

Zero-Gravity Distillation Utilizing the Heat Pipe Principle (Micro-Distillation)

Separation of ethanol-water and methanol-water mixtures has been accomplished experimentally using a horizontal zero-gravity distillation column. A countercurrent flow between liquid and vapor phases was established utilizing the principle of the heat pipe. The concentration profile along the column has been investigated with various product rates. A high degree of separation was achieved in a relatively short column lined with capillary wicks. The flexible column orientation can also be a tremendous advantage as compared to the necessarily vertical operation of the conventional distillation columns.

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SCOPE

Significant improvements have been made recently to increase the efficiency of continuous contact distillation. A wetted-wall column has generally been considered to improve its efficiency. However, poor wetting characteristics are still a problem of such a system. Swathed glass tubes, ground glass tubes, and stainless steel mesh tubes have been used as wetted-wall columns with improved wetting properties so that more effective distillation might be achieved (in the range of low reflux rate).

In order to obtain a uniform wetting surface and an enhanced vapor-liquid contacting surface, materials with capillary action, such as wicks and screen meshes, can be used for the distillation. All conventional distillation columns use gravitational force or centrifugal force (e.g., the British ICI column) to return the con-

densed liquid to the evaporator. By employing materials with capillary action, however, the condensed liquid can be transported back to the evaporator in the absence of or even against gravity. Thus the capillary action provides flexibility in column orientation.

The scope of the present study is to investigate the feasibility of microdistillation (using short columns) in the absence of gravity (or in a horizontal position). Both total reflux conditions and steady state with product removal conditions are studied for separation of binary mixtures. As a possible application in outer space, zero-gravity distillation could be used to separate liquid mixtures for recycle of the materials used.

CONCLUSIONS AND SIGNIFICANCE

A new unit operation has been developed for a horizontal distillation column. Experiments were carried out for binary liquid systems using a glass tube and fiberglass as wick material. The temperature of the evaporator was kept just below the boiling temperature of the liquid mixture and an atmospheric pressure was maintained in the vapor phase. Mixture samples were analyzed to determine the concentration profiles along a total reflux column and a steady state column with product removal.

It has been demonstrated that both the most and the least volatile components of a binary mixture of any composition can be

separated as highly concentrated products without using gravity forces. In contrast to the tall tower and the vertical operation of conventional distillation, this device permits a short column to achieve the same degree of separation in a horizontal position.

A numerical simulation was developed to obtain the concentration profile along the column. A good agreement is shown between the theoretical and experimental profiles. The performance was characterized by the number of transfer units and height of a transfer unit.

BACKGROUND

Heat Pipe

A study of the two-component heat pipe for ethanol-water and methanol-water systems has been conducted by Feldman and Whitlow (1969), Tien (1970), and Tien and Rohani (1972). In the

investigations of the two-component heat pipe, the temperature readings inside the heat pipe were made along the centerline in the vapor phase. These readings were used to determine the vapor or liquid bulk mole compositions at any section along the heat pipe. Cotter (1965), Katzoff (1967), and Barsch et al. (1970) have conducted studies showing that pure components occupy each

end section of a heat pipe, with the more volatile component further from the evaporator at steady state.

Operating limits of the heat pipe include capillary, sonic, entrainment, and boiling limitations. The first heat pipe theory dealing mainly with the capillary limit and temperature characteristics was studied by Cotter (1965). The operating limits were treated by Chi (1976) and Dunn and Reay (1976). Levy (1968) and Kemme (1969) have studied the sonic limitation which usually occurs with operation at low vapor pressure. Tien and Rohani (1974) have presented an analysis of the effects of vapor pressure variation on vapor temperature distribution. Shibayama and Morooka (1980) studied wick characteristics and capillary properties and predicted the maximum heat transfer rates of capillary limits.

Distillation Column

One of the most critical problems in design is the selection of the contacting device to increase the efficiency of the column. Duncan et al. (1942), Minard et al. (1943), and Herman and Kaiser (1944) noted that the use of fiberglass as a packing material appeared to possess favorable properties.

In the rectification of an ethanol-water system of a 12 in. (30 cm) wetted-wall tower, Surowiec and Furnas (1942) indicated that a good rectification device might be a bundle of half-inch (1.27 cm) tubes. To achieve good contact between liquid and vapor flow, Johnstone and Pigford (1942) investigated the wetted-wall tower of an ethanol-water system. Watanabe and Munakata (1976) improved wetting characteristics by utilizing swatched glass, ground glass, and stainless steel mesh tubes to achieve effective distillation in a vacuum wetted-wall column.

