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Vapor–Liquid Equilibrium of Ethanol–Water System in the Presence of Molecular Sieves

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ABSTRACT

Adsorptive distillation is a new process to separate liquid mixtures in a packed distillation column. It depends on using active packing material instead of inert packing material in a packed distillation column. The active packing material can affect the intermolecular forces among the system components and thus alter its vapor–liquid equilibrium (VLE). The VLE of the ethanol–water system at 1 atm was studied using a circulation still in the absence and in the presence of different amounts of 4 Å molecular sieves. The results obtained showed that the VLE of the system was altered in the presence of the molecular sieves, the azeotropic point of the system (at 89.7 mol% ethanol in the normal case) was eliminated and considerable separation was achieved for a mixture of azeotropic composition, and the alteration in the VLE of a given binary mixture is a function of the pore size and the amount of the molecular sieves.

Key Words. VLE; Gibbs energy; Polarizability; Activity coefficient; Ethanol

INTRODUCTION

Although distillation remains the workhorse of separation processes in spite of its large energy consumption, there are cases when it is not the ideal choice, as for example for liquid mixtures of low relative volatility, or when azeotropes are formed. In the latter case the azeotrope may be broken by al-

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tering the column pressure, or by introducing a mass separating agent (MSA), as in azeotropic or extractive distillation. However, this adds substantially to the cost, since additional columns are usually required to remove and recycle the MSA. As a result, alternative processes such as adsorption and pervaporation are considered, and in some cases have replaced azeotropic or extractive distillation, e.g., in ethanol–water separation (8, 9, 11).

An innovative alternative technique was proposed by Yeh et al., (14–16) and later by Abu Al-Rub (1), Abu Al-Rub et al., (2), and Abu Al-Rub and Datta (3–5) who used macroporous stainless steel plates to alter the vapor–liquid equilibrium (VLE) of binary mixtures. They suggested that this was the result of solid–liquid interfacial molecular forces, i.e., dispersion and polar interactions. Abu Al-Rub et al. (2) and Abu Al-Rub and Datta (3, 5) reported some experimental studies involving distillation of different liquid mixtures including azeotropic mixtures by the use of different macroporous plates which altered the VLE of some systems.

Abu Al-Rub (1) proposed another novel technique, called adsorptive distillation, which involves replacement of the inert packing material in packed-bed distillation by an “active” packing material. By active packing material is meant a material which can affect the force field and the intermolecular forces among the system components and thus alters its VLE. Abu Al-Rub (1) and Abu Al-Rub and Datta (unpublished data, 1994) studied the VLE of the ethanol–water system in the presence of 3 Å molecular sieves using a modified Othmer still and found that the VLE for this system was significantly altered and the azeotropic point was shifted. Recently, Abu Al-Rub et al. (6) studied the VLE of ethanol–water mixtures in the presence of molecular sieves using the headspace gas chromatography technique and found that the VLE of the studied system was altered considerably in the presence of 3 and 4 Å molecular sieves.

The objective of this study is to further investigate, theoretically and experimentally, the VLE of the ethanol–water system in the presence of molecular sieves. The VLE of the ethanol–water system will be studied experimentally in the absence and in the presence of different amounts of 4 Å molecular sieves at 760 mmHg. Next, the results obtained will be interpreted thermodynamically.

EXPERIMENTAL APPARATUS AND PROCEDURE

Chemicals

Distilled water and 200 proof ethanol (Midwest Grain Product) were used. The active packing material used in this study was 4 Å molecular sieves with the following specifications: manufacturer, Grace Co.; type, 4 Å; grade, 514;

mesh, 8–12 mesh; effective pore size, 4 Å; base, aluminum silicate; cation, sodium.

Procedure

The still used in this study was an 800-mL circulation still similar to that used by Jaques and Furter (7), Subbaiah (12), and Abu Al-Rub (1) which is a modification of the Othmer still. A detailed description of this still can be found elsewhere (1, 7, 12).

