Density and relative permittivity of (pyridine + 2-methoxyethanol, 2-ethoxyethanol, 2-propoxyethanol, and 2-butoxyethanol) binary mixtures at 298.15 K

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(Received 31 December 2013; final version received 24 January 2014)

The densities and relative permittivities of binary mixtures of pyridine with 2-methoxyethanol, 2-ethoxyethanol, 2-propoxyethanol and 2-butoxyethanol have been measured as a function of composition, at $T = 298.15 \text{ K}$. From the experimental data the excess molar volume ($V^E$) and the deviation in the relative permittivity ($\Delta \varepsilon$) from a mole fraction average have been calculated. The results are discussed in terms of intermolecular interactions and structure of studied binary mixtures.

Keywords: pyridine; alkoxyethanols; density; relative permittivity; intermolecular interaction

1. Introduction

In our previous papers were analysed intermolecular interactions in aliphatic amines with 2-methoxyethanol (ME), 2-ethoxyethanol (EE), 2-propoxyethanol (PE) and 2-butoxyethanol (BE) binary mixtures by measuring their density, viscosity, relative permittivity and $^1\text{H}$-NMR spectra at different temperatures [1–6]. All the results presented in these papers and literature information about the internal structure of amines and alkoxyethanols suggest that the addition of pure amine to alkoxyethanol (or contrariwise) in the studied mixtures would disrupt the self-associated structure in alkoxyethanol causing the appearance in the solutions of free molecules of both the components of the mixed solvent. The free amine, 2-ME, 2-EE (EE), 2-PE and 2-BE molecules may interact by dipole–dipole forces and/or intermolecular hydrogen bonds forming the mixed intermolecular complexes. It results in toughening of the internal structure of the mixed solvents.

In a continuing effort to collect other physicochemical and thermodynamic quantities for the mixtures containing amine and alkoxyethanol and in order to throw more light on hetero-interactions in \{pyridine (PY) + ME, PY + EE, PY + PE and PY + BE\} systems we report in this article densities and relative permittivities, at $T = 298.15 \text{ K}$, for these binary mixtures.

From the experimental results excess molar volume ($V^E$) and deviation in relative permittivity ($\Delta \varepsilon$) from a mole fraction average have been calculated. These structural parameters are very sensitive to the interactions between solute and solvent.

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2. Experiment

2.1. Materials

The following materials with mole fraction purity as stated were used: 2-ME (GC > 0.99 mole fraction), 2-EE (GC > 0.98 mole fraction), 2-PE (GC > 0.99 mole fraction) from Merck–Schuchard (Poznań, Poland) and 2-BE (GC ≥ 0.99 mole fraction) from Fluka (Poznań, Poland) were used. All alkoxyethanols were further purified by the methods described by us previously [1–6]. Additionally pyridine (GC ≥ 0.995 mole fraction) was from Riedel de Haën (Hanover, Germany) and used without further purification. The mixtures were prepared using a Sartorius balance. Conversion to molar quantities was based on the relative atomic mass table of 1985, issued by IUPAC in 1986. The maximum estimated error in the mole fractions is ±1·10^{-4}. Liquids were stored in dry-box over phosphorus pentoxide and degassed by ultrasound just before the experiment. Experimental densities and relative permittivities for the pure solvents, at T = 298.15 K, are compared with the values available in the literature and listed in Table 1. The differences between the measured and literature values can be ascribed to different measurement methods used and to the different purification procedures employed by other authors [7,8].

2.2. Measurements

Solvent densities were measured with a bicapillary-type Lipkin pycnometer, with a capacity of approximately 90 cm³. The maximum error in the density measurements was 2 × 10^{-5} g·cm^{-3}.