Recently, Imperial Chemical Industries (1983) in England claimed that fractional distillation could be achieved under high-gravity conditions using a spinning squat doughnut-shaped drum containing usual packing material.

DESCRIPTION OF MICRO-DISTILLATION

The efficiency of continuous contact distillation may be improved if one introduces a controlled and more efficient means of liquid-vapor contact. This can be achieved in a device similar to the heat pipe. A heat pipe is a very simple device in which a condensible vapor flows from a hot surface to a cold surface where it becomes a liquid that then travels in the opposite direction by capillary action or gravity. At every location along the flow axis, the vapor is in good contact with the liquid.

Suppose that instead of a pure liquid, a binary mixture of liquid is placed in a heat pipe. The same action of countercurrent flow will take place. Since the vapor phase is in contact with the liquid phase at every location of the heat pipe, there will be a continuous exchange of mass between these two phases. Thus, the arrangement is no different from a continuous contact distillation column. If there are no outlets or inlets, this device will produce a total reflux situation at a steady state. Therefore, the binary mixture will be separated in such a way that the more volatile component is concentrated at the low-temperature end and the less volatile component is concentrated at the high-temperature end of the column. A continuous concentration profile will be established along the column connecting those two extreme values.

If a binary feed mixture of any composition between the above two limiting values is introduced into the column at the location where the composition is equally balanced, then this column can continuously separate the binary mixture into two product streams withdrawn from the hot and cold ends of the column. Thus, a continuous distillation can be achieved in a micro-scale column without using gravity forces at all, resulting in "zero-gravity distillation" (or micro-distillation).

THEORY

Mass transfer occurs between the liquid and vapor phases due to heat transfer and diffusion. When the vapor produced by the heat and diffusional mass transfers has the composition y^e , the material balance of the more volatile component in the vapor phase is

$$\frac{d(Gy)}{dz} = K_y a A_v (y^e - y) + J_e y^e \quad (1)$$

Although the micro-distillation column consists of evaporator, adiabatic, and condenser sections, for a simple approximation only the adiabatic section will be considered in calculating the concentration profile by introducing an effective column length:

$$L_{\text{eff}} = L_a + \frac{L_e + L_c}{2} \quad (2)$$

where L_a , L_e , and L_c are the column length of the adiabatic section, evaporator, and condenser respectively.

Since the adiabatic section operates at zero radial heat flux, only the diffusional mass transfer rate should be considered. If the composition of evaporating vapor is in equilibrium, $y^e = y^*$, then Eq. 1 can be rewritten for the adiabatic section:

$$\frac{d(Gy)}{dz} = K_y a A_v (y^* - y) \quad (3)$$

When the vapor phase resistance to mass transfer is controlling, the $(H.T.U.)_{OG}$ of the packed column is nearly constant and independent of the vapor and liquid flow rates. However, as described by Watanabe and Minakata (1976), a constant mass transfer coefficient can be applied to the zero-gravity distillation column because the column operates at low vapor flow rates. Since $K_y a$ is constant and independent of the flow rates, Eq. 3 can easily be converted to a dimensionless equation:

$$\frac{dy}{d\bar{z}} = \frac{G_o N_{OG} (y^* - y)}{G} - \left(\frac{y}{G} \right) \frac{dG}{d\bar{z}} \quad (4)$$

where N_{OG} is $K_y a A_v L_{\text{eff}} / G_o$ and $\bar{z}$ is z / L_{eff} .

The local vapor flow rate, G , relates to the local axial heat flow rate, Q_H , with the assumption of saturated vapor at the local vapor temperature:

$$\frac{dQ_H}{d\bar{z}} = h_{fg}^m \frac{d(Gy)}{d\bar{z}} + h_{fg}^l \frac{d[G(1 - y)]}{d\bar{z}} \quad (5)$$

Since the axial heat flow rate is constant at the adiabatic section, the axial heat flow rate gradient is

$$\frac{dQ_H}{d\bar{z}} = 0 \quad (6)$$

Equations 4 and 6 can be combined by eliminating $dG/d\bar{z}$ to yield a relationship between y and $\bar{z}$:

$$\frac{dy}{d\bar{z}} = (1 - fy)(y^* - y) N_{OG} \frac{G_o}{G} \quad (7)$$

where $f = (h_{fg}^l - h_{fg}^m) / h_{fg}^l$. For the ethanol-water system, $f \ll 1$, which closely approximates the equal molar heats of vaporization assumption.