The pressure was measured using another ebulliometer containing pure boiling water. By connecting the two stills in parallel, we can obtain an absolute manometer. The pressure was controlled using a vacuum pump and an air compressor connected to the condenser. The experiment was conducted under a pressure of 760 ± 1 mmHg. Steady state was attained after 30 minutes, where the temperature was measured, using a calibrated thermometer with a resolution of ±0.01°C. Samples of liquid and condensed vapor were withdrawn and analyzed five times using a Perkin-Elmer Auto System Gas Chromatograph with a 6 ft, $^1/_8$ in. Porapak R column under isothermal conditions at 170°C.

To study the VLE in the presence of the active packing material, different experiments were conducted with different amounts of the molecular sieves. The molecular sieves were first introduced into the still and then 200 mL of the solution was added to the still. Then the above-described experimental procedure was followed. “Fresh” molecular sieves were used for each run. The reproducibility of the results was checked by performing up to three replicate experiments to obtain deviation of the measured data due to random error and was found to be within 0.01%.

RESULTS AND DISCUSSION

The experimental VLE data for the ethanol–water system in the absence of molecular sieves at 1 atm are presented in Table 1 and plotted in Fig. 1 as an x–y diagram. The main objective for studying the VLE for this system in the absence of molecular sieves was to check for the practical “feasibility” of using this still to study the effect of having active packing material on the VLE of binary mixtures. This was done by comparing the results obtained using this still with published results for the given system under the same conditions. Comparison of our results with those published in the literature (10), as shown in Fig. 1, indicates good agreement. Moreover, the data were analyzed using the Wilson equation (13), where the Wilson parameters were found to be $\Lambda_{12} = 0.2101$ and $\Lambda_{21} = 0.7902$. The root-mean-square deviation (RMSD) between the calculated and the experimental y_1 values was found to be 0.0077.

TABLE 1
Experimental Data for the Ethanol–Water System in the Absence of Molecular Sieves at 760 mmHg

x_1	y_1	T (°C)
0	0	99.99
0.0250	0.2301	93.10
0.1101	0.4412	86.81
0.2560	0.5549	82.13
0.4420	0.6210	80.55
0.5521	0.6669	79.51
0.6811	0.7401	78.72
0.7611	0.7918	78.43
0.8950	0.8950	78.12
0.9820	0.9788	78.22
1.0	1.0	78.22

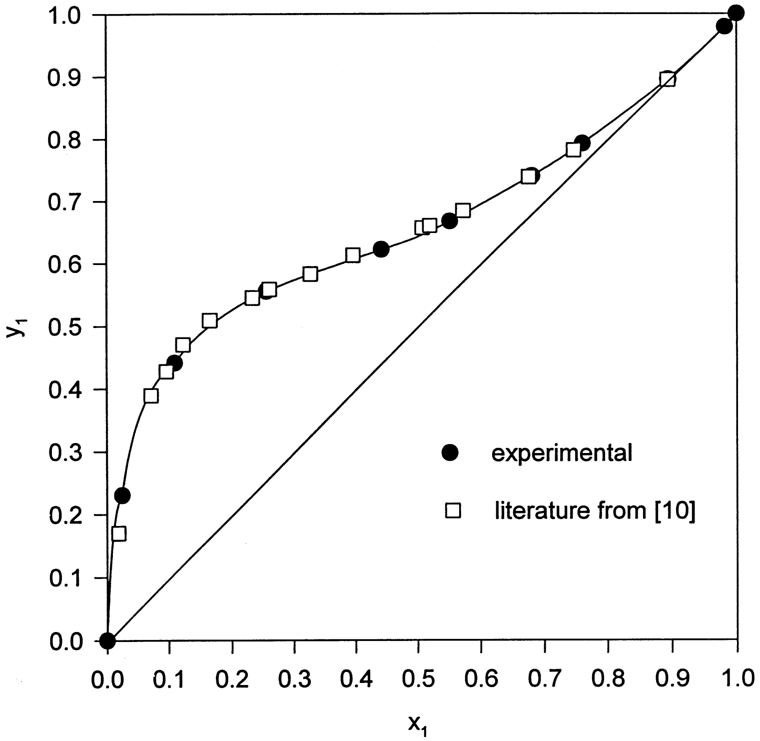

FIG. 1 Vapor–liquid equilibrium of the ethanol–water system at 760 mmHg.

