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Separation of 2-Propanol–Water Mixture with Capillary Porous Plates

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ABSTRACT

Distillation of the 2-propanol–water mixture of different compositions was experimentally studied in a continuous distillation column equipped with polar and nonpolar porous plates as well as normal sieve plates. The results showed that while no separation was achieved in a distillation column with porous carbon plates or with conventional stages, the azeotropic point of this system was broken in a distillation column with polar sintered stainless steel porous plates. A distillate of about 80.0 mol% 2-propanol was obtained for a feed of azeotropic composition, i.e., 68.0 mol% 2-propanol. These results showed that the main factors affecting the separation efficiency in a given porous plate are the polarization of the pure liquids and porous plates as well as the polarization difference between the mixture components.

Key Words. Capillarity; Distillation; Polarizability; 2-Propanol

INTRODUCTION

Anhydrous organic solvents are used in electronic industries to wash the components and the circuits. The resulting water–organic solvent mixture needs to

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be separated so that the organic solvent can be reused in the washing process, thus minimizing cost and pollution (1). 2-Propanol is one of the organic solvents used in this industry. However, the 2-propanol–water mixture has an azeotropic point with a minimum boiling temperature of 80.22°C and 68.0 mol% 2-propanol at atmospheric pressure. This makes removal of anhydrous 2-propanol from a 2-propanol–water mixture by conventional distillation impossible. Another separation step is needed to break the azeotropic point, and this step usually involves extractive or azeotropic distillation which requires the addition of a second liquid to alter the vapor–liquid equilibrium (VLE) of the system by enhancing the relative volatility of one of the components relative to the other. However, the added liquid must subsequently be removed in another distillation column, which reduces the thermal efficiency of the separation process.

Distillation of liquid mixtures using capillary porous plates is a new process which depends upon the use of intermolecular interactions between the solid and liquids to create an "external force field" to alter the "normal" VLE of a given mixture (2–4). Due to porous solid–liquid interactions, a layer with different physicochemical properties from those in the bulk phases may be created next to the solid surface. Another interaction that may result in alteration of the VLE in a porous material is the vapor–liquid interface curvature, as given by the well-known Kelvin equation (5–7).

$$\ln\left(\frac{p^{\mathrm{r}}_{\mathrm{v},0}}{p^{\infty}_{\mathrm{v},0}}\right) = -\frac{2\sigma_0 \tilde{V}_{1,0}}{rRT} \tag{1}$$

Yeh et al. (8–10), Abu Al-Rub and Datta (2, 3), Abu Al-Rub et al. (4), and Wong (11) proved the feasibility of using capillary porous plates to alter the VLE of a given binary solution. Yeh et al. (8, 9) conducted some experimental studies on distillation of different liquid mixtures, including some azeotropic mixtures, by the use of macroporous fractionating plates of sintered stainless steel. The results obtained showed alteration of VLE in the porous plates and resulted in high separation efficiencies for most of the systems studied. Abu Al-Rub et al. (4) reported the results of some experimental studies involving distillation of different liquid mixtures of different polarity, including some azeotropic mixtures, by the use of capillary porous sintered stainless steel plates which were found to have altered the VLE of these liquid mixtures and resulted in high separation efficiencies for some systems. Recently, Wong (11) conducted some experiments on the VLE of some mixtures involving alcohols–water in different porous plates using the headspace gas chromatograph technique. She found that the VLEs of the systems studied were altered in the capillary porous plates used.

Yeh et al. (10) developed an approximate theory to predict the vapor pressure of pure components inside capillary plates. Their theory depends on using the theory of corresponding state for prediction of the component vapor pressure and then using the van der Waals relation for the temperature dependence of surface tension to relate the vapor pressure empirically to the surface tension by eliminating the temperature. Abu Al-Rub and Datta (2, 3) developed an alternative theory to study the vapor pressure of pure liquids as well as the VLE of binary mixtures in porous plates. They showed that the main factors affecting the vapor pressure of pure components and the VLE of a binary mixture are 1) the polarization of the pure liquids, 2) the relative polarization difference of the mixture components, and 3) the polarization of the porous solid. They also developed an accurate theoretical approach to predict the vapor pressure as well as the VLE in porous plates. They showed that the alteration of the VLE of mixtures in capillary porous plates is due to a change in the vapor pressures of the pure components and the activity coefficient of each component in the porous plates.