Since the zero-gravity distillation column is analogous to the packed column, the concept of the overall height of a transfer unit in the vapor phase, $(H.T.U.)_{OG}$, can be employed for evaluating the column's performance in distillation. The values of $(H.T.U.)_{OG}$ are computed by the Chilton and Colburn (1935) equation.

Operating Limit

The pressure drop of the liquid in the saturated wick can be obtained by integrating the liquid pressure gradient along the column:

$$\Delta P_l = \int_z \left(\frac{dP_l}{dz} \right) dz. \quad (8)$$

Since liquid velocity in the wicks is generally very low, the dynamic pressure gradient may be neglected. Thus, the liquid pressure gradient in the direction of liquid flow at steady state is related to the frictional drag forces:

$$\frac{dP_l}{dz} = \frac{4f}{D_H} \left(\frac{\rho_l V_l^2}{2} \right). \quad (9)$$

Applying Darcy's law, the pressure gradient can be expressed in terms of permeability, K_p , and mass flux, m :

$$\frac{dP_l}{dz} = \frac{\mu_p m}{\rho_l K_p A_w}. \quad (10)$$

In order for the zero-gravity distillation column to be able to operate, the pressure rise by capillary force must be greater than or equal to the pressure drop sum. The pressure difference due to the hydrostatic head of the liquid depends upon the positions of the evaporator and condenser. Since the capillary limitation is the most restrictive among all the others—such as boiling limit, sonic limit, and entrainment limit—the maximum heat flow rate of the column can be obtained from the maximum capillary force, $P_{c,max} = 4\sigma/D_c$. In the actual system, the vapor pressure drop is negligibly small in comparison to the liquid phase pressure drop. For the horizontal column, the maximum capillary force can be obtained by integrating the liquid pressure gradient:

$$P_{c,max} = \int_0^z \left(\frac{\mu_l Q_H M}{\rho_l K_p A_w h_{fg}} \right) dz. \quad (11)$$

By introducing a distribution function of heat flow rate, $F(z)$, the maximum heat flow rate, $Q_{H,max}$, can be obtained by:

$$Q_{H,max} = \frac{4\sigma K_p A_w M}{D_c \int_0^z \left[\frac{\nu_l}{h_{fg}} F(z) \right] dz} \quad (12)$$

where the distribution function

$F(z) = z/L_e$ at the evaporator

$F(z) = 1$ at the adiabatic section

$F(z) = \frac{Z - z}{L_c}$ at the condenser

under the assumption of uniform heat addition and loss at the evaporator and the condenser, respectively.

EXPERIMENTAL

A diagram of the apparatus to determine the feasibility of the zero-gravity distillation column is shown in Figure 1. The evaporator and condenser were made from copper tubes (10.6 mm I.D. $\times$ 100 mm long). The adiabatic section consisted of a glass tube (10.5 mm I.D. $\times$ 340 mm long) that was perforated with 3 mm I.D. holes at 30 mm intervals as sampling ports. A wick was constructed by rolling several layers (1.2 mm thickness) of fiberglass (Owens-Corning Fiberglass) together and inserting them into a tube. The wick was held firmly against the wall by an aluminum screen. An outlet 10 mm from each end of evaporator and condenser was con-

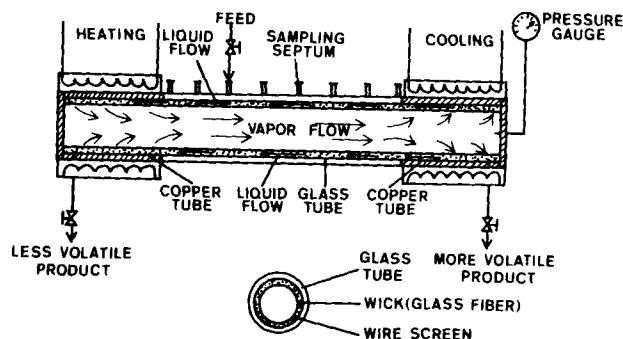


Figure 1. Zero-gravity distillation column.

nected to a needle valve through a copper tube (6.3 mm O.D. $\times$ 20 mm long).