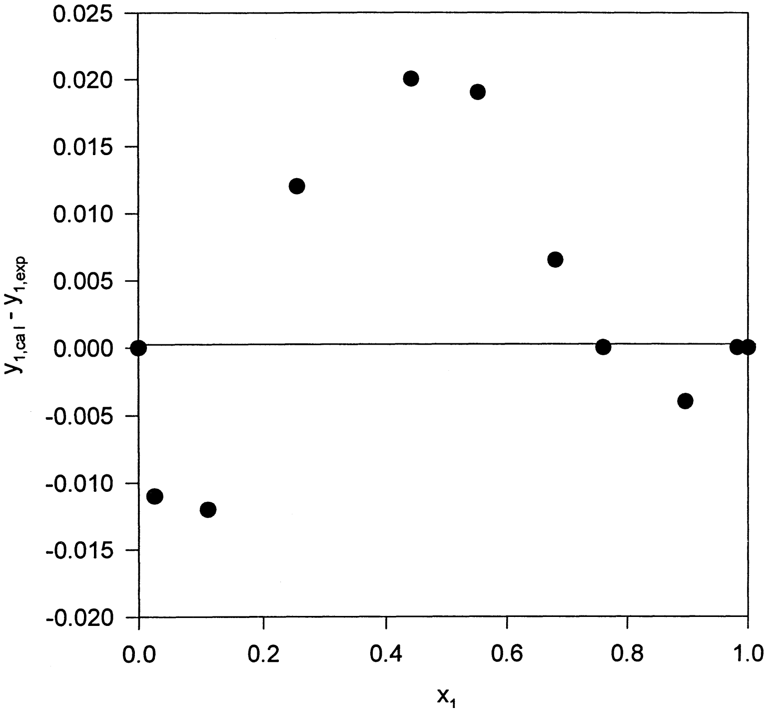

FIG. 2 Ethanol vapor composition variation ($y_{1,\mathrm{cal}} - y_{1,\mathrm{exp}}$) vs x_1.

TABLE 2
Experimental Data for the Ethanol–Water System in the Presence of 300 g of 4 Å Molecular Sieves at 760 mmHg

x_1	y_1	T (°C)
0.1510	0.5201	84.55
0.4110	0.6801	80.42
0.5698	0.7348	79.22
0.6341	0.7722	79.01
0.7011	0.8102	78.61
0.7719	0.8401	78.59
0.8210	0.8851	78.53
0.8950	0.9252	78.46
0.9201	0.9350	78.41
0.9600	0.9658	78.32

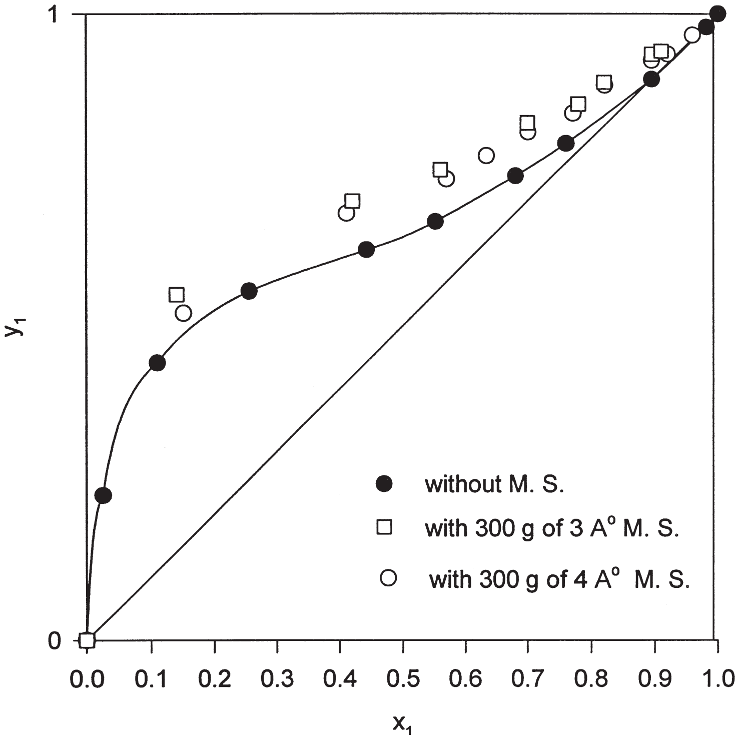

FIG. 3 Vapor–liquid equilibrium of the ethanol–water system with 300 g of molecular sieves at 760 mmHg.

