AN ABSTRACT OF THE THESIS OF

GARY LEE HUNDLEY for the M.S. in Chemical Engineering
(Name) (Degree) (Major)

Date thesis is presented November 28, 1966

Title PERFORMANCE OF A PACKED DISTILLATION COLUMN WITH INDUCTION HEATED PACKING

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(Major professor)

An experimental packed distillation column was constructed and data were taken to evaluate the effects of heating sections of the column packing during the distillation operation. The experimental column was packed with carbon Raschig rings and the heating was accomplished using an induction coil. The carbon tetrachloride and benzene system was studied.

Heating the packing did not increase the separation of the components but it did allow a higher flow rate through the column before flooding conditions developed. The effect of heating relative to the efficiency of a packed column was opposite to the results obtained in an earlier investigation. Reevaluation of the earlier publication indicated that the author's conclusions were based on inappropriate vapor flow rates.
PERFORMANCE OF A PACKED DISTILLATION COLUMN WITH INDUCTION HEATED PACKING

by

GARY LEE HUNDLEY

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Professor of Chemical Engineering
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Typed by Donna L. Olson
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The author wishes to express his grateful appreciation to his wife whose financial assistance made this work possible.
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INTRODUCTION

Distillation is one of the most common and important unit operations in chemical engineering, finding widespread use in many industries. The process of distillation is a method of separating the components of a solution due to the differences in vapor pressure of the constituents. This process depends upon the distribution of the substances between gas and liquid phases, each phase having different composition.

In the operation of a distillation column, vapor leaves the reboiler at the base of the column, travels up through the column and passes out the top where it is condensed. Part of the condensate is returned to the top of the column as liquid reflux. As the vapor passes through the column it contacts the liquid phase travelling downward. Some of the more volatile components of the liquid phase are vaporized and enter the gas phase and some of the less volatile components of the gas phase are condensed in the liquid phase. This mass transfer takes place throughout the column and produces a separation of the components. In order to promote contact between the two phases so that mass transfer may occur, the tower is provided with trays to disperse the gas phase or with packing material to disperse the liquid phase. Both types of towers are in common use in industry. In the case of random packing almost any material
will suffice, provided it is inert to the components being separated and provided it offers a large surface area with a minimum of volume. Most packing materials are specially manufactured for this purpose. A common type of packing material is hollow cylinders called Raschig rings.

Attempts to improve the operating efficiency of packed distillation columns have been mainly directed toward improved packing materials and improving the liquid distribution in the column. In the case of a system in which an appreciable resistance to mass transfer lies in the liquid phase, introducing turbulence into the liquid phase should promote mixing and reduce the concentration gradient in the liquid layer, thereby improving the mass transfer and increasing the efficiency of the column. One method for promoting this turbulence is to increase the flow rates through the column. However this is not always practical, particularly in the case of a column already operating at flow rates near the loading or flooding points.

Danckwerts et al. (1) describe a phenomenon in distillation called the thermal distillation effect. These authors propose that the efficiency of a distillation column can be improved by boiling the liquid phase on the surface of the packing. This mixes the liquid phase, reducing its concentration gradient. The boiling also increases the interfacial area between the gas and liquid phases.
However, the boiling of the liquid can also have a reverse effect on the column efficiency. The slight rise in liquid temperature lessens the chances for the contacting vapor to condense into the liquid phase and the heating vaporizes more of the less volatile component.

In a recent study by Somer (3), an experimental column was operated with electrically heated packing to evaluate the effects of boiling the liquid phase in the column. Somer applied electrical energy to the entire length of carbon packing material in his column and boiled the liquid phase throughout the column. His results indicated an increase in column efficiency. The improvement rose sharply as the flow rates approached flooding velocities.

This study was undertaken to evaluate the effect of heating only in the region of the column where flooding first started to occur. By using induction heating, the specific area of the tower in which flooding occurs could be heated. This should permit a better control and observation of this phenomenon.
EXPERIMENTAL EQUIPMENT

The experimental column and associated equipment are shown schematically in Fig. 1. The column was constructed from a five foot length of three inch diameter, double-tough pyrex glass pipe. The column packing material was supported by a perforated steel plate covered with a copper screen to prevent the packing from dropping through the holes in the plate. The column was packed with 1/4-inch carbon Raschig rings manufactured by the U. S. Stoneware Corp. Before the packing was placed in the column, the packing was washed repeatedly with water to remove carbon dust particles. The column was filled with liquid before the packing was dropped into the column to minimize breakage and to obtain a more dense packing.