To eliminate the resistance of air against the vapor flow, the column was evacuated before loading 20 cm³ of liquid mixture, which was a sufficient amount to saturate the column's wick. The column was operated below the boiling temperature of the system. Then the heat flux was increased gradually until the operating pressure reached about atmospheric pressure. The column was allowed to reach a steady state with constant heating and cooling temperature. This usually required 3–4 h. Samples were taken periodically from the sample ports and analyzed with a gas chromatograph (Hewlett-Packard model 5832A equipped with a TCD and a Porapak-Q column). A second set of samples was taken an hour later and two samples were compared to determine if equilibrium had been established.

When the column reached a steady state at total reflux, the mixture was fed through the first sampling port (120 mm from the end of the evaporator). A diagram of the continuous distillation column is shown in Figure 2. The feed rates were measured by counting the number of drops from the needle (18G 3/2, hypodermic needle, Yale). Condenser and evaporator valves were opened carefully to the product reservoirs (6 cm³ capacity). When these reservoirs were filled with liquid, drain valves were opened to the collectors. Both products were measured by using an electronic balance at 1 h intervals. The vapor pressure in the column was kept at atmospheric pressure during all the runs. After confirming the overall material balance with a constant product rate the column was allowed to reach a steady state. The column operated for approximately 3 h to reach a steady state. During the run, samples were analyzed. When a steady state was reached, the composition and flow rate were measured. With a constant feed composition, the feed rate was changed. The above procedure was repeated with various feed compositions. It was estimated that the concentration distribution data were accurate to ± 1.2 mol %. The estimated error of overall material balance was within 4% of the feed in all cases.

RESULTS AND DISCUSSION

Total Reflux Performance

The liquid phase concentration distribution of three combinations of ethanol-water system and two combinations of methanol-water system at total reflux along the column are graphically illustrated in Figures 3 and 4. The concentration of the more volatile component increased abruptly in a narrow zone called the separation zone; the location of the separation zone depended on the volume ratio of the two components as well as the operating temperature. Increasing the mixture composition shifted the concentration profile parallel to the evaporator, as shown in Figures 3 and 4.

The composition of ethanol at the condenser was 84 mol % (i.e., the azeotropic mixture) from a 20 mol % initial feed mixture composition. In the case of over 30 mol % of initial mixture composition, incomplete separation occurred at the evaporator end, as shown in Figure 3. For the performance of 10 mol % methanol-water system, pure water was obtained at the evaporator, with incomplete separation of methanol at the condenser. However, for the 40 mol % methanol system, almost complete separation was obtained at both ends, as shown in Figure 4.

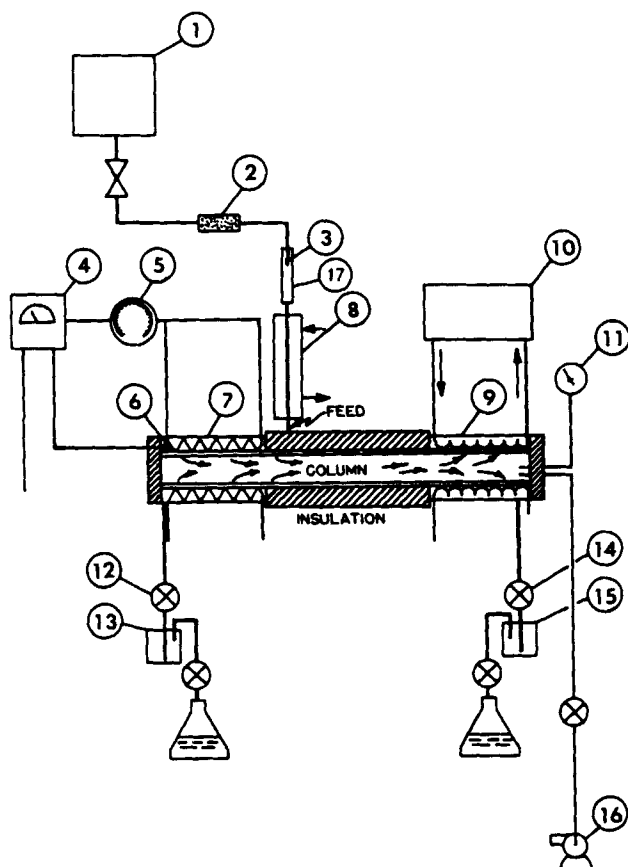


Figure 2. Continuous distillation column.