Figure 2 shows a plot of ethanol vapor composition deviation ($y_{1,\text{cal}} - y_{1,\text{exp}}$) calculated using the Wilson equation against the mole fractions of ethanol in the liquid phase. The results obtained are shown to be in a good agreement over the whole concentration range. This proved the practical feasibility of using the above apparatus to study the effect of molecular sieves on the VLE of binary mixtures.

Effect of Molecular Sieves on VLE

The experimental VLE data for the ethanol-water system in the presence of 300 g of 4 Å molecular sieves at 1 atm are presented in Table 2 and plotted in Fig. 3 as the *x*–*y* diagram. As can be seen from the figure, the VLE diagram has been altered and the azeotropic point has been eliminated. A considerable separation was obtained for a feed having the azeotropic composition.

The relative volatility of ethanol with respect to water, defined as

$$\alpha_{12} \equiv \frac{y_1/x_1}{y_2/x_2} \tag{1}$$

is calculated for the cases with and without the molecular sieves, and the results are shown in Fig. 4. The presence of an azeotropic point can be identified using this figure by noting the neighborhood of a point at which the values of the relative volatility are less and greater than unity, or simply the point at which the relative volatility equals unity. As can be seen from Fig. 4, for the case of no molecular sieves, the relative volatility of ethanol is equal to 1 at an ethanol liquid composition of 0.895, which is similar to that reported in the literature (10). However, Fig. 4 shows the absence of an azeotropic point in the presence of molecular sieves, as the relative volatility is always greater than unity.

The alteration of the VLE in the presence of molecular sieves was due to the effect of the force field created by the molecular sieves on the mixture components. The solid–liquid interactions for the case of liquids in capillary porous media are discussed in detail based on a molecular approach by Abu Al-Rub and Datta (4, 5). The type and the strength of these interactions depend mainly on the nature of the solid and the 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 are the dominant interactions for polar–polar systems; 2) dipole–induced dipole, or induction, interactions, which are dominant if one of the materials is polar while the other is nonpolar; and 3) nonpolar, or dispersion, interactions, which

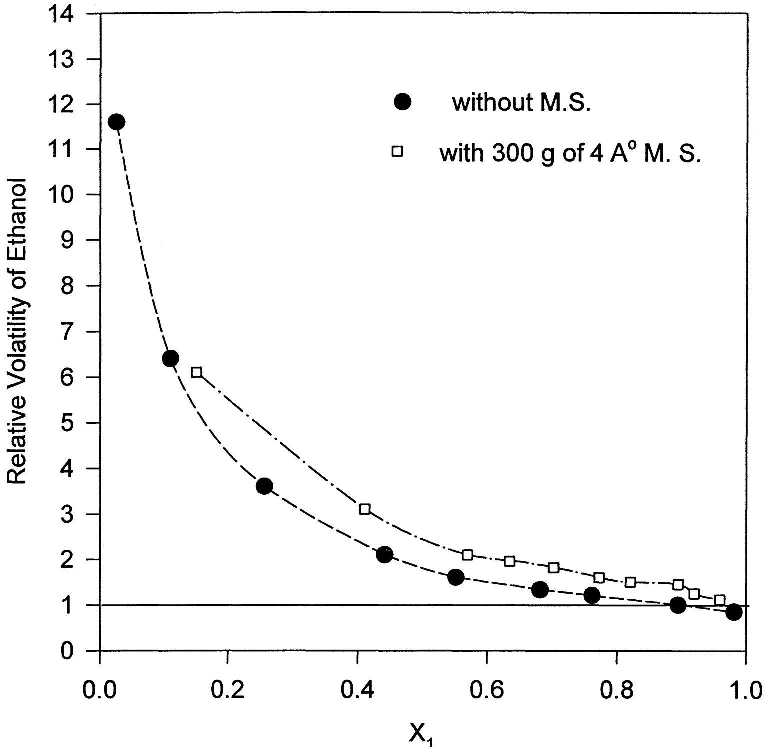

FIG. 4 Relative volatility of the ethanol–water system at 760 mmHg.

are dominant if both materials are nonpolar. Thus, the degree of alteration of the VLE will be a strong function of the polarity of each component as well as of the polarity of the solid.