At each end of the column a pyrex glass tee-fitting was installed to provide suitable openings for the vapor and liquid lines. All vapor lines were formed from 3/4-inch galvanized iron pipe while the liquid lines were formed from 1/4-inch galvanized iron pipe. Attachment of the vapor and liquid lines to the column was accomplished by brazing the pipes into the center of steel plates that were bolted to the glass tees at the top and bottom of the column. Vapor tight joints in the column were obtained using teflon gaskets. All pipe joints were assembled using teflon tape.

The reboiler was constructed from a nine gallon, stainless
Figure 1. Packed Distillation Tower and Associated Equipment
steel tank in which two, 2000-watt copper immersion heaters were installed. These heaters were controlled by a seven KVA Variac operated at 220 volt line voltage. The power into the reboiler was measured with a Welch wattmeter with a 5000 watt capacity. A pressure gage, a sight glass liquid level indicator, and a valve for withdrawing a liquid sample were also installed in the reboiler.

The condenser was a copper shell-and-tube heat exchanger which used two tube passes and a single shell pass. Water was used as the cooling medium in the shell side. The condensate from the condenser flowed by gravity down into a small surge tank that was fitted with a sight gage liquid-level indicator. From the surge tank, the condensate flowed into an Eastern Industries centrifugal pump which was driven by a 1/25-HP motor that was controlled by a one KVA Powerstat. The condensate was pumped through a rotameter to measure the flow and back into the top of the column. The rotameter was a Predictability Flowmeter type 36-541-32 manufactured by the Manostat Corp. The flowmeter used a glass float giving it a maximum capacity of 805 ml per minute of water. After the condensate passed through the flowmeter, it entered a three foot long section of 1/2-inch brass pipe where it was heated to the temperature of the vapor leaving the top of the column. Nichrome wire ribbon, wound around a thin layer of asbestos insulation covering the brass pipe, provided the required energy. Several layers of asbestos
paper and one inch of fiberglass insulation were placed over the Nichrome wire. The heating section had a power rating of 300 watts at 115 volts. The electrical power to the heater wire was controlled by a one KVA Powerstat. A needle valve to control the rate of flow through the rotameter and a valve to withdraw a sample of liquid were installed in the reflux line between the rotameter and heated pipe.

The temperature of the vapor leaving the top of the column and the temperature of the reflux entering the column was measured by iron-constantan thermocouples placed in the flowing streams.

The column was not insulated in order to observe the flow inside the column, particularly in the area of the heated section of packing.

The packing was heated by means of an induction coil. The induction heating was performed using a 30 KW Lepel converter connected to a heating coil of 1/4-inch copper tubing.

No provision was made for measuring the liquid and vapor temperatures in the heated section of packing as a thermocouple or thermometer would be heated by induction and would indicate a higher temperature than the actual temperature.
DESCRIPTION OF SYSTEM STUDIED

The binary system of carbon tetrachloride-benzene was chosen for this investigation. This system was chosen because the vapor-liquid equilibrium curve is very close and parallel to the 45 degree line in the experimental range, thus requiring a large number of transfer units for a small enrichment. Both components are non-polar and have an appreciable difference in their index of refraction. The difference in the index of refraction provided a convenient, accurate method of determining the composition of liquid samples. The liquids were also relatively low boiling and non-corrosive, thereby enabling ordinary materials to be used in the construction of the equipment without worrying about corrosion or high temperatures. Disadvantages to the system are that the components are poisonous and are powerful solvents requiring care to be exercised in choosing suitable gasket materials.

The vapor-liquid equilibrium data at atmospheric pressure was obtained from the results of Fowler and Lim (2). The data are presented in Appendix B and are shown for the experimental range in Figure 2. The data was tested for thermodynamic consistency by the composition-resolution method of testing vapor-liquid equilibrium data of Van Ness (5) and was found to be consistent.