- | | |
|---------------------------|----------------------|
| 1. Storage | 10. Bath circulator |
| 2. Filter | 11. Vacuum gauge |
| 3. Needle | 12. Evaporator valve |
| 4. Temperature controller | 13. Hot reservoir |
| 5. Voltage regulator | 14. Condenser valve |
| 6. Probe | 15. Cold reservoir |
| 7. Heating coil | 16. Vacuum pump |
| 8. Hot water heater | 17. View glass tube |
| 9. Cooling coil | |

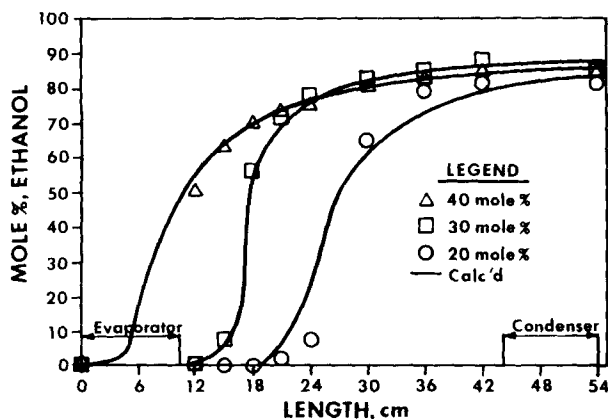


Figure 3. Composition profiles at total reflux for ethanol-water system.

The experimental results showed that if the initial volume composition of the more volatile component is higher than that of the less volatile component at a steady state, some quantity of the more volatile component may still exist in the evaporator. Otherwise, the amount of more volatile component in the evaporator may be completely separated. The separation zone of the methanol-water system has a wider range than that of the ethanol-water system. This result might depend upon the equilibrium slope of the system. These results also demonstrate that substantial separation can be achieved even with a short column.

Due to the essentially pure component at the end of the evaporator, the numerical model must follow a backward integration from the condenser end. The boundary conditions of this initial value problem are:

$$\begin{aligned} \text{at } z = 1; \quad & y_1 = y_c = x_c \\ & T_1 = T_c \\ & G_1 = G_c \end{aligned}$$

$$\begin{aligned} \text{at } z = 0; \quad & y_o = y_e = x_e \\ & T_o = T_e \\ & G_o = G_e \end{aligned}$$

The vapor flow rate, G , should be known; however, measurement of this rate would be very difficult. As one possible assumption, the column was believed to operate at its maximum operating limit, such as the capillary limitation. The maximum heat flux can be obtained by Eq. 12. The vapor flow rate also can be calculated from the maximum heat flux. At total reflux, $G = L$ and $y = x$ at every point along the column. The equation of concentration gradient was solved by using the fourth-order Runge-Kutta method.

The numerical simulation of the total reflux works generally well, but contains some difficulties. These difficulties relate to restrictions in the unknown boundary conditions, which are the vapor flow rates at the evaporator and condenser. Since the maximum liquid flow rates are functions of the concentration profiles, a trial and error method should be used to solve the equations.

Calculated concentration profiles are shown in Figures 3 and 4 in comparison with measured values. The calculated concentration profiles follow the experimental trends closely. Most of the calculated concentration profiles lie slightly below the experimental profiles, and the experimental concentration gradients are steeper than the theoretical gradients.

The differences between the measured and calculated values are probably due to the relatively complicated heat and mass transfer mechanisms in the evaporator and condenser. Small amounts of the liquid condensed in the pressure gauge, outlet tubing, and in the valves of the each end. Thus, the exact working quantities and compositions might be slightly different from the initial feed composition.

Steady State Operation with Products

The concentration profiles along the column were analyzed for various reflux conditions in the first set of experiments. In most experiments the concentration of the product (cold product), waste (hot product), and liquid phase at the feed position were analyzed.