The change in the activity coefficients can be explained on the basis of the fact that in the presence of external fields, the deviation from ideality is due to two contributions: 1) the short-range intermolecular interactions (intrinsic contribution), and 2) the long-range intermolecular interactions (external contribution). Abu Al-Rub and Datta (3) thus showed that

$$\gamma_i = \gamma_{i,0}\gamma_{i,\mathrm{F}} \tag{2}$$

where $\gamma_{i,\mathrm{F}}$ is the activity coefficient due to the presence of external force fields. Thus, from an experimental measurement of the activity coefficients in the absence and in the presence of external fields, the long-range interactions contribution can be obtained.

Heat and mass transfer rates in distillation with capillary porous plates were studied by Scolere (12). Scolere (12) analyzed the process in terms of capillary vaporization, capillary condensate flow, and capillary condensation.

The objective of this study is to study the separation efficiency and the factors affecting the separation of the 2-propanol–water system in continuous distillation with capillary porous sintered stainless steel fractionating plates of 13.5 μm pore diameter and with capillary porous carbon plates of 30 μm pore diameter.

EXPERIMENTAL APPARATUS AND PROCEDURE

VLE of 2-Propanol–Water System

The VLE of the 2-propanol–water mixture without the porous plates at atmospheric pressure was measured using an 800 mL circulation still similar to that used by Subbaiah (13). A detailed description of this still and detailed procedures can be found elsewhere (13).

Distillation of 2-Propanol–Water System

Distillation of the 2-propanol–water mixture with capillary porous fractionating plates was conducted using the Scott Model 9079 (obtained from Scott-Engineering Sciences, a division of A-T-O, Inc.) distillation column shown in Fig. 1, which can be used for plate- or packed-column distillation. As a plate column, it consists of six sieve plate sections, each assembled from a 5-in.-long, 3-in. I.D. glass pipe section, a stainless steel process ring with four connections, and appropriate rubberized asbestos gasketed flanges, and bolts. Each section contains process fittings for feed, pressure measurement, liquid/vapor sampling, and weir-downcomer adjustment. The specifications of the sieve plates used are as follows: diameter of each plate, 3.0 in.; plate thickness, 1/8 in.; hole diameter, 5/32 in.; number of holes, 36; open area, 0.688 in.^{2}; and % open area, 9.7%.

The plate column was assembled as follows: starting with two 6-in. Pyrex glass pipe sections, two cast iron flanges with molded asbestos splitting inserts, two Teflon gaskets, a stainless steel process ring and sieve, or a porous stainless steel plate, the two sections were assembled as shown in the exploded

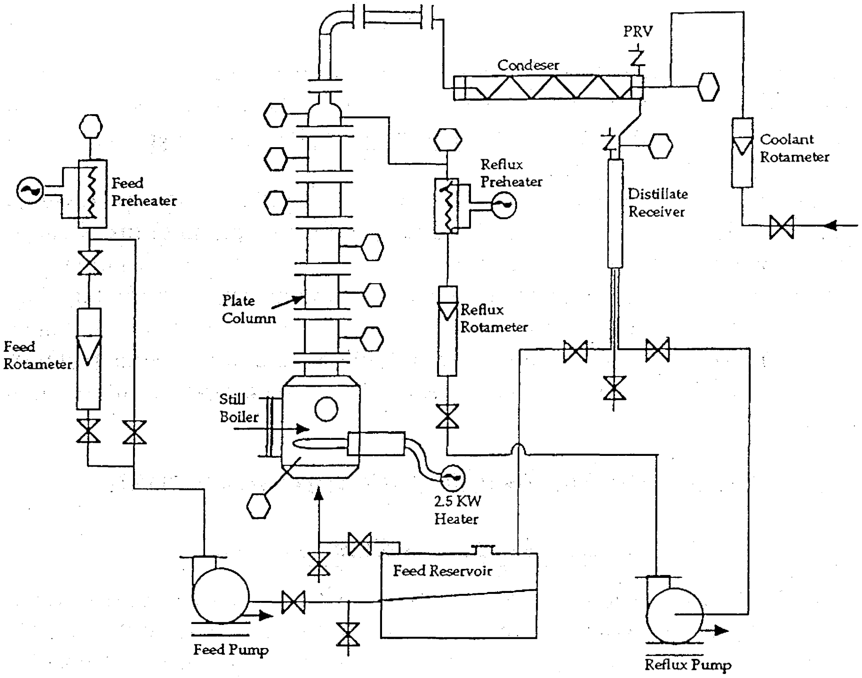

FIG. 1 Scott Model 9079 distillation column.