The vapor-liquid equilibrium data was necessary to determine
the number of transfer units in the column. The number of transfer units was calculated from the equation, \( N_{\text{tog}} = \int_{y_1}^{y_2} \frac{dy}{y^*-y} \). This was evaluated by plotting the function, \( 1/(y^*-y) \), vs. \( y \) and integrating the area under the curve between the limits of the reflux composition, \( y_2 \), and the vapor composition, \( y_1 \), which is in equilibrium with the liquid composition, \( x_w \), in the reboiler. The term, \((y^*-y)\), is the vertical distance between the equilibrium line and the operating line at constant values of liquid composition. The term, \( y \), is the vapor composition on the operating line corresponding to the values of liquid composition. This is illustrated in Figure 3 for a general case that includes a feed stream. In this experimental work, the operating line was the 45 degree line since the column operated at total reflux with no feed streams. A plot of \( 1/(y^*-y) \) vs. \( y \) is shown in Figure 4. This calculation procedure is described in more detail by Treybal (4). The chemicals used in the apparatus were of reagent grade purity. The benzene was manufactured by the J. T. Baker Chemical Co. and the carbon tetrachloride was manufactured by the Matheson Co.
$y^* = \text{mole fraction } \text{CCl}_4 \text{ in vapor}$

$x = \text{mole fraction } \text{CCl}_4 \text{ in liquid}$

Figure 2. Vapor-Liquid Equilibrium for Benzene-Carbon Tetrachloride at 1 atm Pressure
Figure 3. Operating Line Location Relative to Equilibrium Curve
Figure 4. Plot Used to Calculate Number of Transfer Units
OPERATING PROCEDURE

Before any data were taken or before the reagent grade chemicals were placed into the system, the equipment was operated for two days with two different batches of technical grade chemicals to remove any impurities from the system. The equipment was then cleaned and the reagent grade chemicals were placed in the apparatus. Approximately four gallons each of carbon tetrachloride and benzene were used in the system. The chemicals were replenished from time to time as the withdrawal of samples would deplete the amount of reagents in the reboiler.

Power to the reboiler heaters was adjusted by means of the Variac until the desired gas flow rate through the column was obtained. The cooling water to the condenser and the power to the heated section of pipe were adjusted until the reflux into the column was at the same temperature as the vapor leaving the top of the tower. The column was operated at total reflux; all of the vapor leaving the top of the column was returned as liquid reflux. The flow through the flowmeter was adjusted by means of the needle valve in the reflux line and by varying the power to the pump motor until the level in the surge tank held a constant value as indicated by the sight gage. The thermocouples used an ice bath reference junction and were connected to a Leeds and Northrup potentiometer
Model 8662 to measure the generated EMF.

The column was operated approximately five hours before samples of the reflux and liquid in the reboiler were collected in flasks and analyzed for their composition. Two samples were taken an hour apart and had to agree within one percent before conditions were adjusted for a new run. Analysis was accomplished using a Bausch and Lomb Precision Refractometer of the modified Abbe type. The temperature of the refractometer prisms was maintained at a constant value of 25°C by means of a constant temperature bath. A calibration chart was prepared by measuring the index of refraction of samples of known composition.

A 100 ml sample of the reflux was also collected and weighed on an analytical balance to determine the density. The density was necessary to correct the flowmeter reading. The flowmeter was calibrated with water since the density of the reflux was not known before the run. Viscosity corrections were also applied but were found to be insignificant.

The induction converter was operated at its lowest power step of approximately 12 KW and the geometry of the copper induction coil was varied by changing the diameter and number of turns to provide enough heat to the packing to cause the liquid on the surface of the packing to boil but not too much heat so that the liquid on the packing would be completely boiled off. After the proper geometry
of the coil was determined to provide the desired amount of heat, it was not varied during the majority of the runs, nor was the position of the coil varied after the region of the column involving flooding was determined. Two runs were performed with the induction converter at a power setting of two and one run was performed using a larger coil at a power setting of one.