In several instances, lowering the rate of product withdrawal allowed a greater separation of the components. Change in product rate has a large effect on the composition. When the product rate was increased to about 25% of the feed rate (0.8 mol/h), a high concentration of over 74 mol % ethanol was obtained with 20 mol % ethanol-water feed mixture. The rapid decrease in the concentration of product occurred with increasing product rate at 30% of the feed rate (1 mol/h). Similar results occurred in the performance of 20 mol % methanol-water system. 99 mol % methanol was withdrawn from the condenser when the product

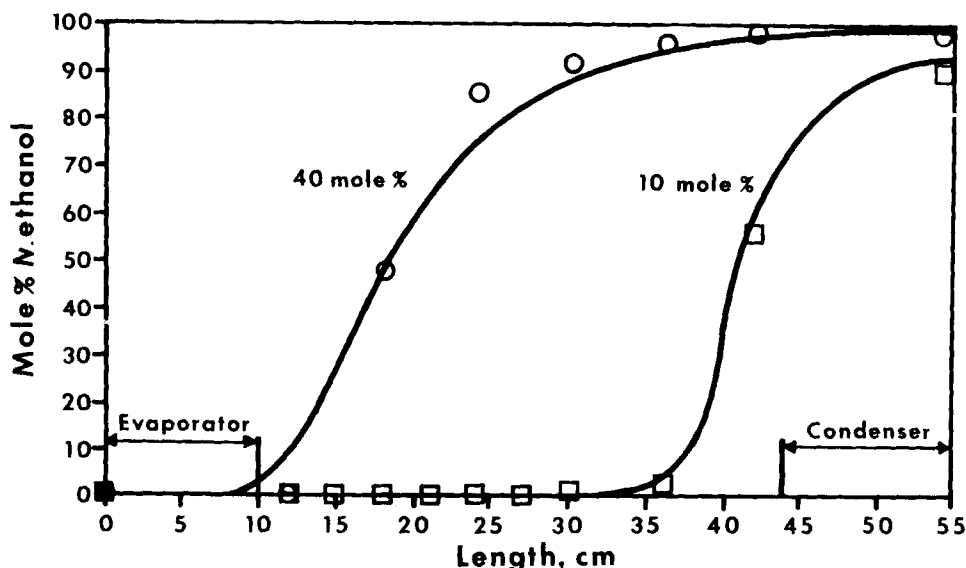


Figure 4. Composition profiles at total reflux for methanol-water system.

rate was about 16% of feed rate (1.1 mol/h). The abrupt concentration drop (to 82 mol %) occurred when the product rate was increased to 24% of the feed rate (1.4 mol/h). On the other hand, the vapor-liquid ratio might be an important factor in the performance of the micro-distillation column.

The calculated results obtained from the numerical model for a partial reflux enricher are shown in Figures 5 and 6 as solid curves. The numerical procedures involved in the simulation were identical to those outlined earlier at total reflux performance. The composition of the liquid phase at the feed position, which was measured experimentally, was used as one of the initial conditions. Vapor compositions were calculated from the operating line equation of enrichment by Billet (1979).

The data in Figure 5 for the 20 mol % ethanol-water feed mixture indicated that as the vapor-liquid ratio increased, the product concentration decreased. The calculated results were in good agreement with experimental data. The results of the 20 mol % methanol-water feed mixture are shown in Figure 6. When the product rate was increased to 4 mol/h the separation zone was spread out over the entire column length. The abrupt drop in product concentration also occurred from 99 to 82 mol %, when

the vapor-liquid ratio was increased to 1.2.

In general, the calculated results were in good agreement with the experimental data at low vapor-liquid ratios. Most of the experimental data show a steeper concentration gradient at the separation zone, than that of the calculated results as like total reflux results.

The calculated $(H.T.U.)_{OG}$ of the methanol-water system is plotted against the slope of the equilibrium curve with the vapor-liquid ratio as the parameter in Figure 7. The straight line represents the approximate expression for $(H.T.U.)_{OG}$ by the least-squares method. The results indicated that $(H.T.U.)_{OG}$ values increased as the vapor-liquid ratios were increased. No effective separation occurred over the vapor-liquid ratio of 1.3. Since the high value of G/L corresponds to a high vapor rate in comparison to the liquid rate through the column, one may conclude that the high vapor velocity had a large resistance in vapor phase. However, it is possible that there might be other factors.

For the purpose of comparison, it is assumed that the column will behave the same as that of the scaled-up column and the total flow rate can be obtained by multiplying the number of tubes and the flow rate in the one-tube column.