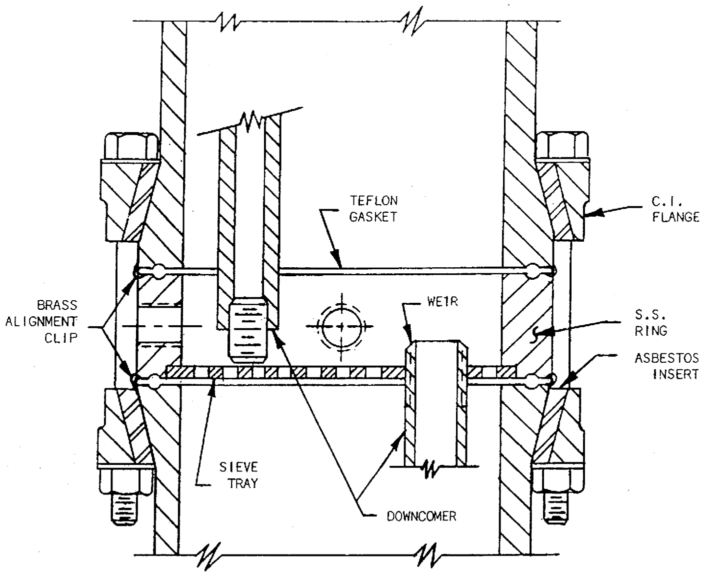

FIG. 2 Plate column assembly (14).

view (Fig. 2). The downcomer was inserted into a weir, and the weir was threaded into the plate. In a similar manner, other sections can be assembled.

The weirs/downcomers were aligned as follows: Beginning with the bottom plate, the plate was rotated until the weir was approximately equidistant between the thermocouple port and the quick-disconnect coupling as shown in Fig. 3. This procedure was repeated for each succeeding section with the alignment for each weir being 180° out-of-phase as one proceeded up the column. The weir height was 3/8 in. A detailed description of this apparatus and assembly procedures can be found elsewhere (14).

The plate column, when assembled, is approximately 30 in. in height, bolted to the boiler at its bottom and to the vapor feed line at its upper end by means of a bell-reducing coupling section and a flexible Teflon expansion joint. On each plate the weir and downcomer are readily accessible for adjustment purposes. In distillation with capillary porous plates, the sieve plates are replaced by porous plates of similar size. This column allows the effects of vaporization, condensation, and vapor–liquid mixing to be viewed and studied under the conditions of dynamic operation. Experiments with two porous plates; porous carbon plates of 30 μm pore diameter and porous sintered stainless steel plates of 13.5 μm pore diameter, and with normal sieve plates were conducted to compare the "efficiency" of the porous plates in distillation.

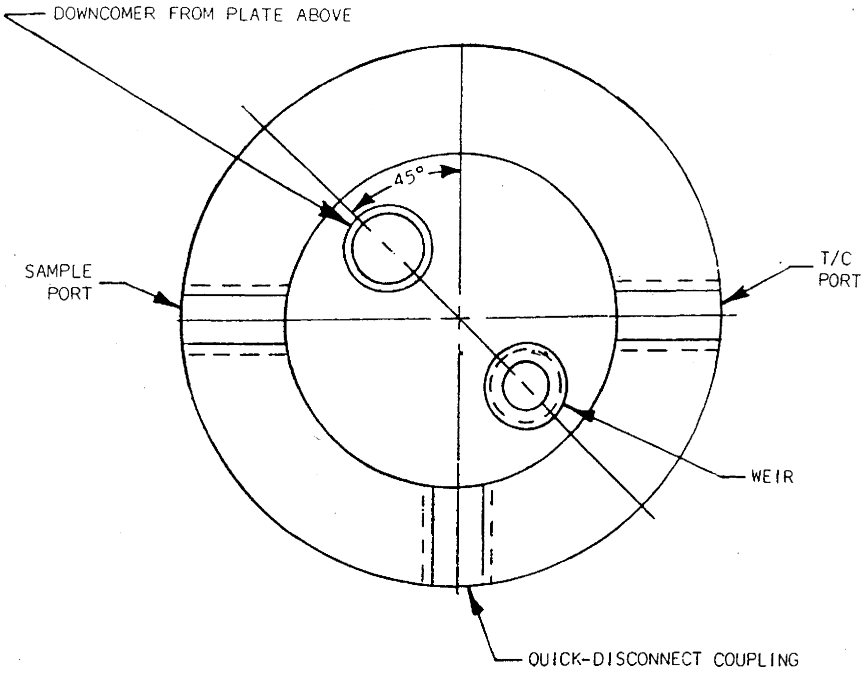

FIG. 3 Plate plan view (14).