For the case of VLE in the presence of molecular sieves, the intermolecular interactions between the molecular sieves, which is considered to be polar, and the most polar compound in the liquid phase will be greater than those between the molecular sieves and the component of less polarity. Thus, the composition of the most polar compound in the vapor phase will be less than that in the absence of molecular sieves under the same conditions. One of the measures of the polarity is the dielectric constant which is related to the total molar polarizability. The larger the value of the dielectric constant, the more polar the material will be. For our system, the dielectric constant at 25°C is 78.5 for water and 24.55 for ethanol. Thus, based on what we have said above, the composition of water in the vapor phase in the presence of molecular sieves is expected to be less than that in the absence of molecular sieves.

Abu Al-Rub and Datta (5) developed a theoretical approach to predict the VLE of binary mixtures in porous media using the "normal," i.e., without molecular sieves, VLE. They showed that the ratio of the relative volatility in porous media, α^{r}_{ij}, to the "normal" relative volatility, α^{∞}_{ij}, is given by

$$\ln \frac{\alpha^{r}_{ij}}{\alpha^{\infty}_{ij}} \cong (\kappa_c - \kappa_s)\left(\frac{\tilde{P}^{l}_{i}}{\tilde{V}^{l}_{i}} - \frac{\tilde{P}^{l}_{j}}{\tilde{V}^{l}_{j}}\right) \tag{2}$$

where $\tilde{P}_i$ and $\tilde{V}_i$ are the molar polarization and molar volume of the *i*th component, respectively, and κ_c and κ_s are empirical parameters that characterize the porous media and the binary mixture, respectively. It is evident from the above equation that in order to predict the VLE of a given binary system in a porous media, the empirical parameters κ_c and κ_s should be known; they are not available for our case. Moreover, the above equation is valid only for a uniform "continuous" force field as in the case of porous plates. For the case of molecular sieves, where the VLE is assumed to be within the pores of each "particle," the assumption of a uniform continuous force field is not valid, and therefore a theoretical analysis would be very difficult.

Effect of Pore Size

The effect of pore size on altering the VLE of ethanol–water mixtures was investigated by comparing the experimental results of this study with those reported by Abu Al-Rub and Datta (unpublished data, 1994). They studied the VLE of ethanol–water mixtures in the presence of 300 g of 3 Å molecular sieves. Figure 3 shows the comparison. As can be seen from this figure, the alteration in the VLE of ethanol–water mixtures in the presence of the 3 Å molecular sieves was "greater" than that in the presence of the 4 Å molecular sieves. This may be attributed to two factors: 1) the intermolecular interactions between the solid and the liquid are inversely proportional to the pore diameter (4, 5, 15). Thus, the intermolecular interactions of the liquids with 3 Å molecular sieves will be greater than these with 4 Å. 2) According to the Kelvin equation, the vapor pressure of the pure components in microporous media is given by (4, 5).

$$\ln\left(\frac{p_{\mathrm{v}}^{\mathrm{r}}}{p_{\mathrm{v}}^{\infty}}\right) = -\frac{2\sigma_0 \tilde{V}_{\mathrm{l}}}{rRT} \tag{3}$$

where $p_{\mathrm{v}}^{\mathrm{r}}$ is the vapor pressure of a pure liquid in a porous media of radius r, p_{v}^{∞} is its vapor pressure over a flat interface, σ_0 is the surface tension, and $\tilde{V}_{\mathrm{l}}$ is the molar volume of the pure liquid. As is evident from this equation, the reduction of the vapor pressure of the pure liquids in the presence of 3 Å molecular sieves will be greater than that in the presence of 4 Å molecular sieves, thus the VLE of the binary mixture in 3 Å molecular sieves will be different from that in 4 Å molecular sieves. This is in accordance with the theory of VLE in porous media developed by Abu Al-Rub and Datta (4, 5). They showed (5) that the alteration of the VLE of binary mixtures in porous media is strongly dependent on the geometric characteristics of the media. They showed that the intermolecular interactions between the liquids and the porous media increase with decreasing pore diameter, which results in a “larger” alteration in the VLE of a given system.