The relative permittivity measurements were carried out at 3 MHz, using a bridge of the type OH 301 (made in Radelcis, Hungary). The thermostatic stainless steel measuring cell was of CIII (1 < ε < 60) type. The cell was calibrated with standard pure liquids, such as acetone, butan-1-ol, N,N-dimethylformamide, dichloromethane and water + dioxane binary mixtures (double distilled, deionised and degassed water with a specific conductance of 1 × 10^{-9} S·m^{-1} was used for these mixtures). All these solvents were of spectrograde quality or higher. The relative permittivities for the standards were taken from the literature [9]. The accuracy in the relative permittivity measurements was ±0.02.

| Solvent | ρ (g cm⁻³) | ε   |
|---------|------------|-----|
| PY      | 0.97815    | 12.48 |
|         | 0.978138 [8] | 12.1 [9,10] |
|         | 0.97824 [9] | 12.40 [11,12] |
| ME      | 0.96029    | 16.96 |
|         | 0.960288 [14] | 16.94 [15] |
| EE      | 0.92515    | 14.03 |
|         | 0.92572 [17] | 13.219 [19] |
| PE      | 0.90819    | 11.50 |
|         | 0.908223 [21] | 13.38 [20] |
| BE      | 0.89629    | 9.35  |
|         | 0.89625 [24] | 9.350 [26] |
|         | 0.8958 [25] | 9.43 [27] |
In the all volumetric and dielectric property measurements a Haake model DC-30 thermostat was used at a constant digital temperature control of ±0.01 K.

3. Results and discussion

The experimental densities ($\rho$) and relative permittivities ($\varepsilon$) obtained from the measurements of the pure solvents and for the binary mixtures are summarised in Table S1.

The variations of the density and relative permittivity with binary composition were studied by using the following equations [28]:

$$\rho(x) = \sum_{j=0}^{5} a_j \cdot x_j^1$$  (1)

and

$$\ln(x) = \sum_{j=0}^{6} \beta_j \cdot x_j^1$$  (2)

which could be fitted to the experimental data at each mixture using a least-squares method. The coefficients $a_j$ and $\beta_j$ of this fitting procedure are listed in Tables S2–S3, along with the standard deviations $\sigma(\rho)$ and $\sigma(\varepsilon)$ for each binary mixture.

The goodness-of-fit of this procedure is ascertained by a mean deviation $\Delta\rho = \pm 1.2 \times 10^{-5}$ g·cm$^{-3}$ for (PY + ME), $\Delta\rho = \pm 0.9 \times 10^{-5}$ g·cm$^{-3}$ for (PY + EE), $\Delta\rho = \pm 0.8 \times 10^{-5}$ g·cm$^{-3}$ for (PY + PE), and $\Delta\rho = \pm 1.0 \times 10^{-5}$ g·cm$^{-3}$ for (PY + BE) binary liquid mixtures.

The goodness-of-fit of this procedure is ascertained by a mean deviation $\Delta\varepsilon = \pm 2.0 \times 10^{-3}$ for (PY + ME), $\Delta\varepsilon = \pm 1.3 \times 10^{-3}$ for (PY + EE), $\Delta\varepsilon = \pm 0.7 \times 10^{-3}$ (PY + PE), and $\Delta\varepsilon = \pm 0.7 \times 10^{-3}$ for (PY + BE) binary liquid mixtures.

From these data the values of the excesses molar volume ($V_E$) and deviations of relative permittivity ($\Delta\varepsilon$) were calculated by using the following relations:

$$V_E = x_1 \cdot M_1 (\rho^{-1} - \rho_1^{-1}) + x_2 \cdot M_2 (\rho^{-1} - \rho_2^{-1})$$  (3)

$$\Delta\varepsilon = \varepsilon - (x_1 \cdot \varepsilon_1 + x_2 \cdot \varepsilon_2)$$  (4)

where $M$ is the molar mass; subscripts 1 and 2 stand for pure components, pyridine and alkoxyethanol, respectively.

The values of $V_E$ and $\Delta\varepsilon$ calculated by using Equations (3) and (4) are included in Table S1.