The porous polar plates used in this study were sintered stainless steel plates with the specifications listed in Table 1. These plates were air blown to remove any dust and then washed for 24 hours in a beaker containing acetone. Next, the plates were dried in a vacuum and then immersed twice in boiling

TABLE 1
Specifications of Sintered Stainless Steel Plates

Manufacturer	Technetics Corporation
Product number	FM 1104
Plate thickness	1/8 inch
Area density	1.04 lb/ft^2
Median pore diameter	13 μm
Pore size range	7–72 μm
Tensile strength	6000 psia
Surface area	39,000 in.^{2}/lb

TABLE 2
Specifications of Porous Carbon Plates

Manufacturer	Union Carbide Corporation
Porosity	52%
Plate thickness	1/8 inch
Bulk density	1.05 g/cm^3
Median pore diameter	30 μm
Flexural strength	400 psia
Compressive strength	600 psia

distilled water for 8 hours, using fresh water each time. The plates were then tested for cleanliness by using fresh distilled water and observing their ability to be wetted completely and instantly by water.

The porous nonpolar plates used in this study were carbon plates with the specifications listed in Table 2. These porous plates were prepared by machining them from a larger sheet of the same material. These plates were air blown to remove any dust and then washed with distilled water, then dried and immersed in 2-propanol for 1 hour, then washed with water, dried, and put in sulfuric acid for 1 hour. Then the plates were twice immersed in boiling distilled water for 8 hours, using fresh water each time. The plates were then tested for perfect cleanliness.

After the column was assembled, 8 gallons of the mixture to be distilled was charged into the feed reservoir, and the heating cycle and the flow of water through the condenser were begun. The system heated up and began boiling within 15 minutes. The temperature was monitored as distillate began to collect in plates over the boiler. Steady state was obtained within 45 minutes. As the distillate collected in the distillate receiver, the reflux, in the case of normal stages, was adjusted to attain the required reflux ratio. Pressure relief valves are located at the top of the distillate receiver and on the shell side of the condenser. These were set to open at approximately 1.0 psig. If for any reason the liquid level in the boiler were to drop below the electrical heating coils, a thermal-overload switch set to respond at 240°F would interrupt the line power. Samples of the distillate were taken every 15 minutes for 5 hours. The samples were analyzed in a Perkin-Elmer AutoSystem Gas Chromatograph using a 6 ft, 1/8 in. Porapak R column under isothermal conditions at 170°C. Calibration was done for each mixture used, and the accuracy of analysis was estimated to be 1%. Each sample was analyzed five times, and the average value was taken as the composition of that sample. The experiments in this study were conducted under total reflux conditions.

The experiments started with a feed composition corresponding to the azeotropic composition. This choice of feed composition allowed a quick conclusion to be made regarding the feasibility of this method for breaking the azeotrope. At the azeotropic composition, of course, the liquid and the vapor have identical compositions and no further separation can be achieved through ordinary distillation. Thus, if distillation in porous media is effective in breaking the azeotrope, we would expect the distillate composition to be higher or lower than the feed composition. However, if this process is not effective, then we expect the distillate composition to be the same as the feed composition.

In the case of distillation with capillary porous plates, it is essential to have a liquid film immobilized within the porous plates. Thus, to ensure that the vapor–liquid was immobilized within the porous plate, the pressure differential of the rising vapor between two plates in the vapor phase should not exceed the capillary pressure calculated by the Laplace–Young equation. If this happens, then the vapor will blow through the capillary passages, thus producing no perceptible effect of the external force field. To ensure that the pressure differential of the vapor is less than the capillary pressure, the heating rate was controlled.

RESULTS AND DISCUSSION

VLE of 2-Propanol–Water System

The experimental data, along with data from literature (15), of VLE for the 2-propanol–water system at 1 atm under normal conditions are shown in Fig. 4. It is clear from this figure that the experimental results of VLE of the 2-propanol–water system found in this study are in agreement with those from the literature. As is evident from this figure, this system has an azeotropic point at about 68.0 mol% 2-propanol. This means that no further distillation can be achieved beyond 68.0 mol% 2-propanol with conventional distillation.