Finally, it is worth mentioning that it is well acknowledged that long-range surface forces can be experienced over hundreds of nanometers (4, 5, 14–16), and may lead to changes in the properties of thin liquid layers on solids when compared to those in the bulk liquid, in particular for polar liquids and at lower temperatures. These include changes in density, viscosity, freezing point, latent heat of vaporization, critical temperature, relative permittivity, and the thermal expansion coefficient of liquids in small pores (4).

Effect of the Molecular Sieves’ Weight

The effect of the weight of molecular sieves on the separation efficiency of the ethanol–water system was studied for two liquid compositions, 40.0 and 89.5 mol% ethanol, and with different amounts of the molecular sieves. The results, shown in Fig. 5, show that the optimum amount of molecular sieves for the studied liquid volume is about 270 g. No further improvement on separation can be achieved by increasing the weight of molecular sieves beyond 270 g. This can be explained as follows: When the amount of molecular sieves is less than the optimum amount, “free” liquid regions exist that will reduce the separation efficiency. By free liquid region we mean a region where the equilibrium between the vapor and the liquid does not occur in the pores of the molecular sieves. This is because the height of the bed of the molecular sieves

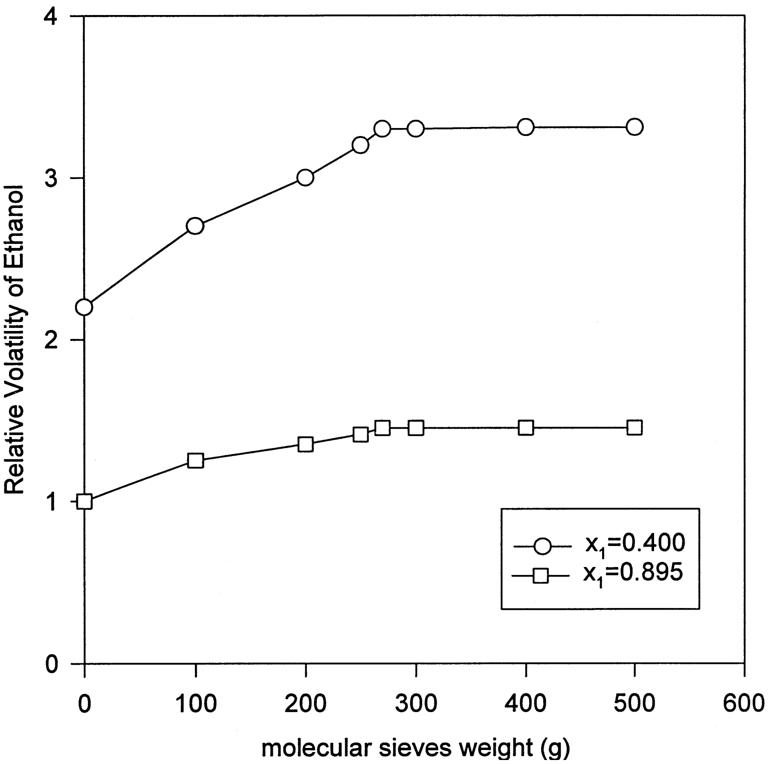

FIG. 5 Effect of molecular sieves' weight on the relative volatility of ethanol at 760 mmHg.

is not enough to prevent vapor molecules from escaping without being contacted with the molecular sieves. At the optimum weight we reach the bed height were no vapors can escape without contacting the molecular sieves. Thus, increasing the amount of molecular sieves beyond the optimum amount will introduce no enhancement of the separation efficiency.

