plate to the reboiler. At total reflux the vapor composition was found by taking a liquid sample leaving the plate and using this composition for the actual vapor composition. This method of determining a vapor composition was found to be simpler and more accurate than direct sampling of the vapor stream itself.

The systems used in Bakowski's experiments include such mixtures as methanol/water, acetone/water, trichloroethylene/toluene, benzene/toluene and water/acetic acid.

Unfortunately, Bakowski failed to publish any flow rates or temperatures. Without these variables and the condenser and reboiler duties, a heat balance around a tray could not be calculated. This limitation causes one to eliminate equation (9) from the set of equilibrium equations thus eliminating certain needed equilibrium values and making the calculation of a generalized tray efficiency impossible.

Studies similar to those of Bakowski's have been performed by Carey, et al.(18), with relation to plate efficiencies in the distillation of binary mixtures. Three separate rectifying columns were employed in the experiments reported. The columns, all measuring 4.0 inches in diameter, contained one, seven, and ten plates with each plate having

the same number of caps. Mixtures of ethanol and water were rectified in the single plate column while the binary mixture used in the multiple-plate columns was benzene/toluene. A complete physical description of the rectifying columns is included in the article.

In all of Carey's work, the distillation operation was conducted at total reflux. At total reflux, the data reported gave compositions of liquid and vapor leaving the tray as well as vapor velocities and reboiler duties. However, if tray temperatures were measured, there were no references given to them for either the vapor or liquid phase. Without this data one cannot calculate enthalpies and for the same reason as outlined in the discussion of Bakowski's data the values for the generalized plate efficiencies are impossible to obtain.

In the Department of Chemical Engineering at New York University, a rectification column has been designed and built primarily for use in their chemical engineering laboratory. A discussion of the construction and operation of this column is given in an article presented by Huffman and Treybal (82).

The complete apparatus consists of a boiler, a column, and a condenser with all the necessary accessories. The column is designed to allow the feed to enter at any plate, permitting even the extremes of running the column as a stripping or as an enriching section. The column has been constructed to permit temperature measurements, rates of flow, and samples of both liquid and vapor at all vital points. In addition, distillation may be performed with open steam, or under pressure or vacuum.

The column contains ten plates of 11.0 inches in diameter with each plate containing two bubble caps. Liquid samples are taken from each plate near the downpipes from that plate while the vapor samples are removed from each plate six inches above the plate.

The experimental data given are for a carbon tetrachloride/toluene mixture. The data given consist of vapor and liquid mole fractions and liquid temperatures on each plate as well as an overall heat balance. However, no vapor temperatures or flow rates for either phase were published. For the same reason as mentioned previously, values for the generalized efficiency are impossible to obtain.

A great deal of work in the field of plate efficiencies and the measurement of related experimental data has been done by J. A. Gerster at the University of Delaware. A

paper (60) published by Gerster discusses plate efficiencies in the separation of C_{μ} hydrocarbons.

The experimental distillation unit was a 13.0 inch diameter column with ten trays spaced 24 inches apart. Each plate was equipped with thirteen bubble caps with an inside diameter of 1.5 inches and a height of 2.5 inches.

Liquid sample inlets were located about 0.5 inches above the trays and directly beneath the center of the downpipes. Vapor sample inlets were placed about 0.5 inches below the tray, exactly at the center of the trays. The thermocouple wells, specially designed for precise temperature measurement and minimum sample holdup were located to coincide with the liquid and vapor sample probes. All of the experimental runs were made at total reflux.

A summary of the data presented gives an overall heat balance with temperatures of the condenser and reboiler. The data also include the vapor and liquid compositions and the liquid rates through the column. A graph of the vapor and liquid tray temperatures is also given. However, due to the sensitive temperature dependence of the equilibrium equations, this graphical representation is inadequate to use in calculating a generalized tray efficiency.

Another article by Gerster, et al.(61), discusses plant performance of a 13 foot diameter extractive distillation

column consisting of two fifty-tray units operated in series.

In addition to measuring compositions, temperatures, pressures, and flow rates of the external streams, the column has been designed to measure vapor and liquid compositions, flow rates, and liquid temperatures of different locations within the tower.

A summary of the data acquired is presented in tabular form. This data consists of vapor and liquid flow rates, liquid temperatures, and compositions for the key components at intermediate locations within the column varying from three to seventeen trays apart. The fact that no complete set of data is given for consecutive trays, makes a generalized plate efficiency calculation impossible. This is explained by the fact that operating data, such as these recorded, are needed for both flow to and from the tray.

Perhaps the most complete set of distillation data from the University of Delaware was published by the American Institute of Chemical Engineers in 1958 as a part of their study of tray efficiencies in distillation columns (149).

The system with the most complete and useful set of data was acetone/benzene mixture published in part V of this report.

These data were recorded for a 2.0 foot diameter column containing five trays. Each tray contained seventeen bubble caps measuring 3.0 inches in diameter and 2.625 inches high.

A summary of the operating conditions and results show that the data recorded were the column pressure, the flow rate condition on tray three, the condenser and reboiler duties, the compositions of vapor and liquid leaving the individual trays, and the temperature of the liquid on each tray. To simplify the calculations, only those runs which were made at total reflux were considered. These data. along with the column specifications given in the report, allow a trial and error calculation for the remaining variable needed to calculate a generalized plate efficiency. This remaining variable is the vapor temperature of the composition leaving the trays. In order to calculate this temperature, the enthalpy data for the acetone/benzene system must be available at the column conditions. If pure component enthalpy data were available, because the mixture is nonideal, the enthalpies of the component mixture could not be calculated as a product of mole fraction and the component enthalpy. After a reasonable literature search, the author was unable to locate any pure component or mixture data and therefore was unable to complete any

calculations of a generalized efficiency.

In all of the experimental data found, at least part of the operating variables needed to calculate a generalized tray efficiency were not given. Excluding the data published by the A.I. Ch.E. Research Committee, all of the data found in the open literature had too many degrees of freedom to make any valid assumptions to use in completing the calculations of a generalized tray efficiency.

CHAPTER IV

CONCLUSIONS AND RECOMMENDATIONS

From the observations of the data found in the literature, the author believes that the measurement of all the necessary data needed to calculate a generalized tray efficiency is possible. This conclusion is based on the fact that all of the data needed were found separately, but unfortunately no one complete set was found in the open literature for the same experimental results. Using what data is available, some assumptions can be made for the missing variables, but there is no way of checking the validity of these assumed values. Work is being done at the present time to obtain a complete set of distillation data.

More work should be done in obtaining useful data to use with the definition of the generalized tray efficiencies to verify their validity and usefulness.

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VITA 2

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