Distillation of 2-Propanol–Water System

Distillation of a 2-propanol–water mixture by a multistage distillation column was conducted under five different column configurations: 1) with four porous sintered stainless steel plates, 2) with five porous sintered stainless steel plates, 3) with six porous sintered stainless steel plates, 4) with six porous carbon plates, and 5) with six normal stages. Unfortunately, a larger number of plates could not be utilized in the current column. The results for a feed composition of 68.0 mol% 2-propanol, which corresponds to the azeotropic composition, under the first, fourth, and fifth column configurations are shown in Table 3 and in Fig. 5. As can be seen from Fig. 5, the distillate composition in the porous sintered stainless steel plates obtained at steady state

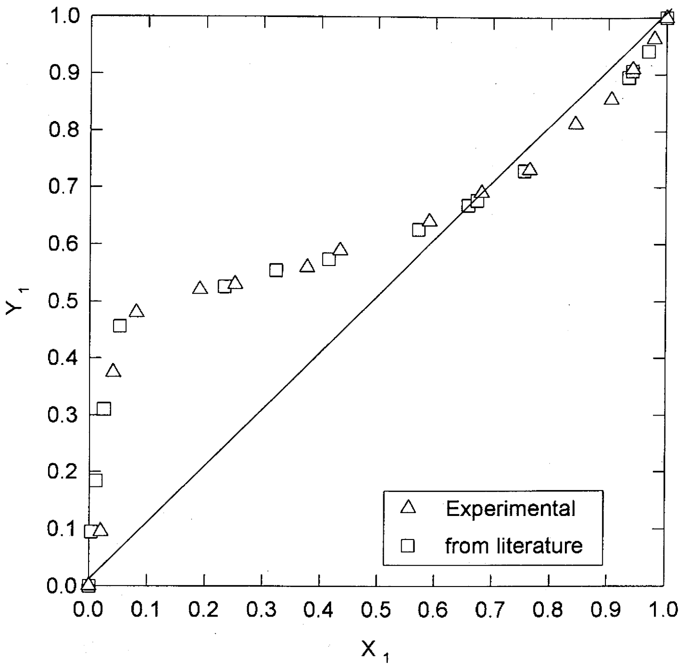

FIG. 4 x_1–y_1 diagram for 2-propanol–water system at 1 atm.

was about 80.0 ± 0.1 mol% 2-propanol, which is considerably different from that of the feed. However, as expected, there was no change in the distillate composition in the six normal stages. Similar results were obtained in the porous carbon plates. These results thus prove the feasibility of using sintered stainless steel plates to separate the 2-propanol–water system with an azeotropic feed composition. However, the porous carbon plates are not suitable porous plates to separate this system. These results can be explained based on the theory developed by Abu Al-Rub and Datta (2, 3). They theorized that the alteration of the VLE of a mixture in a pore is owing to: 1) the close proximity of the solid surface which exerts long-range molecular forces on each liquid, and 2) the curvature of the vapor–liquid interface. While the latter has no significant effect on the VLE of a binary mixture in the porous plates studied due to the large pore size, the former is the main factor in altering the VLE of mixtures in the capillary porous plates studied. The solid–liquid interactions for the case of liquids in porous plates were discussed in more detail based on the molecular approach by Abu Al-Rub and Datta (2). The type and the strength of these interactions depend mainly on the nature of the solid

TABLE 3
Distillation of 2-Propanol–Water System

Mol% 2-propanol in the feed	Mol% 2-propanol in the distillate
a. In Six Porous Sintered Stainless Steel Plates of 13.5 μm Pore Diameter with Distillate Flow Rate of 20 mL/min	
19.00	62.41
24.90	64.20
45.10	69.62
49.09	71.50
56.40	76.03
68.00	80.00
b. In Six Normal Stages with Total Reflux	
19.00	55.00
24.90	56.21
45.10	61.50
49.09	62.39
56.40	64.60
68.00	68.01
c. In Six Porous Carbon Plates of 30 μm Pore Diameter with Distillate Flow Rate of 20 mL/min	
19.00	55.02
24.90	56.29
45.10	62.00
49.09	62.49
56.40	65.10
68.00	68.10

and liquids. The most common interactions are van der Waals interactions which may be divided into three main categories: 1) permanent dipole–dipole, or orientation, interactions, which is the dominant interactions for polar–polar systems; 2) dipole–induced dipole, or induction, interactions, which is dominant if one of the materials is polar while the other is nonpolar; and 3) nonpolar, or dispersion, interactions, which is dominant if both materials are nonpolar.