Thermodynamic Interpretation

To interpret the results obtained thermodynamically, we will use the fact that the excess Gibbs energy is one way of expressing the nonideality of a system. Thus it is related to the interactions of the system components, where

$$\Delta G^{\mathrm{E}} \equiv \Delta G - \Delta G^{\mathrm{Id}} \tag{4}$$

where ΔG^{Id} is the Gibbs energy for an ideal system. Because G is an extensive property, G of a system is given by the sum of the free energies of the constituent phases of the system. For the case of liquids in the presence of external force fields, we have interactions due to the solution components and due to the interaction of each component with the solid phase, i.e.,

$$\Delta G^{\mathrm{E}} = \sum_{\substack{j\,=\,i\\ j\,\neq\, i}}^{n}\sum_{i=1}^{n}\Delta G_{ij}^{\mathrm{E}} + \sum_{i=1}^{n}\Delta G_{i\mathrm{s}}^{\mathrm{E}} \tag{5}$$

For binary solutions this equation reduces to

$$\Delta G^{\mathrm{E}} = \Delta G_{12}^{\mathrm{E}} + \Delta G_{1\mathrm{s}}^{\mathrm{E}} + \Delta G_{2\mathrm{s}}^{\mathrm{E}} \tag{6}$$

Each of the above contributions could be individually a positive or a negative deviation from ideality, with the sum being positive or negative.

Moreover, the interactions due to the solid, as discussed previously, are composed of several molecular interactions which can be grouped into two main groups: 1) dispersion interactions (D), and 2) polar interactions (dipole–dipole and dipole–induced dipole interactions, P). It is then possible to write the second term of Eq. (3) as the sum of these two interactions, i.e.,

$$\sum_{i=1}^{n}\Delta G_{i\mathrm{s}}^{\mathrm{E}} = \sum_{i=1}^{n}\Delta G_{i\mathrm{sD}}^{\mathrm{E}} + \sum_{i=1}^{n}\Delta G_{i\mathrm{sP}}^{\mathrm{E}} \tag{7}$$

However, it is difficult to evaluate each term in the solid contribution. For the sake of studying the overall effect of the solid on the system, we may group all the solid interactions into one unit; call it $\Delta G_{\mathrm{s}}^{\mathrm{E}}$. Thus, for binary mixtures

$$\Delta G^{\mathrm{E}} = \Delta G_{12}^{\mathrm{E}} + \Delta G_{\mathrm{s}}^{\mathrm{E}} \tag{8}$$

The excess Gibbs energy is related, in general, to the activity coefficient by the relation

$$\overline{G}_{i}^{\mathrm{E}} = RT\ln\gamma_i \tag{9}$$

and for the entire solution

$$\widetilde{G}^{\mathrm{E}} = \sum_{i=1}^{n} x_i\widetilde{G}_{i}^{\mathrm{E}} \tag{10}$$

For the case of a liquid mixture in the presence of an active packing material:

$$\Delta G_{i}^{\mathrm{E}} = \Delta G_{i\mathrm{l}}^{\mathrm{E}} + \Delta G_{i\mathrm{s}}^{\mathrm{E}} \tag{11}$$

and

$$\ln\Delta\overline{G}_{i}^{\mathrm{E}} = \ln(\Delta\overline{G}_{i\mathrm{l}}^{\mathrm{E}} + \Delta\overline{G}_{i\mathrm{s}}^{\mathrm{E}}) \tag{12}$$

where

$$\Delta G_{i\mathrm{l}}^{\mathrm{E}} \equiv \sum_{\substack{j\,=\,i\\ j\,\neq\, i}}^{n}\sum_{i=1}^{n}\Delta G_{ij}^{\mathrm{E}} \tag{13}$$