For each mixture the values $V_E$ and $\Delta\varepsilon$ were fitted by a Redlich–Kister polynomial regression of the type:
\[ V^E \text{ (cm}^3 \cdot \text{mol}^{-1}) = x_1 \cdot (1 - x_1) \sum_{j=0}^{5} a_j \cdot (2x_1 - 1)^j \]

and

\[ \Delta \varepsilon = x_1 \cdot (1 - x_1) \sum_{j=0}^{5} b_j \cdot (2x_1 - 1)^j \] (5)

The parameters \( a_j \) and \( b_j \) in Equation (5) were evaluated by the least-squares method. The values of these parameters with standard deviations \( \sigma(V^E) \) and \( \sigma(\Delta \varepsilon) \) are summarised in Tables S4 and S5.

The graphical variations of \( V^E \) and \( \Delta \varepsilon \) values as a function of pyridine mole fraction \( (x_1) \) are shown in Figures 1 and 2.

In general, the values of \( V^E \) and \( \Delta \varepsilon \) depend on several contributions arising from literature [29–36]:

1. The physicochemical properties of pure components of binary mixtures such as their polarity, electron-donor and proton-donor properties (acid-base properties)
2. The internal structure of pure components of the studied mixture
3. The type of intermolecular interactions (physical or chemical forces) between the components of a mixed solvent. The physical contributions comprised of dispersive forces or weak dipole–dipole interactions lead to positive values of \( V^E \) and negative values of \( \Delta \varepsilon \) while specific interactions such as formation of hydrogen bonds lead to negative values of \( V^E \) and positive values of \( \Delta \varepsilon \).
bonds, charge transfer complexes and other complex forming interactions including strong dipole–dipole interactions between the component molecules in the mixture lead to the negative values of $V_E$ and $\Delta \varepsilon$.

Another effect, which should give a negative contribution to the excess volume, is the difference in molecular size between the components in the mixture (this is a geometrical effect). As these differences increase, the more negative should be the contribution to $V_E$.

The excess molar volumes for all presented binary systems are negative over the entire composition range with the minimum of the curves located between 0.45 and 0.50 mole fraction of pyridine as can be seen from Figure 1. The highest negative value is noticed in the mixture with 2-ME and decreases in the sequence:

$$V_{\text{ME}}^{\text{min}} > V_{\text{EE}}^{\text{min}} > V_{\text{PE}}^{\text{min}} > V_{\text{BE}}^{\text{min}}$$

In the same sequence we observe the change of acidic properties ($pK_a$) of the pure studied alkoxyethanols [37]:

$$pK_a(\text{ME}) = 14.8 < pK_a(\text{EE}) = 15.1 < pK_a(\text{PE}) = 15.2 < pK_a(\text{BE}) = 15.3$$

Pyridine is a typical aprotic solvent. The nitrogen atom of the pyridine ring has one lone pair of electrons determining its basic nature (pyridine behaves like a typical tertiary aliphatic amine). Because this pair is not relocated, it may be a proton acceptor, as well as nitrogen atoms tertiary amines. The $pK_a$ for the conjugate acid $\text{PYH}^+$ is 5.21. Pyridine is a strong nucleophile (PY donor number is equal to 33.1) and therefore is easily attacked by
electrophilic agents [38–40]. Alkoxyethanols are very interesting class of solvents, due to the presence of the oxy and hydroxyl groups in the same molecule, which allow self-association via intra- and intermolecular hydrogen bonds. The formation of intramolecular hydrogen bonds is more favourable when the molecules are in the gauche conformations. For these molecules five-, six-, seven- and eight-membered rings, of quite different properties, are formed [41–44].

Taking into account these structural informations and our previous studies [1–6] we can suggest that in the first time a disrupt of the self-associated structure of alkoxyethanols can be observed after the addition of pure pyridine to alkoxyethanols (or contrariwise). The free molecules of alkoxyethanol appear in the solution. The negative values of $\nu^E$ are a consequence of some specific interactions (such as hydrogen bond) between PY, ME, EE, BE and BE free molecules of and the formation of the mixed intermolecular complexes. In the pyridine systems with the studied alkoxyethanols the electron-donating ability of pyridine is very strong bearing in mind free lone electron pair of electronegative nitrogen atom and cross-associated OH–N bonds from complexes in all cases of these systems. Pyridine has six π-electrons at set distance that are delocalised over the ring. The OH–π interaction between alkoxyethanols and pyridine is much weaker than OH–N interaction. Also, because of their planar conformation, molecules of pyridine are very suitable for geometrical fitting of other molecules giving various compact structures.