For liquids in polar porous plates, such as sintered stainless steel plates, the intermolecular interactions of each liquid with the solid will be functions of the molar polarizability of the liquid and the solid (2). Thus, when liquids of close polarity are contacted in polar plates, the VLE will not be significantly altered from the "normal" VLE. However, when there is considerable difference in the polarity of the mixture components, then it is expected that significant alteration in the VLE will be obtained. A measure of the polarizability is the dielectric constant (2, 16–18). For our system, the molar polarizability of 2-propanol at 25°C is 3.93 and the molar polarizability of water at 25°C is 17.56 (19). Thus there is considerable difference in the molar polarizability of 2-propanol and water, which explains the large separation and VLE alteration of the 2-propanol–water mixture.

On the other hand, for nonpolar plates, such as porous carbon plates, the intermolecular interactions between the solid and liquids are mainly those of dispersion forces. These forces are a weak function of the polarity and can be assumed to be the same for all liquids in nonpolar plates (2, 3). Thus, the intermolecular interactions between the components of the 2-propanol–water system will not be affected significantly in porous nonpolar plates, thus no significant alteration in the VLE is expected in these plates. This explains why

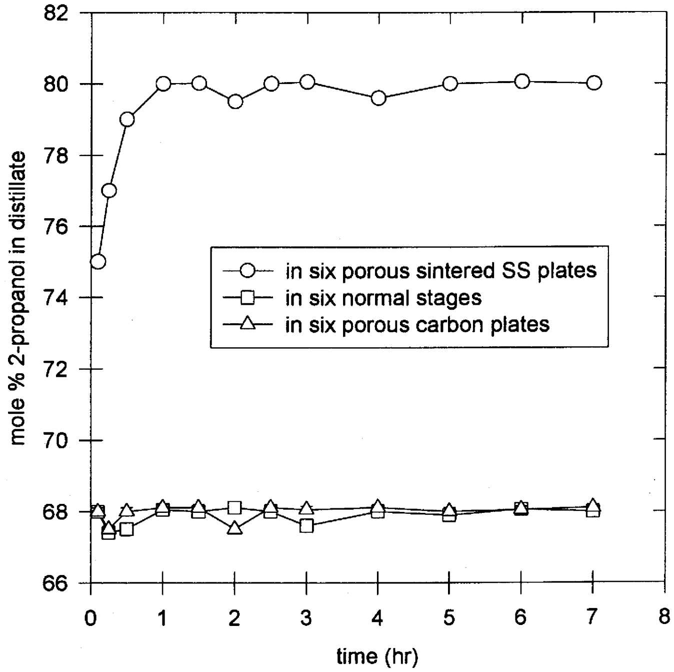

FIG. 5 Continuous distillation of 2-propanol–water system in six stainless steel plates of 13.5 μm pore diameter, in six porous carbon plates, and in six normal stages with a distillate flow rate of 20 mL/min.

the separation results obtained in porous carbon plates were similar to those obtained in normal stages.

Effect of the Number of Stages

It is known that, in general, the separation of mixtures in a distillation process increases with an increase in the number of stages. However, for a feed of azeotropic composition, the number of stages will not enhance separation in a conventional distillation column. The effect of the number of stages, within the limitations of the apparatus used, on the distillate composition for the 2-propanol–water system was studied for the azeotropic feed composition. For the case of normal stages and porous carbon plates, as expected, there was no effect of the number of stages on the distillate composition, i.e., the composition on each tray was similar to the azeotropic composition. For the case of distillation in porous sintered stainless steel plates, the results showed that the distillate composition increased from 77.0 mol% 2-propanol to about 80.0 mol% 2-propanol for a feed of 68.0 mol% 2-propanol when the number of stages was increased from four to six (Fig. 6).