Using Eq. (10) in Eq. (13) results in

$$\ln\gamma_i = \ln\gamma_{i\mathrm{l}} + \ln\gamma_{i\mathrm{s}} \tag{14}$$

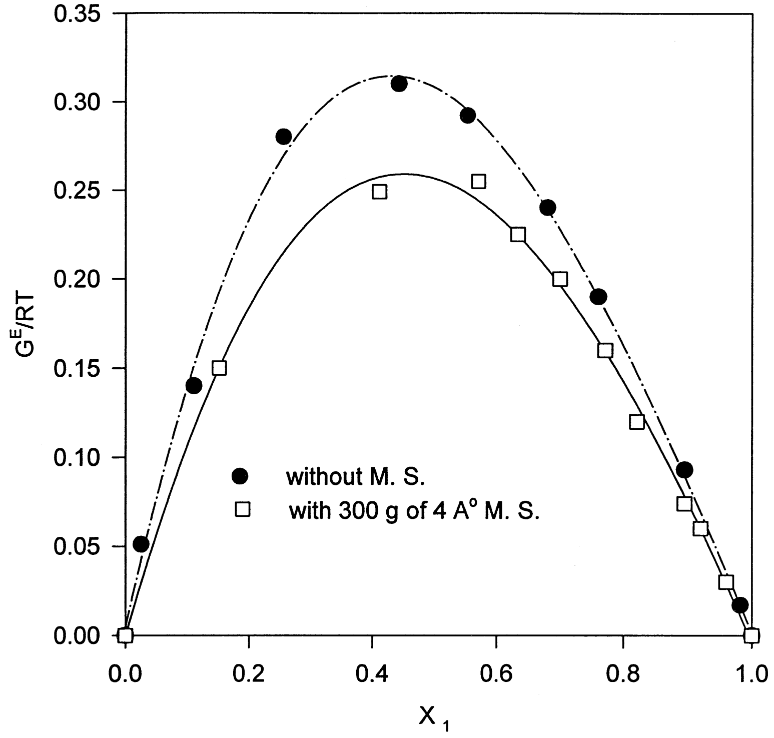

FIG. 6 Molar excess Gibbs energy of the ethanol–water system at 760 mmHg.

or

$$\gamma_i = \gamma_{i1}\gamma_{is} \tag{15}$$

where γ_{is} is the activity coefficient due to the presence of external force fields created by the solid on the liquid. Thus, from an experimental measurement of the activity coefficients in the absence and in the presence of external fields, γ_{is} can be obtained. Note that γ_{i1} and γ_{is} may individually be less than or greater than unity, depending upon positive (unlike molecules are attracted less than like molecules) or negative (unlike molecules are attracted more than like molecules) deviations from ideality.

Figure 6 shows a plot of the molar excess Gibbs energy of the ethanol–water system against mole fractions of ethanol in the liquid phase. As can be seen from this figure, there is a positive deviation from ideality ($\widetilde{G}^{E}/RT > 0$) for this system in the absence and in the presence of molecular sieves. However, this deviation is less in the presence of molecular sieves. Thus, it may be said that the molecular sieves altered the VLE of this system by reducing the deviation from ideality.

CONCLUSIONS

Experimental results obtained for the VLE of the ethanol–water system at 1 atm in the presence of different amounts of 4 Å molecular sieves showed considerable change from those without the molecular sieves. The azeotropic point for this system was eliminated, and a separation of the azeotropic mixture was achieved. The alteration of the VLE is a result of the external force field exerted by the molecular sieves on the mixture's components. These results prove the feasibility of using active packing materials to alter the VLE of binary mixtures.

NOTATION

G	Gibbs energy (J)
p	pressure (mmHg)
$\bar{P}_i$	molar polarization of component i ($m^3 \cdot mol^{-1}$)
n	number of components
r	pore radius (m)
R	universal gas constant = 8.3143 J/mol·K
T	temperature (K)
V	volume (m^3)
x	mole fraction in the liquid phase
y	mole fraction in the vapor phase

Greek Letters

α	relative volatility
γ	activity coefficient
κ_c, κ_s	empirical parameters
Λ_{21}, Λ_{12}	Wilson parameters
σ_0	surface tension of pure liquids ($mN \cdot m^{-1}$)

Subscripts

D	dispersion interactions
i	species i
l	liquid phase
P	polar interactions
s	solid phase
v	vapor phase

Superscripts

E	excess thermodynamic property in the presence of external force fields

id	property at ideal condition
—	partial molar thermodynamic property
∼	molar thermodynamic property
∞	property with flat interface

Abbreviations

MSA	mass separating agent
RMSD	root-mean-square deviation
VLE	vapor–liquid equilibrium

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