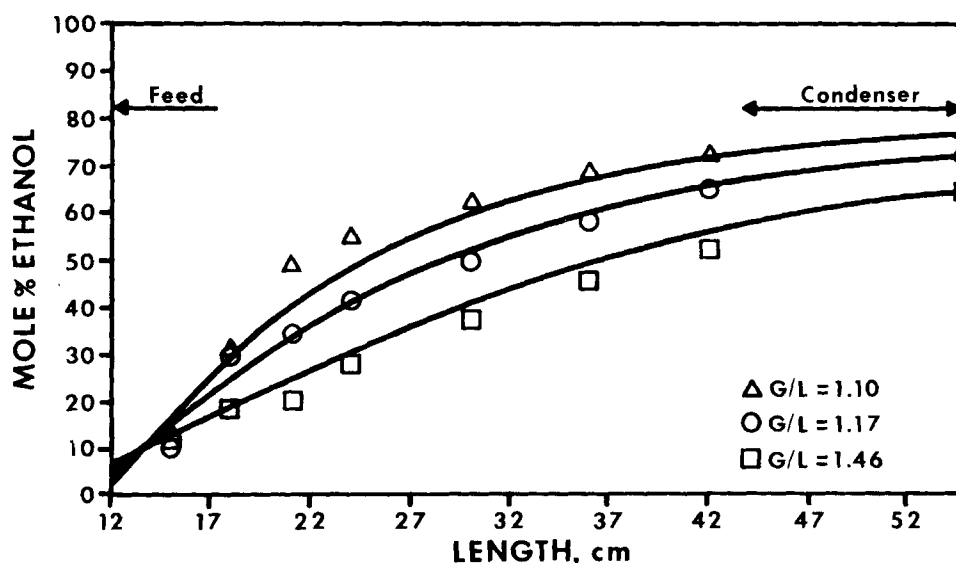


Figure 5. Composition profiles with product for 20 mol % ethanol-water system.

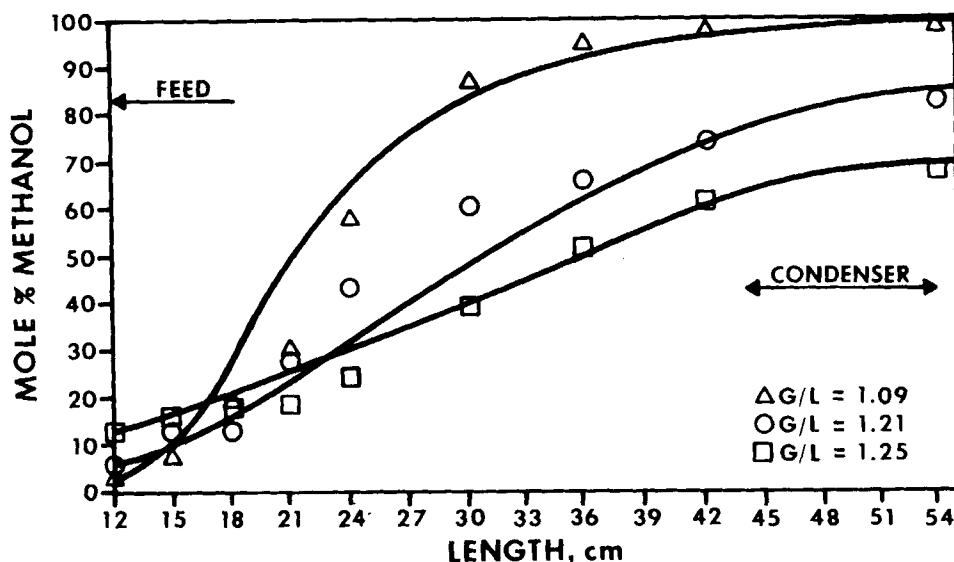


Figure 6. Composition profiles with product for 20 mol % methanol-water system.