A series of runs were obtained by varying the gas flow rate through the column without induction heating. Then a series of runs with induction heating was obtained over the same range of gas flow rates as the previous runs. The number of transfer units was computed for each run and a comparison was made between the runs with and without induction heating on the packing.
ANALYTICAL PROCEDURE

Samples of the reflux and reboiler liquid were taken and analyzed for composition with the refractometer. A 100 ml sample of the reflux was taken in the volumetric flask and weighed to determine reflux density. Additional data taken was reboiler pressure, reboiler heater Variac setting, and reflux and top vapor temperature. The flow meter reading was corrected for density effects by the following equation,

\[ V' = V^0 \sqrt{\frac{2.36 - \rho'}{1.36 \rho'}} \]

where \( V' \) = volume flow rate of liquid in ml/min flowing at conditions where its density is \( \rho' \), \( V^0 \) = volume flow rate of water in ml/min at 70°F, \( \rho' \) = density of liquid in grams/ml at the conditions of flow. This equation was obtained from the predictibility chart supplied with the flowmeter by the manufacturer.

The gas flow rate from the top of the tower was calculated from the equation,

\[ G = \frac{(V') (\rho') (60)}{(MW) (A) (453.6)} \]

where \( MW \) = calculated molecular weight of reflux, \( A \) = cross sectional area of column in ft\(^2\), \( G \) = gas flow rate at top of column in lb-moles/hr-ft\(^2\).

The number of transfer units in the column was calculated as
previously described knowing the top and bottom compositions in the column.

In addition to determining the vapor flow rate at the top of the column, the vapor flow rate entering the column was calculated. This was calculated knowing the power to the reboiler heaters and the latent heat of vaporization of the liquid in the reboiler. No heat losses were assumed in this calculation. The calculated vapor flow rate entering the column should be somewhat greater than the actual flow rate because of the heat losses from the reboiler. Also, the vapor flow rate from the top of the column is lower than the actual flow rate through the column because some vapor is condensed in the column due to heat losses from the column.

The vapor flow rate from the reboiler was calculated from the equation,

\[ G^0 = \frac{3.313 \text{ Btu}/(hr)(\text{watts})(P)}{(\lambda)(A)} \]

where \( P \) = power in watts into reboiler, \( \lambda \) = molar heat of vaporization of reboiler liquid in Btu/lb-mole, \( A \) = cross sectional area of column in ft\(^2\), \( G^0 \) = vapor flow rate entering the column in lb-moles/hr-ft\(^2\). A sample calculation is shown in Appendix C.

It is believed that the largest cause of the data scatter is due to inaccuracies in the reflux flow measurement.
RESULTS AND DISCUSSION

The results of this study are presented in Figures 5 and 6 and in Appendix A. Figure 5 shows the number of transfer units in the column as a function of the vapor flow rate from the top of the column. This curve shows that the number of transfer units with induction heating is less than the number obtained without induction heating at the same vapor flow rate from the top of the column. A few runs were made at a higher heating rate and one at a lower heating rate but these did not appear to be significantly different from the majority of the runs. The results shown in this figure appears to be the opposite of the results of Somer (3). However, Somer presented his data in a misleading manner in that his vapor velocity was the vapor velocity leaving the reboiler. Since the heating of the packing generates vapor in the column, the vapor flow rate from the reboiler is not the true vapor velocity through the column. The vapor flowing from the top of the column is representative of the actual vapor flow within the packing; this is the flow rate used to define the operating line and, in turn, the transfer units within the packed column.

The results of this study are also presented in Figure 6 as a function of the vapor flow rate from the reboiler. When the data is presented in this manner the results are similar to those obtained by Somer, indicating that the heating increases the number of transfer
Figure 5. Number of Transfer Units vs. Vapor Flow Rate From Top of Column
Figure 6. Number of Transfer Units vs. Vapor Flow Rate Entering Column
units at a given vapor flow rate. Since this is a false vapor flow rate, this conclusion is not representative of what actually takes place.

The decrease in number of transfer units with heated packing is believed to be the result of the heated section acting as a partial reboiler. The total height of packing available for mass transfer was reduced; accordingly, the effectiveness of the entire column was reduced.