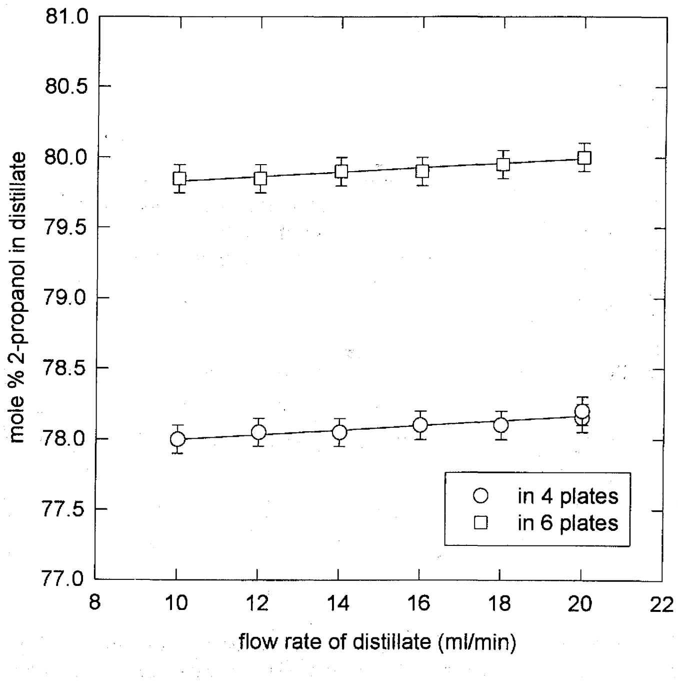

FIG. 6 Effect of distillate flow rate on the distillate composition in stainless steel plates of 13.5 μm pore diameter.

Effect of the Distillate Flow Rate

Distillation of a 2-propanol–water mixture in capillary porous sintered stainless steel plates was conducted with distillate liquid flow rates ranging from 10 to 22 mL/min. The distillate flow rate was controlled by controlling the heating rate of the reboiler. The results are shown in Fig. 6. As can be seen from these results, the effect of the distillate flow rate on the distillate composition is not significant. Although Fig. 6 shows a minor increase in distillate composition with an increase in the flow rate, this increase is not significant and is within the experimental error. Thus, all the experiments in this study were conducted at a flow rate of 20 mL of liquid/min, which is equivalent to a flux of 0.41 mL liquid/cm^2·min through the porous plates.

Pressure Drop across Sintered Stainless Steel Plates

The pressure drop across a porous plate which is completely wetted by the condensed liquid is expected to be small unless there is a heat loss from the column to the surrounding air (8). The pressure drop across the sintered stainless steel plates was measured for different flow rates for the case of six sintered stainless steel plates. The results in Fig. 7 show that the pressure drop

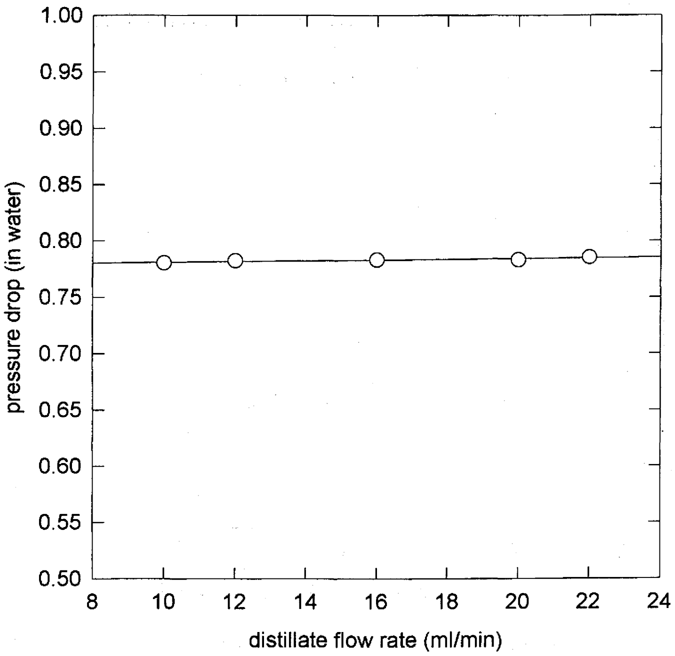

FIG. 7 Pressure drop across porous sintered stainless steel plate vs distillate flow rate.

across the sintered stainless steel plates was about 0.78 in. water. Theoretically, the pressure drop across capillary plates which are completely wetted should be the static pressure of liquid above the plate, which in our case corresponds to less than 1/2 in. water. The difference between the measured and "theoretical" pressure may be attributed to heat losses from the column to the surrounding air and the possible blowing of vapor through larger pores in the plate (8). Moreover, this pressure drop was independent of the distillate flow rate.

Effect of the Feed Composition

Experiments were conducted with different feed compositions, in addition to the azeotropic composition, to study the effect of the feed composition on the "efficiency" of the distillation process. The experiments were conducted in six porous carbon plates, six sintered stainless steel plates, as well as six normal stages. The results of these experiments are given in Table 3. A selec-