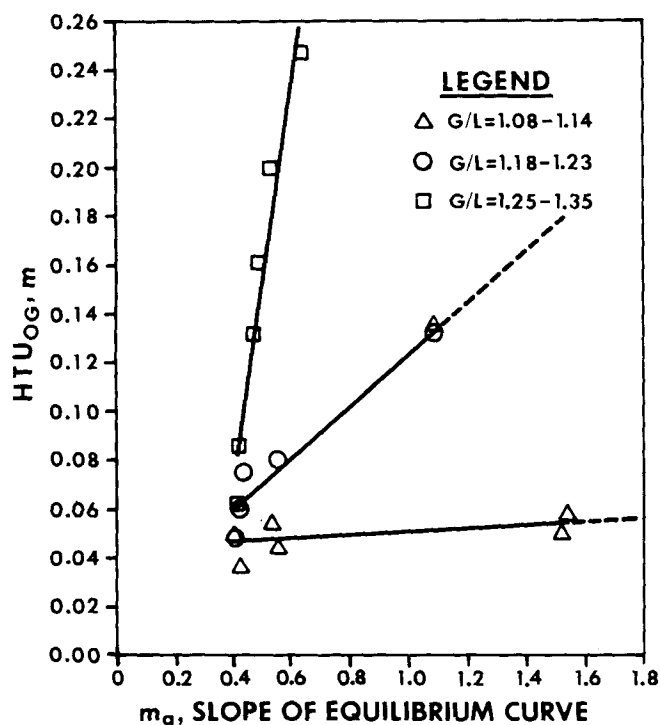


Figure 7. Overall $(H.T.U.)_{OG}$ vs. average slope of vapor-liquid equilibrium curve for methanol-water system.

Five runs on a fiberglass-packed tower of 1.98 m were selected from the work by Minard et al. (1943). The equivalent length was computed for a zero-gravity distillation column. The maximum flow rate of the zero-gravity distillation column was calculated from the maximum heat flow rate, Eq. 12. The results of the computation are tabulated in Table 1. The $(H.T.U.)_{OG}$ for the zero-gravity distillation column was obtained from Figure 7 at a 1.21 vapor:liquid ratio. The result suggests that if difficulties of construction and operation can be overcome, the zero-gravity distillation column may offer the same degree of separation in a horizontal column one-ninth the length of conventional vertical towers.

TABLE 1. COMPUTED COMPARISON OF PACKED AND ZERO-GRAVITY DISTILLATION COLUMN (Z-G-D)

Quantity	Run Number				
	1	2	3	4	5
Vap. flow rate kmol/h·m ²	140.3	146.5	141.4	152.3	182.6
Vap. flow rate of Z-G-D kmol/h·m ²	24.3	24.5	24.9	26.7	26.4
(H.T.U.) _{OG} of packed column, m	1.33	1.56	1.62	2.48	2.19
Computed equivalent (H.T.U.) _{OG} of Z-G-D, m	0.059	0.065	0.068	0.190	0.162
Equivalent length of Z-G-D column, m	0.269	0.236	0.245	0.387	0.405
No. of 10.5 mm tubes for equivalent vapor flow rate	7,430	7,660	7,290	7,330	8,890

*Runs were taken from data of Minard et al. (1943) for packed height 1.98 m fiber glass packing tower.

NOTATION

A	= cross-sectional area, m ²
a	= interfacial area, m ² /m ³
D_c	= capillary diameter, m
D_H	= hydraulic diameter, m
f	= friction factor
G	= vapor flow rate, mol/s
$H.T.U.$	= height of a transfer unit
h_{fg}	= latent heat of vaporization, J/kmol
J_e	= evaporating rate, mol/m ² ·s
K_p	= wick permeability, m ²
K_y	= overall mass transfer coefficient, kmol/m ² ·s
L	= liquid flow rate, mol/s
M	= molecular weight
m	= mass flow rate, mass/s
m_a	= slope of equilibrium curve
Δp	= total pressure drop, Pa
P_c	= capillary pressure, kg/m·s ²
Q_H	= axial heat flow rate, J/s
V	= mean velocity, m/s
x	= mole fraction of liquid phase

y	= mole fraction of vapor phase
Z	= total column length, m
z	= axial distance, m
$\tilde{z}$	= dimensionless axial distance

Greek Letters

ρ	= density, mol/m ³
μ	= viscosity, kg/m·s
σ	= surface tension, N/m
ν	= kinematic viscosity, m ² /s

Superscripts

e	= evaporating
l	= less volatile component
m	= more volatile component
$*$	= equilibrium

Subscripts

a	= adiabatic section
c	= condenser
e	= evaporator
l	= liquid phase
$\max$	= maximum
o	= initial
v	= vapor phase
w	= wick phase

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