One beneficial effect that was observed in the operation of the column with heated packing was that the column could be operated at higher flow rates before flooding started to occur. The experimental column would start to load and flood at the base of the packing. The liquid was held up at the packing support. As the flow rates increased, the column would begin to fill with liquid and the pressure drop would increase through the column. However, when heat was applied to the packing, the flow rates increased in the column and the number of transfer units increased but the column did not start to flood. All the increased liquid flow did not have to pass out of the column and therefore was not held up at the support.

In conclusion, it appears that the heated packing does not increase the separation of the components but it will allow a higher flow rate through the column before flooding will occur. Further study is necessary to evaluate these effects more fully.
BIBLIOGRAPHY


APPENDIX A

EXPERIMENTAL DATA

\(G\) is the measured vapor flow rate from the top of the column, \(\text{lb-moles/hr-ft}^2\); \(G_0\) is the calculated vapor flow rate entering the column, \(\text{lb-moles/hr-ft}^2\).

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**Induction heating - power step 1**

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**Induction heating - power step 2**

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**Induction heating - large coil**

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APPENDIX B

VAPOR-LIQUID EQUILIBRIUM DATA
BY FOWLER AND LIM (2)

Isobaric conditions at 760 mm. Hg

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<th>$x_1$ mole fraction CC\textsubscript{4} in liquid phase</th>
<th>$y_1$ mole fraction CC\textsubscript{4} in vapor phase</th>
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APPENDIX C

SAMPLE CALCULATIONS FOR RUN 9

Data Recorded During Run

Density of reflux = 1.377 g/ml
Rotameter reading = 7.3
Variac setting = 147
Index of refraction of reflux = 1.46886
Index of refraction of reboiler liquid = 1.47799

Calculated Data

Rotameter water flow calibration at reading of 7.3 = 167 ml/min.

The reflux flow rate is calculated from the water calibration flow rate by the formula

\[ V' = V_0 \sqrt{\frac{2.36 - \rho'}{1.36 \rho'}} \]

where \( V_0 \) is the water flow rate, \( \rho' \) is the reflux density, and \( V' \) is the reflux flow rate.

\[ V' = 167 \sqrt{\frac{2.36 - 1.377}{(1.36)(1.377)}} \]

\[ V' = 121 \text{ ml/min} \]

The measured index of refraction for the reflux corresponds to a
CCl₄ mole fraction, y₂, of 0.707 and the reboiler liquid index of refraction corresponds to a CCl₄ mole fraction, xₚ, of 0.481. From the vapor-liquid equilibrium data, the composition of xₚ corresponds to an equilibrium vapor composition, y₁, of 0.505.

The curve in Figure 4 is integrated graphically between the limits of y₁ and y₂ to determine the number of transfer units. The number of transfer units was determined to be 12.3.

The reflux mass flow rate is calculated from the equation

\[ G' = V' \rho' \]

\[ G' = (121)(1.377) \]

\[ = 165 \text{ g/min} \]

\[ = 21.8 \text{ lb/hr} \]

The molecular weight of the reflux was calculated from the equation,

\[ MW = (153.87)y₂ + (78.11)(1 - y₂) \]

\[ MW = 131.4 \]

The vapor flow rate from the top of the tower was calculated from the formula,

\[ G = \frac{G'}{(MW)(A)} \]

where A = cross sectional area of column, ft².

\[ G = \frac{21.8}{(131.4)(0.048)} \]

\[ G = 3.50 \text{ lb-moles/hr-ft}² \]
The variac setting of 147 corresponds to a power input to the reboiler heaters of 1400 watts. The heat of vaporization of the reboiler liquid was calculated by the equation

$$\lambda_{\text{mix}} = (12,854)(x_w) + (13,236)(1 - x_w)$$

$$\lambda_{\text{mix}} = 13,043 \text{ Btu/lb-mole}$$

The vapor flow rate entering the column was calculated from the formula,

$$G^o = \frac{(3.414 \text{ Btu/watt-hr})(P)}{\lambda(A)}$$

where $P$ is power input, watts.

$$G^o = \frac{(3.414)(1400)}{(13,043)(0.048)}$$

$$G^o = 7.63 \text{ lb-moles/hr-ft}^2$$