Additional information about the intermolecular interactions and internal structure of the studied binary mixtures are available from the deviations of relative permittivity ($\Delta \varepsilon$). The deviations in the relative permittivity, presented in Figure 2, are negative over the entire composition range for all the studied systems, with the minimum of the curves located between 0.45 and 0.50 mole fraction of pyridine.

The literature review shows that the study of this extrathermodynamic parameter ($\Delta \varepsilon$) for binary liquid systems represents a unique tool for investigating the formation of intermolecular complexes and provides a valuable aid for determining their stoichiometry and conformation [10–27,36].

The negative values of $\Delta \varepsilon$ confirm that PY and alkoxyethanol molecules are apt to coalesce and form hetero-molecular structures by breaking of the self-associated structures of alkoxyethanols. Additionally, the negative values confirm the formation of H-bonded complexes with decrease in effective number of parallel-aligned dipoles.

It is observed that the highest negative values of $\Delta \varepsilon$ are noticed in the mixture with 2-ME and $\Delta \varepsilon$ values are more positive as an alkyl chain in alkoxyethanol increases:

$$\Delta \varepsilon_{\text{min}}^{\text{ME}} > \Delta \varepsilon_{\text{min}}^{\text{EE}} > \Delta \varepsilon_{\text{min}}^{\text{PE}} > \Delta \varepsilon_{\text{min}}^{\text{BE}}$$

which is evidenced on the strongest interactions between PY and ME molecules. This may be due to the highest values of acidity ($pK_a$) and polarity (see Dimroth and Reichardt solvatochromic parameter of polarity $E^N$ of the pure studied alkoxyethanols [45]) of 2-ME molecules. The highest value of $E^N_T$ is noticed for 2-ME and decreases in the sequence:

$$E^N_T(\text{ME}) = 0.657 \text{ kcal} \cdot \text{mol}^{-1} > E^N_T(\text{EE}) = 0.627 \text{ kcal} \cdot \text{mol}^{-1}$$
$$> E^N_T(\text{PE}) = 0.615 \text{ kcal} \cdot \text{mol}^{-1} > E^N_T(\text{ME}) = 0.596 \text{ kcal} \cdot \text{mol}^{-1}$$

Obtained results seem to indicate that the stable intermolecular complexes of the PY–ME, PY–EE, PY–PE and PY–BE types are respectively formed in the studied binary mixtures. The energy stability of these complexes must be differentiated as it is indicated by plots of changes of $\nu^E$ and $\Delta \varepsilon$ values. The highest energy stabilisation has been noticed
in the complex PY with 2-ME, and it decreases when an alkyl chain of alkoxyethanol in intermolecular complex increases as well as polarity and acidity of alkoxyethanol decreases.

4. Summary and conclusions

Densities and relative permittivities, at $T = 298.15$ K, in the binary mixtures of pyridine with 2-ME or 2-EE or 2-PE and 2-BE have been measured as a function of composition mixed solvent.

From the experimental data excess molar volumes $V^E$ and the deviations in the relative permittivity ($\Delta \varepsilon$) from a mole fraction average have been calculated. All results can be explained in terms of the changes of molecular shapes and intermolecular interactions. Probably, in the studied mixtures, the addition of pure PY to the studied alkoxyethanols (or contrariwise) would disrupt the self-associated structure in alkoxyethanols. The free molecules of alkoxyethanol appear in the solution. The free PY, ME, EE, PE and BE molecules may interact by dipole–dipole forces and/or intermolecular hydrogen bonds forming intermolecular complexes of the type PY–ME, PY–EE, PY–PE and PY–BE. It results in reinforcement of the internal structure of mixed solvents.

Supplemental data

Supplemental data for this article can be accessed [here](#).

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