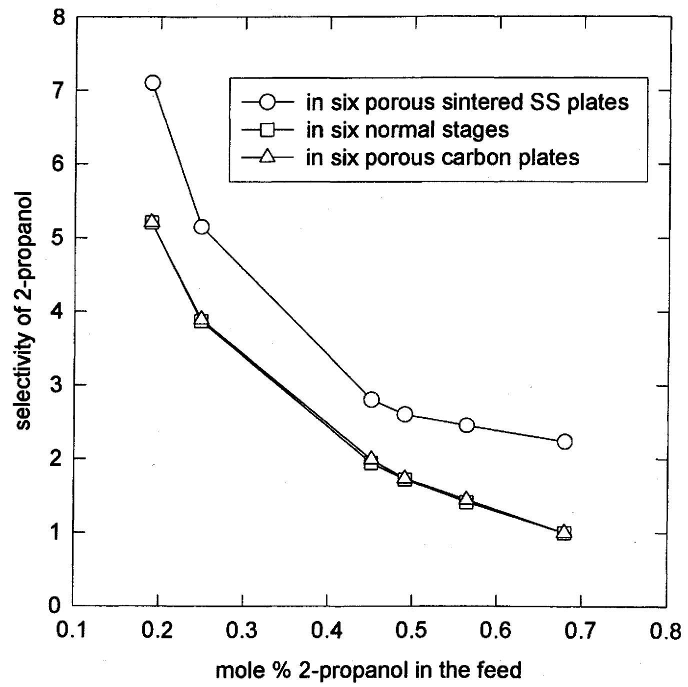

FIG. 8 Selectivity of continuous distillation of 2-propanol–water system as a function of the feed composition.

tivity, S, defined as the ratio of the distillate composition of the two components to that of the feed composition, i.e.,

$$S \equiv \frac{x_{1D}/x_{2D}}{x_{1F}/x_{2F}} \tag{3}$$

may be used as a measure of the efficiency of separation in the continuous separation process (4). The results for steady-state selectivity in distillation are shown in Fig. 8. These results show that the selectivity of the distillation in the three cases was high at low feed concentration, which is also evident from the VLE diagram for the 2-propanol–water system (Fig. 4). Comparison between the results of different plates shows, however, that the selectivity in the sintered stainless steel plates was always greater than that in the porous carbon plates and in the normal stages. The difference is particularly significant at a feed composition around 68.0 mol% 2-propanol, when the selectivity in the porous carbon plates and in the normal stages has an asymptote of $S \rightarrow 1$, while the selectivity in the porous sintered stainless steel plates was always >1.

Further, as can be seen from Table 3(a), a distillate with a composition of 76.03 mol% 2-propanol resulted when the feed composition was 56.4 mol% 2-propanol. This means that separation beyond the normal azeotropic point was achieved even for a feed composition well below the normal azeotropic point. These results show that distillation with sintered stainless steel plates can be used to achieve compositions higher than the azeotropic composition from a low feed composition.

CONCLUSIONS

Distillation of a 2-propanol–water mixture of azeotropic composition was achieved using porous sintered stainless steel plates but not achieved in either porous carbon plates or in normal stages. The selectivity of 2-propanol, which is a measure of separation efficiency, in sintered stainless steel plates was greater than that of either porous carbon plates or normal stages for any feed composition. These results prove the feasibility of using polar capillary porous plates instead of normal plates in a continuous distillation column to alter the VLE of a given system. The main factors affecting the efficiency and the applicability of this process are the polarization of the liquids and the solid. Nonpolar porous plates provide no particular advantage.

ACKNOWLEDGMENT

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NOTATION

p	pressure (kPa; mmHg)
$p_{\mathrm{v}}^{\mathrm{r}}$	vapor pressure over interface of radius of curvature r and in the presence of external fields (kPa; mmHg)
$p_{\mathrm{v},0}^{\infty}$	vapor pressure over a plane interface and in the absence of external fields (kPa; mmHg)
r	radius of curvature; pore radius (d)
R	universal gas constant ($N_{\mathrm{A}}k = 8.3143\ \mathrm{J\cdot mol^{-1}\cdot K^{-1}}$)
T	temperature (K)
$\tilde{V}$	molar volume ($\equiv M/\rho$, $\mathrm{m^3\cdot mol^{-1}}$)
x_i	molar fraction of species i in the liquid phase
y_i	molar fraction of species i in the vapor phase

Greek Letters

σ	surface energy or tension ($\mathrm{mJ\cdot m^{-2}}$ or $\mathrm{mN\cdot m^{-1}}$)
γ	activity coefficient

Subscripts

D	distillate
F	feed
i	species i
l	liquid phase
0	property in the absence of external fields
v	vapor phase

Superscripts

r	property with radius of curvature r
$\sim$	molar thermodynamic property

Abbreviations

C.I.	cast iron
S.S.	stainless steel
VLE	vapor–liquid equilibrium

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